STATUS TABLES FOR
SW-846, THIRD EDITION

FINAL UPDATES I, II, IIA, IIB, III, IIIA, IIIB AND IV
PLUS OTHER NEW AND REVISED SW-846 METHODS
AT THE OSW METHODS WEB SITE

REVISED FEBRUARY 2007

HOW TO USE THIS DOCUMENT

This document provides historical information regarding EPA-published 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 (e.g., chapters).

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

Methods that have "deleted" as the latest status are those methods that have been removed from SW-846 for various reasons, and you will not find that method at the Methods Team web site. See the associated update rulemakings or notices for an explanation regarding why a method was deleted from SW-846.

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

With the publication of the final Methods Innovation Rule, SW-846 and its methods are no longer required in general by any RCRA regulation. See 40 CFR 260.11(a)(11) for a listing of those SW-846 methods that may be still required by the RCRA regulations for the analysis of method-defined parameters.

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

SW-846 METHOD STATUS TABLE

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
0010	--	--	--	--	--	Modified Method 5 Sampling Train
--	--	--	0011 (Up. III)	--	--	Sampling for Selected Aldehyde and Ketone Emissions from Stationary Sources
0020	--	--	--	--	--	Source Assessment Sampling System (SASS)
--	--	--	0023A (Up. III)	--	--	Sampling Method for Polychlorinated Dibenzo-*p*-Dioxins and Polychlorinated Dibenzofuran Emissions from Stationary Sources (Note: This method is a revision of Method 23, 40 CFR Part 60.)
--	--	--	--	25D Referral	--	Determination of the Volatile Organic Concentration of Waste Samples
--	--	--	--	25E Referral	--	Determination of Vapor Phase Organic Concentration in Waste Samples
0030	--	--	--	--	--	Volatile Organic Sampling Train
--	--	--	0031 (Up. III)	--	--	Sampling Method for Volatile Organic Compounds (SMVOC)
--	--	--	0040 (Up. III)	--	--	Sampling of Principal Organic Hazardous Constituents from Combustion Sources Using Tedlar® Bags
--	--	--	0050 (Up. III)	--	--	Isokinetic HCl/Cl_2 Emission Sampling Train
--	--	--	0051 (Up. III)	--	--	Midget Impinger HCl/Cl_2 Emission Sampling Train
--	--	--	0060 (Up. III)	--	--	Determination of Metals in Stack Emissions
--	--	--	0061 (Up. III)	--	--	Determination of Hexavalent Chromium Emissions from Stationary Sources

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	0100 (Up. III)	--	--	Sampling for Formaldehyde and Other Carbonyl Compounds in Indoor Air
--	--	--	--	207 Referral	--	A Method for Measuring Isocyanates in Stationary Source Emissions
1010	--	--	1010A (Up. IIIB)	--	--	Test Methods for Flash Point by Pensky-Martens Closed Cup Tester (Method text is a referral to ASTM Standard D 93-79 or Standard D 93-80)
1020	1020A	--	1020B (Up. IIIB)	--	--	Standard Test Methods for Flash Point by Setaflash (Small Scale) Closed-cup Apparatus (Method text is a referral to ASTM Standard D 3278-78)
--	--	--	1030 (Up. III)	--	--	Ignitability of Solids
--	--	--	--	1040	--	Test Method for Oxidizing Solids
--	--	--	--	1050	--	Test Methods to Determine Substances Likely to Spontaneously Combust
1110	--	--	1110A (Up. IIIB)	--	--	Corrosivity Toward Steel
--	--	--	1120 (Up. III)	--	--	Dermal Corrosion
1310	1310A	--	1310B (Up. IIIB)	--	--	Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test
--	1311	--	--	--	--	Toxicity Characteristic Leaching Procedure
--	--	1312 (Up. II)	--	--	--	Synthetic Precipitation Leaching Procedure
1320	--	--	--	--	--	Multiple Extraction Procedure
1330	1330A	--	--	--	--	Extraction Procedure for Oily Wastes

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
3005	3005A	--	--	--	--	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
3010	3010A	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy
--	--	3015 (Up. II)	--	3015A	--	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
3020	3020A	--	--	--	--	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy
--	--	--	3031 (Up. III)	--	--	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040	--	--	3040A (Up. III)	--	--	Dissolution Procedure for Oils, Greases, or Waxes
3050	3050A	--	3050B (Up. III)	--	--	Acid Digestion of Sediments, Sludges, and Soils
--	--	3051 (Up. II)	--	3051A	--	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
--	--	--	3052 (Up. III)	--	--	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
[3060, in the 2nd Ed.]	--	--	3060A (Up. III)	--	--	Alkaline Digestion for Hexavalent Chromium
--	--	--	--	--	3200 (7/05)	Mercury Species Fractionation and Quantification by Microwave Assisted Extraction, Selective Solvent Extraction and/or Solid Phase Extraction
3500	3500A	--	3500B (Up. III)	3500C	--	Organic Extraction and Sample Preparation
3510	3510A	3510B (Up. II)	3510C (Up. III)	--	--	Separatory Funnel Liquid-Liquid Extraction

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	--		3511 (11/02)	Organic Compounds in Water by Microextraction
3520	3520A	3520B (Up. II)	3520C (Up. III)	--	--	Continuous Liquid-Liquid Extraction
--	--	--	3535 (Up. III)	3535A	--	Solid-Phase Extraction (SPE)
3540	3540A	3540B (Up. II)	3540C (Up. III)	--	--	Soxhlet Extraction
--	--	3541 (Up. II)	--	--	--	Automated Soxhlet Extraction
--	--	--	3542 (Up. III)	--	3542A (5/05)	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)
--	--	--	3545 (Up. III)	3545A	--	Pressurized Fluid Extraction (PFE)
--	--	--	--	3546	--	Microwave Extraction
3550	--	3550A (Up. II)	3550B (Up. III)	3550C	--	Ultrasonic Extraction
--	--	--	3560 (Up. III)	--	--	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
--	--	--	3561 (Up. III)	--	--	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
--	--	--	--	3562	--	Supercritical Fluid Extraction of Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides
--	--	--	--	--	3570 (11/02)	Microscale Solvent Extraction (MSE)
3580	3580A	--	--	--	--	Waste Dilution

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	3585 (Up. III)	--	--	Waste Dilution for Volatile Organics
3600	3600A	3600B (Up. II)	3600C (Up. III)	--	--	Cleanup
3610	3610A	--	3610B (Up. III)	--	--	Alumina Cleanup
3611	3611A	--	3611B (Up. III)	--	--	Alumina Column Cleanup and Separation of Petroleum Wastes
3620	3620A	--	3620B (Up. III)	3620C	--	Florisil Cleanup
3630	3630A	3630B (Up. II)	3630C (Up. III)	--	--	Silica Gel Cleanup
3640	--	3640A (Up. II)	--	--	--	Gel-Permeation Cleanup
3650	3650A	--	3650B (Up. III)	--	--	Acid-Base Partition Cleanup
3660	3660A	--	3660B (Up. III)	--	--	Sulfur Cleanup
--	--	3665 (Up. II)	3665A (Up. III)	--	--	Sulfuric Acid/Permanganate Cleanup
3810	--	--	--	Deleted	--	Headspace
--	--	--	--	3815	--	Screening Solid Samples for Volatile Organics
3820	--	--	--	--	--	Hexadecane Extraction and Screening of Purgeable Organics
--	--	--	4000 (Up. III)	--	--	Immunoassay
--	--	4010 (Up. IIA)	4010A (Up. III)	--	--	Screening for Pentachlorophenol by Immunoassay

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	4015 (Up. III)	--	--	Screening for 2,4-Dichlorophenoxyacetic Acid by Immunoassay
--	--	--	4020 (Up. III)	--	--	Screening for Polychlorinated Biphenyls by Immunoassay
--	--	--	--	--	4025 (10/02)	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay
--	--	--	4030 (Up. III)	--	--	Soil Screening for Petroleum Hydrocarbons by Immunoassay
--	--	--	4035 (Up. III)	--	--	Soil Screening for Polynuclear Aromatic Hydrocarbons by Immunoassay
--	--	--	4040 (Up. III)	--	--	Soil Screening for Toxaphene by Immunoassay
--	--	--	4041 (Up. III)	--	--	Soil Screening for Chlordane by Immunoassay
--	--	--	4042 (Up. III)	--	--	Soil Screening for DDT by Immunoassay
--	--	--	4050 (Up. III)	--	--	TNT Explosives in Soil by Immunoassay
--	--	--	4051 (Up. III)	--	--	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in Soil by Immunoassay
--	--	--	--	4425	--	Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, PCDDs/PCDFs) by a Reporter Gene on a Human Cell Line
--	--	--	--	4670	--	Triazine Herbicides as Atrazine in Water by Quantitative Immunoassay
--	--	--	5000 (Up. III)	--	--	Sample Preparation for Volatile Organic Compounds

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	5021 (Up. III)	--	5021A (6/03)	5021: Volatile Organic Compounds in Soils and Other Solid Sample Matrices Using Equilibrium Headspace Analysis 5021A: Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis
5030	5030A	--	5030B (Up. III)	--	5030C (5/03)	Purge-and-Trap for Aqueous Samples
--	--	--	5031 (Up. III)	--	--	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
--	--	--	5032 (Up. III)	--	--	Volatile Organic Compounds by Vacuum Distillation
--	--	--	5035 (Up. III)	--	5035A (7/02)	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
5040	--	5040A (Up. II)	Deleted (Up. III)	--	--	Analysis of Sorbent Cartridges from Volatile Organic Sampling Train (VOST): Gas Chromatography/Mass Spectrometry Technique
--	--	5041 (Up. II)	5041A (Up. III)	--	--	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)
--	--	5050 (Up. II)	--	--	--	Bomb Preparation Method for Solid Waste
6010	6010A	--	6010B (Up. III)	6010C	--	Inductively Coupled Plasma-Atomic Emission Spectrometry
--	--	6020 (Up. II)	--	6020A	--	Inductively Coupled Plasma-Mass Spectrometry
--	--	--	--	6200	--	Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
--	--	--	--	6500	--	Dissolved Inorganic Anions in Aqueous Matrices by Capillary Ion Electrophoresis
--	--	--	--	6800	--	Elemental and Speciated Isotope Dilution Mass Spectrometry

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	--	--	6850 (1/07)	Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS/MS)
--	--	--	--	--	6860 (1/07)	Perchlorate in Water, Soils and Solid Wastes Using Ion Chromatography/Electrospray Ionization/Mass Spectrometry (IC/ESI/MS or IC/ESI/MS/MS)
7000	7000A	--	--	7000B	--	Flame Atomic Absorption Spectrophotometry
--	--	--	--	7010	--	Graphite Furnace Atomic Absorption Spectrophotometry
7020	--	--	--	Deleted	--	Aluminum (Atomic Absorption, Direct Aspiration)
7040	--	--	--	Deleted	--	Antimony (Atomic Absorption, Direct Aspiration)
7041	--	--	--	Deleted	--	Antimony (Atomic Absorption, Furnace Technique)
7060	--	7060A (Up. II)	--	Deleted	--	Arsenic (Atomic Absorption, Furnace Technique)
7061	7061A	--	--	--	--	Arsenic (Atomic Absorption, Gaseous Hydride)
--	--	7062 (Up. II)	--	--	--	Antimony and Arsenic (Atomic Absorption, Borohydride Reduction)
--	--	--	7063 (Up. III)	--	--	Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)
7080	--	7080A (Up. II)	--	Deleted	--	Barium (Atomic Absorption, Direct Aspiration)
--	7081	--	--	Deleted	--	Barium (Atomic Absorption, Furnace Technique)
7090	--	--	--	Deleted	--	Beryllium (Atomic Absorption, Direct Aspiration)
7091	--	--	--	Deleted	--	Beryllium (Atomic Absorption, Furnace Technique)
7130	--	--	--	Deleted	--	Cadmium (Atomic Absorption, Direct Aspiration)

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
7131	--	7131A (Up. II)	--	Deleted	--	Cadmium (Atomic Absorption, Furnace Technique)
7140	--	--	--	Deleted	--	Calcium (Atomic Absorption, Direct Aspiration)
7190	--	--	--	Deleted	--	Chromium (Atomic Absorption, Direct Aspiration)
7191	--	--	--	Deleted	--	Chromium (Atomic Absorption, Furnace Technique)
7195	--	--	--	--	--	Chromium, Hexavalent (Coprecipitation)
7196	7196A	--	--	--	--	Chromium, Hexavalent (Colorimetric)
7197	--	--	--	--	--	Chromium, Hexavalent (Chelation/Extraction)
7198	--	--	--	--	--	Chromium, Hexavalent (Differential Pulse Polarography)
--	--	--	7199 (Up. III)	--	--	Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography
7200	--	--	--	Deleted	--	Cobalt (Atomic Absorption, Direct Aspiration)
7201	--	--	--	Deleted	--	Cobalt (Atomic Absorption, Furnace Technique)
7210	--	--	--	Deleted	--	Copper (Atomic Absorption, Direct Aspiration)
--	7211	--	--	Deleted	--	Copper (Atomic Absorption, Furnace Technique)
7380	--	--	--	Deleted	--	Iron (Atomic Absorption, Direct Aspiration)
--	7381	--	--	Deleted	--	Iron (Atomic Absorption, Furnace Technique)
7420	--	--	--	Deleted	--	Lead (Atomic Absorption, Direct Aspiration)
7421	--	--	--	Deleted	--	Lead (Atomic Absorption, Furnace Technique)
--	7430	--	--	Deleted	--	Lithium (Atomic Absorption, Direct Aspiration)
7450	--	--	--	Deleted	--	Magnesium (Atomic Absorption, Direct Aspiration)
7460	--	--	--	Deleted	--	Manganese (Atomic Absorption, Direct Aspiration)

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	7461	--	--	Deleted	--	Manganese (Atomic Absorption, Furnace Technique)
7470	--	7470A (Up. II)	--	--	--	Mercury in Liquid Waste (Manual Cold-Vapor Technique)
7471	--	7471A (Up. II)	--	7471B	--	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
--	--	--	7472 (Up. III)	--	--	Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)
--	--	--	--	7473	--	Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry
--	--	--	--	7474	--	Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry
7480	--	--	--	Deleted	--	Molybdenum (Atomic Absorption, Direct Aspiration)
7481	--	--	--	Deleted	--	Molybdenum (Atomic Absorption, Furnace Technique)
7520	--	--	--	Deleted	--	Nickel (Atomic Absorption, Direct Aspiration)
--	--	--	7521 (Up. III)	Deleted	--	Nickel (Atomic Absorption, Furnace Method)
7550	--	--	--	Deleted	--	Osmium (Atomic Absorption, Direct Aspiration)
--	--	--	7580 (Up. III)	--	--	White Phosphorus (P_4) by Solvent Extraction and Gas Chromatography
7610	--	--	--	Deleted	--	Potassium (Atomic Absorption, Direct Aspiration)
7740	--	--	--	Deleted	--	Selenium (Atomic Absorption, Furnace Technique)
7741	--	7741A (Up. II)	--	--	--	Selenium (Atomic Absorption, Gaseous Hydride)
--	--	7742 (Up. II)	--	--	--	Selenium (Atomic Absorption, Borohydride Reduction)

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
7760	7760A	--	--	Deleted	--	Silver (Atomic Absorption, Direct Aspiration)
--	7761	--	--	Deleted	--	Silver (Atomic Absorption, Furnace Technique)
7770	--	--	--	Deleted	--	Sodium (Atomic Absorption, Direct Aspiration)
--	7780	--	--	Deleted	--	Strontium (Atomic Absorption, Direct Aspiration)
7840	--	--	--	Deleted	--	Thallium (Atomic Absorption, Direct Aspiration)
7841	--	--	--	Deleted	--	Thallium (Atomic Absorption, Furnace Technique)
7870	--	--	--	Deleted	--	Tin (Atomic Absorption, Direct Aspiration)
7910	--	--	--	Deleted	--	Vanadium (Atomic Absorption, Direct Aspiration)
7911	--	--	--	Deleted	--	Vanadium (Atomic Absorption, Furnace Technique)
7950	--	--	--	Deleted	--	Zinc (Atomic Absorption, Direct Aspiration)
--	7951	--	--	Deleted	--	Zinc (Atomic Absorption, Furnace Technique)
8000	8000A	--	8000B (Up. III)	--	8000C (3/03)	Determinative Chromatographic Separations
8010	8010A	8010B (Up. II)	Deleted (Up. III)	--	--	Halogenated Volatile Organics by Gas Chromatography
--	8011	--	--	--	--	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography
8015	8015A	--	8015B	8015C	8015D (6/03)	8015C: Nonhalogenated Organics by Gas Chromatography 8015D: Nonhalogenated Organics by Gas Chromatography Using GC/FID
8020	--	8020A (Up. II)	Deleted (Up. III)	--	--	Aromatic Volatile Organics by Gas Chromatography
--	8021	8021A (Up. II)	8021B (Up. III)	--	--	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors

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THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
8030	8030A	--	Deleted (Up. III)	--	--	Acrolein and Acrylonitrile by Gas Chromatography
--	--	8031 (Up. II)	--	--	--	Acrylonitrile by Gas Chromatography
--	--	8032 (Up. II)	8032A (Up. III)	--	--	Acrylamide by Gas Chromatography
--	--	--	8033 (Up. III)	--	--	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection
8040	8040A	--	Deleted (Up. III)	--	--	Phenols by Gas Chromatography
--	--	--	8041 (Up. III)	8041A	--	Phenols by Gas Chromatography
8060	--	--	Deleted (Up. III)	--	--	Phthalate Esters
--	--	8061 (Up. II)	8061A (Up. III)	--	--	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)
--	8070	--	8070A (Up. III)	--	--	Nitrosamines by Gas Chromatography
8080	--	8080A (Up. II)	Deleted (Up. III)	--	--	Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography
--	--	8081 (Up. II)	8081A (Up. III)	8081B	--	Organochlorine Pesticides by Gas Chromatography
--	--	--	8082 (Up. III)	8082A	--	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
				8085	--	Compound-independent Elemental Quantitation of Pesticides by Gas Chromatography with Atomic Emission Detection (GC/AED)

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
8090	--	--	Deleted (Up. III)	--	--	Nitroaromatics and Cyclic Ketones
--	--	--	8091 (Up. III)	--	--	Nitroaromatics and Cyclic Ketones by Gas Chromatography
--	--	--	--	8095	--	Explosives by Gas Chromatography
8100	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons
--	8110	--	Deleted (Up. III)	--	--	Haloethers by Gas Chromatography
--	--	--	8111 (Up. III)	--	--	Haloethers by Gas Chromatography
8120	--	8120A (Up. II)	Deleted (Up. III)	--	--	Chlorinated Hydrocarbons by Gas Chromatography
--	--	8121 (Up. II)	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique
--	--	--	8131 (Up. III)	--	--	Aniline and Selected Derivatives by Gas Chromatography
8140	--	--	Deleted (Up. III)	--	--	Organophosphorus Pesticides
--	8141	8141A (Up. II)	--	8141B	--	Organophosphorus Compounds by Gas Chromatography
8150	8150A	8150B (Up. II)	Deleted (Up. III)	--	--	Chlorinated Herbicides by Gas Chromatography
--	--	8151 (Up. II)	8151A (Up. III)	--	--	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization
8240	8240A	8240B (Up. II)	Deleted (Up. III)	--	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
8250	--	8250A (Up. II)	Deleted (Up. III)	--	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	8260	8260A (Up. II)	8260B (Up. III)	--	8260C (8/06)	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	--	--	--	8261	8261A (10/06)	Volatile Organic Compounds by Vacuum Distillation in Combination with Gas Chromatography/Mass Spectrometry (VD/GC/MS)
--	--	--	--	--	8265 (3/02)	Volatile Organic Compounds in Water, Soil, Soil Gas and Air by Direct Sampling Ion Trap Mass Spectrometry (DSITMS)
8270	8270A	8270B (Up. II)	8270C (Up. III)	8270D	--	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
--	--	8275 (Up. II)	8275A (Up. III)	--	--	Semivolatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)
8280	--	--	8280A (Up. III)	8280B	--	Polychlorinated Dibenzo-*p*-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)
--	--	8290 (Up. II)	--	8290A	--	Polychlorinated Dibenzo-*p*-dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/ High-Resolution Mass Spectrometry (HRGC/HRMS)
8310	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons
--	--	8315 (Up. II)	8315A (Up. III)	--	--	Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)
--	--	8316 (Up. II)	--	--	--	Acrylamide, Acrylonitrile and Acrolein by High Performance Liquid Chromatography (HPLC)
--	--	8318 (Up. II)	--	8318A	--	*N*-Methylcarbamates by High Performance Liquid Chromatography (HPLC)

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	8321 (Up. II)	8321A (Up. III)	8321B	--	Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection
--	--	--	--	--	8323 (1/03)	Determination of Organotins by Micro-liquid Chromatography-electrospray Ion Trap Mass Spectrometry
--	--	--	8325 (Up. III)	--	--	Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry (HPLC/PB/MS)
--	--	8330 (Up. II)	--	8330A	8330B (10/06)	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)
--	--	8331 (Up. II)	--	--	--	Tetrazene by Reverse Phase High Performance Liquid Chromatography (HPLC)
--	--	--	8332 (Up. III)	--	--	Nitroglycerine by High Performance Liquid Chromatography
--	--	8410 (Up. II)	--	--	--	Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column
--	--	--	8430 (Up. III)	--	--	Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR
--	--	--	8440 (Up. III)	--	--	Total Recoverable Petroleum Hydrocarbons by Infrared Spectrophotometry
				8510	--	Colorimetric Screening Procedure for RDX and HMX in Soil
--	--	--	8515 (Up. III)	--	--	Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil
--	--	--	8520 (Up. III)	--	--	Continuous Measurement of Formaldehyde in Ambient Air
--	--	--	--	8535	--	Screening Procedure for Total Volatile Organic Halides in Water
--	--	--	--	8540	--	Pentachlorophenol by UV-induced Colorimetry

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	--	9000	--	Determination of Water in Waste Materials by Karl Fischer Titration
--	--	--	--	9001	--	Determination of Water in Waste Materials by Quantitative Calcium Hydride Reaction
9010	9010A	--	9010B (Up. III) 9010C (Up. IIIB)	--	--	Total and Amenable Cyanide: Distillation
9012	--	--	9012A (Up. III) 9012B (Up. IIIB)	--	--	Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)
--	9013	--	--	--	9013A (11/04)	Cyanide Extraction Procedure for Solids and Oils
--	--	--	9014 (Up. III)	--	--	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
--	--	--	--	--	9015 (11/04)	Metal Cyanide Complexes by Anion Exchange Chromatography and UV Detection
9020	9020A	9020B (Up. II)	--	--	--	Total Organic Halides (TOX)
--	9021	--	--	--	--	Purgeable Organic Halides (POX)
9022	--	--	--	--	--	Total Organic Halides (TOX) by Neutron Activation Analysis
--	--	--	9023 (Up. III)	--	--	Extractable Organic Halides (EOX) in Solids
9030	9030A	--	9030B (Up. III)	--	--	Acid-Soluble and Acid-Insoluble Sulfides: Distillation
--	9031	--	--	--	--	Extractable Sulfides

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	9034 (Up. III)	--	--	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides
9035	--	--	--	--	--	Sulfate (Colorimetric, Automated, Chloranilate)
9036	--	--	--	--	--	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
9038	--	--	--	--	--	Sulfate (Turbidimetric)
9040	--	9040A (Up. II) 9040B (Up. IIB)	9040C (Up. IIIB)	--	--	pH Electrometric Measurement
9041	9041A	--	--	--	--	pH Paper Method
9045	9045A	9045B (Up. II) 9045C (Up. IIB)	9045D (Up. IIIB)	--	--	Soil and Waste pH
9050	--	--	9050A (Up. III)	--	--	Specific Conductance
--	--	9056 (Up. II)	--	9056A	--	Determination of Inorganic Anions by Ion Chromatography
--	--	--	9057 (Up. III)	--	--	Determination of Chloride from HCl/Cl_2 Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography
9060	--	--	9060A (Up. IIIB)	--	--	Total Organic Carbon
9065	--	--	--	--	--	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)
9066	--	--	--	--	--	Phenolics (Colorimetric, Automated 4-AAP with Distillation)

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
9067	--	--	--	--	--	Phenolics (Spectrophotometric, MBTH with Distillation)
9070	--	--	9070 (Up. IIIA) 9070A (Up. IIIB)	--	--	n-Hexane Extractable Material (HEM) for Aqueous Samples (Note: Method text is a referral to Method 1664: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry)
9071	--	9071A (Up. II)	9071B (Up. IIIA)	--	--	n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples
--	--	--	--	9074	--	Turbidimetric Screening Method for Total Recoverable Petroleum Hydrocarbons in Soil
--	--	9075 (Up. II)	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)
--	--	9076 (Up. II)	--	--	--	Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry
--	--	9077 (Up. II)	--	--	--	Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)
--	--	--	9078 (Up. III)	--	--	Screening Test Method for Polychlorinated Biphenyls in Soil
--	--	--	9079 (Up. III)	--	--	Screening Test Method for Polychlorinated Biphenyls in Transformer Oil
9080	--	--	--	--	--	Cation-Exchange Capacity of Soils (Ammonium Acetate)
9081	--	--	--	--	--	Cation-Exchange Capacity of Soils (Sodium Acetate)
9090	9090A	--	--	--	--	Compatibility Test for Wastes and Membrane Liners

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
9095	--	--	9095A (Up. III) 9095B (Up. IIIB)	--	--	Paint Filter Liquids Test
--	--	9096 (Up. II)	--	--	--	Liquid Release Test (LRT) Procedure
9100	--	--	--	--	--	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability
9131	--	--	--	--	--	Total Coliform: Multiple Tube Fermentation Technique
9132	--	--	--	--	--	Total Coliform: Membrane-Filter Technique
9200	--	--	Deleted (Up. III)	--	--	Nitrate
--	--	--	9210 (Up. III)	9210A	--	Potentiometric Determination of Nitrate in Aqueous Samples with an Ion-Selective Electrode
--	--	--	9211 (Up. III)	--	--	Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode
--	--	--	9212 (Up. III)	--	--	Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode
--	--	--	9213 (Up. III)	--	--	Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion-Selective Electrode
--	--	--	9214 (Up. III)	--	--	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode
--	--	--	9215 (Up. III)	--	--	Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion-Selective Electrode

METHOD NUMBER (Date in parenthesis is found at bottom right-hand corner of method)						METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	OTHER METHODS (e.g., at web site)	
--	--	--	--	9216	--	Potentiometric Determination of Nitrite in Aqueous Samples with Ion-Selective Electrode
9250	--	--	--	--	--	Chloride (Colorimetric, Automated Ferricyanide AAI)
9251	--	--	--	--	--	Chloride (Colorimetric, Automated Ferricyanide AAII)
9252	--	9252A (Up. II)	Deleted (Up. III)	--	--	Chloride (Titrimetric, Mercuric Nitrate)
--	--	9253 (Up. II)	--	--	--	Chloride (Titrimetric, Silver Nitrate)
9310	--	--	--	--	--	Gross Alpha and Gross Beta
9315	--	--	--	--	--	Alpha-Emitting Radium Isotopes
9320	--	--	--	--	--	Radium-228
HCN and H_2S Test Methods	HCN and H_2S Test Methods	HCN and H_2S Test Methods (Up. II)	HCN and H_2S Test Methods (Up. III) Deleted (Up. IIIB)	--	--	Test Method to Determine Hydrogen Cyanide Released from Wastes and Test Method to Determine Hydrogen Sulfide Released from Wastes

Note: Draft Update IV Method 9058, "Determination of Perchlorate Using Ion Chromatography with Chemical Suppression Conductivity Detection," and Method 4500, "Mercury in Soil by Immunoassay," were not finalized as part of Final Update IV. See the Final Update IV Federal Register Notice.

STATUS TABLE FOR SW-846 CHAPTER TEXT AND OTHER DOCUMENTS

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) IIIB (11/04)	FINAL UP. IV (2/07)	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, and IIIB)	✓	Rev 7 (2/07)
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)	✓	Rev 4 (2/07)
Chapter Three -- Inorganic Analytes	✓	✓	✓ (Up. II)	✓ (Up. III)	✓	Rev 4 (2/07)
Chapter Four -- Organic Analytes	✓	--	✓ (Up. II)	✓ (Up. III)	✓	Rev 4 (2/07)
Chapter Five -- Miscellaneous Test Methods	✓	--	✓ (Up. II)	✓ (Up. III, IIIA and IIIB)	✓	Rev 5 (2/07)

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) IIIB (11/04)	FINAL UP. IV (2/07)	CURRENT FINAL VERSION
Chapter Six -- Properties	✓	--	✓ (Up. II & IIB)	✓ (Up. III and IIIB)	✓	Rev 5 (2/07)
Chapter Seven -- Characteristics Introduction and Regulatory Definitions	✓	✓	✓ (Up. II)	✓ (Up. III and IIIB)	--	Rev 4 (11/04)
Chapter Eight --Methods for Determining Characteristics	✓	--	✓ (Up. II)	✓ (Up. III and IIIB)	--	Rev 3 (11/04)
Chapter Nine -- Sampling Plan	✓	--	--	--	--	Rev 0 (9/86)
Chapter Ten -- Sampling Methods	✓	--	--	✓ (Up. III)	✓	Rev 3 (2/07)
Chapter Eleven -- Ground Water Monitoring	✓	--	--	--	✓	Rev 1 (2/07)
Chapter Twelve -- Land Treatment Monitoring	✓	--	--	--	--	Rev 0 (9/86)
Chapter Thirteen -- Incineration	✓	--	--	--	--	Rev 0 (9/86)
Appendix -- Company References	✓	--	--	--	--	Rev 0 (9/86)

CHAPTER ONE
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CHAPTER ONE
QUALITY CONTROL

1.0 INTRODUCTION

It is the goal of the U.S. Environmental Protection Agency's (EPA's) quality assurance (QA) program to ensure that all data be scientifically valid, defensible, and of known precision and accuracy. The data should be of sufficient known quality to withstand scientific and legal challenge relative to the use for which the data are obtained. The QA program is management's tool for achieving this goal.

For RCRA analyses, the recommended minimum requirements for a QA program and the associated quality control (QC) procedures are provided in this chapter.

The data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Method-specific QC procedures are incorporated in the individual methods since they are not applied universally.

A total program to generate data of acceptable quality should include both a QA component, which encompasses the management procedures and controls, as well as an operational day-to-day QC component. This chapter defines fundamental elements of such a data collection program. Data collection efforts involve:

1. design of a project plan to achieve the data quality objectives (DQOs);

2. implementation of the project plan; and

3. assessment of the data to determine if the DQOs are met.

The project plan may be a sampling and analysis plan or a waste analysis plan if it covers the QA/QC goals of the Chapter, or it may be a Quality Assurance Project Plan as described later in this chapter.

This chapter identifies the minimal QC components that should be used in the performance of sampling and analyses, including the QC information which should be documented. Guidance is provided to construct QA programs for field and laboratory work conducted in support of the RCRA program.

2.0 QA PROJECT PLAN

It is recommended that all projects which generate environment-related data in support of RCRA have a QA Project Plan (QAPjP) or equivalent. In some instances, a sampling and analysis plan or a waste analysis plan may be equivalent if it covers all of the QA/QC goals outlined in this chapter. In addition, a separate QAPjP need not be prepared for routine analyses or

activities where the procedures to be followed are described in a Standard Operating Procedures manual or similar document and include the elements of a QAPjP. These documents should be available and referenced in the documentation and/or records for the analysis activities. The term "QAPjP" in this chapter refers to any of these QA/QC documents.

The QAPjP should detail the QA/QC goals and protocols for a specific data collection activity. The QAPjP sets forth a plan for sampling and analysis activities that will generate data of a quality commensurate with their intended use. QAPjP elements should include a description of the project and its objectives; a statement of the DQOs of the project; identification of those involved in the data collection and their responsibilities and authorities; reference to (or inclusion of) the specific sample collection and analysis procedures that will be followed for all aspects of the project; enumeration of QC procedures to be followed; and descriptions of all project documentation. Additional elements should be included in the QAPjP if needed to address all quality related aspects of the data collection project. Elements should be omitted only when they are inappropriate for the project or when absence of those elements will not affect the quality of data obtained for the project (see reference 1).

The role and importance of DQOs and project documentation are discussed below in Sections 2.1 through 2.6. Management and organization play a critical role in determining the effectiveness of a QA/QC program and ensuring that all required procedures are followed. Section 2.7 discusses the elements of an organization's QA program that have been found to ensure an effective program. Field operations and laboratory operations (along with applicable QC procedures) are discussed in Sections 3 and 4, respectively.

2.1 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) for the data collection activity describe the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data. This uncertainty is used to specify the quality of the measurement data required, usually in terms of objectives for precision, bias, representativeness, comparability and completeness. The DQOs should be defined prior to the initiation of the field and laboratory work. The field and laboratory organizations performing the work should be aware of the DQOs so that their personnel may make informed decisions during the course of the project to attain those DQOs. More detailed information on DQOs is available from the U.S. EPA Quality Assurance Management Staff (QAMS) (see references 2 and 4).

2.2 PROJECT OBJECTIVES

A statement of the project objectives and how the objectives are to be attained should be concisely stated and sufficiently detailed to permit clear understanding by all parties involved in the data collection effort. This

includes a statement of what problem is to be solved and the information required in the process. It also includes appropriate statements of the DQOs (i.e., the acceptable level of uncertainty in the information).

2.3 SAMPLE COLLECTION

Sampling procedures, locations, equipment, and sample preservation and handling requirements should be specified in the QAPjP. Further details on quality assurance procedures for field operations are described in Section 3 of this chapter. The OSW is developing policies and procedures for sampling in a planned revision of Chapter Nine of this manual. Specific procedures for groundwater sampling are provided in Chapter Eleven of this manual.

2.4 ANALYSIS AND TESTING

Analytes and properties of concern, analytical and testing procedures to be employed, required detection limits, and requirements for precision and bias should be specified. All applicable regulatory requirements and the project DQOs should be considered when developing the specifications. Further details on the procedures for analytical operations are described in Section 4 of this chapter.

2.5 QUALITY CONTROL

The quality assurance program should address both field and laboratory activities. Quality control procedures should be specified for estimating the precision and bias of the data. Recommended minimum requirements for QC samples have been established by EPA and should be met in order to satisfy recommended minimum criteria for acceptable data quality. Further details on procedures for field and laboratory operations are described in Sections 3 and 4, respectively, of this chapter.

2.6 PROJECT DOCUMENTATION

Documents should be prepared and maintained in conjunction with the data collection effort. Project documentation should be sufficient to allow review of all aspects of the work being performed. The QAPjP discussed in Sections 3 and 4 is one important document that should be maintained.

The length of storage time for project records should comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent. It is recommended that documentation be stored for three years from submission of the project final report.

Documentation should be secured in a facility that adequately addresses/minimizes its deterioration for the length of time that it is to be

retained. A system allowing for the expedient retrieval of information should exist.

Access to archived information should be controlled to maintain the integrity of the data. Procedures should be developed to identify those individuals with access to the data.

2.7 ORGANIZATION PERFORMING FIELD OR LABORATORY OPERATIONS

Proper design and structure of the organization facilitates effective and efficient transfer of information and helps to prevent important procedures from being overlooked.

The organizational structure, functional responsibilities, levels of authority, job descriptions, and lines of communication for all project activities should be established and documented. One person may cover more than one organizational function. Each project participant should have a clear understanding of his or her duties and responsibilities and the relationship of those responsibilities to the overall data collection effort.

The management of each organization participating in a project involving data collection activities should establish that organization's operational and QA policies. This information should be documented in the QAPjP. The management should ensure that (1) the appropriate methodologies are followed as documented in the QAPjPs; (2) personnel clearly understand their duties and responsibilities; (3) each staff member has access to appropriate project documents; (4) any deviations from the QAPjP are communicated to the project management and documented; and (5) communication occurs between the field, laboratory, and project management, as specified in the QAPjP. In addition, each organization should ensure that their activities do not increase the risk to humans or the environment at or about the project location. Certain projects may require specific policies or a Health and Safety Plan to provide this assurance.

The management of the participating field or laboratory organization should establish personnel qualifications and training requirements for the project. Each person participating in the project should have the education, training, technical knowledge, and experience, or a combination thereof, to enable that individual to perform assigned functions. Training should be provided for each staff member as necessary to perform their functions properly. Personnel qualifications should be documented in terms of education, experience, and training, and periodically reviewed to ensure adequacy to current responsibilities.

Each participating field organization or laboratory organization should have a designated QA function (i.e., a team or individual trained in QA) to monitor operations to ensure that the equipment, personnel, activities, procedures, and documentation conform with the QAPjP. To the extent possible, the QA monitoring function should be entirely separate from, and independent of,

personnel engaged in the work being monitored. The QA function should be responsible for the QA review.

2.7.1 Performance Evaluation

Performance evaluation studies are used to measure the performance of the laboratory on unknown samples. Performance evaluation samples are typically submitted to the laboratory as blind samples by an independent outside source. The results are compared to predetermined acceptance limits. Performance evaluation samples can also be submitted to the laboratory as part of the QA function during internal assessment of laboratory performance. Records of all performance evaluation studies should be maintained by the laboratory. Problems identified through participation in performance evaluation studies should be immediately investigated and corrected.

2.7.2 Internal Assessment by QA Function

Personnel performing field and laboratory activities are responsible for continually monitoring individual compliance with the QAPjP. The QA function should review procedures, results and calculations to determine compliance with the QAPjP. The results of this internal assessment should be reported to management with requirements for a plan to correct observed deficiencies.

2.7.3 External Assessment

The field and laboratory activities may be reviewed by personnel external to the organization. Such an assessment is an extremely valuable method for identifying overlooked problems. The results of the external assessment should be submitted to management with requirements for a plan to correct observed deficiencies.

2.7.4 On-Site Evaluation

On-site evaluations may be conducted as part of both internal and external assessments. The focus of an on-site evaluation is to evaluate the degree of conformance of project activities with the applicable QAPjP. On-site evaluations may include, but are not limited to, a complete review of facilities, staff, training, instrumentation, procedures, methods, sample collection, analyses, QA policies and procedures related to the generation of environmental data. Records of each evaluation should include the date of the evaluation, location, the areas reviewed, the person performing the evaluation, findings and problems, and actions recommended and taken to resolve problems. Any problems identified that are likely to affect data integrity should be brought immediately to the attention of management.

2.7.4.1 Field Activities

The review of field activities should be conducted by one or more persons knowledgeable in the activities being reviewed and include evaluating, at a minimum, the following subjects:

Completeness of Field Reports -- This review determines whether all requirements for field activities in the QAPjP have been fulfilled, that complete records exist for each field activity, and that the procedures specified in the QAPjP have been implemented. Emphasis on field documentation will help assure sample integrity and sufficient technical information to recreate each field event. The results of this completeness check should be documented, and environmental data affected by incomplete records should be identified.

Identification of Valid Samples -- This review involves interpretation and evaluation of the field records to detect problems affecting the representativeness of environmental samples. Examples of items that might indicate potentially invalid samples include improper well development, improperly screened wells, instability of pH or conductivity, and collection of volatiles near internal combustion engines. The field records should be evaluated against the QAPjP and SOPs. The reviewer should document the sample validity and identify the environmental data associated with any poor or incorrect field work.

Correlation of Field Test Data -- This review involves comparing any available results of field measurements obtained by more than one method. For example, surface geophysical methods should correlate with direct methods of site geologic characterization such as lithologic logs constructed during drilling operations.

Identification of Anomalous Field Test Data -- This review identifies any anomalous field test data. For example, a water temperature for one well that is 5 degrees higher than any other well temperature in the same aquifer should be noted. The reviewer should evaluate the impact of anomalous field measurement results on the associated environmental data.

Validation of Field Analyses -- This review validates and documents all data from field analysis that are generated *in situ* or from a mobile laboratory as specified in Section 2.7.4.2. The reviewer should document whether the QC checks meet the acceptance criteria, and whether corrective actions were taken for any analysis performed when acceptance criteria were exceeded.

2.7.4.2 Laboratory Activities

The review of laboratory data should be conducted by one or more persons knowledgeable in laboratory activities and include evaluating, at a minimum, the following subjects:

Completeness of Laboratory Records -- This review determines whether: (1) all samples and analyses required by the QAPjP have been processed, (2) complete records exist for each analysis and the associated QC samples, and that (3) the procedures specified in the QAPjP have been implemented. The results of the completeness check should be documented, and environmental data affected by incomplete records should be identified.

Evaluation of Data with Respect to Detection and Quantitation Limits -- This review compares analytical results to required quantitation limits. Reviewers should document instances where detection or quantitation limits exceed regulatory limits, action levels, or target concentrations specified in the QAPjP.

Evaluation of Data with Respect to Control Limits -- This review compares the results of QC and calibration check samples to control criteria. Corrective action should be implemented for data not within control limits. The reviewer should check that corrective action reports, and the results of reanalysis, are available. The review should determine whether samples associated with out-of-control QC data are identified in a written record of the data review, and whether an assessment of the utility of such analytical results is recorded.

Review of Holding Time Data -- This review compares sample holding times to those required by the QAPjP, and notes all deviations.

Review of Performance Evaluation (PE) Results -- PE study results can be helpful in evaluating the impact of out-of-control conditions. This review documents any recurring trends or problems evident in PE studies and evaluates their effect on environmental data.

Correlation of Laboratory Data -- This review determines whether the results of data obtained from related laboratory tests, e.g., Purgeable Organic Halides (POX) and Volatile Organics, are documented, and whether the significance of any differences is discussed in the reports.

2.7.5 QA Reports

There should be periodic reporting of pertinent QA/QC information to the project management to allow assessment of the overall effectiveness of the QA program. There are three major types of QA reports to project management:

Periodic Report on Key QA Activities -- Provides summary of key QA activities during the period, stressing measures that are being taken to improve data quality; describes significant quality problems observed and corrective actions taken; reports information regarding any changes in certification/accreditation status; describes involvement in resolution of quality issues with clients or agencies; reports any QA organizational changes; and provides notice of the distribution of revised documents controlled by the QA organization (i.e., procedures).

Report on Measurement Quality Indicators -- Includes the assessment of QC data gathered over the period, the frequency of analyses repeated due to unacceptable QC performance, and, if possible, the reason for the unacceptable performance and corrective action taken.

Reports on QA Assessments -- Includes the results of the assessments and the plan for correcting identified deficiencies; submitted immediately

following any internal or external on-site evaluation or upon receipt of the results of any performance evaluation studies.

3.0 FIELD OPERATIONS

The field operations should be conducted in such a way as to provide reliable information that meets the DQOs. To achieve this, certain minimal policies and procedures should be implemented. The OSW is considering revisions of Chapter Nine and Eleven of this manual. Supplemental information and guidance is available in the RCRA Ground-Water Monitoring Technical Enforcement Guidance Document (TEGD) (Reference 3). The project documentation should contain the information specified below.

3.1 FIELD LOGISTICS

The QAPjP should describe the type(s) of field operations to be performed and the appropriate area(s) in which to perform the work. The QAPjP should address ventilation, protection from extreme weather and temperatures, access to stable power, and provision for water and gases of required purity.

Whenever practical, the sampling site facilities should be examined prior to the start of work to ensure that all required items are available. The actual area of sampling should be examined to ensure that trucks, drilling equipment, and personnel have adequate access to the site.

The determination as to whether sample shipping is necessary should be made during planning for the project. This need is established by evaluating the analyses to be performed, sample holding times, and location of the site and the laboratory. Shipping or transporting of samples to a laboratory should be done within a timeframe such that recommended holding times are met.

Samples should be packaged, labelled, preserved (e.g., preservative added, iced, etc.), and documented in an area which is free of contamination and provides for secure storage. The level of custody and whether sample storage is needed should be addressed in the QAPjP.

Storage areas for solvents, reagents, standards, and reference materials should be adequate to preserve their identity, concentration, purity, and stability prior to use.

Decontamination of sampling equipment may be performed at the location where sampling occurs, prior to going to the sampling site, or in designated areas near the sampling site. Project documentation should specify where and how this work is accomplished. If decontamination is to be done at the site, water and solvents of appropriate purity should be available. The method of accomplishing decontamination, including the required materials, solvents, and water purity should be specified.

During the sampling process and during on-site or in situ analyses, waste materials are sometimes generated. The method for storage and disposal of these waste materials that complies with applicable local, state and Federal regulations should be specified. Adequate facilities should be provided for the collection and storage of all wastes, and these facilities should be operated so as to minimize environmental contamination. Waste storage and disposal facilities should comply with applicable federal, state, and local regulations.

The location of long-term and short-term storage for field records, and the measures to ensure the integrity of the data should be specified.

3.2 EQUIPMENT/INSTRUMENTATION

The equipment, instrumentation, and supplies at the sampling site should be specified and should be appropriate to accomplish the activities planned. The equipment and instrumentation should meet the requirements of specifications, methods, and procedures as specified in the QAPjP.

3.3 OPERATING PROCEDURES

The QAPjP should describe or make reference to all field activities that may affect data quality. For routinely performed activities, standard operating procedures (SOPs) are often prepared to ensure consistency and to save time and effort in preparing QAPjPs. Any deviation from an established procedure during a data collection activity should be documented. The procedures should be available for the indicated activities, and should include, at a minimum, the information described below.

3.3.1 Sample Management

The numbering and labeling system, chain-of-custody procedures, and how the samples are to be tracked from collection to shipment or receipt by the laboratory should be specified. Sample management procedures should also specify the holding times, volumes of sample required by the laboratory, required preservatives, and shipping requirements.

3.3.2 Reagent/Standard Preparation

The procedures describing how to prepare standards and reagents should be specified. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and record keeping for stocks and dilutions should be included.

3.3.3 Decontamination

The procedures describing decontamination of field equipment before and during the sample collection process should be specified. These procedures

should include cleaning materials used, the order of washing and rinsing with the cleaning materials, requirements for protecting or covering cleaned equipment, and procedures for disposing of cleaning materials.

3.3.4 Sample Collection

The procedures describing how the sampling operations are actually performed in the field should be specified. A simple reference to standard methods is not sufficient, unless a procedure is performed exactly as described in the published method. Methods from source documents published by the EPA, American Society for Testing and Materials, U.S. Department of the Interior, National Water Well Association, American Petroleum Institute, or other recognized organizations with appropriate expertise should be used, if possible. The procedures for sample collection should include at least the following:

- Applicability of the procedure,
- Equipment required,
- Detailed description of procedures to be followed in collecting the samples,
- Common problems encountered and corrective actions to be followed, and
- Precautions to be taken.

3.3.5 Field Measurements

The procedures describing all methods used in the field to determine a chemical or physical parameter should be described in detail. The procedures should address criteria from Section 4, as appropriate.

3.3.6 Equipment Calibration And Maintenance

The procedures describing how to ensure that field equipment and instrumentation are in working order should be specified. These describe calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, and service arrangements for equipment. Calibration and maintenance of field equipment and instrumentation should be in accordance with manufacturers' specifications or applicable test specifications and should be documented.

3.3.7 Corrective Action

The procedures describing how to identify and correct deficiencies in the sample collection process should be specified. These should include specific steps to take in correcting deficiencies such as performing additional decontamination of equipment, resampling, or additional training of field personnel. The procedures should specify that each corrective action should be documented with a description of the deficiency and the corrective action taken,

and should include the person(s) responsible for implementing the corrective action.

3.3.8 Data Reduction and Validation

The procedures describing how to compute results from field measurements and to review and validate these data should be specified. They should include all formulas used to calculate results and procedures used to independently verify that field measurement results are correct.

3.3.9 Reporting

The procedures describing the process for reporting the results of field activities should be specified.

3.3.10 Records Management

The procedures describing the means for generating, controlling, and archiving project-specific records and field operations records should be specified. These procedures should detail record generation and control and the requirements for record retention, including type, time, security, and retrieval and disposal authorities.

Project-specific records relate to field work performed for a project. These records may include correspondence, chain-of-custody records, field notes, all reports issued as a result of the work, and procedures used.

Field operations records document overall field operations and may include equipment performance and maintenance logs, personnel files, general field procedures, and corrective action reports.

3.3.11 Waste Disposal

The procedures describing the methods for disposal of waste materials resulting from field operations should be specified.

3.4 FIELD QA AND QC REQUIREMENTS

The QAPjP should describe how the following elements of the field QC program will be implemented.

3.4.1 Control Samples

Control samples are QC samples that are introduced into a process to monitor the performance of the system. Control samples, which may include blanks (e.g., trip, equipment, and laboratory), duplicates, spikes, analytical standards, and reference materials, can be used in different phases of the data collection process beginning with sampling and continuing through transportation, storage, and analysis.

Each day of sampling, at least one field duplicate and one equipment rinsate should be collected for each matrix sampled. If this frequency is not appropriate for the sampling equipment and method, then the appropriate changes should be clearly identified in the QAPjP. When samples are collected for volatile organic analysis, a trip blank is also recommended for each day that samples are collected. In addition, for each sampling batch (20 samples of one matrix type), enough volume should be collected for at least one sample so as to allow the laboratory to prepare one matrix spike and either one matrix duplicate or one matrix spike duplicate for each analytical method employed. This means that the following control samples are recommended:

- Field duplicate (one per day per matrix type)
- Equipment rinsate (one per day per matrix type)
- Trip blank (one per day, volatile organics only)
- Matrix spike (one per batch [20 samples of each matrix type])
- Matrix duplicate or matrix spike duplicate (one per batch)

Additional control samples may be necessary in order to assure data quality to meet the project-specific DQOs.

3.4.2 Acceptance Criteria

Procedures should be in place for establishing acceptance criteria for field activities described in the QAPjP. Acceptance criteria may be qualitative or quantitative. Field events or data that fall outside of established acceptance criteria may indicate a problem with the sampling process that should be investigated.

3.4.3 Deviations

All deviations from plan should be documented as to the extent of, and reason for, the deviation. Any activity not performed in accordance with procedures or QAPjPs is considered a deviation from plan. Deviations from plan may or may not affect data quality.

3.4.4 Corrective Action

Errors, deficiencies, deviations, certain field events, or data that fall outside established acceptance criteria should be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the system. The investigation of the problem and any subsequent corrective action taken should be documented.

3.4.5 Data Handling

All field measurement data should be reduced according to protocols described or referenced in the QAPjP. Computer programs used for data reduction should be validated before use and verified on a regular basis. All information used in the calculations should be recorded to enable reconstruction of the final result at a later date.

Data should be reported in accordance with the requirements of the end-user as described in the QAPjP.

3.5 QUALITY ASSURANCE REVIEW

The QA Review consists of internal and external assessments to ensure that QA/QC procedures are in use and to ensure that field staff conform to these procedures. QA review should be conducted as deemed appropriate and necessary.

3.6 FIELD RECORDS

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning project activities. These records, particularly those that are anticipated to be used as evidentiary data, should directly support current or ongoing technical studies and activities and should provide the historical evidence needed for later reviews and analyses. Records should be legible, identifiable, and retrievable and protected against damage, deterioration, or loss. The discussion in this section (3.6) outlines recommended procedures for record keeping. Organizations which conduct field sampling should develop appropriate record keeping procedures which satisfy relevant technical and legal requirements.

Field records generally consist of bound field notebooks with prenumbered pages, sample collection forms, personnel qualification and training forms, sample location maps, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and field change request forms. All records should be written in indelible ink.

Procedures for reviewing, approving, and revising field records should be clearly defined, with the lines of authority included. It is recommended that all documentation errors should be corrected by drawing a single line through the error so it remains legible and should be initialed by the responsible individual, along with the date of change. The correction should be written adjacent to the error.

Records should include (but are not limited to) the following:

Calibration Records & Traceability of Standards/Reagents -- Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program should include provisions for documentation of frequency, conditions, standards, and records reflecting the calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used. A program for verifying and documenting the accuracy of all working standards against primary grade standards should be routinely followed.

Sample Collection -- To ensure maximum utility of the sampling effort and resulting data, documentation of the sampling protocol, as performed in the field, is essential. It is recommended that sample collection records contain, at a minimum, the names of persons conducting the activity, sample number, sample location, equipment used, climatic conditions, documentation of adherence to protocol, and unusual observations. The actual sample collection record is usually one of the following: a bound field notebook with prenumbered pages, a pre-printed form, or digitized information on a computer tape or disc.

Chain-of-Custody Records -- The chain-of-custody involving the possession of samples from the time they are obtained until they are disposed or shipped off-site should be documented as specified in the QAPjP and should include the following information: (1) the project name; (2) signatures of samplers; (3) the sample number, date and time of collection, and grab or composite sample designation; (4) signatures of individuals involved in sample transfer; and (5) if applicable, the air bill or other shipping number.

Maps and Drawings -- Project planning documents and reports often contain maps. The maps are used to document the location of sample collection points and monitoring wells and as a means of presenting environmental data. Information used to prepare maps and drawings is normally obtained through field surveys, property surveys, surveys of monitoring wells, aerial photography or photogrammetric mapping. The final, approved maps and/or drawings should have a revision number and date and should be subject to the same controls as other project records.

QC Samples -- Documentation for generation of QC samples, such as trip and equipment rinsate blanks, duplicate samples, and any field spikes should be maintained.

Deviations -- All deviations from procedural documents and the QAPjP should be recorded in the site logbook.

Reports -- A copy of any report issued and any supporting documentation should be retained.

4.0 LABORATORY OPERATIONS

The laboratory should conduct its operations in such a way as to provide reliable information. To achieve this, certain minimal policies and procedures should be implemented.

4.1 FACILITIES

The QAPjP should address all facility-related issues that may impact project data quality. Each laboratory should be of suitable size and

construction to facilitate the proper conduct of the analyses. Adequate bench space or working area per analyst should be provided. The space requirement per analyst depends on the equipment or apparatus that is being utilized, the number of samples that the analyst is expected to handle at any one time, and the number of operations that are to be performed concurrently by a single analyst. Other issues to be considered include, but are not limited to, ventilation, lighting, control of dust and drafts, protection from extreme temperatures, and access to a source of stable power.

Laboratories should be designed so that there is adequate separation of functions to ensure that no laboratory activity has an adverse effect on the analyses. The laboratory may require specialized facilities such as a perchloric acid hood or glovebox.

Separate space for laboratory operations and appropriate ancillary support should be provided, as needed, for the performance of routine and specialized procedures.

As necessary to ensure secure storage and prevent contamination or misidentification, there should be adequate facilities for receipt and storage of samples. The level of custody required and any special requirements for storage such as refrigeration should be described in planning documents.

Storage areas for reagents, solvents, standards, and reference materials should be adequate to preserve their identity, concentration, purity, and stability.

Adequate facilities should be provided for the collection and storage of all wastes, and these facilities should be operated so as to minimize environmental contamination. Waste storage and disposal facilities should comply with applicable federal, state, and local regulations.

The location of long-term and short-term storage of laboratory records and the measures to ensure the integrity of the data should be specified.

4.2 EQUIPMENT/INSTRUMENTATION

Equipment and instrumentation should meet the requirements and specifications of the specific test methods and other procedures as specified in the QAPjP. The laboratory should maintain an equipment/instrument description list that includes the manufacturer, model number, year of purchase, accessories, and any modifications, updates, or upgrades that have been made.

4.3 OPERATING PROCEDURES

The QAPjP should describe or make reference to all laboratory activities that may affect data quality. For routinely performed activities, SOPs are often prepared to ensure consistency and to save time and effort in preparing QAPjPs.

Any deviation from an established procedure during a data collection activity should be documented. It is recommended that procedures be available for the indicated activities, and include, at a minimum, the information described below.

4.3.1 Sample Management

The procedures describing the receipt, handling, scheduling, and storage of samples should be specified.

Sample Receipt and Handling -- These procedures describe the precautions to be used in opening sample shipment containers and how to verify that chain-of-custody has been maintained, examine samples for damage, check for proper preservatives and temperature, and log samples into the laboratory sample streams.

Sample Scheduling -- These procedures describe the sample scheduling in the laboratory and includes procedures used to ensure that holding time requirements are met.

Sample Storage -- These procedures describe the storage conditions for all samples, verification and documentation of daily storage temperature, and how to ensure that custody of the samples is maintained while in the laboratory.

4.3.2 Reagent/Standard Preparation

The procedures describing how to prepare standards and reagents should be specified. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and recordkeeping for stocks and dilutions should be included.

4.3.3 General Laboratory Techniques

The procedures describing all essentials of laboratory operations that are not addressed elsewhere should be specified. These techniques should include, but are not limited to, glassware cleaning procedures, operation of analytical balances, pipetting techniques, and use of volumetric glassware.

4.3.4 Test Methods

Procedures for test methods describing how the analyses are actually performed in the laboratory should be specified. A simple reference to standard methods is not sufficient, unless the analysis is performed exactly as described in the published method. Whenever methods from SW-846 are not appropriate, recognized methods from source documents published by the EPA, American Public Health Association (APHA), American Society for Testing and Materials (ASTM), the National Institute for Occupational Safety and Health (NIOSH), or other recognized organizations with appropriate expertise should be used, if possible.

The documentation of the actual laboratory procedures for analytical methods should include the following:

Sample Preparation and Analysis Procedures -- These include applicable holding time, extraction, digestion, or preparation steps as appropriate to the method; procedures for determining the appropriate dilution to analyze; and any other information required to perform the analysis accurately and consistently.

Instrument Standardization -- This includes concentration(s) and frequency of analysis of calibration standards, linear range of the method, and calibration acceptance criteria.

Sample Data -- This includes recording requirements and documentation including sample identification number, analyst, data verification, date of analysis and verification, and computational method(s).

Precision and Bias -- This includes all analytes for which the method is applicable and the conditions for use of this information.

Detection and Reporting Limits -- This includes all analytes in the method.

Test-Specific QC -- This describes QC activities applicable to the specific test and references any applicable QC procedures.

4.3.5 Equipment Calibration and Maintenance

The procedures describing how to ensure that laboratory equipment and instrumentation are in working order should be specified. These procedures include calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, service arrangements for all equipment, and spare parts available in-house. Calibration and maintenance of laboratory equipment and instrumentation should be in accordance with manufacturers' specifications or applicable test specifications and should be documented.

4.3.6 QC

The type, purpose, and frequency of QC samples to be analyzed in the laboratory and the acceptance criteria should be specified. Information should include the applicability of the QC sample to the analytical process, the statistical treatment of the data, and the responsibility of laboratory staff and management in generating and using the data. Further details on development of project-specific QC protocols are described in Section 4.4.

4.3.7 Corrective Action

The procedures describing how to identify and correct deficiencies in the analytical process should be specified. These should include specific steps to take in correcting the deficiencies such as preparation of new standards and

reagents, recalibration and restandardization of equipment, reanalysis of samples, or additional training of laboratory personnel in methods and procedures. The procedures should specify that each corrective action should be documented with a description of the deficiency and the corrective action taken, and should include the person(s) responsible for implementing the corrective action.

4.3.8 Data Reduction and Validation

The procedures describing how to review and validate the data should be specified. They should include procedures for computing and interpreting the results from QC samples, and independent procedures to verify that the analytical results are reported correctly. In addition, routine procedures used to monitor precision and bias, including evaluations of reagent, equipment rinsate, and trip blanks, calibration standards, control samples, duplicate and matrix spike samples, and surrogate recovery, should be detailed in the procedures. More detailed validation procedures should be performed when required in the contract or QAPjP.

4.3.9 Reporting

The procedures describing the process for reporting the analytical results should be specified.

4.3.10 Records Management

The procedures describing the means for generating, controlling, and archiving laboratory records should be specified. The procedures should detail record generation and control, and the requirements for record retention, including type, time, security, and retrieval and disposal authorities.

Project-specific records may include correspondence, chain-of-custody records, request for analysis, calibration data records, raw and finished analytical and QC data, data reports, and procedures used.

Laboratory operations records may include laboratory notebooks, instrument performance logs and maintenance logs in bound notebooks with prenumbered pages; laboratory benchsheets; software documentation; control charts; reference material certification; personnel files; laboratory procedures; and corrective action reports.

4.3.11 Waste Disposal

The procedures describing the methods for disposal of chemicals including standard and reagent solutions, process waste, and samples should be specified.

4.4 LABORATORY QA AND QC PROCEDURES

The QAPjP should describe how the following required elements of the laboratory QC program are to be implemented.

4.4.1 Method Proficiency

Procedures should be in place for demonstrating proficiency with each analytical method routinely used in the laboratory. These should include procedures for demonstrating the precision and bias of the method as performed by the laboratory and procedures for determining the method detection limit (MDL). All terminology, procedures and frequency of determinations associated with the laboratory's establishment of the MDL and the reporting limit should be well-defined and well-documented. Documented precision, bias, and MDL information should be maintained for all methods performed in the laboratory.

4.4.2 Control Limits

Procedures should be in place for establishing and updating control limits for analysis. Control limits should be established to evaluate laboratory precision and bias based on the analysis of control samples. Typically, control limits for bias are based on the historical mean recovery plus or minus three standard deviation units, and control limits for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units. Procedures should be in place for monitoring historical performance and should include graphical (control charts) and/or tabular presentations of the data.

4.4.3 Laboratory Control Procedures

Procedures should be in place for demonstrating that the laboratory is in control during each data collection activity. Analytical data generated with laboratory control samples that fall within prescribed limits are judged to be generated while the laboratory was in control. Data generated with laboratory control samples that fall outside the established control limits are judged to be generated during an "out-of-control" situation. These data are considered suspect and should be repeated or reported with qualifiers.

Laboratory Control Samples -- Laboratory control samples should be analyzed for each analytical method when appropriate for the method. A laboratory control sample consists of either a control matrix spiked with analytes representative of the target analytes or a certified reference material.

Laboratory control sample(s) should be analyzed with each batch of samples processed to verify that the precision and bias of the analytical process are within control limits. The results of the laboratory control sample(s) are compared to control limits established for both precision and bias to determine usability of the data.

Method Blank -- When appropriate for the method, a method blank should be analyzed with each batch of samples processed to assess contamination

levels in the laboratory. Guidelines should be in place for accepting or rejecting data based on the level of contamination in the blank.

Procedures should be in place for documenting the effect of the matrix on method performance. When appropriate for the method, there should be at least one matrix spike and either one matrix duplicate or one matrix spike duplicate per analytical batch. Additional control samples may be necessary to assure data quality to meet the project-specific DQOs.

Matrix-Specific Bias -- Procedures should be in place for determining the bias of the method due to the matrix. These procedures should include preparation and analysis of matrix spikes, selection and use of surrogates for organic methods, and the method of standard additions for metal and inorganic methods. When the concentration of the analyte in the sample is greater than 0.1%, no spike is necessary.

Matrix-Specific Precision -- Procedures should be in place for determining the precision of the method for a specific matrix. These procedures should include analysis of matrix duplicates and/or matrix spike duplicates. The frequency of use of these techniques should be based on the DQO for the data collection activity.

Matrix-Specific Detection Limit -- Procedures should be in place for determining the MDL for a specific matrix type (e.g., wastewater treatment sludge, contaminated soil, etc).

4.4.4 Deviations

Any activity not performed in accordance with laboratory procedures or QAPjPs is considered a deviation from plan. All deviations from plan should be documented as to the extent of, and reason for, the deviation.

4.4.5 Corrective Action

Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria should be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken should be documented.

4.4.6 Data Handling

Data resulting from the analyses of samples should be reduced according to protocols described in the laboratory procedures. Computer programs used for data reduction should be validated before use and verified on a regular basis. All information used in the calculations (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, and blank- or background-correction protocols) should be recorded in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry

weight for solids, extract volume, dilution factor used) should also be maintained in order to enable reconstruction of the final result at a later date.

All data should be reviewed by a second analyst or supervisor according to laboratory procedures to ensure that calculations are correct and to detect transcription errors. Spot checks should be performed on computer calculations to verify program validity. Errors detected in the review process should be referred to the analyst(s) for corrective action. Data should be reported in accordance with the requirements of the end-user. It is recommended that the supporting documentation include at a minimum:

- Laboratory name and address.
- Sample information (including unique sample identification, sample collection date and time, date of sample receipt, and date(s) of sample preparation and analysis).
- Analytical results reported with an appropriate number of significant figures.
- Detection limits that reflect dilutions, interferences, or correction for equivalent dry weight.
- Method reference.
- Appropriate QC results (correlation with sample batch should be traceable and documented).
- Data qualifiers with appropriate references and narrative on the quality of the results.

4.5 QUALITY ASSURANCE REVIEW

The QA review consists of internal and external assessments to ensure that QA/QC procedures are in use and to ensure that laboratory staff conform to these procedures. QA review should be conducted as deemed appropriate and necessary.

4.6 LABORATORY RECORDS

Records provide the direct evidence and support for the necessary technical interpretations, judgements, and discussions concerning project activities. These records, particularly those that are anticipated to be used as evidentiary data, should directly support technical studies and activities, and provide the historical evidence needed for later reviews and analyses. Records should be legible, identifiable, and retrievable, and protected against damage, deterioration, or loss. The discussion in this section (4.6) outlines recommended procedures for record keeping. Organizations which conduct field

sampling should develop appropriate record keeping procedures which satisfy relevant technical and legal requirements.

Laboratory records generally consist of bound notebooks with prenumbered pages, personnel qualification and training forms, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and analytical change request forms. All records should be written in indelible ink.

Procedures for reviewing, approving, and revising laboratory records should be clearly defined, with the lines of authority included. Any documentation errors should be corrected by drawing a single line through the error so that it remains legible and should be initialed by the responsible individual, along with the date of change. The correction is written adjacent to the error.

Strip-chart recorder printouts should be signed by the person who performed the instrumental analysis. If corrections need to be made in computerized data, a system parallel to the corrections for handwritten data should be in place.

Records of sample management should be available to permit the re-creation of an analytical event for review in the case of an audit or investigation of a dubious result.

Laboratory records should include, at least, the following:

Operating Procedures -- Procedures should be available to those performing the task outlined. Any revisions to laboratory procedures should be written, dated, and distributed to all affected individuals to ensure implementation of changes. Areas covered by operating procedures are given in Sections 3.3 and 4.3.

Quality Assurance Plans -- The QAPjP should be on file.

Equipment Maintenance Documentation -- A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, the maintenance guidelines of the equipment manufacturer should be followed. When maintenance is necessary, it should be documented in either standard forms or in logbooks. Maintenance procedures should be clearly defined and written for each measurement system and required support equipment.

Proficiency -- Proficiency information on all compounds reported should be maintained and should include (1) precision; (2) bias; (3) method detection limits; (4) spike recovery, where applicable; (5) surrogate recovery, where applicable; (6) checks on reagent purity, where applicable; and (7) checks on glassware cleanliness, where applicable.

Calibration Records & Traceability of Standards/Reagents -- Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program should include provisions for documenting frequency, conditions, standards, and records reflecting the

calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used. A program for verifying and documenting the accuracy and traceability of all working standards against appropriate primary grade standards or the highest quality standards available should be routinely followed.

Sample Management -- All required records pertaining to sample management should be maintained and updated regularly. These include chain-of-custody forms, sample receipt forms, and sample disposition records.

Original Data -- The raw data and calculated results for all samples should be maintained in laboratory notebooks, logs, benchsheets, files or other sample tracking or data entry forms. Instrumental output should be stored in a computer file or a hardcopy report.

QC Data -- The raw data and calculated results for all QC and field samples and standards should be maintained in the manner described in the preceding paragraph. Documentation should allow correlation of sample results with associated QC data. Documentation should also include the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes, and matrix spike duplicates.

Correspondence -- Project correspondence can provide evidence supporting technical interpretations. Correspondence pertinent to the project should be kept and placed in the project files.

Deviations -- All deviations from procedural and planning documents should be recorded in laboratory notebooks. Deviations from QAPjPs should be reviewed and approved by the authorized personnel who performed the original technical review or by their designees.

Final Report -- A copy of any report issued and any supporting documentation should be retained.

5.0 DEFINITIONS

The following terms are defined for use in this document:

ACCURACY — The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

BATCH: — A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit (see Section 3.4.1 for field

samples and Section 4.4.3 for laboratory samples). For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

BIAS: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike). Thus, the bias (B) due to matrix effects based on a matrix spike is calculated as:

$$B = (x_s - x_u) - K$$

where:

x_s = measured value for spiked sample,
x_u = measured value for unspiked sample, and
K = known value of the spike in the sample.

Using the following equation yields the percent recovery (%R).

$$\%R = 100\ (x_s - x_u)/\ K$$

BLANK: see Equipment Rinsate, Method Blank, Trip Blank.

CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.

DATA QUALITY OBJECTIVES (DQOs): A statement of the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data (see reference 2, EPA/QAMS, July 16, 1986). This is qualitatively distinct from quality measurements such as precision, bias, and detection limit.

DATA VALIDATION: The process of evaluating the available data against the project DQOs to make sure that the objectives are met. Data validation may be very rigorous, or cursory, depending on project DQOs. The available data reviewed will include analytical results, field QC data and lab QC data, and may also include field records.

DUPLICATE: see Matrix Duplicate, Field Duplicate, Matrix Spike Duplicate.

EQUIPMENT BLANK: see Equipment Rinsate.

EQUIPMENT RINSATE: A sample of analyte-free media which has been used to

	rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.
ESTIMATED QUANTITATION LIMIT (EQL):	The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs in SW-846 are provided for guidance and may not always be achievable.
FIELD DUPLICATES:	Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.
LABORATORY CONTROL SAMPLE:	A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.
MATRIX:	The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.
MATRIX DUPLICATE:	An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.
MATRIX SPIKE:	An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.
MATRIX SPIKE DUPLICATES:	Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.
METHOD BLANK:	An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern should not be higher than the highest of either:

(1)The method detection limit, or

(2)Five percent of the regulatory limit for that analyte, or

(3)Five percent of the measured concentration in the sample.

METHOD DETECTION LIMIT (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of three analyses of a matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL, where the t-statistic is obtained from standard references or the table below.

No. of samples:	t-statistic
3	6.96
4	4.54
5	3.75
6	3.36
7	3.14
8	3.00
9	2.90
10	2.82

Estimate the MDL as follows:
Obtain the concentration value that corresponds to:

a) an instrument signal/noise ratio within the range of 2.5 to 5.0, or

b) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

Determine the variance (S^2) for each analyte as follows:

$$s^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n}(x_i - \bar{x})^2\right]$$

where x_i = the ith measurement of the variable x and $\bar{x}$ = the average value of x;

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

Determine the standard deviation (s) for each analyte as follows:

$$s = (S^2)^{1/2}$$

Determine the MDL for each analyte as follows:

$$MDL = t_{(n-1,\ \alpha = .99)}(s)$$

where $t_{(n-1,\ \alpha = .99)}$ is the one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99 percent level.

ORGANIC-FREE REAGENT WATER:

For volatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. Organic-free reagent water may also be prepared by boiling water for 15 minutes and, subsequently, while maintaining the temperature at 90°C, bubbling a contaminant-free inert gas through the water for 1 hour.

For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

PRECISION:

The agreement among a set of replicate measurements

without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are the relative standard deviation (RSD) or the coefficient of variation (CV),

$$RSD = CV = 100\ S/\bar{x},$$

where:
x = the arithmetic mean of the x_i measurements, and S = variance; and the relative percent difference (RPD) when only two samples are available.

$$RPD = 100\ [(x_1 - x_2)/\{(x_1 + x_2)/2\}].$$

PROJECT: Single or multiple data collection activities that are related through the same planning sequence.

QUALITY ASSURANCE PROJECT PLAN (QAPjP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

RCRA: The Resource Conservation and Recovery Act.

REAGENT BLANK: See Method Blank.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

SPLIT SAMPLES: Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.

STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

TRIP BLANK: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

6.0 REFERENCES

1. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, Washington, DC 20460.

2. Development of Data Quality Objectives, Description of Stages I and II, July 16, 1986, Quality Assurance Management Staff, ORD, U.S. EPA, Washington, DC 20460.

3. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, September, 1986, Office of Waste Programs Enforcement. OSWER, U.S. EPA, Washington, DC, 20460.

4. DQO Training Software, Version 6.5, December, 1988, Quality Assurance Management Staff, ORD, U.S. EPA, Washington, DC 20460.

5. Preparing Perfect Project Plans, EPA/600/9-89/087, October 1989, Risk Reduction Engineering Laboratory (Guy Simes), Cincinnati OH.

6. ASTM Method D 1129-77, Specification for Reagent Water. 1991 Annual Book of ASTM Standards. Volume 11.01 Water and Environmental Technology.

7. Generation of Environmental Data Related to Waste Management Activities (Draft). February 1989. ASTM.

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* Definition of term.

CHAPTER TWO

CHOOSING THE CORRECT PROCEDURE

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in these methods are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

2.0 INTRODUCTION

The purpose of this chapter is to aid the analyst in choosing the appropriate methods for sample analyses, based upon the sample matrix and the analytes to be determined. The ultimate responsibility for producing reliable analytical results lies with the entity subject to the regulation. Therefore, members of the regulated community are advised to refer to this chapter and to consult with knowledgeable laboratory personnel when choosing the most appropriate suite of analytical methods. In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements.

SW-846 analytical methods are written as quantitative trace analytical methods to demonstrate that a waste does not contain analytes of concern that cause it to be managed as a hazardous waste. As such, these methods typically contain relatively stringent recommended quality control (QC) criteria appropriate to trace analyses. However, if a particular application does not require data of this quality, less stringent QC criteria may and should be used.

The choice of the appropriate sequence of analytical methods depends on the information sought and on the experience of the analyst. Appropriate selection is confirmed by the usability of data (i.e., adequate for its intended use). The use of the recommended procedures, whether they are approved or mandatory, does not release the analyst from demonstrating the correct execution of the method.

Sec. 2.1 provides guidance regarding the analytical flexibility inherent to SW-846 methods and the precedence of various QC criteria. Sec. 2.2 reviews the information required to choose the correct combination of methods for an analytical procedure. Sec. 2.3 provides useful information on implementing the method selection guidance for organic analyses. Sec. 2.4 provides guidance on choosing procedures for characteristic analyses. Sec. 2.5 provides guidance on the determination of analytes in groundwater. Finally, Sec. 2.6 provides information regarding choosing procedures for inorganic analyte analyses. Tables and figures referenced in this chapter are sequentially located after the last page chapter text.

2.1 GUIDANCE REGARDING FLEXIBILITY INHERENT TO SW-846 METHODS AND THE PRECEDENCE OF SW-846 QUALITY CONTROL CRITERIA

The specific products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency for use in the method. Glassware, reagents, supplies, equipment and settings other than those listed in this manual may be employed, provided that method performance appropriate for the intended RCRA application has been documented. Such performance includes consideration of precision, accuracy (or bias), recovery, representativeness, comparability, and sensitivity (quantitation or reporting limits) relative to the data quality objectives for the intended use of the analytical results. In response to this inherent flexibility, if an alternative analytical procedure is employed, then EPA expects the laboratory to demonstrate and document that the procedure is capable of providing appropriate performance for its intended application. This demonstration must not be performed after the fact, but as part of the laboratory's initial demonstration of proficiency with the method. The documentation should be in writing, maintained in the laboratory, and available for inspection upon request by authorized representatives of the appropriate regulatory authorities. The documentation should include the performance data as well as a detailed description of the procedural steps as performed (i.e., a written standard operating procedure).

Given this allowance for flexibility, EPA wishes to emphasize that this manual also contains 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 <u>not</u> subject to the flexibility afforded in other methods.

Analysts and data users are advised that even for those analytes that are not method-defined, different procedures may produce some difference in results. Common examples include the differences in recoveries of phenolic compounds extracted from water by separatory funnel (Method 3510) and continuous liquid-liquid (Method 3520) extraction techniques, differences in recoveries of many compounds between Soxhlet (Method 3540) and ultrasonic (Method 3550) extraction techniques, and differences resulting from the choice of acid digestion of metals (Method 3050) or microwave digestion (Method 3051). Where practical, the Agency has included guidance in the individual methods regarding known potential problems, and analysts are advised to review this information carefully in choosing or modifying analytical procedures. Chapter One describes a variety of QC procedures that may be used to evaluate the quality of the analytical results. Additional QC procedures may be described in the individual methods. The results of these QC procedures should be used by the analyst to evaluate if the choice of the analytical procedures and/or any modifications are appropriate to generate data of the quality necessary to satisfy the data quality needs of the intended application.

The performance data included in the SW-846 methods are <u>not</u> intended to be used as absolute QC acceptance criteria for method performance. The data are intended to be guidance, by providing typical method performance in typical matrices, to assist the analyst in selection of the appropriate method for the intended application. In addition, it is the responsibility of the laboratory to establish actual operating parameters and in-house QC acceptance criteria, based on its own laboratory SOPs and in-house QC program, to demonstrate appropriate performance of the methods used in that laboratory for the RCRA analytical applications for which they are intended.

The regulated community is further advised that the methods here or from other sources need only be used for those specific analytes of concern that are subject to regulation or other monitoring requirements. The fact that a method provides a long list of analytes does not mean that each of those analytes is subject to any or all regulations, or that all of those analytes must be analyzed each time the method is employed, or that all of the analytes can be analyzed using a single sample preparation procedure. It is EPA's intention that the target analyte list for any procedure includes those analytes necessary to meet the data quality objectives of the project, i.e., those analytes subject to monitoring requirements and set out in a RCRA permit (or other applicable regulation), plus those analytes used in the methods for QC purposes, such as surrogates, internal standards, system performance check compounds, etc. Additional analytes, not included on the analyte list of a particular method(s) but needed for a specific project, may be analyzed by that particular method(s), if appropriate performance can be demonstrated for the analytes of concern in the matrices of concern at the levels of concern.

2.1.1 Trace analysis vs. macroanalysis

Through the choice of sample size and concentration procedures, the methods presented in SW-846 were designed to address the problem of "trace" analyses (<1000 ppm), and have been developed for an optimized working range. These methods are also applicable to "minor" (1000 ppm - 10,000 ppm) and "major" (>10,000 ppm) analyses, as well, through use of appropriate sample preparation techniques that result in analyte concentrations within that optimized range. Such sample preparation techniques include:

1. adjustment of size of sample prepared for analysis (for homogeneous samples),
2. adjustment of injection volumes,
3. dilution or concentration of sample,
4. elimination of concentration steps prescribed for "trace" analyses, and
5. direct injection (of samples to be analyzed for volatile constituents).

The performance data presented in each of these methods were generated from "trace" analyses, and may not be applicable to "minor" and "major" analyses. Generally, extraction efficiency improves as concentration increases.

CAUTION: Great care should be taken when performing trace analyses after the analysis of concentrated samples, given the possibility of contamination.

2.1.2 Choice of apparatus and preparation of reagents

Since many types and sizes of glassware and supplies are commercially available, and since it is possible to prepare reagents and standards in many different ways, the apparatus, reagents, and volumes included in these methods may be replaced by any similar types as long as this substitution does not affect the overall quality of the analyses.

2.1.3 Quality control criteria precedence

Chapter One contains general quality control (QC) guidance for analyses using SW-846 methods. QC guidance specific to a given analytical technique (e.g., extraction, cleanup, sample introduction, or analysis) may be found in Methods 3500, 3600, 5000, 7000, and 8000. Method-specific QC criteria may be found in Sec. 8.0 of most older individual methods, in Sec. 9.0 of newer methods, or in Sec. 11.0 of some air sampling methods. When inconsistencies exist between the information in these locations, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One.

2.2 INFORMATION NECESSARY FOR CHOOSING THE CORRECT PROCEDURE

In order to choose the correct combination of methods to comprise the appropriate analytical procedure, some basic information is necessary. This includes information on:

- The physical state of the sample
- The analytes of interest
- The sensitivity or quantitation limits needed
- The analytical objective
- Whether the purpose is quantitation or monitoring
- What sample containers and preservation will be used and what holding times may apply

2.2.1 Physical state(s) of sample

The phase characteristics of the sample must be known. There are several general categories of phases into which the sample may be categorized, including:

Aqueous	Oil or other Organic Liquid
Sludge	Stack Sampling (VOST) Condensate
TCLP or EP Extract	Multiphase Sample
Solid	
Groundwater	

There may be a substantial degree of overlap between the phases listed above and it may be useful to further divide these phases in certain instances. A multiphase sample may be a combination of aqueous, organic liquid, sludge, and/or solid phases, and generally must undergo a phase separation as the first step in the analytical procedure.

2.2.2 Analytes of interest

Analytes may be divided into various classes, based on the determinative methods which are used to identify and quantify them. The most basic differentiation is between organic (e.g., carbon-containing) analytes and inorganic (e.g., metals and anions) analytes.

Table 2-1 is an alphabetical list of analytes cited within the SW-846 organic determinative methods (excludes immunoassay and other screening methods). These analytes have been evaluated by those methods. The methods may also be applicable to other analytes that are similar to those listed. Tables 2-2 through 2-38 list the analytes for each organic determinative method. Table 2-39 indicates which methods are applicable to inorganic analytes.

NOTE: Analysts should review the discussion in Sec. 2.1 of this chapter with regard to the presence of an analyte in a method versus the need for its analysis for a given project.

2.2.3 Sensitivity or quantitation limits

Some regulations may require a specific sensitivity or quantitation limit for an analysis, as in the determination of analytes for the Toxicity Characteristic (TC). Drinking water quantitation limits, for those specific organic and metallic analytes covered by the National Primary Drinking Water Regulations, are desired in the analysis of groundwater.

2.2.4 Analytical objective

Knowledge of the analytical objective is essential in the choice of sample preparation procedures and in the selection of a determinative method. This is especially true when the

sample has more than one phase. Knowledge of the analytical objective may not be possible or desirable at all management levels, but that information should be included in the project planning document and transmitted to the analytical laboratory management to ensure that the correct techniques are used during the analytical effort.

2.2.5 Quantitation or monitoring

The strategy for quantitation of compounds in environmental or process samples may be contrasted with the strategy for collecting monitoring data. Quantitation samples define initial conditions. When there is little information available about the composition of the sample source, e.g., a well or process stream, mass spectral identification of organic analytes leads to fewer false positive results. Thus, the most practical form of quantitation for organic analytes is often mass spectral identification. However, where the sensitivity requirements exceed those that can be achieved using mass spectral methods (e.g., GC/MS or HPLC/MS), it may be necessary to employ a more sensitive quantitation method (e.g., electron capture). In these instances, the risk of false positive results may be minimized by confirming the results through a second analysis with a dissimilar detector or chromatographic column. Thus, the choice of technique for organic analytes may be governed by the quantitation limit requirements and potential interferants.

Similarly, the choice of technique for metals is governed by the quantitation limit requirements and potential interferants.

In contrast, monitoring samples are analyzed to confirm existing and on-going conditions, tracking the presence or absence of known constituents in an environmental or process matrix. In well-defined matrices and under stable analytical conditions, less compound-specific quantitation modes may be used, as the risk of false positive results is less.

2.2.6 Sample preservation and holding times

Table 2-40 provides information regarding recommended sample preservation techniques, sample holding times, and other information. Similar information may be found in Table 3-1 of Chapter Three (inorganic analytes) and Table 4-1 of Chapter Four (organic analytes). Samples need to be extracted and analyzed within the recommended holding times for the results to be considered reflective of native concentrations as collected. Analytical data generated outside of the recommended holding times should typically be considered as minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold, but cannot be used to demonstrate that a waste is not hazardous. However, regarding the information in Table 2-40, a longer holding time may be appropriate if it can be demonstrated that reported concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

2.3 CHOOSING PROCEDURES FOR ORGANIC ANALYSES

Figure 2-1 summarizes the organic analysis options available in SW-846.

2.3.1 Extraction and sample preparation procedures for organic analytes

SW-846 methods for preparing samples for organic analytes are shown in Table 2-41. Method 3500 and associated methods should be consulted for further details on preparing the sample for analysis.

2.3.1.1 Aqueous samples

Methods 3510, 3520, and 3535 may be used for extraction of the semivolatile organic compounds from aqueous samples. The choice of a preparative method depends on the sample. Method 3510, a separatory funnel liquid-liquid extraction technique, is appropriate for samples which will not form a persistent emulsion interface between the sample and the extraction solvent. The formation of an emulsion that cannot be broken up by mechanical techniques will prevent proper extraction of the sample. Method 3520, a continuous liquid-liquid extraction technique, may be used for any aqueous sample and will minimize emulsion formation.

Method 3535 is solid-phase extraction technique that has been tested for organochlorine pesticides, phthalate esters, polychlorinated biphenyls (PCBs), organophosphorus pesticides, nitroaromatics and nitramines, and some explosive compounds, and may be applicable to other semivolatile and extractable compounds as well. The aqueous sample is passed through a solid sorbent material which traps the analytes. They are then eluted from the solid-phase sorbent with a small volume of organic solvent. This technique may be used to minimize the volumes of organic solvents that are employed, but may not be appropriate for aqueous samples with high suspended solids contents.

2.3.1.1.1 Acidic extraction of phenols and acid analytes

The solvent extract obtained by performing Method 3510, 3520, or 3535 at a pH less than or equal to 2 will contain the phenols and acid/neutral extractable organics of interest, and may contain some mildly basic compounds. The particular pH extraction conditions needs to be defined during the project planning process based on the desired target analytes and performance goals.

2.3.1.1.2 Basic or neutral extraction of semivolatile analytes

The solvent extract obtained by performing Method 3510, 3520, or 3535 at a basic pH will contain the organic bases of interest, if acid extraction is performed first. It will also contain the neutral compounds of interest, if acid extraction is not performed. Refer to Table 1 in the extraction methods (3510 and/or 3520) for guidance on the requirements for pH adjustment prior to extraction and analysis.

2.3.1.2 Solid samples

Soxhlet extraction (Methods 3540, 3541 and 3542), pressurized fluid extraction (Method 3545), microwave extraction (Method 3546) and ultrasonic extraction (Method 3550) may be used with solid samples. Consolidated samples should be ground finely enough to pass through a 1-mm sieve. In limited applications, waste dilution (Methods 3580 and 3585) may be used if the entire sample is soluble in the specified solvent.

Methods 3540, 3541, 3542, 3545, 3546 and 3550 are neutral-pH extraction techniques and therefore, depending on the analysis requirements, acid-base partition cleanup (Method 3650) may be necessary. Method 3650 will only be needed if chromatographic interferences are severe enough to prevent quantitation of the analytes of interest. This separation will be most important if a GC method is chosen for analysis of the sample. If GC/MS is used, the ion selectivity of the technique may compensate for chromatographic interferences.

There are three extraction procedures for solid samples that employ supercritical fluid extraction (SFE). Method 3560 is a technique for the extraction of petroleum hydrocarbons from various solid matrices using carbon dioxide at elevated temperature and pressure. Method 3561 may be used to selectively extract polynuclear aromatic hydrocarbons (PAHs) from solid matrices using supercritical carbon dioxide and appropriate modifiers, based on the determinative procedure to be used. Method 3562 may be used to selectively extract organochlorine pesticides or PCBs from solid matrices using supercritical carbon dioxide.

2.3.1.3 Oils and organic liquids

Method 3580, waste dilution, may be used to prepare oils and organic liquid samples for analysis of semivolatile and extractable organic analytes by GC or GC/MS. Method 3585 may be employed for the preparation of these matrices for volatiles analysis by GC or GC/MS. To avoid overloading the analytical detection system, care must be exercised to ensure that proper dilutions are made. Methods 3580 and 3585 give guidance on performing waste dilutions.

To remove interferences for semivolatiles and extractables, Method 3611 (Alumina cleanup) may be performed on an oil sample directly, without prior sample preparation.

Method 3650 is the only other preparative procedure for oils and other organic liquids. This procedure is a back extraction into an aqueous phase. It is generally introduced as a cleanup procedure for extracts rather than as a preparative procedure. Oils generally have a high concentration of semivolatile compounds and, therefore, preparation by Method 3650 should be done on a relatively small aliquot of the sample. Generally, extraction of 1 mL of oil will be sufficient to obtain a saturated aqueous phase and avoid emulsions.

NOTE: The use of traditional extraction techniques, i.e., 3510, 3520, 3535, 3540, 3541, 3545, 3546, and 3550, is neither suitable nor recommended for use in these matrices due to a high potential for hydrocarbon interferences and decreased determinative method sensitivity, i.e., poor analytical performance.

2.3.1.4 Sludge samples

Determining the appropriate methods for analysis of sludges is complicated because of the lack of precise definitions of sludges with respect to the relative percent of liquid and solid components. There is no set ratio of liquid to solid which enables the analyst to determine which of the three extraction methods cited is the most appropriate. Sludges may be classified into three categories: liquid sludges, solid sludges, and emulsions, but with appreciable overlap.

If the sample is an organic sludge (solid material and organic liquid, as opposed to an aqueous sludge), the sample should be handled as a multiphase sample.

2.3.1.4.1 Liquid sludges

Method 3510 or Method 3520 may be applicable to sludges that behave like, and have the consistency of, aqueous liquids. Ultrasonic extraction (Method 3550) and Soxhlet-type (Method 3540 series) procedures will, most likely, be ineffective because of the overwhelming presence of the liquid aqueous phase.

2.3.1.4.2 Solid sludges

Soxhlet extraction (Methods 3540 and 3541), pressurized fluid (Method 3545) extraction, microwave extraction (Method 3546) and ultrasonic extraction (Method 3550) will be more effective when applied to sludge samples that resemble solids. Samples may be dried or centrifuged to form solid materials for subsequent determination of semivolatile compounds.

Using Method 3650, Acid-Base Partition Cleanup, on the extract may be necessary, depending on whether chromatographic interferences prevent determination of the analytes of interest.

2.3.1.4.3 Emulsions

Attempts should be made to break up and separate the phases of an emulsion. Several techniques are effective in breaking emulsions or separating the phases of emulsions, including:

1. Freezing/thawing -- Certain emulsions will separate if exposed to temperatures below 0 °C.

2. Salting out -- Addition of a salt to make the aqueous phase of an emulsion too polar to support a less polar phase promotes separation.

3. Centrifugation -- Centrifugal force may separate emulsion components by density.

4. Addition of water or ethanol -- Emulsion polymers may be destabilized when a preponderance of the aqueous phase is added.

5. Forced filtering through glass wool -- Many emulsions can be broken by forcing the emulsion through a pad of Pyrex glass wool in a drying column using a slight amount of air pressure (using a rubber bulb usually provides sufficient pressure).

If techniques for breaking emulsions fail, use Method 3520. If the emulsion can be broken, the different phases (aqueous, solid, or organic liquid) may then be analyzed separately.

2.3.1.5 Multiphase samples

Choice of the procedure for separating multiphase samples is highly dependent on the objective of the analysis. With a sample in which some of the phases tend to separate rapidly, the percent weight or volume of each phase should be calculated and each phase should be individually analyzed for the required analytes.

An alternate approach is to obtain a homogeneous sample and attempt a single analysis on the combination of phases. This approach will give no information on the abundance of the analytes in the individual phases other than what can be implied by solubility.

A third alternative is to select phases of interest and to analyze only those selected phases. This tactic must be consistent with the sampling/analysis objectives or it will yield

insufficient information for the time and resources expended. The phases selected should be compared with Figure 2-1 and Table 2-41 for further guidance.

2.3.2 Cleanup procedures

Cleanup procedure selection is determined by the analytes of interest within the extract. Each analyte type in Table 2-42, Cleanup Methods for Organic Analyte Extracts, corresponds to one or more of the possible determinative methods available in the manual. However, the necessity of performing cleanup may also depend upon the matrix from which the extract was developed. Cleanup of a sample may be done exactly as instructed in the cleanup method for some of the analytes. There are some instances when cleanup using one of the methods may only proceed after the procedure is modified to optimize recovery and separation. Several cleanup techniques may be possible for each analyte category. The information provided is not meant to imply that any or all of these methods must be used for the analysis to be acceptable. Extracts with components which interfere with spectral or chromatographic determinations are expected to be subjected to cleanup procedures.

The analyst in consultation with the regulator, customer and other project planning participants, as necessary, must determine the necessity for cleanup procedures, as there are no clear cut criteria for indicating their use. Method 3600 and associated methods should be consulted for further details on extract cleanup.

2.3.3 Determinative procedures

In Table 2-43. the determinative methods for organic analytes are divided into four categories, specifically: gas chromatography/mass spectrometry (GC/MS); gas chromatography (GC) with electromagnetic spectrometric (ES) detectors, i.e., Fourier Transform infrared (FT-IR) or atomic emission (AES); specific quantitation methods, i.e., gas chromatography (GC) with specific non-MS detectors; and high performance liquid chromatography (HPLC). This division is intended to help an analyst choose which determinative method will apply. Under each analyte column, SW-846 method numbers are indicated, if appropriate, for the determination of the analyte. A blank has been left if no chromatographic determinative method is available.

Generally, the MS procedures are more specific but less sensitive than the appropriate gas chromatographic/specific quantitation or ES method.

Method 8000 gives a general description of the techniques of gas chromatography and high performance liquid chromatography. Method 8000 should be consulted prior to application of any of the gas chromatographic methods.

Method 8081 (organochlorine pesticides), Method 8082 (polychlorinated biphenyls), Method 8141 (organophosphorus pesticides), and Method 8151 (chlorinated herbicides), are preferred over GC/MS because of the combination of selectivity and sensitivity of the flame photometric, nitrogen-phosphorus, and electron capture detectors.

Method 8260 is a GC/MS method for volatile analytes, which employs a capillary column. A variety of sample introduction techniques may be used with Method 8260, including Methods 5021, 5030, 5031, 5035, 5041, and 3585. A GC with a selective detector is also useful for the determination of volatile organic compounds in a monitoring scenario, as described in Sec. 2.2.5.

Method 8270 is a GC/MS method for semivolatile analytes, which employs a capillary column. Method 8410 is another capillary GC method for semivolatile analytes which uses a

Fourier Transform IR (FT-IR) detector. Method 8085 is a capillary GC method for pesticides which uses an atomic emission detector (AES).

Table 2-43 lists several GC and HPLC methods that apply to only a small number of analytes. Methods 8031 and 8033 are GC methods for acrolein, acrylonitrile, and acetonitrile. Methods 8315 and 8316 are HPLC methods for these three analytes. Method 8316 also addresses acrylamide, which may be analyzed by Method 8032.

HPLC methods have been developed for other types of analytes, most notably N-methyl carbamates (Method 8318); azo dyes, phenoxy acid herbicides, carbamates, and organophosphorus pesticides (Method 8321); PAHs (Method 8310); explosives (Methods 8330, 8331, and 8332); and some volatile organics (Methods 8315 and 8316).

Method 8430 utilizes a fourier transform infrared spectrometer (FT-IR) coupled to a gas chromatograph to determine bis(2-chloroethyl) ether and its hydrolysis products. The sample is introduced by direct aqueous injection. Method 8440 may be employed for the determination of total recoverable petroleum hydrocarbons (TRPH) in solid samples by infrared (IR) spectrophotometry. The samples may be extracted with supercritical carbon dioxide, using Method 3560.

2.4 CHOOSING PROCEDURES FOR CHARACTERISTIC ANALYSES

2.4.1 Figure 2-2 outlines a sequence for determining if a waste exhibits one or more of the characteristics of a hazardous waste.

2.4.2 EP and TCLP extracts

The leachate obtained from using either the EP (Figure 2-3A) or the TCLP (Figure 2-3B) is an aqueous sample, and therefore, requires further solvent extraction prior to the analysis of semivolatile compounds.

The TCLP leachate is solvent extracted with methylene chloride at a pH <2 and at a pH >11 by either Method 3510 or 3520. The leachate may also be extracted as received for organochlorine pesticides and semivolatiles and at pH <1.0 for phenoxyacid herbicides using the solid phase extraction (SPE) disk option in Method 3535. The best recoveries are usually obtained using either Method 3520 or Method 3535.

The solvent extract obtained by performing either Method 3510 or 3520 at an acidic pH will contain the acid/neutral compounds of interest. Refer to the specific determinative method for guidance on the pH requirements for extraction prior to analysis. Method 5031 (azeotropic distillation) may be used as an effective preparative method for pyridine.

Due to the high concentration of acetate in the TCLP extract, it is recommended that purge-and-trap be used to introduce the volatile sample into the gas chromatograph.

The EP and TCLP extracts can also be digested using acids (Method 3010, 3015, or 3020) and analyzed for metals using a 6000 or 7000 series method (Figures 2-3A and 2-3B).

2.5 CHOOSING PROCEDURES FOR GROUNDWATER ANALYSES

Appropriate analysis schemes for the determination of analytes in groundwater are presented in Figures 2-4A, 2-4B, and 2-4C. Quantitation limits for the inorganic analytes should correspond to the drinking water limits, where such limits are available.

2.5.1 Special techniques for inorganic analytes

All atomic absorption analyses should employ appropriate background correction systems whenever spectral interferences could be present. Several background correction techniques are employed in modern atomic absorption spectrometers. Matrix modification can complement background correction in some cases. Since no approach to interference correction is completely effective in all cases, the analyst should attempt to verify the adequacy of correction. If the interferant is known (e.g., high concentrations of iron in the determination of selenium), accurate analyses of synthetic solutions of the interferant (with and without analyte) could establish the efficacy of the background correction. If the nature of the interferant is not established, good agreement of analytical results using two substantially different wavelengths could substantiate the adequacy of the background correction.

To reduce matrix interferences, all graphite furnace atomic absorption (GFAA) analyses should be performed using techniques which maximize an isothermal environment within the furnace cell. Data indicate that two such techniques, L'vov platform and the delayed atomization cuvette (DAC), are equivalent in this respect, and produce high quality results.

All furnace atomic absorption analysis should be carried out using the best matrix modifier for the analysis. Some examples of modifiers are listed below. (See also the appropriate methods.)

Element(s)	Modifier(s)
As and Se	Nickel nitrate, palladium
Pb	Phosphoric acid, ammonium phosphate, palladium
Cd	Ammonium phosphate, palladium
Sb	Ammonium nitrate, palladium
Tl	Platinum, palladium

ICP, AA, and GFAA calibration standards need to match the acid composition and strength of the acids contained in the samples. Acid strengths of the calibration standards should be stated in the raw data. When using a method which permits the use of internal standardization, and the internal standardization option is being used, matrix matching is not required.

2.6 CHOOSING PROCEDURES FOR INORGANIC ANALYSES

Methods for preparing different sample matrices for inorganic analyses are shown in Table 2-44. Guidance regarding the use of leaching and digestive methods for inorganic analysis is provided in Table 2-45.

2.7 REFERENCES

1. M. J. Barcelona, "TOC Determinations in Ground Water," Ground Water 1984, 22(1), 18-24.

2. R. Riggin, et al.; Development and Evaluation of Methods for Total Organic Halide and Purgeable Organic Halide in Wastewater; 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, 1984; EPA-600/4-84-008.

3. G. McKee, et al.; Determination of Inorganic Anions in Water by Ion Chromatography (Technical addition to Methods for Chemical Analysis of Water and Wastewater, EPA 600/4-79-020); U.S. Environmental Protection Agency; Environmental Monitoring and Support Laboratory; ORD Publication Offices of Center for Environmental Research Information; Cincinnati, OH, 1984; EPA-600/4-84-017.

TABLE 2-1

DETERMINATIVE METHODS FOR ORGANIC ANALYTES
Analytes are listed in alphabetical order and alternative analyte names are in parenthesis.
The applicable method listing does not include immunoassay or screening methods.

Analyte	Applicable Method
Abate (Temephos)	8085
Acenaphthene	8100, 8270, 8275, 8310, 8410
Acenaphthylene	8100, 8270, 8275, 8310, 8410
Acetaldehyde	8315
Acetone	8015, 8260, 8261, 8315
Acetonitrile	8015, 8033, 8260, 8261
Acetophenone	8261, 8270
2-Acetylaminofluorene	8270
1-Acetyl-2-thiourea	8270
Acifluorfen	8085, 8151
Acrolein (Propenal)	8015, 8260, 8261, 8315, 8316
Acrylamide	8032, 8316
Acrylonitrile	8015, 8031, 8260, 8261, 8316
Alachlor	8081,8085
Aldicarb (Temik)	8318, 8321
Aldicarb sulfone	8318, 8321
Aldicarb sulfoxide	8321
Aldrin	8081, 8085, 8270
Allyl alcohol	8015, 8260
Allyl chloride	8021, 8260, 8261
Ametryn	8085
2-Aminoanthraquinone	8270
Aminoazobenzene	8270
4-Aminobiphenyl	8270
Aminocarb	8321
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	8095, 8330
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	8095, 8330
3-Amino-9-ethylcarbazole	8270
t-Amyl alcohol (TAA)	8015
t-Amyl ethyl ether (TAEE, 4,4-Dimethyl-3-oxahexane)	8015, 8261
t-Amyl methyl ether (TAME)	8015, 8261
Anilazine	8270
Aniline	8131, 8261, 8270
o-Anisidine	8270
Anthracene	8100, 8270, 8275, 8310, 8410
Aramite	8270
Aroclor-1016 (PCB-1016)	8082, 8270
Aroclor-1221 (PCB-1221)	8082, 8270
Aroclor-1232 (PCB-1232)	8082, 8270
Aroclor-1242 (PCB-1242)	8082, 8270
Aroclor-1248 (PCB-1248)	8082, 8270
Aroclor-1254 (PCB-1254)	8082, 8270
Aroclor-1260 (PCB-1260)	8082, 8270
Aspon	8141
Asulam	8321

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Atraton	8085
Atrazine	8041, 8085, 8141
Azinphos-ethyl (Ethyl guthion)	8085, 8141
Azinphos-methyl (Guthion)	8085, 8141, 8270
Barban	8270, 8321
Baygon (Propoxur)	8318, 8321
Bendiocarb	8141, 8318, 8321
Benefin	8091
Benfluralin	8085
Benomyl	8321
Bentazon	8151
Benzal chloride	8121
Benzaldehyde	8315
Benz(a)anthracene	8100, 8270, 8275, 8310, 8410
Benzene	8015, 8021, 8260, 8261
Benzenethiol (Thiophenol)	8270
Benzidine	8270, 8325
Benzo(b)fluoranthene	8100, 8270, 8275, 8310
Benzo(j)fluoranthene	8100
Benzo(k)fluoranthene	8100, 8270, 8275, 8310
Benzoic acid	8270, 8410
Benzo(g,h,i)perylene	8100, 8270, 8275, 8310
Benzo(a)pyrene	8100, 8270, 8275, 8310, 8410
p-Benzoquinone	8270
Benzotrichloride	8121
Benzoylprop ethyl	8325
Benzyl alcohol	8270
Benzyl chloride	8021, 8121, 8260
α-BHC (α-Hexachlorocyclohexane)	8081, 8085, 8121, 8270
β-BHC (β-Hexachlorocyclohexane)	8081, 8085, 8121, 8270
δ-BHC (δ-Hexachlorocyclohexane)	8081, 8085, 8121, 8270
γ-BHC (Lindane, γ-Hexachlorocyclohexane)	8081, 8085, 8121, 8270
Bis(2-chloroethoxy)methane	8111, 8270, 8410
Bis(2-chloroethyl) ether	8111, 8270, 8410, 8430
Bis(2-chloroethyl)sulfide	8260
Bis(2-chloroisopropyl) ether	8021, 8111, 8270, 8410
Bis(2-n-butoxyethyl) phthalate	8061
Bis(2-ethoxyethyl) phthalate	8061
Bis(2-ethylhexyl) phthalate	8061, 8270, 8410
Bis(2-methoxyethyl) phthalate	8061
Bis(4-methyl-2-pentyl)-phthalate	8061
Bolstar (Sulprofos)	8085, 8141
Bromacil	8085, 8321
Brominal (Bromoxynil)	8085, 8270
Bromoacetone	8021, 8260
4-Bromoaniline	8131
Bromobenzene	8021, 8260
Bromochloromethane	8021, 8260, 8261

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
2-Bromo-6-chloro-4-nitroaniline	8131
Bromodichloromethane	8021, 8260, 8261
2-Bromo-4,6-dinitroaniline	8131
Bromoform	8021, 8260, 8261
Bromomethane	8021, 8260, 8261
4-Bromophenyl phenyl ether	8111, 8270, 8275, 8410
Bromoxynil (Brominal)	8085, 8270
Butachlor	8085
Butanal	8315
1-Butanol (*n*-Butyl alcohol, *n*-Butanol)	8260
n-Butanol (1-Butanol, *n*-Butyl alcohol)	8260
2-Butanone (Methyl ethyl ketone, MEK)	8015, 8260, 8261
Butifos (DEF)	8085
Butralin	8091
n-Butyl alcohol (1-Butanol, *n*-Butanol)	8260
t-Butyl alcohol	8015, 8260
Butylate	8085, 8141, 8321
n-Butylbenzene	8021, 8260, 8261
sec-Butylbenzene	8021, 8260, 8261
tert-Butylbenzene	8021, 8260, 8261
Butyl benzyl phthalate	8061, 8270, 8410
2-*sec*-Butyl-4,6-dinitrophenol (DNBP, Dinoseb)	8041, 8085, 8151, 8270, 8321
Captafol	8081, 8085, 8270
Captan	8085, 8270
Carbaryl (Sevin)	8270, 8318, 8321, 8325
Carbendazim	8321
Carbofuran (Furaden)	8270, 8318, 8321
Carbofuran phenol	8321
Carbon disulfide	8260, 8261
Carbon tetrachloride	8021, 8260, 8261, 8535
Carbophenothion	8081, 8085, 8141, 8270
Carbosulfan	8321
Carboxin	8085
Casoron (Dichlobenil)	8085
Chloral hydrate	8260
Chloramben	8151
Chlordane (NOS)	8081, 8270
cis-Chlordane	8081
trans-Chlordane	8085, 8081
Chlorfenvinphos	8141, 8270
Chloroacetonitrile	8260
2-Chloroaniline	8131
3-Chloroaniline	8131
4-Chloroaniline	8131, 8270, 8410
Chlorobenzene	8021, 8260, 8261
Chlorobenzilate	8081, 8270
2-Chlorobiphenyl	8082, 8275
2-Chloro-1,3-butadiene (Chloroprene)	8021, 8260

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
1-Chlorobutane	8260
Chlorodibromomethane (Dibromochloromethane)	8021, 8260, 8261
2-Chloro-4,6-dinitroaniline	8131
1-Chloro-2,4-dinitrobenzene	8091
1-Chloro-3,4-dinitrobenzene	8091
Chloroethane	8021, 8260, 8261
2-Chloroethanol	8021, 8260, 8430
2-(2-Chloroethoxy)ethanol	8430
2-Chloroethyl vinyl ether	8021, 8260
Chloroform	8021, 8260, 8261
1-Chlorohexane	8260
Chloromethane	8021, 8260, 8261
5-Chloro-2-methylaniline	8270
Chloromethyl methyl ether	8021
2-Chloro-5-methylphenol	8041
4-Chloro-2-methylphenol	8041
4-Chloro-3-methylphenol	8041, 8270, 8410
3-(Chloromethyl)pyridine hydrochloride	8270
1-Chloronaphthalene	8270, 8275
2-Chloronaphthalene	8121, 8270, 8410
Chloroneb	8081
2-Chloro-4-nitroaniline	8131
4-Chloro-2-nitroaniline	8131
1-Chloro-2-nitrobenzene	8091
1-Chloro-4-nitrobenzene	8091
2-Chloro-6-nitrotoluene	8091
4-Chloro-2-nitrotoluene	8091
4-Chloro-3-nitrotoluene	8091
2-Chlorophenol	8041, 8270, 8410
3-Chlorophenol	8041
4-Chlorophenol	8410
4-Chloro-1,2-phenylenediamine	8270
4-Chloro-1,3-phenylenediamine	8270
4-Chlorophenyl phenyl ether	8111, 8270, 8410
2-Chlorophenyl 4-nitrophenyl ether	8111
3-Chlorophenyl 4-nitrophenyl ether	8111
4-Chlorophenyl 4-nitrophenyl ether	8111
o-Chlorophenyl thiourea	8325
Chloroprene (2-Chloro-1,3-butadiene)	8021, 8260
3-Chloropropionitrile	8260
Chloropropham	8085, 8321
Chloropropylate	8081
Chlorothalonil	8081
2-Chlorotoluene	8021, 8260, 8261
4-Chlorotoluene	8021, 8260, 8261
Chloroxuron	8321
Chlorpyrifos	8085, 8141
Chlorpyrifos methyl	8141

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Chlorthalonil (Daconil)	8085
Chrysene	8100, 8270, 8275, 8310, 8410
Coumaphos	8085, 8141, 8270
p-Cresidine	8270
o-Cresol (2-Methylphenol)	8041, 8270, 8410
m-Cresol (3-Methylphenol)	8041, 8270
p-Cresol (4-Methylphenol)	8041, 8270, 8410
Crotonaldehyde	8015, 8260, 8315
Crotoxyphos	8141, 8270
m-Cumenyl methylcarbamate	8318, 8321
Cyanazine	8085
Cycloate	8085
Cyclohexanone	8315
2-Cyclohexyl-4,6-dinitrophenol	8041, 8270
2,4-D	8151, 8321
2,4-D (acid)	8085
2,4-D (butoxyethanol ester)	8321
2,4-D (ethylhexyl ester)	8321
Dacthal (DCPA)	8081, 8085
Daconil (Chlorthalonil)	8085
Dalapon	8151, 8321
2,4-DB	8151, 8321
2,4-DB (acid)	8085
DBCP (1,2-Dibromo-3-chloropropane)	8011, 8021, 8081, 8260, 8261, 8270
2,4-D, butoxyethanol ester	8321
DCM (Dichloromethane, Methylene chloride)	8021, 8260, 8261
DCPA (Dacthal)	8081, 8085
DCPA diacid	8151
2,4'-DDD	8085
4,4'-DDD	8081, 8085, 8270
2,4'-DDE	8085
4,4'-DDE	8081, 8085, 8270
2,4'-DDT	8085
4,4'-DDT	8081, 8085, 8270
DDVP (Dichlorvos, Dichlorovos)	8085, 8141, 8270, 8321
2,2',3,3'4,4'5,5',6,6'-Decachlorobiphenyl	8275
Decanal	8315
DEF (Butifos)	8085
Demeton-O, and Demeton-S	8085, 8141, 8270
2,4-D, ethylhexyl ester	8321
Diallate	8081, 8085, 8270
Diamyl phthalate	8061
2,4-Diaminotoluene	8270
Diazinon	8085, 8141
Dibenz(*a,h*)acridine	8100
Dibenz(*a,j*)acridine	8100, 8270
Dibenz(*a,h*)anthracene	8100, 8270, 8275, 8310
7H-Dibenzo(*c,g*)carbazole	8100

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Dibenzofuran	8270, 8275, 8410
Dibenzo(*a,e*)pyrene	8100, 8270
Dibenzo(*a,h*)pyrene	8100
Dibenzo(*a,i*)pyrene	8100
Dibenzothiophene	8275
Dibromochloromethane (Chlorodibromomethane)	8021, 8260, 8261
1,2-Dibromo-3-chloropropane (DBCP)	8011, 8021, 8081, 8260, 8261, 8270
Dibromofluoromethane	8260
Dibromomethane	8021, 8260, 8261
1,2-Dibromoethane (EDB, Ethylene dibromide)	8011, 8021, 8260
2,6-Dibromo-4-nitroaniline	8131
2,4-Dibromophenyl 4-nitrophenyl ether	8111
Di-*n*-butyl phthalate	8061, 8270, 8410
Dicamba	8085, 8151, 8321
Dichlobenil (Casoron)	8085
Dichlone	8081, 8270
Dichloran	8081
3,4-Dichloroaniline	8131
1,2-Dichlorobenzene	8021, 8121, 8260, 8261, 8270, 8410
1,3-Dichlorobenzene	8021, 8121, 8260, 8261, 8270, 8410
1,4-Dichlorobenzene	8021, 8121, 8260, 8261, 8270, 8410
3,3'-Dichlorobenzidine	8270,8325
3,5-Dichlorobenzoic acid	8085, 8151
2,3-Dichlorobiphenyl	8082
3,3'-Dichlorobiphenyl	8275
cis-1,4-Dichloro-2-butene	8260, 8261
trans-1,4-Dichloro-2-butene	8260, 8261
Dichlorodifluoromethane	8021, 8260, 8261
1,1-Dichloroethane	8021, 8260, 8261
1,2-Dichloroethane	8021, 8260, 8261
1,1-Dichloroethene (Vinylidene chloride)	8021, 8260, 8261
cis-1,2-Dichloroethene	8021, 8260, 8261
trans-1,2-Dichloroethene	8021, 8260, 8261
Dichlorofenthion	8141
Dichloromethane (DCM, Methylene chloride)	8021, 8260, 8261
2,6-Dichloro-4-nitroaniline	8131
2,3-Dichloronitrobenzene	8091
2,4-Dichloronitrobenzene	8091
3,5-Dichloronitrobenzene	8091
3,4-Dichloronitrobenzene	8091
2,5-Dichloronitrobenzene	8091
2,3-Dichlorophenol	8041
2,4-Dichlorophenol	8041, 8270, 8410
2,5-Dichlorophenol	8041
2,6-Dichlorophenol	8041, 8270
3,4-Dichlorophenol	8041
3,5-Dichlorophenol	8041
2,4-Dichlorophenol 3-methyl-4-nitrophenyl ether	8111

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
2,3-Dichlorophenyl 4-nitrophenyl ether	8111
2,4-Dichlorophenyl 4-nitrophenyl ether	8111
2,5-Dichlorophenyl 4-nitrophenyl ether	8111
2,6-Dichlorophenyl 4-nitrophenyl ether	8111
3,4-Dichlorophenyl 4-nitrophenyl ether	8111
3,5-Dichlorophenyl 4-nitrophenyl ether	8111
Dichloroprop (Dichlorprop)	8085, 8151, 8321
1,2-Dichloropropane	8021, 8260, 8261
1,3-Dichloropropane	8021, 8260, 8261
2,2-Dichloropropane	8021, 8260, 8261
1,3-Dichloro-2-propanol	8021, 8260
1,1-Dichloropropene	8021, 8260, 8261
cis-1,3-Dichloropropene	8021, 8260, 8261
trans-1,3-Dichloropropene	8021, 8260, 8261
Dichlorovos (DDVP, Dichlorvos)	8085, 8141, 8270, 8321
Dichlorprop (Dichloroprop)	8085, 8151, 8321
Dichlorvos (DDVP, Dichlorovos)	8085, 8141, 8270, 8321
Dicrotophos	8141, 8270
Diclofol (Kelthane)	8085
Diclofop-methyl	8085
Dicofol	8081
Dicyclohexyl phthalate	8061
Dieldrin	8081, 8085, 8270
1,2,3,4-Diepoxybutane	8260
Diesel range organics (DRO)	8015
Diethylene glycol	8430
Diethyl ether	8015, 8260, 8261
Diethyl phthalate	8061, 8270, 8410
Diethylstilbestrol	8270
Diethyl sulfate	8270
Dihexyl phthalate	8061
Diisobutyl phthalate	8061
Diisopropyl ether (DIPE)	8015, 8261
Dimethoate	8141, 8270,8085, 8321
3,3'-Dimethoxybenzidine	8270, 8325
Dimethylaminoazobenzene	8270
2,5-Dimethylbenzaldehyde	8315
7,12-Dimethylbenz(a)anthracene	8270
3,3'-Dimethylbenzidine	8270, 8325
4,4-Dimethyl-3-oxahexane (*t*-Amyl ethyl ether, TAEE)	8015, 8261
α,α-Dimethylphenethylamine	8270
2,3-Dimethylphenol	8041
2,4-Dimethylphenol	8041, 8270
2,5-Dimethylphenol	8041
2,6-Dimethylphenol	8041
3,4-Dimethylphenol	8041
Dimethyl phthalate	8061, 8270, 8410
Dinitramine	8091

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
2,4-Dinitroaniline	8131
3,5-Dinitroaniline	8095
1,2-Dinitrobenzene	8091, 8270
1,3-Dinitrobenzene (1,3-DNB)	8091, 8095, 8270, 8330
1,4-Dinitrobenzene	8091, 8270
4,6-Dinitro-2-methylphenol	8270, 8410
2,4-Dinitrophenol	8041, 8270, 8410
2,5-Dinitrophenol	8041
2,4-Dinitrotoluene (2,4-DNT)	8091, 8095, 8270, 8330, 8410
2,6-Dinitrotoluene (2,6-DNT)	8091, 8095, 8270, 8330, 8410
Dinocap	8270
Dinonyl phthalate	8061
Dinoseb (2-*sec*-Butyl-4,6-dinitrophenol, DNBP)	8041, 8085, 8151, 8270, 8321
Di-*n*-octyl phthalate	8061, 8270, 8410
Dioxacarb	8318
1,4-Dioxane	8260, 8261
Dioxathion	8085, 8141
Di-*n*-propyl phthalate	8410
DIPE (Diisopropyl ether)	8015, 8261
Diphenamid	8085
Diphenylamine	8270
5,5-Diphenylhydantoin	8270
1,2-Diphenylhydrazine	8270
Disperse Blue 3	8321
Disperse Blue 14	8321
Disperse Brown 1	8321
Disperse Orange 3	8321
Disperse Orange 30	8321
Disperse Red 1	8321
Disperse Red 5	8321
Disperse Red 13	8321
Disperse Red 60	8321
Disperse Yellow 5	8321
Disulfoton	8085, 8141, 8270, 8321
Diuron	8085, 8321, 8325
1,3-DNB (1,3-Dinitrobenzene)	8091, 8095, 8270, 8330
DNBP (2-*sec*-Butyl-4,6-dinitrophenol, Dinoseb)	8041, 8085, 8151, 8270, 8321
2,4-DNT (2,4-Dinitrotoluene)	8091, 8095, 8270, 8330, 8410
2,6-DNT (2,6-Dinitrotoluene)	8091, 8270, 8330, 8410
EDB (1,2-Dibromoethane, Ethylene dibromide)	8011, 8021, 8260
Endosulfan I	8081, 8085, 8270
Endosulfan II	8081, 8085, 8270
Endosulfan sulfate	8081, 8085, 8270
Endrin	8081, 8085, 8270
Endrin aldehyde	8081, 8085, 8270
Endrin ketone	8081, 8085, 8270
Epichlorohydrin	8021, 8260
EPN	8141, 8085, 8270

Analyte	Applicable Method(s)
Eptam (EPTC)	8085, 8141, 8321
EPTC (Eptam)	8085, 8141, 8321
ETBE (Ethyl *tert*-butyl ether)	8015, 8261
Ethalfluralin (Sonalan)	8085
Ethanol	8015, 8260, 8261
Ethion	8085, 8141, 8270
Ethoprop	8085, 8141
Ethyl acetate	8015, 8260, 8261
Ethyl benzene	8015, 8021, 8260, 8261
Ethyl carbamate	8270
Ethyl cyanide (Propionitrile)	8015, 8260, 8261
Ethylene dibromide (EDB, 1,2-Dibromoethane)	8011, 8021, 8260
Ethylene glycol	8430
Ethyl guthion (Azinphos-ethyl)	8085, 8141
Ethylene oxide	8015, 8260
Ethyl methacrylate	8260, 8261
Ethyl methanesulfonate	8270
Ethyl *tert*-butyl ether (ETBE)	8015, 8261
Etridiazole	8081
Famphur	8141, 8270, 8321
Fenamiphos	8085
Fenarimol	8085
Fenitrothion	8085, 8141
Fensulfothion	8085, 8141, 8270, 8321
Fenthion	8085, 8141, 8270
Fenuron	8321
Fluchloralin	8270
Fluometuron	8321
Fluoranthene	8100, 8270, 8275, 8310, 8410
Fluorene	8100, 8270, 8275, 8310, 8410
Fluridone	8085
Fonophos	8085, 8141
Formaldehyde	8315
Formetanate hydrochloride	8318, 8321
Furaden (Carbofuran)	8270, 8318, 8321
Gardona (Tetrachlovinphos, Stirophos)	80858141, 8270
Garlon (Triclopyr)	8085
Gasoline range organics (GRO)	8015
Guthion (Azinphos-methyl)	8085, 8141, 8270
Halowax-1000	8081
Halowax-1001	8081
Halowax-1013	8081
Halowax-1014	8081
Halowax-1051	8081
Halowax-1099	8081
Heptachlor	8081, 8085, 8270
2,2',3,3',4,4',5-Heptachlorobiphenyl	8082, 8275
2,2',3,4,4',5,5'-Heptachlorobiphenyl	8082, 8275

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
2,2',3,4,4',5',6-Heptachlorobiphenyl	8082
2,2',3,4',5,5',6-Heptachlorobiphenyl	8082, 8275
Heptachlor epoxide	8081, 8085, 8270
Heptanal	8315
Hexachlorobenzene	8081, 8085, 8121, 8270, 8275, 8410
2,2',3,3,4,4'-Hexachlorobiphenyl	8275
2,2',3,4,4',5'-Hexachlorobiphenyl	8082, 8275
2,2',3,4,5,5'-Hexachlorobiphenyl	8082
2,2',3,5,5',6-Hexachlorobiphenyl	8082
2,2',4,4',5,5'-Hexachlorobiphenyl	8082
Hexachlorobutadiene (1,3-Hexachlorobutadiene)	8021, 8121, 8260, 8261, 8270, 8410
α-Hexachlorocyclohexane (α-BHC)	8081, 8085, 8121, 8270
β-Hexachlorocyclohexane (β-BHC)	8081, 8085, 8121, 8270
δ-Hexachlorocyclohexane (δ-BHC)	8081, 8085, 8121, 8270
γ-Hexachlorocyclohexane (γ-BHC, Lindane)	8081, 8085, 8121, 8270
Hexachlorocyclopentadiene	8081, 8085, 8121, 8270, 8410
Hexachloroethane	8121, 8260, 8270, 8410
Hexachlorophene	8270
Hexachloropropene	8141, 8270
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	8095, 8330, 8510
Hexamethyl phosphoramide (HMPA)	, 8270
Hexanal	8315
2-Hexanone	8260, 8261
Hexazinone	8085
Hexyl 2-ethylhexyl phthalate	8061
HMPA (Hexamethyl phosphoramide)	8141, 8270
HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	8095, 8330
1,2,3,4,6,7,8-HpCDD	8280, 8290
HpCDD, total	8280, 8290
1,2,3,4,6,7,8-HpCDF	8280, 8290
1,2,3,4,7,8,9-HpCDF	8280, 8290
HpCDF, total	8280, 8290
1,2,3,4,7,8-HxCDD	8280, 8290
1,2,3,6,7,8-HxCDD	8280, 8290
1,2,3,7,8,9-HxCDD	8280, 8290
HxCDD, total	8280, 8290
1,2,3,4,7,8-HxCDF	8280, 8290
1,2,3,6,7,8-HxCDF	8280, 8290
1,2,3,7,8,9-HxCDF	8280, 8290
2,3,4,6,7,8-HxCDF	8280, 8290
HxCDF	8280, 8290
Hydroquinone	8270
3-Hydroxycarbofuran	8318, 8321
5-Hydroxydicamba	8151
2-Hydroxypropionitrile	8260
Igran (Terbutryn)	8085
Imidan (Phosmet)	8085, 8141, 8270
Indeno(1,2,3-cd)pyrene	8100, 8270, 8275, 8310

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Iodomethane (Methyl iodide)	8260, 8261
Ioxynil	8085
Isobutyl alcohol (2-Methyl-1-propanol)	8260, 8261
Isodrin	8081, 8270
Isophorone	8270, 8410
Isopropalin	8091
Isopropyl alcohol (2-Propanol)	8015, 8260
Isopropylbenzene	8021, 8260
p-Isopropyltoluene	8021, 8260, 8261
Isosafrole	8270
Isovaleraldehyde	8315
Kelthane (Diclofol)	8085
Kepone	8270
Kerb (Pronamide)	8085, 8270
Lannate (Methomyl)	8318, 8321
Leptophos	8141, 8270
Lindane (γ-Hexachlorocyclohexane, γ-BHC)	8081, 8085, 8121, 8270
Linuron (Lorox)	8321, 8325
Lorox (Linuron)	8321, 8325
Malathion	8085, 8141, 8270
Maleic anhydride	8270
Malononitrile	8260
MCPA	8151, 8321
MCPA (acid)	8085
MCPP	8151, 8321
MCPP (acid)	8085
MEK (Methyl ethyl ketone, 2-Butanone)	8015, 8260, 8261
Merphos	8085, 8141, 8321
Mestranol	8270
Mesurol (Methiocarb)	8141, 8318, 8321
Methacrylonitrile	8260, 8261
Metalaxyl	8085
Methanol	8015, 8260
Methapyrilene	8270
Methiocarb (Mesurol)	8141, 8318, 8321
Methomyl (Lannate)	8318, 8321
Methoxychlor	8081, 8085, 8270
Methyl acrylate	8260
Methyl chlorpyrifos	8085
Methyl-*tert*-butyl ether (MTBE)	8015, 8260, 8261
3-Methylcholanthrene	8100, 8270
2-Methyl-4,6-dinitrophenol	8041
4,4'-Methylenebis(2-chloroaniline)	8270
4,4'-Methylenebis(*N,N*-dimethylaniline)	8270
Methyl ethyl ketone (MEK, 2-Butanone)	8015, 8260, 8261
Methylene chloride (Dichloromethane, DCM)	8021, 8260, 8261
Methyl iodide (Iodomethane)	8260, 8261
Methyl isobutyl ketone (MIBK, 4-Methyl-2-pentanone)	8260, 8261

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Methyl methacrylate	8260, 8261
Methyl methanesulfonate	8270
1-Methylnaphthalene	8261
2-Methylnaphthalene	8261, 8270, 8410
Methyl paraoxon	8085
Methyl parathion (Parathion, methyl)	8085, 8270, 8141, 8321
4-Methyl-2-pentanone (MIBK, Methyl isobutyl ketone)	8260, 8261
2-Methylphenol (*o*-Cresol)	8041, 8270, 8410
3-Methylphenol (*m*-Cresol)	8041, 8270
4-Methylphenol (*p*-Cresol)	8041, 8270, 8410
2-Methyl-1-propanol (Isobutyl alcohol)	8260, 8261
2-Methyl-2-propanol (*t*-Butyl alcohol)	8015, 8260
2-Methylpyridine (2-Picoline)	8015, 8260, 82618270
Methyl-2,4,6-trinitrophenyl-nitramine (Tetryl)	8330
Metolachlor	8085
Metolcarb	8318, 8321
Metribuzin	8085
Mevinphos	8085, 8141, 8270
Mexacarbate	8270, 8318, 8321
MGK-264	8085
MIBK (Methyl isobutyl ketone, 4-Methyl-2-pentanone)	8260, 8261
Mirex	8081, 8085, 8270
Molinate	8085, 8141, 8321
Monocrotophos	8141, 8270, 8321
Monuron	8321, 8325
MTBE (Methyl-*tert*-butyl ether)	8015, 8260, 8261
Naled	8141, 8270, 8321
Naphthalene	8021, 8100, 8260, 8261, 8270, 8275, 8310, 8410
Napropamide	8085
NB (Nitrobenzene)	8091, 8095, 8260, 8270, 8330, 8410
1,2-Naphthoquinone	8091
1,4-Naphthoquinone	8270, 8091
1-Naphthylamine	8270
2-Naphthylamine	8270
Neburon	8321
Nicotine	8270
5-Nitroacenaphthene	8270
2-Nitroaniline	8131, 8270, 8410
3-Nitroaniline	8131, 8270, 8410
4-Nitroaniline	8131, 8270, 8410
5-Nitro-*o*-anisidine	8270
Nitrobenzene (NB)	8091, 8095, 8260, 8270, 8330, 8410
4-Nitrobiphenyl	8270
Nitrofen	8081, 8270
Nitroglycerin	8095, 8332
2-Nitrophenol	8041, 8270, 8410
3-Nitrophenol	8041
4-Nitrophenol	8041, 8085, 8151, 8270, 8410

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
4-Nitrophenyl phenyl ether	8111
2-Nitropropane	8260
Nitroquinoline-1-oxide	8270
N-Nitroso-di-*n*-butylamine (*N*-Nitrosodibutylamine)	8015, 8260, 8261, 8270
N-Nitrosodiethylamine	8261, 8270
N-Nitrosodimethylamine	8070, 8261, 8270, 8410
N-Nitrosodiphenylamine	8070, 8270, 8410
N-Nitroso-di-*n*-propylamine	8070, 8261, 8270, 8410
N-Nitrosomethylethylamine	8261, 8270
N-Nitrosomorpholine	8270
N-Nitrosopiperidine	8270
N-Nitrosopyrrolidine	8270
2-Nitrotoluene (*o*-Nitrotoluene, 2-NT)	8091, 8095, 8330
3-Nitrotoluene (*m*-Nitrotoluene, 3-NT)	8091, 8095, 8330
4-Nitrotoluene (*p*-Nitrotoluene, 4-NT)	8091, 8095, 8330
o-Nitrotoluene (2-Nitrotoluene, 2-NT)	8091, 8095, 8330
m-Nitrotoluene (3-Nitrotoluene, 3-NT)	8091, 8095, 8330
p-Nitrotoluene (4-Nitrotoluene, 4-NT)	8091, 8095, 8330
5-Nitro-*o*-toluidine	8270
trans-Nonachlor	8081
2,2'3,3'4,4'5,5'6-Nonachlorobiphenyl	8082, 8275
Nonanal	8315
Norflurazon	8085
2-NT (2-Nitrotoluene, *o*-Nitrotoluene)	8091, 8095, 8330
3-NT (3-Nitrotoluene, *m*-Nitrotoluene)	8091, 8095, 8330
4-NT (4-Nitrotoluene, *p*-Nitrotoluene)	8091, 8095, 8330
OCDD	8280, 8290
OCDF	8280, 8290
2,2',3,3',4,4'5,5'-Octachlorobiphenyl	8275
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	8095, 8330
Octamethyl pyrophosphoramide	8270
Octanal	8315
Oxamyl	8318, 8321
4,4'-Oxydianiline	8270
Oxyfluorfen	8085
Paraldehyde	8015, 8260
Parathion	8085, 8270
Parathion, ethyl	8141
Parathion, methyl	8085, 8270, 8141, 8321
PCB-1016 (Aroclor-1016)	8082, 8270
PCB-1221 (Aroclor-1221)	8082, 8270
PCB-1232 (Aroclor-1232)	8082, 8270
PCB-1242 (Aroclor-1242)	8082, 8270
PCB-1248 (Aroclor-1248)	8082, 8270
PCB-1254 (Aroclor-1254)	8082, 8270
PCB-1260 (Aroclor-1260)	8082, 8270
PCBs, as congeners	8082
PCNB (Pentachloronitrobenzene)	8081, 8091, 8270

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Pebulate	8085, 8141, 8321
1,2,3,7,8-PeCDD	8280, 8290
PeCDD, total	8280, 8290
1,2,3,7,8-PeCDF	8280, 8290
2,3,4,7,8-PeCDF	8280, 8290
PeCDF, total	8280, 8290
Pendimethaline (Penoxalin)	8085, 8091
Penoxalin (Pendimethaline)	8085, 8091
Pentachlorobenzene	8121, 8270
2,2',3,4,5'-Pentachlorobiphenyl	8082
2,3',4,4',5-Pentachlorobiphenyl	8275
2,2',4,5,5'-Pentachlorobiphenyl	8082, 8275
2,3,3',4',6-Pentachlorobiphenyl	8082
Pentachloroethane	8260, 8261
Pentachloronitrobenzene (PCNB)	8081, 8091, 8270
Pentachlorophenol	8041, 8085, 8151, 8270, 8410
Pentaerythritoltetranitrate	8095
Pentafluorobenzene	8260
Pentanal (Valeraldehyde)	8315
2-Pentanone	8015, 8260
Perchloroethylene (Tetrachloroethene, Tetrachloroethylene)	8021, 8260, 8261
Permethrin (*cis* + *trans*)	8081
Perthane	8081
Phenacetin	8270
Phenanthrene	8100, 8270, 8275, 8310, 8410
Phenobarbital	8270
Phenol	8041, 8270, 8410
1,4-Phenylenediamine	8270
1,2-Phenylenediamine (*o*-Phenylenediamine)	8141, 8321
Phorate	8085, 8141, 8270, 8321
Phosalone	8270
Phosmet (Imidan)	8085, 8141, 8270
Phosphamidon	8085, 8141, 8270
Phthalic anhydride	8270
Physostigmine	8321
Physostigmine salicylate	8321
Picloram	8085, 8151
2-Picoline (2-Methylpyridine)	8015, 8260, 8261, 8270
Piperonyl sulfoxide	8270
Polychlorinated biphenyls (PCBs), as Aroclors or congeners	8082, 8270
Profluralin	8085, 8091
Pramitol 5p (Prometon)	8085
Promecarb	8318, 8321
Prometon (Pramitol 5p)	8085
Prometryn	8085
Pronamide (Kerb)	8085, 8270
Propachlor (Ramrod)	8081, 8085
Propanal (Propionaldehyde)	8315

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
1-Propanol (*n*-Propyl alcohol)	8015, 8260
2-Propanol (Isopropyl alcohol)	8015, 8260
Propargite (S-181)	8085
Propargyl alcohol	8260
Propazine	8085
Propenal (Acrolein)	8015, 8260, 8261, 8315, 8316
Propetamidophos	8085
Propham	8141, 8321
ß-Propiolactone	8260
Propionaldehyde (Propanal)	8315
Propionitrile (Ethyl cyanide)	8015, 8260, 8261
Propoxur (Baygon)	8318, 8321
n-Propylalcohol (1-Propanol)	8015, 8260
n-Propylamine	8260
n-Propylbenzene	8021, 8260, 8261
Propylthiouracil	8270
Prosulfocarb	8141, 8321
Prothiophos (Tokuthion)	8141
Pyrene	8100, 8270, 8275, 8310, 8410
Pyridine	8015, 8260, 8261
Ramrod (Propachlor)	8085
RDX (Hexahydro-1,3,5-trinitro-1,3,5-triazine)	8095, 8330
Resorcinol	8270
Ronnel	8085, 8141
Rotenone	8325
S-181 (Propargite)	8085
Safrole	8270
Sevin (Carbaryl)	8270, 8318, 8321, 8325
Siduron	8321, 8325
Simazine	8085, 8141
Silvex (2,4,5-TP)	8085, 8151, 8321
Solvent Red 3	8321
Solvent Red 23	8321
Sonalan (Ethalfluralin)	8085
Stirophos (Tetrachlorvinphos, Gardona)	8085, 8141, 8270
Strobane	8081
Strychnine	8270
Styrene	8021, 8260, 8261
Sulfallate	8270
Sulfotepp	8085, 8141
Sulprofos (Bolstar)	8085, 8141
2,4,5-T	8151, 8321
2,4,5-T (acid)	8085
2,4,5-TB	8085
TAA (t-Amyl alcohol)	8015
TAEE (t-Amyl ethyl ether, 4,4-Dimethyl-3-oxahexane)	8015, 8261
TAME (t-Amyl methyl ether)	8015, 8261
2,4,5-T, butoxyethanol ester	8321

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
2,4,5-T, butyl ester	8321
2,3,7,8-TCDD	8280, 8290
TCDD, total	8280, 8290
2,3,7,8-TCDF	8280, 8290
TCDF, total	8280, 8290
Tebuthiuron	8085, 8321
Temephos (Abate)	8085
Temik (Aldicarb)	8318, 8321
TEPP (Tetraethyl pyrophosphate)	8141, 8270
Terbacil	8085
Terbufos	8141, 8270
Terbutryn (Igran)	8085
1,2,3,4-Tetrachlorobenzene	8121
1,2,3,5-Tetrachlorobenzene	8121
1,2,4,5-Tetrachlorobenzene	8121, 8270
2,2',3,5'-Tetrachlorobiphenyl	8082, 8275
2,2',4,5'-Tetrachlorobiphenyl	8275
2,2',5,5'-Tetrachlorobiphenyl	8082, 8275
2,3',4,4'-Tetrachlorobiphenyl	8082, 8275
1,1,1,2-Tetrachloroethane	8021, 8260
1,1,2,2-Tetrachloroethane	8021, 8260, 8261
Tetrachloroethene (Perchloroethylene, Tetrachloroethylene)	8021, 8260, 8261
2,3,4,5-Tetrachloronitrobenzene	8091
2,3,5,6-Tetrachloronitrobenzene	8091
2,3,4,5-Tetrachlorophenol	8041, 8085
2,3,4,6-Tetrachlorophenol	8041, 8085, 8270
2,3,5,6-Tetrachlorophenol	8041
Tetrachlorvinphos (Stirophos, Gardona)	8085, 8141, 8270
Tetraethyl dithiopyrophosphate	8270
Tetraethyl pyrophosphate (TEPP)	8141, 8270
Tetrahydrofuran (THF)	8261
THF (Tetrahydrofuran)	8261
Tetrazene	8331
Tetryl (Methyl-2,4,6-trinitrophenylnitramine)	8330
Thiodicarb	8318, 8321
Thiofanox	8321
Thiophanate-methyl	8321
Thionazin (Zinophos)	8141, 8270
Thiophenol (Benzenethiol)	8270
1,3,5-TNB (1,3,5-Trinitrobenzene)	8095, 8270, 8330
2,4,6-TNT (2,4,6-Trinitrotoluene)	8095, 8330
TOCP (Tri-*o*-cresylphosphate)	8141
Tokuthion (Prothiofos)	8141
m-Tolualdehyde	8315
o-Tolualdehyde	8315
p-Tolualdehyde	8315
Toluene	8015, 8021, 8260, 8261
Toluene diisocyanate	8270

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
o-Toluidine	8015, 8260, 8261, 8270
Toxaphene	8081, 8270
2,4,5-TP (Silvex)	8085, 8151, 8321
Treflan (Trifluralin)	8081, 8085, 8091, 8270
Triademefon	8085
Triallate	8085, 8141, 8321
Triclopyr (Garlon)	8085
Trichlorfon	8141, 8321
2,4,6-Trichloroaniline	8131
2,4,5-Trichloroaniline	8131
1,2,3-Trichlorobenzene	8021, 8121, 8260, 8261
1,2,4-Trichlorobenzene	8021, 8121, 8260, 8261, 8270, 8275, 8410
1,3,5-Trichlorobenzene	8121
2,2',5-Trichlorobiphenyl	8082, 8275
2,3',5-Trichlorobiphenyl	8275
2,4',5-Trichlorobiphenyl	8082, 8275
1,1,1-Trichloroethane	8021, 8260, 8261
1,1,2-Trichloroethane	8021, 8260, 8261
Trichloroethene (Trichloroethylene)	8021, 8260, 8261, 8535
Trichlorofluoromethane	8021, 8260, 8261
Trichloronate	8141
1,2,3-Trichloro-4-nitrobenzene	8091
1,2,4-Trichloro-5-nitrobenzene	8091
2,4,6-Trichloronitrobenzene	8091
2,3,4-Trichlorophenol	8041
2,3,5-Trichlorophenol	8041
2,3,6-Trichlorophenol	8041
2,4,5-Trichlorophenol	8041, 8085, 8270, 8410
2,4,6-Trichlorophenol	8041, 8085, 8270, 8410
2,3,4-Trichlorophenyl 4-nitrophenyl ether	8111
2,3,5-Trichlorophenyl 4-nitrophenyl ether	8111
2,3,6-Trichlorophenyl 4-nitrophenyl ether	8111
2,4,5-Trichlorophenyl 4-nitrophenyl ether	8111
2,4,6-Trichlorophenyl 4-nitrophenyl ether	8111
3,4,5-Trichlorophenyl 4-nitrophenyl ether	8111
1,2,3-Trichloropropane	8021, 8260, 8261
Tri-*o*-cresylphosphate (TOCP)	8141
Triethylamine	8015
O,O,O-Triethyl phosphorothioate	8270
Trifluralin (Treflan)	8081, 8085, 8091, 8270
Trihalomethanes	8535
2,4,5-Trimethylaniline	8270
1,2,4-Trimethylbenzene	8021, 8260, 8261
1,3,5-Trimethylbenzene	8021, 8260, 8261
Trimethyl phosphate	8270
1,3,5-Trinitrobenzene (1,3,5-TNB)	8095, 8270, 8330
2,4,6-Trinitrophenylmethylnitramine	8095
2,4,6-Trinitrotoluene (2,4,6-TNT)	8095, 8330

TABLE 2-1
(continued)

Analyte	Applicable Method(s)
Tris-BP (Tris(2,3-dibromopropyl) phosphate)	8270, 8321
Tri-*p*-tolyl phosphate	8270
Tris(2,3-dibromopropyl) phosphate (Tris-BP)	8270, 8321
Valeraldehyde (Pentanal)	8315
Vernolate	8085
Vinyl acetate	8260
Vinyl chloride	8021, 8260, 8261
Vinylidene chloride (1,1-Dichloroethene)	8021, 8260, 8261
m-Xylene	8015, 8021, 8260, 8261
o-Xylene	8015, 8021, 8260, 8261
p-Xylene	8015, 8021, 8260, 8261
Zinophos (Thionazin)	8141, 8270

TABLE 2-2

METHOD 8011 (MICROEXTRACTION AND GAS CHROMATOGRAPHY)

1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)

TABLE 2-3

METHOD 8015 (GC/FID) - NONHALOGENATED VOLATILES

Acetone	Ethyl *tert*-butyl ether (ETBE)
Acetonitrile	Gasoline range organics (GRO)
Acrolein	Isopropyl alcohol
Acrylonitrile	Methanol
Allyl alcohol	Methyl ethyl ketone (MEK, 2-Butanone)
t-Amyl alcohol (TAA)	*N*-Nitroso-di-*n*-butylamine
t-Amyl ethyl ether (TAEE)	Paraldehyde
t-Amyl methyl ether (TAME)	2-Pentanone
Benzene	2-Picoline
t-Butyl alcohol	1-Propanol (*n*-Propyl alcohol)
Crotonaldehyde	Propionitrile
Diesel range organics (DRO)	Pyridine
Diethyl ether	Toluene
Diisopropyl ether (DIPE)	*o*-Toluidine
Ethanol	*o*-Xylene
Ethyl acetate	*m*-Xylene
Ethyl benzene	*p*-Xylene
Ethylene oxide	Triethylamine

TABLE 2-4

METHOD 8021 (GC, PHOTOIONIZATION AND ELECTROLYTIC CONDUCTIVITY DETECTORS) - AROMATIC AND HALOGENATED VOLATILES

Allyl chloride	*cis*-1,2-Dichloroethene
Benzene	*trans*-1,2-Dichloroethene
Benzyl chloride	1,2-Dichloropropane
Bis(2-chloroisopropyl) ether	1,3-Dichloropropane
Bromoacetone	2,2-Dichloropropane
Bromobenzene	1,3-Dichloro-2-propanol
Bromochloromethane	1,1-Dichloropropene
Bromodichloromethane	*cis*-1,3-Dichloropropene
Bromoform	*trans*-1,3-Dichloropropene
Bromomethane	Epichlorhydrin
n-Butylbenzene	Ethylbenzene
sec-Butylbenzene	Hexachlorobutadiene
tert-Butylbenzene	Isopropylbenzene
Carbon tetrachloride	*p*-Isopropyltoluene
Chlorobenzene	Methylene chloride
Chlorodibromomethane	Naphthalene
Chloroethane	*n*-Propylbenzene
2-Chloroethanol	Styrene
2-Chloroethyl vinyl ether	1,1,1,2-Tetrachloroethane
Chloroform	1,1,2,2-Tetrachloroethane
Chloromethyl methyl ether	Tetrachloroethene
Chloroprene	Toluene
Chloromethane	1,2,3-Trichlorobenzene
2-Chlorotoluene	1,2,4-Trichlorobenzene
4-Chlorotoluene	1,1,1-Trichloroethane
1,2-Dibromo-3-chloropropane	1,1,2-Trichloroethane
1,2-Dibromoethane	Trichloroethene
Dibromomethane	Trichlorofluoromethane
1,2-Dichlorobenzene	1,2,3-Trichloropropane
1,3-Dichlorobenzene	1,2,4-Trimethylbenzene
1,4-Dichlorobenzene	1,3,5-Trimethylbenzene
Dichlorodifluoromethane	Vinyl chloride
1,1-Dichloroethane	*o*-Xylene
1,2-Dichloroethane	*m*-Xylene
1,1-Dichloroethene	*p*-Xylene

TABLE 2-5

METHODS 8031 AND 8033 (GC WITH NITROGEN-PHOSPHORUS DETECTION)
AND METHOD 8032 (GC WITH ELECTRON CAPTURE DETECTION)

Method 8031: Acrylonitrile
Method 8032: Acrylamide
Method 8033: Acetonitrile

TABLE 2-6

METHOD 8041 (GC) - PHENOLS

2-Chloro-5-methylphenol	2,5-Dinitrophenol
4-Chloro-2-methylphenol	Dinoseb (2-*sec*-butyl-4,6-dinitro phenol)
4-Chloro-3-methylphenol	2-Methyl-4,6-dinitrophenol
2-Chlorophenol	2-Methylphenol (*o*-Cresol)
3-Chlorophenol	4-Methylphenol (*p*-Cresol)
4-Chlorophenol	2-Nitrophenol
2-Cyclohexyl-4,6-dinitrophenol	3-Nitrophenol
2,3-Dichlorophenol	4-Nitrophenol
2,4-Dichlorophenol	Pentachlorophenol
2,5-Dichlorophenol	Phenol
2,6-Dichlorophenol	2,3,4,5-Tetrachlorophenol
3,4-Dichlorophenol	2,3,4,6-Tetrachlorophenol
3,5-Dichlorophenol	2,3,5,6-Tetrachlorophenol
2,3-Dimethylphenol	2,3,4-Trichlorophenol
2,4-Dimethylphenol	2,3,5-Trichlorophenol
2,5-Dimethylphenol	2,3,6-Trichlorophenol
2,6-Dimethylphenol	2,4,5-Trichlorophenol
3,4-Dimethylphenol	2,4,6-Trichlorophenol
2,4-Dinitrophenol	

TABLE 2-7

METHOD 8061 (GC/ECD) - PHTHALATE ESTERS

Benzyl benzoate	Dihexyl phthalate
Bis(2-*n*-butoxyethyl) phthalate	Diisobutyl phthalate
Bis(2-ethoxyethyl) phthalate	Di-*n*-butyl phthalate
Bis(2-ethylhexyl) phthalate	Diethyl phthalate
Bis(2-methoxyethyl) phthalate	Dinonyl phthalate
Bis(4-methyl-2-pentyl)-phthalate	Dimethyl phthalate
Butyl benzyl phthalate	Di-*n*-octyl phthalate
Diamyl phthalate	Hexyl 2-ethylhexyl phthalate
Dicyclohexyl phthalate	

TABLE 2-8

METHOD 8070 (GC) - NITROSAMINES

N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosodi-*n*-propylamine

TABLE 2-9

METHOD 8081 (GC) - ORGANOCHLORINE PESTICIDES

Alachlor	Diallate	Hexachlorobenzene
Aldrin	Dichlone	Hexachlorocyclopentadiene
α-BHC	Dichloran	Isodrin
β-BHC	Dicofol	Methoxychlor
δ-BHC	Dieldrin	Mirex
γ-BHC (Lindane)	Endosulfan I	Nitrofen
Captafol	Endosulfan II	*trans*-Nonachlor
Carbophenothion	Endosulfan sulfate	Pentachloronitrobenzene (PCNB)
cis-Chlordane	Endrin	Permethrin (*cis* + *trans*)
trans-Chlordane	Endrin aldehyde	Perthane
Chlordane (NOS)	Endrin ketone	Propachlor
Chlorobenzilate	Etridiazole	Strobane
Chloroneb	Halowax-1000	Toxaphene
Chloropropylate	Halowax-1001	Trifluralin
Chlorothalonil	Halowax-1013	
DBCP	Halowax-1014	
Dacthal (DCPA)	Halowax-1051	
4,4'-DDD	Halowax-1099	
4,4'-DDE	Heptachlor	
4,4'-DDT	Heptachlor epoxide	

TABLE 2-10

METHOD 8082 (GC) - POLYCHLORINATED BIPHENYLS

Aroclor 1016	2,3',4,4'-Tetrachlorobiphenyl
Aroclor 1221	2,2',3,4,5'-Pentachlorobiphenyl
Aroclor 1232	2,2',4,5,5'-Pentachlorobiphenyl
Aroclor 1242	2,3,3',4',6-Pentachlorobiphenyl
Aroclor 1248	2,2',3,4,4',5'-Hexachlorobiphenyl
Aroclor 1254	2,2',3,4,5,5'-Hexachlorobiphenyl
Aroclor 1260	2,2',3,5,5',6-Hexachlorobiphenyl
PCBs as congeners	2,2',4,4',5,5'-Hexachlorobiphenyl
2-Chlorobiphenyl	2,2',3,3',4,4',5-Heptachlorobiphenyl
2,3-Dichlorobiphenyl	2,2',3,4,4',5,5'-Heptachlorobiphenyl
2,2',5-Trichlorobiphenyl	2,2',3,4,4',5',6-Heptachlorobiphenyl
2,4',5-Trichlorobiphenyl	2,2',3,4',5,5',6-Heptachlorobiphenyl
2,2',3,5'-Tetrachlorobiphenyl	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl
2,2',5,5'-Tetrachlorobiphenyl	

TABLE 2-11

METHOD 8085 (GC/AED) - PESTICIDES

Abate (Temephos)	Dichlorprop	Metolachlor
Acifluorfen	Dichlorvos (DDVP)	Metribuzin
Alachlor	Diclofol (Kelthane)	Mevinphos
Aldrin	Diclofop-methyl	MGK-264
Ametryn	Dieldrin	Mirex
Atraton	Dimethoate	Molinate
Atrazine	Dinoseb	Napropamide
Azinphos ethyl (Ethyl guthion)	Dioxathion	Norflurazon
Azinphos methyl (Guthion)	Diphenamid	4-Nitrophenol
Benfluralin	Disulfoton (Disyston)	Oxyfluorfen
α-BHC	Diuron	Parathion
β-BHC	Endosulfan I	Pebulate
δ-BHC	Endosulfan II	Pendimethalin
γ-BHC (Lindane)	Endosulfan sulfate	Pentachlorophenol (PCP)
Bromacil	Endrin	Phorate
Bromoxynil (Brominal)	Endrin aldehyde	Phosphamidon
Butachlor	Endrin ketone	Picloram
Butylate	EPN	Profluralin
Captafol	Eptam (EPTC)	Prometon (Pramitol 5p)
Captan	Ethalfluralin (Sonalan)	Prometryn
Carbophenothion	Ethion	Pronamide (Kerb)
Carboxin	Ethoprop	Propachlor (Ramrod)
trans-Chlordane	Fenamiphos	Propargite (S-181)
Chlorpropham	Fenarimol	Propazine
Chlorpyrifos	Fenitrothion	Propetamidophos
Chlorthalonil (Daconil)	Fensulfothion	Ronnel
Cyanazine	Fluridone	Simazine
Cycloate	Fonofos	Sulfotepp
2,4-D acid	Gardona (Tetrachlovinphos)	Sulprofos (Bolstar)

TABLE 2-11
(continued)

Coumaphos	Fenthion	Silvex
2,4-DB acid	Heptachlor	2,4,5-T acid
DCPA (Dacthal)	Heptachlor epoxide	2,4,5-TB
2,4'-DDD	Hexachlorobenzene	Tebuthiuron
4,4'-DDD	Hexachlorocyclopentadiene	Terbacil
2,4'-DDE	Hexazinone	Terbutryn (Igran)
4,4'-DDE	Imidan (Phosmet)	2,3,4,5-Tetrachlorophenol
2,4'-DDT	Ioxynil	2,3,4,6-Tetrachlorophenol
4,4'-DDT	Malathion	Triademefon
DEF (Butifos)	MCPA acid	Triallate
Demeton-O	MCPP acid	Triclopyr (Garlon)
Demeton-S	Merphos	2,4,5-Trichlorophenol
Diallate	Metalaxyl	2,4,6-Trichlorophenol
Diazinon	Methoxychlor	Trifluralin (Treflan)
Dicamba	Methyl chlorpyrifos	Vernolate
Dichlobenil (Casoron)	Methyl paraoxon	
3,5-Dichlorobenzoic acid	Methyl parathion	

TABLE 2-12

METHOD 8091 (GC) - NITROAROMATICS AND CYCLIC KETONES

Benefin	2,4-Dinitrotoluene
Butralin	2,6-Dinitrotoluene
1-Chloro-2,4-dinitrobenzene	Isopropalin
1-Chloro-3,4-dinitrobenzene	1,2-Naphthoquinone
1-Chloro-2-nitrobenzene	1,4-Naphthoquinone
1-Chloro-4-nitrobenzene	Nitrobenzene
2-Chloro-6-nitrotoluene	2-Nitrotoluene
4-Chloro-2-nitrotoluene	3-Nitrotoluene
4-Chloro-3-nitrotoluene	4-Nitrotoluene
2,3-Dichloronitrobenzene	Penoxalin [Pendimethalin]
2,4-Dichloronitrobenzene	Pentachloronitrobenzene
3,5-Dichloronitrobenzene	Profluralin
3,4-Dichloronitrobenzene	2,3,4,5-Tetrachloronitrobenzene
2,5-Dichloronitrobenzene	2,3,5,6-Tetrachloronitrobenzene
Dinitramine	1,2,3-Trichloro-4-nitrobenzene
1,2-Dinitrobenzene	1,2,4-Trichloro-5-nitrobenzene
1,3-Dinitrobenzene	2,4,6-Trichloronitrobenzene
1,4-Dinitrobenzene	Trifluralin

TABLE 2-13

METHOD 8095 (GC) - EXPLOSIVES

2-Amino-4,6-dinitrotoluene	2-Nitrotoluene
4-Amino-2,6-dinitrotoluene	3-Nitrotoluene
3,5-Dinitroaniline	4-Nitrotoluene
1,3-Dinitrobenzene	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
2,4-Dinitrotoluene	Pentaerythritoltetranitrate
2,6-Dinitrotoluene	1,3,5-Trinitrobenzene
Hexahydro-1,3,5-trinitro-1,3,5-triazine	2,4,6-Trinitrophenylmethylnitramine
Nitrobenzene	2,4,6-Trinitrotoluene
Nitroglycerine	

TABLE 2-14

METHOD 8100 - POLYNUCLEAR AROMATIC HYDROCARBONS

Acenaphthene	Dibenz(*a,h*)anthracene
Acenaphthylene	7H-Dibenzo(*c,g*)carbazole
Anthracene	Dibenzo(*a,e*)pyrene
Benz(*a*)anthracene	Dibenzo(*a,h*)pyrene
Benzo(*b*)fluoranthene	Dibenzo(*a,i*)pyrene
Benzo(*j*)fluoranthene	Fluoranthene
Benzo(*k*)fluoranthene	Fluorene
Benzo(*g,h,i*)perylene	Indeno(1,2,3-*cd*)pyrene
Benzo(*a*)pyrene	3-Methylcholanthrene
Chrysene	Naphthalene
Dibenz(*a,h*)acridine	Phenanthrene
Dibenz(*a,j*)acridine	Pyrene

TABLE 2-15

METHOD 8111 (GC) - HALOETHERS

Bis(2-chloroethoxy)methane	2,5-Dichlorophenyl 4-nitrophenyl ether
Bis(2-chloroethyl) ether	2,4-Dichlorophenyl 4-nitrophenyl ether
Bis(2-chloroisopropyl) ether	2,3-Dichlorophenyl 4-nitrophenyl ether
4-Bromophenyl phenyl ether	3,4-Dichlorophenyl 4-nitrophenyl ether
4-Chlorophenyl phenyl ether	4-Nitrophenyl phenyl ether
2-Chlorophenyl 4-nitrophenyl ether	2,4,6-Trichlorophenyl 4-nitrophenyl ether
3-Chlorophenyl 4-nitrophenyl ether	2,3,6-Trichlorophenyl 4-nitrophenyl ether
4-Chlorophenyl 4-nitrophenyl ether	2,3,5-Trichlorophenyl 4-nitrophenyl ether
2,4-Dibromophenyl 4-nitrophenyl ether	2,4,5-Trichlorophenyl 4-nitrophenyl ether
2,4-Dichlorophenyl 3-methyl-4-nitrophenyl ether	3,4,5-Trichlorophenyl 4-nitrophenyl ether
2,6-Dichlorophenyl 4-nitrophenyl ether	2,3,4-Trichlorophenyl 4-nitrophenyl ether
3,5-Dichlorophenyl 4-nitrophenyl ether	

TABLE 2-16

METHOD 8121 (GC) - CHLORINATED HYDROCARBONS

Benzal chloride	δ-Hexachlorocyclohexane (δ-BHC)
Benzotrichloride	γ-Hexachlorocyclohexane (γ-BHC)
Benzyl chloride	Hexachlorocyclopentadiene
2-Chloronaphthalene	Hexachloroethane
1,2-Dichlorobenzene	Pentachlorobenzene
1,3-Dichlorobenzene	1,2,3,4-Tetrachlorobenzene
1,4-Dichlorobenzene	1,2,3,5-Tetrachlorobenzene
Hexachlorobenzene	1,2,4,5-Tetrachlorobenzene
Hexachlorobutadiene	1,2,3-Trichlorobenzene
α-Hexachlorocyclohexane (α-BHC)	1,2,4-Trichlorobenzene
β-Hexachlorocyclohexane (β-BHC)	1,3,5-Trichlorobenzene

TABLE 2-17

METHOD 8131 (GC) - ANILINE AND SELECTED DERIVATIVES

Aniline	2,6-Dibromo-4-nitroaniline
4-Bromoaniline	3,4-Dichloroaniline
2-Bromo-6-chloro-4-nitroanilne	2,6-Dichloro-4-nitroaniline
2-Bromo-4,6-dintroaniline	2,4-Dinitroaniline
2-Chloroaniline	2-Nitroaniline
3-Chloroaniline	3-Nitroaniline
4-Chloroaniline	4-Nitroaniline
2-Chloro-4,6-dinitroaniline	2,4,6-Trichloroaniline
2-Chloro-4-nitroaniline	2,4,5-Trichloroaniline
4-Chloro-2-nitroaniline	

TABLE 2-18

METHOD 8141 (GC) - ORGANOPHOSPHORUS COMPOUNDS

Aspon	Disulfoton	Parathion, methyl
Atrazine	EPN	Pebulate
Azinphos-ethyl	EPTC	*o*-Phenylenediamine
Azinphos-methyl	Ethion	Phorate
Bendiocarb	Ethoprop	Phosmet
Bolstar (Sulprofos)	Famphur	Phosphamidon
Butylate	Fenitrothion	Propham
Carbophenothion	Fensulfothion	Prosulfocarb
Chlorfenvinphos	Fenthion	Ronnel
Chlorpyrifos	Fonophos	Simazine
Chlorpyrifos methyl	Hexamethyl phosphoramide (HMPA)	Stirophos (Tetrachlorvinphos, Gardona)
Coumaphos	Leptophos	Sulfotepp
Crotoxyphos	Malathion	Tetraethyl pyrophosphate (TEPP)
Demeton-O, and -S	Merphos	Terbufos
Diazinon	Methiocarb	Triallate
Dichlorofenthion	Mevinphos	Thionazin (Zinophos)
Dichlorvos (DDVP)	Molinate	Tokuthion (Prothiofos)
Dicrotophos	Monocrotophos	Trichlorfon
Dimethoate	Naled	Trichloronate
Dioxathion	Parathion, ethyl	Tri-*o*-cresyl phosphate (TOCP)

TABLE 2-19

METHOD 8151 (GC USING METHYLATION OR PENTAFLUOROBENZYLATION DERIVATIZATION) - CHLORINATED HERBICIDES

Acifluorfen	Dicamba	MCPP
Bentazon	3,5-Dichlorobenzoic acid	4-Nitrophenol
Chloramben	Dichloroprop	Pentachlorophenol
2,4-D	Dinoseb	Picloram
Dalapon	5-Hydroxydicamba	2,4,5-TP (Silvex)
2,4-DB	MCPA	2,4,5-T
DCPA diacid		

TABLE 2-20

METHOD 8260 (GC/MS) - VOLATILE ORGANIC COMPOUNDS

Acetone	Dibromofluoromethane	Methylene chloride
Acetonitrile	Dibromomethane	Methyl acrylate
Acrolein (Propenal)	1,2-Dichlorobenzene	Methyl methacrylate
Acrylonitrile	1,3-Dichlorobenzene	4-Methyl-2-pentanone (MIBK)
Allyl alcohol	1,4-Dichlorobenzene	Naphthalene
Allyl chloride	*cis*-1,4-Dichloro-2-butene	Nitrobenzene
Benzene	*trans*-1,4-Dichloro-2-butene	2-Nitropropane
Benzyl chloride	Dichlorodifluoromethane	*N*-Nitroso-di-*n*-butylamine
Bis(2-chloroethyl)-sulfide	1,1-Dichloroethane	Paraldehyde
Bromoacetone	1,2-Dichloroethane	Pentachloroethane
Bromobenzene	1,1-Dichloroethene	Pentafluorobenzene
Bromochloromethane	*cis*-1,2-Dichloroethene	2-Pentanone
Bromodichloromethane	*trans*-1,2-Dichloroethene	2-Picoline
Bromoform	1,2-Dichloropropane	1-Propanol
Bromomethane	1,3-Dichloropropane	2-Propanol
n-Butanol	2,2-Dichloropropane	Propargyl alcohol
2-Butanone (MEK)	1,3-Dichloro-2-propanol	ß-Propiolactone
t-Butyl alcohol	1,1-Dichloropropene	Propionitrile (Ethyl cyanide)
n-Butylbenzene	*cis*-1,3-Dichloropropene	*n*-Propylamine
sec-Butylbenzene	*trans*-1,3-Dichloropropene	*n*-Propylbenzene
tert-Butylbenzene	1,2,3,4-Diepoxybutane	Pyridine
Carbon disulfide	Diethyl ether	Styrene
Carbon tetrachloride	1,4-Dioxane	1,1,1,2-Tetrachloroethane
Chloral hydrate	Epichlorohydrin	1,1,2,2-Tetrachloroethane
Chloroacetonitrile	Ethanol	Tetrachloroethene
Chlorobenzene	Ethyl acetate	Toluene

TABLE 2-20
(continued)

1-Chlorobutane	Ethylbenzene	*o*-Toluidine
Chlorodibromomethane	Ethylene oxide	1,2,3-Trichlorobenzene
Chloroethane	Ethyl methacrylate	1,2,4-Trichlorobenzene
2-Chloroethanol	Hexachlorobutadiene	1,1,1-Trichloroethane
2-Chloroethyl vinyl ether	Hexachloroethane	1,1,2-Trichloroethane
Chloroform	2-Hexanone	Trichloroethene
1-Chlorohexane	2-Hydroxypropionitrile	Trichlorofluoromethane
Chloromethane	Iodomethane	1,2,3-Trichloropropane
Chloroprene	Isobutyl alcohol	1,2,4-Trimethylbenzene
3-Chloropropionitrile	Isopropylbenzene	1,3,5-Trimethylbenzene
2-Chlorotoluene	*p*-Isopropyltoluene	Vinyl acetate
4-Chlorotoluene	Malononitrile	Vinyl chloride
Crotonaldehyde	Methacrylonitrile	*o*-Xylene
1,2-Dibromo-3-chloropropane	Methanol	*m*-Xylene
1,2-Dibromoethane	Methyl-*t*-butyl ether	*p*-Xylene

TABLE 2-21

METHOD 8261 (VD/GC/MS) - VOLATILE ORGANIC COMPOUNDS

Acetone	1,3-Dichlorobenzene	Methacrylonitrile
Acetonitrile	1,4-Dichlorobenzene	Methyl *t*-butyl ether (MTBE)
Acetophenone	*cis*-1,4-Dichloro-2-butene	Methylene chloride
Acrolein	*trans*-1,4-Dichloro-2-butene	Methyl methacrylate
Acrylonitrile	Dichlorodifluoromethane	1-Methylnaphthalene
Allyl Chloride	1,1-Dichloroethane	2-Methylnaphthalene
t-Amyl ethyl ether (TAEE) (4,4-Dimethyl-3-oxahexane)	1,2-Dichloroethane	4-Methyl-2-pentanone
t-Amyl methyl ether (TAME)	1,1-Dichloroethene	Naphthalene
Aniline	*trans*-1,2-Dichloroethene	*N*-Nitrosodimethylamine
Benzene	*cis*-1,2-Dichloroethene	*N*-Nitrosodi-*n*-propylamine
Bromochloromethane	1,2-Dichloropropane	*N*-Nitrosomethylethylamine
Bromodichloromethane	1,3-Dichloropropane	*N*-Nitrosodibutylamine
Bromoform	2,2-Dichloropropane	*N*-Nitrosodiethylamine
Bromomethane	1,1-Dichloropropene	Pentachloroethane
2-Butanone	*cis*-1,3-Dichloropropene	2-Picoline
n-Butylbenzene	*trans*-1,3-Dichloropropene	Propionitrile
sec-Butylbenzene	Diethyl ether	*n*-Propylbenzene
tert-Butylbenzene	Diisopropyl ether (DIPE)	Pyridine
Carbon disulfide	1,4-Dioxane	Styrene
Carbon tetrachloride	Ethanol	1,1,2,2-Tetrachloroethane
Chlorobenzene	Ethyl acetate	Tetrachloroethene
Chlorodibromomethane	Ethylbenzene	Tetrahydrofuran
Chloroethane	Ethyl *t*-butyl ether (ETBE)	Toluene
Chloroform	Ethyl methacrylate	*o*-Toluidine
Chloromethane	Hexachlorobutadiene	1,2,3-Trichlorobenzene
2-Chlorotoluene	2-Hexanone	1,2,4-Trichlorobenzene
4-Chlorotoluene	Iodomethane	1,1,1-Trichloroethane
1,2-Dibromo-3-chloropropane	Isobutyl alcohol	1,1,2-Trichloroethane
Dibromomethane	Isopropylbenzene	Trichloroethene
1,2-Dichlorobenzene	*p*-Isopropyltoluene	Trichlorofluoromethane

TABLE 2-21
(continued)

1,2,3-Trichloropropane	*o*-Xylene
1,2,4-Trimethylbenzene	*m*-Xylene
1,3,5-Trimethylbenzene	*p*-Xylene
Vinyl chloride	

TABLE 2-22

METHOD 8270 (GC/MS) - SEMIVOLATILE ORGANIC COMPOUNDS

Acenaphthene	Endrin aldehyde
Acenaphthylene	Endrin ketone
Acetophenone	EPN
2-Acetylaminofluorene	Ethion
1-Acetyl-2-thiourea	Ethyl carbamate
Aldrin	Ethyl methanesulfonate
2-Aminoanthraquinone	Famphur
Aminoazobenzene	Fensulfothion
4-Aminobiphenyl	Fenthion
3-Amino-9-ethylcarbazole	Fluchloralin
Anilazine	Fluoranthene
Aniline	Fluorene
o-Anisidine	Heptachlor
Anthracene	Heptachlor epoxide
Aramite	Hexachlorobenzene
Aroclor-1016	Hexachlorobutadiene
Aroclor-1221	Hexachlorocyclopentadiene
Aroclor-1232	Hexachloroethane
Aroclor-1242	Hexachlorophene
Aroclor-1248	Hexachloropropene
Aroclor-1254	Hexamethylphosphoramide

TABLE 2-22
(continued)

Aroclor-1260	Hydroquinone
Azinphos-methyl	Indeno(1,2,3-cd)pyrene
Barban	Isodrin
Benz(a)anthracene	Isophorone
Benzidine	Isosafrole
Benzo(b)fluoranthene	Kepone
Benzo(k)fluoranthene	Leptophos
Benzoic acid	Malathion
Benzo(g,h,i)perylene	Maleic anhydride
Benzo(a)pyrene	Mestranol
p-Benzoquinone	Methapyrilene
Benzyl alcohol	Methoxychlor
α-BHC	3-Methylcholanthrene
β-BHC	4,4'-Methylenebis(2-chloroaniline)
δ-BHC	4,4'-Methylenebis(*N,N*-dimethylaniline)
γ-BHC (Lindane)	Methyl methanesulfonate
Bis(2-chloroethoxy)-methane	2-Methylnaphthalene
Bis(2-chloroethyl)ether	Methyl parathion
Bis(2-chloroisopropyl)ether	2-Methylphenol
Bis(2-ethylhexyl)phthalate	3-Methylphenol
4-Bromophenyl phenyl ether	4-Methylphenol
Bromoxynil	Mevinphos
Butyl benzyl phthalate	Mexacarbate
Captafol	Mirex
Captan	Monocrotophos
Carbaryl	Naled
Carbofuran	Naphthalene
Carbophenothion	1,4-Naphthoquinone
Chlordane (NOS)	1-Naphthylamine
Chlorfenvinphos	2-Naphthylamine

TABLE 2-22
(continued)

4-Chloroaniline	Nicotine
Chlorobenzilate	5-Nitroacenaphthene
5-Chloro-2-methylaniline	2-Nitroaniline
4-Chloro-3-methylphenol	3-Nitroaniline
3-(Chloromethyl)pyridine hydrochloride	4-Nitroaniline
1-Chloronaphthalene	5-Nitro-*o*-anisidine
2-Chloronaphthalene	Nitrobenzene
2-Chlorophenol	4-Nitrobiphenyl
4-Chloro-1,2-phenylenediamine	Nitrofen
4-Chloro-1,3-phenylenediamine	2-Nitrophenol
4-Chlorophenyl phenyl ether	4-Nitrophenol
Chrysene	Nitroquinoline-1-oxide
Coumaphos	*N*-Nitrosodi-*n*-butylamine
p-Cresidine	*N*-Nitrosodiethylamine
Crotoxyphos	*N*-Nitrosodimethylamine
2-Cyclohexyl-4,6-dinitrophenol	*N*-Nitrosodiphenylamine
4,4'-DDD	*N*-Nitrosodi-*n*-propylamine
4,4'-DDE	*N*-Nitrosomethylethylamine
4,4'-DDT	*N*-Nitrosomorpholine
Demeton-O	*N*-Nitrosopiperidine
Demeton-S	*N*-Nitrosopyrrolidine
Diallate (*cis* or *trans*)	5-Nitro-*o*-toluidine
2,4-Diaminotoluene	Octamethyl pyrophosphoramide
Dibenz(*a,j*)acridine	4,4'-Oxydianiline
Dibenz(*a,h*)anthracene	Parathion
Dibenzofuran	Pentachlorobenzene
Dibenzo(*a,e*)pyrene	Pentachloronitrobenzene
1,2-Dibromo-3-chloropropane	Pentachlorophenol
Di-*n*-butyl phthalate	Phenacetin
Dichlone	Phenanthrene

TABLE 2-22
(continued)

1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
3,3'-Dichlorobenzidine
2,4-Dichlorophenol
2,6-Dichlorophenol
Dichlorovos
Dicrotophos
Dieldrin
Diethyl phthalate
Diethylstilbestrol
Diethyl sulfate
Dimethoate
3,3'-Dimethoxybenzidine
Dimethylaminoazobenzene
7,12-Dimethylbenz(*a*)anthracene
3,3'-Dimethylbenzidine
α,α-Dimethylphenethylamine
2,4-Dimethylphenol
Dimethyl phthalate
1,2-Dinitrobenzene
1,3-Dinitrobenzene
1,4-Dinitrobenzene
4,6-Dinitro-2-methylphenol
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Dinocap
Di-*n*-octyl phthalate
Diphenylamine
Phenobarbital
Phenol
1,4-Phenylenediamine
Phorate
Phosalone
Phosmet
Phosphamidion
Phthalic anhydride
2-Picoline (2-Methylpyridine)
Piperonyl sulfoxide
Pronamide
Propylthiouracil
Pyrene
Resorcinol
Safrole
Strychnine
Sulfallate
Terbufos
1,2,4,5-Tetrachlorobenzene
2,3,4,6-Tetrachlorophenol
Tetrachlorvinphos
Tetraethyl dithiopyrophosphate
Tetraethyl pyrophosphate
Thionazine
Thiophenol (Benzenethiol)
Toluene diisocyanate
o-Toluidine
Toxaphene
1,2,4-Trichlorobenzene
2,4,5-Trichlorophenol

TABLE 2-22
(continued)

5,5-Diphenylhydantoin	2,4,6-Trichlorophenol
1,2-Diphenylhydrazine	*O,O,O*-Triethylphosphorothioate
Dinoseb	Trifluralin
Disulfoton	2,4,5-Trimethylaniline
Endosulfan I	Trimethyl phosphate
Endosulfan II	1,3,5-Trinitrobenzene
Endosulfan sulfate	Tris(2,3-dibromopropyl)phosphate
Endrin	Tri-*p*-tolyl phosphate

TABLE 2-23

METHOD 8275 (TE/GC/MS) - SEMIVOLATILE ORGANIC COMPOUNDS

Acenaphthene	1,2,4-Trichlorobenzene
Acenaphthylene	2-Chlorobiphenyl
Anthracene	3,3'-Dichlorobiphenyl
Benz(*a*)anthracene	2,2',5-Trichlorobiphenyl
Benzo(*a*)pyrene	2,3',5-Trichlorobiphenyl
Benzo(*b*)fluoranthene	2,4',5-Trichlorobiphenyl
Benzo(*g,h,i*)perylene	2,2',5,5'-Tetrachlorobiphenyl
Benzo(*k*)fluoranthene	2,2'4,5'-Tetrachlorobiphenyl
4-Bromophenyl phenyl ether	2,2'3,5'-Tetrachlorobiphenyl
1-Chloronaphthalene	2,3',4,4'-Tetrachlorobiphenyl
Chrysene	2,2',4,5,5'-Pentachlorobiphenyl
Dibenzofuran	2,3',4,4',5-Pentachlorobiphenyl
Dibenz(*a,h*)anthracene	2,2',3,4,4',5'- Hexachlorobiphenyl
Dibenzothiophene	2,2',3,3',4,4'- Hexachlorobiphenyl
Fluoranthene	2,2',3,4',5,5',6- Heptachlorobiphenyl
Fluorene	2,2',3,4,4',5,5'- Heptachlorobiphenyl
Hexachlorobenzene	2,2',3,3',4,4',5- Heptachlorobiphenyl
Indeno(1,2,3-*cd*)pyrene	2,2',3,3',4,4',5,5'- Octachlorobiphenyl
Naphthalene	2,2',3,3'4,4',5,5',6- Nonachlorobiphenyl
Phenanthrene	2,2',3,3'4,4',5,5',6,6'- Decachlorobiphenyl
Pyrene	

TABLE 2-24

METHODS 8280 (HRGC/LRMS) AND 8290 (HRGC/HRMS) -
POLYCHLORINATED DIBENZO-*p*-DIOXINS (PCDDs)
AND POLYCHLORINATED DIBENZOFURANS (PCDFs)

2,3,7,8-TCDD	1,2,3,7,8-PeCDF
TCDD, total	2,3,4,7,8-PeCDF
1,2,3,7,8-PeCDD	PeCDF, total
PeCDD, total	1,2,3,4,7,8-HxCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,6,7,8-HxCDD	1,2,3,7,8,9-HxCDF
1,2,3,7,8,9-HxCDD	2,3,4,6,7,8-HxCDF
HxCDD, total	HxCDF, total
1,2,3,4,6,7,8-HpCDD	1,2,3,4,6,7,8-HpCDF
HpCDD, total	1,2,3,4,7,8,9-HpCDF
OCDD	HpCDF, total
2,3,7,8-TCDF	OCDF
TCDF, total	

TABLE 2-25

METHOD 8310 (HPLC) - POLYNUCLEAR AROMATIC HYDROCARBONS

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(*a,h*)anthracene
Anthracene	Fluoranthene
Benz(*a*)anthracene	Fluorene
Benzo(*a*)pyrene	Indeno(1,2,3-*cd*)pyrene
Benzo(*b*)fluoranthene	Naphthalene
Benzo(*g,h,i*)perylene	Phenanthrene
Benzo(*k*)fluoranthene	Pyrene

TABLE 2-26

METHOD 8315 - CARBONYL COMPOUNDS

Acetaldehyde	Decanal	Octanal
Acetone	2,5-Dimethylbenzaldehyde	Pentanal (Valeraldehyde)
Acrolein	Formaldehyde	Propanal (Propionaldehyde)
Benzaldehyde	Heptanal	*m*-Tolualdehyde
Butanal (Butyraldehyde)	Hexanal (Hexaldehyde)	*o*-Tolualdehyde
Crotonaldehyde	Isovaleraldehyde	*p*-Tolualdehyde
Cyclohexanone	Nonanal	

TABLE 2-27

METHOD 8316 (HPLC)

Acrylamide
Acrylonitrile
Acrolein

TABLE 2-28

METHOD 8318 (HPLC) - *N*-METHYLCARBAMATES

Aldicarb (Temik)	Dioxacarb	Mexacarbate
Aldicarb sulfone	Formetanate hydrochloride	Oxamyl
Bendiocarb	3-Hydroxycarbofuran	Promecarb
Carbaryl (Sevin)	Methiocarb (Mesurol)	Propoxur (Baygon)
Carbofuran (Furadan)	Methomyl (Lannate)	Thiodicarb
m-Cumenyl methylcarbamate	Metolcarb	

TABLE 2-29

METHOD 8321 (HPLC/TS/MS) - NONVOLATILE ORGANIC COMPOUNDS

Azo Dyes	Carbamates
Disperse Red 1	Aldicarb
Disperse Red 5	Aldicarb sulfone
Disperse Red 13	Aldicarb sulfoxide
Disperse Yellow 5	Aminocarb
Disperse Orange 3	Barban
Disperse Orange 30	Benomyl
Disperse Brown 1	Bendiocarb
Solvent Red 3	Bromacil
Solvent Red 23	Butylate
	Carbaryl
	Carbendazim
Chlorinated Phenoxyacid Compounds	Carbofuran
2,4-D	Carbofuran phenol
2,4-D, butoxyethanol ester	Carbosulfan
2,4-D, ethylhexyl ester	Chloropropham
2,4-DB	Chloroxuron
Dalapon	*m*-Cumenyl methyl carbamate
Dicamba	Diuron
Dichlorprop	EPTC
Dinoseb	Fenuron
MCPA	Fluometuron
MCPP	Formetanate hydrochloride
Silvex (2,4,5-TP)	3-Hydroxycarbofuran
2,4,5-T	Linuron
2,4,5-T, butyl ester	Methiocarb
2,4,5-T, butoxyethanol ester	Methomyl
	Metolcarb

TABLE 2-29
(continued)

Organophosphorus Compounds	Carbamates (cont.)
Asulam	Mexacarbate
Fensulfothion	Molinate
Dichlorvos (DDVP)	Monuron
Dimethoate	Neburon
Disulfoton	Oxamyl
Parathion methyl	Pebulate
Merphos	*o*-Phenylenediamine
Methomyl	Physostigmine
Monocrotophos	Physostigmine salicylate
Famphur	Promecarb
Naled	Propham
Phorate	Propoxur
Trichlorfon	Prosulfocarb
Thiofanox	Siduron
Tris(2,3-dibromopropyl) phosphate (Tris-BP)	Tebuthiuron
	Thiodicarb
Anthraquinone Dyes	Thiophanate-methyl
Disperse Blue 3	Triallate
Disperse Blue 14	
Disperse Red 60	

TABLE 2-30

METHOD 8325 (HPLC/PB/MS) - NONVOLATILE ORGANIC COMPOUNDS

Benzidine	3,3'-Dimethylbenzidine
Benzoylprop ethyl	Diuron
Carbaryl	Linuron (Lorox)
o-Chlorophenyl thiourea	Monuron
3,3'-Dichlorobenzidine	Rotenone
3,3'-Dimethoxybenzidine	Siduron

TABLE 2-31

METHOD 8330 (HPLC) - NITROAROMATICS AND NITRAMINES

4-Amino-2,6-dinitrotoluene (4-Am-DNT)	Nitrobenzene (NB)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	2-Nitrotoluene (2-NT)
1,3-Dinitrobenzene (1,3-DNB)	3-Nitrotoluene (3-NT)
2,4-Dinitrotoluene (2,4-DNT)	4-Nitrotoluene (4-NT)
2,6-Dinitrotoluene (2,6-DNT)	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1,3,5-Trinitrobenzene (1,3,5-TNB)
Methyl-2,4,6-trinitrophenyl-nitramine (Tetryl)	2,4,6-Trinitrotoluene (2,4,6-TNT)

TABLE 2-32

METHOD 8331 (HPLC)

Tetrazene

TABLE 2-33

METHOD 8332 (HPLC)

Nitroglycerine

TABLE 2-34

METHOD 8410 - SEMIVOLATILE ORGANIC COMPOUNDS

Acenaphthene	2,6-Dinitrotoluene
Acenaphthylene	Di-*n*-octyl phthalate
Anthracene	Di-*n*-propyl phthalate
Benzo(*a*)anthracene	Fluoranthene
Benzo(*a*)pyrene	Fluorene
Benzoic acid	Hexachlorobenzene
Bis(2-chloroethoxy)methane	1,3-Hexachlorobutadiene
Bis(2-chloroethyl) ether	Hexachlorocyclopentadiene
Bis(2-chloroisopropyl) ether	Hexachloroethane
Bis(2-ethylhexyl) phthalate	Isophorone
4-Bromophenyl phenyl ether	2-Methylnaphthalene
Butyl benzyl phthalate	2-Methylphenol
4-Chloroaniline	4-Methylphenol
4-Chloro-3-methylphenol	Naphthalene
2-Chloronaphthalene	2-Nitroaniline
2-Chlorophenol	3-Nitroaniline
4-Chlorophenol	4-Nitroaniline
4-Chlorophenyl phenyl ether	Nitrobenzene
Chrysene	2-Nitrophenol
Dibenzofuran	4-Nitrophenol
Di-*n*-butyl phthalate	*N*-Nitrosodimethylamine
1,2-Dichlorobenzene	*N*-Nitrosodiphenylamine
1,3-Dichlorobenzene	*N*-Nitroso-di-*n*-propylamine
1,4-Dichlorobenzene	Pentachlorophenol
2,4-Dichlorophenol	Phenanthrene
Diethyl phthalate	Phenol
Dimethyl phthalate	Pyrene
4,6-Dinitro-2-methylphenol	1,2,4-Trichlorobenzene
2,4-Dinitrophenol	2,4,5-Trichlorophenol
2,4-Dinitrotoluene	2,4,6-Trichlorophenol

TABLE 2-35

METHOD 8430 (GC/FT-IR) - BIS(2-CHLOROETHYL) ETHER
AND ITS HYDROLYSIS PRODUCTS

Bis(2-chloroethyl) ether
2-Chloroethanol
2-(2-Chloroethoxy)ethanol
Diethylene glycol
Ethylene glycol

TABLE 2-36

METHOD 8510 (COLORIMETRIC SCREENING) - RDX AND HMX

Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)

TABLE 2-37

METHOD 8535 (COLORIMETRIC SCREENING) - VOLATILE ORGANIC HALIDES

Trichloroethylene
Perchloroethylene (Tetrachloroethene)
Carbon tetrachloride
Trihalomethanes

TABLE 2-38

METHOD 8540 (UV-INDUCED COLORIMETRY) - PENTACHLOROPHENOL

Pentachlorophenol

TABLE 2-39

DETERMINATIVE METHODS FOR INORGANIC ANALYTES

Analyte	Applicable Methods
Aluminum	6010, 6020, 7000, 7010
Antimony	6010, 6020, 6200, 6800, 7000, 7062
Arsenic	6010, 6020, 6200, 7010, 7061, 7062, 7063
Barium	6010, 6020, 6200, 6800, 7000, 7010
Beryllium	6010, 6020, 7000, 7010
Boron	6010, 6800
Bromide	6500, 9056, 9211
Cadmium	6010, 6020, 6200, 6800, 7000, 7010
Calcium	6010, 6020, 6200, 6800, 7000
Chloride	6500, 9056, 9057, 9212, 9250, 9251, 9253
Chromium	6010, 6020, 6200, 6800, 7000, 7010
Chromium, hexavalent	6800, 7195, 7196, 7197, 7198, 7199
Cobalt	6010, 6020, 6200, 7000, 7010
Copper	6010, 6020, 6200, 6800, 7000, 7010
Cyanide	9010, 9012, 9013, 9213
Fluoride	6500, 9056, 9214
Iron	6010, 6020, 6200, 6800, 7000, 7010
Lead	6010, 6020, 6200, 6800, 7000, 7010
Lithium	6010, 7000
Magnesium	6010, 6020, 6800, 7000
Manganese	6010, 6020, 6200, 7000, 7010
Mercury	6010, 6020, 6200, 6800, 7470, 7471, 7472, 7473, 7474
Molybdenum	6010, 6200, 6800, 7000, 7010
Nickel	6010, 6020, 6200, 6800, 7000, 7010
Nitrate	6500, 9056, 9210
Nitrite	6500, 9056, 9216
Osmium	7000
Phosphate	6500, 9056
Phosphorus	6010
Phosphorus, white	7580
Potassium	6010, 6020, 6200, 6800, 7000
Rubidium	6200
Selenium	6010, 6020, 6200, 6800, 7010, 7741, 7742
Silver	6010, 6020, 6200, 6800, 7000, 7010
Silica	6010
Sodium	6010, 6020, 7000
Strontium	6010, 6200, 6800, 7000
Sulfate	6500, 9035, 9036, 9038, 9056
Sulfide	9030, 9031, 9215
Thallium	6010, 6020, 6200, 6800, 7000, 7010
Thorium	6200
Tin	6010, 6200, 7000
Titanium	6010, 6200
Vanadium	6010, 6020, 6200, 6800, 7000, 7010
Zinc	6010, 6020, 6200, 6800, 7000, 7010
Zirconium	6200

TABLE 2-40(A)

RECOMMENDED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR ORGANIC ANALYTES[a]
(Note: Footnotes are located on the last page of the table.)

VOLATILE ORGANICS

Sample Matrix	Container	Preservative[1]	Holding Time[2]
Concentrated waste samples	Method 5035: See method. Method 5021: See method. Methods 5031 and 5032: See methods. Use PTFE-lined lids for all procedures.	Cool to ≤6 °C.	14 days
Aqueous samples with no residual chlorine present	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Cool to ≤6 °C and adjust pH to less than 2 with H_2SO_4, HCl, or solid $NaHSO_4$	14 days
		If carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples.	*7 days*
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	*7 days*

VOLATILE ORGANICS (continued)			
Sample Matrix	Container	Preservative[1]	Holding Time[2]
Aqueous samples WITH residual chlorine present	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to ≤6 °C and adjust pH to less than 2 with H_2SO_4, HCl, or solid $NaHSO_4$.	14 days
		If carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples.	*7 days*
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	*7 days*
Acrolein and acrylonitrile in aqueous samples	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Adjust to pH 4-5. Cool to ≤6 °C. *These compounds are highly reactive and should be analyzed as soon as possible.*	7days
Solid samples (e.g. soils, sediments, sludges, ash)	Method 5035: See method. Method 5021: See method. Methods 5031 and 5032: See methods.	See the individual methods.	14 days
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	7 days

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES AND HERBICIDES

Sample Matrix	Container	Preservative[1]	Holding Time[2]
Concentrated waste samples	125-mL wide-mouth glass with PTFE-lined lid	None	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.
Aqueous samples with no residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Cool to ≤6 °C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES AND HERBICIDES (continued)

Sample Matrix	Container	Preservative[1]	Holding Time[2]
Aqueous samples WITH residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤6 °C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Solid samples (e.g. soils, sediments, sludges, ash)	250-mL wide-mouth glass container with PTFE-lined lid	Cool to ≤6 °C.	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.

POLYCHLORINATED BIPHENYLS, POLYCHLORINATED DIBENZO-*p*-DIOXINS, AND POLYCHLORINATED DIBENZOFURANS

Sample Matrix	Container	Preservative[1]	Holding Time[2]
Concentrated waste samples	125-mL wide-mouth glass with PTFE-lined lid	None	None
Aqueous samples with no residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Cool to ≤6 °C.	None

POLYCHLORINATED BIPHENYLS, POLYCHLORINATED DIBENZO-*p*-DIOXINS, AND POLYCHLORINATED DIBENZOFURANS (continued)

Sample Matrix	Container	Preservative[1]	Holding Time[2]
Aqueous samples WITH residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤6 °C.	None
Solid samples (e.g. soils, sediments, sludges, ash)	250-mL wide-mouth glass container with PTFE-lined lid.	Cool to ≤6 °C.	None

[a] The information presented in this table does not represent EPA requirements, but rather it is intended solely as guidance. Selection of containers, preservation techniques and applicable holding times should be based on the stated project-specific data quality objectives. See Chapter Three, Chapter Four, or the individual methods for more information.

[1] The exact sample, extract, and standard storage temperature should be based on project-specific requirements and/or manufacturer's recommendations for commercially available standards. Furthermore, alternative storage temperatures may be appropriate based on demonstrated analyte stability in a given matrix, provided the stated data quality objectives for a project-specific application are still attainable.

[2] A longer holding time may be appropriate if it can be demonstrated that the reported analyte concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

TABLE 2-40(B)

RECOMMENDED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR INORGANIC AND OTHER ANALYTES IN AQUEOUS MATRICES[a] (SEE CHAPTER THREE FOR MORE DETAILED GUIDANCE, INCLUDING REGARDING SOLID MATRICES)

Name	Container[1]	Preservation[2]	Holding Time[3]
Inorganic Tests:			
Chloride	P, G	None required	28 days
Cyanide, total and amenable to chlorination	P, G	Cool to ≤6 °C; if oxidizing agents present add 5 mL 0.1N $NaAsO_2$ per L or 0.06 g of ascorbic acid per L; adjust pH>12 with 50% NaOH. See Method 9010 for other interferences.	14 days
Hydrogen ion (pH)	P, G	None required	As soon as possible
Nitrate	P, G	Cool to ≤6 °C.	48 hours
Sulfate	P, G	Cool to ≤6 °C.	28 days
Sulfide	P, G	Cool to ≤6 °C , add zinc acetate NaOH to pH >9	7 days
Metals:			
Chromium VI	P, G	Cool to ≤6 °C.	24 hours
Mercury	P, G	HNO_3 to pH<2	28 days
All Other Metals	P, G	HNO_3 to pH<2	6 months
Hexane Extractable Material (HEM; Oil and grease)	G	Cool ≤6 °C, HCl or H_2SO_4 to pH<2	28 days
Organic carbon, total (TOC)	P, G	Cool to ≤6 °C, store in dark HCl or H_2SO_4 to pH<2	28 days
Radiological Tests:			
Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months

[a] The information presented in this table does not represent EPA requirements, but rather it is intended solely as guidance. Selection of containers, preservation techniques and applicable holding times should be based on the stated project-specific data quality objectives. See Chapter Three, Chapter Four, or the individual methods for more information.

[1] Polyethylene (P) or glass (G)

[2] The exact sample, extract, and standard storage temperature should be based on project-specific requirements and/or manufacturer's recommendations for commercially available standards. Furthermore, alternative storage temperatures may be appropriate based on demonstrated analyte stability in a given matrix, provided the stated data quality objectives for a project-specific application are still attainable.

[3] A longer holding time may be appropriate if it can be demonstrated that the reported analyte concentrations are not adversely affected by preservation, storage and analyses performed outside the recommended holding times.

TABLE 2-41

PREPARATION METHODS FOR ORGANIC ANALYTES
(Note: Footnotes are located on the last page of the table.)

	Matrix			
Analyte Type	Aqueous[1]	Solids	Sludges and Emulsions[1,2]	Organic Liquids, Tars, Oils
Acid Extractable	3510 3520 (pH ≤ 2)	3540 3541 3542[13] 3545 3546 3550	3520 (pH ≤ 2)	3650 3580[3]
Acrolein[12], Acrylonitrile[12], and Acetonitrile	5031 5032[12]	5031 5032[12]	5031 5032[12]	3585
Acrylamide	8032[4]			
Aniline and Selected Derivatives	3510 3520 (pH >11) 5031[11]	3540 3541 3545 3550	3520 (pH >11)	3580[3]
Aromatic Volatiles	5021 5030 5032	5021 5032 5035	5030 5032	3585
Base/Neutral Extractable	3510 3520 (pH >11)	3540 3541 3542[13] 3545 3546 3550	3520 (pH >11)	3650 3580[3]
Carbamates	8318[5] 8321	8318[5] 8321	8318[5]	8318[5]
Chlorinated Herbicides	3535 (pH < 1) 8151[6] (pH ≤ 2) 8321	3545 3546 8151[6] 8321	8151[6] (pH ≤ 2)	3580[3]
Chlorinated Hydrocarbons	3510 3520 (pH as received)	3540 3541 3550	3520 (pH as received)	3580[3]
Dyes	3510 3520	3540 3541 3545 3550		
Explosives	3535 8330[7] 8331[8]	8330[7] 8331[8]		
Formaldehyde	8315[9]	8315[9]		

TABLE 2-41
(continued)

Analyte Type	Matrix: Aqueous[1]	Solids	Sludges and Emulsions[1,2]	Organic Liquids, Tars, Oils
Haloethers	3510 3520	3540 3541 3545 3550		
Halogenated Volatiles	5021 5030 5032	5021 5032 5035	5030	3585
Nitroaromatics and Cyclic Ketones	3510 3520 (pH 5-9) 3535	3540 3541 3545 3550	3520 (pH 5-9)	3580[3]
Nitrosamines	3510 3520	3540 3541 3545 3550		
Non-halogenated Volatiles	5021 5031 5032	5021 5031 5032	5021 5031 5032	5032 3585
Organochlorine Pesticides	3510 3520 3535 (pH 5-9)	3540 3541 3545 3546 3550 3562	3520 (pH 5-9)	3580[3]
Organophosphorus Pesticides	3510 3520 (pH 5-8) 3535	3540 3541 3545 3546	3520 (pH 5-8)	3580[3]
Phenols	3510 3520 (pH $\leq$ 2) 3535	3540 3541 3545 3546 3550 3562	3520 (pH $\leq$ 2)	3650 3580[3]
Phthalate Esters	3510 3520 3535 (pH 5-7)	3540 3541 3545 3546 3550	3520 (pH 5- 7)	3580[3]
Polychlorinated Biphenyls	3510 3520 3535 (pH 5-9)	3540 3541 3545 3546 3562	3520 (pH 5-9)	3580[3]

TABLE 2-41
(continued)

Analyte Type	Matrix			
	Aqueous[1]	Solids	Sludges and Emulsions[1,2]	Organic Liquids, Tars, Oils
PCDDs and PCDFs	8280[10] 8290[10]	3545 3546 8280[10] 8290[10]	8280[10] 8290[10]	8280[10] 8290[10]
Polynuclear Aromatic Hydrocarbons	3510 3520 (pH as received)	3540 3541 3545 3546 3550 3561	3520 (pH as received)	3580[3]
Volatile Organics	5021 5030 5031 5032	5021 5031 5032 5035	5021 5030 5031 5032	3585

1 The pH at which extraction should be performed is shown in parentheses.
2 If attempts to break an emulsion are unsuccessful, these methods may be used.
3 Method 3580 is only appropriate if the sample is soluble in the specified solvent.
4 Method 8032 contains the extraction, cleanup, and determinative procedures for this analyte.
5 Method 8318 contains the extraction, cleanup, and determinative procedures for these analytes.
6 Method 8151 contains the extraction, cleanup, and determinative procedures for these analytes.
7 Method 8330 contains the extraction, cleanup, and determinative procedures for these analytes.
8 Method 8331 is for Tetrazene only, and contains the extraction, cleanup, and determinative procedures for this analyte.
9 Method 8315 contains the extraction, cleanup, and determinative procedures for this analyte.
10 Methods 8280 and 8290 contain the extraction, cleanup, and determinative procedures for these analytes.
11 Method 5031 may be used when only aniline is to be determined.
12 Method 5032 may be used for acrolein and acrylonitrile.
13 Method 3542 is used for extraction of semivolatiles from stack samples collected using Method 0010.

TABLE 2-42

CLEANUP METHODS FOR ORGANIC ANALYTE EXTRACTS

Analyte Type	Method
Acid Extractable	3650, 3640
Base/Neutral Extractable	3650, 3640
Carbamates	8318[1]
Chlorinated Herbicides	8151[2]
Chlorinated Hydrocarbons	3620, 3640
Haloethers	3620, 3640
Nitroaromatics & Cyclic Ketones	3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine Pesticides	3620, 3630, 3640 3660
Organophosphorus Pesticides	3620
Phenols	3630, 3640, 3650 8041[3]
Phthalate Esters	3610, 3611, 3620 3640
Polychlorinated Biphenyls	3620, 3630, 3640 3660, 3665
Polychlorinated Dibenzo-*p*-Dioxins and Polychlorinated Dibenzofurans	8280[4] 8290[4]
Polynuclear Aromatic Hydrocarbons	3610, 3611 3630, 3640, 3650

[1] Method 8318 contains the extraction, cleanup, and determinative procedures for these analytes.
[2] Method 8151 contains the extraction, cleanup, and determinative procedures for these analytes.
[3] Method 8041 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered using GC/FID.
[4] Methods 8280 and 8290 contain the extraction, cleanup, and determinative procedures for these analytes.

TABLE 2-43

DETERMINATIVE METHODS ORGANIC ANALYTES

Analyte Type	GC/MS Method	Specific GC Method[6]	HPLC Method
Acid Extractable	8270	8410[6]	
Acrolein, Acrylonitrile, Acetonitrile	8260, 8261	8015, 8031, 8033[1]	8315[2], 8316
Acrylamide	8260	8032	8316
Aniline and Selected Derivatives	8270	8131	
Aromatic Volatiles	8260, 8261	8021	
Base/Neutral Extractable	8270	8410[6]	8325[4]
Carbamates			8318, 8321
Chlorinated Herbicides	8270[3]	8151	8321
Chlorinated Hydrocarbons	8270	8121	
Diesel Range Organics (DRO)		8015, 8440[7]	
Dyes			8321
Explosives		8095	8330, 8331, 8332
Formaldehyde			8315
Gasoline Range Organics (GRO)		8015	
Haloethers	8270	8111	
Halogenated Volatiles	8260, 8261	8011, 8021	
Nitroaromatics and Cyclic Ketones	8270	8091	8330[5]
Nitrosoamines	8270	8070	
Non-halogenated Volatiles	8260	8015	8315
Organochlorine Pesticides	8270[3]	8081, 8085[6]	
Organophosphorus Pesticides	8270[3]	8141, 8085[6]	8321
Phenols	8270	8041, 8410[6]	
Phthalate Esters	8270	8061, 8410[6]	
Polychlorinated Biphenyls	8270[3]	8082	
PCDDs and PCDFs	8280, 8290		
Polynuclear Aromatic Hydrocarbons	8270	8100, 8410[6]	8310
Volatile Organics	8260, 8261	8011, 8015, 8021, 8031, 8032, 8033	8315, 8316

[1] Of these analytes, Method 8033 is for acetonitrile only.
[2] Of these analytes, Method 8315 is for acrolein only.
[3] This method is an alternative confirmation method, not the method of choice.
[4] Benzidines and related compounds.
[5] Nitroaromatics (see "Explosives").
[6] Includes GC/ES methods, e.g., Methods 8085 and 8410.
[7] FT-IR determinative method only. Does not use GC.

TABLE 2-44

PREPARATION METHODS FOR INORGANIC ANALYSES [1]

Matrix	Method
Surface water	3005, 3010, 3015, 3020
Groundwater	3005, 3010, 3015, 3020
Extracts	3010, 3015, 3020
Aqueous samples containing suspended solids	3010, 3015, 3020
Oils	3031, 3040, 3051, 3052[2]
Oil sludges	3031, 3052[2]
Tars	3031, 3052[2]
Waxes	3031, 3040, 3052[2]
Paints	3031, 3052[2]
Paint sludges	3031, 3052[2]
Petroleum products	3031, 3040, 3052[2]
Sediments	3050, 3051, 3052[2], 3060[3]
Sludges	3050, 3051, 3052[2], 3060[3]
Soil samples	3050, 3051, 3052[2], 3060[3]
Ashes	3052[2]
Biological tissues	3052[2]

[1]It is the responsibility of the analyst to refer to each analytical method to determine applicability of the chosen method to a specific waste type and target analyte.

[2]For total decomposition analysis ONLY.

[3]For the analysis of samples for hexavalent chromium ONLY.

TABLE 2-45

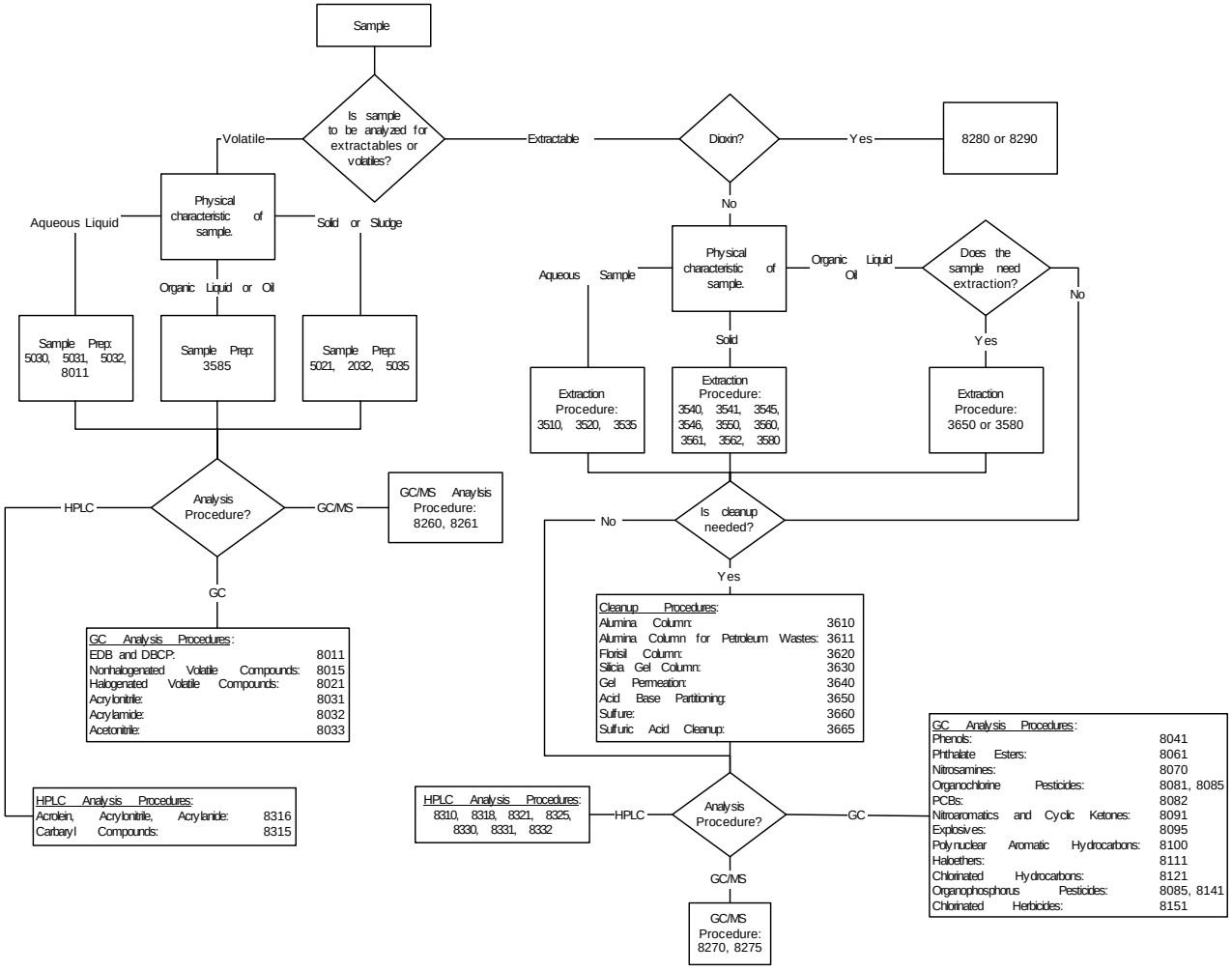
USE OF LEACHING, EXTRACTION AND DIGESTION METHODS
FOR INORGANIC ANALYSIS (In order of increasing strength)

Method	Reagents & Conditions	Use
1310	Dilute acetic acid	Simulate leaching that would result from codisposal of a solid waste and municipal waste in a sanitary landfill[1]
1311	Extraction Fluid # 1 -- Dilute glacial acetic acid and NaOH, pH 4.93 ± 0.05 Extraction Fluid # 2 -- Dilute glacial acetic acid, pH 2.88 ± 0.05	Simulate leaching that would result from codisposal of a solid waste and municipal waste in a sanitary landfill[1]
1312	Dilute H_2SO_4 and HNO_3 (synthetic acid rain)	Simulate acid rain leaching of a waste
1320	Dilute H_2SO_4 and HNO_3 (synthetic acid rain)	Simulate long-term acid rain leaching of a waste
3005	HNO_3, heat	Surface water and groundwater
3010	HNO_3, HCl, heat	Aqueous samples and extracts
3015	HNO_3 or alternatively HNO_3 and HCl, (pressure, heat)	Aqueous samples and extracts
3020	HNO_3, heat	Aqueous samples and extracts for GFAA work only
3031	Potassium permanganate, H_2SO_4, HNO_3, HCl, heat	Oils, oily sludges, tars, waxes, paint, paint sludge, and other viscous petroleum products
3040	Solvent (e.g., xylene, kerosene, or MIBK)	Dissolution of oils, oily wastes, greases and waxes
3050	HNO_3 and H_2O_2, heat (for GFAA or ICPMS) HNO_3, H_2O_2, and HCl, heat (for ICP-AES or FLAA)	Sediments, soils, and sludges
3051	HNO_3, or alternatively HNO_3 and HCl, microwave assisted (pressure, heat)	Sludges, sediments, soils and oils
3052	HNO_3, HF, HCl (optional) H_2O_2 (optional), heat, pressure	Siliceous, organic and other complex matrices for total sample decomposition
3060A	Na_2CO_3/NaOH, heat	Soils, sludges, sediments and some industrial wastes for the analysis of hexavalent chromium only.

[1] As described in the respective background documents developed in support of the rulemakings which added required use of these methods to the Toxicity Characteristic regulation (Method 1311 replaced Method 1310 for Toxicity Characteristic determinations on March 29, 1990, 55 FR 11862).

FIGURE 2-1

ORGANIC ANALYSIS OPTIONS FOR SOLID AND LIQUID MATRICES



For illustrative purposes only. See the disclaimer and Sec. 2.1 for information on the flexibility inherent in SW-846 methods.

FIGURE 2-2
SCHEMATIC OF SEQUENCE TO DETERMINE
IF A WASTE IS HAZARDOUS BY CHARACTERISTIC

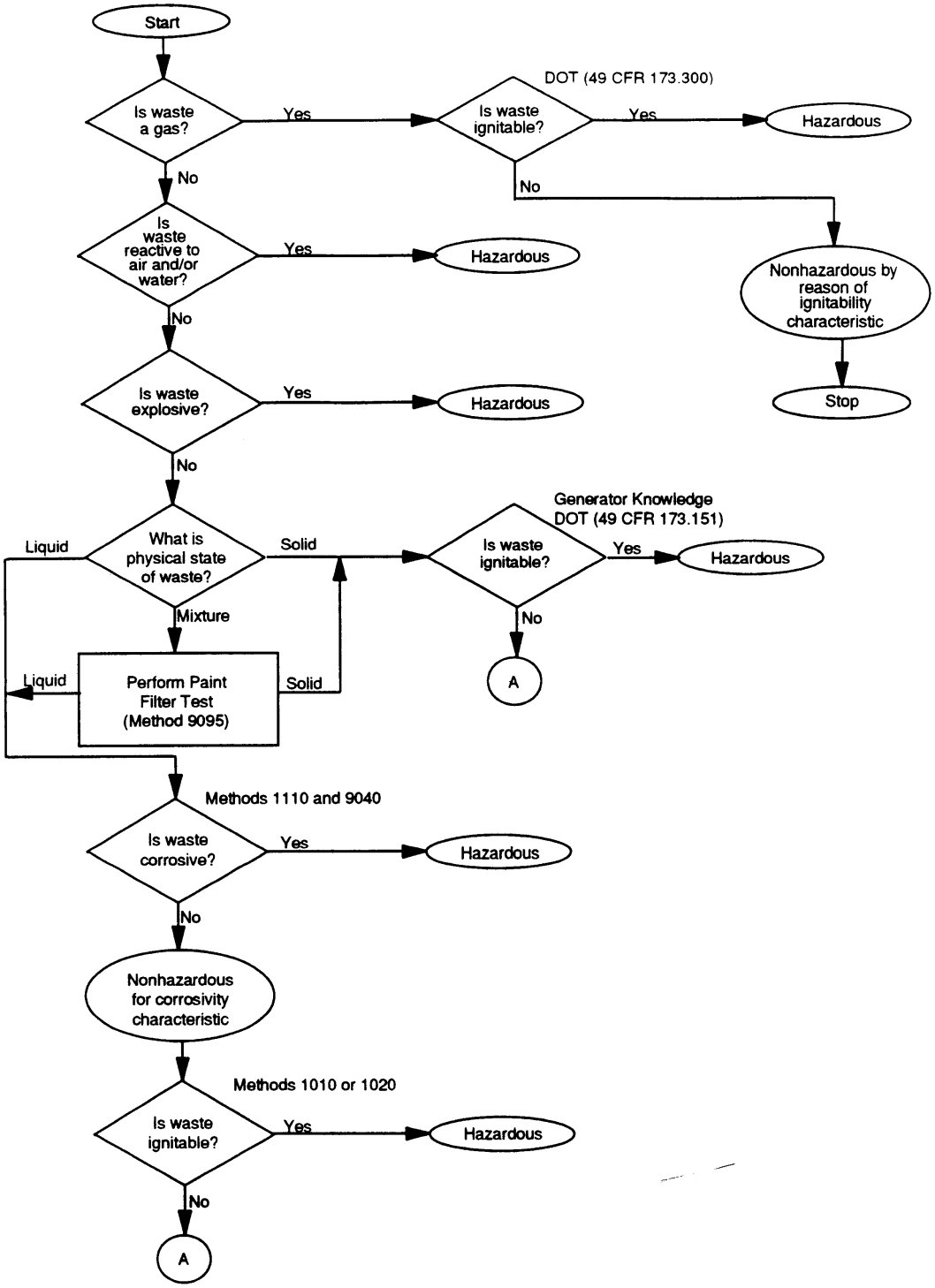


FIGURE 2-2
(continued)

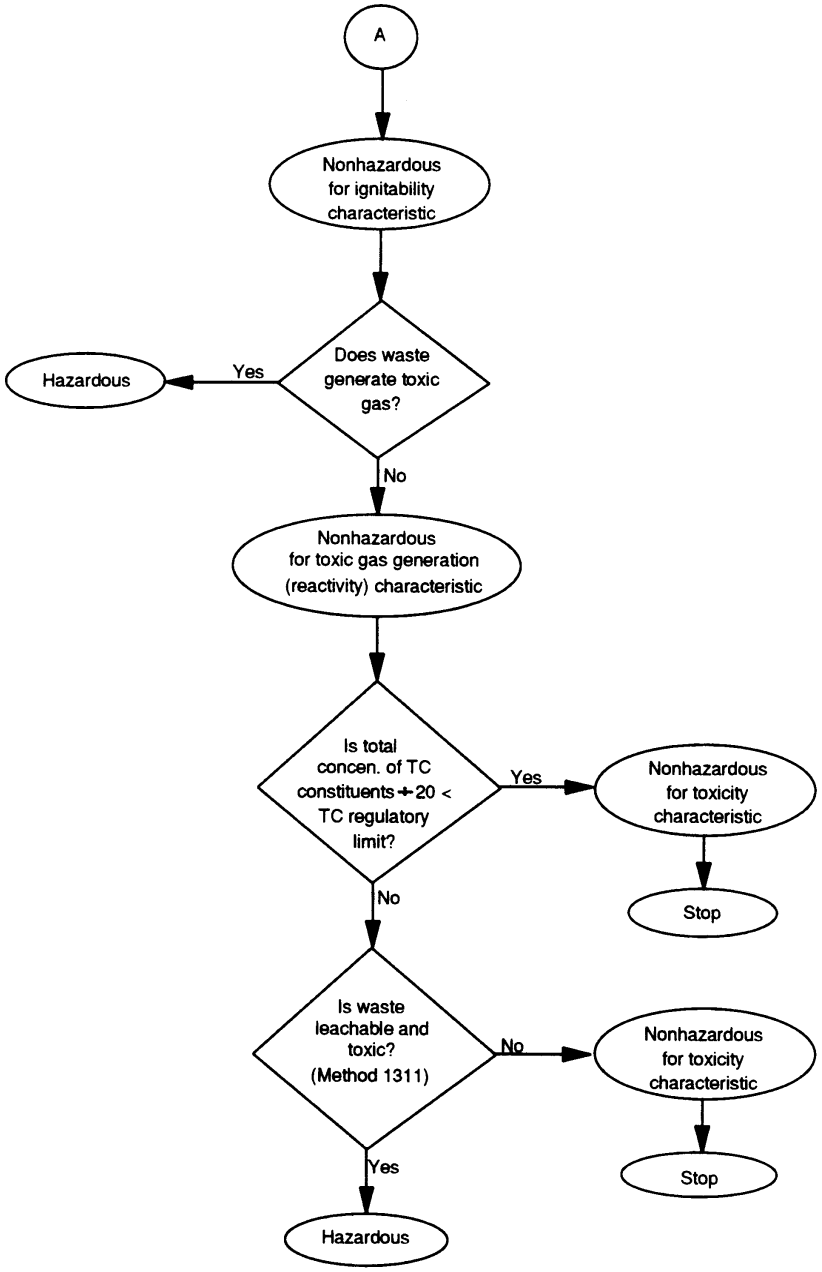


FIGURE 2-3A
RECOMMENDED SW-846 METHODS FOR ANALYSIS OF EP LEACHATES

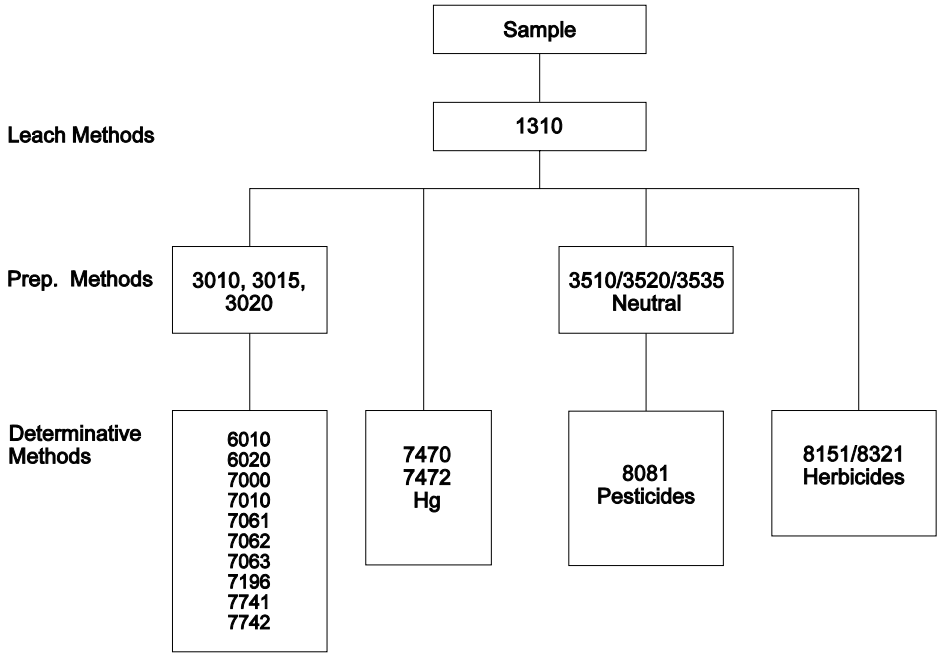


FIGURE 2-3B

RECOMMENDED SW-846 METHODS FOR ANALYSIS OF TCLP LEACHATES

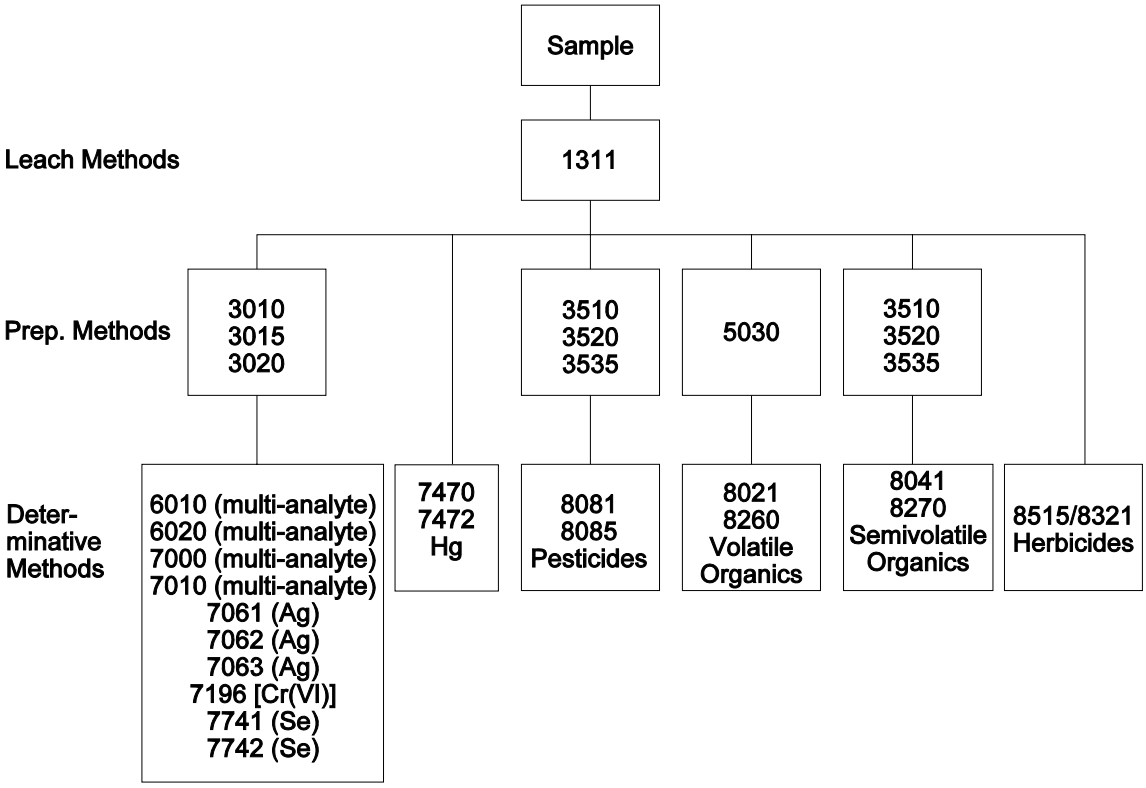
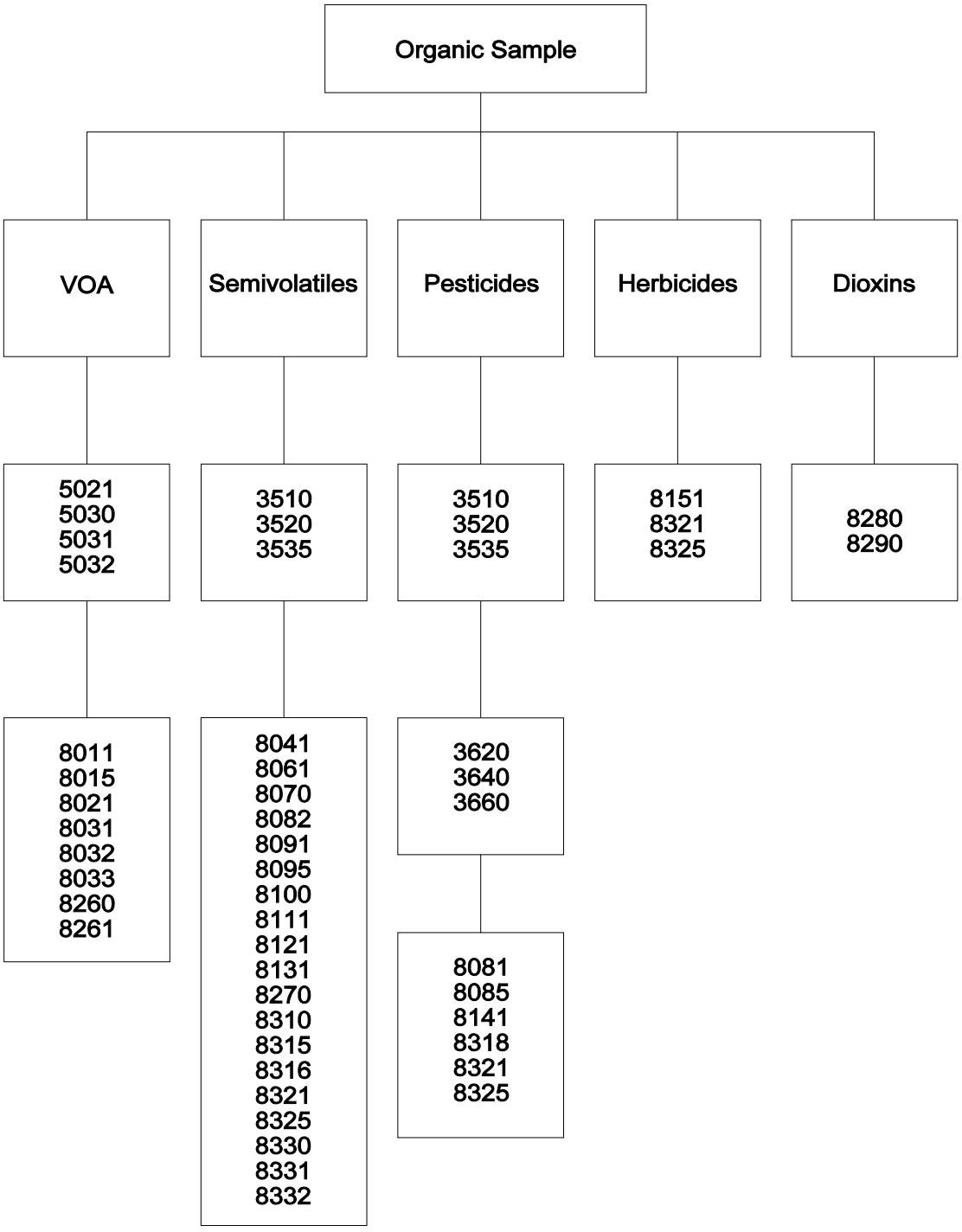
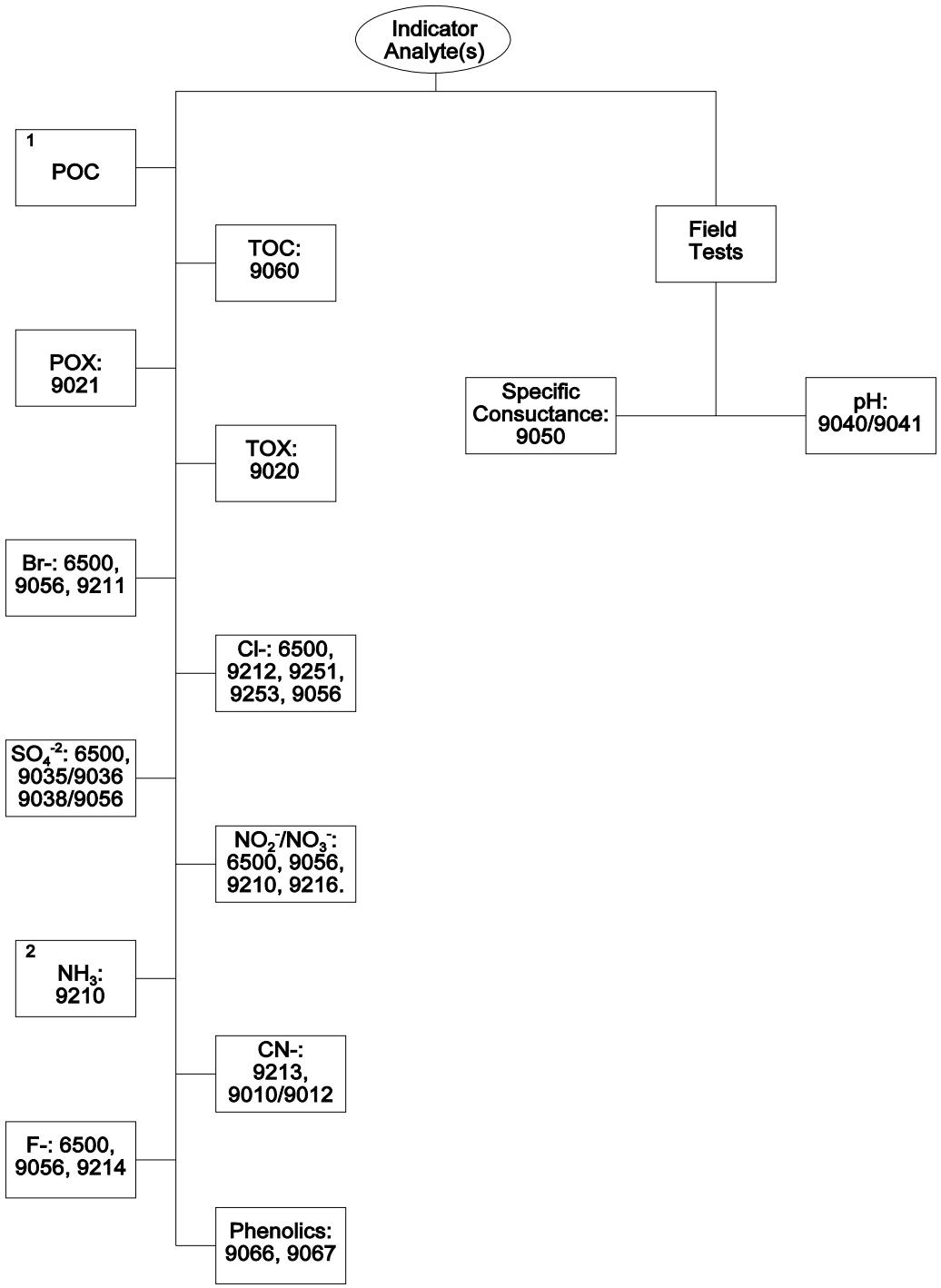


FIGURE 2-4A
GROUNDWATER ANALYSIS - ORGANIC ANALYTES



For illustrative purposes only. See the disclaimer and Sec. 2.1 for information on the flexibility inherent in SW-846 methods.

FIGURE 2-4B
GROUNDWATER ANALYSIS - INDICATOR ANALYTES

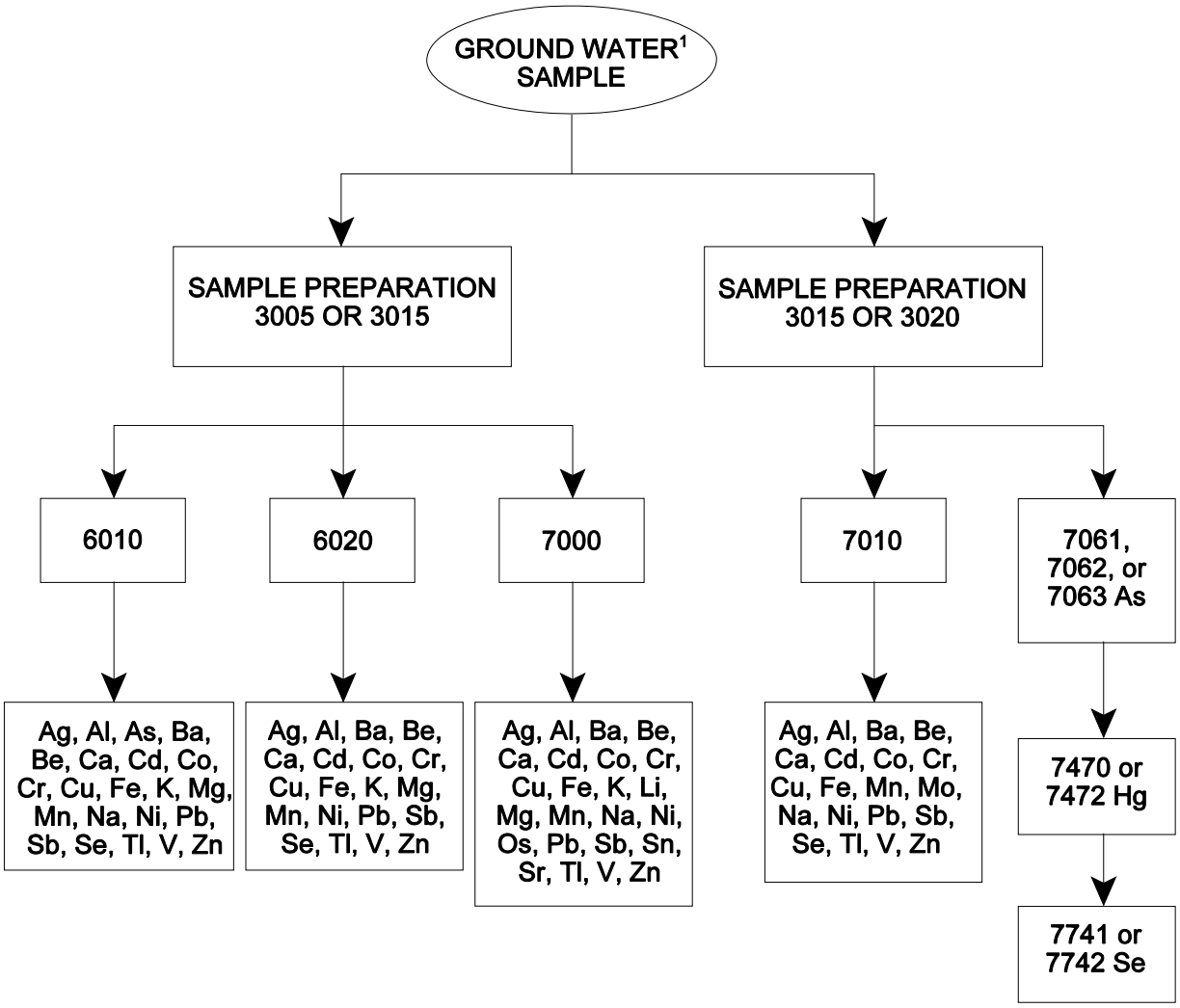


1- Barcelona 1984, (See Reference 1)
2- Riggin, 1984, (See Reference 2)

For illustrative purposes only. See the disclaimer and Sec. 2.1 regarding the flexibility inherent in SW-846 methods.

FIGURE 2-4C

GROUNDWATER ANALYSIS - INORGANIC ANALYTES



1. When analyzing for total dissolved metals, digestion is not necessary if the samples are filtered to the same concentration as the standards.

For illustrative purposes only. See the disclaimer and Sec. 2.1 regarding the flexibility inherent in SW-846 methods.

CHAPTER THREE

INORGANIC ANALYTES

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. 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 judgments necessary to meet the data quality objectives or needs for the intended use of the data.

3.1 INTRODUCTION

This chapter provides guidance for the analysis of inorganic analytes in a variety of matrices. The analytical methods are written as specific steps in the overall analysis scheme -- sample handling and preservation, sample digestion or preparation, and sample analysis for specific inorganic components. From these methods, the analyst should assemble a total analytical protocol which is appropriate for the sample to be analyzed and for the information required. This introduction discusses the options available in general terms, provides background information on the analytical techniques, and highlights some of the considerations to be made when selecting a total analysis protocol.

3.2 DEFINITIONS

The following terms are relevant for the determination of inorganic analytes:

Calibration blank: A volume of reagent water prepared with the same amounts of acids or other reagents as were the standards and samples.

Calibration curve: The functional relationship between analytical response and target analyte concentration determined for a series of calibration standards. The calibration curve is obtained by plotting the analytical response versus concentration and performing a regression analysis of the data.

Calibration standards: A series of solutions containing the target analyte at known and varying concentrations used by the analyst for instrument calibration (i.e., preparation of the calibration curve).

Continuing calibration verification (CCV): A solution containing a known concentration of analyte derived from the same source as the calibration standards. The CCV is used to assure calibration accuracy during each analysis run. It should be run for each analyte as described in the particular analytical method. At a minimum, it should be analyzed at the beginning of the run and after the last analytical sample. The CCV concentration should be at or near the mid-range levels of the calibration curve.

Dissolved metals: The concentration of metals determined in an aqueous sample after the sample is filtered through a 0.45-µm filter (see Method 3005).

Initial calibration verification (ICV) standard: A certified or independently-prepared solution from a source other than used for the calibration standards and used to

verify the accuracy of the initial calibration. For ICP analysis, it should be run at each wavelength used in the analysis.

Instrument detection limit (IDL): Typically used in metals analysis to evaluate the instrument noise level and response changes over time for analytes of interest. IDLs can be estimated by calculating the average of the standard deviations of three analytical runs performed on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once every three months or at a project-specific designated frequency and the associated documentation kept with the instrument log book.

Interference check sample (ICS): A solution containing both interfering and analyte elements of known concentration that can be used in metals ICP and ICP-MS analysis to verify background and inter-element correction factors.

Laboratory control sample (LCS): A volume of reagent water spiked with known concentrations of analytes and carried through the same preparation and analysis procedure as a sample. It is used to monitor analyte loss/recovery. The LCS may either be prepared from the same source as the calibration standards or independently of the calibration standards. An independently prepared LCS may either be obtained as or prepared from a certified reference solution or prepared from a certified reagent solid or from an alternate lot reagent solid relative to the calibration standards source. For each analytical batch, at least one LCS should be prepared from the same source as the calibration standards. In this way, if the recoveries of both the LCS and the matrix spike are outside the acceptance limits, the analyst will be able to determine whether the problem is due to a calibration error or a matrix interference.

Linear dynamic range: In either ICP-AES and ICP-MS analysis based on a one-point calibration, the concentration range above the highest calibration point over which the functional relationship between analyte signal and analyte concentration remains linear. A sample result that falls within the linear dynamic range is considered valid and may be reported, thus avoiding the need to dilute and reanalyze the sample.

Method blank: A volume of reagent water processed through each sample preparation procedure. Analysis of a method blank is used to assess contamination from the laboratory environment, sample processing equipment, and/or reagents.

Lower limit of quantitation (LLOQ): The lowest point of quantitation, or in most cases, the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels based on the stated project requirements. Analysis of a standard prepared at the LLOQ concentration level or use of the LLOQ as the lowest point calibration standard provides confirmation of the established quantitation sensitivity of the method. The LLOQ recovery should be within 50% of the true value, or some other mutually agreed upon recovery based upon the project-specific data quality objectives, in order to verify the data reporting limit.
.
Method of standard addition (MSA): An alternative calibration procedure employed when the signal response of the analyte of interest is different in a particular matrix

than when it is in reagent water. The procedure is generally reserved for analyzing complex matrices. The standard addition technique involves the addition of known amounts of the target analyte to each of a series of replicate sample aliquots. The final concentrations of the sample replicates should span the calibration range of the method. The analytical responses versus the standard addition concentration for each of the replicates is plotted. After performing a linear regression, the curve is extrapolated to the x-axis. The analyte concentration in the original unspiked sample is equal to the inverse of the x-intercept. See Method 7000, for more information.

Optimum concentration range: In metals analysis, a concentration range, below which scale expansion should be used, and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating conditions employed.

Sample holding time: The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed. Different times may be specified for holding field samples prior to extraction, digestion, or other such preparation procedures versus holding prepared samples (e.g. an extract or a solution resulting from a sample digestion) prior to analysis.

Sensitivity: The ability of an analytical technique or instrument to discriminate between small differences in analyte concentration (Reference 1). For metals analysis, the following methods are commonly employed to determine sensitivity.

(a) Atomic absorption (AA): The concentration of metal, in mg/L, that produces a transmission of 1%.

(b) Graphite furnace AA (GFAA): The mass of analyte required to give a response of 0.044 absorbance-seconds.

(c) Inductively coupled plasma (ICP): The average of the standard deviations of three runs of a reagent blank solution on three non-consecutive days with seven consecutive measurements per day.

Suspended metals: The concentration of metals determined in the portion of an aqueous sample that is retained by a 0.45-µm filter (Method 3005).

Total acid soluble/recoverable metals: The concentration of metals determined in an unfiltered sample following digestion using hot mineral acid by Methods 3005, 3010, 3015, 3020, 3050, or 3051.

Total metals: The concentration of metals determined in a sample following digestion by Method 3052.

3.3 SAFETY

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

The toxicity or carcinogenicity of each reagent used in these methods has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level by whatever means available. The following additional references to laboratory safety are available:

1. "Carcinogens - Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

2. "Handbook of Chemical Health and Safety," American Chemical Society, Oxford University Press, New York, 2001.

3. "NIOSH Pocket Guide to Chemical Hazards," Department of Health and Human Services, Centers for Disease Control, National Institute for Occupational Safety and Health, Publication No. 2005-149, September 2005.

4. "Occupational Safety and Health Standards," 29 CFR Part 1910, Occupational Safety and Health Administration, Department of Labor.

5. "Safety in Academic Chemistry Laboratories," 7th Edition, Volumes 1 and 2, American Chemical Society, Committee on Chemical Safety, Washington, D.C., 2003.

3.4 SAMPLING CONSIDERATIONS

3.4.1 Sample Collection

The fundamental goal of all field sampling activities is to collect samples that are representative of the water, soil or waste from which they were collected. Thus, representative sampling may be considered to be the sampling analog to analytical accuracy. Of equal importance is sampling precision for ensuring consistency both within a single sampling event and between sampling events conducted over time. Sampling imprecision can rival analytical imprecision as a source of measurement error. High quality field practices are, therefore, necessary for generating representative samples on a consistent basis. Sampling quality assurance includes the development of a quality assurance plan, data quality objectives and the generation of field quality control samples including equipment rinsates, trip blanks and field duplicates. Regardless of the specific program needs, the documentation of all relevant field and sample information is the final essential component of a sampling event for providing evidence that proper procedures and quality assurance were performed during sample collection. Use of inadequate field procedures and documentation can jeopardize an entire sampling program despite adequate planning, analytical facilities, and personnel.

While advances in analytical sensitivity are continuing to be made that allow for quantification of environmental contaminants at ultra-trace levels (i.e., < 0.1 ppb), clean sampling techniques are consequently being devised and practiced in order to minimize or eliminate sources of contamination during the collection of samples intended for ultra-trace contaminant testing. Such clean sampling and analysis techniques are not generally needed or required under the RCRA program and are beyond the scope of this chapter. However, as an introduction to this topic, Sec. 3.5 provides a more detailed discussion on the special category and requirements of clean analysis for determining constituents at ultra-trace levels.

3.4.2 Sample Containers

Sample container materials can introduce either positive or negative errors in measurement, particularly at low or ultra-trace levels, by contributing contaminants through leaching or surface desorption, or by depleting concentrations through adsorption. Additionally, the sample containers should be compatible with the reagents used for sample preservation. Thus, the collection and containment of the sample prior to analysis requires particular attention. Sample contamination introduced through field collection activities including sample containment and shipment can be assessed from the analysis of equipment rinsates and trip blanks. Guidelines on the selection of appropriate sample container materials for the collection of inorganic analytical samples are provided in Table 3-1.

3.4.3 Cleaning of Sample Containers

Sample containers should be scrupulously clean so as not to introduce contaminants that could interfere with quantification of the target analyte(s). This is of particular importance when determining trace or ultra-trace analyte concentration levels. The following cleaning sequence has been determined to be adequate to minimize contamination in the sample bottle, whether borosilicate glass, linear polyethylene, polypropylene, or PTFE:

- Detergent
- Tap water
- 1:1 HNO_3
- Tap water
- 1:1 HCl
- Tap water
- Reagent water

NOTE: **Chromic acid should not be used to clean glassware**, especially if chromium is to be included in the analytical scheme. Commercial, non-chromate products (e.g., Nochromix) may be used in place of chromic acid, if adequate cleaning is documented by an analytical quality control program. Chromic acid should also not be used with plastic bottles.

3.4.4 Sample Handling and Preservation

Sample holding times, recommended collection volumes or masses and recommended digestion volumes, and preservatives are listed in Table 3-1. The sample collection and digestion amounts depend on the combination of digestion or extraction and determinative procedures that will be employed for a given sample as well as the sensitivity that is required for a specific project. Likewise, the use of alternative preservatives to those indicated in Table 3-2 may be necessary depending on the objectives of the project. In all cases, the sample quantity that is collected should be representative of the bulk material whenever feasible.

3.4.5 Sample Preparation

For all non-speciated digestion methods, great reduction in analytical variability can be achieved through the use of appropriate sample preparation procedures. Generally, a reduction in subsampling variance can be accomplished by reducing the sample particle size, and homogeneously mixing the resulting fines. Under most circumstances, it is

recommended that the sample be analyzed without drying. If it is necessary to report the analytical data on a dry-weight basis, then a separate aliquot may be analyzed for moisture content and the wet-weight data corrected accordingly.

If the sample cannot be well-mixed and homogenized in the form in which it was received by the laboratory, then air- or oven-drying at 30 °C or less, crushing, sieving, grinding, and mixing should be performed as needed or feasible to homogenize the sample until the subsampling variance is less than the data quality objectives of the analysis. While proper sample preparation generally produces great reduction in analytical variability, it should be noted that in certain unusual circumstances there could be loss of volatile metals (e.g., Hg, organometallics) or irreversible chemical changes (e.g., precipitation of insoluble species, change in valence state) caused by inappropriate sample preparation procedures.

Variability due to sample heterogeneity is assessed by analyzing individually prepared sample replicates. Variability inherent in the analytical determinative procedure is assessed by matrix spiking of individually digested samples.

TABLE 3-1

MATERIALS FOR USE IN SAMPLE COLLECTION FOR
INORGANIC ANALYTE DETERMINATIONS

Analyte	Recommended Container Material
Metals	PTFE, plastic, glass
Chloride	PTFE, plastic, glass
Cyanide	PTFE, plastic
Fluoride	PTFE, plastic
Nitrate	PTFE, plastic, glass
pH	PTFE, plastic, glass
Specific Conductance	PTFE, plastic, glass
Sulfate	PTFE, plastic, glass
Sulfide	PTFE, plastic, glass

[a]These recommendations are intended as guidance only. The selection of sample container should be made based on the nature of the sample, the intended end use of the data and the project data quality objectives.

TABLE 3-2

RECOMMENDED SAMPLE HOLDING TIMES, PRESERVATION, COLLECTION QUANTITIES, AND DIGESTION VOLUMES FOR SELECTED INORGANIC ANALYTE DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES [a,b]

Analyte	Matrix	Fraction	Minimum Collection Volume/Mass	Preservation [1]	Digestion Volume	Holding Time [2]
Metals (except Hg and Cr^{6+})	Aqueous	Total	600 mL	HNO_3 to pH<2	100 mL	6 months
		Dissolved	600 mL	Filter on site; HNO_3 to pH<2	100 mL	6 months
		Suspended	600 mL	Filter on site;	100 mL	6 months
	Solid	Total	200 g	None	2 g	6 months
Hexavalent chromium	Aqueous		400 mL	≤6 °C	100 mL	24 hours
	Solid		100 g	≤6 °C		30 days to extraction
				≤6 °C	2.5 g	7 days from extraction to analysis
Mercury	Aqueous	Total	400 mL	HNO_3 to pH<2	100 mL	28 days
		Dissolved	400 mL	Filter; HNO_3 to pH<2	100 mL	28 days
	Solid	Total	200 g	≤6 °C	0.2 g	28 days
Chloride	Aqueous		50 mL	≤6 °C	—	28 days
Cyanide	Aqueous		500 mL	≤6 °C; NaOH to pH>12	—	14 days
	Solid		5 g	≤6 °C	—	14 days
Fluoride	Aqueous		300 mL	≤6 °C	—	28 days

TABLE 3-2

RECOMMENDED SAMPLE HOLDING TIMES, PRESERVATION, COLLECTION QUANTITIES, AND DIGESTION VOLUMES FOR SELECTED INORGANIC ANALYTE DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES [a,b]

Analyte	Matrix	Fraction	Minimum Collection Volume/Mass	Preservation [1]	Digestion Volume	Holding Time [2]
Nitrate	Aqueous		1000 mL	≤6 °C	—	28 days
Hexane Extractable Material (HEM; Oil & Grease)	Aqueous		1000 mL	≤6 °C HCl or H_2SO_4 to pH <2	—	28 days
	Solid		100 g	≤6 °C HCl or H_2SO_4 to pH <2; when practical		28 days
pH	Aqueous		25 mL	NA	—	Analyze immediately
	Solid		20 g	NA	—	Analyze immediately
Specific Conductance	Aqueous		100 mL	NA	—	Analyze immediately
Sulfate	Aqueous		50 mL	≤6 °C	—	28 days
Sulfide	Aqueous		100 mL	4 drops 2N zinc acetate/100 mL sample; NaOH to pH>9; Minimize aeration; Store headspace free at ≤6 °C	—	7 days
	Solid			Fill sample surface with 2N zinc acetate until moistened; Store headspace free at ≤6 °C	—	7 days

TABLE 3-2

RECOMMENDED SAMPLE HOLDING TIMES, PRESERVATION, COLLECTION QUANTITIES, AND DIGESTION VOLUMES FOR SELECTED INORGANIC ANALYTE DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES [a,b]

Analyte	Matrix	Fraction	Minimum Collection Volume/Mass	Preservation [1]	Digestion Volume	Holding Time [2]
Organic Carbon, Total (TOC)	Aqueous		200 mL	≤6 °C store in dark HCl or H_2SO_4 to pH <2;	—	28 days
	Solid		100 g	≤6 °C	—	28 days

[a] These recommendations are intended as guidance only. The selection of sample and digestion volumes and preservation and holding times should be made based on the nature of the sample, the intended end use of the data and the data quality objectives.

[b] Additional sample quantities may need to be collected in order to allow for the preparation and analysis of QC samples, such as matrix spikes and duplicates.

[1] The exact sample extract, and standard storage temperature should be based on project-specific requirements and/or manufacturer's recommendations for standards. Alternative temperatures may be appropriate based on demonstrated analyte stability within a matrix, provided the data quality objectives for a specific project are still attainable.

[2] A longer holding time may be appropriate if it can be demonstrated that the reported analyte concentrations are not adversely affected by preservation, storage and analyses performed outside the recommended holding times.

3.5 SPECIAL CONSIDERATIONS FOR DETERMINING INORGANIC ANALYTES AT ULTRA-TRACE CONCENTRATION LEVELS

3.5.1 Clean Sampling Techniques

For the determination of ultra-trace analyte concentrations in environmental samples, it is essential that samples be collected and subsequently managed using techniques specifically designed to minimize sample contamination from field collection activities and to ensure target analyte stability. Such techniques represent a special category of sampling procedures designed specifically for ultra-trace analyses and are commonly referred to as clean or ultra-clean sampling procedures. Clean sampling methods are generally not intended for the determination of discharges from industrial facilities. Rather, they are primarily applicable for the determination of ambient element concentrations at levels of 0.1 ppb or less. At these concentrations, the opportunity for sample contamination during sample collection or analysis in the laboratory is significant and should be managed accordingly. Figure 3-1 provides a demonstration of the impact of clean sampling and analysis techniques on data obtained for estuarine waters. Clean sampling typically involves the following key steps:

- Special container pre-cleaning and pre-packaging requirements
- Specific sampling equipment and container materials selection
- Specific cleaning protocols for sampling equipment
- Equipment and container blank determinations prior to field use
- "Clean hands/dirty hands" sample collection techniques based on a 2-person sampling crew
    - Dirty hands sampler manages sampling equipment only
    - Clean hands sampler manages the sample container
- Special sample packaging prior to shipment
- Use of a laboratory trained and properly equipped to perform clean analysis of the analytes of interest

Given the laboratory resources required to perform clean analysis techniques, it is paramount that samples be collected using ultra-clean techniques and conditions in the field. Otherwise, subsequent analytical efforts become futile. The information provided in this section is intended only as an introduction to the topic of clean sampling. Specific guidelines for clean sampling may be found in Reference 2 and other sources.

3.5.2 Clean Analysis and the Analytical Blank

The significant role of the analytical blank in chemical analysis of trace metals cannot be overemphasized. Sensitive instrumentation such as ICP-MS, ICP-AES, and GFAA requires that sample preparation be at least as sophisticated as the instruments used for analysis. The analytical blank is normally a primary source of error in ultra-trace element analysis. Ultra-trace analysis is as dependent on control of the analytical blank as it is on the accuracy and precision of the instrument making the measurement. Inability to control contamination, is frequently the limiting factor in trace (parts per million (ppm) to parts per billion (ppb)) and ultra-trace (ppb to parts per trillion (ppt)) analysis. Analytical blank contributions occur from the following four major sources (References 3 through 7):

- The atmosphere in which the sample preparation and analysis are conducted
- The purity of the reagents used in sample preparation, including all reagents and the quantities added directly to the sample
- The materials and equipment used in digestion or extraction vessels that come in contact with the sample during the sample preparation and analysis

- The analyst's technique and skill in preparing the samples and performing the analyses

The four primary areas that affect the analytical blank can be demonstrated using standard reference materials in analysis. Table 3-3 illustrates and isolates the main blank influencing parameters: environment, reagents, materials, and analyst skills. The skill of the analyst was kept constant as the same analyst changed the environment, reagents, and combinations of these parameters in the analysis (see Reference 6). The trace elements in glass (TEG) standard reference material from the National Institute of Standards and Technology (NIST) was used to keep sample homogeneity constant and to permit removal of the sampling error by using sample sizes in which appropriate homogeneity had previously been demonstrated.

It is important to note that the relationship of the precision and measurement remained relatively constant. This relationship yields no information about the accuracy of the data. The significance of the first two major sources of contamination, environment and reagents, can be evaluated. In the example above, the contamination in the laboratory air and in the acid used for the reagent blank altered the accuracy of the example above by over two orders of magnitude for both lead and silver. The larger influence of the two sources in this example is the laboratory environment in which the samples were prepared.

3.5.2.1 Sample Preparation and Analysis Atmosphere

The atmosphere in which the sample is prepared is a major source of contamination for most target analytes when analyzing at ultra-trace levels. With the exception of some rare constituents, contamination from airborne sources represents the most significant of the four main contamination sources. To illustrate this point, Table 3-4 presents concentrations of lead found in samples of ambient air.

This contamination can also be seen in the comparison of 58,000 particles per liter of air measured in a normal laboratory in Pittsburgh, PA, and inside a clean chamber in an adjacent laboratory five meters away. Figure 3-2 demonstrates the dramatic difference between the two environments. Cost-effective methods of creating clean chambers for sample preparation are documented along with this data in Reference 4.

Any laboratory air that comes into contact with the sample may deposit some portion of its concentration into the sample. The sample is especially vulnerable to this transfer when it is being decomposed in acid. The acid will leach particles from the air, resulting in unwanted ions in solution, mixing with those of the sample.

To prevent air from contaminating a sample for ultra-trace analysis, the sample should be processed in a clean environment. This is much easier to accomplish than it may appear at first. These precautions are becoming state-of-the-art in many analytical and environmental laboratories. The prevention of airborne contamination is most frequently dealt with by employing a laminar flow clean bench or a clean laboratory facility. Instructions are referenced for the construction of both from component parts; both are relatively inexpensive and uncomplicated, once the concepts are understood (Reference 4).

There are many sources of airborne contamination. Several of the sources have been described and their particle size ranges are provided in Figure 3-3. These sources primarily provide particulates in discrete size ranges. Depending on whether the laboratory is located in an industrial, urban, or rural area, or near the sea, the distribution of these source particles will be different, as will their composition. The vertical dashed line in Figure 3 indicates the particle size cutoff, usually 0.5 µm, for the high efficiency particulate air (HEPA) filter used to prevent particulate contamination. Particles above this size cannot pass through a HEPA filter that is in good working order. These filters are in common use today (References 4 and 8.

The definition of clean air is derived from Federal Standard 209a, which defines cleanliness levels. Table 3-5 lists these conditions. "Laminar flow" is directed coherent air movement that does not contain any turbulence.

A dramatic reduction in airborne contaminants can be achieved by using HEPA-filtered air in laminar-flow clean hoods or entire clean laboratories. Table 3-6 demonstrates the dramatic differences in airborne contaminant concentrations in an ordinary laboratory, a clean laboratory, and a clean hood inside a clean laboratory.

3.5.2.2 Reagent Purity for Ultra-trace Analysis

The purity of the reagents used for acid decomposition, leaching, and extraction is extremely important to the overall level of the blank. Reagents have very different purities, depending on their processing grade and purpose. Frequently, the analyst should purchase special reagents, or purify lesser-grade reagents prior to use, in order to minimize the analytical blank.

In addition to the purity of the reagents, the reagent quantity that is added to the sample is also significant. When reagents are added, they bring with them elemental and molecular components that exist as contaminants. The more reagent that is used in excess of the stoichiometric reaction, the greater the potential for blank contamination. Reagents of high purity should either be purchased or produced in the laboratory.

In the preparation of high purity reagents, there is only one significant and practical choice for the method of purification, i.e., sub-boiling distillation (References 9 through 11). Different from normal distillation, sub-boiling distillation uses an infrared radiation source to heat the reagent to a temperature just below the boiling point. This use prevents the formation of bubbles that rise and burst at the surface of the liquid. Thus, the aerosolized solution particles are left in solution and prevented from physically transporting contaminants throughout the distillation apparatus. Sub-boiling distillation is a slower but very reliable method of purifying all of the common mineral acids and many organic reagents used in analytical methods. It relies exclusively on the vapor pressure of the reagent, and contaminant, and can therefore be specifically optimized for purification of the mineral acids if the object is to remove metal ions. Of all acids, nitric acid, for a variety of reasons, can be purified to excellent quality. Sources for sub-boiling apparatus equipment and methods for constructing one are provided in the references. Purchasing sub-boiling acids from commercial sources is also an option. Construction or purchase of sub-boiling reagent purification equipment may be cost effective for some laboratories depending on the quantity of reagents required for sample throughput.

3.5.2.3 Materials for Sample Preparation, Storage, and Analysis

For ultra-trace analyses, only certain materials are preferred for use in the construction of sample vessels and instrument components that come into contact with the sample. Over the past two decades, materials identified as being non-contaminating have become the top choices for bottles, beakers, reaction vessels, storage containers, nebulizers, and instrument components for trace and ultra-trace analysis. The materials are the same as those currently being used in many digestion vessels, bomb liners, and microwave vessels. The materials are characterized by being thermally durable, chemically resistant or inert, non-contaminating, and possessing appropriate compression and tensile strength. Table 3-7 lists, in order of preference, several types of, non-contaminating materials that are chemically inert to most acid reactions. These materials have been evaluated and tested extensively for their potential to contaminate (References 4, 6, 7, 12, and 13).

With the exception of polyethylene, the materials listed in Table 3-7 are those most commonly for sample preparation vessels, both atmospheric pressure vessels and closed vessel liners, that come into contact with the sample. These materials are the most stable to acid reactions (with the exception of quartz and glass if hydrofluoric acid is used). Fluoropolymers are the most common and were adapted from other chemical uses for application in pressure systems. The fluoropolymers, TFM, PFA and TFE or PTFE have the highest range of use temperatures for most plastics, ranging from 270-300 °C. They are also chemically inert to the majority of mineral acids and combinations thereof. Sulfuric acid has a boiling point of approximately 330 °C and can damage all fluoropolymers by melting them. Quartz and glass can safely contain sulfuric acid at these high temperatures, but borosilicate glass is not appropriate for ultra-trace elemental analysis (References 7 and 13). Glass actually forms a gel layer that hydrates and leaches, transferring contaminants from the glass to the sample solution. While these quantities may be considered minute, they would be detected in blanks and samples undergoing ultra-trace analyses.

Polyethylene is suitable for storage of diluted samples after decomposition, but it does not have a thermal-use temperature appropriate for decomposition. It is also not sufficiently inert to be useful as a decomposition vessel or vessel liner, similar to polycarbonate and polypropylene. The low cost of polyethylene and its relative inertness to cool, weakly acidic solutions make it an excellent storage container for trace element solutions (Reference 4).

3.5.2.4 Analytical Technique and Synergistic Equipment

The fourth significant source of analytical blank contamination is the skill of the analyst and the appropriateness of the technique being performed. Analytical blank control has been explained as the combination of atmosphere, reagent, material, and protocol being performed. Also, the skill and awareness of the analyst as well as the way in which the combinations of the aforementioned clean chemistry techniques are applied will have a significant effect on the final contamination error and analytical blank control. Sample preparation instrumentation may also assist in these protocols. For example, microwave sample preparation assists each of these parameters in synergistic ways, thus lowering the analytical blank, improving blank precision, and enhancing overall

quality control and transferability of methods. Some instrumentation and fundamental processes involved in specific sample preparation procedures assists the analyst by incorporating useful clean chemistry concepts into instrumentation and method structure. Such instrumentation is pertinent since microwave methods now exist that provide sample preparation for leaching or total analysis of many target analytes simultaneously. As an example, the skill of the analyst with regard to clean chemistry is assisted by the method structure and microwave equipment as indicated below:

- If a closed or controlled atmospheric microwave vessel is prepared in a clean hood and sealed before leaving the clean environment, the sample will not be affected by atmospheric contamination during the reaction, since it has not been removed from a clean environment.

- The vessel materials described previously might not normally be used by many laboratories, and therefore the advantages of the fluoropolymers would not be realized if they were not required in most microwave reaction vessels as they commonly are.

- The time that the sample spends in decomposition, leaching, or extraction may be reduced from hours to minutes, thus reducing the potential leaching of contaminants from the container walls.

- Because most microwave systems are sealed systems, evaporation of the reagent before it reacts productively is prevented and smaller quantities of reagents are used, thus preventing excess and unnecessary accumulation of contaminants in the blank.

By reducing the exposure variables, the blank is consequently reduced in size and is more consistent. An example of these components working together has been provided in the literature, where analysis under different conditions has verified these conclusions (References 4, 14 and 15). The example illustrates the isolation of the blank optimization areas: environment, reagents, materials, and analysis skills. The skill of the analyst is kept more constant as the instrument dictates more clean, chemically-appropriate procedures.

3.6 REAGENT PURITY

The purity of the reagents used for sample preservation, acid decomposition, leaching, extraction and analysis is extremely important relative to preventing or minimizing sample contamination. Reagents have very different purities, depending on their processing grade and purpose. Reagent grade, ACS grade or better are recommended for use with most SW-846 methods. Sample contamination introduced through sample preservation, handling, preparation and analysis is assessed from the analysis of method blanks.

TABLE 3-3

EXAMPLES OF THE ANALYTICAL BLANK INFLUENCE ON
ULTRA-TRACE ANALYSIS OF ELEMENTS IN GLASS

Conditions	Pb (ng)	Ag (ng)
Initial analysis of TEG* standard	330 ± 250	970 ± 500
Analysis using sub-boiled distilled acids	260 ± 200	--
Analysis in a Class 100 hood	20 ± 8	207 ± 200
Analysis using sub-boiled acids in a Class 100 hood	2 ± 1	3 ± 2

* TEG = Trace element in glass

Data are taken from Reference 6.

TABLE 3-4

EXAMPLES OF LEAD CONCENTRATIONS IN AIR

Site	Lead Concentration ($\mu g/m^3$)	Source
Downtown St. Louis, MO	18.84	Reference 16
Rural park, Southeastern MO	0.77	Reference 17
NIST Laboratory, MD	0.4	Reference 6

TABLE 3-5

CLEANLINESS LEVELS IN FEDERAL STANDARD 209A[a]

Class	Maximum Contamination in Work Area (particles/ft^{-3})
100	100 particles > 0.5 μm 0 particles > 5.0 μm
10,000	10,000 particles > 0.5 μm 65 particles > 5.0 μm
100,000	100,000 particles > 0.5 μm 700 particles > 5.0 μm

[a]The Federal standard required the use of laminar-flow equipment to attain this level of cleanliness. Since measurement of dust particles smaller than 0.5 μm introduces substantial errors, 0.5 μm has been adopted as the criterion of measurement.

Data are taken from Reference 8.

TABLE 3-6

PARTICULATE CONCENTRATIONS IN LABORATORY AIR

	Concentration ($\mu g/m^3$)			
Location	Iron	Copper	Lead	Cadmium
Ordinary laboratory	0.2	0.02	0.4	0.002
Clean room	0.001	0.002	0.0002	ND
Clean hood	0.0009	0.007	0.0003	0.0002

ND = Not Detected

Data are taken from Reference 17.

TABLE 3-7

NON-CONTAMINATING MATERIALS AND FOR USE AS DECOMPOSITION VESSELS AND SAMPLE CONTAINERS IN ULTRA-TRACE ANALYSES

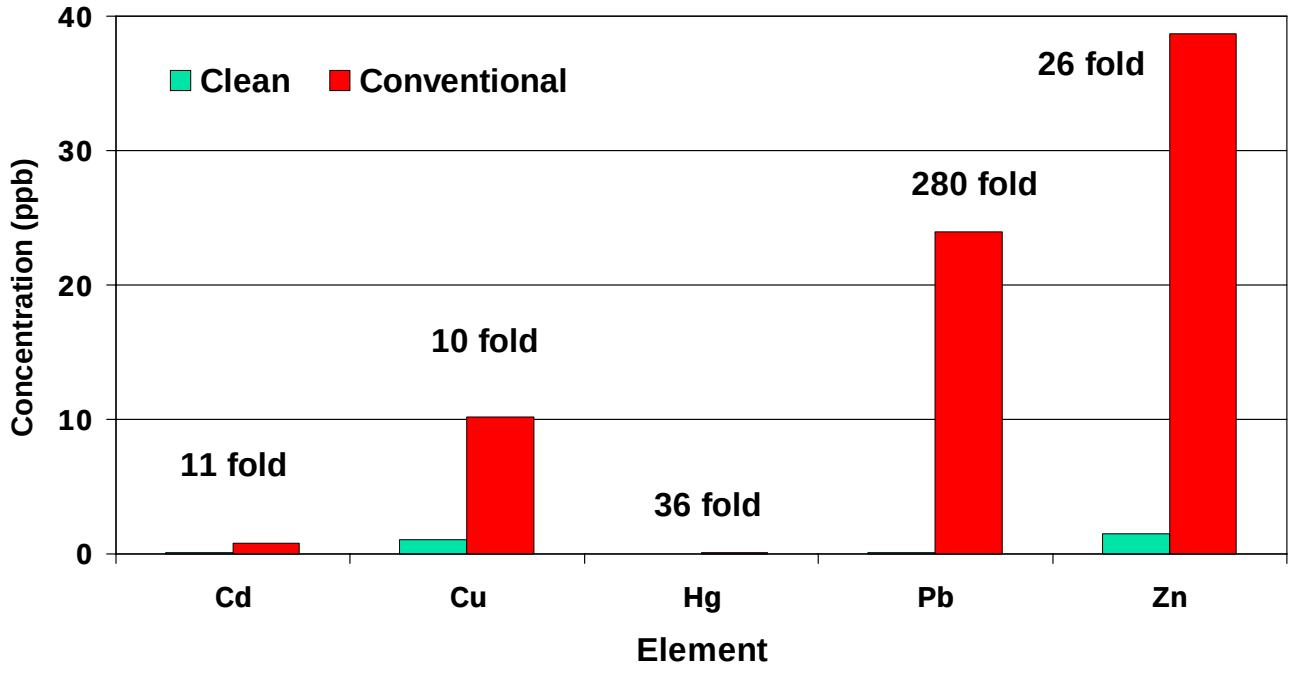
Listed from highest to lowest preference for use in sample containment
Fluoropolymers: PFA*, TFM, TFE*, FEP*, Tefzel*
Quartz - Synthetic
Polyethylene (suitable for storage only, not for acid digestion)
Quartz - Natural
Borosilicate Glass

* Various forms of PTFE

Data are taken from Reference 8.

FIGURE 3-1

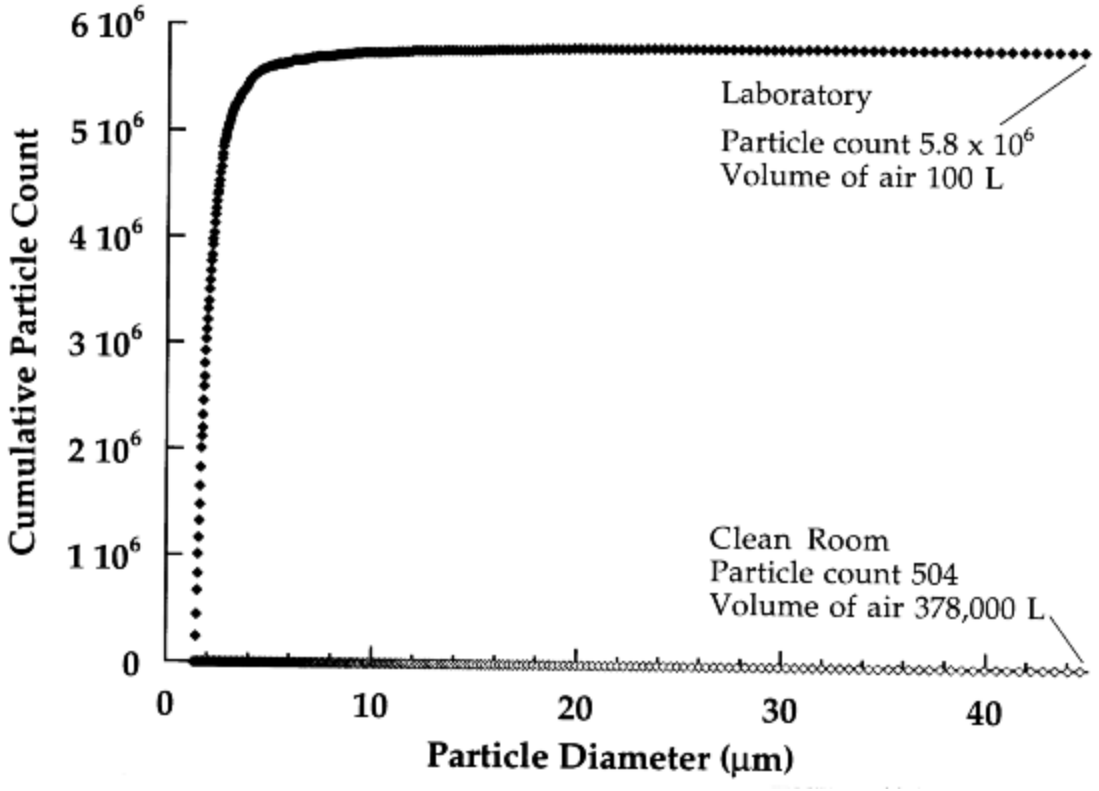
COMPARISON OF CLEAN VERSUS CONVENTIONAL SAMPLING AND ANALYSIS TECHNIQUES USED IN THE ANALYSIS OF SOUTH TEXAS ESTUARY WATERS



Taken from Reference 18.

FIGURE 3-2

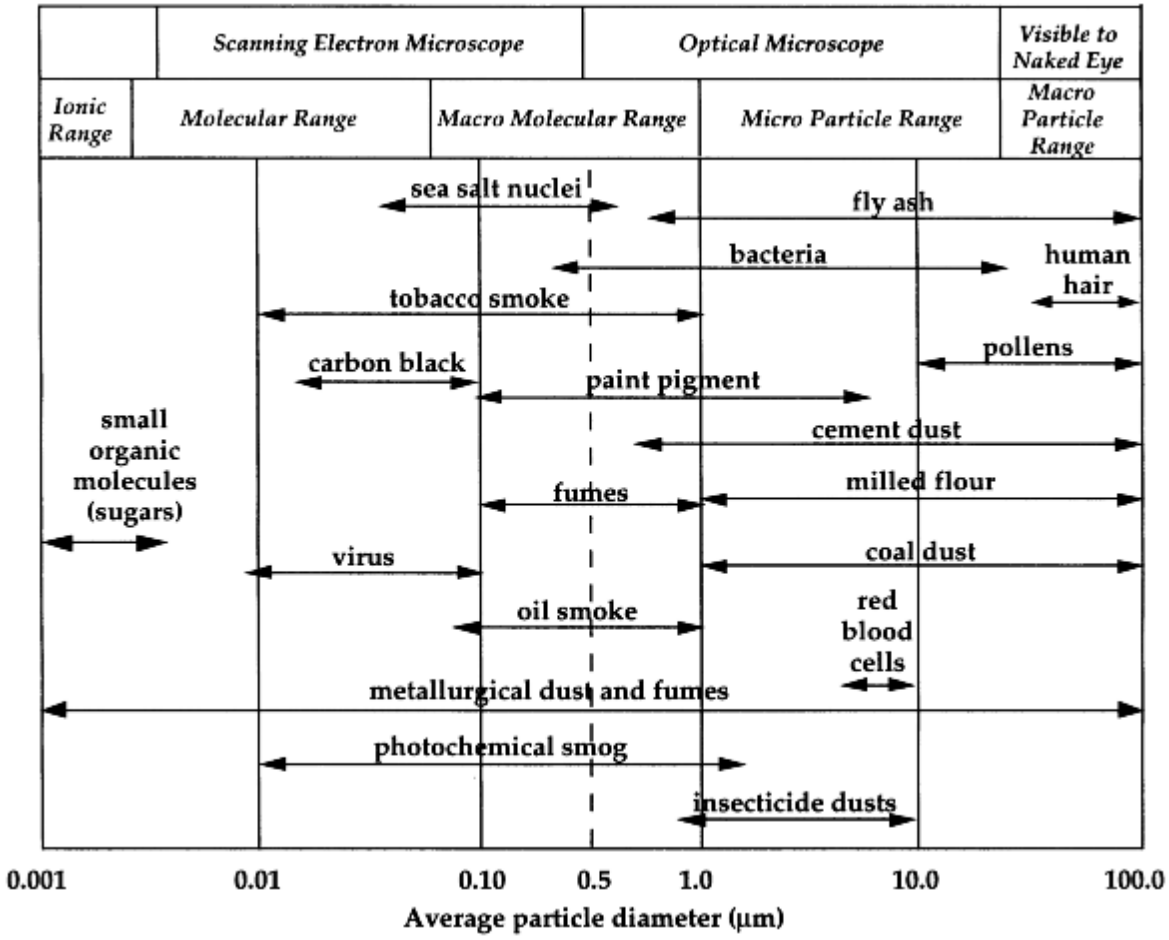
COMPARISON OF PARTICLE COUNT ANALYSIS OF A CLEAN ROOM AND
A STANDARD LABORATORY AT DUQUESNE UNIVERSITY IN PITTSBURGH, PA



Taken from Reference 4.

FIGURE 3-3

PARTICLE SIZE COMPARISON CHART FOR COMMON PARTICULATES



Taken from Reference 4, 19.

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3.8 SAMPLE DIGESTION 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. 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 judgments necessary to meet the data quality objectives or needs for the intended use of the data.

NOTE: Many of the methods listed below employ HCl in the digestion process. Chlorine is an interferant in ICP/MS analysis and its use in sample digestion is discouraged except when absolutely necessary or when the instrument manufacturer has indicated that the use of HCl will not adversely affect the equipment and accurate quantitation of the desired target analytes.

The methods in SW-846 for sample digestion or dissolution include:

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

This method may be used for the preparation of ground water and surface water samples for total recoverable and dissolved metal determinations by FLAA, ICP-AES, or ICP-MS. The unfiltered or filtered sample is heated with dilute HCl and HNO_3 prior to metal determination.

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

This method may be used for the preparation of waste samples for total recoverable metal determinations by FLAA, ICP-AES, or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid. The method is applicable to aqueous samples, leachates, and mobility-procedure extracts.

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

This method may be used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total recoverable metal determinations by FLAA, GFAA, ICP-AES, or ICP-MS. Nitric acid and hydrochloric acid are added to the sample in a PTFE digestion vessel and heated in a microwave unit prior to metals determination.

Method 3020A: Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy

This method may be used for the preparation of waste samples for total recoverable metals determinations by GFAA or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with nitric acid. The method is applicable to aqueous samples, leachates, and mobility-procedure extracts.

Method 3031: Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry

This method may be used for the preparation of waste oils, oil sludges, tars, waxes, paints, paint sludges and other viscous petroleum products for analysis by FLAA, GFAA, and

ICP-AES. The samples are vigorously digested with nitric acid, sulfuric acid, hydrochloric acid, and potassium permanganate prior to analysis.

Method 3040A: Dissolution Procedure for Oils, Greases, or Waxes

This method may be used for the preparation of oily waste samples for determination of soluble metals by FLAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content.

Method 3050B: Acid Digestion of Sediments, Sludges, and Soils

This method may be used for the preparation of waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples.

Method 3051A: Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils

This method may be used for the preparation of sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid and hydrochloric acid are added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination.

Method 3052: Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices

This method may be used for the preparation of siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total metals analysis by FLAA, CVAA, GFAA, ICP-AES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis.

Method 3060A: Alkaline Digestion for Hexavalent Chromium

This method may be used for the preparation of soils, sludges, sediments and similar waste materials for hexavalent chromium determination. The samples are digested and heated to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

3.9 METHODS FOR DETERMINATION OF INORGANIC ANALYTES

This section of the manual contains analytical techniques for trace inorganic analyte determinations. Instrumental techniques include:

- Inductively coupled argon plasma atomic emission spectrometry (ICP-AES),
- Inductively coupled plasma mass spectrometry (ICP-MS),
- Direct-aspiration or flame atomic absorption spectrophotometry (FLAA),
- Graphite furnace atomic absorption spectrophotometry (GFAA),
- Hydride-generation atomic absorption spectrometry (HGAA),
- Cold-vapor atomic absorption spectrometry (CVAA),
- X-ray fluorescence (XRF),
- Ion chromatography (IC)
- Capillary electrophoresis (CE)
- Speciated isotope dilution mass spectrometry (SIDMS) and
- Several procedures for hexavalent chromium analysis.

Each of these (except the individual hexavalent chromium analyses) is discussed briefly below. Some advantages, disadvantages, and cautions for the analysis of wastes are provided.

Prior to employing the above 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. 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 judgments necessary to meet the data quality objectives or needs for the intended use of the data.

ICP-AES allows simultaneous or rapid sequential determination of many elements in a short time. Aerosol samples are introduced into an extremely hot plasma source which vaporizes, atomizes, ionizes and electronically excites the sample components. Upon exiting the plasma, the electronically excited analytes emit characteristic photons that are detected via emission spectrometry. A primary disadvantage of ICP-AES is the occurrence of background radiation from other elements and the plasma gases. Although all ICP-AES instruments utilize high-resolution optics and background correction to minimize these interferences, analysis of trace levels of inorganic analytes in the presence of a large excess of a single analyte is difficult. Examples would be trace levels of inorganic analytes in an alloy or trace metals in a limed (high calcium) waste. ICP-AES and FLAA have comparable detection limits (within a factor of 4) except that ICP-AES exhibits greater sensitivity for refractories (Al, Ba, etc.). FLAA, in general, will exhibit lower detection limits than either ICP-AES or FLAA.

ICP-MS allows sensitive, simultaneous determination of many elements in a short time frame using MS detection in place of AES. In general ICP-MS exhibits greater sensitivity than either GFAA, FLAA or ICP-AES for most elements. The greatest disadvantage of ICP-MS is isobaric elemental interferences. These are caused by different elements forming atomic ions with the same nominal mass-to-charge ratio. Mathematical correction for interfering ions can minimize these interferences.

FLAA direct-aspiration determinations, as opposed to ICP-AES or ICP-MS, are normally completed as single-element analyses and are relatively free of interelement spectral interferences. Either a nitrous-oxide/acetylene or air/acetylene flame is used as an energy source for dissociating the aspirated sample into the free atomic state, making analyte atoms available for absorption of light and spectrophotometric detection. In the analysis of some

elements, the temperature or type of flame used is critical. If the proper flame and analytical conditions are not used, chemical and ionization interferences can occur.

GFAA replaces the flame with an electrically-heated graphite furnace. The furnace allows for gradual heating of the sample aliquot in several stages. Thus, the processes of dissolution, drying, decomposition of organic and inorganic molecules and salts, and formation of atoms, which should occur in a flame or ICP in a few milliseconds may be allowed to occur over a much longer time period and at controlled temperatures in the furnace. This allows an experienced analyst to remove unwanted matrix components by using temperature programming and/or matrix modifiers. The major advantage of this technique is that it affords extremely low detection limits. It is the easiest to perform on relatively clean samples. Because this technique is so sensitive, interferences can be a real problem; finding the optimum combination of digestion, heating times and temperatures, and matrix modifiers can be a challenge for complex matrices.

HGAA utilizes a chemical reduction to reduce and separate arsenic or selenium selectively from a sample digestate. The technique therefore has the advantage of being able to isolate these two elements from complex samples which may cause interferences for other analytical procedures. Significant interferences have been reported when any of the following is present: (1) easily reduced metals (Cu, Ag, Hg); (2) high concentrations of transition metals (>200 mg/L); (3) oxidizing agents (oxides of nitrogen) remaining following sample digestion.

CVAA uses a chemical reduction to reduce mercury selectively. The procedure is extremely sensitive, but is subject to interferences from some volatile organics, chlorine, and sulfur compounds.

XRF uses sealed radioisotope sources to irradiate samples with X-rays. When a sample is irradiated with X-rays, the source X-rays may undergo either scattering or absorption by sample atoms. This later process is known as the photoelectric effect. When an atom absorbs the source X-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of X-rays characteristic of the given atom. The emission of X-rays, in this manner, is termed X-ray fluorescence.

IC generally refers to the separation of ions through ion exchange chromatography. In this technique, an aqueous sample is injected into a mobile solution that is carried into a chromatography column. As the sample travels through the column, the sample analytes are temporarily retained on the column, the stationary phase, via electrostatic forces. The separated analytes are identified as they are released from the column based on their retention time. Detection and quantification in IC is most commonly performed using conductivity detection. IC is typically used for the determination of anionic analytes in waste samples.

CE refers to the electrophorectic separation of ions dissolved or suspended in an electrolyte. Samples are introduced into a capillary tube containing an electrolytic buffer. Under the application of an electric field the cations in the sample migrate toward the negatively charged electrode (cathode) and the anions migrate toward the positively charged electrode (anode). This technique may be coupled with a variety of determinative techniques for quantitative analysis. Inorganic anions can be determined in environmental samples using CE and indirect UV detection, in which analytes are detected and quantified based on proportional decreases in the absorbance of the buffer solution. CE is a complementary technique to IC and typically offers shorter analysis times than IC.

SIDMS is a quantitative method for determining elemental species based on the measurement of isotope ratio(s) in each species of a nuclide using mass spectrometry after speciated isotope dilution. Samples are mixed with one or more isotopic spikes which have different isotopic abundances and are artificially converted to chemical forms corresponding to the species to be analyzed. The spiked samples are then subjected to the separation of the species and the measurement of the altered isotope ratios in each species. Both species concentrations and species conversions can be mathematically derived.

The following methods are included in this section:

Method 6010C: Inductively Coupled Plasma-Atomic Emission Spectrometry
Method 6020A: Inductively Coupled Plasma-Mass Spectrometry
Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
Method 6500: Dissolved Inorganic Anions in Aqueous Matrices by Capillary Ion Electrophoresis
Method 6800: Elemental and Speciated Isotope Dilution Mass Spectrometry
Method 7000B: Flame Atomic Absorption Spectrophotometry
Method 7010: Graphite Furnace Atomic Absorption Spectrophotometry
Method 7061A: Arsenic (Atomic Absorption, Gaseous Hydride)
Method 7062: Antimony and Arsenic (Atomic Absorption, Borohydride Reduction)
Method 7063: Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltametry (ASV)
Method 7195: Chromium, Hexavalent (Coprecipitation)
Method 7196A: Chromium, Hexavalent (Colorimetric)
Method 7197: Chromium, Hexavalent (Chelation/Extraction)
Method 7198: Chromium, Hexavalent (Differential Pulse Polarography)
Method 7199: Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography
Method 7470A: Mercury in Liquid Waste (Manual Cold-Vapor Technique)
Method 7471B: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
Method 7472: Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltametry (ASV)
Method 7473: Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry
Method 7474: Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry
Method 7580: White Phosphorus (P_4) by Solvent Extraction and Gas Chromatography
Method 7741A: Selenium (Atomic Absorption, Gaseous Hydride)
Method 7742: Selenium (Atomic Absorption, Borohydride Reduction)

CHAPTER FOUR

ORGANIC ANALYTES

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. 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 this chapter 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.

4.1 SAMPLING CONSIDERATIONS

4.1.1 Introduction

Following the initial and critical step of designing a sampling plan (Chapter Nine) is the implementation of that plan such that a representative sample of the solid waste is collected. Once the sample has been collected it must be stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. The sample type, type of containers and their preparation, possible forms of contamination, and preservation methods are all items which must be thoroughly examined in order to maintain the integrity of the samples. This section highlights considerations which must be addressed in order to maintain a sample's integrity and representativeness. This section is, however, applicable only to trace analyses.

Quality Control (QC) requirements need not be met for all compounds presented in the Table of Analytes for the method in use, rather, the requirements must be met for all compounds reported. A report of non-detect is considered a quantitative report, and must meet all applicable QC requirements for that compound and the method used.

4.1.2 Sample handling and preservation

This section deals separately with volatile and semivolatile organics. Refer to Chapter Two and Table 4-1 of this section for sample containers, sample preservation, and sample holding time information.

Volatile organics

Samples that contain analytes that are subject to biological degradation prior to analysis need to be preserved. Samples where aromatic hydrocarbons are target analytes, which are most subject to biological degradation, need to be preserved, unless they are to be analyzed immediately on-site, even if other VOA compound classes are present. Chemical preservation may be inappropriate for highly reactive compounds, e.g., styrene, vinyl chloride, 2-chloroethyl vinyl ether, acrylamide, etc., since it may accelerate loss by polymerization or other rapid chemical reaction. Samples for which chlorinated aliphatic hydrocarbons are the only target analytes generally do not need to be preserved. However, all aqueous samples containing free chlorine must be preserved with a dechlorinating agent in order to prevent formation of trihalomethanes and other possible chemical reactions.

Although VOA samples may be held for up to 7 days unpreserved or 14 days or longer preserved, it is not recommended as good laboratory practice to hold them that long. VOA samples should be run as soon as possible after receipt by the laboratory. Samples containing highly reactive compounds, e.g., styrene, vinyl chloride, 2-chloroethyl vinyl ether, acrylamide, etc., as target analytes should not be preserved and should be analyzed as soon as they are received in the laboratory.

Standard 40-mL glass screw-cap VOA vials with PTFE-lined silicone septa may be used for liquid matrices. Special 40-mL VOA vials for purge-and-trap of solid samples are described in Method 5035. VOA vials for headspace analysis of solid samples are described in Method 5021. Standard 125-mL wide-mouth glass containers may be used for Methods 5031 and 5032 for high concentration samples only. However, the sampling procedures described in Method 5035 may minimize sample preparation analyte loss better than the procedures described in Methods 5031 and 5032. The vials and septa should be washed with soap and water and rinsed with distilled deionized water. After thoroughly cleaning the vials and septa, they should be placed in an oven and dried at 100 °C for approximately one hour.

NOTE: Do not heat the septa for extended periods of time (i.e., more than one hour, because the silicone begins to slowly degrade at 105 °C).

When collecting the samples, liquids and solids should be introduced into the vials gently to reduce agitation which might drive off volatile compounds.

In general, liquid samples should be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. The vials should be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The sample should be hermetically sealed in the vial at the time of sampling, and must not be opened prior to analysis to preserve their integrity.

- Due to differing solubility and diffusion properties of gases in LIQUID matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and should not invalidate a sample for volatiles analysis.

- The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial, (e.g. carbonate-containing samples preserved with acid). Studies conducted by the USEPA (EMSL-Ci, unpublished data) indicate that "pea-sized" bubbles (i.e., bubbles not exceeding 1/4 inch or 6 mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are more susceptible to variations in gas solubility than are groundwater samples.

– Pre-testing of a representative soil or aqueous sample, prior to collection, with acid or bisulfate may show effervescence if carbonaceous materials are present. If bubbling occurs during chemical preservation, an increased potential for loss of volatile constituents exists and samples should therefore be collected without preserving with acid or bisulfate.

Immediately prior to analysis of liquid samples, the aliquot to be analyzed should be taken from the vial using the instructions from the appropriate sample introduction technique:

- For smaller analysis volumes, a gas-tight syringe may be inserted directly through the septum of the vial to withdraw the sample.

- For larger analysis volumes, (e.g. purge-and-trap analyses) the sample may be carefully poured into the syringe barrel. Opening a volatile sample to pour a sample into a syringe destroys the validity of the sample for future analysis. Therefore, if there is only one VOA vial, it is strongly recommended that the analyst fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly.

If these guidelines are not followed, then the validity of the data generated from the samples may be suspect.

VOA vials for samples with solid or semi-solid matrices (e.g., sludges) should be filled according to the guidance given in the appropriate 5000 series sample introduction method (see Table 4-1) to be used. When 125-mL wide-mouth glass containers are used for high-concentration samples only, the containers should be filled as completely as possible. The 125-mL vials should be tapped slightly as they are filled to try and eliminate as much free air space as possible. A minimum of two vials should also be filled per sample location.

At least two VOA vials should be filled and labeled immediately at the point at which the sample is collected. They should NOT be filled near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the samples. The two vials from each sampling location should then be sealed in separate plastic bags to prevent cross-contamination between samples, particularly if the sampled waste is suspected of containing high levels of volatile organics. (Activated carbon may also be included in the bags to prevent cross-contamination from highly contaminated samples.) VOA samples may also be contaminated by diffusion of volatile organics through the septum during shipment and storage. To monitor possible contamination, a trip blank prepared from organic-free reagent water (as defined in Chapter One) should be carried throughout the sampling, storage, and shipping process. Reactive compounds such as 2-chloroethyl vinyl ether, vinyl chloride, and styrene can readily be lost under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.

Semivolatile organics (including pesticides, PCBs and herbicides)

Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing (see Sec. 4.1.4 for specific instructions on glassware cleaning). The sample containers should be of glass, and have screw-caps with PTFE-lined septa. In situations where PTFE liners are not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's

gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank.

4.1.3 Safety

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

Safety should always be the primary consideration in the collection of samples. A thorough understanding of the waste production process, as well as all of the potential hazards making up the waste, should be investigated whenever possible. The site should be evaluated just prior to sampling to determine additional safety measures. Minimum protection of gloves and safety glasses should be worn to prevent sample contact with the skin and eyes. A respirator should be worn even when working outdoors if organic vapors are present. More hazardous sampling missions may require the use of supplied air and special clothing.

4.1.4 Cleaning of glassware

In the analysis of samples containing components in the parts per billion range, the preparation of scrupulously clean glassware is necessary. Failure to do so can lead to a myriad of problems in the interpretation of the final chromatograms due to the presence of extraneous peaks resulting from contamination. Particular care must be taken with glassware such as Soxhlet extractors, Kuderna-Danish evaporative concentrators, sampling-train components, or any other glassware coming in contact with an extract that will be evaporated to a smaller volume. The process of concentrating the compounds of interest in this operation may similarly concentrate the contaminating substance(s), which distort the results.

The basic cleaning steps are:

1. Removal of surface residuals immediately after use;

2. Hot soak to loosen and float most particulate material;

3. Hot water rinse to flush away floated particulates;

4. Soak with an oxidizing agent to destroy traces of organic compounds;

5. Hot water rinse to flush away materials loosened by the deep penetrant soak;

6. Distilled water rinse to remove metallic deposits from the tap water;

7. Alcohol, e.g., isopropanol or methanol, rinse to flush off any final traces of organic materials and remove the water; and

8. Flushing the item immediately before use with some of the same solvent that will be used in the analysis.

Comments regarding each of the eight fundamental steps are discussed here in the order in which they appeared above:

Step 1: As soon as possible after glassware (i.e., beakers, pipets, flasks, or bottles) has come in contact with sample or standards, the glassware should be flushed with alcohol before it is placed in the hot detergent soak. If this is not done, the soak bath may serve to contaminate all other glassware placed therein.

Step 2: The hot soak consists of a bath of a suitable detergent in water of 50 °C or higher. The detergent, powder or liquid, should be entirely synthetic and not a fatty acid base. There are very few areas of the country where the water hardness is sufficiently low to avoid the formation of some hard-water scum resulting from the reaction between calcium and magnesium salts with a fatty acid soap. This hard-water scum or curd would have an affinity particularly for many chlorinated compounds and, being almost wholly water-insoluble, would deposit on all glassware in the bath in a thin film.

There are many suitable detergents on the wholesale and retail market. Most of the common liquid dishwashing detergents sold at retail are satisfactory but are more expensive than other comparable products sold industrially. Alconox, in powder or tablet form, is manufactured by Alconox, Inc., New York, and is marketed by a number of laboratory supply firms. Sparkleen, another powdered product, is distributed by Fisher Scientific Company.

Step 3: No comments required.

Step 4: **Chromic acid should not be used to clean glassware.** Commercial, non-chromate products (e.g., Nochromix) may be used in place of chromic acid, if adequate cleaning is documented by an analytical quality control program. Chromic acid should also not be used with plastic bottles.

The potential hazards of using chromic-sulfuric acid mixture are great and have been well publicized. There are now commercially available substitutes that possess the advantage of safety in handling. These are biodegradable concentrates with a claimed cleaning strength equal to the chromic acid solution. They are alkaline, equivalent to ca. 0.1 N NaOH upon dilution, and are claimed to remove dried blood, silicone greases, distillation residues, insoluble organic residues, etc. They are further claimed to remove radioactive traces and will not attack glass or exert a corrosive effect on skin or clothing. One such product is "Chem Solv 2157," manufactured by Mallinckrodt and available through laboratory supply firms. Another comparable product is "Detex," a product of Borer-Chemie, Solothurn, Switzerland. Other similarly effective products are Nochromix (Godax Laboratories) and Contrad 70 (Decon Labs).

Steps 5, 6, and 7: No comments required.

Step 8: There is always a possibility that between the time of washing and the next use, the glassware could pick up some contamination from either the air or direct contact. To prevent this, it is good practice to flush the item immediately before use with some of the same solvent that will be used in the analysis.

The drying and storage of the cleaned glassware is of critical importance to prevent the beneficial effects of the scrupulous cleaning from being nullified. Pegboard drying is not recommended. It is recommended that laboratory glassware and equipment be dried at 100 °C. Under no circumstances should such small items be left in the open without protective covering. The dust cloud raised by the daily sweeping of the laboratory floor can most effectively recontaminate the clean glassware.

As an alternate to solvent rinsing, the glassware can be heated to a minimum of 300 °C to vaporize any organics. Do not use this high temperature treatment on volumetric glassware, glassware with ground glass joints, or sintered glassware.

4.1.5 High concentration samples

Cross contamination of trace concentration samples may occur when prepared in the same laboratory with high concentration samples. Ideally, if both type samples are being handled, a laboratory and glassware dedicated solely to the preparation of high concentration samples would be available for this purpose. If this is not feasible, as a minimum when preparing high concentration samples, disposable glassware should be used or, at least, glassware dedicated entirely to the high concentration samples. Avoid cleaning glassware used for both trace and high concentration samples in the same area.

TABLE 4-1
RECOMMENDED SAMPLE CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES[a]
(Note: Footnotes are located on the last page of the table.)

VOLATILE ORGANICS

Sample Matrix	Container	Preservative[1]	Holding Time[1]
Concentrated waste samples	Method 5035: See the method. Method 5021: See the method. Methods 5031 and 5032: See the methods. Use PTFE-lined lids for all procedures.	Cool to ≤6 °C.	14 days
Aqueous samples with no residual chlorine present	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Cool to ≤6 °C and adjust pH to less than 2 with H_2SO_4, HCl, or solid $NaHSO_4$	14 days
		If carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples.	*7 days*
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	*7 days*

VOLATILE ORGANICS (continued)			
Sample Matrix	Container	Preservative[1]	Holding Time[1]
Aqueous samples WITH residual chlorine present	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to ≤6 °C and adjust pH to less than 2 with H_2SO_4, HCl, or solid $NaHSO_4$.	14 days
		If carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples.	*7 days*
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	*7 days*
Acrolein and acrylonitrile in aqueous samples	Methods 5030, 5031, and 5032: 2 x 40-mL vials with PTFE-lined septum caps	Adjust to pH 4-5. Cool to ≤6 °C. *These compounds are highly reactive and should be analyzed as soon as possible.*	7days
Solid samples (e.g. soils, sediments, sludges, ash)	Method 5035: See the method. Method 5021: See the method. Methods 5031 and 5032: See the methods.	See the individual methods.	14 days
		If vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.	7 days

TABLE 4-1 (Continued)

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES AND HERBICIDES

Sample Matrix	Container	Preservative[1]	Holding Time[1]
Concentrated waste samples	125-mL wide-mouth glass with PTFE-lined lid	None	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.
Aqueous samples with no residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Cool to $\leq$6 °C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.

TABLE 4-1 (Continued)

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES AND HERBICIDES (continued)			
Sample Matrix	Container	Preservative[1]	Holding Time[2]
Aqueous samples WITH residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤6 °C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Solid samples (e.g. soils, sediments, sludges, ash)	250-mL wide-mouth glass container with PTFE-lined lid	Cool to ≤6 °C.	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.

POLYCHLORINATED BIPHENYLS, POLYCHLORINATED DIBENZO-*p*-DIOXINS, AND POLYCHLORINATED DIBENZOFURANS			
Sample Matrix	Container	Preservative[1]	Holding Time[2]
Concentrated waste samples	125-mL wide-mouth glass with PTFE-lined lid	None	None
Aqueous samples with no residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Cool to ≤6 °C.	None

TABLE 4-1 (Continued)

POLYCHLORINATED BIPHENYLS, POLYCHLORINATED DIBENZO-*p*-DIOXINS, AND POLYCHLORINATED DIBENZOFURANS (continued)			
Sample Matrix	Container	Preservative[1]	Holding Time[2]
Aqueous samples WITH residual chlorine present	4 x 1-L amber glass container with PTFE-lined lid, or other size, as appropriate, to allow use of entire sample for analysis.	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to ≤6 °C	None
Solid samples (e.g. soils, sediments, sludges, ash)	250-mL wide-mouth glass container with PTFE-lined lid.	Cool to ≤6 °C.	None

[a] The information presented in this table does not represent EPA requirements, but rather it is intended solely as guidance. Selection of containers, preservation techniques and applicable holding times should be based on the stated project-specific data quality objectives.

[1] The exact sample, extract, and standard storage temperature should be based on project-specific requirements and/or manufacturer's recommendations for commercially available standards. Furthermore, alternative storage temperatures may be appropriate based on demonstrated analyte stability in a given matrix, provided the stated data quality objectives for a project-specific application are still attainable.

[2] A longer holding time may be appropriate if it can be demonstrated that the reported analyte concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

4.2 SAMPLE PREPARATION 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. 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.

4.2.1 Extractions and preparations

The following methods are included in this section:

Method 3500C:	Organic Extraction and Sample Preparation
Method 3510C:	Separatory Funnel Liquid-Liquid Extraction
Method 3520C:	Continuous Liquid-Liquid Extraction
Method 3535A:	Solid-Phase Extraction (SPE)
Method 3540C:	Soxhlet Extraction
Method 3541:	Automated Soxhlet Extraction
Method 3542:	Extraction of Semivolatile Analytes Collected Using Method 0010 (Modified Method 5 Sampling Train)
Method 3545A:	Pressurized Fluid Extraction (PFE)
Method 3546:	Microwave Extraction
Method 3550C:	Ultrasonic Extraction
Method 3560:	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
Method 3561:	Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
Method 3562:	Supercritical Fluid Extraction of Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides
Method 3580A:	Waste Dilution
Method 3585:	Waste Dilution for Volatile Organics
Method 5000:	Sample Preparation for Volatile Organic Compounds
Method 5021:	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
Method 5030B:	Purge-and-Trap for Aqueous Samples
Method 5031:	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
Method 5032:	Volatile Organic Compounds by Vacuum Distillation
Method 5035:	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
Method 5041A:	Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST)

4.2 Sample preparation 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. 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.

4.2.2 Cleanup

The following methods are included in this section:

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

4.3 DETERMINATION OF ORGANIC ANALYTES

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. 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.

4.3.1 Gas chromatographic methods

The following methods are included in this section:

Method 8000B:	Determinative Chromatographic Separations
Method 8011:	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography
Method 8015C:	Nonhalogenated Organics by Gas Chromatography
Method 8021B:	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors
Method 8031:	Acrylonitrile by Gas Chromatography
Method 8032A:	Acrylamide by Gas Chromatography
Method 8033:	Acetonitrile by Gas Chromatography with Nitrogen-Phosphorus Detection
Method 8041A:	Phenols by Gas Chromatography
Method 8061A:	Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC/ECD)
Method 8070A:	Nitrosamines by Gas Chromatography
Method 8081B:	Organochlorine Pesticides by Gas Chromatography
Method 8082A:	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
Method 8085:	Compound-independent Elemental Quantitation of Pesticides by Gas Chromatography with Atomic Emission Detection (GC/AED)
Method 8091:	Nitroaromatics and Cyclic Ketones by Gas Chromatography
Method 8095:	Explosives by Gas Chromatography
Method 8100:	Polynuclear Aromatic Hydrocarbons
Method 8111:	Haloethers by Gas Chromatography
Method 8121:	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique
Method 8131:	Aniline and Selected Derivatives by Gas Chromatography
Method 8141B:	Organophosphorus Compounds by Gas Chromatography
Method 8151A:	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization

4.3.2 Gas chromatographic/mass spectrometric 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. 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 included in this section:

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

Method 8261: Volatile Organic Compounds by Vacuum Distillation in Combination with Gas Chromatography/Mass Spectrometry (VD/GC/MS)

Method 8270D: 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 8280B: Polychlorinated Dibenzo-*p*-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)

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

Appendix A: Procedures for the Collection, Handling, Analysis, and Reporting of Wipe Tests Performed within the Laboratory

4.3.3 High performance liquid chromatographic 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. 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 included in this section:

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 8318A:	*N*-Methylcarbamates by High Performance Liquid Chromatography (HPLC)
Method 8321B:	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 8330A:	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

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. 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 included in this section:

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

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. 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 method is included in this section:

Method 8520: Continuous Measurement of Formaldehyde in Ambient Air

4.4 IMMUNOASSAY 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. 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 included in this section:

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
Method 4425:	Screening Extracts of Environmental Samples for Planar Organic Compounds (PAHs, PCBs, PCDDs/PCDFs) by a Reporter Gene on a Human Cell Line
Method 4670:	Triazine Herbicides as Atrazine in Water by Quantitative Immunoassay

4.5 MISCELLANEOUS SCREENING 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. 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 included in this section:

Method 3815:	Screening Solid Samples for Volatile Organics
Method 3820:	Hexadecane Extraction and Screening of Purgeable Organics
Method 8510:	Colorimetric Screening Procedure for RDX and HMX in Soil
Method 8515:	Colorimetric Screening Method for Trinitrotoluene (TNT) in Soil
Method 8535:	Screening Procedure for Total Volatile Organic Halides in Water
Method 8540:	Pentachlorophenol by UV-Induced Colorimetry
Method 9074:	Turbidimetric Screening Method for Total Recoverable Petroleum Hydrocarbons in Soil
Method 9078:	Screening Test Method for Polychlorinated Biphenyls in Soil
Method 9079:	Screening Test Method for Polychlorinated Biphenyls in Transformer Oil

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. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in each procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.

The following methods are found in Chapter Five:

Method 5050:	Bomb Preparation Method for Solid Waste
Method 9000:	Determination of Water in Waste Materials by Karl Fischer Titration
Method 9001:	Determination of Water in Waste Materials by Quantitative Calcium Hydride Reaction
Method 9010B:	Total and Amenable Cyanide: Distillation
Method 9012A:	Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)
Method 9013:	Cyanide Extraction Procedure for Solids and Oils
Method 9014:	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
Method 9020B:	Total Organic Halides (TOX)
Method 9021:	Purgeable Organic Halides (POX)
Method 9022:	Total Organic Halides (TOX) by Neutron Activation Analysis
Method 9023:	Extractable Organic Halides (EOX) in Solids
Method 9030B:	Acid-Soluble and Acid-Insoluble Sulfides: Distillation
Method 9031:	Extractable Sulfides
Method 9034:	Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides
Method 9035:	Sulfate (Colorimetric, Automated, Chloranilate)
Method 9036:	Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
Method 9038:	Sulfate (Turbidimetric)
Method 9056A:	Determination of Inorganic Anions by Ion Chromatography
Method 9057:	Determination of Chloride from HCl/Cl_2 Emission Sampling Train (Methods 0050 and 0051) by Anion Chromatography
Method 9060A:	Total Organic Carbon
Method 9065:	Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)
Method 9066:	Phenolics (Colorimetric, Automated 4-AAP with Distillation)
Method 9067:	Phenolics (Spectrophotometric, MBTH with Distillation)
Method 9070A:	*n*-Hexane Extractable Material (HEM) for Aqueous Samples

Method 9071B: *n*-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples

Method 9075: Test Method for Total Chlorine in New and Used Petroleum Products by X-Ray Fluorescence Spectrometry (XRF)

Method 9076: Test Method for Total Chlorine in New and Used Petroleum Products by Oxidative Combustion and Microcoulometry

Method 9077: Test Methods for Total Chlorine in New and Used Petroleum Products (Field Test Kit Methods)

- **Method A:** Fixed End Point Test Kit Method
- **Method B:** Reverse Titration Quantitative End Point Test Kit Method
- **Method C:** Direct Titration Quantitative End Point Test Kit Method

Method 9131: Total Coliform: Multiple Tube Fermentation Technique

Method 9132: Total Coliform: Membrane-Filter Technique

Method 9210A: Potentiometric Determination of Nitrate in Aqueous Samples with an Ion-Selective Electrode

Method 9211: Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode

Method 9212: Potentiometric Determination of Chloride in Aqueous Samples with Ion-Selective Electrode

Method 9213: Potentiometric Determination of Cyanide in Aqueous Samples and Distillates with Ion-Selective Electrode

Method 9214: Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode

Method 9215: Potentiometric Determination of Sulfide in Aqueous Samples and Distillates with Ion-Selective Electrode

Method 9216: Potentiometric Determination of Nitrate in Aqueous Samples with Ion-Selective Electrode

Method 9250: Chloride (Colorimetric, Automated Ferricyanide AAI)

Method 9251: Chloride (Colorimetric, Automated Ferricyanide AAII)

Method 9253: Chloride (Titrimetric, Silver Nitrate)

Method 9320: Radium-228

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:

Method 1030:	Ignitability of Solids
Method 1040:	Test Method for Oxidizing Solids
Method 1050:	Test Methods to Determine Substances Likely to Spontaneously Combust
Method 1120:	Dermal Corrosion
Method 1312:	Synthetic Precipitation Leaching Procedure
Method 1320:	Multiple Extraction Procedure
Method 1330A:	Extraction Procedure for Oily Wastes
Method 9041A:	pH Paper Method
Method 9045D:	Soil and Waste pH
Method 9050A:	Specific Conductance
Method 9080:	Cation-Exchange Capacity of Soils (Ammonium Acetate)
Method 9081:	Cation-Exchange Capacity of Soils (Sodium Acetate)
Method 9090A:	Compatibility Test for Wastes and Membrane Liners
Method 9095B:	Paint Filter Liquids Test
Method 9096:	Liquid Release Test (LRT) Procedure
	Appendix A: Liquid Release Test Pre-Test
Method 9100:	Saturated Hydraulic Conductivity, Saturated Leachate Conductivity, and Intrinsic Permeability
Method 9310:	Gross Alpha and Gross Beta
Method 9315:	Alpha-Emitting Radium Isotopes

CHAPTER SEVEN

CHARACTERISTICS INTRODUCTION AND REGULATORY DEFINITIONS

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

7.1 IGNITABILITY

7.1.1 Introduction

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

7.1.2 Regulatory Definition

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

7.2 CORROSIVITY

7.2.1 Introduction

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

1. Mobilize toxic metals if discharged into a landfill environment;

2. Corrode handling, storage, transportation, and management equipment; or

3. Destroy human or animal tissue in the event of inadvertent contact.

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

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

7.2.2 Regulatory Definition

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

7.3 REACTIVITY

7.3.1 Introduction

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

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

7.3.2 Regulatory Definition

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

7.4 TOXICITY CHARACTERISTIC LEACHING PROCEDURE

7.4.1 Introduction

The Toxicity Characteristic Leaching Procedure (TCLP) is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill. This test is designed to simulate leaching that takes place in a sanitary landfill only. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A subsample of a waste is extracted with the appropriate buffered acetic acid solution for 18 $\pm$ 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

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

1. Separation Procedure

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

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

2. Particle Size Reduction

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

3. Extraction of Solid Material

The solid material from Step 2 is extracted for 18 $\pm$ 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 µm glass fiber filter. If compatible, the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

5. Testing (Analysis) of TCLP Extract

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

7.4.3 Regulatory Definition

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

Under the Land Disposal Restrictions regulations (40 CFR, Part 268), a restricted waste identified in 40 CFR 268.40 cannot be land disposed if a TCLP extract of the waste or a TCLP extract of the treatment residue of the waste exceeds the values shown in the table of 40 CFR 268.40 for any hazardous constituent listed in the table for that waste. If a waste contains

<0.5% filterable solids, the waste itself, after filtering, is considered to be the extract for the purposes of analysis.

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

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

[1]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.
[2]Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

FIGURE 3.

TOXICITY CHARACTERISTIC LEACHING PROCEDURE FLOWCHART

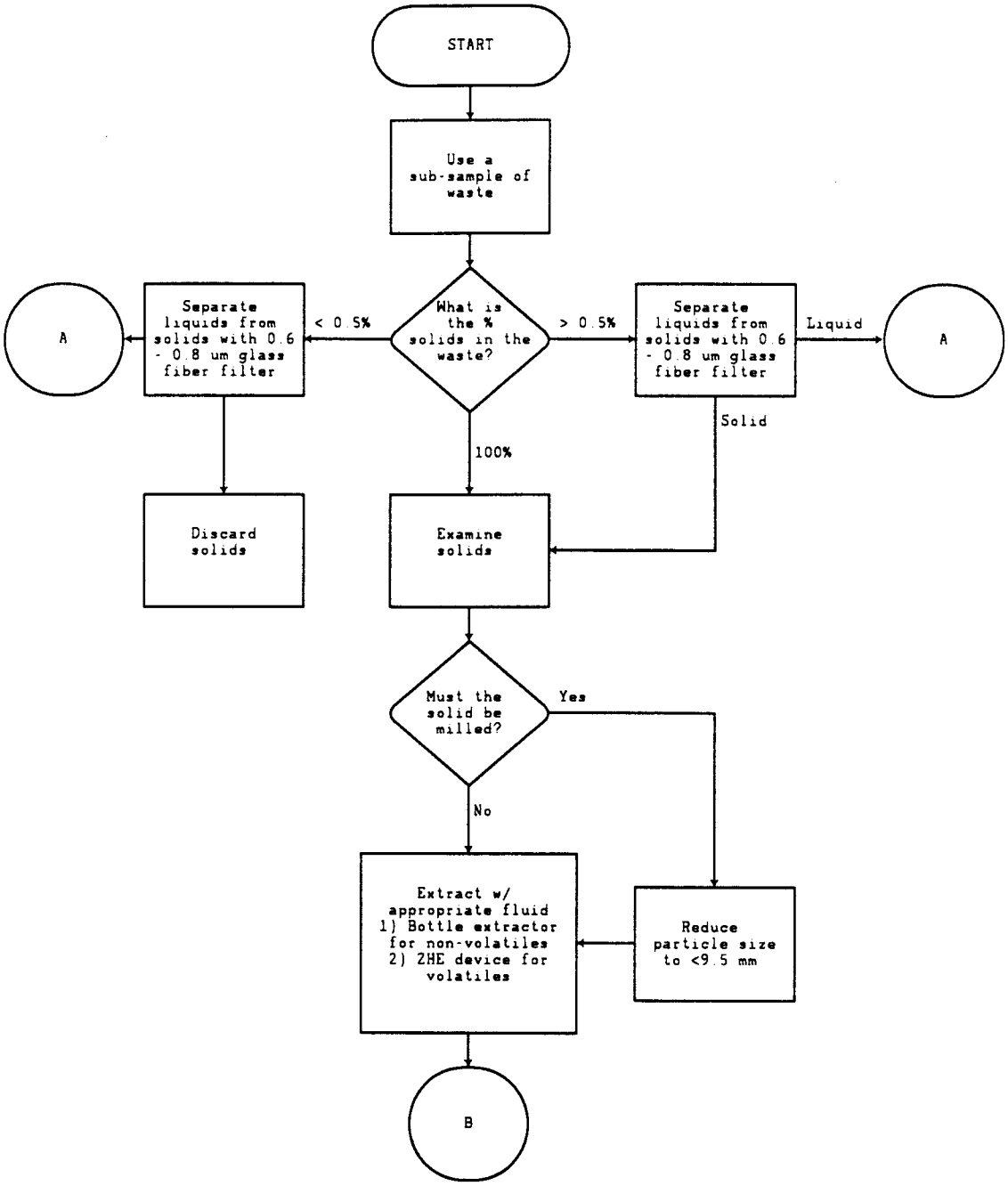
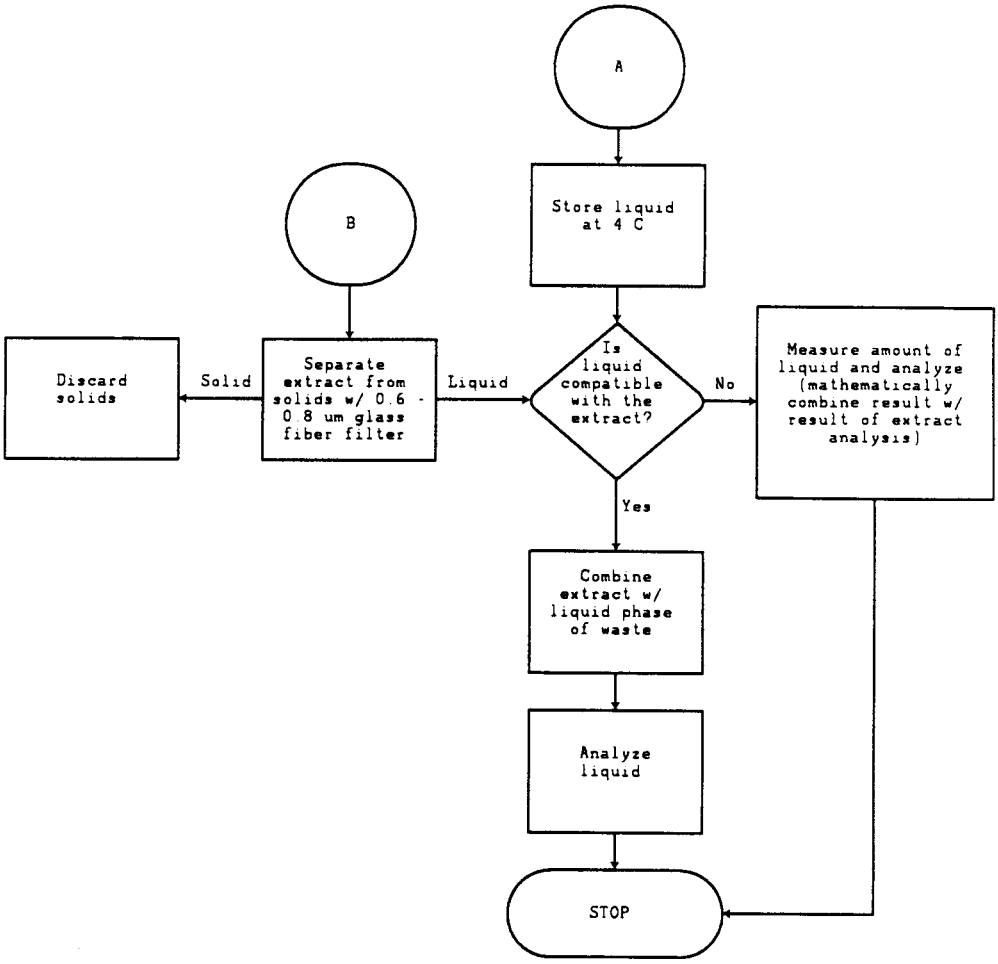


FIGURE 3 (continued)



CHAPTER EIGHT

METHODS FOR DETERMINING CHARACTERISTICS

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

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

8.1 Ignitability

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

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

Method 1010A: Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

Method 1020B : Standard Test Methods for Flash Point by Setaflash (Small Scale) Closed-cup Apparatus

8.2 Corrosivity

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

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

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

8.3 Toxicity

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

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

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