

GUIDE TO  
**ENVIRONMENTAL  
ANALYTICAL  
METHODS**  
5th Edition

*Edited by*  
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## INTRODUCTION TO 5<sup>th</sup> EDITION

The fifth edition of the *Guide to Environmental Analytical Methods* covers the massive changes that have continued to take place to EPA methods, affecting organic and inorganic determinative methods along with preparative and sampling procedures.

Since the last edition of "*The Guide*," EPA SW846 has been updated with many new methods; existing methods have been revised; and more methods removed. For instance the large number of individual flame AA and graphite furnace AA methods for metals have been collapsed into general Methods 7000B and 7010, respectively. Update IV to SW-846 has been out in distribution for several years however the legal status of the update is uncertain in many States as the Office of Solid Waste has not promulgated the update, choosing instead to hold fast to the idea of SW-846 as guidance rather than regulation. I have chosen to update this book to Update IV of the Third Edition of SW-846 as many laboratories are already using it. Previous editions of this book present appropriate information about earlier SW-846 versions. The changes from SW846 Final Update III are so expansive that the Method Status Table in Appendix D of this fifth edition has been revised, listing current promulgated methods and available more recently revised versions.

The CLP data are extensively revised to reflect the newest Statements of Work: inorganics ILM05.2, December, 2001, and organics OLM04.2, May, 1999 and OLC03.2, December, 2000. Further, tabular data on the CLP organics have been expanded to include the low concentration method in a side-by-side comparison with the multi-media, multi-concentration Statement of Work. The inorganic SOW is noteworthy as no FLAA nor GFAA techniques are included in it and the dreaded GFAA decision flowchart has been dropped.

An important addition to the fifth edition is tabular comparison of multi-anion analysis by ion chromatography and capillary electrophoresis. These analytical techniques are becoming more visible within the industry and the regulatory framework. The increased number of practitioners of these methods justifies their inclusion.

The discussion and data relating to the analysis of PCB congeners in SW-846 Method 8082A has been deleted, as it is an inadequate method for the stated objective. The Office of Water is working on Method 1668, which is a high resolution mass spectrometer technique capable of identifying and quantitating most of the 209 PCB congeners including the individual planar (toxic) PCB.

All EPA 200 series, 500 series, 600 series, and methods found in the 20th Edition of *Standard Methods for the Examination of Water and Wastewater* contained in the book have been reviewed for changes and revised where necessary.

It must be kept in the forefront that despite the wishes and practices of many in the laboratory community, EPA still maintains a strict separation between program areas. Thus EPA Method 8270 (regardless of version) is not a permissible replacement for EPA Method 625 for the analysis of semivolatile organics in wastewater. Let the reader be aware that even though they may think Method 8270 is the better method, Clean Water Act regulations still require the use of Method 625. And Method 625 has easily recognizable differences from Method 8270.

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## HISTORY and ACKNOWLEDGMENTS

The *Guide to Environmental Analytical Methods* was originally developed from a method comparison document called *Variability in Protocols* (VIP) published by the EPA Risk Reduction Engineering Laboratory in Cincinnati, Ohio. VIP provided a useful tool for understanding similarities and differences among commonly used environmental test methods. This concept was greatly expanded by Northeast Analytical's staff with the first publication of the *Guide to Environmental Analytical Methods* in 1992. Now in its fifth edition, and under new editorship, *The Guide* has become a popular reference tool for all who work in the environmental field.

Dr. Roy-Keith Smith retired in June, 2000 from his former position as Quality Assurance Manager and Analytical Methods Manager with a prominent commercial environmental laboratory in the Southeast. His retirement has actually provided more time for him to delve into the intricacies of the environmental industry, unhampered by the day-to-day distractions of the laboratory. Besides writing, Dr. Smith has been busy serving as an expert witness for environmental lawsuits, where his specialty of foundation destruction has been widely appreciated, and for teaching, presenting seminars, and laboratory consultations. He can be reached through Genium Publishing, [www.genium.com](http://www.genium.com).

## Analytical Methods - Organic Constituents

The numerous analytical methods for organic constituent testing have been organized into five major categories:

- Herbicides (chlorinated) identified by gas chromatography
- Pesticides (organochlorine) and PCBs identified by gas chromatography
- Semivolatile organic compounds identified by gas chromatography/mass spectroscopy
- Volatile organic compounds identified by gas chromatography/mass spectroscopy
- Volatile organic compounds (aromatic and halogenated) identified by gas chromatography

Within each category there is a section called *Method Comparison* that summarizes the key method requirements and components for each testing method. The methods are compared on a side-by-side basis, which allows the user to quickly compare key aspects of the test methods. Where appropriate the user is referred to a table in Appendix A that provides details about an aspect of the test method. The tables in Appendix A are taken directly from the regulations.

For determining if a specific analyte is analyzed by a test method, each category has an *Analyte Listing* of all compounds addressed by the test methods within the category. This is also presented on a side-by-side basis so that the user can quickly see which method(s) cover the specific compound in question.

If you want to find a test method for a specific analyte but don't know what category(ies) it belongs to, refer to Appendix B. Appendix B is an alphabetical listing of all organic analytes specified in the method comparison sections. Use this appendix to identify the category to turn to for additional information.

## SW-846 Method 8000B

Unlike most Methods published by EPA and the various Standards organizations, the complete intent and guidance of SW-846, the EPA Office of Solid Waste Methods Manual, is not present in an individual Method such as Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry. Instead SW-846 is broken down into sampling guidance, sample preparation guidance, and sample analysis guidance, followed by specific Methods that implement the guidance. Thus to say that Method 8270D was performed on a sample actually means that a host of Methods in SW-846 were used, specifically Chapter 1 and Chapter 2, the quality control and Method selection chapters, followed by Methods 3500B (Sample preparation guidance) and a specific sample preparation technique such as Method 3520C (continuous liquid-liquid extraction). Next the general directions for use of analytical instruments in organic analysis, Method 8000B, was consulted prior to the use of the specific determinative Method, 8270D.

Method 8000B is essential to successful use of any chromatographic Methods in SW-846. Method 8000B has the following contents:

- Section 1. Introduction and Applicability
- Section 2. Summary
- Section 3. Interferences/Chromatographic Performance
- Section 4. Apparatus and Materials (GC Inlet Systems, flow control, columns, detectors, HPLC inlet systems, pumps, columns, ovens, detectors, and data systems)
- Section 5. Reagents
- Section 6. Sample Collection, Preservation, and Handling
- Section 7. Procedure (Extraction, cleanup and separation, initial calibration, calibration linearity, retention time windows, calibration verification, chromatographic analysis, compound identification, calculations, and system maintenance.)
- Section 8. Quality Control (chromatographic performance, required instrument quality control, initial demonstration of proficiency, matrix spike and laboratory control samples, surrogate recoveries, generating performance criteria for quality control measures.)
- Section 9. Method Performance (MDL definition and procedure for determination.)
- Section 10. References

All of Method 8000B is important, however if any sections can be said to be critical they are Sections 7 and 8. All of the chromatographic Methods in SW-846 describe a calibration procedure, however they are very scant on description of options. Method 8000B exhaustively describes the options for the cases where linear calibration is not achievable. One particular item of concern is the number of concentration standards needed in a non-linear calibration. Five standards are required for a linear calibration, six for a second order (quadratic equation) calibration, and seven for a third order equation.

Method Comparison

Determination of Chlorinated Herbicides by GC

Method ⇒ Parameter ↓	SW-846 Method 8151A	EPA 500 Series Method 515.4 <sup>i</sup>	EPA 500 Series Method 515.2	Standard Methods Method 6640 <sup>ii</sup>
<b>Applicability</b>	Groundwater, soils, sludges, and non-water miscible wastes	Groundwater, and drinking water	Groundwater and drinking water	Groundwater, surface, and drinking water
<b>Number of Analytes (3)</b>	19 total	17 total	14 total	9 total
<b>Method Validation (4)</b>	Extract and analyze 4 replicates of a QC check sample. Compare accuracy and precision results to Table 4 (see page A-126). Note: accuracy and precision limits not listed for PFB derivatives of herbicides.	Extract and analyze a minimum of 4 to 7 replicates of QC check sample, Concentration 5 ug/L. Analyte recovery must be within ±20% of fortified concentration. RSD of measurements must be ≤20%. Perform MDL study	Extract and analyze a minimum of 4 replicates of a QC check sample. Concentration 10-20 times the MDL or at a mid point in the calibration Results must be within 40% of fortified concentration. RSD must be <30%	Extract and analyze 4 replicates of QC check standards. Results must be within ±30% of values specified in Table 6640: VI. (see page A-112) Determine MDL (6)
<b>QC Check Standards/Samples</b>	Analyze one laboratory control sample per 20 samples or each batch of samples whichever is more frequent. Compare results to laboratory established limits.  Laboratory can use 70-130% as interim limits until in house limits are developed.	Analyze one QC check sample at least once a quarter; Concentration should be 10 times the EDL or a conc. which is near the mid point of the calibration. Compare %R to laboratory established limits, if available. If laboratory limits not available analyte recovery must be ± 20% of fortified value. Laboratory established limits must not exceed ±20%.	Analyze one QC check sample (Laboratory Fortified Blank) per 20 samples or one per sample set (all samples extracted within a 24 hr period), whichever is more frequent. Concentration should be 10 times the EDL or a conc. which is near the mid point of the calibration. Compare %R to laboratory established limits, if available. If laboratory limits not available analyte recovery must be ±40% of fortified value. Laboratory established limits must not exceed ±40%.	Analyze one QC check sample (Laboratory Fortified Blank) per 20 samples or one per sample set (all samples extracted within a 24 hr period), whichever is more frequent. Concentration should be within 2 times the concentration listed in Table 6640: VI (see page A-112). Compare %R to laboratory established limits, if available, or Table 6640: VI and VII (see pages A-104)
<b>Method Detection Limit</b>	MDLs listed in Table 1 (see page A -125) for diazo-methane derivatives of herbicides. MDLs for PFB derivatives are not listed in the method.	MDLs and EDLs listed in Table 3. Extract and analyze a minimum of 7 laboratory fortified blanks. Use Table 3 concentrations as guide. Calculate MDL.	MDLs listed in Table 2 (see page A-38).  Extract and analyze a minimum of 7 laboratory fortified blanks. Use Table 2 concentrations as guide. Calculate MDL.	MDLs listed in Table 6640:I (see page A-111).  EQLs measured at approximately 5-10 times the MDL.

Notes:

- (1) EPA Method 515.4, Revision 1.0, April, 2000
- (2) 20th Edition Standard methods for the Examination of Water and Wastewater 1998; the method does not include a base-catalyzed hydrolysis step and is thus not approved for use by EPA.
- (3) Analyte lists may vary among methods; a smaller list in one method is not necessarily a subset of a larger list in another method.
- (4) Initial, one-time, demonstration of ability to generate acceptable accuracy and precision. Procedure may need to be repeated if changes in instrumentation or methodology occur.

Method Comparison

Determination of Chlorinated Herbicides by GC (Continued)

Method ⇒ Parameter ↓	SW-846 Method 8151A	EPA 500 Series Method 515.4	EPA 500 Series Method 515.2	Standard Methods Method 6640
<b>Standard Solution Expiration (5)</b>	Stock standards: Not specified. Working Standard solution: Prepare daily. Calibration standards: Prepare daily	Stock standards: Not specified. Calibration standards: Prepare daily.	Stock standards: Not specified. Calibration standards: Prepare daily.	Stock standards: Not specified. Calibration standards: Prepare daily.
<b>Initial Calibration</b>	<p>Minimum of 5 levels, lowest near but above MDL.</p> <p><b>Option 1:</b> If CF or RF %RSD ≤20 or if mean %RSD of all analytes ≤20 then use avg. CF or RF. Alternatively use a cal. curve (see options below).</p> <p><b>Option 2:</b> If CF or RF %RSD&gt;20 or if %RSD of all analytes &gt;20, then use cal. curve:</p> <p>a) linear cal.using least squares regression;</p> <p>b) linear cal. using weighted least squares regression.</p> <p><b>Requirements:</b> r ≥ 0.99, not forced through origin, do not use origin as a cal. point.</p> <p><b>Option 3:</b> Where instrument response is non-linear over wide working range or above procedures fail acceptance criteria, non-linear cal. may be employed.</p> <p><b>Requirements:</b> COD ≥ 0.99, not forced through origin, do not use origin as a cal. point, other requirements exist pending curve fitting model chosen.</p>	<p>Calibration is by extracting calibration standards from water. 5 levels of calibration are required. Pre-esterified standards may not be used. Lowest concentration calibration standard is at the minimum reporting level.</p>	<p>Calibration levels required:</p> <p>20x range: 3 calibration standards.</p> <p>50x range: 4 calibration standards.</p> <p>100x range: 5 calibration standards.</p> <p>Lowest level should be near but above MDL.</p> <p>If %RSD ≤30, linearity assumed and average RF used.</p> <p>Alternatively use a calibration curve.</p> <p>Calibration standards are to be extracted the same as samples.</p>	<p>Minimum of 3 levels (5 recommended), use calibration levels listed in Table 6640: IV (see page A-111). If %RSD ≤ 20, linearity assumed and average RF used. Alternatively use a calibration curve or a single point calibration. Single point calibration standard must produce response within 20% of unknowns.</p>

**Notes:**

(5) Indicates maximum usage time. If comparisons to QC check standards indicate a problem, more frequent preparation may be necessary.

Method Comparison

Determination of Chlorinated Herbicides by GC (Continued)

Method ⇒ Parameter ↓	SW-846 Method 8151A	EPA 500 Series Method 515.4	EPA 500 Series Method 515.2	Standard Methods Method 6640
<b>Continuing Calibration</b>	<p>A mid-level calibration check standard must be analyzed at the beginning of each 12 hour shift, or more frequently if warranted. If average response of all analytes is not within <math>\pm 15\%</math> of predicted response, recalibrate. Samples which contain target compounds above the reportable limit must be bracketed by an acceptable calibration check standard.</p> <p>Standard RT must fall within daily RT window or system is out of control.</p> <p>Samples injected after criteria was exceeded must be reanalyzed.</p>	<p>A calibration check standard at the minimum reporting level for each analyte must be analyzed at the beginning of analysis day. If not within <math>\pm 50\%</math> of predicted response, recalibrate. A mid- or high- level calibration check standard is then run after each 10 samples. These must be within 70-130% of true value</p> <p>Verify calibration standards at least quarterly using an independent source.</p>	<p>One or more calibration standards analyzed daily. If not within <math>\pm 30\%</math> of predicted response, recalibrate.</p> <p>Derivatized calibration standards can be used up to 14 days.</p> <p>Verify calibration standards at least quarterly using an independent source.</p>	<p>A calibration check standard must be analyzed at the beginning and end of analysis day. If not within <math>\pm 20\%</math> of predicted response, recalibrate and reanalyze samples.</p>
<b>Surrogate Standards</b>	<p>One/two surrogates added to each sample (avoid use of deuterated analogs) 2,4-Dichlorophenylacetic acid (DCAA) at 0.5 mg/L in extract is recommended. Results must fall within laboratory established limits.</p>	<p>2,4-Dichlorophenylacetic acid (DCAA) at 25 ng/mL in sample. %R = 70-130.</p>	<p>2,4-Dichlorophenylacetic acid (DCAA) at 0.1 <math>\mu\text{g/ml}</math> in extract. %R = 60-140.</p>	<p>2,3,5,6-Tetrafluorobenzoic acid %R=70-130 compared to the average daily calibration standard.</p>
<b>Internal Standards</b>	<p>Optional. If used 4,4'-Dibromooctafluorobiphenyl (DBOB) or others if there is DBOB interference. (DBOB) at 0.25 <math>\mu\text{g/mL}</math> in extract.</p>	<p>4,4'-Dibromooctafluorobiphenyl (DBOB) at 0.25 <math>\mu\text{g/mL}</math> in extraction solvent. Sample IS response must be <math>\pm 50\%</math> of calibration response</p>	<p>Optional. If used 4,4'-Dibromooctafluorobiphenyl (DBOB) at .020 <math>\mu\text{g/ml}</math> in extract. Sample IS response must be <math>\pm 30\%</math> of daily standard response.</p>	<p>Optional. If used, 1,2,3-Trichloropropane. Sample IS response must be <math>\pm 30\%</math> of daily standard response.</p>

Method Comparison

Determination of Chlorinated Herbicides by GC (Continued)

Method ⇒ Parameter ↓	SW-846 Method 8151A	EPA 500 Series Method 515.4	EPA 500 Series Method 515.2	Standard Methods Method 6640
<b>Accuracy/ Precision</b>	One MS/MSD per 20 samples or each batch of samples, whichever is more frequent. Compare results to laboratory established limits. (See also: <b>QC Check Standards/Samples</b> .) Laboratory can use 70-130% as interim limits until in-house limits are developed.	One MS (Laboratory Fortified Sample Matrix) per 10 samples or each sample set, whichever is more frequent. Compare %R to laboratory established limits. Recovery should be 70-130%. Duplicates analyzed at a rate of 1 per batch. Analyze a QC sample from an external source at least quarterly. (See also: <b>QC Check Standards/Samples</b> ).	One MS (Laboratory Fortified Sample Matrix) per 10 samples or each sample set, whichever is more frequent. Compare %R to laboratory established limits. Analyze a QC sample from an external source at least quarterly. (See also: <b>QC Check Standards/Samples</b> ).	One MS (Matrix with known additions) per 10 samples or at least one per month. Compare %R to laboratory established limits. Analyze a QC sample from an external source at least quarterly. (See also: <b>QC Check Standards/Samples</b> ).
<b>Blanks</b>	One method blank per extraction batch (up to 20 samples) or when there is a change in reagents, whichever is more frequent.	One method blank with each batch of samples extracted.	One method blank with each batch of samples extracted or when new reagents are used.	One method blank with each batch of samples extracted or when new reagents are used.
<b>Preservation/ Storage Conditions</b>	If residual chlorine present, add sodium thiosulfate. Store at 4 °C. Protect from light.	Sodium sulfite added to sample container.. Store at less than 6 °C in the dark. Store extracts at less than 0°C in the dark.	Adjust pH to <2 by adding HCL at sampling site. Sodium thiosulfate if residual chlorine present (aqueous). Protect from light. Store at 4 °C.	Store at 4 °C. Protect from light. Sodium thiosulfate if residual chlorine present. Adjust pH to <2 at sampling site.
<b>Holding Time (6)</b>	Extraction: 7 days (aqueous). 14 days (solids). Analysis: 40 days after extraction.	Extraction: 14 days. Analysis: 21 days after extraction.	Extraction: 14 days. Analysis: 14 days after extraction.	Extraction: 14 days. Analysis: 14 days after extraction.
<b>Field Sample Amount (7)</b>	1 liter (aqueous). 4 oz. (solid) glass container. Teflon lined top.	40 mL glass container Teflon lined top.	250 mL amber glass container. Teflon lined top.	40 mL glass container Teflon lined top. Collect samples in quadruplicate.
<b>Amount for Extraction</b>	1 liter (aqueous). 30 grams (low level solid). 2 grams (medium level solid).	40 mL	250 mL	30 mL

Notes:

- (6) Unless otherwise indicated, holding times are from the date of sample collection.
- (7) Approximate volumes to be gathered for analysis. Additional volumes are required for the generation of QC data.

Method Comparison

**Determination of Chlorinated Herbicides by GC (Continued)**

Method → Parameter ↓	SW-846 Method 8151A	EPA 500 Series Method 515.4	EPA 500 Series Method 515.2	Standard Methods Method 6640
<b>Other Criteria (Method Specific)</b>	When doubt exists in compound identification, second column or GC/MS confirmation should be used.	Laboratory Performance Check Sample analyzed daily to monitor instrument sensitivity, column performance, and chromatographic performance.  Compound identification, verified by second column confirmation	Instrument Performance Check Sample analyzed daily to monitor instrument sensitivity, column performance, and chromatographic performance.  Compare results to Table 11 (see page A-38). When doubt exists in compound identification, second column confirmation or additional qualitative technique must be used.	Use at least two columns for identification and quantification.  Laboratory Performance Check Sample analyzed daily to monitor instrument sensitivity, column performance, and chromatographic performance. Compare results to Table 6640: VIII (see page A-113).

**Notes:**

- (8) MDL determination for Standard Methods 6000 Series: Analyze a minimum of seven check samples (concentration = 0.2 times MCL or 10 times estimated MDL.). Average percent recovery should be 80% to 120% of true value with %RSD ≤ 35%. Use results to determine MDLs. Broader acceptance ranges exist for some compounds with lower extraction efficiency and are indicated in the specific method.
- (9) For SW-846 Method 8151A, either diazomethane or pentafluorobenzyl bromide may be used for esterification.

Analyte Cross-Reference

Determination of Chlorinated Herbicides by GC

Chemical Name	CAS Number	SW-846 Method 8151A	EPA 500 Series Method 515.4	EPA 500 Series Method 515.2	Std. Methods Method 6640
Acifluorfen	50594-66-6	•	•	•	
Bentazon	25057-89-0	•	•	•	•
Chloramben	133-90-4	•	•		
2,4-D	94-75-7	•	•	•	•
Dalapon	75-99-0	•	•		•
2,4'-DB	94-82-6	•	•	•	
Dacthal	1861-32-1			•	
DCPA acid metabolites	N/A	•	•		
Dicamba	1918-00-9	•	•	•	•
3,5-Dichlorobenzoic acid	51-36-5	•	•	•	
Dichloroprop	120-36-5	•	•	•	
Dinoseb	88-85-7	•	•	•	•
5-Hydroxydicamba	7600-50-2	•		•	
MCPA	94-74-6	•			
MCPP	93-65-2	•			
4-Nitrophenol	100-02-7	•			
Pentachlorophenol	87-86-5	•	•	•	•
Picloram	1918-02-1	•	•	•	•
2,4,5-T	93-76-5	•	•	•	•
2,4,5-TP(Silvex)	93-72-1	•	•	•	•
Quinclorac	84087-01-4		•		

## Analytical Methods - Inorganic Constituents

The numerous analytical methods for inorganic constituent testing have been organized into six major categories:

- Cyanide (total and amenable)
- Organic carbon (total)
- Mercury identified by cold vapor atomic absorption
- Trace metals identified by flame and graphite furnace atomic absorption spectroscopy
- Trace metals identified by inductively coupled plasma technique
- Trace metals identified by inductively coupled plasma/mass spectroscopy

Within each category there is a section called *Method Comparison* that summarizes the key method requirements and components for each testing method. The methods are compared on a side-by-side basis, which allows the user to quickly compare key aspects of the test methods. Where appropriate the user is referred to a table in Appendix A that provides details about an aspect of the test method. The tables in Appendix A are taken directly from the regulations.

For determining if a specific inorganic analyte is analyzed by a test method, each category that identifies more than one specific analyte has an *Analyte Listing* of all analytes addressed by the test methods within the category. This is also presented on a side-by-side basis so that the user can quickly see which method(s) cover the specific inorganic analyte in question.

If you want to find a test method for a specific inorganic analyte but don't know what category(ies) it belongs to, refer to Appendix C. Appendix C is an alphabetical listing of all inorganic analytes specified in the method comparison sections. Use this appendix to identify the category to turn to for additional information.

Method Comparison

Multi-Analyte Analysis of Anions

Method ⇒ Parameter ↓	EPA 300.1 <sup>1</sup>	SW-846 6500 <sup>2</sup>	SW-846 9056A <sup>3</sup>	SM 4110 <sup>4</sup>	SM 4140 <sup>5</sup>
<b>Instrument</b>	Ion Chromatograph	Capillary Electrophoresis	Ion Chromatograph	Ion Chromatograph	Capillary Electrophoresis
<b>Number of Analytes</b>	10 <sup>6</sup>	7	7	6	11
<b>Method Validation</b>	Initial analysis of a Quality Control Sample and determination of MDL for all analytes followed by quarterly re-analysis of the Quality Control Sample and semi-annual MDL determination	None specified	None specified	Assessment of individual capability, performance evaluation samples (Method 4020)	Analyze 7 replicates of a certified performance evaluation sample, mean of each analyte must be within 95% confidence interval for the sample; semi-annual performance of performance evaluation samples
<b>QC Check Standards/Samples</b>	Analysis of a Laboratory Fortified Blank with every batch of samples	One laboratory control sample with each batch of samples	One Laboratory Control sample with each batch of samples; recoveries between 80-120%	Laboratory control samples (Method 4020)	Laboratory control samples (Method 4020)
<b>Method Detection Limit</b>	Determined by 40 CFR 136, Appendix B; MDL range from 0.5 µg/L to 20 µg/L depending on analyte	Depends on injected sample size and sensitivity of detector; nominally <0.1 mg/L	Depends on injected sample size and sensitivity of detector; nominally <0.02 mg/L	Depends on injected sample size and sensitivity of detector; nominally <0.1 mg/L	Depends on injected sample size; nominally <0.1 mg/L
<b>Standard Solution Expiration</b>	Chlorite 2 weeks; nitrite and phosphate 1 month; all others 6 mos; fresh working standards prepared daily	One month for nitrite; others not specified	One month when stored at 4C; fresh working standards prepared weekly	At least one month; prepare working standards fresh daily	At least three months; prepare working standards fresh daily

<sup>1</sup> Revision 1.0, with errata sheet, April, 1999

<sup>2</sup> Update IVA, January, 1998

<sup>3</sup> Update IVB, November, 2000

<sup>4</sup> 20th Edition, 1998

<sup>5</sup> 20th Edition, 1998

<sup>6</sup> Combined analytes of Methods 300.0 Part A and 300.0 Part B

## **Sampling Procedures and Preparation**

This section provides a tabulation of environmental sample parameters including method references, sample amounts required for analysis, container specifications, sample preservation requirements, and extraction and analysis hold times. This information is useful in maintaining sample integrity as well as compliance with correct sampling procedures as specified in EPA regulation.

Also provided in this section is a summary of sample extraction and sample cleanup methods approved for various sample matrix types for each of the analytical methods listed. The user should note that for EPA SW-846 the extraction and clean-up methods are cited with their own method numbers.

## Sample Extraction and Clean-up Methods for Municipal and Industrial Discharge

EPA Method Name	Method #	Preparation and Extraction				Cleanup Methods			
		Purge-and-Trap	Liquid/Liquid Extraction	Base Wash	Derivatization	Florisil Clmn Clean-up	Alumina Clmn Clean-up	Silica Gel Clmn Clean-up	Sulfur Clean-up (Hg)
Purgeable Halocarbons	601	•							
Purgeable Aromatics	602	•							
Acrolein and Acrylonitrile	603	•							
Phenols	604		•		•			•	
Hexachlorophene and Dichlorophen	604.1*		•	•					
Benzidines	605		•						
Phthalate Esters	606		•			•	•		
Nitrosamines	607		•			•	•		
Organochlorine Pesticides and PCBs	608		•			•			•
Organochlorine Pesticides	608.1		•			•			
Organochlorine Pesticides	608.2		•			•		•	
Nitroaromatics and Isophorone	609		•			•			
Polynuclear Aromatic Hydrocarbons	610		•					•	
Haloethers	611		•			•			
Chlorinated Hydrocarbons	612		•			•			
2,3,7,8-Tetrachlorodibenzo-p-dioxin	613		•	•			•	•	
Organophosphorus Pesticides	614*		•			•			•
Organophosphorus Pesticides	614.1*		•					•	
Chlorinated Herbicides	615*		•		•				
Carbon, Hydrogen, and Oxygen containing pesticides	616*		•					•	
Organohalide pesticides and PCBs	617*		•			•			•
Volatile Pesticides	618*		•						
Triazine Pesticides	619*		•			•			
Diphenylamine	620*		•					•	
Organophosphorus Pesticides	622*		•						
Purgeables	624	•							
Base Neutrals and Acids	625		•						
Dinitroaniline Pesticides	627*		•						
Cyanazine	629*		•						
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Dithiocarbamate Pesticides	630.1 <sup>(1)</sup>								

# APPENDIX A

This is a compilation of some of the tables, exhibits, etc., found in the methods. If the method comparison refers to a specific table, that table will be found in this appendix. Only tables and exhibits referred to in the method comparison are found in this appendix.

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## Appendix B

All organic analytes mentioned in any of the test methods covered in the method comparison sections in this reference are listed in alphabetical order followed by their CAS number. If the analyte is included in one of the listed test methods it is marked with a “•” or one of the following letters in the appropriate column. “S” for Surrogate compounds, “I” for Internal Standard compounds, or “B” if the compound is both a Surrogate and an Internal Standard.

## Organic Analyte Cross-Reference to Methods

**Key:**

S = Surrogate

I = Internal Standard

B = Both Surrogate and Internal  
Standard

Analyte	CAS Number	Herbicides				Pesticides & PCBs						Semivolatile Organic						
		SW846 - 8151A	EPA 500 - 515.4	EPA 500 - 515.2	Std. Methods 6640	SW846 - 8081B	SW846 - 8082A	EPA500 - 508	EPA600 - 608	Std. Methods 6630	CLP-PEST - MC	SW846 - 8270D	EPA500 - 525.2	EPA600 - 625	Std. Methods 6410	CLP - SVOA - MC	CLP - SVOA - LC	CLP - PEST - LC
Acenaphthene	83-32-9											•						
Acenaphthene d10	15067-26-2											—	—					
Acenaphthylene	208-96-8											•	•	•	•	•	•	
Acetone	67-64-1																	
Acetonitrile	75-05-8																	
Acetophenone	98-86-2											•		•		•	•	
2-Acetylaminofluorene	53-96-3											•						
1-Acetyl-2-thiourea	591-08-2											•						
Acifluorfen	50594-66-6	•	•	•														
Acrolein	107-02-8																	
Acrylonitrile	107-13-1																	
Alachlor	15972-60-8					•							•					
Aldrin	309-00-2					•	•	•	•	•	•	•	•	•				•
Allyl alcohol	107-18-6																	
Allyl chloride	107-05-1																	
Ametryn	834-12-8												•					
2-Aminoanthraquinone	117-79-3											•						
Aminoazobenzene	60-09-3											•						
4-Aminobiphenyl	92-67-1											•						
3-Amino-9-ethylcarbazole	132-32-1											•						
Anilazine	101-05-3											•						
Aniline	62-53-3											•		•				
Aniline d5	4165-61-1													B	B			
o-Anisidine	90-04-0											•						
Anthracene	120-12-7											•	•	•	•	•	•	
Anthracene d10	1719-06-8													B	B			
Aramite	140-57-8											•						
Aroclor-1016	12674-11-2					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1221	11104-28-2					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1232	11141-16-5					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1242	53469-21-9					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1248	12672-29-6					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1254	11097-69-1					•	•	•	•	•	•	•	•	•	•			•
Aroclor-1260	11096-82-5					•	•	•	•	•	•	•	•	•	•			•
Atraton	1610-17-9											•						
Atrazine	1912-24-9											•			•	•		
Azabenzene	110-86-1																	
Azinphos methyl	86-50-0											•						
Barban	101-27-9											•						

Organic Analyte Cross-Reference to Methods

Volatile Organic by GC/MS						Volatile Organic by GC						Analyte					
SW846 - 8260B	EPA500 - 524.2	EPA600 - 624	Std. Methods 6210B	Std. Methods 6210C	Std. Methods 6210D	CLP - VOA - MC	CLP - VOA - LC	EPA600 - 602	Std. Methods 6220B	Std. Methods 6220C	SW-846 - 8021B		EPA500 - 502.2	EPA600 - 601	Std. Methods 6230B	Std. Methods 6230C	Std. Methods 6230D
																	Acenaphthene
																	Acenaphthene d10
																	Acenaphthylene
	•	•					•	•									Acetone
	•																Acetonitrile
																	Acetophenone
																	2-Acetylaminofluorene
																	1-Acetyl-2-thiourea
																	Acifluorfen
	•																Acrolein
	•	•															Acrylonitrile
																	Alachlor
																	Aldrin
	•																Allyl alcohol
	•	•									•						Allyl chloride
																	Ametryn
																	2-Aminoanthraquinone
																	Aminoazobenzene
																	4-Aminobiphenyl
																	3-Amino-9-ethylcarbazole
																	Anilazine
																	Aniline
																	Aniline d5
																	o-Anisidine
																	Anthracene
																	Anthracene d10
																	Aramite
																	Aroclor-1016
																	Aroclor-1221
																	Aroclor-1232
																	Aroclor-1242
																	Aroclor-1248
																	Aroclor-1254
																	Aroclor-1260
																	Atraton
																	Atrazine
																	Azabenzene
																	Azinphos methyl
																	Barban

## Inorganic Analyte Cross-Reference to Methods

**APPENDIX C**

Inorganic analytes mentioned in the categories of Trace Metals identified by Flame and Graphite Furnace Atomic Absorption Spectroscopy, Trace Metals identified by Inductively Coupled Plasma (ICP), and Trace Metals identified by Inductively Coupled Plasma/Mass Spectroscopy are listed in alphabetical order. If the analyte is included in one of the listed test methods it is noted with a "\*" in the appropriate column.

**Determination of Total Trace Metals Cross-Reference**

Metal	Symbol	CAS	Flame and Graphite Furnace AA Spect. (p. 74)				ICP (p. 80)				ICP-MS (p. 86)	
			SW-846 7000 Series	EPA 200 Series	Std Methods 3000 Series	CLP Inorgan Method	SW-846 6010C	EPA 200.7	Std. Methods 3120	CLP Inorgan Method	SW-846 6020A	EPA 200.8
Aluminum	Al	7429-90-5	*	*	*		*	*	*	*	*	*
Antimony	Sb	7440-36-0	*	*	*	*	*	*	*	*	*	*
Arsenic	As	7440-38-2	*	*	*	*	*	*	*	*	*	*
Barium	Ba	7440-39-3	*	*	*		*	*	*	*	*	*
Beryllium	Be	7440-41-7	*	*	*	*	*	*	*	*	*	*
Bismuth	Bi	7440-69-9			*							
Boron	B	7440-42-8					*	*	*			
Cadmium	Cd	7440-43-9	*	*	*	*	*	*	*	*	*	*
Calcium	Ca	7440-70-2	*	*	*	*	*	*	*	*	*	
Cerium	Ce	7440-45-1						*				
Cesium	Cs	7440-46-2			*							
Chromium	Cr	7440-47-3	*	*	*	*	*	*	*	*	*	*
Cobalt	Co	7440-48-4	*	*	*		*	*	*	*	*	*
Copper	Cu	7440-50-8	*	*	*		*	*	*	*	*	*
Gold	Au	7440-57-5		*	*							
Iridium	Ir	7439-88-5		*	*							
Iron	Fe	7439-89-6	*	*	*		*	*	*	*	*	
Lead	Pb	7439-92-1	*	*	*	*	*	*	*	*	*	*
Lithium	Li	7439-93-2	*		*		*	*	*			
Magnesium	Mg	7439-95-4	*	*	*	*	*	*	*	*	*	
Manganese	Mn	7439-96-5	*	*	*		*	*	*	*	*	*
Mercury(a)	Hg	7439-97-6	*	*	*	*	*	*		*	*	*
Molybdenum	Mo	7439-98-7	*	*	*		*	*	*			*
Nickel	Ni	7440-02-0	*	*	*		*	*	*	*	*	*
Osmium	Os	7440-04-2	*	*	*							
Palladium	Pd	7440-05-3		*	*							
Phosphorus	P	7723-14-0					*	*				
Platinum	Pt	7440-06-4		*	*							
Potassium	K	7440-09-7	*	*	*	*	*	*	*	*	*	
Rhenium	Re	7440-15-5		*	*							

## APPENDIX D

### **STATUS TABLES FOR SW-846, THIRD EDITION AND DRAFT UPDATES IVA AND IVB REVISED NOVEMBER 2000**

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

Use the "SW-846 Method Status Table" as a reference guide to identify the historical and current status of published SW-846 methods. It also identifies the current location of the method in the manual. Methods in this status table are listed sequentially by method number. This order is different from that in the hard copy manual, where the methods are grouped by purpose and included as part of one of the chapters.

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

## APPENDIX E

Contained in this section are definitions and explanations of terms and abbreviations used in this book or commonly in use in this field.

**AA.** Atomic Absorption.

**Absorb.** To soak up. The incorporation of a liquid into a solid substance, as by capillary, osmotic, solvent, or chemical action.

**Absorbance.** A measure of the decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \frac{I(\text{solvent})}{I(\text{solution})} = \log \frac{I_0}{I}$$

Where,  $I$  = radiation intensity

**Absorption.** To absorb. The process of incorporating a substance (liquid or gas) into the body of another substance (solid).

**Accuracy.** Accuracy means the nearness of a result or the mean ( $\bar{x}$ ) of a set of results to the true value. Accuracy is assessed by means of reference samples and percent recoveries.

**Acid.** An inorganic or organic compound that 1) reacts with metals to yield hydrogen; 2) reacts with a base to form a salt; 3) dissociates in water to yield hydrogen ions; 4) has a pH of less than 7.0; and 5) neutralizes bases or alkalies. All acids contain hydrogen and turn litmus paper red. They are corrosive to human tissue and are to be handled with care. See Base; pH.

**Action Levels.** Typically action levels are considered regulatory levels set by federal government agencies such as the EPA, the Food and Drug Administration (FDA), and the U.S. Department of Agriculture. The presence of a contaminant in high enough concentration to warrant action or a response under regulatory guidelines.

**Adsorb.** To attract and retain gas, liquid, or dissolved substances on the surface of another material.

**Adsorbtion.** To adsorb. Adhesion of molecules of gas, liquid, or dissolved solids to a surface. Adsorbtion is a surface phenomenon.

**Aliquot.** A measured portion of a sample taken for analysis.

**Alkali.** Any compound having highly basic properties; i.e., one that readily ionizes in aqueous solution to yield OH anions, with a pH above 7, and turns litmus paper blue. Examples are oxides and hydroxides of certain metals belonging to group IA of the periodic table (Li, Na, K, Rb, Cs, Fr). Ammonia and amines may also be alkaline. Alkalies are caustic and dissolve human tissue. Treat alkali burns by quickly washing the affected area with large amounts of water for at least 15 min. Common commercial alkalies are sodium carbonate (soda ash), caustic soda and caustic potash, lime, lye, waterglass, regular mortar, Portland cement, and bicarbonate of soda. See Acid; Base; pH.

**Alpha Particle.** A particle emitted from radioactive decay. The particle is a helium nucleus consisting of two neutrons and two protons.

**Ambient.** Usual or surrounding conditions of temperatures, humidity, etc.

**Analysis Date/Time.** The date and military time (24 hour clock) of the introduction of the sample, standard, or blank into the analysis system.

**Analyte.** The element or ion compound an analyst seeks to determine; the element of interest.

**Analytical Batch.** The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g. groundwater, sludge, ash, etc.)

## APPENDIX F

Source information used to compile this manual is the latest promulgated versions of methods commonly available to analytical testing laboratories and environmental engineering companies. In some cases newer proposed versions of methods exist. These are not included in this reference because their use is not universally accepted. The latest versions of the inorganic and organic Statements of Work for the Contract Laboratory Program were used, though some laboratories may still have contracts using an older Statement of Work version. SW-846 third edition, methods, including promulgated updates to December 1996, are included in this reference. Appendix D is a detailed explanation of SW-846 third edition and its updates.

### REFERENCES

#### U.S. Environmental Protection Agency

**National Primary Drinking Water Regulations** – 40 CFR Part 141; July 1, 1988.

**Contract Laboratory Program – User's Guide to the Contract Laboratory Program.** EPA/540/P-91/002, January 1991, Office of Emergency and Remedial Response. Washington, DC

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**EPA100-400 Series** – Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

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**EPA Method 300** – Revision 1.0, with errata sheet, April, 1999, available at [www.epa.gov](http://www.epa.gov)

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**EPA Method 515.4** – Revision 1.0, April, 2000, available at [www.epa.gov](http://www.epa.gov)

**EPA 600 Series** – 40 CFR, Part 136, Revised as of July 1, 1995. Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

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