Protocol of Accepted Drinking Water Testing Methods

Version 2.0

Protecting our environment.

Ontario

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Laboratory Services Branch
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1. INTRODUCTION

This document sets out testing methods that may be used for the conduct of tests of Ontario drinking water. Under the *Safe Drinking Water Act, 2002*, only licensed laboratories located in Ontario and eligible out-of-province laboratories may conduct tests on samples of drinking water. In addition, a laboratory must be accredited for the selected method by: the Standards Council of Canada (SCC); by the Canadian Association for Laboratory Accreditation (CALA); by an accreditation body that, in the Director's opinion, is equivalent to the SCC; or the laboratory must be authorized by the Director to conduct a particular test without accreditation, in accordance with the provisions of the Act. In addition to these requirements, a laboratory must use validated methods for the parameters outlined in this document, to conduct drinking water tests, including demonstrating that they meet or exceed the required **reporting detection limit (RDL)**.

The methods that follow are derived from one of five sources:

1. Methods used by the Ministry of the Environment (MOE) Laboratory Services Branch (LaSB) for the testing of drinking water for which the laboratory has been accredited through the SCC or CALA. Requests for these methods can be sent to LaboratoryServicesBranch@ontario.ca. These methods are indicated as **LaSB Method.**

2. Methods as described in the reference Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, and 21st Edition, 2005, American Public Health Association, American Waterworks Association (AWWA), Water Environmental Federation. These methods are indicated as **AWWA Method.**

3. Methods of the United States Environmental Protection Agency (US EPA) available through the USEPA (www.epa.gov), the National Technical Information Service, Springfield, Virginia, or the National Environmental Methods Index (www.nemi.gov). These methods are indicated as **US EPA Method.**

4. Methods of ASTM International (formerly the American Society for Testing and Materials) (ASTM), Vol. 11.01 and 11.02, 2008, available from ASTM International, www.astm.org. These methods are indicated as **ASTM Method.**

5. Methods approved by AOAC International (formerly the Association of Official Analytical Chemists) in the "Official Methods of Analysis of AOAC International", 18th edition, revision 2 2007, revision 3 2010, available from AOAC International, www.aoac.org. These methods are indicated as **AOAC Method**.

A laboratory that uses a validated, accredited method derived from a source different from those above, must receive Ministry approval by the Director in accordance with the Act. The laboratory must provide a copy of the test procedure used, validation data for the method demonstrating that they meet or exceed the required **reporting detection limit (RDL)**, and any other documentation requested which demonstrates that the method is suitable and appropriate for use on drinking water matrices. If it is determined that the test is suitable and appropriate, the Director may add the test to this protocol as an accepted testing method, or may authorize a specific laboratory under its licence to conduct the test as permitted under the Act. Details of these requirements are provided in the document *Ministry of the Environment Protocol for Acceptance of Alternate Methods (PAAM), Version 1.4, January 2005*, PIBS #5297e, available on the MOE internet site at http://www.ontario.ca/drinkingwater/stel01_046888.pdf.

The methods are listed by the type of parameter for which each method may be used and grouped as listed in the Schedules of Ontario Regulation (O. Reg.) 169/03. Also listed are additional parameters that may be required under a Certificate of Approval, an Order or another directive issued by the MOE. The standard for each parameter, as set out in the Ontario Drinking Water Quality Standards (ODWQS) (O. Reg. 169/03), is set out first, followed by the RDL. The methods are grouped as follows: Schedule 1, Microbiological Parameters; Schedule 2, Chemical Parameters; Additional Parameters (Certificate of Approval or Ministry Orders). Not all of the additional parameters have limits or RDLs available.

NOTE: The units for the ODWQS are those listed in O. Reg. 169/03. The units for RDL are in the required reporting units for the Drinking Water Information System (DWIS).

2. SCHEDULE 1, MICROBIOLOGICAL PARAMETERS

2.1. Total Coliforms

PARAMETER	**ODWQS**	
Methodology	Membrane Filtration	Presence/Absence
Total Coliforms	0 CFU/100 mL	Absent/100 mL

Reporting units for Total Coliforms are dependant on the methodology used. The ODWQS values provided in the table above include the appropriate reporting unit for each methodology. (See Section 5, Acronyms for a description of the reporting units.)

Method Principle: The principle of the method described refers to all of the methods listed for Total Coliforms (including detection and/or enumeration). A measured volume of water (100 mL) is analyzed quantitatively (using an enumeration technique such as membrane filtration) or qualitatively (using the Presence-Absence [P-A] procedure, or equivalent). Incubation for Total Coliforms is at 35.5°C for 24±2 hours (or 48±3 hours, for P-A tests).

Coliforms are classically described as being Gram-negative, non spore-forming, rod-shaped bacteria which ferment lactose to produce acid and gas within 48 hours. Coliforms are commonly isolated from plants, soils and sediments. These bacteria are members of the Family *Enterobacteriaceae*. One enteric bacteria (*Escherichia coli*), is associated with intestines of warm-blooded mammals (including humans) and is commonly isolated from faecal material.

The following LaSB methods apply to the testing of <u>both</u> Total Coliforms (this section) and *Escherichia coli (E. coli)* (Section 2.2 below). They are repeated in both sections.

LaSB Methods: Method E3407 – Membrane Filtration Method Using DC-Agar for the Simultaneous Detection of Total Coliforms and *Escherichia coli*

Method E3226 – Detection of Coliform Bacteria (including *Escherichia coli*) and Other Indicators of Deteriorating Water Quality in Drinking Water by the Presence-Absence Procedure

Method E3371 – Membrane Filtration Method for the Detection and Enumeration of Total Coliforms, *Escherichia coli, Pseudomonas aeruginosa*, and Fecal Streptococci in Environmental Samples

AWWA Methods:[1] Method 9221 – Multiple-Tube Fermentation Technique for Members of the Coliform Group [excluding 9221 E – Fecal Coliform Procedure]

Method 9222 – Membrane Filter Technique for Members of the Coliform Group [excluding 9222 C – Delayed-Incubation Total Coliform Procedure and 9222 E – Delayed-Incubation Fecal Coliform Procedure]

Method 9223 – Enzyme Substrate Coliform Test

US EPA Methods: Method 1604, Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

Enzyme substrate methods as approved by the EPA, 40 CFR Part 122,136, et al, March 12, 2007

AOAC Method: Method CCA-210, Combined *E. coli* and Total Coliform Test (AOAC certificate number 010801)

2.2. *Escherichia coli*

PARAMETER	**ODWQS**	
Methodology	Membrane Filtration	Presence/Absence
Escherichia coli (E. coli)	0 CFU/100 mL	Absent/100 mL

Reporting units for *E. coli* are dependant on the methodology used. The ODWQS values provided in the table above include the appropriate reporting unit for each methodology.

Method Principle: *Escherichia coli* are Gram-negative, facultatively anaerobic, non spore-forming, lactose-fermenting, rod-shaped bacteria which are oxidase negative. They produce indole from tryptophan and acids during the fermentation of sugars, giving a positive methyl red test. They do not, however, produce acetylmethylcarbinol as an end product of fermentation thus, giving a negative Vogues-Proskauer test. Furthermore, they cannot utilize citrate as the sole source of carbon for growth. In addition, most *E. coli* strains (approximately 95%) have the unique ability, among the lactose-fermenting members of the family Enterobacteriaceae, to produce the enzyme ß-D-glucuronidase.

Historically, the presence of both *E. coli* and/or faecal coliforms has been described as an indication of sewage or faecal contamination as they are commonly isolated from the intestinal tract of warm-blood animals. Currently, *E. coli* is considered to be the most specific indicator of faecal contamination in the assessment of water quality for the following reasons:

[1] *Standard Methods, 20th and 21st Ed.*

1) *E. coli* isolates are normally present in the feces of warm-blooded animals (including humans) at higher densities (10^7 to 10^8 cells per gram) than other lactose-fermenting members of the family *Enterobacteriaceae*.

2) *E. coli* isolates may persist for periods of time, once outside the human or warm-blooded animal intestine but they generally do not multiply in water or wastewater in temperate climates.

The following LaSB Methods apply to the testing of both Total Coliforms (Section 2.1 above) and *E. coli* (this section). They are repeated in both sections.

LaSB Methods:	Method E3407 – Membrane Filtration Method Using DC Agar for the Simultaneous Detection of Total Coliforms and *Escherichia coli.*
	Method E3226 – Detection of Coliform Bacteria (including *Escherichia coli*) and Other Indicators of Deteriorating Water Quality in Drinking Water by the Presence-Absence Procedure
	Method E3371 – Membrane Filtration Method for the Detection and Enumeration of Total Coliforms, *Escherichia coli, Pseudomonas aeruginosa*, and Fecal Streptococci in Environmental Samples
AWWA Methods: [2]	Method 9221 – Multiple-Tube Fermentation Technique for Members of the Coliform Group [excluding 9221 E – Fecal Coliform Procedure]
	Method 9222 – Membrane-Filter Technique for Members of the Coliform Group [excluding 9222 C – Delayed-Incubation Total Coliform Procedure and 9222 E – Delayed-Incubation Fecal Coliform Procedure]
	Method 9223 – Enzyme Substrate Coliform Test
US EPA Methods:	Method 1103.1, *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC)
	Method 1603, *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC)
	Method 1604, Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)
	Enzyme substrate methods as approved by the EPA, 40 CFR Part 122,136, et al, March 12, 2007
ASTM Method:[3]	Method D5392-93 (2006), Standard Test Method for Isolation and Enumeration of *E. coli* in Water by the Two-Step Membrane Filtration Procedure
AOAC Method:	Method CCA-210, Combined *E. coli* and Total Coliform Test (AOAC certificate number 010801)

[2] *Standard Methods, 20th and 21st Ed.*
[3] Vol. 11.02, 2008

2.3. Heterotrophic Plate Count

PARAMETER	Technique	Reporting Units
Heterotrophic Bacteria (Heterotrophic Plate Count)	Spread Plate Pour Plate Membrane Filtration	Count/CFU/0.1 mL Count/CFU/1 mL Count/CFU/1 to 100 mL

NOTE: The requirement to report Hetertrophic Plate Count as part of the Ontario Drinking Water Quality Standards was revoked in June 2006 (O. Reg. 169/03 as amended to O. Reg. 248/06, s. 1). However, an individual drinking water system may be required to report this parameter in a Ministry Order or Certificate of Approval.

LaSB Method: Method E3408 – The Spread Plate Method for the Enumeration of Aerobic, Heterotrophic Bacteria in Drinking Water

Method Principle: Plate count agar is inoculated with a volume of sample (0.1 mL). A turntable and spreader are then used to ensure consistent spreading of the inoculum over the entire surface of the agar plate. The plates are then inverted and incubated at 35±0.5°C for 48±3 hours.

Heterotrophic bacteria capable of growing in the presence of air will produce colonies (CFU's) on the surface of the agar over the course of an incubation period of 48±3 hours at 35±0.5°C. All bacterial colonies which appear at the end of the 48±3 hour incubation period are counted. This count per plate represents the number of heterotrophic bacterial colony forming units per 0.1 mL of sample. The count is then divided by the volume of the inoculum (0.1 mL) to obtain the number of colony forming units (bacterial count) per mL.

AWWA Method: [4] Method 9215 – Heterotrophic Plate Count

2.4. Clostridium

PARAMETER	
Clostridium perfringens	P/A/100 mL

LaSB Method: N/A

ASTM Method:[5] D5916-96 (2002), Standard Test Method for Detection and Enumeration of *Clostridium perfringens* from Water and Extracted Sediments by Membrane Filtration (MF)

[4] *Standard Methods, 20th and 21st Ed.*
[5] Vol. 11.02, 2008

2.5. Cryptosporidium

PARAMETER	
Cryptosporidium	P/A/100 mL

LaSB Method: N/A

AWWA Methods: [6] Method 9711 B – *Giardia* and *Cryptosporidium* Methods

US EPA Methods: Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA

Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA

ICR Protozoan Method for Detecting *Giardia* Cysts and *Cryptosporidium* Oocysts in Water by a Fluorescent Antibody Procedure (EPA/814-B-95-003, June 1995)

3. SCHEDULE 2, CHEMICAL PARAMETERS

3.1. Volatile Organic Compounds (VOCs)

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
1,1-Dichloroethylene	75-35-4	0.014	1.4
1,2-Dichlorobenzene	95-50-1	0.2	20
1,2-Dichloroethane	107-06-2	0.005	0.5
1,4-Dichlorobenzene	106-46-7	0.005	0.5
Benzene	71-43-2	0.005	0.5
Carbon Tetrachloride	56-23-5	0.005	0.5
Dichloromethane	75-09-2	0.05	5
Ethylbenzene *	100-41-4	0.0024 *	1.2
Monochlorobenzene	108-90-7	0.08	8
Tetrachloroethylene (perchloroethylene)	127-18-4	0.03	3
Toluene *	108-88-3	0.024 *	2.4
Trichloroethylene	79-01-6	0.005	0.5
Trihalomethanes: Bromoform	75-25-2	0.1	10 **
Trihalomethanes: Bromodichloromethane	75-27-4	0.1	10 **
Trihalomethanes: Chloroform	67-66-3	0.1	10 **
Trihalomethanes: Chlorodibromomethane	124-48-1	0.1	10 **
Xylene, Total *	n/a	0.3 *	150
Xylene, -ortho *	95-47-6		0.5
Xylene, -m/p *	108-38-3/106-42-3		0.5
Vinyl Chloride	75-01-4	0.002	0.2

* These parameters are not part of Schedule 2 in O. Reg. 169/03, but may be found in individual Certificates of Approval or Ministry Orders. The methods for analysis for these parameters fall into the same category as those volatile organic compounds listed in Schedule 2.

[6] *Standard Methods, 21st Ed.*

** RDL applies to the sum of bromoform, bromodichloromethane, chloroform and chlorodibromomethane to determine Total Trihalomethanes.

LaSB Method:	E3144 – The Determination of Volatile Organic Compounds in Raw and Treated Drinking Water by Purge and Trap Capillary Gas Chromatography-Flame Ionization Detection/Mass Selective Detection (GC-FID/MSD)
Method Principle:	Volatile organic compounds (VOCs) in water are determined by purge-and-trap technique, followed by capillary gas chromatography (GC) with simultaneous flame ionization and quadrupole mass spectrometric detection (MSD). The VOCs are purged from the sample with a stream of helium at ambient temperature onto an adsorbent trap. They are then thermally desorbed and, prior to being introduced into the oven of the GC, are cryo-focused with liquid nitrogen at the head of the capillary column. Target compounds are quantified by a linear, dual point, external standard primary FID method and secondary target ion MSD method and identified on the basis of retention time conformity and the presence of MS qualifier peaks of selected m/z values.
AWWA Methods: [7]	Method 6200 B – Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method
	Method 6200 C – Purge and Trap Capillary-Column Gas Chromatographic Method
US EPA Methods:	Method 502.2, Rev 2.1, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series
	Method 524.2, Rev 4.1, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry
	Method 524.3, Version 1.0, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry
	Method 551.1, Rev 1.0, Chlorinated Disinfection By-Products and Chlorinated Solvents by Liquid-Liquid Extraction and GC with an Electron Capture Detector
	SW-846, Method 5030C, Purge-and-Trap for Aqueous Samples
	SW-846, Method 8021B, Aromatic and Halogenated Volatiles by Gas Chromatography using Photoionization and/or Electrolytic Conductivity Detectors
	SW-846, Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
ASTM Methods:[8]	Method D3973-85(2003), Standard Test Method for Low-Molecular Weight Halogenated Hydrocarbons in Water

[7] *Standard Methods, 20th and 21st Ed.*
[8] Vol. 11.02, 2008

Method D5790-95(2006), Standard Test Method for Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry

3.2. Trace Metals

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Antimony (Sb)	7440-36-0	0.006	0.6
Arsenic (As)	7440-38-2	0.025	2.5
Barium (Ba)	7440-39-3	1.0	100
Boron (B)	7440-42-8	5.0	500
Cadmium (Cd)	7440-43-9	0.005	0.5
Chromium (Cr)	7440-47-3	0.05	5
Lead (Pb)	7439-92-1	0.010	2
Selenium (Se)	7782-49-2	0.01	5
Uranium (U)	7740-61-1	0.02	10

LaSB Method: E3473 – The Determination of Trace Metals in Potable Waters by Dynamic Reaction Cell (DRC) Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)

Method Principle: Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is an analytical technique for trace element determinations in a variety of sample matrices. An inductively coupled argon plasma (ICP) is used as the ion source. A quadrupole mass spectrometer measures the number of ions on the basis of their respective mass-to-charge ratio (m/z). A Dynamic Reaction Cell (DRC) also consists of a cell containing an additional quadrupole, with reaction cell gases (ammonia, NH_3, and methane, CH_4, are used in this method) added to break up interferences present in the samples. Typical interferences that DRC mode is used to break up are $^{38}Ar^{14}N^{1}H$ on ^{51}V, $^{40}Ar^{35}Cl$ on ^{75}As, $^{40}Ar^{37}Cl$ on ^{77}Se, $^{40}Ar^{12}C$ on ^{52}Cr, $^{40}Ar^{14}N$ on ^{54}Fe, $^{40}Ar^{16}O^{1}H$ on ^{57}Fe.

LaSB Method: E3051 – The Determination of Trace Metals in Potable Waters by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) (2007)

Method Principle: Inductively coupled plasma-mass spectroscopy (ICP-MS) is used for the detection of trace metals in drinking water samples. An inductively coupled argon plasma (ICAP or ICP) is used as an ion source to directly aspirate an aliquot of sample. A quadrupole mass spectrometer measures the number of ions on the basis of their respective mass to charge ratios (m/z).

Other Methods:

The following methods have been developed by the US EPA, AWWA, and ASTM for the analysis of various trace elements in drinking water. This list is not all-inclusive, and similar methods from other sources may also be applicable to the analysis of trace elements in drinking water. To be acceptable to

MOE for the analysis of Ontario drinking water samples, the laboratory must demonstrate that the method used meets or exceeds the applicable RDL listed in the table above.

Method	Sb	As	Ba	B	Cd	Cr	Pb	Se	U
US EPA – 200.5, Rev 4.2	√	√	√	‡	√	√	√	√	√
US EPA – 200.7, Rev 4.4		‡	√	‡	√	√			
US EPA – 200.8, Rev 5.4	√	√	√		√	√	√	√	
US EPA – 200.9, Rev 2.2	√ *	√			√	√	√	√	
US EPA – 200.15, Rev 1.2			†	†	†	†		†	
US EPA – Metals (AAS – Direct)		†	†					†	
US EPA – Metals (AAS – Furnace)		†	†		†	†	†	†	
US EPA – 206.2		†							
US EPA – 206.3		†							
US EPA – 208.1			†						
US EPA – 208.2			†						
US EPA – 212.3				†					
US EPA – 213.2					†				
US EPA – 218.2						†			
US EPA – 218.3						†			
US EPA – 239.2							†		
US EPA – 270.2								†	
US EPA – 270.3								†	
US EPA – 6010C			†	†		†			
US EPA – 6020A	†	†	†		†	†	†		
US EPA – 7000B (AAS)			†						
US EPA – 7000B (GF-AAS)		†	†		†	†	†		
US EPA – 7061A		†							
US EPA – 7062		†							
US EPA – 7063		†							
US EPA – 7080A			†						
US EPA – 7081			†						
US EPA – 7131A					†				
US EPA – 7191						†			
US EPA – 7421							†		
US EPA – 7740								†	
US EPA – 7741A								†	
Standard Methods 20th/21st Ed – 3120B		‡	√	‡	‡	√	‡	‡	
Standard Methods 20th/21st Ed – 3111B					‡	‡		‡	
Standard Methods 20th/21st Ed – 3111D			√						
Standard Methods 20th/21st Ed – 3113B	√	√	√		√	√	√	√	
Standard Methods 20th 21st Ed – 3114B		√						√	
Standard Methods 20th 21st Ed – 3114C		‡						‡	
Standard Methods 20th/21st Ed – 3125B	‡	‡	‡		‡	‡	‡	‡	√
Standard Methods 20th/21st Ed – 3130B							‡		
Standard Methods 20th/21st Ed – 4500B				‡					
Standard Methods 20th/21st Ed – 4500C				‡					
ASTM – D1687-02C						‡			

Method	Sb	As	Ba	B	Cd	Cr	Pb	Se	U
ASTM – D1976-07		‡*		‡	‡*	‡*	‡*	‡*	
ASTM – D2972-03B		√							
ASTM – D2972-03C		√							
ASTM – D3082-03				‡					
ASTM – D3559-03C							‡		
ASTM – D3559-03D							√ *		
ASTM – D3697-07	√								
ASTM – D3859-03A								√	
ASTM – D3859-03B								√	
ASTM – D3919-04		‡		‡	‡	‡	‡	‡	
ASTM – D4382-02			‡						
ASTM – D4691-02			‡		‡	‡		‡	
ASTM – D5174-07									√
ASTM – D5673–05	‡	‡	‡		‡	‡	‡	‡	√

Legend: √ Recommended by US EPA ‡ Available in Standard Methods 20th and 21st Ed., or from ASTM
† Availabe in US EPA SW-846 or EPA 600/4-79/020 or EPA 600/R-94/111

* See NOTE at method citation below.

US EPA Methods:

Method 200.5, Rev 4.2, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma – Atomic Emission Spectrometry

Method 200.7 Rev 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma -Atomic Emission Spectrometry

Method 200.8 Rev 5.4, Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry

Method 200.9 Rev 2.2, Determination of Trace Elements by Stabilized Temperature Graphite Furnace AA Spectrometry;

NOTE: This method is approved by the US EPA but the published MDL is higher than the MOE required RDL. Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL

Method 200.15 Rev 1.2, Determination of Metals and Trace Elements in Water by Ultrasonic Nebulization Inductively Coupled Plasma-Atomic Emission Spectrometry

Metals, Rev 2 (Atomic Absorption Methods), EPA/600/4-79/020 [includes Direct Aspiration and Furnace Techniques]

Method 206.2, Arsenic (Atomic absorption, furnace technique)

Method 206.3, Arsenic (Atomic Absorption – gaseous hydride)

Method 208.1, Barium (Atomic Absorption, direct aspiration)

Method 208.2, Barium (Atomic Absorption, furnace technique)

Method 212.3, Boron (Colorimetric, Curcumin)

Method 213.2, Cadmium (Atomic Absorption, furnace technique)

Method 218.2, Chromium (Atomic Absorption, furnace technique)

Method 218.3, Chromium (Atomic Absorption, chelation-extraction)

Method 239.2, Lead (Atomic Absorption, furnace technique)

Method 270.2, Selenium (Atomic Absorption, furnace technique)

Method 270.3, Selenium (Atomic Absorption, gaseous hydride)

SW-846, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry

SW-846, Method 6020A, Inductively Coupled Plasma-Mass Spectrometry

SW-846, Method 7000B, Atomic Absorption Methods

SW-846, Method 7060A, Arsenic (Atomic Absorption, Furnace Technique)

SW-846, Method 7061A, Arsenic (Atomic Absorption, Gaseous Hydride)

SW-846, Method 7062, Antimony and Arsenic (Atomic Absorption, Borohydride Reduction) [Arsenic only]

SW-846, Method 7063, Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)

SW-846, Method 7080A, Barium (Atomic Absorption, Direct Aspiration)

SW-846, Method 7081, Barium (Atomic Absorption, Furnace Technique)

SW-846, Method 7131A, Cadmium (Atomic Absorption, Furnace Technique)

SW-846, Method 7191, Chromium (Atomic Absorption, Furnace Technique)

SW-846, Method 7421, Lead (Atomic Absorption, Furnace Technique)

SW-846, Method 7740, Selenium (Atomic Absorption, Furnace Technique)

SW-846, Method 7741A, Selenium (Atomic Absorption, Gaseous Hydride)

SW-846, Method 7742, Selenium (Atomic Absorption, Borohydride Reduction)

AWWA Methods: [9]	Method 3111 B – Metals by Flame Atomic Absorption Spectrometry - Direct Air-Acetylene Flame Method
	Method 3111 D – Metals by Flame Atomic Absorption Spectrometry - Direct Nitrous Oxide-Acetylene Flame Method
	Method 3113 B – Metals by Electrothermal Atomic Absorption Spectrometric Method
	Method 3114 B – Arsenic and Selenium by Hydride Generation/Atomic Absorption Spectrometry - Manual Method
	Method 3114 C – Arsenic and Selenium by Continuous Hydride Generation/Atomic Absorption Spectrometric Method
	Method 3120 B – Metals by Plasma Emission Spectroscopy-Inductively Coupled Plasma (ICP) Method
	Method 3125 B – Metals by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) Method
	Method 3130 – Metals by Anodic Stripping Voltammetry [3130 B - Lead only]
	Method 4500-B B – Boron – Curcumin Method (Colourimetry)
	Method 4500-B C – Boron – Carmine Method (Colourimetry)
ASTM Methods:[10]	Method D1687-02C (reapproved 2007), Standard Test Method for Chromium in Water, Atomic Absorption, Graphite Furnace
	Method D1976-07, Standard Test Method for Elements in Water by ICP-AES; NOTE: Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL
	Method D2972-03B, Standard Test Method for Arsenic in Water, Atomic Absorption, Hydride Generation
	Method D2972-03C, Standard Test Method for Arsenic in Water, Atomic Absorption, Graphite Furnace
	Method D3082-03, Standard Test Method for Boron in Water (Colourimetry)
	Method D3559-03C, Standard Test Method for Lead in Water, Differential Pulse Anodic Stripping Voltammetry
	Method D3559-03D, Standard Test Method for Lead in Water, Atomic Absorption, Graphite Furnace

[9] *Standard Methods, 20th and 21st Ed.*
[10] Vol. 11.01 and 11.02, 2008

NOTE: This method is approved by the US EPA but the published MDL is higher than the MOE required RDL. Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL.

Method D3697-07, Standard Test Method for Antimony in Water

NOTE: Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL

Method D3859-03A, Standard Test Method for Selenium in Water, Gaseous Hydride AAS

Method D3859-03B, Standard Test Method for Selenium in Water, Graphite Furnace AAS

Method D3919-04, Standard Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry

Method D4382-02 (reapproved 2007), Standard Test Method for Barium in Water, Atomic Absorption Spectrophotometry, Graphite Furnace

Method D4691-02 (reapproved 2007), Standard Practice for Measuring Elements in Water by Flame AAS

Method D5174-07, Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry

Method D5673-05, Standard Test Method for Elements in Water by Inductively Coupled Plasma-Mass Spectrometry

3.3. Mercury

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Mercury	7439-97-6	0.001	0.1

LaSB Method: E3060 – The Determination of Mercury in Water by Cold Vapour-Flameless Atomic Absorption Spectrophotometry (CV-FAAS)

Method Principle: Mercury in the water sample is oxidized to its divalent ion form (Hg^{+2}) by an acid digestion procedure. It is then reduced by a stannous chloride solution to its elemental form. An air (or argon) stream carries the mercury vapour into a flow-through absorption cell positioned between a light source, set at 253.7 nm wavelength and a detector. The amount of light absorption is proportional to the concentration of mercury in the sample.

US EPA Methods: Method 245.1 Rev 3.0, Determination of Mercury by Cold Vapor Atomic Absorption Spectrometry – Manual

NOTE: This method is approved by the US EPA but the published MDL is higher than the MOE required RDL. Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL

Method 245.2, Mercury (Automated Cold Vapor Technique)

NOTE: This method is approved by the US EPA but the published MDL is higher than the MOE required RDL. Laboratories wishing to use this method must demonstrate that they can meet the required MOE RDL

Method 245.7 Rev 2.0, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry

Method 200.8 Rev 5.4, Trace Metals by ICP/Mass Spectrometry

SW-846, Method 7472, Mercury in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV)

AWWA Method: [11] Method 3112 B – Metals by Cold-Vapor Atomic Absorption Spectrometry

ASTM Method:[12] Method D3223-02 (reapproved 2007), Standard Test for Total Mercury in Water

3.4. Nitrite, Nitrate, and Nitrate + Nitrite

PARAMETER	CAS Number	ODWQS mg/L	RDL mg/L
Nitrate (as nitrogen)	14797-55-8	10.0	1
Nitrate + Nitrite (as nitrogen)	n/a	10.0	1
Nitrite (as nitrogen)	14797-65-0	1.0	0.1

LaSB Method: E3364 – The Determination of Ammonia Nitrogen, Nitrite Nitrogen, Nitrite plus Nitrate Nitrogen and Reactive Ortho-Phosphate in Surface Water, Drinking Waters and Ground Waters by Colourimetry.

Method Principle: Nitrite is determined as one of four automated nutrient tests performed simultaneously on the same aliquot of sample. Nitrite forms a diazotization product with sulphanilamide which is then coupled with N(1-naphthyl) ethylenediamine dihydrochloride at pH 1 ±0.1. A light red colour is produced. The absorbance of the solution is measured at 520 nm and the concentration of nitrite is determined by comparison with a known set of standards.

For nitrite plus nitrate, samples are analyzed via an automated colourimetric procedure, which entails converting nitrate to nitrite, and then analyzing the sample for nitrite; the original nitrite concentration is included in the result because nitrite anions are not affected by the preliminary reduction step. Nitrate is reduced to nitrite by heating (37°C) an aliquot of sample with hydrazine in alkaline media; this reaction is catalyzed by the addition of cupric ion.

[11] *Standard Methods, 20th and 21st Ed.*
[12] Vol. 11.01, 2008

Subsequently, an azo dye is formed in acid media by diazotizing sulphanilamide with nitrite and coupling the product with N (l-naphthyl) ethylenediamine dihydrochloride. The absorbance of the light red azo dye is measured at 520 nm and the concentration of nitrate nitrogen plus nitrite nitrogen is determined by comparison with a similarly treated series of mixed standards.

Nitrate may be calculated by subtracting the Nitrite result from the Nitrite plus Nitrate result.

US EPA Methods:

Method 300.0 Rev 2.1, Determination of Inorganic Anions by Ion Chromatography

Method 300.1 Rev 1.0, Determination of Inorganic Anions in Drinking Water by Ion Chromatography

Method 352.1, Nitrogen, Nitrate (Colorimetric, Brucine)

Method 353.1 Rev 1, Nitrogen, Nitrate-Nitrite, (Colorimetric, Automated, Hydrazine Reduction)

Method 353.2 Rev 2.0, Determination of Nitrate-Nitrite Nitrogen by Automated Colorimetry

Method 353.3, Nitrogen, Nitrate-Nitrite (Spectrophotometric, Cadmium Reduction)

Method 354.1, Nitrogen, Nitrite (Spectrophotometric)

SW-846, Method 9056, Determination of Inorganic Anions by Ion Chromatography

SW-846, Method 9200, Nitrate

AWWA Methods: [13]

Method 4110 B – Determination of Anions by Ion Chromatography with Chemical Suppression of Eluent Conductivity

Method 4110 C – Determination of Anions by Single-Column Ion Chromatography with Direct Conductivity Detection

Method 4500-NO_2^- B – Colorimetric Method

Method 4500-NO_3^- D – Nitrate Electrode Method

Method 4500-NO_3^- E – Cadmium Reduction Method

Method 4500-NO_3^- F – Automated Cadmium Reduction Method

Method 4500-NO_3^- H – Automated Hydrazine Reduction Method

Method 4500-NO_3^- I – Cadmium Reduction Flow Injection Method

[13] *Standard Methods, 20th and 21st Ed.*

ASTM Methods:[14] Method D4327-03, Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

Method D3867-04A, Standard Test Method for Nitrite-Nitrate in Water, Automated Cadmium Reduction

Method D3867-04B, Standard Test Method for Nitrite-Nitrate in Water, Manual Cadmium Reduction

Method D6508-00 (2005), Standard Test Method for Determination of Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte

3.5. Triazines (N-Containing Herbicides)

PARAMETER	CAS Number	ODWQS mg/L	RDL ng/L
Alachlor	15972-60-8	0.005	500
Atrazine + N-dealkylated metabolites	1912-24-9/	0.005	1000
Cyanazine	21725-46-2	0.01	1000
Metolachlor	51218-45-2	0.05	5000
Metribuzin	21087-64-9	0.08	8000
Prometryne	7287-19-6	0.001	250
Simazine	122-34-9	0.01	1000

LaSB Method: E3435 – The Determination of Triazine Pesticides in Water Matrices by Gas Chromatography-Time of Flight-Mass Spectrometry

Method Principle: Samples are made basic (pH >12) and solvent-extracted for triazine target compounds. Two deuterated triazine surrogate standards are added to an 800 mL water sample which is extracted with dichloromethane using a liquid/liquid extraction technique. The sample extracts for triazine compounds are dried, concentrated and re-constituted in a measured amount of iso-octane before analysis by capillary Gas Chromatography-Time of Flight Mass Spectrometry (GC-TOFMS).

LaSB Method: E3121 – The Determination of Triazine Herbicides in Water, Soils, Vegetation, and Toxicity Characteristic Leaching Procedure (TCLP) Leachate by Gas Chromatography/Mass Spectrometry (GC/MS) (2002)

Method Principle: Samples are made basic (pH > 12) and solvent-extracted. The sample extract is passed through sodium sulphate to remove excess water, evaporated to dryness and re-constituted in a measured amount of solvent prior to analysis by capillary gas chromatography/mass spectrometry (GC/MS) for these target compounds

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US EPA Methods: Method 505 Rev 2.1, Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products by Microextraction and Gas Chromatography

Method 507 Rev 2.1, Determination of Nitrogen- and Phosphorus-Containing Pesticides by Gas Chromatography with a Nitrogen Phosphorus Detector

Method 508.1 Rev 2.0, Chlorinated Pesticides, Herbicides and Organohalides by Liquid-Solid Extraction and GC with an Electron Capture Detector

Method 525.2 Rev 2.0, Organic Compounds by Liquid-Solid Extraction and Capillary Column GC/Mass Spectrometry

Method 527, Rev 1.0, Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

Method 536, Ver 1.0, Determination of Triazine Pesticides and Their Degradates in Drinking Water by Liquid Chromatography Electrospray Ionization Tandem Mass Spectrometry (LC/ESI-MS/MS)

Method 551.1, Rev 1.0, Determination of Chlorinated Disinfection By-Products and Chlorinated Solvents by Liquid-Liquid Extraction and Gas Chromatography with an Electron Capture Detector

SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction

SW-846, Method 3520C, Continuous Liquid-Liquid Extraction

SW-846, Method 3535, Solid-Phase Extraction (SPE)

SW-846, Method 8000C, Determinative Chromatographic Separations

ASTM Method:[15] Method D5475-93 (2002), Standard Test Method for Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector

3.6. Carbamates

PARAMETER	CAS Number	ODWQS mg/L	RDL ng/L
Aldicarb	116-06-3	0.009	9000
Bendiocarb	22781-23-3	0.04	7500
Carbaryl	63-25-2	0.09	9000
Carbofuran	1563-66-2	0.09	9000
Triallate	2302-17-5	0.23	23000

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LaSB Method:	E3438 – The Determination of Carbamates in Environmental Matrices by High Performance Liquid Chromatography and Tandem-Mass Spectrometry (LC/MS-MS) Analysis
Method Principle:	Samples are extracted by solid phase extraction, re-constituted in acetonitrile prior to analysis by high performance liquid chromatography with a tandem mass spectrometric detector (LC/MS-MS). LC/MS-MS analysis is carried out using an electrospray ionization source (ESI) in positive ionization and Multiple Reaction Monitoring (MRM) scan mode. MRM is a high- sensitivity type of MS/MS scan mode which is used for very specific target compound analysis. In this scan mode, a precursor ion with specific mass-to-charge ratio (m/z) is selected in the first quadrupole (Q1). It's then allowed to enter the collision cell (Q2) and fragmented by collision with neutral gas molecules (N2) in a process referred to as Collisionally Activated Dissociation (CAD) to produce product ions. A product ion with specific m/z is then selected in the third quadrupole (Q3) and sent for detection. The combination of that specific precursor ion from Q1 and its most abundant product ion from Q3 is chosen for use in the Multiple Reaction Monitoring (MRM) quantitative analysis. In addition to MRM, scheduled MRM (sMRM) can be used to ensure there are enough data points used to define a chromatographic peak to achieve a higher signal-to-noise (SNR) ratio. This is automatically done by the software such that the optimum dwell time of each MRM transition at its specific retention time will be used to achieve the best SNR possible. Identification of the target compound is done by comparing the LC retention time (RT) of the specific target MRM transition present in the sample with the RT of that target MRM transition in the standard. Upon positive identification of the target compound, quantification of the compound is done by the integrated area of that specific peak of the MRM chromatogram using internal standard calibration protocols and d6-dimethoate is used as an internal standard for volume correction.
LaSB Method:	E3158 – The Determination of Carbamates in Water by High Pressure Liquid Chromatography-Ultraviolet (HPLC-UV) Detection. (2000)
Method Principle:	Samples are solvent-extracted. The sample extract is passed through sodium sulphate to remove excess water, evaporated to dryness and re-constituted in a measured amount of solvent prior to analysis by high performance liquid chromatography with a UV detector
US EPA Methods:	Method 531.1 Rev 3.1, Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivatization Method 531.2, Rev 1.0, Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction

SW-846, Method 3520C, Continuous Liquid-Liquid Extraction

SW-846, Method 3535A, Solid-Phase Extraction (SPE)

SW-846, Method 8000C, Determinative Chromatographic Separations

SW-846, Method 8318, N-Methylcarbamates by High Performance Liquid Chromatography (HPLC)

SW-846, Method 8321C, Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection

AWWA Method: [16] Method 6610 B – Carbamate Pesticides, High-Performance Liquid Chromatographic Method

ASTM Method:[17] D5315-04, Standard Test Method for N-Methyl-Carbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection High-Performance Liquid Chromatography with Post-Column Derivatization

3.7. Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)

PARAMETER	CAS Number	ODWQS mg/L	RDL ng/L
Aldrin + Dieldrin	309-00-2/60-57-1	0.0007	70
Chlordane	57-74-9	0.007	700
Heptachlor + Heptachlor epoxide	76-44-8/1024-57-3	0.003	300
DDT+metabolites : p,p-DDD	72-54-8	0.03	3000
DDT+metabolites : p,p-DDE	72-55-9	0.03	3000
DDT+metabolites : o,p-DDT	789-02-6	0.03	3000
DDT+metabolites : p,p-DDT	50-29-3	0.03	3000
Lindane (Total)	58-89-9	0.004	400
Methoxychlor	72-43-5	0.9	90000
Polychlorinated Biphenyls (PCB)*	1336-36-3	0.003	300
Trifluralin	1582-09-8	0.045	4500

* sum of Arochlor 1254 and 1260

LaSB Method: E3400 – The Determination of Organochlorine Pesticides, Chlorobenzenes, PCB Aroclors, and Toxaphenes in Water, Effluent, and Waste Water by Hexane Microextraction and Gas Chromatography-Mass Spectrometry (GC- MS)

Method Principle: An approximately 800 mL water sample is extracted with 5 mL hexane. The extract is concentrated, reconstituted in toluene, and analyzed by gas chromatography-mass spectrometry (GC/MS) for the target compounds.

[16] *Standard Methods, 20th and 21st Ed.*
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	Qualitative identification of target compounds is achieved by comparison of target compound retention times (RT), intensities and ratios of target and qualifier ions with those of a calibration standard mixture. Quantitation of the target compounds as detected in a field sample is achieved by comparison of the target ion abundances with those from a calibration standard mix prepared in the laboratory. This method uses d_{10}-fluoranthene as an internal standard and 1,3,5-tribromobenzene is used as a surrogate to monitor method performance.
US EPA Methods:	Method 505 Rev 2.1, Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products by Microextraction and Gas Chromatography
	Method 508, Rev 3.1, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector
	Method 508.1 Rev 2.0, Chlorinated Pesticides, Herbicides and Organohalides by Liquid-Solid Extraction and GC with an Electron Capture Detector
	Method 525.2 Rev 2.0, Organic Compounds by Liquid-Solid Extraction and Capillary Column GC/Mass Spectrometry
	Method 527, Rev 1.0, Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)
	Method 551.1, Rev 1.0, Determination of Chlorinated Disinfection By-Products and Chlorinated Solvents by Liquid-Liquid Extraction and Gas Chromatography with an Electron Capture Detector
	SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction
	SW-846, Method 3520C, Continuous Liquid-Liquid Extraction
	SW-846, Method 3535A, Solid-Phase Extraction (SPE)
	SW-846, Method 8000C, Determinative Chromatographic Separations
	SW-846, Method 8081B, Organochlorine Pesticides by Gas Chromatography
	SW-846, Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography
AWWA Method: [18]	Method 6630 C – Organochlorine Pesticides, Liquid-Liquid Extraction Gas Chromatographic Method II
ASTM Methods:[19]	Method D5175-91 (2003), Standard Test Method for Organohalide Pesticides and Polychorinated Biphenyls in Water by Microextraction and Gas Chromatography

[18] *Standard Methods, 20th and 21st Ed.*

Method D5812-96 (2002), Standard Test Method for Determination of Organochlorine Pesticides in Water by Capillary Column Gas Chromatography

3.8. Organophosphorus Pesticides

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Azinphos-methyl	86-50-0	0.02	2
Chlorpyrifos	2921-88-2	0.09	9
Diazinon	333-41-5	0.02	2
Dimethoate	60-51-5	0.02	2.5
Malathion	121-75-5	0.19	19
Parathion (ethyl)	56-38-2	0.05	5
Phorate	298-02-2	0.002	0.5
Temephos	3383-96-8	0.28	28
Terbufos	13071-79-9	0.001	0.5

LaSB Method: E3437 – The Determination of Organophosphorus Pesticides in Environmental Matrices by High Performance Liquid Chromatography and Tandem Mass Spectrometry-Mass Spectrometry (LC-MS-MS) Analysis

Method Principle: Samples are extracted by solid phase extraction method. The sample extracts are condensed using nitrogen blow-down technique and re-constituted in acetonitrile prior to analysis by high performance liquid chromatography with a tandem mass spectrometric detector (LC/MS/MS).

LC/MS/MS analysis is carried out by using an electrospray ionization source (ESI) in positive ionization and Multiple Reaction Monitoring (MRM) scan mode. MRM is a high-sensitivity type of MS/MS scan mode which is used for very specific target compound analysis. In this scan mode, a precursor ion with specific mass-to-charge ratio (m/z) is selected in the first quadrupole (Q1). It is then allowed to enter the collision cell (Q2) and fragmented by collision with neutral gas molecules (N2) in a process referred to as Collisionally Activated Dissociation (CAD) to produce product ions. A product ion with specific m/z is then selected in the third quadrupole (Q3) and sent for detection. The combination of that specific precursor ion form Q1 and its most abundant product ion form Q3 is chosen for use in the Multiple Reaction Monitoring (MRM) quantitative analysis. In addition to MRM, scheduled MRM (sMRM) can be used to ensure there are enough data points used to define a chromatographic peak to achieve a higher signal-to-noise (SNR) ratio. This is automatically done by the software such that the optimum dwell time of each MRM transition at its specific retention time will be used to achieve the best SNR possible.

Identification of the target compound is done by comparing the LC retention time (RT) of the specific target MRM transition present in the sample with the RT of that target MRM transition in the standard. Identification of a target compound is confirmed by implementing a second MRM pair as a qualifier. Upon

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identification, quantification of the target compound is done by the integrated area of the specific peak of the first MRM chromatogram using internal calibration protocols and D6-Malathion as an internal standard.

LaSB Method: E3389 – The Determination of Organophosphorus Pesticides in Water by High Performance Liquid Chromatography-Ultraviolet (HPLC-UV) Detection (2000)

Method Principle: Samples are passed through pre-conditioned solid phase extraction (SPE) cartridges. Target analytes are eluted from the cartridge using ethyl acetate. The sample extract is evaporated to dryness and reconstituted in 1.0 mL acetonitrile prior to analysis by HPLC with UV detection.

US EPA Methods: Method 507 Rev 2.1, Determination of Nitrogen- and Phosphorus-Containing Pesticides by Gas Chromatography with a Nitrogen Phosphorus Detector

Method 526, Rev 1.0, Determination of Selected Semivolatile Organic Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

Method 527, Rev 1.0, Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction

SW-846, Method 3520C, Continuous Liquid-Liquid Extraction

SW-846, Method 3535A, Solid-Phase Extraction (SPE)

SW-846, Method 8000C, Determinative Chromatographic Separations

SW-846, Method 8141B, Organophosphorus Compounds by Gas Chromatography

ASTM Method:[20] Method D5475-93 (2002), Standard Test Method for Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector

3.9. Chlorophenols (CPs) & Phenoxy Acids (PAs)

PARAMETER	CAS Numbers	ODWQS mg/L	RDL ng/L
2,4,5-Trichlorophenoxy acetic Acid (2,4,5-T)	93-76-5	0.28	28000
2,4-Dichlorophenoxy acetic acid (2,4-D)	94-75-7	0.1	10000
Bromoxynil	1689-84-5	0.005	500
Dicamba	1918-00-9	0.12	12000
Diclofop-methyl	51338-27-3	0.009	900
Dinoseb	88-85-7	0.01	1000

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PARAMETER	CAS Numbers	ODWQS mg/L	RDL ng/L
Picloram	1918-02-1	0.19	19000
2,3,4,6-Tetrachlorophenol	58-90-2	0.1	10000
2,4,6-Trichlorophenol	88-06-2	0.005	500
2,4-Dichlorophenol	120-83-2	0.9	90000
Pentachlorophenol	87-86-5	0.06	6000

LaSB Method: E3119 – The Determination of Chlorophenols (CPs) and Phenoxyacid Herbicides (PAs) in Environmental Matrices by Gas Chromatography-Mass Spectrometric (GC/MS) Analysis

Method Principle: Water samples are acidified (pH $\leq$ 2) and passed through pre-conditioned C_{18} solid phase extraction (SPE) cartridges. SPE cartridges are then dried and target compounds eluted using a small volume of solvent. The column elute is treated with diazomethane (to methylate target compounds), evaporated and finally diluted with solvent to 1.0 mL. This extract is analyzed by gas chromatography with a mass spectrometer (GC/MS). CPs and PAs are quantified as their corresponding anisoles and methyl esters, respectively.

US EPA Methods: Method 515.2 Rev 1.1, Determination of Chlorinated Acids in Water using Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector

Method 515.1 Rev 4.0, Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector

Method 515.3 Rev 1.0, Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with Electron Capture Detection

Method 515.4 Rev 1.0, Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Microextraction, Derivatization and Fast Gas Chromatography with Electron Capture Detection

Method 525.2, Rev 2.0, Organic Compounds by Liquid-Solid Extraction and Capillary Column GC/Mass Spectrometry

Method 526, Rev 1.0, Determination of Selected Semivolatile Organic Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

Method 528, Rev 1.0, Determination of Phenols in Drinking Water by Solid Phase Extraction and Capillary Column Gas chromatography/Mass Spectrometry (GC/MS)

Method 555, Rev 1.0, Determination of Chlorinated Acids in Water by High Performance Liquid Chromatography with a Photodiode Array Ultraviolet Detector

SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction

SW-846, Method 3520C, Continuous Liquid-Liquid Extraction

SW-846, Method 3535A, Solid-Phase Extraction (SPE)

SW-846, Method 8000C, Determinative Chromatographic Separations

SW-846, Method 8041A, Phenols by Gas Chromatography

SW-846, Method 8150, Chlorinated Herbicides

SW-846, Method 8151A, Chlorinated Herbicides by GC using Methylation or Pentafluorobenzylation Derivatization

SW-846, Method 8321B, Solvent Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection

AWWA Method: [21] Method 6640 B – Acidic Herbicide Compounds, Micro Liquid-Liquid Extraction Gas Chromatographic Method

ASTM Method:[22] Method D5317-98 (2003), Standard Test Method for Determination of Chlorinated Organic Acid Compounds in Water by Gas Chromatography with an Electron Capture Detector

3.10. Quaternary Ammonium Compounds

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Diquat (as the dibromide)	85-00-7	0.07	7
Paraquat (as the dichloride)	1910-42-5	0.01	1

LaSB Method: E3417 – The Determination of Diquat and Paraquat in Water and Environmental Matrices by Liquid Chromatography-(Electrospray Ionization) Mass Spectrometry (LC-(ESI)MS)

Method Principle: Water samples are extracted using mixed-mode, polymer based solid phase extraction (SPE) cartridges at neutral pH. Vegetation and soil matrices are extracted in an acidic medium, diluted with NANOpure™ water, and adjusted to pH 6.5 prior to SPE cleanup. The SPE cartridge is pre-conditioned with water and methanol. Analytes are eluted using 1M ammonium chloride in an aqueous solution of 50 % methanol. Deuterated paraquat and diquat are used as method surrogates, as well as internal standard calibration for their respective native analogs for LC-(ESI) MS analysis.

[21] *Standard Methods, 20th and 21st Ed.*
[22] Vol. 11.02, 2008

US EPA Method: Method 549.2 Rev 1.0, Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and HPLC with a Photodiode Array UV Detector

3.11. Urea Derivative

PARAMETER	CAS Number	ODWQS mg/L	RDL ng/L
Diuron	330-54-1	0.15	15000

LaSB Method: E3436 – The Determination of Phenyl Ureas in Environmental Matrices by High Performance Liquid Chromatography and Mass Spectrometry-Mass Spectrometry (LC/MS-MS) Analysis

Method Principle: Samples are extracted by solid phase extraction method, re-constituted in acetonitrile prior to analysis by high performance liquid chromatography with a tandem mass spectrometric detector (LC/MS-MS).

LC/MS-MS analysis is carried out using an electrospray ionization source (ESI) in positive ionization and Multiple Reaction Monitoring (MRM) scan mode. MRM is a high- sensitivity type of MS/MS scan mode which is used for very specific target compound analysis. In this scan mode, a precursor ion with specific mass-to-charge ratio (m/z) is selected in the first quadrupole (Q1). It's then allowed to enter the collision cell (Q2) and fragmented by collision with neutral gas molecules (N2) in a process referred to as Collisionally Activated Dissociation (CAD) to produce product ions. A product ion with specific m/z is then selected in the third quadrupole (Q3) and sent for detection. The combination of that specific precursor ion from Q1 and its most abundant product ion from Q3 is chosen for use in the Multiple Reaction Monitoring (MRM) quantitative analysis. In addition to MRM, scheduled MRM (sMRM) can be used to ensure there are enough data points used to define a chromatographic peak to achieve a higher signal-to-noise (SNR) ratio. This is automatically done by the software such that the optimum dwell time of each MRM transition at its specific retention time will be used to achieve the best SNR possible.

Identification of the target compound is done by comparing the LC retention time (RT) of the specific target MRM transition present in the sample with the RT of that target MRM transition in the standard. Upon positive identification of the target compound, quantification of the compound is done by the integrated area of that specific peak of the MRM chromatogram using internal standard calibration protocols and d6-dimethoate is used as an internal standard for volume correction.

LaSB Method: E3230 – The Determination of Phenyl Ureas in Water by High Performance Liquid Chromatography-Ultraviolet (HPLC-UV) Detection. (2000)

Method Principle: Samples are solvent-extracted. The sample extract is passed through sodium sulphate to remove excess water, evaporated to dryness and re-constituted in a measured amount of solvent prior to analysis by high performance liquid chromatography with UV detector.

US EPA Methods: Method 532 Rev 1.0, Determination of Phenylurea Compounds in Drinking Water by Solid Phase Extraction and High Performance Liquid Chromatography with UV Detection

Method 553 Rev 1.1, Determination of Benzidines and Nitrogen-Containing Pesticides in Water by Liquid-Liquid Extraction or Liquid-Solid Extraction and Reverse Phase High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry

SW-846, Method 3510C, Separatory Funnel Liquid-Liquid Extraction

SW-846, Method 3520C, Continuous Liquid-Liquid Extraction

SW-846, Method 3535A, Solid-Phase Extraction (SPE)

SW-846, Method 8000C, Determinative Chromatographic Separations

SW-846, Method 8321B, Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection

SW-846, Method 8325, Solvent Extractable Nonvolatile Compounds by High Performance Liquid Chromatography/Particle Beam//Mass Spectrometry (HPLC/PB/MS)

3.12. Glyphosate

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Glyphosate	1071-83-6	0.28	28

LaSB Method: E3415 – The Determination of Glyphosate and Aminomethylphosphonic Acid (AMPA) in Environmental Matrices by High Performance Liquid Chromatography-Electrospray Ionization-Mass Spectrometry (HPLC-ESI-MS)

Method Principle: Neutral sample conditions are used for the extraction of water and vegetation matrices.

This method uses the derivatization of glyphosate and AMPA with 9-fluorenyl methoxycarbonyl chloride (FMOC-Cl), Figure 1, prior to HPLC analysis to enhance the retention of the target compounds, i.e. move them out of the void volume, and their separation using a C-18 reversed phase column.

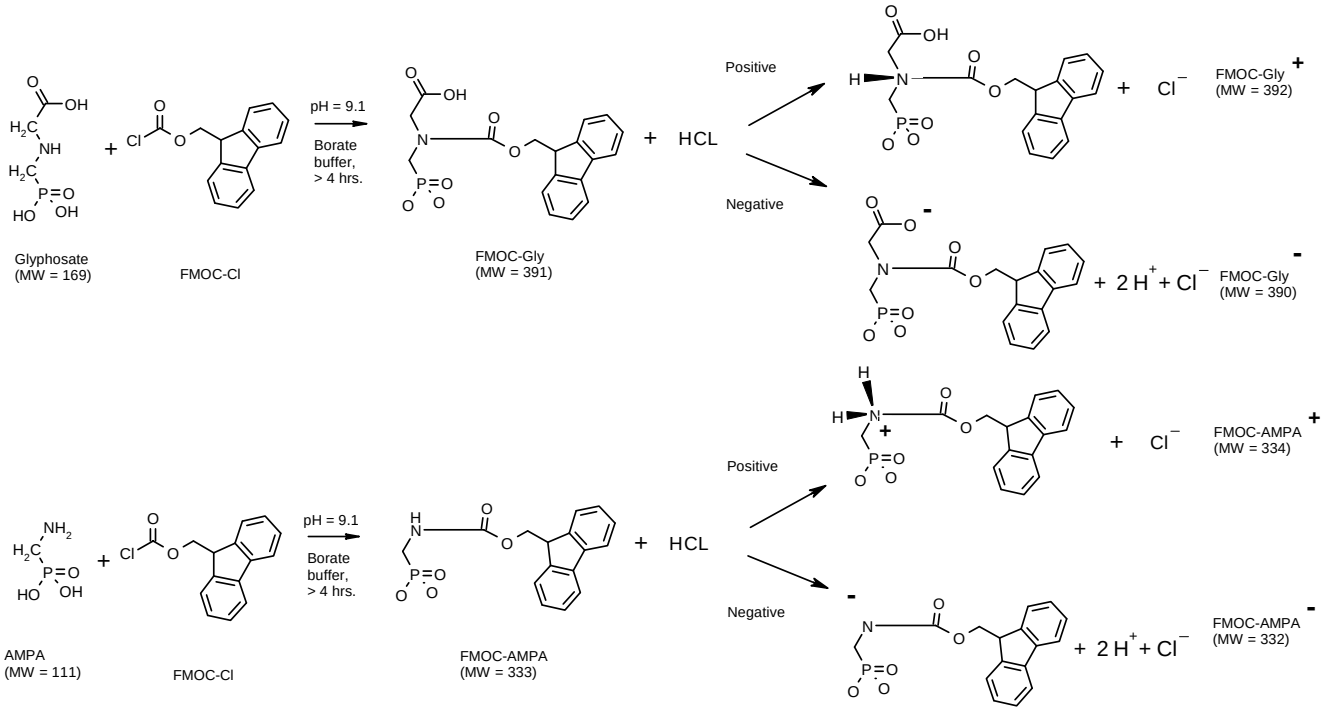
The identification is achieved by the HPLC retention time (within 0.2 minutes of established retention time), the molecular ion of the reaction product at pH 9.1 (FMOC-Gly m/z 390) and the fragment ion of the reaction product FMOC-AMPA (m/z 110), in negative ESI-MS (see the reaction scheme listed in Figure 1). Confirmation ions m/z 168, 332 for FMOC-Glyphosate and FMOC-AMPA are also used.

FMOC-Gly is not available commercially and is prepared in-house. The quantitation of the glyphosate and AMPA in water is done by using a standard prepared in NANOpureTM water and reacted with FMOC-Cl and borate buffer.

Isotope dilution quantitation is used for glyphosate analysis. Quantitation of AMPA is done through the external standard method as there is no correlation between the AMPA and 13C,15N-Glyphosate.

Retention times of FMOC-Gly and AMPA may shift depending upon the species of the plant and origin of samples. Therefore, standard addition of glyphosate into the final sample extract may be used for further confirmation of the identification of glyphosate in the vegetation matrices.

Figure 1: Reaction Scheme for Glyphosate with FMOC-Cl Detection Scheme for AMPA



US EPA Method: Method 547, Determination of Glyphosate in Drinking Water by Direct Aqueous Injection HPLC, Post Column Derivatization, and Fluorescence Detector

AWWA Method: [23] Method 6651 – Glyphosate Herbicide by Liquid Chromatographic Post-Column Fluorescence Method.

[23] *Standard Methods, 20th and 21st Ed.*

3.13. Fluoride

PARAMETER	ODWQS mg/L	RDL mg/L
Fluoride	1.5	0.15

LaSB Method: E3172 – The Determination of Fluoride and Sulphate in Water, Leachates and Effluents by Automated Ion Chromatography

Method Principle: Via ion chromatography (IC), fluoride is separated from other anions by using columns packed with ion exchange resin, and an eluent solution of sodium carbonate/sodium bicarbonate.

After separation, fluoride is converted to its acid form by ion exchange using a suppressor and its concentrations are determined from the conductivity of the acid produced. A conductivity meter measures the conductivity of each anionic species against the carbonic acid eluent background. The identity of the species is determined by their retention times. A pre-column (guard column) is used to trap foreign matter, thereby extending the life of the separator column.

US EPA Methods: Method 300.0 Rev 2.1, Determination of Inorganic Anions by Ion Chromatography

Method 300.1 Rev 1.0, Determination of Inorganic Anions in Drinking Water by Ion Chromatography

Method 340.1, Rev 2, Fluoride, Total (Colorimetric, SPADNS with Bellack Distillation)

Method 340.2, Rev 1, Fluoride (Potentiometric, Ion Selective Electrode)

Method 340.3, Fluoride (Colorimetric, Automated Complexone)

SW-846, Method 9056A, Determination of Inorganic Anions by Ion Chromatography

AWWA Methods: [24] Method 4500_F $^-$ B – Preliminary Distillation Step

Method 4500_F $^-$ C – Ion-Selective Electrode Method

Method 4500_F $^-$ D – SPADNS Method (Colourimetry)

Method 4500_F $^-$ E – Complexone Method (Automated Alizarin, Colourimetry)

Method 4500_F $^-$ G – Ion-Selective Electrode Flow Injection Analysis

Method 4110 B – Determination of Anions by Ion Chromatography with Chemical Suppression of Eluent Conductivity

[24] *Standard Methods, 20th and 21st Ed.*

Method 4110 C – Determination of Anions by Single-Column Ion Chromatography with Direct Conductivity Detection

ASTM Methods: [25] Method D4327-03, Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

Method D1179-04 B, Standard Test Method for Fluoride Ion in Water, Ion Selective Electrode

Method D6508-00 (2005), Standard Test Method for Determination of Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte

3.14. Benzo(a)pyrene

PARAMETER	CAS Number	ODWQS mg/L	RDL ng/L
Benzo(a)pyrene	50-32-8	0.00001	10

LaSB Method: E3480 – The Determination of Polycyclic Aromatic Hydrocarbons in Water Matrices by Gas Chromatography/Time-of-Flight-Mass Spectrometry (GC/TOF-MS)

Method Principle: Samples are adjusted to pH range 5 to 9 and solvent-extracted for Polycyclic Aromatic Hydrocarbons (PAH) target compounds. The sample extract for PAH compounds is concentrated prior to analysis by GC/TOF-MS for the target PAH compounds. Qualitative identification of the target compounds is achieved by comparison of target compound retention times (RT) and full spectral library match with that of a calibration standard mixture. Quantitation of the target compounds as detected in samples is achieved by comparison of areas of the molecular ion (target ion) with that from a calibration standard mix prepared in the laboratory.

The following compounds are used as internal standards for PAH analysis: d_{10}-phenanthrene, d_{10}-fluoranthene, d_{10}-chrysene, d_{12}-benzo(a)pyrene, d_{12}-benz(a)anthracene, and d_{12}-indeno(1,2,3-c,d)pyrene .

NOTE: LaSB formerly combined the analysis of PAHs and Triazines within one method document, E3435. As of 2010, the PAH target list was separated from the previous method and assigned a new number. The Triazine target list still remains in the E3435 document. There was no change in the method principle for the PAH analysis. Laboratories who have based their PAH method on earlier versions of E3435 may wish to request a copy of this method as a current reference.

LaSB Method: E3399 – The Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Aqueous Matrices by Liquid-Liquid Micro-Extraction (LLME) and Gas Chromatography-Mass Spectrometry (GC/MS) (2008)

[25] Vol. 11.01 and 11.02, 2008

Method Principle: Six deuterated PAH internal standards are added to an 800 mL water sample which is extracted with 5 mL toluene using a liquid/liquid microextraction technique. The extract is concentrated and analyzed by gas chromatography-mass spectrometry (GC-MS) for the target compounds.

Qualitative identification of target compounds is achieved by comparison of target compound retention times (RT), intensities and ratios of target and qualifier ions with that of a calibration standard mixture. Quantitation of the target compounds as detected in a field sample is achieved by comparison of the target ion abundance with that from a calibration standard mix prepared in the Laboratory. The following compounds are used as Internal standards for this method: d_{10}-phenanthrene, d_{10}-fluoranthene, d_{12}-chrysene, d_{12}-benzo(a)pyrene, d_{12}-benz(a)anthracene, and d_{14}-dibenz(a,h)anthracene.

US EPA Methods: Method 525.2 Rev 2.0, Organic Compounds by Liquid-Solid Extraction and Capillary Column GC/Mass Spectrometry

Method 550, Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Drinking Water by Liquid-Liquid Extraction and HPLC with Coupled Ultraviolet and Fluorescence Detection

Method 550.1, Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Drinking Water by Liquid-Solid Extraction and HPLC with Coupled Ultraviolet and Fluorescence Detection

3.15. Cyanide

PARAMETER	CAS Number	ODWQS mg/L	RDL mg/L
Cyanide (free)	57-12-5	0.2	0.02

LaSB Method: E3015 – The Determination of Free and Total Cyanide in Environmental Samples by Colourimetry.

Method Principle: Total cyanide includes the free simple cyanides (such as HCN and KCN), and weakly bound cyanides (such as $Ni(CN)_4$), as well as those complexed cyanides that decompose to form free cyanides that distill out as HCN in an acidic environment. Aqueous samples are introduced directly to the continuous flow system from an autosampler. The distillation step isolates HCN under specific acidic conditions. The sequential combination of UV digestion plus distillation yields the measurement of "total cyanide". Interferences from thiocyanate are removed using a borosilicate glass coil in a UV-B chamber (wavelengths above 290 nM). When thiocyanate is not dissociated during UV digestion, it does not carry over in the distillation step. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine-T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nM.

Free cyanides are the simple and weakly dissociable cyanides that form HCN upon acidification to pH 4.0. Free cyanide is determined as above, but with two changes; the elimination of the UV digestion, which avoids the conversion of the complexed cyanides to free cyanide and the replacement of the pure-deionized water reagent with a 10 mg/L zinc sulphate solution, for the elimination of interference from complexed iron cyanides

US EPA Methods: SW-846, Method 9010C, Total and Amenable Cyanide: Distillation

SW-846, Method 9012B, Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation)

SW-846, Method 9014, Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide [Spectrophotometric method only]

AWWA Methods: [26] Method 4500-CN^- G – Cyanides Amenable to Chlorination after Distillation

Method 4500-CN- H – Cyanides Amenable to Chlorination without Distillation (Short-Cut Method)

ASTM Methods:[27] Method D2036-06 B, Standard Test Method for Cyanides in Water, Cyanides Amenable to Chlorination by Difference

Method D2036-06 D, Standard Test Method for Cyanides in Water, Cyanides Amenable to Chlorination without Distillation, Short-Cut Method

Method D4282-02, Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion

Method D7237-06, Standard Test Method for Aquatic Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection

3.16. Dioxins and Furans – Toxic Equivalent Quantity

PARAMETER	ODWQS mg/L	RDL pg TEQ 2,3,7,8-TCDD/L
Dioxin and Furan	0.000000015	7.5

LaSB Method: E3418 – The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD), Polychlorinated Dibenzofurans (PCDF) and Dioxin-like Polychlorinated Biphenyls (DLPCBs) in Environmental Matrices by Gas Chromatography-Mass Spectrometry (GC/MS).

Method Principle: Drinking water sample volumes are measured and recorded. If any of the samples contain particulate, then the samples must be extracted using the groundwater/effluent Empore disk method. Samples are liquid/liquid extracted

[26] *Standard Methods, 20th and 21st Ed.*
[27] Vol. 11.02, 2008

with pentane. A 2-stage chromatographic clean-up procedure is used to remove potential chemical interferences.

This analytical method is used to determine the concentrations of PCDDs, PCDFs and DLPCBs in a variety of matrices using isotope dilution with mass spectrometric detection. All samples are fortified prior to sample extraction, digestion or elution with known amounts of [$^{13}C_{12}$ -] isotopically labelled PCDDs/PCDFs and/or DLPCBs. All PCDDs/PCDFs and DLPCBs are quantified against these labelled standards. Sample extracts are cleaned using a 2-stage column (silica/alumina) when PCDDs/PCDFs only are requested or no interferences such as polychlorinated diphenyl ethers (PCDPEs) are expected in the sample. If PCDDs/PCDFs and DLPCBs are requested or if a sample is highly contaminated with bulk interferences, a carbon cleanup procedure must be incorporated (3-stage).

US EPA Method: Method 1613B, Tetra- Through Octa-Chlorinated Dioxins by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry

3.16.1 Dioxins and Furans – Calculation of Toxic Equivalent Quantity (TEQ)

There are a total of 210 dioxins and furans. Only 17 are toxic (2,3,7,8-substituted congeners) and their toxicity is normalized to 2378-TCDD (the most toxic). The TEQ is determined (as shown in the following example) by multiplying the concentration of each detected 2,3,7,8-substituted congener by its respective toxic equivalent factor (TEF) to determine its toxic equivalence (TE). The TEFs in the following table are those provided by the World Health Organization (WHO), 2006, as amended from time to time. Laboratories must identify the source of the TEFs used for their calculations. For the 2,3,7,8-substituted congeners that are not detected, half of the detection limit is multiplied by the TEF to determine the TE for that congener. This converts each of the congeners to 2378-TCDD toxic equivalents. The sum of the 17 toxic equivalents (TEs) gives the TEQ (toxic equivalent quantity) for the sample normalized to 2378-TCDD. The result is 1.7 pg/L, which is well below the 15 pg/L ODWQS.

TEQ EXAMPLE

Compound	CAS Number	Conc. pg/L	EDL pg/L	TEF pg/L	TE /congener pg/L
2,3,7,8-TCDD	1746-01-6	ND	1.1	1	0.55
1,2,3,7,8-PeCDD	40321-76-4	ND	1	1	0.5
1,2,3,4,7,8-HxCDD	39227-28-6	ND	1.2	0.1	0.06
1,2,3,6,7,8-HxCDD	57653-85-7	ND	0.89	0.1	0.045
1,2,3,7,8,9-HxCDD	19408-74-3	ND	1	0.1	0.05
1,2,3,4,6,7,8-HpCDD	35822-46-9	ND	1.1	0.01	0.0055
OCDD	3268-87-9	3.4		0.0001	0.00034
2,3,7,8-TCDF	51207-31-9	ND	1	0.1	0.05
1,2,3,7,8-PeCDF	57117-41-6	ND	1	0.05	0.025
2,3,4,7,8-PeCDF	57117-31-4	ND	1	0.5	0.25
1,2,3,4,7,8-HxCDF	70648-26-9	ND	0.82	0.1	0.041
1,2,3,6,7,8-HxCDF	57117-44-9	ND	1.1	0.1	0.055
2,3,4,6,7,8-HxCDF	60851-34-5	ND	1.1	0.1	0.055
1,2,3,7,8,9-HxCDF	72918-21-9	ND	1.2	0.1	0.06

TEQ EXAMPLE

Compound	CAS Number	Conc. pg/L	EDL pg/L	TEF pg/L	TE /congener pg/L
1,2,3,4,6,7,8-HpCDF	67562-39-4	ND	0.95	0.01	0.0048
1,2,3,4,7,8,9-HpCDF	5567-89-7	ND	1	0.01	0.005
OCDF	39001-02-0	1.8		0.0001	0.00018
TOTAL TEQ 2,3,7,8-TCDD (0.5 DL) = 1.75 pg/L					

TEQ = Toxic Equivalent Quantity = sum of individual TE/congener
EDL = Estimated Detection Limit
TEF = Toxic Equivalent Factor (WHO, 2006)
TE/congener = Toxic Equivalence / congener

3.17. Nitrilotriacetic Acid (NTA)

PARAMETER	CAS Number	ODWQS mg/L	RDL mg/L
Nitrilotriacetic Acid (NTA)	139-13-9	0.4	0.05

LaSB Method: E3406 – The Determination of Nitrilotriacetic Acid (NTA) in Aqueous Samples by Automated Ion Chromatography (IC).

Method Principle: Using ion chromatography (IC), nitrilotriacetic acid (NTA) is separated from other anions using columns packed with ion exchange resin and an eluent solution of sodium hydroxide. The eluent is passed through an anion trap column (ATC-1) to remove impurities which may be present in the working eluent solution.

The sample is passed through a cation trap column (in house design) made of Amberlite IR-120(H) resin to remove calcium, magnesium and iron from the sample matrix before analysis. The sample is then injected into the eluent stream and passes through a pre-column (AG-11). The pre-column removes any foreign matter entering the analytical system, extending the life of the separator column (AS-11). After separation, the anions are converted to their acid forms by ion exchange using a micromembrane suppressor and their concentrations are determined from the conductivity of the NTA produced. A conductivity meter measures the conductivity of each anionic species against the water background. The identities of the species are determined by their retention times.

US EPA Method: Method 430.2, NTA (Colorimetric, Automated, Zinc-Zincon)

3.18. N-nitrosodimethylamine (NDMA)

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
N-Nitrosodimethylamine (NDMA)	62-75-9	0.000009	0.00099

LaSB Method: E3388 – The Determination of N-nitrosamines in Water by Gas Chromatography-High Resolution Mass Spectrometry (GC-HRMS)

Method Principle: This method is designed to identify and quantify N-nitrosodimethylamine (NDMA), N-nitrosomethylethylamine (NMEA), N-nitrosodiethylamine (NDEA), N-nitrosodi-n-propylamine (NDPA), N-nitrosodi-n-butylamine (NDBA), N-nitrosopyrrolidine (NPYR), N-nitrosopiperidine (NPIP) and N-nitrosomorpholine (NMOR) in water by adsorption onto Ambersorb 572, elution into an organic solvent and analysis by high resolution (capillary column) gas chromatography-high resolution mass spectrometry (GC-HRMS).

The internal standards d_6-NDMA and d_{14}-N-nitrosodi-n-propylamine (d_{14}-NDPA) are added to a 500 mL aliquot of the sample. After the addition of Ambersorb 572, the bottle is rolled at 50 rpm on a roller apparatus for 1 hour. The granular adsorbent is isolated by filtration on filter paper and allowed to air dry or under ultrahigh purity (UHP) nitrogen for a minimum of 1 hour. The Ambersorb 572 is transferred to a 2 mL autosampler vial and 400 µL of dichloromethane are added. The vial is capped and the contents are analyzed by GC-HRMS. NDMA and NDPA are quantified by isotope dilution with d_6-NDMA and d_{14}-NDPA, respectively. The other six N-nitrosamines (NMEA, NDEA, NDBA, NPYR, NPIP and NMOR) are quantified using the internal standard d_{14}-NDPA.

NOTE: The LaSB method E3291 – The Determination of N-nitrosodimethylamine (NDMA) in Water by Gas Chromatography-High Resolution Mass Spectrometry (GC-HRMS) was discontinued as a separate method in May 2008. The method principles of E3291 are the same as E3388, which includes additional parameters.

US EPA Method: Method 521, Rev 1.0, Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS)

3.19. Bromate

PARAMETER	CAS Number	ODWQS mg/L	RDL mg/L
Bromate	15541-45-4	0.01	0.005

LaSB Method: E3434 – The Determination of Bromide in Drinking/Source Water by Ion Chromatography with Conductivity Detection and Bromate in Drinking Water with the Addition of Postcolumn Reagent and a UV/Visible Detector

Method Principle: Using ion chromatography (IC), bromate and bromide are separated from other anions using columns packed with ion exchange resin and an eluent solution of sodium carbonate.

The sample is introduced into an ion chromatograph. The ions of interest are separated using a guard column, analytical column, suppressor device, electrochemical detector, a postcolumn delivery system (pneumatically controlled), a heated postcolumn reaction coil and an ultraviolet/visible (UV/VIS) absorbance detector.

US EPA Methods: Method 317.0 Rev 2.0, Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis.

Method 326.0, Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography Incorporating the Addition of a Suppressor Acidified Postcolumn Reagent for Trace Bromate Analysis.

Method 300.1, Determination of Inorganic Anions in Drinking Water by Ion Chromatography.

Method 321.8, Rev 1.0, Determination of Bromate in Drinking Waters by Ion Chromatography Inductively Coupled-Plasma-Mass Spectrometry

Method 326.0, Rev 1.0, Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography Incorporating the Addition of a Suppressor Acidified Postcolumn Reagent for Trace Bromate Analysis

AWWA Method: [28] Method 4110 D – Ion Chromatographic Determination of Oxyhalides and Bromide

ASTM Method:[29] Method D6581-00 (2005), Standard Test Method for Bromate, Bromide, Chlorate, and Chlorite in Drinking Water by Chemically Suppressed Ion Chromatography

3.20. Microcystin LR

PARAMETER	CAS Number	ODWQS mg/L	RDL µg/L
Microcystin LR	106021-96-9	0.0015	0.15

LaSB Method: E3450 – The Determination of Microcystins and Anatoxin-A in Water By Liquid Chromatography-(Electrospray Ionization) Tandem Mass Spectrometry [LC-(ESI) MS/MS]

Method Principle: This method is designed to identify and quantify total (free + intracellular) microcystins and anatoxin-A in water by isolation on octadecyl-functionalised (C_{18}) silica gel and analysis by liquid chromatography-(electrospray ionisation) tandem mass spectrometry [LC-(ESI)MS/MS]. Microcystins-LR, -RR, -LA, -YR and anatoxin-A are determined quantitatively by multi-point calibration.

Nodularin is used as the internal standard.

The sample is filtered through a glass fiber, Type C (GC/C) filter and the filtrate is saved. The cells are lysed by freeze-drying and the intracellular toxins are extracted with a 75% methanol/25% water solution + 0.1% trifluoroacetic acid

[28] *Standard Methods, 21st Ed.*
[29] Vol. 11.01, 2006

(TFA). This extract is added to the filtrate. The 500 mL aliquot samples are taken to pH 9.5 to 10.0 using a borax buffer solution. The internal standard nodularin is then added to the sample. C18 silica gel is then added to the filtrate. The sample is rolled for 1 hour and the sample is filtered. The C18 silica gel is air dried for approximately 2 hours. The analytes are desorbed with a methanol/TFA solution. The extract is then centrifuge-filtered, evaporated to dryness and redissolved in a 0.1% tridecafluoroheptanoic acid (TDFHA) + 50% methanol / 50% water solution. Quantitation is done by an internal standard method.

3.20.1 Screening Tests for Total Microcystins

As of December 14, 2009, O. Reg. 248/03 was amended to allow the use of approved screening tests.

LaSB Method: E3469 – The Screening and Semi-Quantitative Analysis of Water Samples for Microcystins by Enzyme-Linked Immunosorbent Assay (ELISA)

Method Principle: ELISA is the most prevalent immunoassay technique utilized for environmental analyses. The immunoassay test products available from manufacturers are devised for specific analytes but follow the same general principles. The fundamental concept governing all immunoassays is the lock and key fit between the analyte molecule and the binding sites of the antibody. Immunoassays can be performed in either liquid-phase or solid-phase. An immunosorbent is created when a known amount of antibody is immobilized on a solid-phase support (such as a disposable plastic tube or microtiter plate). The type of immunoassay employed in this method is known as competitive ELISA.

Competitive ELISA methodology is based on three biochemical principles. First, the antibody on a solid-phase (immunosorbent) allows the separation of the antibody-bound analyte from the unbound materials by washing. Second, the analyte of interest is covalently linked to an enzyme to form an enzyme-analyte conjugate with the enzyme serving as a label. After the first step, the conjugate is added and allowed to bind to any remaining antibody not yet saturated with the analyte in the sample. The concentration and cross-reactivity of the analyte are both attributes that determine the affinity of the analyte to bind the antibody and hence its ability to inhibit subsequent binding of the microcystin-enzyme conjugate to the same antibody. Excess unbound conjugates are removed by washing. The final step involves the addition of a chromogenic substrate, which is turned over by the enzyme linked to the conjugate that is antibody-bound. The product of the enzymatic reaction follows the principles of Beer's Law and is measured using a spectrophotometer.

In the absence of microcystin in the sample, maximum binding of the conjugate to the antibodies occurs and would be retained after the washing step. Subsequent addition of substrate and chromogen would yield a maximum colour signal (absorbance). The maximum absorbance value produced by the negative control is regarded as the zero baseline (B0). Conversely, the presence of microcystin in the sample inhibits the conjugate from binding to the antibodies thereby reducing the absorbance. Thus, the intensity of the colour product is inversely proportional to the analyte concentration, typical of a competitive inhibition reaction. The

absorbance values of the sample are be expressed as a percentage of the zero baseline (%B0).

The general analytical protocol used in screening of the microcystin samples involves the following steps:

i) Cell wall rupturing by freeze-thawing
ii) ELISA
iii) Spectrophotometer measurement
iv) Data processing and reporting

Other Methods: The MOE does not endorse specific manufacturers of supplies for immunoassay techniques. The laboratory is recommended to use microtiter plates and is required to provide performance data.

3.21. Chloramines

PARAMETER	ODWQS mg/L	RDL mg/L
Chloramines	3.0	1.5

LaSB Method: N/A

AWWA Methods: [30] Method 4500-Cl D – Amperometric Titration Method

Method 4500-Cl F – DPD Ferrous Titrimetric Method

Method 4500-Cl G – DPD Colorimetric Method

4. ADDITIONAL PARAMETERS (CERTIFICATE OF APPROVAL OR MINISTRY ORDERS)

4.1. Sodium

PARAMETER	CAS Number	Standard mg/L	RDL mg/L
Sodium	7440-23-5	20	2

LaSB Method: E3171 – The Determination of Cations in Aqueous Samples by Atomic Absorption Spectrophotometry (AAS)

Method Principle: An automated atomic absorption method is used to measure the concentration of calcium, magnesium, sodium and potassium ions. Prior to sample aspiration as a fine mist into the air-acetylene flame of the AAS, the sample is automatically mixed with either lanthanum chloride, a releasing agent for calcium and

[30] *Standard Methods, 20th and 21st Ed.*

magnesium analysis, or caesium chloride, an ionization suppressant, for the analysis of sodium and potassium.

Light is emitted from a hollow cathode lamp and is directed through a flame into a monochromator and onto a detector that is set at a characteristic wavelength for each of the parameters (Ca 422.7 nm, Mg 285.2 nm, Na 589.0 nm, K 766.5 nm). The atoms of interest are heated in the flame, absorb the light at its particular wavelength and the detector measures the decreased intensity of the resulting beam. The amount of light absorbed is directly proportional to the concentration of the parameter in the sample. By comparing the sample with known standards, the sample concentration can be calculated.

NOTE: The LaSB method E3271 – The Determination of Cations in Water, Sewage, Health Samples, Industrial Waste, Leachates and Landfills by Atomic Absorption Spectrophotometry was discontinued as a separate method in August 2006, and merged with E3171. The method principles of the two methods are identical, and differed only by the calibration ranges for the target parameters.

US EPA Methods: Method 200.5, Rev 4.2, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry

Method 200.7 Rev 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry

Metals, Rev 2 (Atomic Absorption Methods), EPA/600/4-79/020

Method 273.1 Rev 1, Sodium (Atomic Absorption, direct aspiration)

Method 273.2, Sodium (Atomic Absorption, furnace technique)

SW-846, Method 7000B, Atomic Absorption Methods

AWWA Methods: [31] Method 3111 B – Metals by Flame Atomic Absorption Spectrometry - Direct Air-Acetylene Flame Method

Method 3500 B – Sodium by Flame Emission Photometric Method

Method 3120 B – Metals by Plasma Emission Spectroscopy - Inductively Coupled Plasma (ICP) Method

ASTM Methods:[32] Method D4191-03, Standard Test Method for Sodium in Water by Atomic Absorption Spectrophotometry

Method D6919-03, Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography

[31] *Standard Methods, 20th and 21st Ed.*
[32] Vol. 11.01 and 11.02, 2008

4.2. Ammonia

PARAMETER
Ammonia (as Nitrogen)

LaSB Method:	E3364 – The Determination of Ammonia Nitrogen, Nitrite Nitrogen, Nitrite plus Nitrate Nitrogen and Reactive Ortho-Phosphate in Surface Water, Drinking Waters and Ground Waters by Colourimetry.
Method Principle:	Ammonia is determined as one of four automated nutrient tests performed simultaneously on the same aliquot of sample. The procedure, is based on the formation of indophenol blue in buffered alkaline media, includes a blanking system to compensate for the sample matrix. In the colour formation stream, ammonia is converted to indophenol blue using sodium nitroprusside as catalyst. The blanking stream differs from the colour formation stream in only one respect: the flow of the catalyst reagent is replaced by an equal flow of Pure Water that has been further deionized, Pure-DW.
US EPA Methods:	Method 350.1, Rev 2.0, Determination of Ammonia Nitrogen by Semi-Automated Colorimetry
	Method 350.2, Rev 1, Nitrogen, Ammonia (Colourimetric; Titrimetric; Potentiometric – Distillation Procedure)
	Method 350.3, Nitrogen, Ammonia (Potentiometric, Ion Selective Electrode)
AWWA Methods: [33]	Method 4500-NH_3 B – Preliminary Distillation Step
	Method 4500-NH3 C – Titrimetric Method
	Method 4500-NH3 D – Ammonia-Selective Electrode
	Method 4500-NH3 E – Ammonia-Selective Electrode Method Using Known Addition
	Method 4500-NH3 F – Phenate Method
	Method 4500-NH3 G – Automated Phenate Method
	Method 4500-NH3 H – Flow Injection Analysis
ASTM Methods:[34]	Method D1426-03, Standard Test Methods for Ammonia Nitrogen in Water
	Method D6919-03, Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography

[33] *Standard Methods, 20th and 21st Ed.*
[34] Vol. 11.01 and 11.02, 2008

4.3. Biochemical Oxygen Demand (5-Day BOD)

PARAMETER
Biochemical Oxygen Demand (5-Day BOD)

LaSB Method: E3182 – The Determination of Biochemical Oxygen Demand In Surface Water and Sewage Effluents by Dissolved Oxygen Meter

Method Principle: The BOD is a measure of the dissolved oxygen depletion during a 5-day incubation period at a specified temperature (20°C). The units are milligrams per litre as molecular oxygen. Modifications are introduced, if necessary, to ensure a suitable level of bacterial activity.

The biochemical oxygen demand is determined by analyzing a sample or dilution thereof with Dilution Water. The dilution should be such that about 50% of the dissolved oxygen is depleted after 5 days incubation. The dissolved oxygen is determined as soon as possible after preparation and again after the incubation period. The BOD is expressed as the amount of dissolved oxygen in mg utilized by 1 litre of sample during a 5-day incubation period at 20°C.

The principle of the oxygen electrode is based on the relationship between the concentration of dissolved oxygen in a sample and the current generated by its reduction under controlled conditions. The electrode probe contains an electrolytic cell separated from test sample by a Teflon membrane which is permeable to dissolved oxygen. When the probe is immersed in a sample, a portion of the oxygen in the sample diffuses through the membrane into the electrolytic cell and is reduced at the cathode. The resultant change in current is directly proportional to the oxygen concentration present in the sample and is read out digitally as mg/L as O2. A suitable velocity of water across the membrane is maintained by a motorized stirrer and a built-in thermistor compensates for temperature variations.

US EPA Method: Method 405.1, Biochemical Oxygen Demand (BOD) - 5 Days, 20°C

AWWA Method: [35] Method 5210 B – 5-Day BOD Test

4.4. Bromide, Chlorate and Chlorite

PARAMETER
Bromide
Chlorate
Chlorite

LaSB Method: E3434 – The Determination of Bromide in Source Water by Ion Chromatography/Electrochemical Detection and Trace Levels of Bromate in

[35] *Standard Methods, 20th and 21st Ed.*

Ozonated Drinking Water with the Addition of Postcolumn Reagent and a UV/Visible Detector

Method Principle: Using ion chromatography (IC), bromate and bromide are separated from other anions using columns packed with ion exchange resin and an eluent solution of sodium carbonate.

The sample is introduced into an ion chromatograph. The ions of interest are separated using a guard column, analytical column, suppressor device, electrochemical detector, a postcolumn delivery system (pneumatically controlled), a heated postcolumn reaction coil and an ultraviolet/visible (UV/VIS) absorbance detector.

NOTE: The LaSB method does not currently include chlorate and chlorite.

US EPA Methods: Method 300.0 Rev 2.1, Determination of Inorganic Anions by Ion Chromatography

Method 300.1 Rev 1.0, Determination of Inorganic Anions in Drinking Water by Ion Chromatography

Method 317.0 Rev 2.0, Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis.

Method 326.0, Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography Incorporating the Addition of a Suppressor Acidified Postcolumn Reagent for Trace Bromate Analysis.

Method 327.0 Rev 1.1, Determination of Chlorine Dioxide and Chlorite Ion in Drinking Water Using Lissamine Green B and Horseradish Peroxidase with Detection by Visible Spectrophotometry

SW-846, Method 9056A, Determination of Inorganic Anions by Ion Chromatography

SW-846, Method 9211, Potentiometric Determination of Bromide in Aqueous Samples with Ion-Selective Electrode

AWWA Methods: [36] Method 4110 B – Determination of Anions by Ion Chromatography with Chemical Suppression of Eluent Conductivity

Method 4110 C – Determination of Anions by Single-Column Ion Chromatography with Direct Conductivity Detection

Method 4110 D – Ion Chromatographic Determination of Oxyhalides and Bromide

Method 4500-ClO_2 D – Amperometric Method II (Chlorate and chlorite only)

[36] *Standard Methods, 20th and 21st Ed.*

ASTM Methods:[37] Method D1246-05, Standard Test Method for Bromide Ion in Water

Method D4327-03, Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

Method D6581-00 (2005), Standard Test Method for Bromate, Bromide, Chlorate, and Chlorite in Drinking Water by Chemically Suppressed Ion Chromatography

4.5. Chemical Oxygen Demand

PARAMETER
Chemical Oxygen Demand (COD)

LaSB Method: E3170 – The Determination of Chemical Oxygen Demand (COD) in Domestic and Surface Waters by Colourimetry

Method Principle: Samples are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. Concentrated sulphuric acid containing silver sulphate as a catalyst is added and the mixture is digested in a mechanical-convection oven for 3 hours at 149°C ±2°C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

US EPA Methods: Method 410.2 Rev 2, Chemical Oxygen Demand (Titrimetric, Low Level)

Method 410.4 Rev 2.0, Determination of Chemical Oxygen Demand by Semi-automated Colorimetry

AWWA Methods: [38] Method 5220 B – Open Reflux Method

Method 5220 C – Closed Reflux, Titrimetric Method

Method 5220 D – Closed Reflux, Colorimetric Method

ASTM Methods:[39] Method D1252-06, Standard Test Methods for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Water

Method D6697-01, Standard Test Method for Determination for Chemical Oxygen Demand (Manganese III Oxygen Demand) of Water

[37] Vol. 11.01 and 11.02, 2008
[38] *Standard Methods, 20th and 21st Ed.*
[39] Vol. 11.02, 2008

4.6. Haloacetic Acids

PARAMETER	CAS Number
Monochloroacetic acid (MCAA)	79-11-8
Dichloroacetic acid (DCAA)	79-43-6
Trichloroacetic acid (TCAA)	76-03-9
Monobromoacetic acid (MBAA)	79-08-3
Bromochloroacetic acid (BCAA)	5589-96-3
Dibromoacetic acid (DBAA)	631-64-1
2,2-dichloropropanoic acid [Dalapon] (2,2-DCPA)	75-99-0
Iodoacetic Acid (IAA)	64-69-7
Bromodichloroacetic Acid (BDCAA)	71133-14-7
Chlorodibromoacetic Acid (CDBAA)	5278-95-5
Tribromoacetic Acid (TBAA)	75-96-7

LaSB Method: E3478 – The Determination of Haloacetic Acids (HAAs) and 2,2-Dichloropropionic Acid (2,2-DCPA) in Raw and Treated Water by Direct Aqueous Injection Liquid Chromatography- Tandem Mass Spectrometry (LC-MS/MS)

Method Principle: This is a direct aqueous injection liquid chromatography-tandem mass spectrometry (LC-MS/MS) method applicable to the determination of the parameters listed in Table 1 in drinking water, raw water and any intermediate steps in the water treatment process. Analytes are identified and quantified by comparing Multiple Reaction Monitoring (MRM) transitions of the precursor and product ions to those of the target compounds in the calibration standard curve.

Key characteristics of the method are:
- the use of a five level calibration curve during the LC-MS/MS analysis, correcting for the impact of changes to instrumental linearity;
- method control and instrumental control charts are provided for each run;
- new instrument calibration and control standards are prepared with each batch of samples.

LaSB Method: E3383 – The Determination of Haloacetic Acids (HAA) And Dalapon (2,2-Dichloropropanoic Acid) in Drinking Waters By Liquid-Liquid Extraction, Using Derivatization and Gas Chromatography - Mass Spectrometry (GC-MS) (2008)

Method Principle: This is a gas chromatographic-mass spectrometer (GC-MS) method applicable to the determination of the target parameters in drinking water, raw water and any intermediate step in the water treatment process. This method involves liquid-liquid extraction at pH < 0.5 (for improved extraction efficiency) and derivatization with diazomethane (to improve chromatographic properties).The analytes are qualitatively identified and quantified by their corresponding methyl esters.

Qualitative identification of target compounds is achieved by comparing target compound retention times (RTs) and intensities and ratios of target and qualifier ions to that of a calibration standard. quantitation of the target compounds detected in a field sample is achieved by comparing the target ion abundance with that of a calibration standard prepared in the laboratory.

US EPA Methods: Method 552, Determination of Haloacetic Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization, and Gas Chromatography with Electron Capture Detection (GC/ECD)

Method 552.1, Rev 1.0, Determination of Haloacetic Acids and Dalapon in Drinking Water by Ion-Exchange Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector

Method 552.2, Rev 1.0, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with an Electron Capture Detector

Method 552.3, Rev 1.0, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Gas chromatography with Electron Capture Detection

AWWA Method: [40] Method 6251 B – Micro Liquid-Liquid Extraction Gas Chromatographic Method

4.7. Hexavalent Chromium

PARAMETER	CAS Number
Hexavalent Chromium (Cr VI)	11104-59-9

LaSB Method: E3056 – The Determination of Hexavalent Chromium in Waters, Landfill Leachates and Effluents By Colourimetry

Method Principle: 1,5-diphenylcarbohydrazide reacts with chromium VI to give a reddish-purple colour, the absorption of which is measured spectrophotometrically at a wavelength of 540 nm.

US EPA Methods: Method 218.4, Chromium, Hexavalent (Atomic Absorption, chelation-extraction)

Method 218.5, Chromium, Hexavalent (Atomic Absorption, Furnace Technique)

Method 218.6 Rev 3.3, Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography

SW-846, Method 7197, Chromium, Hexavalent (Chelation/Extraction)

[40] *Standard Methods, 20th and 21st Ed.*

SW-846 Method 7198, Chromium, Hexavalent (Differential Pulse Polarography)

SW-846, Method 7199, Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography

AWWA Methods: [41] Method 3500-Cr B – Colorimetric Method

Method 3500-Cr C – Ion Chromatographic Method

ASTM Method:[42] Method D5257-03, Standard Test Method for Dissolved Hexavalent Chromium in Water by Ion Chromatography

4.8. Ortho-phosphate

PARAMETER
Ortho-phosphate

LaSB Method: E3364 – The Determination of Ammonia Nitrogen, Nitrite Nitrogen, Nitrite plus Nitrate Nitrogen and Reactive Ortho-Phosphate in Surface Water, Drinking Waters and Ground Waters by Colourimetry.

Method Principle: Phosphorus (as ortho-phosphate) is determined as one of four automated nutrient tests performed simultaneously on the same aliquot of sample. Ortho-phosphate is determined by the formation of a phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent. The absorbance of the solution is measured at 880 nm and the concentration of phosphorus is determined by comparison with a known set of standards.

US EPA Methods: Method 300.0 Rev 2.1, Determination of Inorganic Anions by Ion Chromatography

Method 300.1 Rev 1.0, Determination of Inorganic Anions in Drinking Water by Ion Chromatography

Method 365.1 Rev 2.0, Determination of Phosphorus by Semi-Automated Colorimetry

Method 365.2 Rev 2.0, Phosphorus, All Forms (Colorimetric, Ascorbic Acid, Single Reagent)

Method 365.3, Phosphorus, All Forms (Colorimetric, Ascorbic Acid, Two Reagent)

SW-846, Method 9056A, Determination of Inorganic Anions by Ion Chromatography

[41] *Standard Methods, 20th and 21st Ed.*
[42] Vol. 11.01, 2008

AWWA Methods: [43] Method 4500-P C – Vanadomolybdophosphoric Acid Colorimetric Method

Method 4500-P E – Ascorbic Acid Method

Method 4500-P F – Automated Ascorbic Acid Reduction Method

Method 4500-P G – Flow Injection Analysis for Orthophosphate

Method 4110 B – Determination of Anions by Ion Chromatography with Chemical Suppression of Eluent Conductivity

Method 4110 C – Determination of Anions by Single-Column Ion Chromatography with Direct Conductivity Detection

ASTM Methods:[44] Method D4327-03, Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

Method D6508-00 (2005), Standard Test Method for Determination of Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte

4.9. Phenolic Compounds – Total (4AAP)

PARAMETER
Phenol (4AAP)

LaSB Method: E3179 – The Determination of Phenolic Compounds in Water, Industrial Wastes, Landfill Leachates and Sewage by Colourimetry

Method Principle: The 4-aminoantipyrine (4-AAP) automated colourimetric method with an automated distillation step is used for routine river, lake, drinking water, industrial wastes, landfill leachates and sewage samples. An unfiltered sample (preserved to pH 1.5–2 with sulphuric acid) aliquot enters the autoanalyzer system at the automated continuous -flow distillation module. The distillate is mixed with a tartrate-borax buffer, pH 9.4, and 4-AAP to produce an antipyrine dye which is oxidized by alkaline ferricyanide. The absorbance of the red antipyrine dye is measured colourimetrically in a 5 cm flow cell at 505 nm.

The term "phenolic compounds" is applied here to those hydroxy-derivatives of benzene which react under the conditions of the tests, with the reagents used. The percentage composition of the phenolic compounds present in a given sample is unpredictable. A standard mixture could not, therefore, be applicable to all samples. For this reason phenol is used as standard and any colour produced by reaction with the reagent is reported as phenol.

[43] *Standard Methods, 20th and 21st Ed.*
[44] Vol. 11.01 and 11.02, 2008

US EPA Methods: Method 420.2, Phenolics, Total Recoverable (Colorimetric, Automated 4-AAP with Distillation)

Method 420.3, Phenolics, Total Recoverable (Spectrophotometric, MBTH with Distillation)

Method 420.4 Rev 1.0, Determination of Total Recoverable Phenolics by Semi-Automated Colorimetry

SW-846, Method 9066, Phenolics (Colorimetric, Automated 4-AAP with Distillation)

SW-846, Method 9067, Phenolics (Spectrophotometric, MBTH with Distillation)

AWWA Methods: [45] Method 5530 B – Cleanup Procedure

Method 5530 C – Chloroform Extraction Method

Method 5530 D – Direct Photometric Method

ASTM Method:[46] Method D1783-01 (reapproved 2007), Standard Test Methods for Phenolic Compounds in Water

4.10. Silica

PARAMETER
Silica

LaSB Method: E3370 – The Determination of Molybdate Reactive Silicates and Dissolved Carbon in Water, Industrial Waste and Precipitation by Colourimetry

Method Principle: The method used is based upon the formation of the molybdenum heteropoly blue complex and measures only dissolved reactive silicate anions. Silica, in the soluble colloidal form or the insoluble polymerized form, does not react and, therefore, is not detected by this method. The extent to which the various forms of silica may be hydrolysed to a molybdate reactive form under the test conditions is presently unknown. Similarly, it is not known to what extent dissolved poly-silicates and silica are present in natural waters. The detection criterion of this method is significantly better than measurement by atomic absorption spectrometry (AAS).

Ammonium molybdate at pH 1.2 reacts with silicates to produce a yellow molybdosilicic acid complex. Since phosphates also react to produce a yellow phosphomolybdate complex under the same test conditions, oxalic acid is added to destroy the phosphorus chromophore. A reducing agent, ascorbic acid, is used to convert the yellow molybdosilicic acid to the heteropoly blue complex. An

[45] *Standard Methods, 20th and 21st Ed.*
[46] Vol. 11.02, 2008

auto-analyzer system is used to measure the absorbance of the coloured solution at 660 nm. The absorbance is proportional to the reactive silicate concentration in the original sample.

US EPA Methods: Method 370.1 Rev 1, Silica, Dissolved (Colorimetric)

Method 200.5, Rev 4.2, Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma – Atomic Emission Spectrometry

Method 200.7 Rev 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma - Atomic Emission Spectrometry

AWWA Methods: [47] Method 4500-SiO_2 C – Molybdosilicate Method

Method 4500- SiO2 D – Heteropoly Blue Method

Method 4500-SiO2 E – Automated Method for Molybdate-Reactive Silica

Method 4500-SiO2 F – Flow Injection Analysis for Molybdate-Reactive Silicate

ASTM Method:[48] Method D859-05, Standard Test Method for Silica in Water

4.11. Taste and Odour Compounds

PARAMETER	CAS Number
2-methylisoborneol (MIB)	2371-42-8
Geosmin	19700-21-1
2-isopropyl-3-methoxypyrazine (IPMP)	25773-40-4
2-isobutyl-3-methoxypyrazine (IBMP)	24683-00-9
2,3,6-trichloroanisole (236-TCA)	50375-10-5
2,4,6-trichloroanisole (246-TCA)	87-40-1

LaSB Method: E3310 – The Determination of Taste and Odour Compounds in Water by Gas Chromatography-High Resolution Mass Spectrometry (GC-HRMS)

Method Principle: This method is designed to identify and quantify geosmin, 2-methylisoborneol, 2-isopropyl-3-methoxypyrazine (IPMP), 2-isobutyl-3-methoxypyrazine (IBMP), 2,3,6-trichloroanisole (236-TCA) and 2,4,6-trichloroanisole (246-TCA) in water by adsorption onto the granular adsorbent Ambersorb 572, elution into an organic solvent and analysis by high resolution (capillary column) gas chromatography-high resolution mass spectrometry (GC-HRMS).

The internal standards d_3-geosmin, d_3-2-MIB, 2-sec-butyl-3-methoxypyrazine (s-BMP) and d_5-246-TCA are added to the sample (1 L) upon receipt at the laboratory. A 500 mL aliquot of the sample is analysed. After the addition of

[47] *Standard Methods, 20th and 21st Ed.*
[48] Vol. 11.01, 2008

Ambersorb 572, the bottle is rolled at 50 rpm on a roller apparatus for 1 hour. The granular adsorbent is isolated by filtration on filter paper and allowed to air dry for 1 hour, minimum. The Ambersorb 572 is transferred to a 2 mL autosampler vial and approximately 400 µL of dichloromethane are added. The vial is capped and the contents are analyzed by GC-HRMS. Geosmin, 2-MIB and 246-TCA are quantified by isotope dilution with d_3-geosmin, d_3-2-MIB and d_5-246-TCA respectively. IPMP/ IBMP as well as 236-TCA, are quantified using the internal standards sBMP and d_5-246-TCA respectively.

AWWA Methods: [49] Method 6040 B – Closed-Loop Stripping, Gas Chromatographic/Mass Spectrometric Analysis

Method 6410 B – Liquid-Liquid Extraction Gas Chromatographic/Mass Spectrometric Method; NOTE: the laboratory must be able to demonstrate that it can achieve an MDL of 5 ng/L or lower for this method to be acceptable to the MOE for the analysis of Ontario drinking water samples.

AWWA Method: [50] Method 6040 D – Solid-Phase Microextraction (SPME)

4.12. Total Kjeldahl Nitrogen (TKN)

PARAMETER
Total Kjeldahl Nitrogen (TKN)

LaSB Method: E3367 – The Determination of Total Kjeldahl Nitrogen and Total Phosphorus in Water and Precipitation by Colourimetry

Method Principle: Samples are analyzed for TKN via a semi-automated procedure that includes batch digestion, automated neutralization, and automated colourimetry. Aliquots of samples are digested, in block digesters, with Kjeldahl's reagent to convert the tri-negative nitrogen content of organic compounds to ammonium ions (highly acidic media). After restoring the volume integrity, the sample is presented to an auto-analyzer system where it is neutralized in two stages, and then analyzed for ammonia species using phenate-hypochlorite colourimetry. The absorbance of the blue dye is measured at 630 nm. The TKN concentration is determined by comparison with a known set of standards.

US EPA Methods: Method 351.1, Rev 2, Nitrogen, Kjeldahl, Total (Colorimetric, Automated Phenate)

Method 351.2, Rev 2.0, Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry

Method 351.3, Rev 2, Nitrogen, Kjeldahl, Total (Colorimetric; Titrimetric; Potentiometric)

[49] *Standard Methods, 20th and 21st Ed.*
[50] *Standard Methods, 21st Ed.*

Method 351.4, Nitrogen, Kjeldahl, Total (Potentiometric, Ion Selective Electrode)

AWWA Methods: [51] Method 4500-N_{org} B – Macro-Kjeldahl Method

Method 4500-Norg C – Semi-Micro-Kjeldahl Method

Method 4500-Norg D – Block Digestion and Flow Injection Analysis

Method 4500-P J – Persulfate Method for Simultaneous Determination of Total Nitrogen and Total Phosphorus

ASTM Method: [52] D3590-02 (reapproved 2007), Standard Test Methods for Total Kjeldahl Nitrogen in Water

4.13. Total Phosphorus

PARAMETER
Total Phosphorus

LaSB Method: E3367 – The Determination of Total Kjeldahl Nitrogen and Total Phosphorus in Water and Precipitation by Colourimetry

Method Principle: For the determination of total phosphorus, samples are digested in sulphuric acid-mercuric oxide-potassium sulphate media using 3 block digesters maintained at 180°C, 210°C and 360°C. The orthophosphate content in the digestate is determined by the formation of a reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent. Acidity levels are controlled throughout analysis.

US EPA Methods: Method 365.1 Rev 2.0, Determination of Phosphorus by Semi-Automated Colorimetry

Method 365.2 Rev 2.0, Phosphorus, All Forms (Colorimetric, Ascorbic Acid, Single Reagent)

Method 365.3, Phosphorus, All Forms (Colorimetric, Ascorbic Acid, Two Reagent)

Method 365.4 - Phosphorus, Total (Colorimetric, Automated, Block Digestor AA II)

Method 200.7 Rev 4.4, Determination of Metals and Trace Elements by ICP-Atomic Emission Spectrometry

AWWA Methods: [53] Method 4500-P B – Sample Preparation

[51] *Standard Methods, 20th and 21st Ed.*
[52] Vol. 11.02, 2008

Method 4500-P E – Ascorbic Acid Method

Method 4500-P F – Automated Ascorbic Acid Reduction Method

Method 4500-P H – Manual Digestion and Flow Injection Analysis for Total Phosphorus

Method 4500-P I – In-line UV/Persulphate Digestion and Flow Injection Analysis for Total Phosphorus

Method 4500-P J – Persulfate Method for Simultaneous Determination of Total Nitrogen and Total Phosphorus

4.14. Formaldehyde

PARAMETER	CAS Number
Formaldehyde	50-00-0

LaSB Method: E3428 – The Determination of Formaldehyde in Environmental Matrices by Gas Chromatography-Mass Spectrometry [GC-MS] (2004)

Method Principle: This method was designed to identify and quantify formaldehyde (FORM) in water by reacting the aldehyde with 2,4-dinitrophenylhydrazine (DNPH) to form a hydrazone derivative which is extracted from the sample by liquid/liquid extraction and analyzed by gas chromatography-mass spectrometry [GC-MS].

The internal standard d_2-formaldehyde is added to a 150 mL aliquot of the sample. An acidified 2,4-dinitrophenylhydrazine (DNPH) solution is added to the sample and allowed to react for 0.5 hr. The 2,4-dinitrophenylhydrazone derivative (FORM-DNPH) is formed and extracted from the water sample by partitioning into hexane. The hexane extract is washed with water, dried through sodium sulphate and concentrated to approximately 100 µL. This extract is analyzed by GC-MS or GC-HRMS. Quantitation of formaldehyde is done by isotope dilution with d_2-formaldehyde and monitoring the molecular ions of the DNPH derivatives at m/z 210 and m/z 212 respectively.

US EPA Methods: Method 1667A, Determination of Formaldehyde, Isobutyraldehyde and Furfural in *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater*, EPA 821/B-98-002, July 1998.

Method 554 Rev 1.0, Determination of Carbonyl compounds in Drinking Water by Dinitrophenylhydrazine Derivatization and High Performance Liquid Chromatography

[53] *Standard Methods, 20th and 21st Ed.*

AWWA Method: [54] Method 6252 B – Disinfection By-Products: Aldehydes by PFBHA Liquid-Liquid Extraction Gas Chromatographic Method

4.15. Emerging Complex Contaminants

Emerging complex contaminants are under continual review by various government jurisdictions. In many cases, analytical methods are in development and reference methods are not yet established for these compounds. The following parameters and groups of compounds fall into this category, but this list is not comprehensive. A limited number of methodologies are provided, but the list is not comprehensive and analysis is not limited to these methods. The principles of "Good Laboratory Practice" should be applied for any method use to analyze drinking water samples for these types of compounds and complete method validation records must be maintained.

PARAMETER/COMPOUND GROUP
Pharmaceuticals and Personal Care Products (PPCP)
Brominated Flame Retardants (BFR)
PFOS (Perfluorooctane Sulfonates)
PFOA (Perfluorooctanoic Acid)
Nonylphenol & Ethoxylates

LaSB Method: E3430 – The Determination of Polybrominated Diphenyl Ethers (PBDEs) in Environmental Matrices By Gas Chromatography-High Resolution Mass Spectrometry (GC-HRMS)

Method Principle: This analytical method is used to determine the concentrations of tri to deca substituted PBDEs in a variety of matrices using gas chromatography-high resolution mass spectrometric detection. Aqueous sample volumes are measured and recorded. Samples are filtered using a C_{18} solid phase adsorption disk. PBDEs from particulate matter are trapped on top of the disk while the soluble analytes are adsorbed. Toluene/ethanol is used to extract the PBDEs from the disk and particulate matter. The extracts are cleaned using a single stage silica (acid/base/$AgNO_3$) cleanup. All samples are fortified with at least one [$^{13}C_{12}$] isotopically labelled congener for each homologue group (quantification standard). All PBDEs are quantified against these corresponding [$^{13}C_{12}$] labelled internal standards.

LaSB Method: E3457 - The Determination of Perfluorinated Alkyl Compounds (PFCs) in Environmental Matrices by Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

Method Principle: This method is designed to identify and quantify 11 perfluorinated alkyl compounds in various environmental matrices. Solid samples (sediments, soils, biosolids and fish) are homogenized, mixed with an ion paring agent (TBAS), extracted with methyl tert-butyl ether (MTBE), evaporated to dryness and

[54] *Standard Methods, 20th and 21st Ed.*

reconstituted in methanol. Water samples are either analysed directly or concentrated by solid phase extraction prior to instrument analysis depending on detection limit requirements. All target compounds are analysed by C18 reversed phase liquid chromatography-negative electrospray tandem mass spectrometry [LC-(ESI$^-$)MS/MS].

C13-labeled surrogate ($^{13}C_4$-PFOA) and internal standards ($^{13}C_2$-PFOA, $^{13}C_4$-PFOS, $^{13}C_5$-PFNA, $^{13}C_2$-PFDA and $^{13}C_2$-PFDoA) are added during sample processing/instrument analysis to monitor method performance and compensate for instrument variability. PFCs are quantified by internal standard methods using a multipoint external calibration curve.

US EPA Methods: Method 527, Rev 1.0, Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

Method 537, Ver 1.0, Determination of Selected Perfluorinated Alkyl Acids in drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)

ASTM Method:[55] D7065-06, Standard Test Methods for Determination of Nonylphenol, Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethyoxylate and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass Spectrometry

5. ACRONYMS

4-AAP	4-aminoantipyrine
AAS	Atomic Absorption Spectroscopy
AES	Atomic Emission Spectroscopy
AMPA	Aminomethyl-phosphonic Acid
ASTM	ASTM International (formerly the American Society for Testing and Materials)
ASV	Anodic Stripping Voltammetry
AWWA	American Waterworks Association
CALA	Canadian Association for Laboratory Accreditation
CAS	Chemical Abstract Services
CBs	Chlorinated Benzenes
CFU	Colony Forming Units
CV-AAS	Cold Vapour-Flameless Atomic Absorption Spectrophotometry
DC Agar	Differential Coliform Agar
E. coli	*Escherichia coli*
ELISA	Enzyme-Linked Immunosorbent Assay
ESI-MS	Electro-Spray Ionization Mass Spectrometry
GC/MS	Gas Chromatography-Mass Spectrometry
GC-HRMS	Gas Chromatography-High Resolution Mass Spectrometry
HPLC	High Performance Liquid Chromatography

[55] Vol. 11.02, 2008

IC	Ion Chromatography
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
LaSB	Laboratory Services Branch
mg/L	Milligrams per Litre
MIB	2-methylisoborneol
MOE	Ministry of the Environment (Ontario)
MPN	Most Probable Number
MS-MS	Tandem Mass Spectrometry
NDMA	N-Nitrosodimethylamine
O Reg	Ontario Regulation
OCs	Organochlorine Pesticides
ODWQS	Ontario Drinking Water Quality Standards
P/A or P-A	Presence/Absence [applies to microbiological tests]
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
RDL	Reporting Detection Limit
SCC	Standards Council of Canada
SPE	Solid Phase Extraction
TCLP	Toxicity Characteristic Leaching Procedure
TEF	Toxic Equivalent Factor
TEQ	Toxic Equivalent Quantity
TKN	Total Kjeldahl Nitrogen
US EPA	United States Environmental Protection Agency
UV	Ultraviolet
WHO	World Health Organization
µg/L	Micrograms per Litre

6. HISTORY OF CHANGES

6.1 Version 1.0, March 17, 2008

Section 1

- added e-mail address for requesting copies of LaSB methods
- updated AWWA method references to include 21st Edition, 2005
- updated ASTM method references to the 2006 edition
- added AOAC International as a source of accepted methods
- added reference to MOE document *Protocol for Acceptance of Alternate Methods*

NOTE: Additions and deletions of specific methods in Sections 2 and 3 have not been listed.

Section 2

- reformatted description of method principle for Total Coliforms (2.1) and *E. coli* (2.2)
- removed references to Fecal Coliforms

- removed references to Total Coliform background counts and included note that Heterotrophic Plate Count is no longer required to be reported (2.3)
- added sections 2.4 *Clostridium* and 2.5 *Cryptosporidium*

Section 3

- added CAS (Chemical Abstract Service) registry number for all applicable parameters
- updated ODWQS and RDL for Trichloroethylene to reflect changes made in 2006
- removed trace metals that are considered operational parameters, i.e. not included in O Reg 169/03 (3.2)
- updated significant figures for ODWQS for lead (O. Reg. 169/03 amended to O. Reg. 242/07)
- changed units for RDLs of Triazines (3.5) to correspond to units required in DWIS
- changed units for RDLs of Carbamates (3.6) to correspond to units required in DWIS; also corrected RDL of Aldicarb to $1/10^{th}$ of the ODWQS
- changed units for RDLs of Organochlorine Pesticides (3.7) to correspond to units required in DWIS
- changed units for RDLs of Chlorophenols & Phenoxy Acids (3.9) to correspond to units required in DWIS
- changed units for RDLs of Urea Derivative (3.11) to correspond to units required in DWIS; also added LaSB method reference number
- corrected the equations for the reactions (Figure 1) for Glyphosate (3.12)
- clarified analytical technique (colourimetry) for several methods referenced for Fluoride (3.13)
- updated toxic equivalent factors (TEF) for dioxins and furans from NATO values in the June 2003 version of this document to the 2006 values from the World Health Organization (WHO); included phrase that these values may be amended from time to time; included direction that laboratories must identify the source of the TEFs used for their calculations
- added section 3.20 Microcystin LR and 3.21 Chloramines

Section 4

- added CAS (Chemical Abstract Service) registry number for all applicable parameters
- removed references to the following operational parameters (i.e. not included in O Reg 169/03): Alkalinity, Chloride, Colour, Dissolved Organic Carbon, Hardness, pH, Sulphate, Sulphite, Total Dissolved Solids, and Turbidity
- added the following new parameters or target groups:

Ammonia (4.2)
Biochemical Oxygen Demand (4.3)
Bromide, Chlorate and Chlorite (4.4)
Chemical Oxygen Demand (4.5)
Haloacetic Acids (4.6)
Hexavalent Chromium (4.7)
Ortho-phosphate (4.8)
Phenolic Compounds – Total [4AAP] (4.9)
Silica (4.10)
Taste and Odour Compounds (4.11)
Total Kjeldahl Nitrogen (4.12)
Total Phosphorus (4.13)
Formaldehyde (4.14)
Emerging Complex Contaminants (4.15)

Section 5

- added list of Acronyms

Section 6

- added History of Changes

6.2 Version 2.0, May, 2010

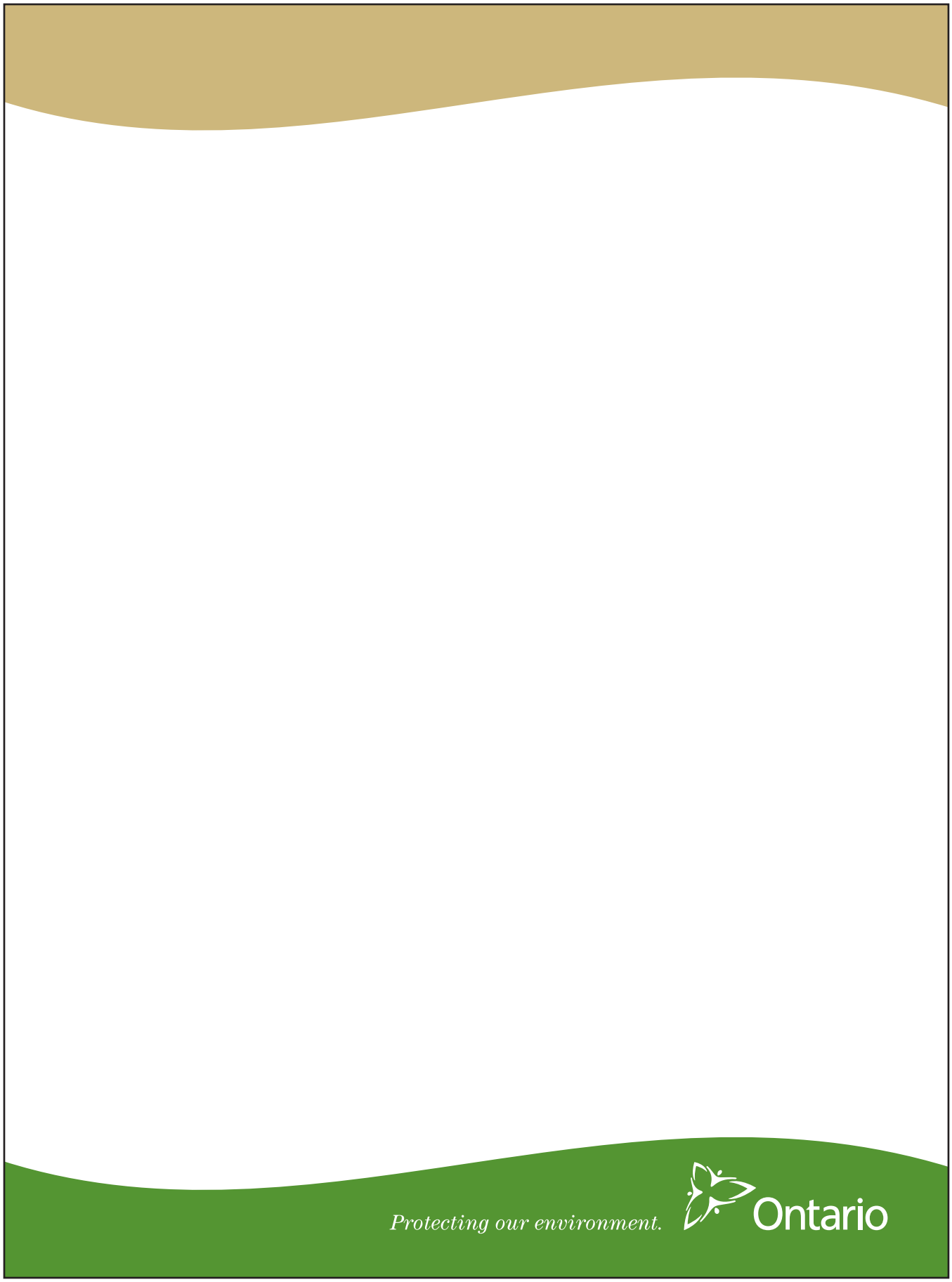
Section 1

- added Canadian Association for Laboratory Accreditation (CALA) as an approved accrediting body
- updated ASTM method references to the 2008 edition

NOTE: Additions and deletions of specific methods in Sections 2 and 3 have not been listed.

Section 2

- added section 2.20.1 Screening Tests for Total Microcystins
- updated acronyms


Protecting our environment.
Ontario