Asbestos in Water by Transmission Electron Microscopy (TEM) - Prescriptive

Parameter

Asbestos in water

Analytical Method

Analysis of Asbestos Fibers > 10 microns by Transmission Electron Microscopy (TEM)

Introduction

Asbestos is a general term for fibrous silicate minerals of the serpentine and amphibole mineral groups, which are widely distributed throughout the Earth's crust. Six commercially important minerals are generally characterized as asbestos. Chrysotile is the only member of the serpentine group and the form of asbestos that is mined in Canada. The amphiboles include crocidolite, amosite, tremolite, anthophyllite and actinolite. Asbestos is ubiquitous in the environment as a result of its extensive industrial use and the dissemination of fibres from natural sources. It is introduced into water by the dissolution of asbestos-containing minerals and ores and from industrial effluents, atmospheric pollution and, in some cases, asbestos-cement (A/C) pipe in distribution systems (Health Canada). BC has adopted the US EPA Drinking Water Guideline of 7 mf/L for total asbestos in water.

Method Summary

Water samples are submitted to the laboratory in polyethylene or glass bottles. Samples are shaken and known aliquots of each sample are filtered through either 0.22 µm mixed cellulose ester filters or 0.1µm polycarbonate filters. A carbon extraction replica is prepared from a portion of the filter, transferred to grids and examined in the TEM. Asbestos fibers are identified by selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXS). Only asbestos structures > 10µm are counted.

This method is prescriptive. The EPA 100.2 reference method must be followed exactly as written, except for minor deviations where indicated in the text below. The EPA 100.2 method was developed for drinking waters. This method is extended to include non-potable waters.

MDL(s) and EMS Analyte Code(s)

Analyte	CAS #	Approx. MDL mf/L	EMS Analyte Code
Amosite (grunerite)	12172-73-5	0.2	n/a
Chrysotile	12001-29-5	0.2	n/a
Crocidolite	12002-28-4	0.2	n/a
Actinolite	13768-00-8	0.2	n/a
Anthophyllite	17068-78-9	0.2	n/a
Tremolite	14567-72-8	0.2	n/a
Asbestos	1332-21-4	0.2	FIBR

Asbestos may exist as any of the above-listed minerals. CSR standards are for “Asbestos”, which represents the sum of the above-listed minerals. MDL is matrix specific. 0.2 mf/L (million fibres per litre) is achievable on clean samples. Particulates may necessitate increase of DLs. DLs below the BC CSR standard of 7 mf/L are fit for purpose.

EMS Method Code

Refer to EMS Parameter Dictionary on the ministry website for all current EMS codes.

Matrix

Drinking Water, Surface Water, Groundwater

Interferences and Precautions

Samples with substantial particulate may interfere, raising the detection limit. If the detection limit is raised beyond 7 mf/L additional sample preparation may be required.

If samples are suspected to contain high levels of organic contaminants, or if algal or bacterial growth causes interference with asbestos measurement to limits sufficient for evaluation of CSR standards, the ozone-UV treatment procedure described within EPA 100.2 may be conducted.

Sample Handling and Preservation

Glass or polyethylene bottles (500 ml or larger is recommended), no preservative, store at ≤ 6°C (do not freeze).

Stability

Holding Time: Samples should be filtered within 48 hours of sampling if possible to minimize algal growth. Maximum holding time prior to filtration is 7 days.

Storage: Store at ≤ 6°C (do not freeze) until filtration.

Procedure

The following is a brief summary of the EPA 100.2 procedure. Additional detail in the EPA method must be followed, except where differences are noted herein.

Water samples are submitted to the laboratory in polyethylene or glass bottles. Samples are shaken, placed in an ultrasonic bath for 15 minutes, and shaken again. A known aliquot, minimum 10 mL for a 25 mL filter and 50 mL for a 47 mm filter is filtered through either 0.22μm mixed cellulose ester filters (MCE) or 0.1μm polycarbonate filters.

The filter is dried and small sections removed from the middle of each filter. The sections are treated as follows:

For MCE filters, a few drops of a clearing solution (35% dimethyl formamide, 15% glacial acetic acid, 50% water) or acetone are placed a clean, labeled slide. Use just enough solution to saturate the filter. Carefully place the filter segment into the solution. Remove any excess solution. Dry the slide at 65 – 70°C. The slides are then plasma ashed (required for 0.22 μm filters, recommended for 0.1 μm).

The filter segments are then carbon coated using a high vacuum evaporation unit. Portions of the carbon coated segments are placed carbon side up in Jaffe washer on TEM grids and allowed to stand until the filter is adequately dissolved.

The samples are allowed to dry and are submitted for TEM analysis as per the reference method. Asbestos fibers are identified by selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXS). Only asbestos structures > 10μm are counted. Counting can be stopped at the completion of the grid opening in which an analytical sensitivity of 0.2 MFL or at the completion of the grid in opening which contains the 100th structure, whichever comes first. A minimum of 4 grid openings must be analyzed even if this results in the counting of more than 100 asbestos fibers over 10 μm in length.

For samples with high levels of particulate, it the required MDL cannot be achieved, additional preparation is required. Samples are dried, ashed, treated with HCl, reconstituted with DI water, filtered and processed as above. See NYS Environmental Laboratory Approval Program, Certification Manual, Item 198.4 (ELAP 198.4) for details.

Quality Control

Summary of QC Requirements*

QC Component	Minimum Frequency	Minimum Data Quality Objectives
Method Blank (MB)	One per 20 samples	Less than reported DL
Lab Duplicates (DUP) Re-Prepared Sample	One per 50 samples	50% RPD [or within 2 fibre counts for low level results]
Verified Count – Inter-analyst	One per 25 samples	50% RPD [or within 2 fibre counts for low level results]
Verified Count – Intra-analyst	One per 25 samples	50% RPD [or within 2 fibre counts for low level results]
If DQOs are not met, repeat testing or report qualified test results.		

Method Blank: A portion of DI water is filtered and analyzed as a Method Blank to control for lab contamination.

Lab Duplicates: For this method, a Lab Duplicate refers to a re-prepared sample. A separate portion of homogenized water sample is filtered, prepared, and counted. Counting may be inter-analyst or intra-analyst.

Verified Count – Inter-analyst: The same grid openings on the same sample and same filter are re-read by the same analyst.

Verified Count – Intra-analyst: The same grid openings on the same sample and same filter are re-read by a different analyst.

Reference Material: A CRM or SRM should be analyzed at least annually by each analyst.

See ELAP 198.4 for more details about recommended QC practices for asbestos methods.

References

1. EPA 100.2, Determination of Asbestos Structures over 10 µm in Length in Drinking Water, EPA/600/R-94/134, June 1994.
2. EPAP 198.4. NYS Environmental Laboratory Approval Program, Certification Manual, Item 198.4.
3. Health Canada 1989. Guidelines for Canadian Drinking Water Quality: Guideline Technical Document – Asbestos, March 1989.

Revision History

Sept 15, 2017 First version added to BC Lab Manual in support of 2017 CSR updates.