



United States  
Environmental Protection  
Agency

# PROPOSED UPDATE IIIB

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



**PROPOSED UPDATE IIIB**  
**TEST METHODS FOR EVALUATING SOLID WASTE**  
**PHYSICAL/CHEMICAL METHODS**  
**(SW-846) THIRD EDITION**

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

**Contents:**

1. **Cover sheet.** (What you are currently reading)
2. **Instructions.** This section explains how Proposed Update IIIB 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. **Proposed Update IIIB Table of Contents.** The Table of Contents (dated August 2002) lists all of the methods (as updated by Third Edition Final Updates I, II, IIA, IIB, III, and IIIA and Proposed Update IIIB) in the order of appearance in the manual. It does not reflect Draft Update IVA or B methods because the Agency plans to publish Final Update IIIB before the final versions of the Draft Update IVA or B methods.
5. **Revised Chapter Five and revised Methods 9010C, 9012B, 9060A, and 9070A.**
6. **Revised Chapter Six and revised Methods 9045D and 9095B.**
7. **Revised Chapter Seven**
8. **Revised Chapter Eight and revised 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, and III, IIIA, and Draft Updates IVA and IVB. (Currently, the methods and chapters of Final Update IIIA and Draft Update IVB are not available from GPO, but are available on-line at [www.epa.gov/SW-846/](http://www.epa.gov/SW-846/) or on paper from NTIS, (800) 553-6847.) This package contains Proposed Update IIIB.

These instructions describe how to get your basic manual up-to-date and what to do with your Proposed Update IIIB package. A number of SW-846 update packages have been released to the public since the original Third Edition was released. The dates and labels on these packages can be confusing. 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 defined as individuals that have received copies of the Third Edition and other SW-846 updates (including proposed updates) in the past and have just received only this Proposed Update IIIB package in the mail.

**New subscribers** are defined as individuals who have recently (6-8 weeks) placed an order with the GPO and have 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 Proposed Update IIIB.

## **INCORPORATING PROPOSED UPDATE IIIB IN YOUR MANUAL**

Proposed Update IIIB is printed on buff colored paper and has the date of "August 2002" in the lower right hand corner of each page. This package contains revised material proposed for addition to SW-846. Because it is a proposed update and not yet final, its proposed methods cannot yet be used for compliance with required uses of SW-846 methods in the RCRA regulations. 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 may 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 should augment the manual with other binders of your choice. The instructions below can be generally 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 your Proposed Update IIIB (those who obtained their manual from other sources, may also find this information useful in putting it together):

- Place the original Third Edition of SW-846 (September 1986) in the properly labeled four 3-ring notebooks according to the instructions in Final Update III.
- Incorporate Final Update I (July 1992) into the manual according to the instructions in Final Update III.
- Incorporate Final Updates II (September 1994) and IIA (August 1993) into the manual according to the instructions in Final Update III.
- Incorporate Final Update IIB (January 1995) into the manual according to the instructions in Final Update III.
- Incorporate Final Update III (December 1996) into the manual according to its instructions.
- 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.

Finally, incorporate Proposed Update IIIB into the manual without removing any white pages, or keep the colored proposed update in a separate binder of your choice.

**PREVIOUS GPO SUBSCRIBERS** - If you are a previous subscriber, it is important to establish exactly what is currently contained in your manual before addressing Proposed Update IIIB. If your manual is properly updated, 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 any other dates.

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. You may keep proposed or draft update pages in the manual if you wish, but those methods are not promulgated final methods and thus cannot be used for required uses of SW-846 methods in the RCRA regulations.

Finally, incorporate Proposed Update IIIB into the manual without removing any white pages, or keep the colored proposed update in a separate binder of your choice.

## UPDATE HISTORY OF SW-846

(Finalized updates are printed in bold and underlined.)

Package	Date	Paper Color	Status of Package
<b><u>Third Edition</u></b>	September 1986	White	<b>Finalized (Promulgated)</b>
Proposed Update I	December 1987	Green	Obsolete
Final Update I	November 1990	White	Obsolete! Never formally finalized.
Proposed Update II	November 1990	Blue	Obsolete! Never formally proposed.
<b><u>Final Update I</u></b>	July 1992	White	<b>Finalized (Promulgated)</b>
Proposed Update II	November 1992	Yellow	Obsolete
Proposed Update IIA	October 1992	White	Obsolete
<b><u>Final Update IIA</u></b> (Method 4010, included with Final Update II.)	August 1993	White	<b>Finalized (Promulgated)</b>
<b><u>Final Update II</u></b>	September 1994	White	<b>Finalized (Promulgated)</b>
<b><u>Final Update IIB</u></b>	January 1995	White	<b>Finalized (Promulgated)</b>
Proposed Update III	January 1995	Pink	Proposed
<b><u>Final Update III</u></b>	December 1996	White	<b>Finalized (Promulgated)</b>
Draft Update IVA	January 1998	Salmon	Draft
<b><u>Final Update IIIA*</u></b>	April 1998	White	<b>Finalized (Promulgated)</b>
Draft Update IVB	Nov. 2000, may be later if revised again and added to web site	Only available at <a href="http://www.epa.gov/SW-846">www.epa.gov /SW-846</a>	Draft
Proposed Update IIIB	August 2002	Buff	Proposed

\*Contains revised Table of Contents, revised Chapter Five, and revised Methods 9070 and 9071B.

## **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](mailto: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 METHODS FOUND IN:

FINAL UPDATES I, II, IIA, IIB, III, AND IIIA  
DRAFT UPDATES IVA AND IVB

OTHER DRAFT METHODS AT THE OSW METHODS WEB SITE AS OF OCTOBER 2002  
AND PROPOSED UPDATE IIIB

REVISED OCTOBER 2002

## HOW TO USE THIS DOCUMENT

This document provides historical status information on 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 current status of published SW-846 methods. Methods in this status table are listed sequentially by method number. Use the "Status Table for SW-846 Chapter Text and Other Documents" as a reference guide to identify the historical and current status of published chapters and other SW-846 documents (e.g., the Disclaimer).

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 METH | STATUS TABLE**  
October 2002

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
0010	--	--	--	--	--	--	--	Modified Method 5 Sampling Train	0010 Rev 0 (9/86)
--	--	--	0011 (Up. III)	--	--	--	--	Sampling for Selected Aldehyde and Ketone Emissions from Stationary Sources	0011 Rev 0 (12/96)
0020	--	--	--	--	--	--	--	Source Assessment Sampling System (SASS)	0020 Rev 0 (9/86)
--	--	--	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	0023A Rev 1 (12/96)
--	--	--	--	--	--	25D Referral	--	Determination of the Volatile Organic Content of Waste Samples	See 40 CFR 60, App. A
--	--	--	--	--	--	25E Referral	--	Determination of Vapor Phase Organic Concentration in Waste Samples	See 40 CFR 60, App. A
0030	--	--	--	--	--	--	--	Volatile Organic Sampling Train	0030 Rev 0 (9/86)
--	--	--	0031 (Up. III)	--	--	--	--	Sampling Method for Volatile Organic Compounds (SMVOC)	0031 Rev 0 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	0040 (Up. III)	--	--	--	--	Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar® Bags	0040 Rev 0 (12/96)
--	--	--	0050 (Up. III)	--	--	--	--	Isokinetic HCl/Cl <sub>2</sub> Emission Sampling Train	0050 Rev 0 (12/96)
--	--	--	0051 (Up. III)	--	--	--	--	Midget Impinger HCl/Cl <sub>2</sub> Emission Sampling Train	0051 Rev 0 (12/96)
--	--	--	0060 (Up. III)	--	--	--	--	Determination of Metals in Stack Emissions	0060 Rev 0 (12/96)
--	--	--	0061 (Up. III)	--	--	--	--	Determination of Hexavalent Chromium Emissions from Stationary Sources	0061 Rev 0 (12/96)
--	--	--	0100 (Up. III)	--	--	--	--	Sampling for Formaldehyde and Other Carbonyl Compounds in Indoor Air	0100 Rev 0 (12/96)
--	--	--	--	--	--	207-1 Referral	--	Sampling Method for Isocyanates	Not Promul-gated
--	--	--	--	--	--	207-2 Referral	--	Analysis for Isocyanates by High Performance Liquid Chromatography (HPLC)	Not Promul-gated

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
1010	--	--	--	1010A	--	--	--	Pensky-Martens Closed-Cup Method for Determining Ignitability	1010 Rev 0 (9/86)
1020	1020A	--	--	1020B	--	--	--	1020A (Promulgated): Setaflash Closed-Cup Method for Determining Ignitability  1020B (Proposed Up. IIIB): Small Scale Closed-Cup Method for Determining Ignitability	1020A Rev 1 (7/92)
--	--	--	1030 (Up. III)	--	--	--	--	Ignitability of Solids	1030 Rev 0 (12/96)
--	--	--	--	--	--	1040	--	Test Method for Oxidizing Solids	Not Promulgated
--	--	--	--	--	--	1050	--	Test Methods to Determine Substances Likely to Spontaneously Combust	Not Promulgated
1110	--	--	--	1110A	--	--	--	Corrosivity Toward Steel	1110 Rev 0 (9/86)
--	--	--	1120 (Up. III)	--	--	--	--	Dermal Corrosion	1120 Rev 0 (12/96)
1310	1310A	--	--	1310B	--	--	--	Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test	1310A Rev 1 (7/92)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	1311	--	--	--	--	--	--	Toxicity Characteristic Leaching Procedure	1311 Rev 0 (7/92)
--	--	1312 (Up. II)	--	--	--	--	--	Synthetic Precipitation Leaching Procedure	1312 Rev 0 (9/94)
1320	--	--	--	--	--	--	--	Multiple Extraction Procedure	1320 Rev 0 (9/86)
1330	1330A	--	--	--	--	--	--	Extraction Procedure for Oily Wastes	1330A Rev 1 (7/92)
3005	3005A	--	--	--	--	--	--	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy	3005A Rev 1 (7/92)
3010	3010A	--	--	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy	3010A Rev 1 (7/92)
--	--	3015 (Up. II)	--	--	3015A	--	--	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts	3015 Rev 0 (9/94)
3020	3020A	--	--	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy	3020A Rev 1 (7/92)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	3031 (Up. III)	--	--	--	--	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry	3031 Rev 0 (12/96)
3040	--	--	3040A (Up. III)	--	--	--	--	Dissolution Procedure for Oils, Greases, or Waxes	3040A Rev 1 (12/96)
3050	3050A	--	3050B (Up. III)	--	--	--	--	Acid Digestion of Sediments, Sludges, and Soils	3050B Rev 2 (12/96)
--	--	3051 (Up. II)	--	--	3051A	--	--	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils	3051 Rev 0 (9/94)
--	--	--	3052 (Up. III)	--	--	--	--	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices	3052 Rev 0 (12/96)
[3060, in the 2nd Ed.]	--	--	3060A (Up. III)	--	--	--	--	Alkaline Digestion for Hexavalent Chromium	3060A Rev 1 (12/96)
3500	3500A	--	3500B (Up. III)	--	--	3500C	--	Organic Extraction and Sample Preparation	3500B Rev 2 (12/96)
3510	3510A	3510B (Up. II)	3510C (Up. III)	--	--	--	--	Separatory Funnel Liquid-Liquid Extraction	3510C Rev 3 (12/96)
3520	3520A	3520B (Up. II)	3520C (Up. III)	--	--	--	--	Continuous Liquid-Liquid Extraction	3520C Rev 3 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	3535 (Up. III)	--	3535A	3535A (Replaces IVA version)	--	Solid-Phase Extraction (SPE)	3535 Rev 0 (12/96)
3540	3540A	3540B (Up. II)	3540C (Up. III)	--	--	--	--	Soxhlet Extraction	3540C Rev 3 (12/96)
--	--	3541 (Up. II)	--	--	--	--	--	Automated Soxhlet Extraction	3541 Rev 0 (9/94)
--	--	--	3542 (Up. III)	--	--	--	--	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)	3542 Rev 0 (12/96)
--	--	--	3545 (Up. III)	--	3545A	3545A (Replaces IVA version)	--	Pressurized Fluid Extraction (PFE)	3545 Rev 0 (12/96)
--	--	--	--	--	--	3546	--	Microwave Extraction	Not Promulgated
3550	--	3550A (Up. II)	3550B (Up. III)	--	--	3550C	--	Ultrasonic Extraction	3550B Rev 2 (12/96)
--	--	--	3560 (Up. III)	--	--	--	--	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons	3560 Rev 0 (12/96)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	3561 (Up. III)	--	--	--	--	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons	3561 Rev 0 (12/96)
--	--	--	--	--	3562	--	--	Supercritical Fluid Extraction of Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides	Not Promulgated
3580	3580A	--	--	--	--	--	--	Waste Dilution	3580A Rev 1 (7/92)
--	--	--	3585 (Up. III)	--	--	--	--	Waste Dilution for Volatile Organics	3585 Rev 0 (12/96)
3600	3600A	3600B (Up. II)	3600C (Up. III)	--	--	--	--	Cleanup	3600C Rev 3 (12/96)
3610	3610A	--	3610B (Up. III)	--	--	--	--	Alumina Cleanup	3610B Rev 2 (12/96)
3611	3611A	--	3611B (Up. III)	--	--	--	--	Alumina Column Cleanup and Separation of Petroleum Wastes	3611B Rev 2 (12/96)
3620	3620A	--	3620B (Up. III)	--	--	3620C	--	Florisil Cleanup	3620B Rev 2 (12/96)
3630	3630A	3630B (Up. II)	3630C (Up. III)	--	--	--	--	Silica Gel Cleanup	3630C Rev 3 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
3640	--	3640A (Up. II)	--	--	--	--	--	Gel-Permeation Cleanup	3640A Rev 1 (9/94)
3650	3650A	--	3650B (Up. III)	--	--	--	--	Acid-Base Partition Cleanup	3650B Rev 2 (12/96)
3660	3660A	--	3660B (Up. III)	--	--	--	--	Sulfur Cleanup	3660B Rev 2 (12/96)
--	--	3665 (Up. II)	3665A (Up. III)	--	--	--	--	Sulfuric Acid/Permanganate Cleanup	3665A Rev 1 (12/96)
3810	--	--	--	--	Noticed for removal from SW-846	--	--	Headspace	3810 Rev 0 (9/86)
--	--	--	--	--	--	3815	--	Screening Solid Samples for Volatile Organics	Not Promulgated
3820	--	--	--	--	--	--	--	Hexadecane Extraction and Screening of Purgeable Organics	3820 Rev 0 (9/86)
--	--	--	4000 (Up. III)	--	--	--	--	Immunoassay	4000 Rev 0 (12/96)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	4010 (Up. IIA)	4010A (Up. III)	--	--	--	--	Screening for Pentachlorophenol by Immunoassay	4010A Rev 1 (12/96)
--	--	--	4015 (Up. III)	--	--	--	--	Screening for 2,4-Dichlorophenoxyacetic Acid by Immunoassay	4015 Rev 0 (12/96)
--	--	--	4020 (Up. III)	--	--	--	--	Screening for Polychlorinated Biphenyls by Immunoassay	4020 Rev 0 (12/96)
--	--	--	4030 (Up. III)	--	--	--	--	Soil Screening for Petroleum Hydrocarbons by Immunoassay	4030 Rev 0 (12/96)
--	--	--	4035 (Up. III)	--	--	--	--	Soil Screening for Polynuclear Aromatic Hydrocarbons by Immunoassay	4035 Rev 0 (12/96)
--	--	--	4040 (Up. III)	--	--	--	--	Soil Screening for Toxaphene by Immunoassay	4040 Rev 0 (12/96)
--	--	--	4041 (Up. III)	--	--	--	--	Soil Screening for Chlordane by Immunoassay	4041 Rev 0 (12/96)
--	--	--	4042 (Up. III)	--	--	--	--	Soil Screening for DDT by Immunoassay	4042 Rev 0 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	--	--	--	--	4025 (10/02)	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay	Not Promulgated
--	--	--	4050 (Up. III)	--	--	--	--	TNT Explosives in Soil by Immunoassay	4050 Rev 0 (12/96)
--	--	--	4051 (Up. III)	--	--	--	--	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in Soil by Immunoassay	4051 Rev 0 (12/96)
--	--	--	--	--	--	4425	--	Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, PCDDs/PCDFs) by a Reporter Gene on a Human Cell Line	Not Promulgated
--	--	--	--	--	4500	--	--	Mercury in Soil by Immunoassay	Not Promulgated
--	--	--	--	--	4670	--	--	Triazine Herbicides as Atrazine in Water by Quantitative Immunoassay	Not Promulgated
--	--	--	5000 (Up. III)	--	--	--	--	Sample Preparation for Volatile Organic Compounds	5000 Rev 0 (12/96)
--	--	--	5021 (Up. III)	--	--	--	--	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis	5021 Rev 0 (12/96)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
5030	5030A	--	5030B (Up. III)	--	--	--	--	Purge-and-Trap for Aqueous Samples	5030B Rev 2 (12/96)
--	--	--	5031 (Up. III)	--	--	--	--	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation	5031 Rev 0 (12/96)
--	--	--	5032 (Up. III)	--	--	--	--	Volatile Organic Compounds by Vacuum Distillation	5032 Rev 0 (12/96)
--	--	--	5035 (Up. III)	--	--	--	5035A (7/02)	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples	5035 Rev 0 (12/96)
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	Deleted from SW-846
--	--	5041 (Up. II)	5041A (Up. III)	--	--	--	--	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)	5041A Rev 1 (12/96)
--	--	5050 (Up. II)	--	--	--	--	--	Bomb Preparation Method for Solid Waste	5050 Rev 0 (9/94)
6010	6010A	--	6010B (Up. III)	--	--	6010C	--	Inductively Coupled Plasma-Atomic Emission Spectrometry	6010B Rev 2 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	6020 (Up. II)	--	--	6020A	--	--	Inductively Coupled Plasma - Mass Spectrometry	6020 Rev 0 (9/94)
--	--	--	--	--	6200	--	--	Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment	Not Promulgated
--	--	--	--	--	6500	--	--	Dissolved Inorganic Anions in Aqueous Matrices by Capillary Ion Electrophoresis	Not Promulgated
--	--	--	--	--	6800	--	--	Elemental and Speciated Isotope Dilution Mass Spectrometry	Not Promulgated
7000	7000A	--	--	--	7000B	--	--	7000A (Promulgated): Atomic Absorption Methods 7000B (Draft Up. IVA): Flame Atomic Absorption Spectrophotometry	7000A Rev 1 (7/92)
--	--	--	--	--	7010	--	--	Graphite Furnace Atomic Absorption Spectrophotometry	Not Promulgated
7020	--	--	--	--	Noticed for removal from SW-846	--	--	Aluminum (Atomic Absorption, Direct Aspiration)	7020 Rev 0 (9/86)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
7040	--	--	--	--	Noticed for removal from SW-846	--	--	Antimony (Atomic Absorption, Direct Aspiration)	7040 Rev 0 (9/86)
7041	--	--	--	--	Noticed for removal from SW-846	--	--	Antimony (Atomic Absorption, Furnace Technique)	7041 Rev 0 (9/86)
7060	--	7060A (Up. II)	--	--	Noticed for removal from SW-846	--	--	Arsenic (Atomic Absorption, Furnace Technique)	7060A Rev 1 (9/94)
7061	7061A	--	--	--	--	--	--	Arsenic (Atomic Absorption, Gaseous Hydride)	7061A Rev 1 (7/92)
--	--	7062 (Up. II)	--	--	--	--	--	Antimony and Arsenic (Atomic Absorption, Borohydride Reduction)	7062 Rev 0 (9/94)
--	--	--	7063 (Up. III)	--	--	--	--	Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)	7063 Rev 0 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
7080	--	7080A (Up. II)	--	--	Noticed for removal from SW-846	--	--	Barium (Atomic Absorption, Direct Aspiration)	7080A Rev 1 (9/94)
--	7081	--	--	--	Noticed for removal from SW-846	--	--	Barium (Atomic Absorption, Furnace Technique)	7081 Rev 0 (7/92)
7090	--	--	--	--	Noticed for removal from SW-846	--	--	Beryllium (Atomic Absorption, Direct Aspiration)	7090 Rev 0 (9/86)
7091	--	--	--	--	Noticed for removal from SW-846	--	--	Beryllium (Atomic Absorption, Furnace Technique)	7091 Rev 0 (9/86)
7130	--	--	--	--	Noticed for removal from SW-846	--	--	Cadmium (Atomic Absorption, Direct Aspiration)	7130 Rev 0 (9/86)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
7131	--	7131A (Up. II)	--	--	Noticed for removal from SW-846	--	--	Cadmium (Atomic Absorption, Furnace Technique)	7131A Rev 1 (9/94)
7140	--	--	--	--	Noticed for removal from SW-846	--	--	Calcium (Atomic Absorption, Direct Aspiration)	7140 Rev 0 (9/86)
7190	--	--	--	--	Noticed for removal from SW-846	--	--	Chromium (Atomic Absorption, Direct Aspiration)	7190 Rev 0 (9/86)
7191	--	--	--	--	Noticed for removal from SW-846	--	--	Chromium (Atomic Absorption, Furnace Technique)	7191 Rev 0 (9/86)
7195	--	--	--	--	--	--	--	Chromium, Hexavalent (Coprecipitation)	7195 Rev 0 (9/86)
7196	7196A	--	--	--	--	--	--	Chromium, Hexavalent (Colorimetric)	7196A Rev 1 (7/92)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
7197	--	--	--	--	--	--	--	Chromium, Hexavalent (Chelation/Extraction)	7197 Rev 0 (9/86)
7198	--	--	--	--	--	--	--	Chromium, Hexavalent (Differential Pulse Polarography)	7198 Rev 0 (9/86)
--	--	--	7199 (Up. III)	--	--	--	--	Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography	7199 Rev 0 (12/96)
7200	--	--	--	--	Noticed for removal from SW-846	--	--	Cobalt (Atomic Absorption, Direct Aspiration)	7200 Rev 0 (9/86)
7201	--	--	--	--	Noticed for removal from SW-846	--	--	Cobalt (Atomic Absorption, Furnace Technique)	7201 Rev 0 (9/86)
7210	--	--	--	--	Noticed for removal from SW-846	--	--	Copper (Atomic Absorption, Direct Aspiration)	7210 Rev 0 (9/86)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	7211	--	--	--	Noticed for removal from SW-846	--	--	Copper (Atomic Absorption, Furnace Technique)	7211 Rev 0 (7/92)
7380	--	--	--	--	Noticed for removal from SW-846	--	--	Iron (Atomic Absorption, Direct Aspiration)	7380 Rev 0 (9/86)
--	7381	--	--	--	Noticed for removal from SW-846	--	--	Iron (Atomic Absorption, Furnace Technique)	7381 Rev 0 (7/92)
7420	--	--	--	--	Noticed for removal from SW-846	--	--	Lead (Atomic Absorption, Direct Aspiration)	7420 Rev 0 (9/86)
7421	--	--	--	--	Noticed for removal from SW-846	--	--	Lead (Atomic Absorption, Furnace Technique)	7421 Rev 0 (9/86)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	7430	--	--	--	Noticed for removal from SW-846	--	--	Lithium (Atomic Absorption, Direct Aspiration)	7430 Rev 0 (7/92)
7450	--	--	--	--	Noticed for removal from SW-846	--	--	Magnesium (Atomic Absorption, Direct Aspiration)	7450 Rev 0 (9/86)
7460	--	--	--	--	Noticed for removal from SW-846	--	--	Manganese (Atomic Absorption, Direct Aspiration)	7460 Rev 0 (9/86)
--	7461	--	--	--	Noticed for removal from SW-846	--	--	Manganese (Atomic Absorption, Furnace Technique)	7461 Rev 0 (7/92)
7470	--	7470A (Up. II)	--	--	--	--	--	Mercury in Liquid Waste (Manual Cold-Vapor Technique)	7470A Rev 1 (9/94)
7471	--	7471A (Up. II)	--	--	7471B	--	--	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)	7471A Rev 1 (9/94)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	7472 (Up. III)	--	--	--	--	Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)	7472 Rev 0 (12/96)
--	--	--	--	--	7473	--	--	Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry	Not Promulgated
--	--	--	--	--	7474	--	--	Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry	Not Promulgated
7480	--	--	--	--	Noticed for removal from SW-846	--	--	Molybdenum (Atomic Absorption, Direct Aspiration)	7480 Rev 0 (9/86)
7481	--	--	--	--	Noticed for removal from SW-846	--	--	Molybdenum (Atomic Absorption, Furnace Technique)	7481 Rev 0 (9/86)
7520	--	--	--	--	Noticed for removal from SW-846	--	--	Nickel (Atomic Absorption, Direct Aspiration)	7520 Rev 0 (9/86)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	7521 (Up. III)	--	Noticed for removal from SW-846	--	--	Nickel (Atomic Absorption, Furnace Method)	7521 Rev 0 (12/96)
7550	--	--	--	--	Noticed for removal from SW-846	--	--	Osmium (Atomic Absorption, Direct Aspiration)	7550 Rev 0 (9/86)
--	--	--	7580 (Up. III)	--	--	--	--	White Phosphorus (P <sub>4</sub> ) by Solvent Extraction and Gas Chromatography	7580 Rev 0 (12/96)
7610	--	--	--	--	Noticed for removal from SW-846	--	--	Potassium (Atomic Absorption, Direct Aspiration)	7610 Rev 0 (9/86)
7740	--	--	--	--	Noticed for removal from SW-846	--	--	Selenium (Atomic Absorption, Furnace Technique)	7740 Rev 0 (9/86)
7741	--	7741A (Up. II)	--	--	--	--	--	Selenium (Atomic Absorption, Gaseous Hydride)	7741A Rev 1 (9/94)

## SW-846 IOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	7742 (Up. II)	--	--	--	--	--	Selenium (Atomic Absorption, Borohydride Reduction)	7742 Rev 0 (9/94)
7760	7760A	--	--	--	Noticed for removal from SW-846	--	--	Silver (Atomic Absorption, Direct Aspiration)	7760A Rev 1 (7/92)
--	7761	--	--	--	Noticed for removal from SW-846	--	--	Silver (Atomic Absorption, Furnace Technique)	7761 Rev 0 (7/92)
7770	--	--	--	--	Noticed for removal from SW-846	--	--	Sodium (Atomic Absorption, Direct Aspiration)	7770 Rev 0 (9/86)
--	7780	--	--	--	Noticed for removal from SW-846	--	--	Strontium (Atomic Absorption, Direct Aspiration)	7780 Rev 0 (7/92)
7840	--	--	--	--	Noticed for removal from SW-846	--	--	Thallium (Atomic Absorption, Direct Aspiration)	7840 Rev 0 (9/86)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
7841	--	--	--	--	Noticed for removal from SW-846	--	--	Thallium (Atomic Absorption, Furnace Technique)	7841 Rev 0 (9/86)
7870	--	--	--	--	Noticed for removal from SW-846	--	--	Tin (Atomic Absorption, Direct Aspiration)	7870 Rev 0 (9/86)
7910	--	--	--	--	Noticed for removal from SW-846	--	--	Vanadium (Atomic Absorption, Direct Aspiration)	7910 Rev 0 (9/86)
7911	--	--	--	--	Noticed for removal from SW-846	--	--	Vanadium (Atomic Absorption, Furnace Technique)	7911 Rev 0 (9/86)
7950	--	--	--	--	Noticed for removal from SW-846	--	--	Zinc (Atomic Absorption, Direct Aspiration)	7950 Rev 0 (9/86)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	7951	--	--	--	Noticed for removal from SW-846	--	--	Zinc (Atomic Absorption, Furnace Technique)	7951 Rev 0 (7/92)
8000	8000A	--	8000B (Up. III)	--	--	--	--	Determinative Chromatographic Separations	8000B Rev 2 (12/96)
8010	8010A	8010B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Halogenated Volatile Organics by Gas Chromatography	Deleted from SW-846
--	8011	--	--	--	--	--	--	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography	8011 Rev 0 (7/92)
8015	8015A	--	8015B	--	--	8015C	--	Nonhalogenated Organics Using GC/FID	8015B Rev 2 (12/96)
8020	--	8020A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Aromatic Volatile Organics by Gas Chromatography	Deleted from SW-846
--	8021	8021A (Up. II)	8021B (Up. III)	--	--	--	--	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors	8021B Rev 2 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
8030	8030A	--	Deleted from SW-846 (Up. III)	--	--	--	--	Acrolein and Acrylonitrile by Gas Chromatography	Deleted from SW-846
--	--	8031 (Up. II)	--	--	--	--	--	Acrylonitrile by Gas Chromatography	8031 Rev 0 (9/94)
--	--	8032 (Up. II)	8032A (Up. III)	--	--	--	--	Acrylamide by Gas Chromatography	8032A Rev 1 (12/96)
--	--	--	8033 (Up. III)	--	--	--	--	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection	8033 Rev 0 (12/96)
8040	8040A	--	Deleted from SW-846 (Up. III)	--	--	--	--	Phenols by Gas Chromatography	Deleted from SW-846
--	--	--	8041 (Up. III)	--	--	8041A	--	Phenols by Gas Chromatography	8041 Rev 0 (12/96)
8060	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Phthalate Esters	Deleted from SW-846
--	--	8061 (Up. II)	8061A (Up. III)	--	--	--	--	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)	8061A Rev 1 (12/96)

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	8070	--	8070A (Up. III)	--	--	--	--	Nitrosamines by Gas Chromatography	8070A Rev 1 (12/96)
8080	--	8080A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography	Deleted from SW-846
--	--	8081 (Up. II)	8081A (Up. III)	--	8081B	8081B (Replaces IVA version)	--	Organochlorine Pesticides by Gas Chromatography	8081A Rev 1 (12/96)
--	--	--	8082 (Up. III)	--	8082A	8082A (Replaces IVA version)	--	Polychlorinated Biphenyls (PCBs) by Gas Chromatography	8082 Rev 0 (12/96)
				--		8085	--	Compound-independent Elemental Quantitation of Pesticides by Gas Chromatography with Atomic Emission Detection (GC/AED)	Not Promulgated
8090	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Nitroaromatics and Cyclic Ketones	Deleted from SW-846
--	--	--	8091 (Up. III)	--	--	--	--	Nitroaromatics and Cyclic Ketones by Gas Chromatography	8091 Rev 0 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	--	--	--	8095	--	Explosives by Gas Chromatography	Not Promulgated
8100	--	--	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons	8100 Rev 0 (9/86)
--	8110	--	Deleted from SW-846 (Up. III)	--	--	--	--	Haloethers by Gas Chromatography	Deleted from SW-846
--	--	--	8111 (Up. III)	--	--	--	--	Haloethers by Gas Chromatography	8111 Rev 0 (12/96)
8120	--	8120A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography	Deleted from SW-846
--	--	8121 (Up. II)	--	--	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique	8121 Rev 0 (9/94)
--	--	--	8131 (Up. III)	--	--	--	--	Aniline and Selected Derivatives by Gas Chromatography	8131 Rev 0 (12/96)
8140	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Organophosphorus Pesticides	Deleted from SW-846

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	8141	8141A (Up. II)	--	--	8141B	8141B (Replaces IVA version)	--	8141A (Promulgated): Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique  8141B (Draft Up. IVB): Organophosphorus Compounds by Gas Chromatography	8141A Rev 1 (9/94)
8150	8150A	8150B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Chlorinated Herbicides by Gas Chromatography	Deleted from SW-846
--	--	8151 (Up. II)	8151A (Up. III)	--	--	--	--	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzyl Derivatization	8151A Rev 1 (12/96)
8240	8240A	8240B (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Deleted from SW-846
8250	--	8250A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Deleted from SW-846
--	8260	8260A (Up. II)	8260B (Up. III)	--	--	--	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	8260B Rev 2 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	--	--	--	8261	--	Volatile Organic Compounds by Vacuum Distillation in Combination with Gas Chromatography/Mass Spectrometry (VD/GC/MS)	Not Promul-gated
--	--	--	--	--	--	--	8265 (3/02)	Volatile Organic Compounds in Water, Soil, Soil Gas and Air by Direct Sampling Ion Trap Mass Spectrometry (DSITMS)	Not Promul-gated
8270	8270A	8270B (Up. II)	8270C (Up. III)	--	8270D	--	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	8270C Rev 3 (12/96)
--	--	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)	8275A Rev 1 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
8280	--	--	8280A (Up. III)	--	8280B	--	--	8280A (Promulgated): The Analysis of Polychlorinated Dibenzo- <i>p</i> -Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)  8280B (Draft Up. IVA): Polychlorinated Dibenzo- <i>p</i> -Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)	8280A Rev 1 (12/96)
--	--	8290 (Up. II)	--	--	8290A	--	--	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/ High-Resolution Mass Spectrometry (HRGC/HRMS)	8290 Rev 0 (9/94)
8310	--	--	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons	8310 Rev 0 (9/86)
--	--	8315 (Up. II)	8315A (Up. III)	--	--	--	--	Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)	8315A Rev 1 (12/96)
--	--	8316 (Up. II)	--	--	--	--	--	Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)	8316 Rev 0 (9/94)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	8318 (Up. II)	--	--	--	8318A	--	N-Methylcarbamates by High Performance Liquid Chromatography (HPLC)	8318 Rev 0 (9/94)
--	--	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	8321A Rev 1 (12/96)
--	--	--	8325 (Up. III)	--	--	--	--	Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry (HPLC/PB/MS)	8325 Rev 0 (12/96)
--	--	8330 (Up. II)	--	--	8330A	--	--	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)	8330 Rev 0 (9/94)
--	--	8331 (Up. II)	--	--	--	--	--	Tetrazene by Reverse Phase High Performance Liquid Chromatography (HPLC)	8331 Rev 0 (9/94)
--	--	--	8332 (Up. III)	--	--	--	--	Nitroglycerine by High Performance Liquid Chromatography	8332 Rev 0 (12/96)
--	--	8410 (Up. II)	--	--	--	--	--	Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column	8410 Rev 0 (9/94)

## SW-84 HOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	8430 (Up. III)	--	--	--	--	Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR	8430 Rev 0 (12/96)
--	--	--	8440 (Up. III)	--	--	--	--	Total Recoverable Petroleum Hydrocarbons by Infrared Spectrophotometry	8440 Rev 0 (12/96)
				--		8510	--	Colorimetric Screening Procedure for RDX and HMX in Soil	Not Promulgated
--	--	--	8515 (Up. III)	--	--	--	--	Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil	8515 Rev 0 (12/96)
--	--	--	8520 (Up. III)	--	--	--	--	Continuous Measurement of Formaldehyde in Ambient Air	8520 Rev 0 (12/96)
--	--	--	--	--	--	8535	--	Screening Procedure for Total Volatile Organic Halides in Water	Not Promulgated
--	--	--	--	--	--	8540	--	Pentachlorophenol by UV-induced Colorimetry	Not Promulgated
--	--	--	--	--	9000	--	--	Determination of Water in Waste Materials by Karl Fischer Titration	Not Promulgated
--	--	--	--	--	9001	--	--	Determination of Water in Waste Materials by Quantitative Calcium Hydride Reaction	Not Promulgated
9010	9010A	--	9010B (Up. III)	9010C	--	--	--	Total and Amenable Cyanide: Distillation	9010B Rev 2 (12/96)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
9012	--	--	9012A (Up. III)	9012B	--	--	--	Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)	9012A Rev 1 (12/96)
--	9013	--	--	--	--	--	--	Cyanide Extraction Procedure for Solids and Oils	9013 Rev 0 (7/92)
--	--	--	9014 (Up. III)	--	--	--	--	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide	9014 Rev 0 (12/96)
9020	9020A	9020B (Up. II)	--	--	--	--	--	Total Organic Halides (TOX)	9020B Rev 2 (9/94)
--	9021	--	--	--	--	--	--	Purgeable Organic Halides (POX)	9021 Rev 0 (7/92)
9022	--	--	--	--	--	--	--	Total Organic Halides (TOX) by Neutron Activation Analysis	9022 Rev 0 (9/86)
--	--	--	9023 (Up. III)	--	--	--	--	Extractable Organic Halides (EOX) in Solids	9023 Rev 0 (12/96)
9030	9030A	--	9030B (Up. III)	--	--	--	--	Acid-Soluble and Acid-Insoluble Sulfides: Distillation	9030B Rev 2 (12/96)
--	9031	--	--	--	--	--	--	Extractable Sulfides	9031 Rev 0 (7/92)

## SW-846 METHOD STATUS TABLE (10/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	9034 (Up. III)	--	--	--	--	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides	9034 Rev 0 (12/96)
9035	--	--	--	--	--	--	--	Sulfate (Colorimetric, Automated, Chloranilate)	9035 Rev 0 (9/86)
9036	--	--	--	--	--	--	--	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)	9036 Rev 0 (9/86)
9038	--	--	--	--	--	--	--	Sulfate (Turbidimetric)	9038 Rev 0 (9/86)
9040	--	9040A (Up. II) 9040B (Up. IIB)	--	9040C	--	--	--	pH Electrometric Measurement	9040B Rev 2 (1/95)
9041	9041A	--	--	--	--	--	--	pH Paper Method	9041A Rev 1 (7/92)
9045	9045A	9045B (Up. II) 9045C (Up. IIB)	--	9045D	--	--	--	Soil and Waste pH	9045C Rev 3 (1/95)

## SW-846 METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
9050	--	--	9050A (Up. III)	--	--	--	--	Specific Conductance	9050A Rev 1 (12/96)
--	--	9056 (Up. II)	--	--	--	9056A	--	Determination of Inorganic Anions by Ion Chromatography	9056 Rev 0 (9/94)
--	--	--	9057 (Up. III)	--	--	--	--	Determination of Chloride from HCl/Cl <sub>2</sub> Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography	9057 Rev 0 (12/96)
				--		9058	--	Determination of Perchlorate Using Ion Chromatography with Chemical Suppression Conductivity Detection	Not Promulgated
9060	--	--	--	9060A	--	--	--	Total Organic Carbon	9060 Rev 0 (9/86)
9065	--	--	--	--	--	--	--	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)	9065 Rev 0 (9/86)
9066	--	--	--	--	--	--	--	Phenolics (Colorimetric, Automated 4-AAP with Distillation)	9066 Rev 0 (9/86)
9067	--	--	--	--	--	--	--	Phenolics (Spectrophotometric, MBTH with Distillation)	9067 Rev 0 (9/86)

SW-846 METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
9070	--	--	9070 Title and text replaced with referral to Method 1664 (Up. IIIA)	9070A (Suffix and title added)	--	--	--	9070 (Final Update IIIA): No title 9070A (Prop. Up. IIIB): n-Hexane Extractable Material (HEM) for Aqueous Samples  Text refers reader 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	The current promulgated version of Method 1664 <sup>1</sup> is Rev. A
9071	--	9071A (Up. II)	9071B (Up. IIIA)	--	--	--	--	n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples	9071B Rev 2 (4/98)
--	--	--	--	--	9074	--	--	Turbidimetric Screening Method for Total Recoverable Petroleum Hydrocarbons in Soil	Not Promulgated
--	--	9075 (Up. II)	--	--	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)	9075 Rev 0 (9/94)

<sup>1</sup> Method 1664 (EPA Publication No. EPA-821-R-98-002) is available via the Internet at <http://www.epa.gov/OST>. It is also available from the National Technical Information Service (NTIS), NTIS Publication No. PB99-121949 (call 703-605-6000 or 800-553-6847).

## SW-846 METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMUL-GATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	9076 (Up. II)	--	--	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry	9076 Rev 0 (9/94)
--	--	9077 (Up. II)	--	--	--	--	--	Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)	9077 Rev 0 (9/94)
--	--	--	9078 (Up. III)	--	--	--	--	Screening Test Method for Polychlorinated Biphenyls in Soil	9078 Rev 0 (12/96)
--	--	--	9079 (Up. III)	--	--	--	--	Screening Test Method for Polychlorinated Biphenyls in Transformer Oil	9079 Rev 0 (12/96)
9080	--	--	--	--	--	--	--	Cation-Exchange Capacity of Soils (Ammonium Acetate)	9080 Rev 0 (9/86)
9081	--	--	--	--	--	--	--	Cation-Exchange Capacity of Soils (Sodium Acetate)	9081 Rev 0 (9/86)
9090	9090A	--	--	--	--	--	--	Compatibility Test for Wastes and Membrane Liners	9090A Rev 1 (7/92)
9095	--	--	9095A (Up. III)	9095B	--	--	--	Paint Filter Liquids Test	9095A Rev 1 (12/96)

## SW-846 METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	--	9213 (Up. III)	--	--	--	--	Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion-Selective Electrode	9213 Rev 0 (12/96)
--	--	--	9214 (Up. III)	--	--	--	--	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode	9214 Rev 0 (12/96)
--	--	--	9215 (Up. III)	--	--	--	--	Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion-Selective Electrode	9215 Rev 0 (12/96)
--	--	--	--	--	9216	--	--	Potentiometric Determination of Nitrite in Aqueous Samples with Ion-Selective Electrode	Not Promulgated
9250	--	--	--	--	--	--	--	Chloride (Colorimetric, Automated Ferricyanide AAI)	9250 Rev 0 (9/86)
9251	--	--	--	--	--	--	--	Chloride (Colorimetric, Automated Ferricyanide AAI)	9251 Rev 0 (9/86)
9252	--	9252A (Up. II)	Deleted from SW-846 (Up. III)	--	--	--	--	Chloride (Titrimetric, Mercuric Nitrate)	Deleted from SW-846
--	--	9253 (Up. II)	--	--	--	--	--	Chloride (Titrimetric, Silver Nitrate)	9253 Rev 0 (9/94)

## SW-8—METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
--	--	9096 (Up. II)	--	--	--	--	--	Liquid Release Test (LRT) Procedure	9096 Rev 0 (9/94)
9100	--	--	--	--	--	--	--	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability	9100 Rev 0 (9/86)
9131	--	--	--	--	--	--	--	Total Coliform: Multiple Tube Fermentation Technique	9131 Rev 0 (9/86)
9132	--	--	--	--	--	--	--	Total Coliform: Membrane-Filter Technique	9132 Rev 0 (9/86)
9200	--	--	Deleted from SW-846 (Up. III)	--	--	--	--	Nitrate	Deleted from SW-846
--	--	--	9210 (Up. III)	--	--	9210A	--	Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode	9210 Rev 0 (12/96)
--	--	--	9211 (Up. III)	--	--	--	--	Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode	9211 Rev 0 (12/96)
--	--	--	9212 (Up. III)	--	--	--	--	Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode	9212 Rev 0 (12/96)

## 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	PROP 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 5 (4/98)
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)
Chapter Four -- Organic Analytes	✓	--	✓ (Up. II)	✓ (Up. III)	✓	✓ (Replaces IVA version)	--	Rev 3 (12/96)

## SW-846 METHOD STATUS TABLE (8/02), CONTINUED

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

METHOD NUMBER								METHOD TITLE	CURRENT PROMULGATED METHOD
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)	PROP. UP. IIIB (8/02)	DRAFT UP. IVA (1/98)	DRAFT UP. IVB (11/00)	OTHER NEW SW-846 METHODS		
9310	--	--	--	--	--	--	--	Gross Alpha and Gross Beta	9310 Rev 0 (9/86)
9315	--	--	--	--	--	--	--	Alpha-Emitting Radium Isotopes	9315 Rev 0 (9/86)
9320	--	--	--	--	--	--	--	Radium-228	9320 Rev 0 (9/86)
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	Guidance Only Rev 3 (12/96)
H <sub>2</sub> S Test Meth.	H <sub>2</sub> S Test Meth.	H <sub>2</sub> S Test Method (Up. II)	H <sub>2</sub> S Test Method (Up. III)	Proposed Removal from SW-846	--	--	--	Test Method to Determine Hydrogen Sulfide Released from Wastes	Guidance Only Rev 3 (12/96)

TITLE	THIRD ED. (9/86)	FINAL UP. I (7/92)	FIN. L II (9/94) IIA (8/93) IIB (1/95)	FINAL UP. III (12/96) IIIA 4/98)	DRAFT UP. IVA	DRAFT UP. IVB	PROP UP. IIIB	CURRENT FINAL VERSION
Chapter Five -- Miscellaneous Test Methods	✓	--	✓ (Up. II)	✓ (Up. III & IIIA)	✓	✓ (Replaces IVA version)	✓	Rev 3 (4/98)
Chapter Six -- Properties	✓	--	✓ (Up. II & IIB)	✓ (Up. III)	--	✓	✓	Rev 2 (12/96)
Chapter Seven -- Characteristics Introduction and Regulatory Definitions	✓	✓	✓ (Up. II)	✓ (Up. III)	--	--	✓	Rev 3 (12/96)
Chapter Eight --Methods for Determining Characteristics	✓	--	✓ (Up. II)	✓ (Up. III)	--	--	✓	Rev 2 (12/96)
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)
Appendix -- Company References	✓	--	--	--	--	--	--	Rev 0 (9/86)

# TABLE OF CONTENTS

-----  
VOLUME ONE

SECTION A  
-----

DISCLAIMER

ABSTRACT

TABLE OF CONTENTS

METHOD INDEX AND CONVERSION TABLE

PREFACE

ACKNOWLEDGEMENTS

PART I METHODS FOR ANALYTES AND PROPERTIES

CHAPTER ONE -- QUALITY CONTROL

- 1.0 Introduction
- 2.0 QA Project Plan
- 3.0 Field Operations
- 4.0 Laboratory Operations
- 5.0 Definitions
- 6.0 References

CHAPTER TWO -- CHOOSING THE CORRECT PROCEDURE

- 2.1 Purpose
- 2.2 Required Information
- 2.3 Implementing the Guidance
- 2.4 Characteristics
- 2.5 Ground Water
- 2.6 References

CHAPTER THREE -- INORGANIC ANALYTES

- 3.1 Sampling Considerations
- 3.2 Sample Preparation Methods

**Method 3005A:** Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy

**Method 3010A:** Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy

**Method 3015:** Microwave Assisted Acid Digestion of Aqueous Samples and Extracts

<b>Method 3020A:</b>	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy
<b>Method 3031:</b>	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
<b>Method 3040A:</b>	Dissolution Procedure for Oils, Greases, or Waxes
<b>Method 3050B:</b>	Acid Digestion of Sediments, Sludges, and Soils
<b>Method 3051:</b>	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
<b>Method 3052:</b>	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
<b>Method 3060A:</b>	Alkaline Digestion for Hexavalent Chromium

### 3.3 Methods for Determination of Inorganic Analytes

<b>Method 6010B:</b>	Inductively Coupled Plasma-Atomic Emission Spectrometry
<b>Method 6020:</b>	Inductively Coupled Plasma-Mass Spectrometry
<b>Method 7000A:</b>	Atomic Absorption Methods
<b>Method 7020:</b>	Aluminum (Atomic Absorption, Direct Aspiration)
<b>Method 7040:</b>	Antimony (Atomic Absorption, Direct Aspiration)
<b>Method 7041:</b>	Antimony (Atomic Absorption, Furnace Technique)
<b>Method 7060A:</b>	Arsenic (Atomic Absorption, Furnace Technique)
<b>Method 7061A:</b>	Arsenic (Atomic Absorption, Gaseous Hydride)
<b>Method 7062:</b>	Antimony and Arsenic (Atomic Absorption, Borohydride Reduction)
<b>Method 7063:</b>	Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)
<b>Method 7080A:</b>	Barium (Atomic Absorption, Direct Aspiration)
<b>Method 7081:</b>	Barium (Atomic Absorption, Furnace Technique)
<b>Method 7090:</b>	Beryllium (Atomic Absorption, Direct Aspiration)
<b>Method 7091:</b>	Beryllium (Atomic Absorption, Furnace Technique)
<b>Method 7130:</b>	Cadmium (Atomic Absorption, Direct Aspiration)
<b>Method 7131A:</b>	Cadmium (Atomic Absorption, Furnace Technique)
<b>Method 7140:</b>	Calcium (Atomic Absorption, Direct Aspiration)
<b>Method 7190:</b>	Chromium (Atomic Absorption, Direct Aspiration)
<b>Method 7191:</b>	Chromium (Atomic Absorption, Furnace Technique)
<b>Method 7195:</b>	Chromium, Hexavalent (Coprecipitation)
<b>Method 7196A:</b>	Chromium, Hexavalent (Colorimetric)
<b>Method 7197:</b>	Chromium, Hexavalent (Chelation/Extraction)
<b>Method 7198:</b>	Chromium, Hexavalent (Differential Pulse Polarography)
<b>Method 7199:</b>	Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography
<b>Method 7200:</b>	Cobalt (Atomic Absorption, Direct Aspiration)
<b>Method 7201:</b>	Cobalt (Atomic Absorption, Furnace Technique)
<b>Method 7210:</b>	Copper (Atomic Absorption, Direct Aspiration)
<b>Method 7211:</b>	Copper (Atomic Absorption, Furnace Technique)
<b>Method 7380:</b>	Iron (Atomic Absorption, Direct Aspiration)
<b>Method 7381:</b>	Iron (Atomic Absorption, Furnace Technique)
<b>Method 7420:</b>	Lead (Atomic Absorption, Direct Aspiration)
<b>Method 7421:</b>	Lead (Atomic Absorption, Furnace Technique)

<b>Method 7430:</b>	Lithium (Atomic Absorption, Direct Aspiration)
<b>Method 7450:</b>	Magnesium (Atomic Absorption, Direct Aspiration)
<b>Method 7460:</b>	Manganese (Atomic Absorption, Direct Aspiration)
<b>Method 7461:</b>	Manganese (Atomic Absorption, Furnace Technique)
<b>Method 7470A:</b>	Mercury in Liquid Waste (Manual Cold-Vapor Technique)
<b>Method 7471A:</b>	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
<b>Method 7472:</b>	Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)
<b>Method 7480:</b>	Molybdenum (Atomic Absorption, Direct Aspiration)
<b>Method 7481:</b>	Molybdenum (Atomic Absorption, Furnace Technique)
<b>Method 7520:</b>	Nickel (Atomic Absorption, Direct Aspiration)
<b>Method 7521:</b>	Nickel (Atomic Absorption, Furnace Method)
<b>Method 7550:</b>	Osmium (Atomic Absorption, Direct Aspiration)
<b>Method 7580:</b>	White Phosphorus (P <sub>4</sub> ) by Solvent Extraction and Gas Chromatography
<b>Method 7610:</b>	Potassium (Atomic Absorption, Direct Aspiration)
<b>Method 7740:</b>	Selenium (Atomic Absorption, Furnace Technique)
<b>Method 7741A:</b>	Selenium (Atomic Absorption, Gaseous Hydride)
<b>Method 7742:</b>	Selenium (Atomic Absorption, Borohydride Reduction)
<b>Method 7760A:</b>	Silver (Atomic Absorption, Direct Aspiration)
<b>Method 7761:</b>	Silver (Atomic Absorption, Furnace Technique)
<b>Method 7770:</b>	Sodium (Atomic Absorption, Direct Aspiration)
<b>Method 7780:</b>	Strontium (Atomic Absorption, Direct Aspiration)
<b>Method 7840:</b>	Thallium (Atomic Absorption, Direct Aspiration)
<b>Method 7841:</b>	Thallium (Atomic Absorption, Furnace Technique)
<b>Method 7870:</b>	Tin (Atomic Absorption, Direct Aspiration)
<b>Method 7910:</b>	Vanadium (Atomic Absorption, Direct Aspiration)
<b>Method 7911:</b>	Vanadium (Atomic Absorption, Furnace Technique)
<b>Method 7950:</b>	Zinc (Atomic Absorption, Direct Aspiration)
<b>Method 7951:</b>	Zinc (Atomic Absorption, Furnace Technique)

## 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 promulgated revision of the method, even though the method number does not include the appropriate letter suffix.

-----  
VOLUME ONE

SECTION B  
-----

DISCLAIMER

ABSTRACT

TABLE OF CONTENTS

METHOD INDEX AND CONVERSION TABLE

PREFACE

ACKNOWLEDGEMENTS

CHAPTER ONE, REPRINTED -- QUALITY CONTROL

- 1.0 Introduction
- 2.0 QA Project Plan
- 3.0 Field Operations
- 4.0 Laboratory Operations
- 5.0 Definitions
- 6.0 References

CHAPTER FOUR -- ORGANIC ANALYTES

- 4.1 Sampling Considerations
- 4.2 Sample Preparation Methods

4.2.1 Extractions and Preparations

<b>Method 3500B:</b>	Organic Extraction and Sample Preparation
<b>Method 3510C:</b>	Separatory Funnel Liquid-Liquid Extraction
<b>Method 3520C:</b>	Continuous Liquid-Liquid Extraction
<b>Method 3535:</b>	Solid-Phase Extraction (SPE)
<b>Method 3540C:</b>	Soxhlet Extraction
<b>Method 3541:</b>	Automated Soxhlet Extraction
<b>Method 3542:</b>	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)
<b>Method 3545:</b>	Pressurized Fluid Extraction (PFE)
<b>Method 3550B:</b>	Ultrasonic Extraction
<b>Method 3560:</b>	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
<b>Method 3561:</b>	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
<b>Method 3580A:</b>	Waste Dilution
<b>Method 3585:</b>	Waste Dilution for Volatile Organics
<b>Method 5000:</b>	Sample Preparation for Volatile Organic Compounds
<b>Method 5021:</b>	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
<b>Method 5030B:</b>	Purge-and-Trap for Aqueous Samples

<b>Method 5031:</b>	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
<b>Method 5032:</b>	Volatile Organic Compounds by Vacuum Distillation
<b>Method 5035:</b>	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
<b>Method 5041A:</b>	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)

#### 4.2.2 Cleanup

<b>Method 3600C:</b>	Cleanup
<b>Method 3610B:</b>	Alumina Cleanup
<b>Method 3611B:</b>	Alumina Column Cleanup and Separation of Petroleum Wastes
<b>Method 3620B:</b>	Florisil Cleanup
<b>Method 3630C:</b>	Silica Gel Cleanup
<b>Method 3640A:</b>	Gel-Permeation Cleanup
<b>Method 3650B:</b>	Acid-Base Partition Cleanup
<b>Method 3660B:</b>	Sulfur Cleanup
<b>Method 3665A:</b>	Sulfuric Acid/Permanganate Cleanup

### 4.3 Determination of Organic Analytes

#### 4.3.1 Gas Chromatographic Methods

<b>Method 8000B:</b>	Determinative Chromatographic Separations
<b>Method 8011:</b>	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography
<b>Method 8015B:</b>	Nonhalogenated Organics Using GC/FID
<b>Method 8021B:</b>	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors
<b>Method 8031:</b>	Acrylonitrile by Gas Chromatography
<b>Method 8032A:</b>	Acrylamide by Gas Chromatography
<b>Method 8033:</b>	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection
<b>Method 8041:</b>	Phenols by Gas Chromatography
<b>Method 8061A:</b>	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)
<b>Method 8070A:</b>	Nitrosamines by Gas Chromatography
<b>Method 8081A:</b>	Organochlorine Pesticides by Gas Chromatography
<b>Method 8082:</b>	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
<b>Method 8091:</b>	Nitroaromatics and Cyclic Ketones by Gas Chromatography
<b>Method 8100:</b>	Polynuclear Aromatic Hydrocarbons
<b>Method 8111:</b>	Haloethers by Gas Chromatography
<b>Method 8121:</b>	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique
<b>Method 8131:</b>	Aniline and Selected Derivatives by Gas Chromatography
<b>Method 8141A:</b>	Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique

**Method 8151A:** Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization

#### 4.3.2 Gas Chromatographic/Mass Spectroscopic Methods

**Method 8260B:** Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

**Method 8270C:** Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

**Method 8275A:** Semivolatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)

**Method 8280A:** The Analysis of Polychlorinated Dibenzo-*p*-Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)

**Method 8290:** Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)

**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

**Method 8310:** Polynuclear Aromatic Hydrocarbons

**Method 8315A:** Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)

**Appendix A:** Recrystallization of 2,4-Dinitrophenylhydrazine (DNPH)

**Method 8316:** Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)

**Method 8318:** N-Methylcarbamates by High Performance Liquid Chromatography (HPLC)

**Method 8321A:** Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection

**Method 8325:** Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry (HPLC/PB/MS)

**Method 8330:** Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)

**Method 8331:** Tetrazene by Reverse Phase High Performance Liquid Chromatography (HPLC)

**Method 8332:** Nitroglycerine by High Performance Liquid Chromatography

#### 4.3.4 Infrared Methods

**Method 8410:** Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column

**Method 8430:** Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR

**Method 8440:** Total Recoverable Petroleum Hydrocarbons by Infrared Spectrophotometry

#### 4.3.5 Miscellaneous Spectrometric Methods

**Method 8520:** Continuous Measurement of Formaldehyde in Ambient Air

#### 4.4 Immunoassay Methods

**Method 4000:** Immunoassay

**Method 4010A:** Screening for Pentachlorophenol by Immunoassay

**Method 4015:** Screening for 2,4-Dichlorophenoxyacetic Acid by Immunoassay

**Method 4020:** Screening for Polychlorinated Biphenyls by Immunoassay

**Method 4030:** Soil Screening for Petroleum Hydrocarbons by Immunoassay

**Method 4035:** Soil Screening for Polynuclear Aromatic Hydrocarbons by Immunoassay

**Method 4040:** Soil Screening for Toxaphene by Immunoassay

**Method 4041:** Soil Screening for Chlordane by Immunoassay

**Method 4042:** Soil Screening for DDT by Immunoassay

**Method 4050:** TNT Explosives in Soil by Immunoassay

**Method 4051:** Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in Soil by Immunoassay

#### 4.5 Miscellaneous Screening Methods

**Method 3810:** Headspace

**Method 3820:** Hexadecane Extraction and Screening of Purgeable Organics

**Method 8515:** Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil

**Method 9078:** Screening Test Method for Polychlorinated Biphenyls in Soil

**Method 9079:** Screening Test Method for Polychlorinated Biphenyls in Transformer Oil

### 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 promulgated revision of the method, even though the method number does not include the appropriate letter suffix.

-----  
VOLUME ONE

SECTION C  
-----

DISCLAIMER

ABSTRACT

TABLE OF CONTENTS

METHOD INDEX AND CONVERSION TABLE

PREFACE

CHAPTER ONE, REPRINTED -- QUALITY CONTROL

- 1.0 Introduction
- 2.0 QA Project Plan
- 3.0 Field Operations
- 4.0 Laboratory Operations
- 5.0 Definitions
- 6.0 References

CHAPTER FIVE -- MISCELLANEOUS TEST METHODS

<b>Method 5050:</b>	Bomb Preparation Method for Solid Waste
<b>Method 9010C:</b>	Total and Amenable Cyanide: Distillation
<b>Method 9012B:</b>	Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation)
<b>Method 9013:</b>	Cyanide Extraction Procedure for Solids and Oils
<b>Method 9014:</b>	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
<b>Method 9020B:</b>	Total Organic Halides (TOX)
<b>Method 9021:</b>	Purgeable Organic Halides (POX)
<b>Method 9022:</b>	Total Organic Halides (TOX) by Neutron Activation Analysis
<b>Method 9023:</b>	Extractable Organic Halides (EOX) in Solids
<b>Method 9030B:</b>	Acid-Soluble and Acid-Insoluble Sulfides: Distillation
<b>Method 9031:</b>	Extractable Sulfides
<b>Method 9034:</b>	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides
<b>Method 9035:</b>	Sulfate (Colorimetric, Automated, Chloranilate)
<b>Method 9036:</b>	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
<b>Method 9038:</b>	Sulfate (Turbidimetric)
<b>Method 9056:</b>	Determination of Inorganic Anions by Ion Chromatography
<b>Method 9057:</b>	Determination of Chloride from HCl/Cl <sub>2</sub> Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography
<b>Method 9060A:</b>	Total Organic Carbon
<b>Method 9065:</b>	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)
<b>Method 9066:</b>	Phenolics (Colorimetric, Automated 4-AAP with Distillation)
<b>Method 9067:</b>	Phenolics (Spectrophotometric, MBTH with Distillation)
<b>Method 9070A:</b>	n-Hexane Extractable Material (HEM) for Aqueous Samples

<b>Method 9071B:</b>	n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples
<b>Method 9075:</b>	Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)
<b>Method 9076:</b>	Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry
<b>Method 9077:</b>	Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)
<b>Method A:</b>	Fixed End Point Test Kit Method
<b>Method B:</b>	Reverse Titration Quantitative End Point Test Kit Method
<b>Method C:</b>	Direct Titration Quantitative End Point Test Kit Method
<b>Method 9131:</b>	Total Coliform: Multiple Tube Fermentation Technique
<b>Method 9132:</b>	Total Coliform: Membrane-Filter Technique
<b>Method 9210:</b>	Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode
<b>Method 9211:</b>	Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode
<b>Method 9212:</b>	Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode
<b>Method 9213:</b>	Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion-Selective Electrode
<b>Method 9214:</b>	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode
<b>Method 9215:</b>	Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion-Selective Electrode
<b>Method 9250:</b>	Chloride (Colorimetric, Automated Ferricyanide AAI)
<b>Method 9251:</b>	Chloride (Colorimetric, Automated Ferricyanide AAI)
<b>Method 9253:</b>	Chloride (Titrimetric, Silver Nitrate)
<b>Method 9320:</b>	Radium-228

## CHAPTER SIX -- PROPERTIES

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

**Method 9310:** Gross Alpha and Gross Beta  
**Method 9315:** Alpha-Emitting Radium Isotopes

## PART II CHARACTERISTICS

### CHAPTER SEVEN -- CHARACTERISTICS INTRODUCTION AND REGULATORY DEFINITIONS

- 7.1 Ignitability
- 7.2 Corrosivity
- 7.3 Reactivity
- 7.4 Toxicity Characteristic Leaching Procedure

### CHAPTER EIGHT -- METHODS FOR DETERMINING CHARACTERISTICS

- 8.1 Ignitability
  - Method 1010A:** Pensky-Martens Closed-Cup Method for Determining Ignitability
  - Method 1020B:** Small Scale Closed Cup Method for Determining Ignitability
- 8.2 Corrosivity
  - Method 9040C:** pH Electrometric Measurement
  - Method 1110A:** Corrosivity Toward Steel
- 8.3 Reactivity
- 8.4 Toxicity
  - Method 1310B:** Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test
  - Method 1311:** Toxicity Characteristic Leaching Procedure

### 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 promulgated revision of the method, even though the method number does not include the appropriate letter suffix.

-----  
VOLUME TWO  
-----

DISCLAIMER  
ABSTRACT  
TABLE OF CONTENTS  
METHOD INDEX AND CONVERSION TABLE  
PREFACE

CHAPTER ONE, REPRINTED -- QUALITY CONTROL

- 1.0 Introduction
- 2.0 QA Project Plan
- 3.0 Field Operations
- 4.0 Laboratory Operations
- 5.0 Definitions
- 6.0 References

PART III SAMPLING

CHAPTER NINE -- SAMPLING PLAN

- 9.1 Design and Development
- 9.2 Implementation

CHAPTER TEN -- SAMPLING METHODS

- Method 0010:** Modified Method 5 Sampling Train
- Appendix A:** Preparation of XAD-2 Sorbent Resin
- Appendix B:** Total Chromatographable Organic Material Analysis
- Method 0011:** Sampling for Selected Aldehyde and Ketone Emissions from Stationary Sources
- Method 0020:** Source Assessment Sampling System (SASS)
- Method 0023A:** Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran Emissions from Stationary Sources
- Method 0030:** Volatile Organic Sampling Train
- Method 0031:** Sampling Method for Volatile Organic Compounds (SMVOC)
- Method 0040:** Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar® Bags
- Method 0050:** Isokinetic HCl/Cl<sub>2</sub> Emission Sampling Train
- Method 0051:** Midget Impinger HCl/Cl<sub>2</sub> Emission Sampling Train
- Method 0060:** Determination of Metals in Stack Emissions
- Method 0061:** Determination of Hexavalent Chromium Emissions from Stationary Sources
- Method 0100:** Sampling for Formaldehyde and Other Carbonyl Compounds in Indoor Air

## PART IV MONITORING

### CHAPTER ELEVEN -- GROUND WATER MONITORING

- 11.1 Background and Objectives
- 11.2 Relationship to the Regulations and to Other Documents
- 11.3 Revisions and Additions
- 11.4 Acceptable Designs and Practices
- 11.5 Unacceptable Designs and Practices

### CHAPTER TWELVE -- LAND TREATMENT MONITORING

- 12.1 Background
- 12.2 Treatment Zone
- 12.3 Regulatory Definition
- 12.4 Monitoring and Sampling Strategy
- 12.5 Analysis
- 12.6 References and Bibliography

### CHAPTER THIRTEEN -- INCINERATION

- 13.1 Introduction
- 13.2 Regulatory Definition
- 13.3 Waste Characterization Strategy
- 13.4 Stack-Gas Effluent Characterization Strategy
- 13.5 Additional Effluent Characterization Strategy
- 13.6 Selection of Specific Sampling and Analysis Methods
- 13.7 References

### 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 promulgated revision of the method, even though the method number does not include the appropriate letter suffix.

## 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. (Note: 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:

<b>Method 5050:</b>	Bomb Preparation Method for Solid Waste
<b>Method 9010C:</b>	Total and Amenable Cyanide: Distillation
<b>Method 9012B:</b>	Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation)
<b>Method 9013:</b>	Cyanide Extraction Procedure for Solids and Oils
<b>Method 9014:</b>	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
<b>Method 9020B:</b>	Total Organic Halides (TOX)
<b>Method 9021:</b>	Purgeable Organic Halides (POX)
<b>Method 9022:</b>	Total Organic Halides (TOX) by Neutron Activation Analysis
<b>Method 9023:</b>	Extractable Organic Halides (EOX) in Solids
<b>Method 9030B:</b>	Acid-Soluble and Acid-Insoluble Sulfides: Distillation
<b>Method 9031:</b>	Extractable Sulfides
<b>Method 9034:</b>	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides
<b>Method 9035:</b>	Sulfate (Colorimetric, Automated, Chloranilate)
<b>Method 9036:</b>	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
<b>Method 9038:</b>	Sulfate (Turbidimetric)
<b>Method 9056:</b>	Determination of Inorganic Anions by Ion Chromatography
<b>Method 9057:</b>	Determination of Chloride from HCl/Cl <sub>2</sub> Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography
<b>Method 9060A:</b>	Total Organic Carbon
<b>Method 9065:</b>	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)
<b>Method 9066:</b>	Phenolics (Colorimetric, Automated 4-AAP with Distillation)
<b>Method 9067:</b>	Phenolics (Spectrophotometric, MBTH with Distillation)
<b>Method 9070A:</b>	n-Hexane Extractable Material (HEM) for Aqueous Samples
<b>Method 9071B:</b>	n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples
<b>Method 9075:</b>	Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)

<b>Method 9076:</b>	Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry
<b>Method 9077:</b>	Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)
<b>Method A:</b>	Fixed End Point Test Kit Method
<b>Method B:</b>	Reverse Titration Quantitative End Point Test Kit Method
<b>Method C:</b>	Direct Titration Quantitative End Point Test Kit Method
<b>Method 9131:</b>	Total Coliform: Multiple Tube Fermentation Technique
<b>Method 9132:</b>	Total Coliform: Membrane-Filter Technique
<b>Method 9210:</b>	Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode
<b>Method 9211:</b>	Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode
<b>Method 9212:</b>	Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode
<b>Method 9213:</b>	Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion-Selective Electrode
<b>Method 9214:</b>	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode
<b>Method 9215:</b>	Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion-Selective Electrode
<b>Method 9250:</b>	Chloride (Colorimetric, Automated Ferricyanide AAI)
<b>Method 9251:</b>	Chloride (Colorimetric, Automated Ferricyanide AAI)
<b>Method 9253:</b>	Chloride (Titrimetric, Silver Nitrate)
<b>Method 9320:</b>	Radium-228

## METHOD 9010C

### TOTAL AND AMENABLE CYANIDE: DISTILLATION

#### 1.0 SCOPE AND APPLICATION

1.1 Method 9010 is 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. Method 9010 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. 6.8 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),  $\text{NaAsO}_2$ . Dissolve 3.2 g  $\text{NaAsO}_2$  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%),  $\text{NaOH}$ . Commercially available.

5.3.4 Acetic acid (1.6M)  $\text{CH}_3\text{COOH}$ . Dilute one part of concentrated acetic acid with 9 parts of water.

5.3.5 2,2,4-Trimethylpentane,  $\text{C}_8\text{H}_{18}$ .

5.3.6 Hexane,  $\text{C}_6\text{H}_{14}$ .

5.3.7 Chloroform,  $\text{CHCl}_3$ .

#### 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),  $\text{NaOH}$ . Dissolve 50 g of  $\text{NaOH}$  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  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  in 100 mL of water. While stirring, add 250 mL of glacial acetic acid,  $\text{CH}_3\text{COOH}$ . 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),  $\text{H}_2\text{SO}_4$ . Slowly and carefully add 500 mL of concentrated  $\text{H}_2\text{SO}_4$  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  $\text{CN}^-$ , the standard deviations for precision were  $\pm 0.005$  to  $\pm 0.094$ , respectively. In a single laboratory using mixed industrial and domestic waste samples at concentrations of 0.28 and 0.62 mg/L  $\text{CN}^-$ , 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"; ATSM: Philadelphia, PA, 1985.; D1193-77.
2. 1982 Annual Book ASTM Standards, Part 19; "Standard Test Methods for Cyanide in Water"; ASTM: Philadelphia, PA, 1982; 2036-82.
3. Bark, L.S.; Higson, H.G. Talanta 1964, 2, 471-479.
4. Britton, P.; Winter, J.; Kroner, R.C. "EPA Method Study 12, Cyanide in Water"; final report to the U.S. Environmental Protection Agency. National Technical Information Service: Springfield, VA, 1984; PB80-196674.

5. Casey, J.P.; Bright, J.W.; Helms, B.D. "Nitrosation Interference in Distillation Tests for Cyanide"; Gulf Coast Waste Disposal Authority: Houston, Texas.
6. Egekeze, J.O.; Oehne, F.W. J. Anal. Toxicology 1979, 3, 119.
7. Elly, C.T. J. Water Pollution Control Federation 1968, 40, 848-856.
8. Fuller, W. Cyanide in the Environment; Van Zyl, D., Ed.; Proceedings of Symposium; December, 1984.
9. Gottfried, G.J. "Precision, Accuracy, and MDL Statements for EPA Methods 9010, 9030, 9060, 7520, 7521, 7550, 7551, 7910, and 7911"; final report to the U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory. Biospheric: Cincinnati, OH, 1984.
10. Methods for Chemical Analysis of Water and Wastes; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1983; EPA-600/4-79-020.
11. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.
12. Standard Methods for the Examination of Water and Wastewater, 18th ed.; Greenberg, A.E.; Clesceri, L.S.; Eaton, A.D.; Eds.; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, DC, 1992.
13. Umaña, M.; Beach, J.; Sheldon, L. "Revisions to Method 9010"; final report to the U.S. Environmental Protection Agency. Office of Solid Waste. Research Triangle Institute: Research Triangle Park, NC, 1986.
14. Umaña, M.; Sheldon, L. "Interim Report: Literature Review"; interim report to the U.S. Environmental Protection Agency. Office of Solid Waste. Research Triangle Institute: Research Triangle Park, NC, 1986.

FIGURE 1.  
APPARATUS FOR CYANIDE DISTILLATION

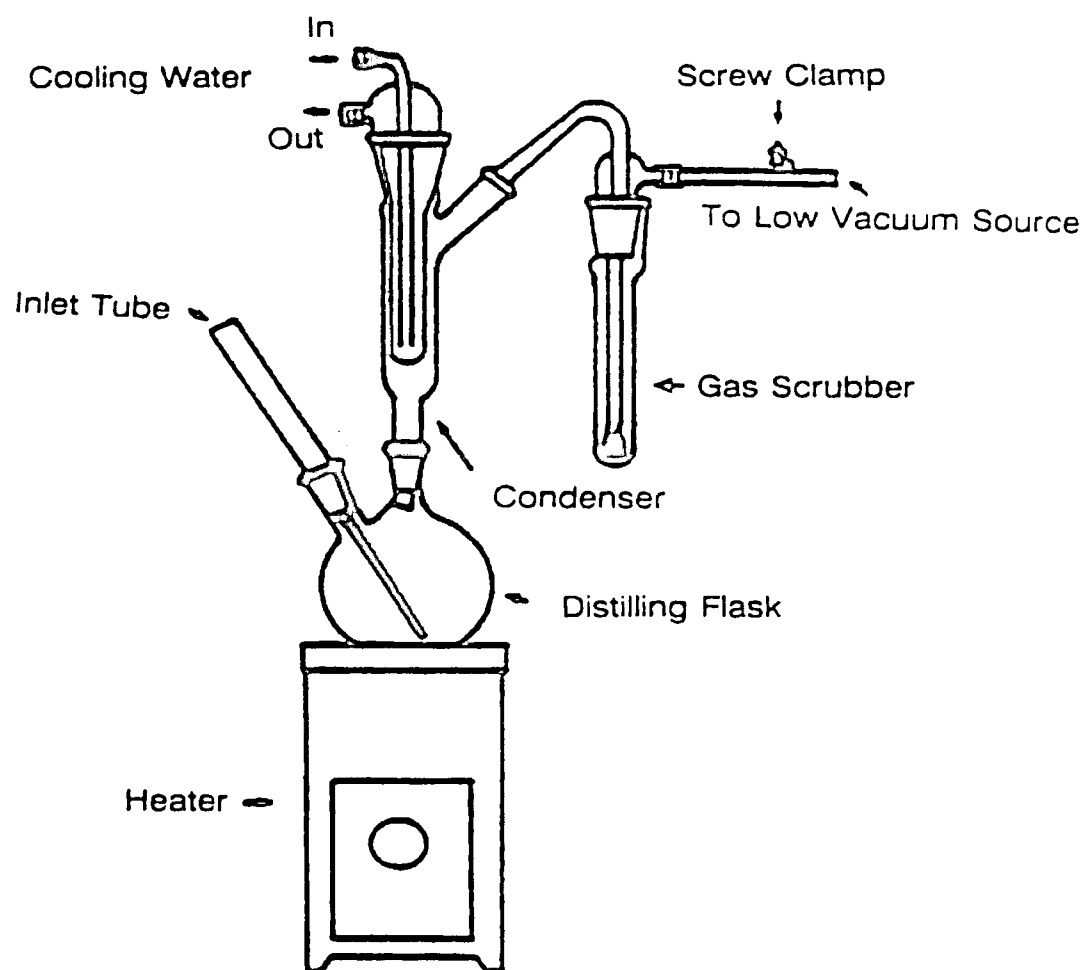
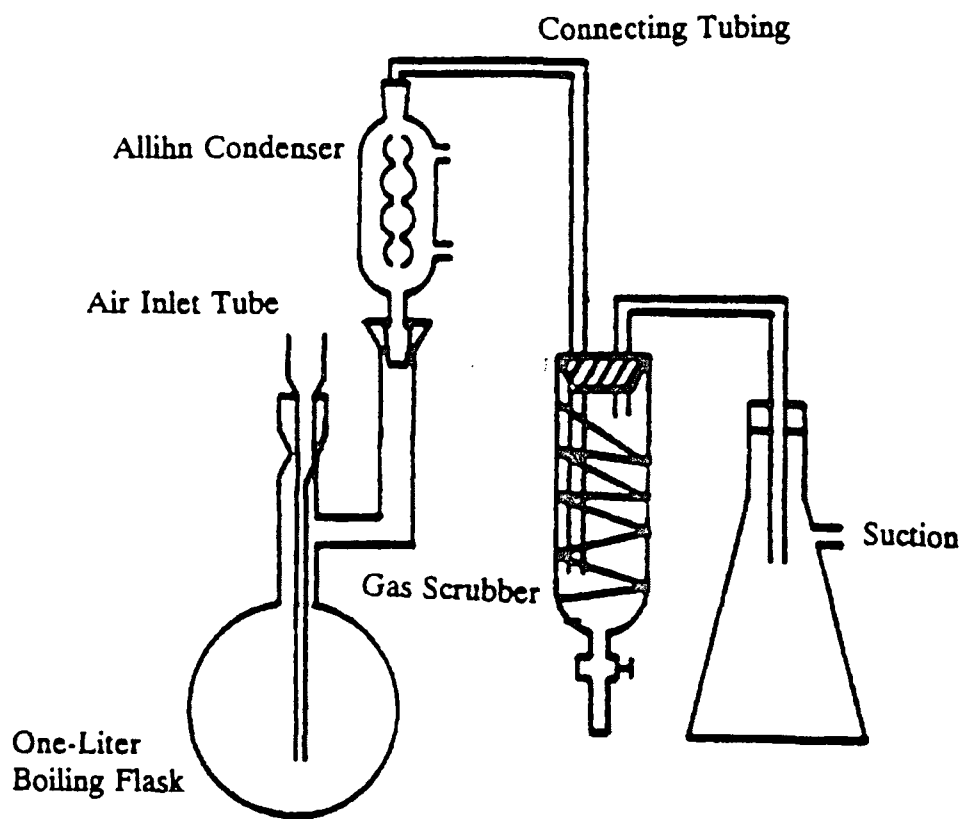
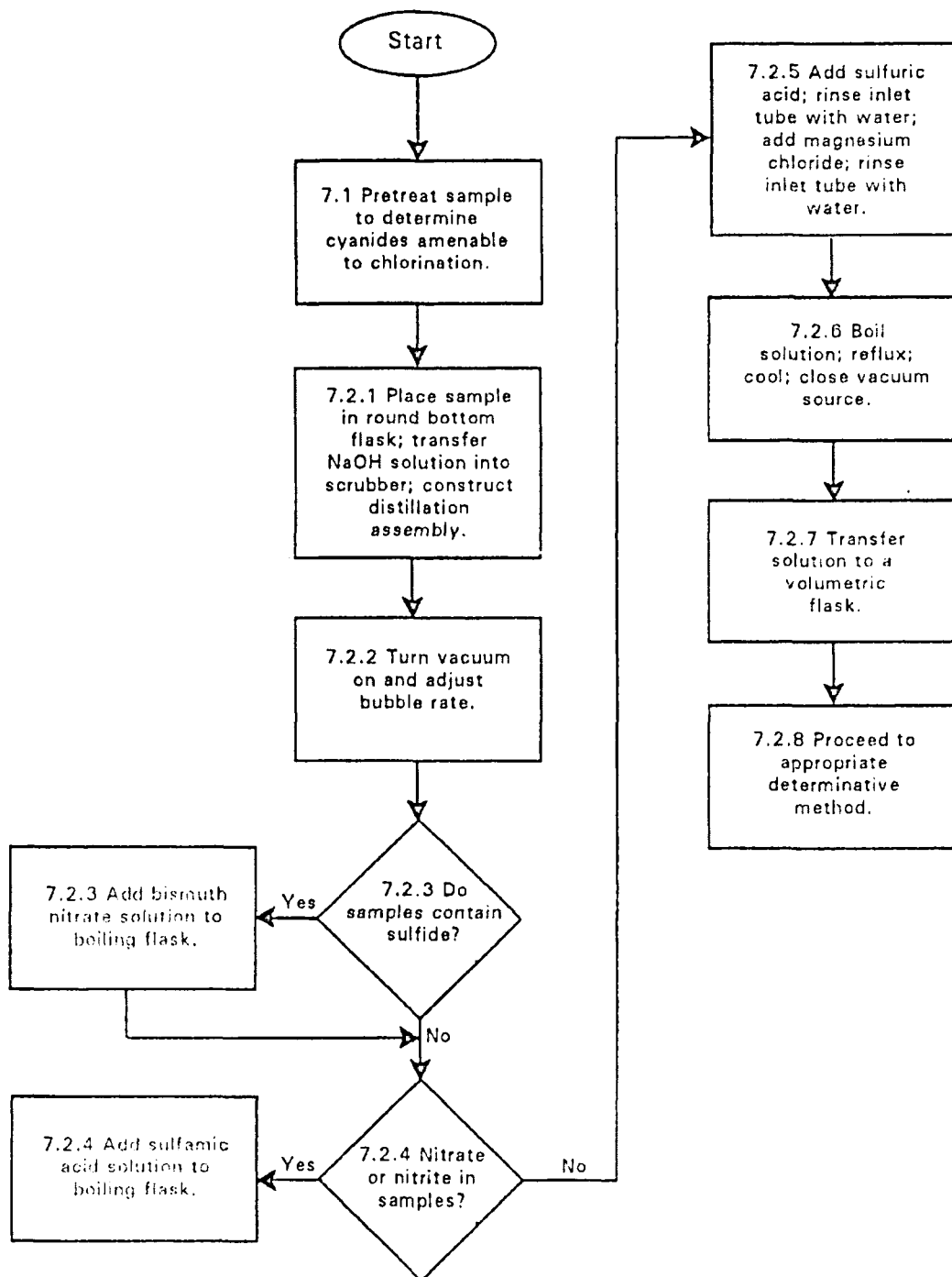


FIGURE 2.  
APPARATUS FOR CYANIDE DISTILLATION



# 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 Method 9012 is used to determine the concentration of inorganic cyanide (CAS Registry Number 57-12-5) in wastes or leachate. The 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 Method 9012.

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 sulfami 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),  $\text{NaAsO}_2$ . Dissolve 3.2 g  $\text{NaAsO}_2$  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%),  $\text{NaOH}$ . Commercially available.

5.3.4 Acetic acid (1.6M)  $\text{CH}_3\text{COOH}$ . Dilute one part of concentrated acetic acid with 9 parts of water.

5.3.5 2,2,4-Trimethylpentane,  $\text{C}_8\text{H}_{18}$ .

5.3.6 Hexane,  $\text{C}_6\text{H}_{14}$ .

5.3.7 Chloroform,  $\text{CHCl}_3$ .

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),  $\text{NaOH}$ . Dissolve 50 g of  $\text{NaOH}$  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  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  in 100 mL of water. While stirring, add 250 mL of glacial acetic acid,  $\text{CH}_3\text{COOH}$ . 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),  $\text{H}_2\text{SO}_4$ . Slowly and carefully add 500 mL of concentrated  $\text{H}_2\text{SO}_4$  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 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 minutes. 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 <sup>-</sup> )	Concentration (µg CN <sup>-</sup> /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  $\mu\text{g/L}$  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  $\mu\text{g/L}$ . 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

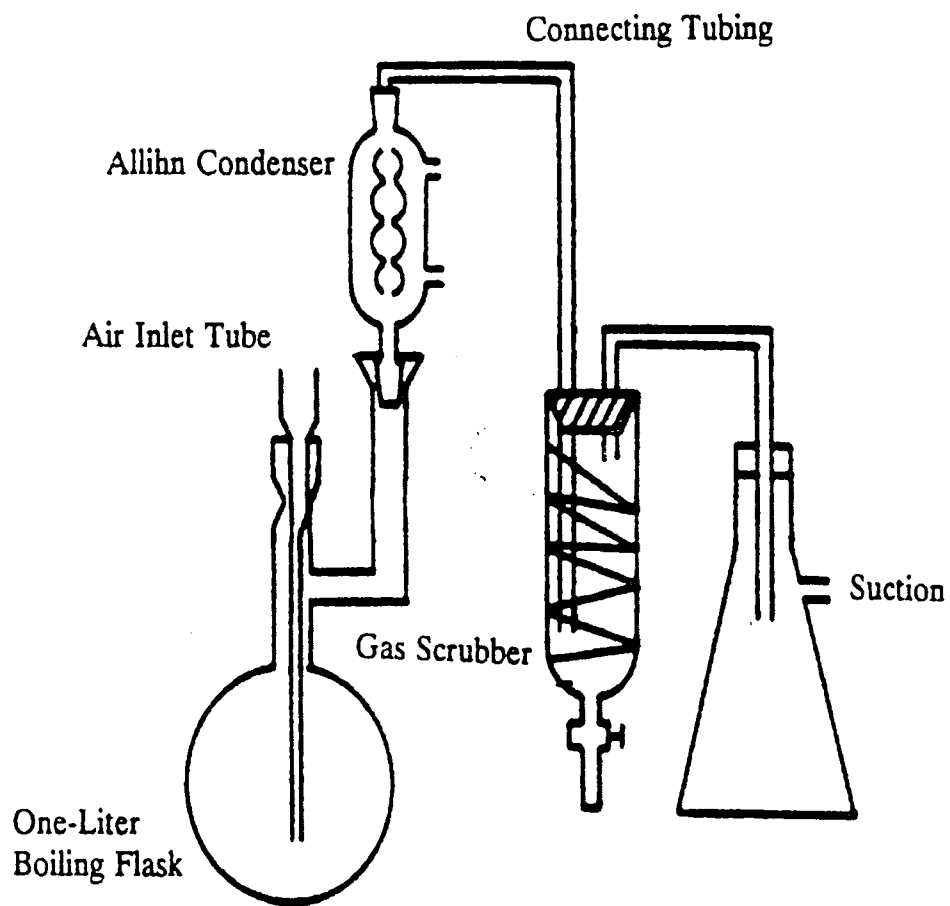
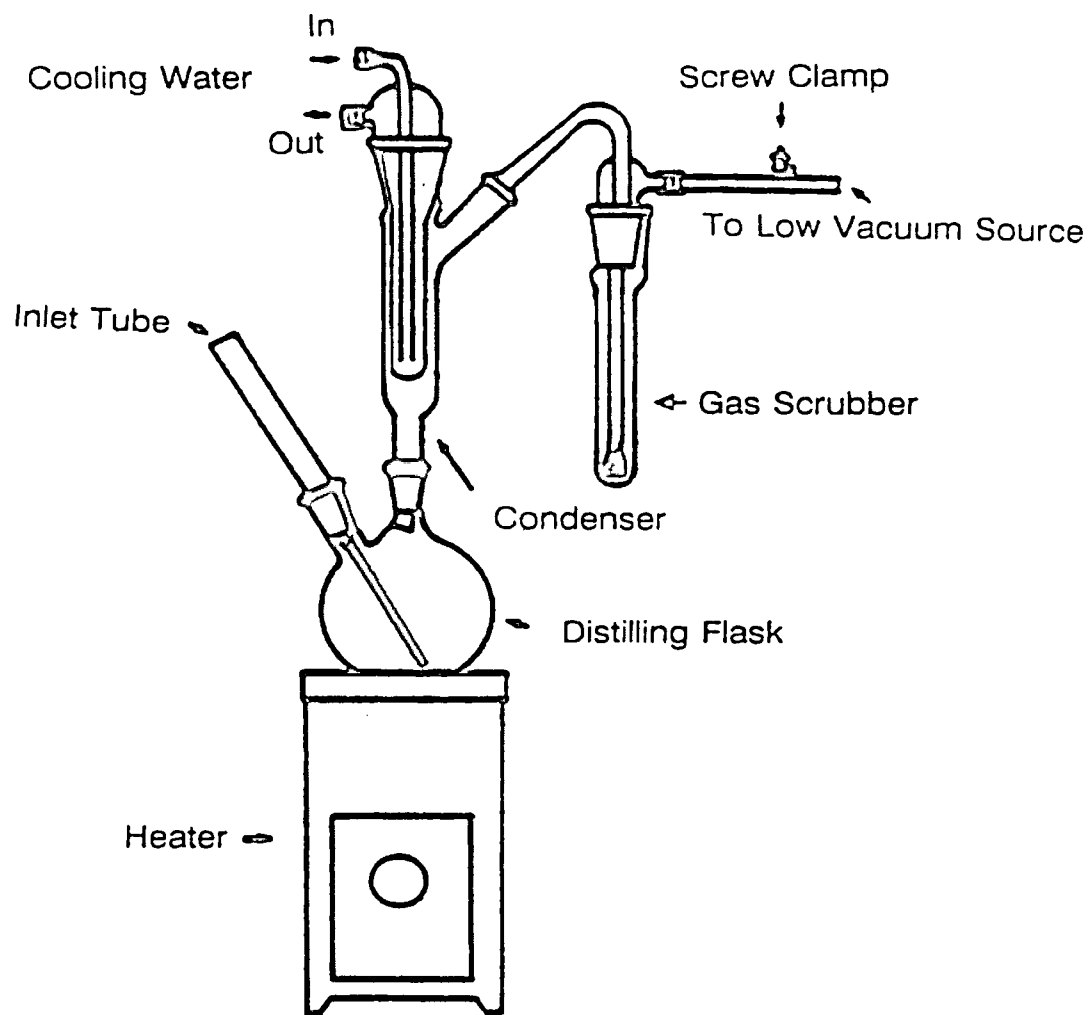


Figure 2. Cyanide Distillation Apparatus



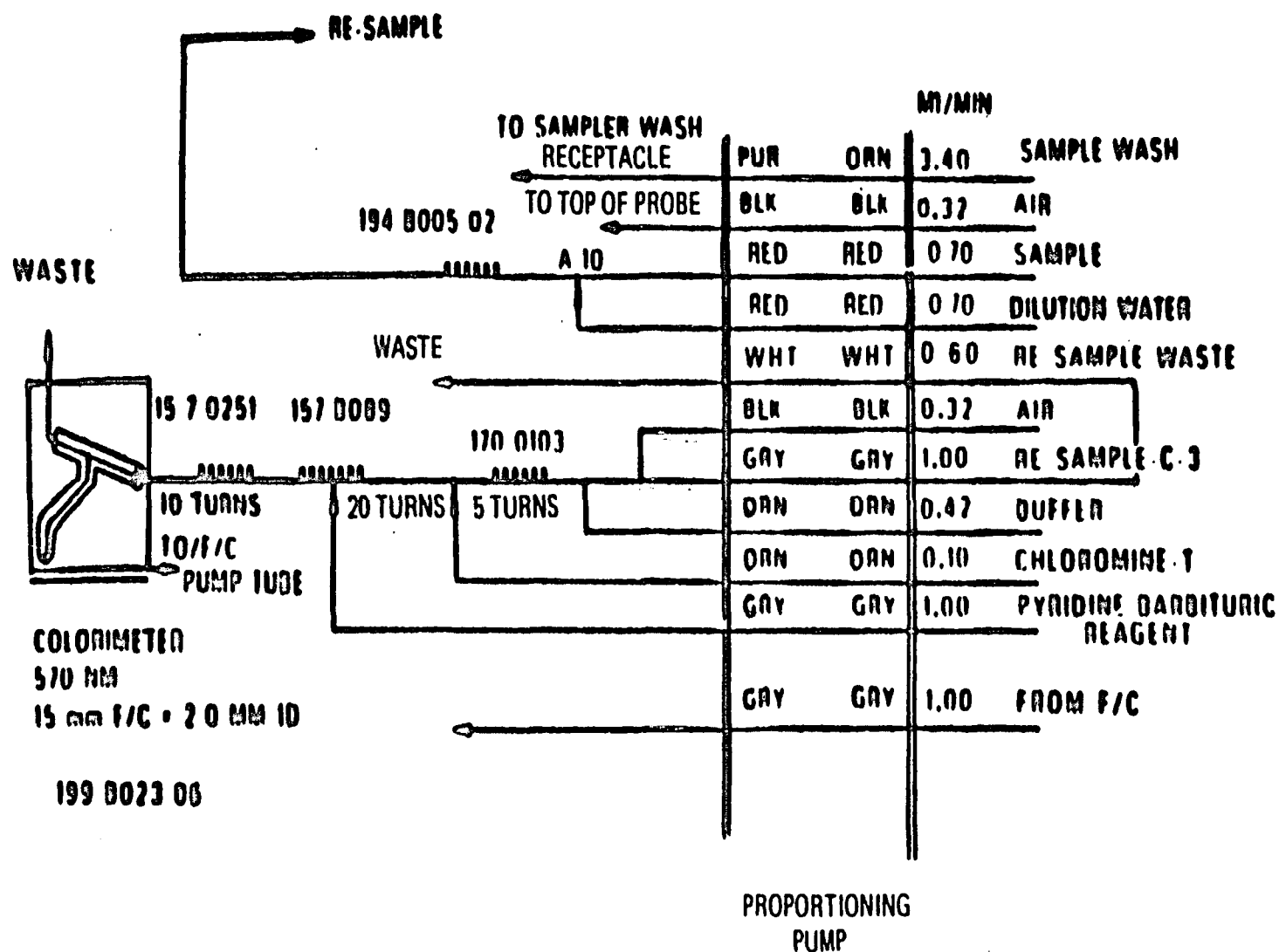
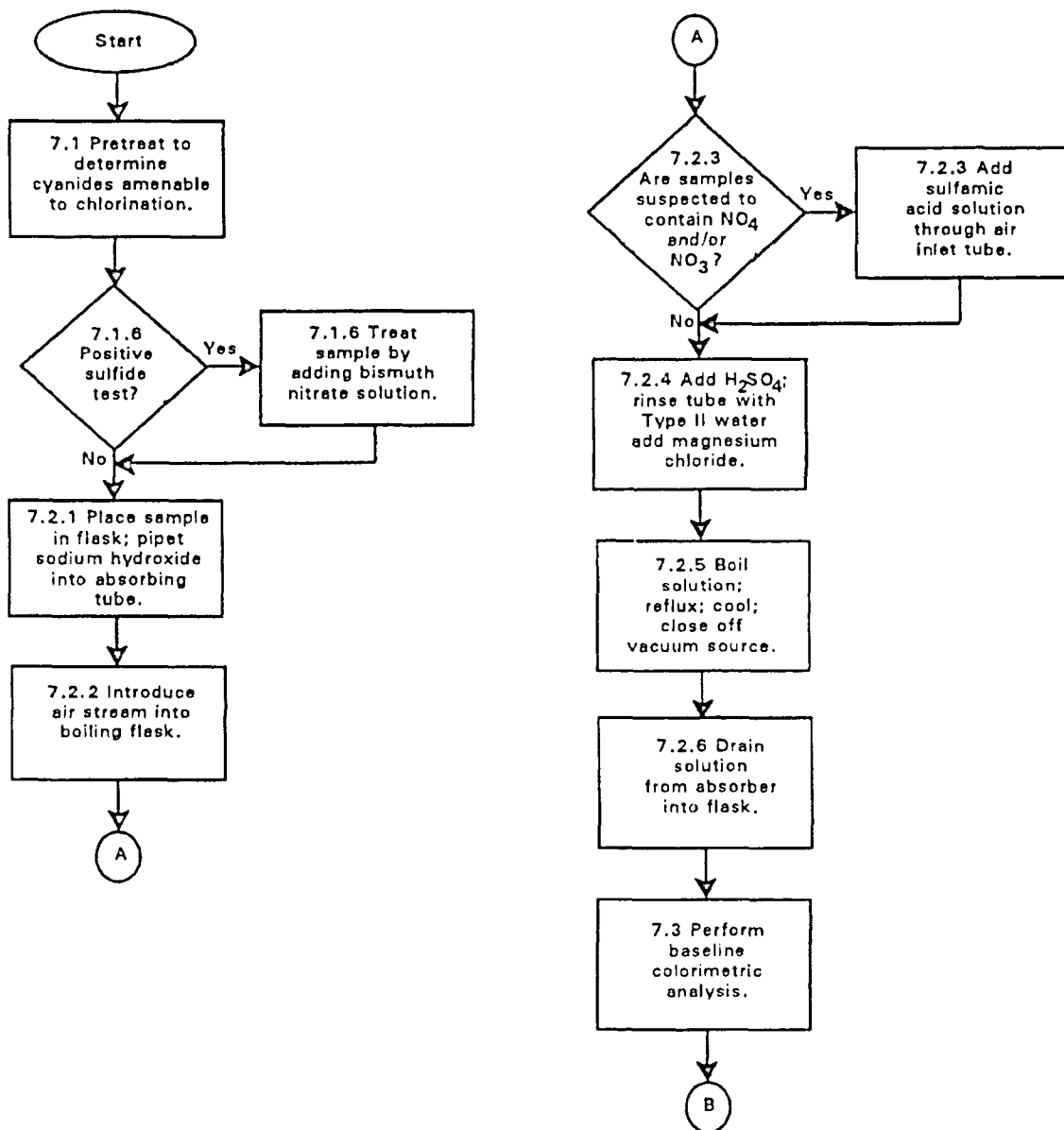


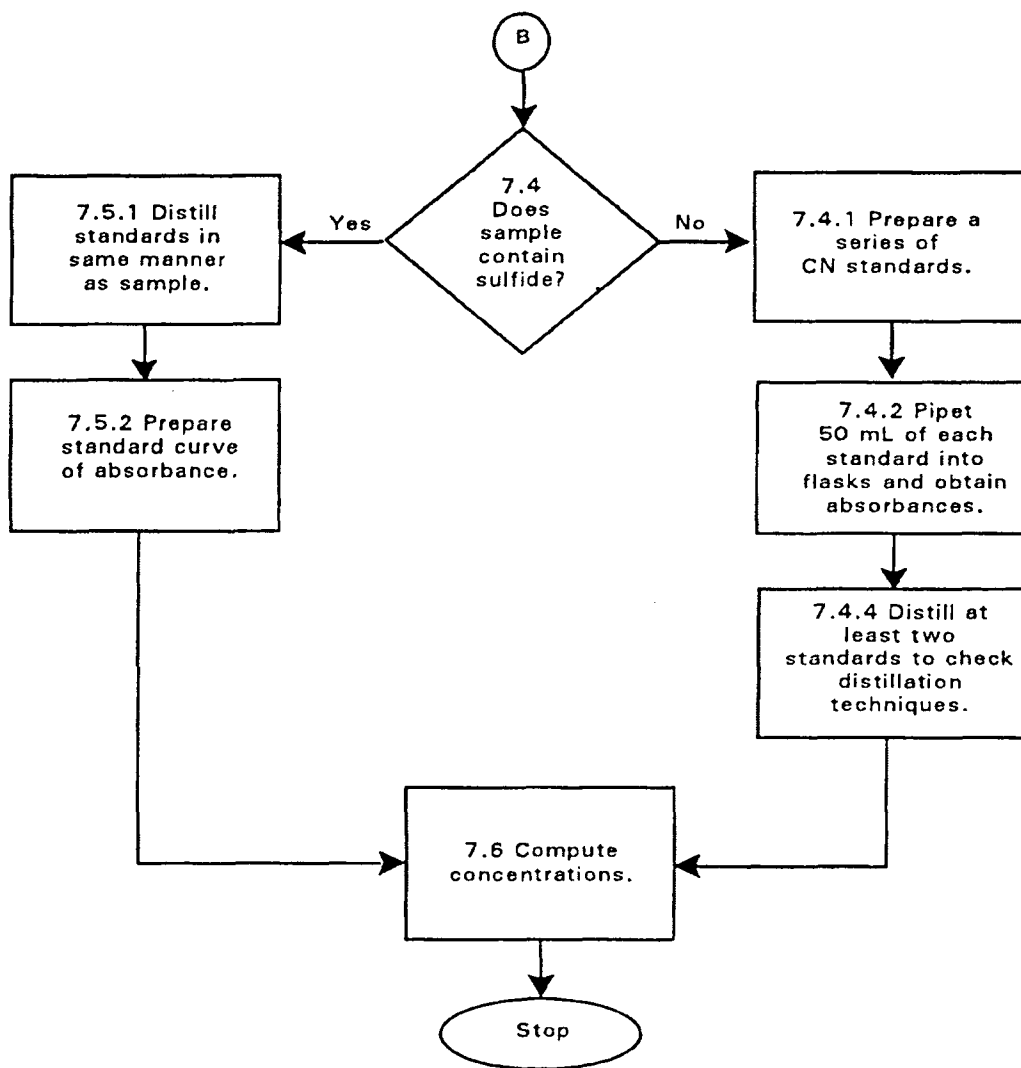
Figure 3. Cyanide Manifold AA11

# 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 Method 9060 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 Sections 2.0 and 3.0.

1.2 Method 9060 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 ( $\text{CO}_2$ ) by either catalytic combustion or wet chemical oxidation. The  $\text{CO}_2$  formed is then either measured directly by an infrared detector or converted to methane ( $\text{CH}_4$ ) and measured by a flame ionization detector. The amount of  $\text{CO}_2$  or  $\text{CH}_4$  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 Method 9060 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  $\text{CO}_2$  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 CO<sub>2</sub>.

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  $\text{H}_2\text{SO}_4$ .

## 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.

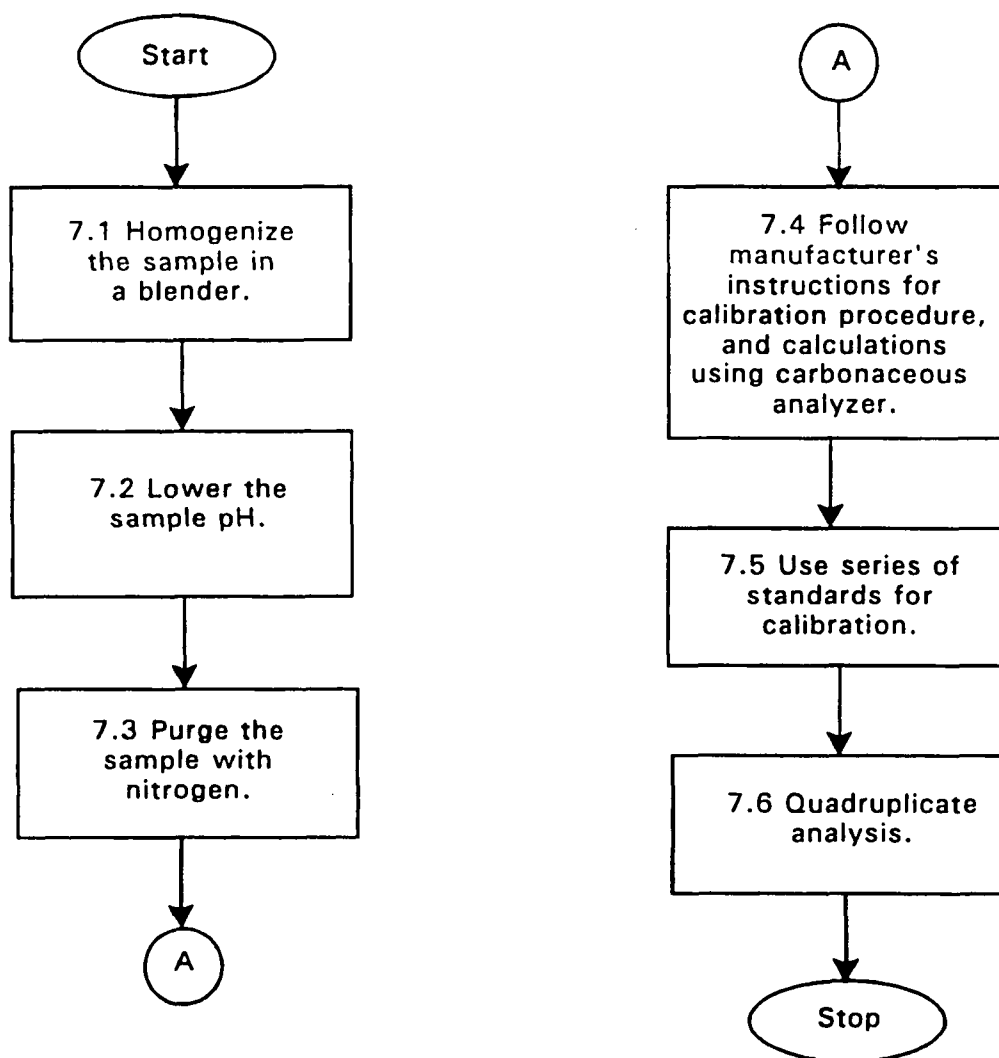
## 9.0 METHOD PERFORMANCE

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, Publication No. EPA-821-R-98-002, for this method procedure.

## CHAPTER SIX

### PROPERTIES

This chapter addresses procedures for method-defined parameters, where the analytical result is wholly dependant on the process used to make the measurement. 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 Chapter Six:

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

## METHOD 9045D

### SOIL AND WASTE pH

#### 1.0 SCOPE AND APPLICATION

1.1 Method 9045 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 minutes. 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 hour 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 minutes. 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 minutes 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

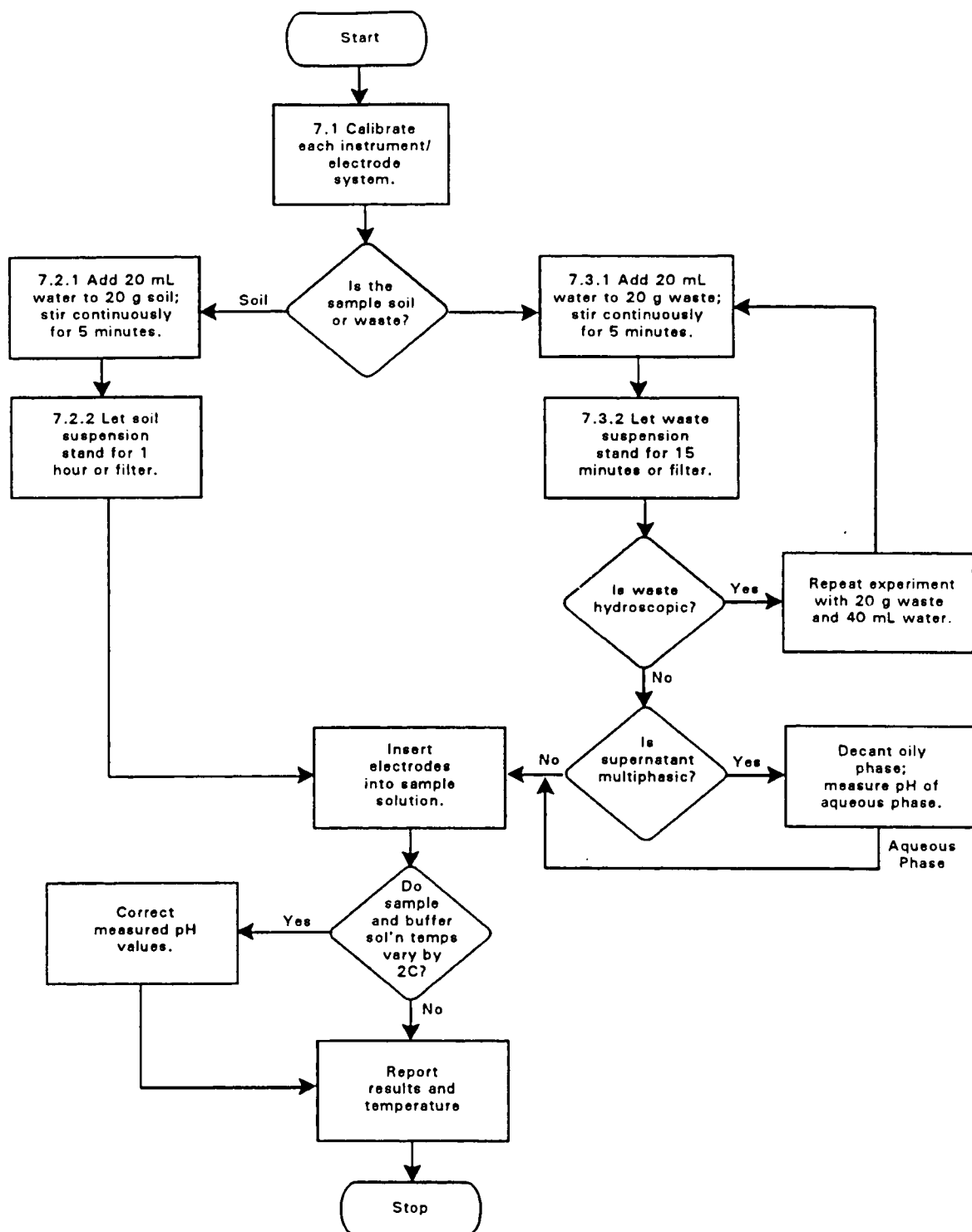
9.1 No data provided.

## 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 overflow 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, 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.

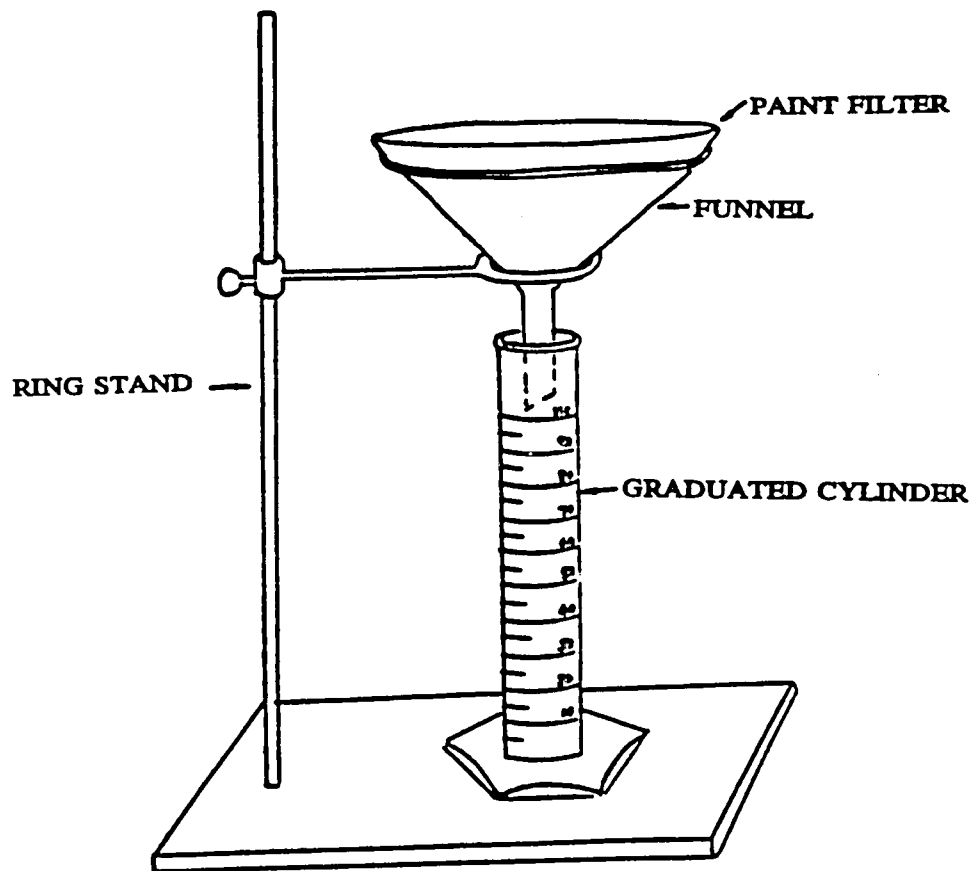
## 9.0 METHOD PERFORMANCE

9.1 No data provided.

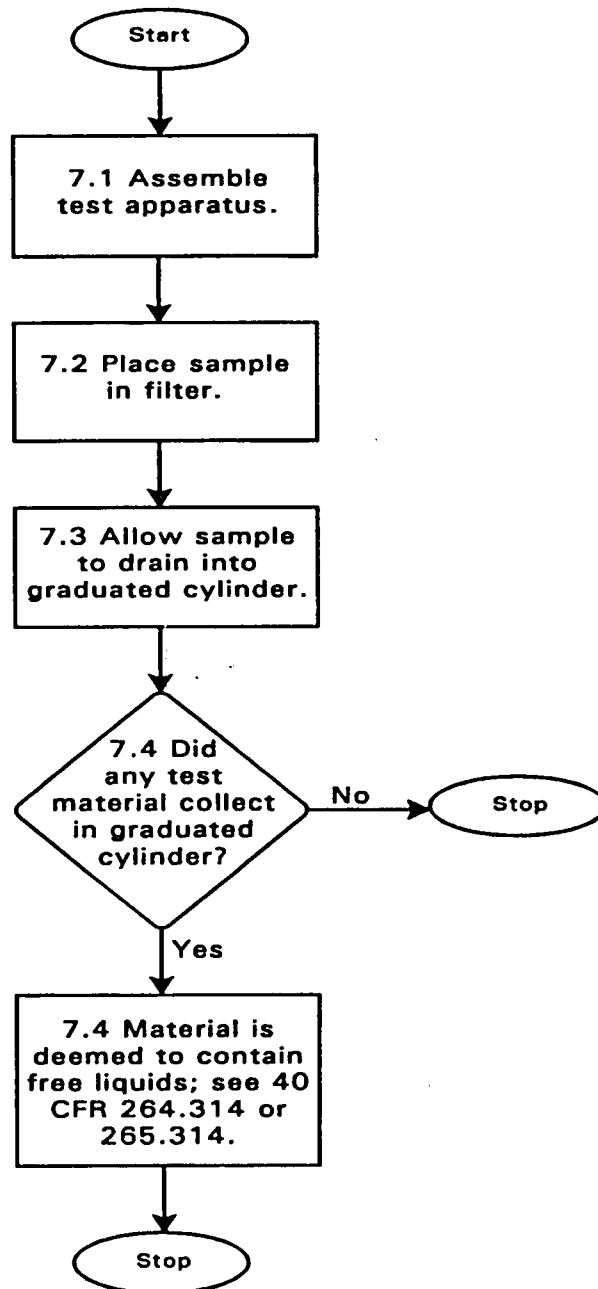
## **10.0 REFERENCES**

**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

This section discusses the hazardous characteristic of ignitability. The regulatory background of this characteristic is summarized, and the regulatory definition of ignitability is presented. The two testing methods associated with this characteristic, Methods 1010 and 1020, can be found in Chapter Eight.

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.

#### 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  $18 \pm 2$  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

The TCLP consists of five steps (refer to Figure 3):

##### 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  $\mu\text{m}$  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  $\text{cm}^2$ , 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  $18 \pm 2$  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  $\mu\text{m}$  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, 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 does 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 <sup>1</sup>
m-Cresol	200.0 <sup>1</sup>
p-Cresol	200.0 <sup>1</sup>
Cresol	200.0 <sup>1</sup>
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 <sup>2</sup>
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13 <sup>2</sup>
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 <sup>2</sup>
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

<sup>1</sup>If 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.

<sup>2</sup>Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

FIGURE 3.

TOXICITY CHARACTERISTIC LEACHING PROCEDURE FLOWCHART

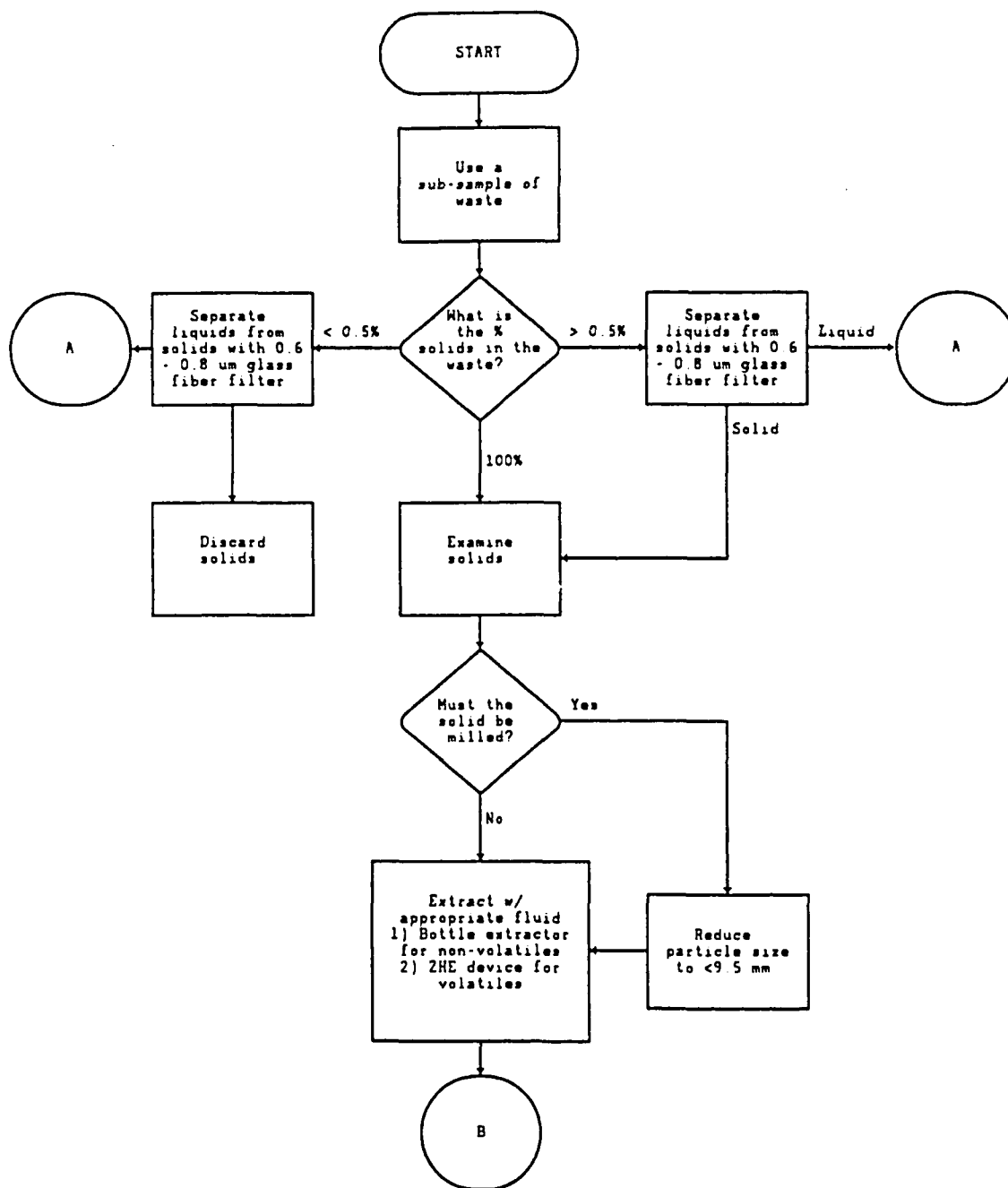
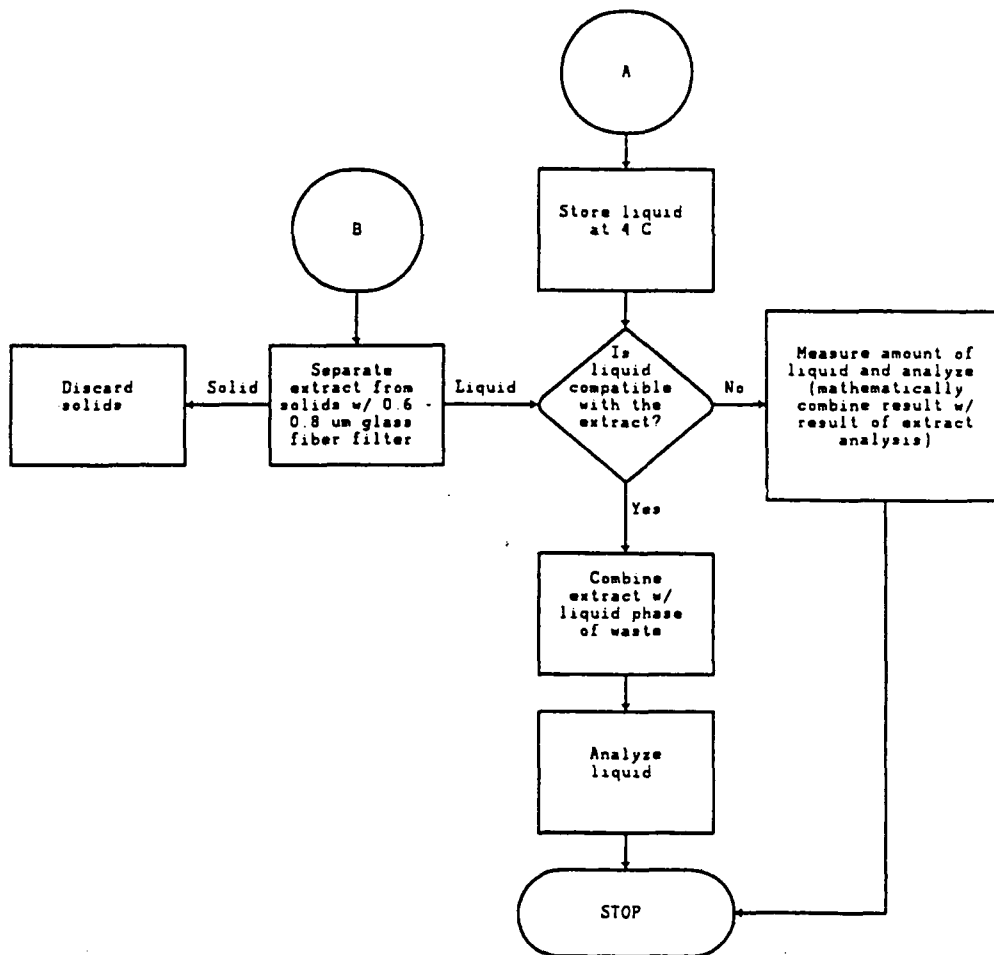


FIGURE 3 (continued)



## CHAPTER EIGHT

### METHODS FOR DETERMINING CHARACTERISTICS

This chapter addresses procedures for 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.

## 8.1 Ignitability

This chapter addresses procedures for 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. 8.1 of this chapter:

<b>Method 1010A:</b>	Pensky-Martens Closed-Cup Method for Determining Ignitability
<b>Method 1020B :</b>	Small Scale Closed-Cup Method for Determining Ignitability

## METHOD 1010A

### PENSKY-MARTENS CLOSED-CUP METHOD FOR DETERMINING IGNITABILITY

#### 1.0 SCOPE AND APPLICATION

1.1 Method 1010 uses the Pensky-Martens closed-cup tester to determine the flash point of liquids including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids can also be tested using this method.

1.2 This method is one of two method options required by 40 CFR 261.21(a)(1) in the determination of the hazardous waste ignitability characteristic. Method 1020 is the other method option.

#### 2.0 SUMMARY OF METHOD

2.1 The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

2.2 For complete instructions on how to conduct a test by this method, see Reference 4 below, "D 93-99c, Standard Test Methods for Flash-Point by Pensky-Martens Closed Cup Tester."

#### 3.0 METHOD PERFORMANCE

3.1 The Pensky-Martens and Setaflash Closed Testers (Revision 0 of Method 1020) were evaluated using five industrial waste mixtures and p-xylene. The results of these studies are shown below in °F along with other data. The sample footnote numbers refer to the source documents identified under Sec. 4.0 of this method.

<u>Sample</u>	<u>Pensky-Martens (°F)</u>	<u>Setaflash (°F)</u>
1 <sup>2</sup>	143.7 ± 1.5	139.3 ± 2.1
2 <sup>2</sup>	144.7 ± 4.5	129.7 ± 0.6
3 <sup>2</sup>	93.7 ± 1.5	97.7 ± 1.2
4 <sup>2</sup>	198.0 ± 4.0	185.3 ± 0.6
5 <sup>2</sup>	119.3 ± 3.1	122.7 ± 2.5
p-xylene <sup>2</sup>	81.3 ± 1.1	79.3 ± 0.6
p-xylene <sup>3</sup>	77.7 ± 0.5 <sup>a</sup>	--
Tanker oil	125, 135	--
Tanker oil	180, 180	--
Tanker oil	110, 110	--
DIBK/xylene	102 ± 4 <sup>b</sup>	107

<sup>a</sup>12 determinations over five-day period. <sup>b</sup>75/25 v/v analyzed by four laboratories.

#### 4.0 REFERENCES

1. D 93-80, Test Methods for Flash Point by Pensky-Martens Closed Tester, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, 04.09, 1986.
2. Umana, M., Gutknecht, W., Salmons, C., et al., Evaluation of Ignitability Methods (Liquids), EPA/600/S4-85/053, 1985.
3. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.
4. D 93-99c, Standard Test Methods for Flash-Point by Pensky-Martens Closed Cup Tester, originally published by the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Available from Global Engineering Documents, 15 Iverness Way East, Englewood, CO 80112, 1-800-854-7179, <http://global.ihs.com>

## METHOD 1020B

### SMALL SCALE CLOSED-CUP METHOD FOR DETERMINING IGNITABILITY

#### 1.0 SCOPE AND APPLICATION

1.1 Method 1020 makes use of the small scale closed-cup apparatus (formerly the Setaflash closed tester) to determine the flash point of liquids that have flash points between 0° and 110 °C (32 and 230 °F) and viscosities lower than 150 stokes at 25 °C (77 °F).

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

1.3 This method is one of two method options required in 40 CFR 261.21(a)(1) for the determination of the hazardous waste ignitability characteristic. Method 1010 (Pensky-Martens Closed-Cup Method for Determining Ignitability) is the other method option. Liquids that tend to form surface films under test conditions or those that contain non-filterable suspended solids should be tested for the hazardous waste ignitability characteristic using Method 1010.

#### 2.0 SUMMARY OF METHOD

2.1 By means of a syringe, 2-mL of sample is introduced through a leak-proof entry port into the tightly closed small scale tester or directly into the cup which has been brought to within 3 °C (5 °F) below the expected flash point.

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

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

2.4 For the complete instructions on how to conduct the ignitability test by this method, see Reference 4 below, "D 3278-96, Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus."

#### 3.0 METHOD PERFORMANCE

See Method 1010.

#### 4.0 REFERENCES

1. D 3278-78, Test Method for Flash Point of Liquids by Setaflash Closed Tester, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

2. Umana, M., Gutknecht, W., Salmons, C., et al., Evaluation of Ignitability Methods (Liquids), EPA/600/S4-85/053, 1985.
3. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.
4. D 3278-96, Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA. <http://www.astm.org/>. Also available from Global Engineering Documents, 15 Iverness Way East, Englewood, CO 80112, 1-800-854-7179, <http://global.ihs.com>.

## 8.2 Corrosivity

This chapter addresses procedures for 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:

<b>Method 9040C:</b>	pH Electrometric Measurement
<b>Method 1110A:</b>	Corrosivity Toward Steel

## METHOD 9040C

### pH ELECTROMETRIC MEASUREMENT

#### 1.0 SCOPE AND APPLICATION

1.1 Method 9040 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  $25 \pm 1^\circ\text{C}$  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  $2^{\circ}\text{C}$  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

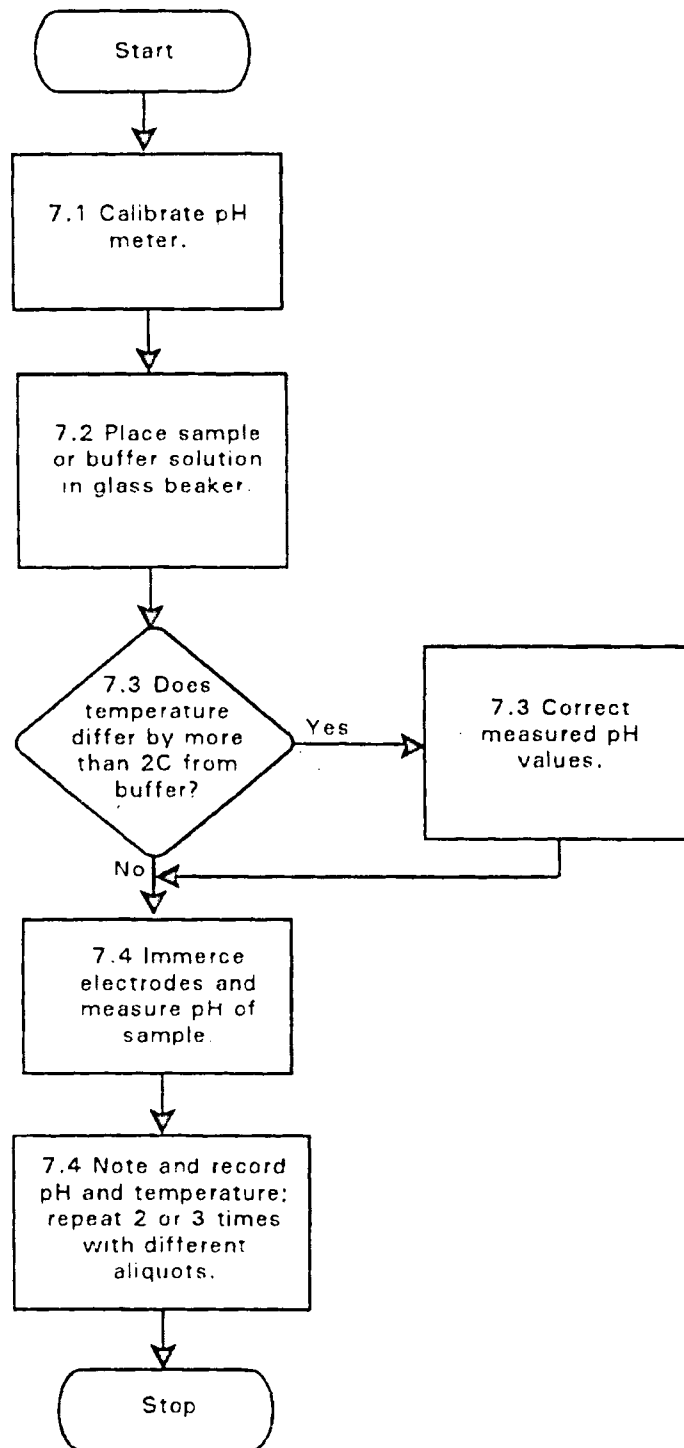
9.1 Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing exact increments of hydrogen-hydroxyl ions, with the following results:

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 Method 1110 is used to measure 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 (0.0001 in.) or 2-3 mg/cm<sup>2</sup> 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/cm<sup>2</sup>.

## 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 (SnCl<sub>2</sub>).

5.5 Antimony chloride (SbCl<sub>3</sub>).

## 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 Paragraph 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 <sub>2</sub> + 20 g/L SbCl <sub>3</sub>	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 <sub>2</sub> SO <sub>4</sub>
Anode:	Carbon or lead
Cathode:	Steel coupon
Cathode current density:	20 amp/cm <sup>2</sup> (129 amp/in. <sup>2</sup> )
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:

$$\text{Corrosion Rate (mmpy)} = \frac{\text{weight loss} \times 11.145}{\text{area} \times \text{time}}$$

where: weight loss is in milligrams,  
area is in square centimeters,  
time is in hours, 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.

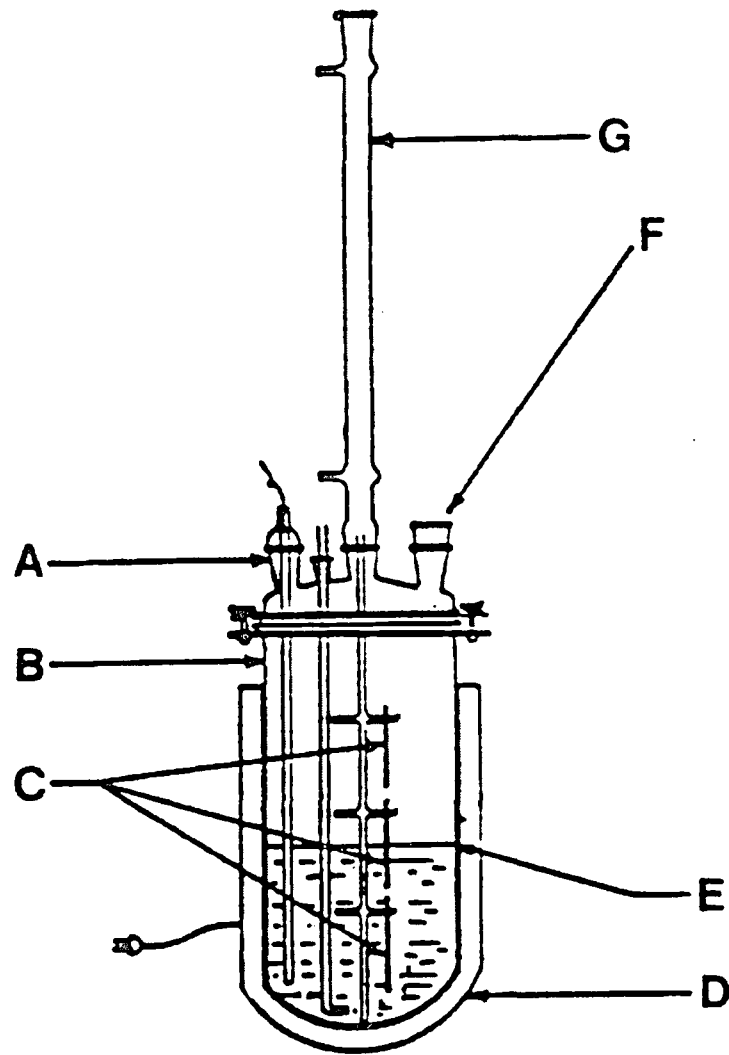
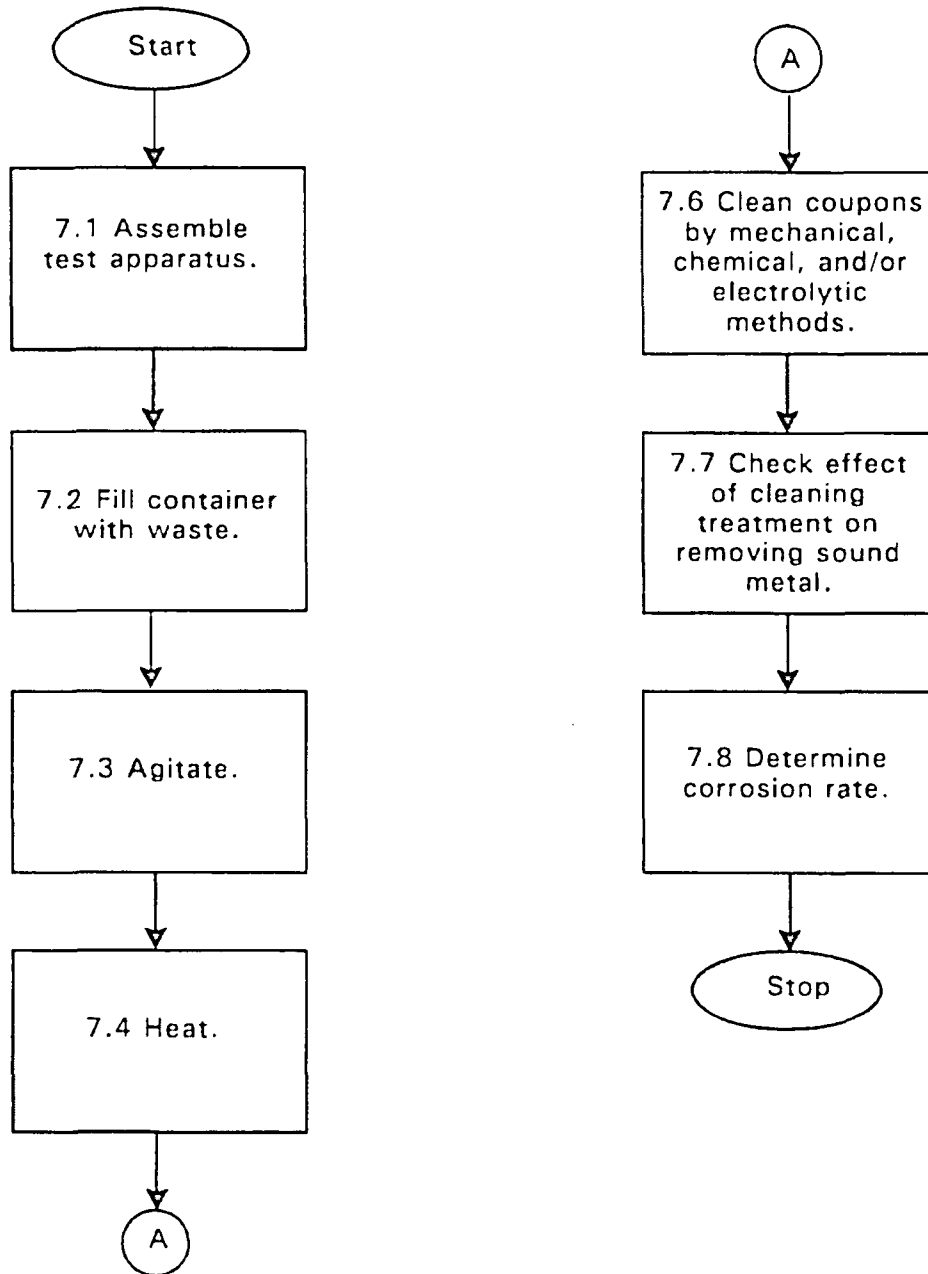


Figure 1. Typical resin flask that can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of the flask top is such that more sophisticated apparatus can be added as required by the specific test being conducted. A = thermowell, B = resin flask, C = specimens 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 Reactivity

There are no required SW-846 methods for the determination of the characteristic of reactivity.



## 8.4 Toxicity

This chapter addresses procedures for 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. 8.4 of this chapter:

- |                      |  |
|----------------------|--|
| <b>Method 1310B:</b> | Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test |
| <b>Method 1311:</b>  | 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  $5 \pm 0.2$ , 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 1-3 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- $\mu$ 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/cm<sup>3</sup> (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),  $\text{CH}_3\text{COOH}$ . 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  $\pm 0.01$  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- $\mu$ 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  $\pm 0.01$  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:

$$W = W_r - W_t$$

where :

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

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

$W_t$  = 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  $5.0 \pm 0.2$  by slowly adding 0.5N acetic acid. If the pH is  $\leq 5.0$ , 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  $5.0 \pm 0.2$ . 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  $5.0 \pm 0.2$ . 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  $5.0 \pm 0.2$  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 <sup>a</sup>
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

<sup>a</sup> 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

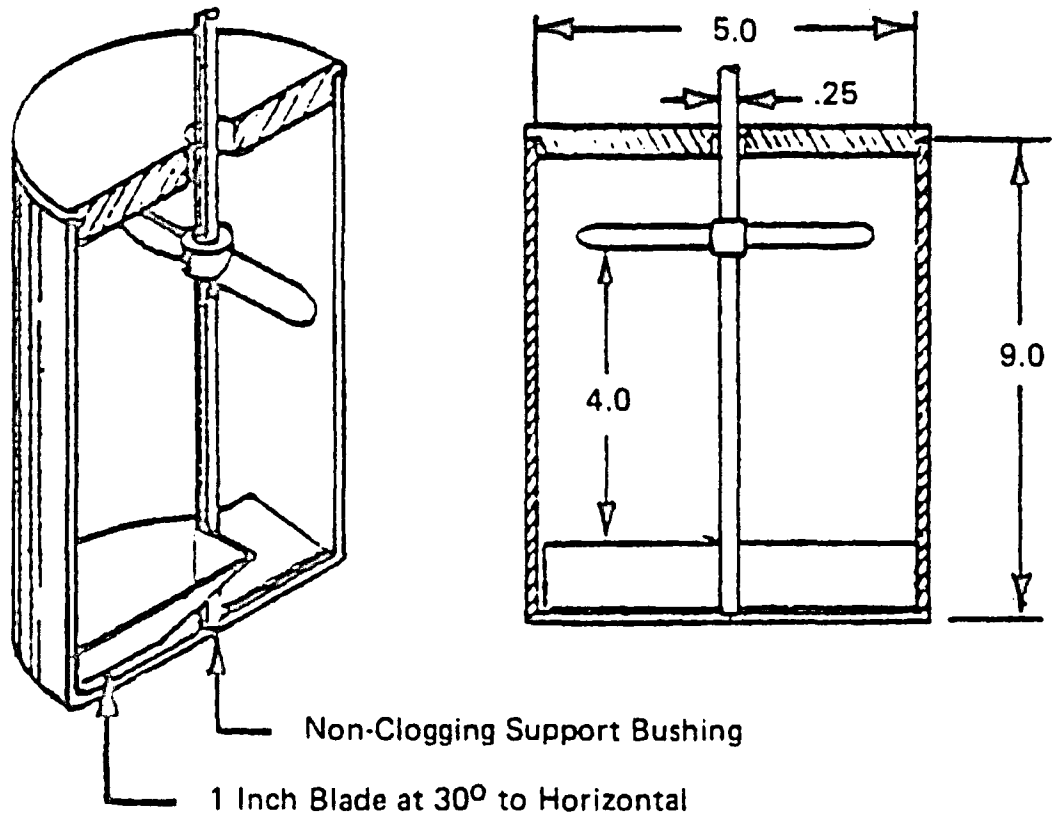


FIGURE 2.  
ROTARY EXTRACTOR

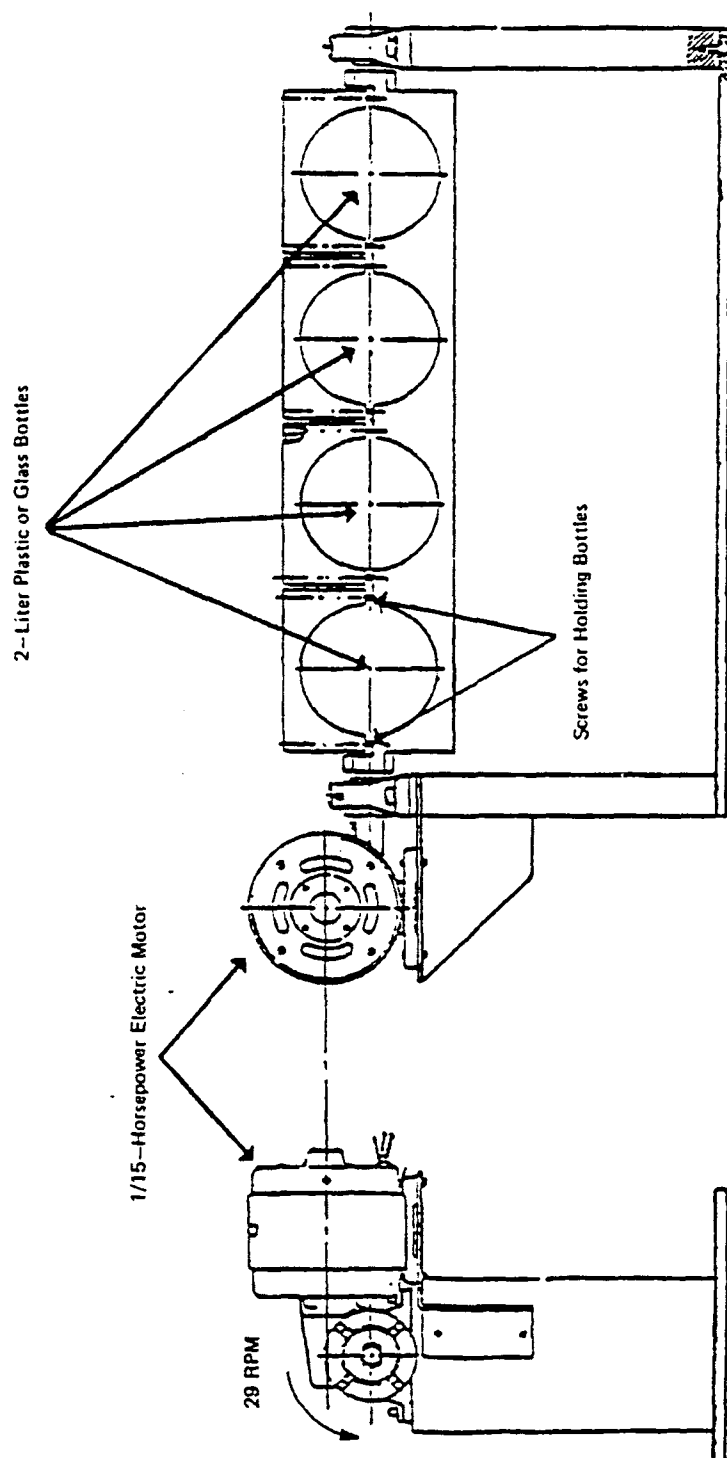


FIGURE 3.  
EPRI EXTRACTOR

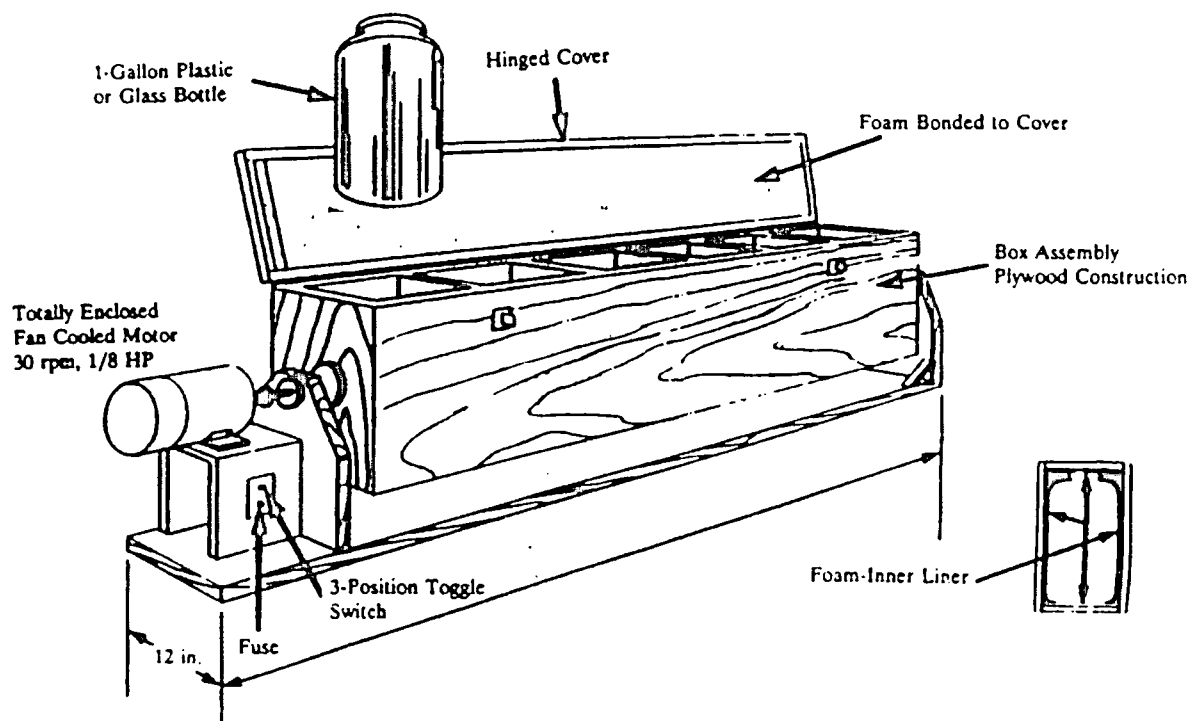
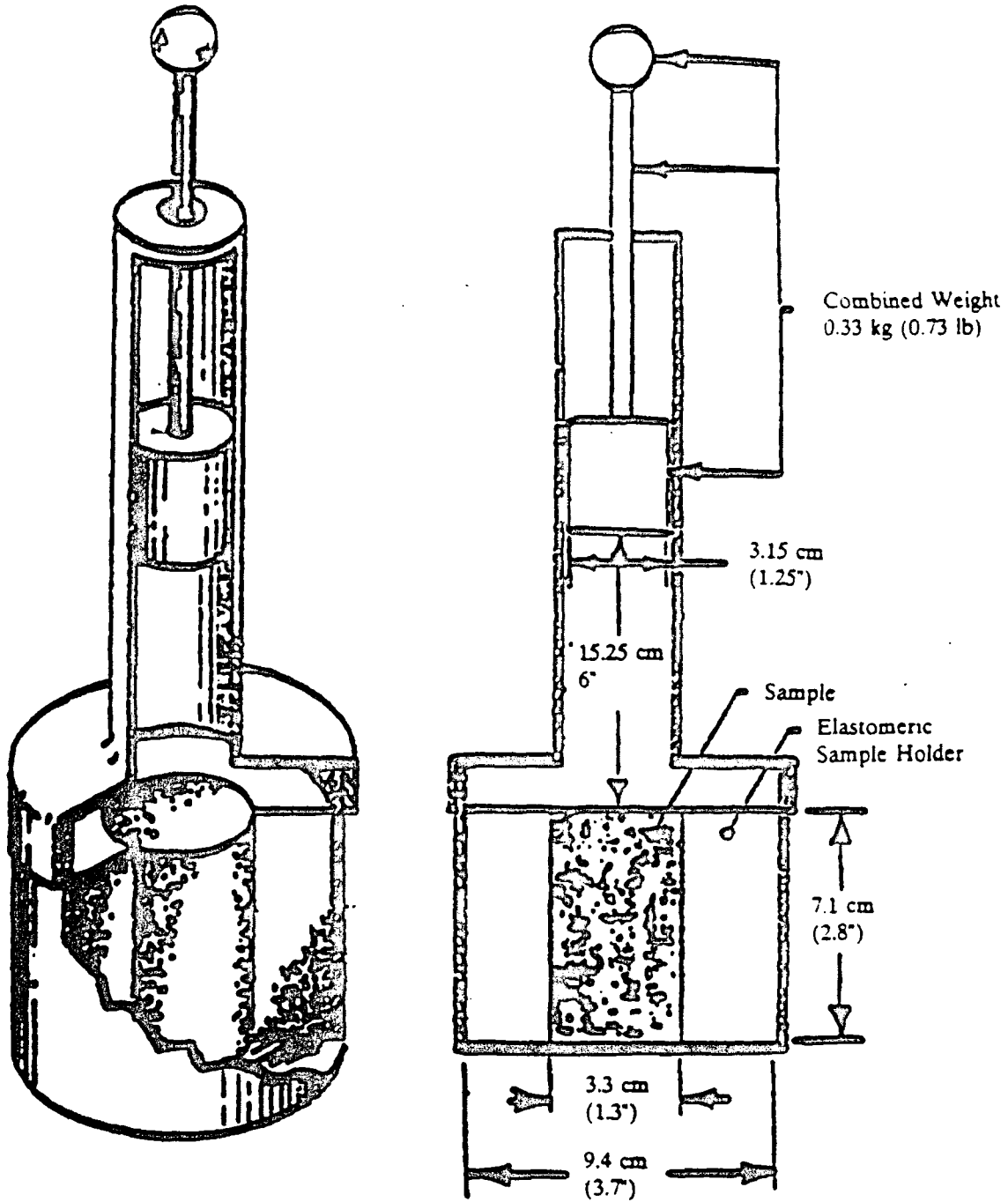
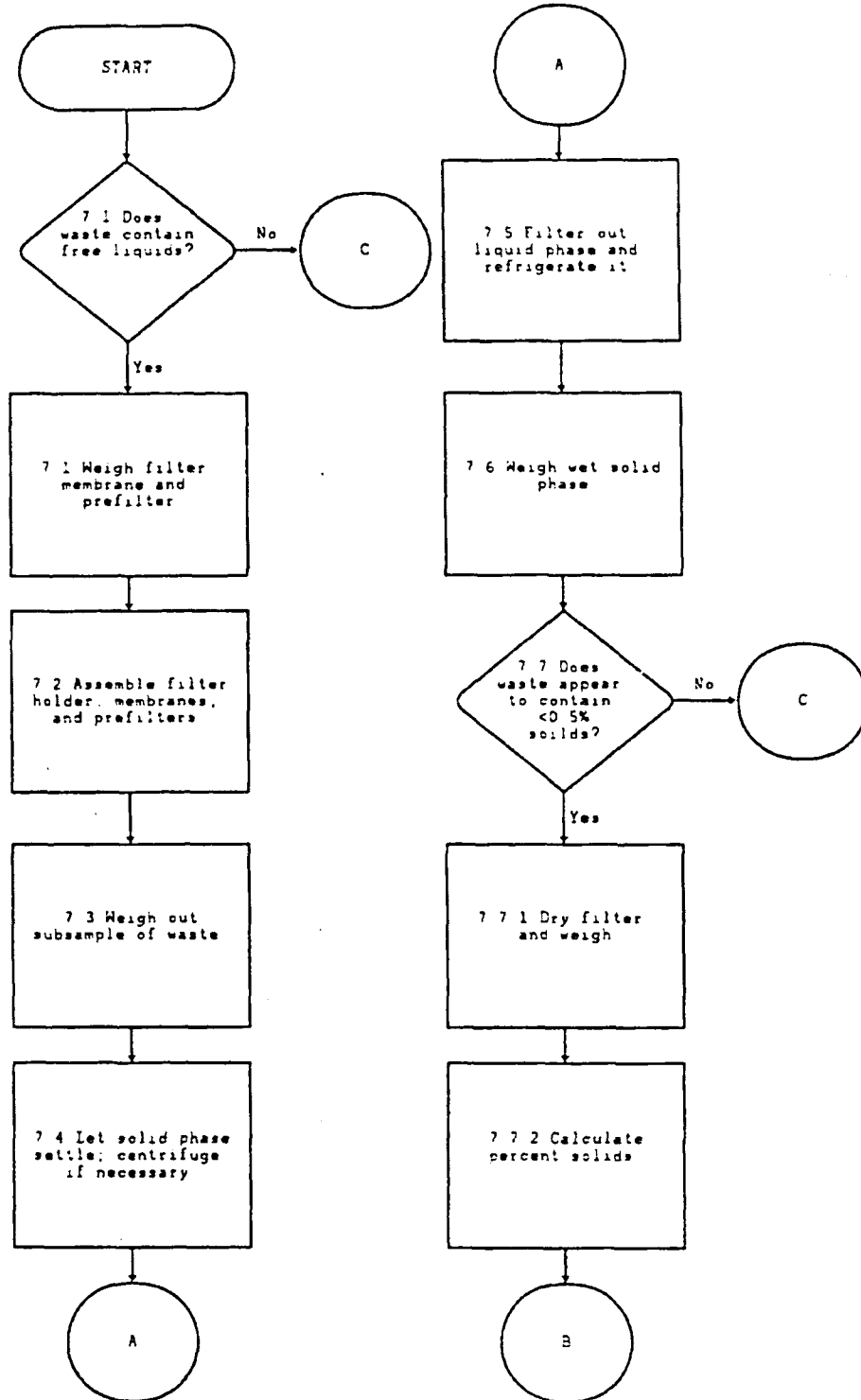


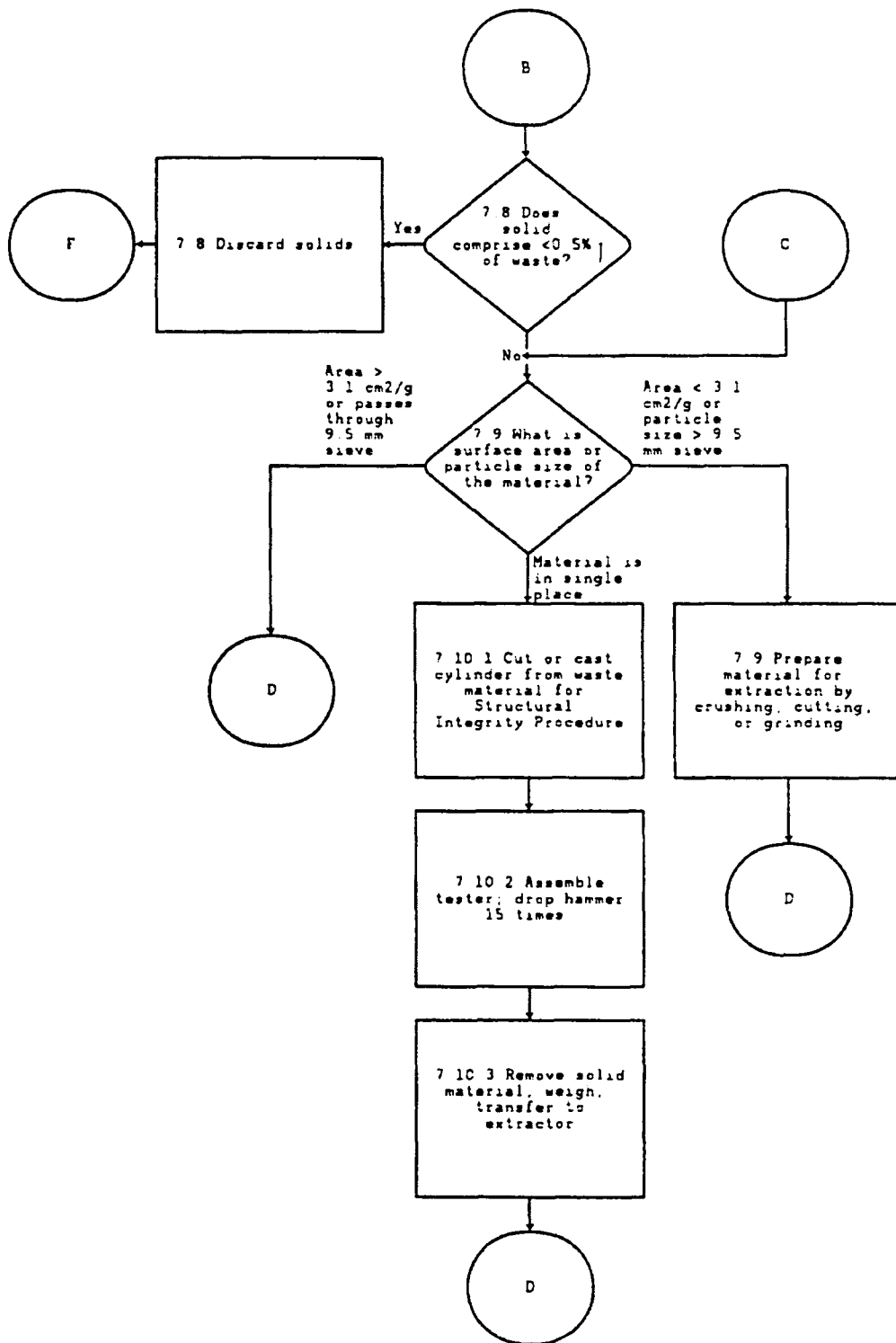
FIGURE 4.  
COMPACTION TESTER



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



METHOD 1310B  
(Continued)



METHOD 1310B  
(Continued)

