

# CCME, Vol. 4: Compendium of Analytical Methods for Contaminated Sites

## TECHNICAL BULLETIN

In 2016, the Canadian Council of Ministers of the Environment (CCME) published a new guidance manual for ["Environmental Site Characterization in Support of Environmental and Human Health Risk Assessment"](#). This manual was prepared by a joint private and public sector working group headed by Maxxam staff and extensively reviewed by CCME's Soil Quality Guidelines Task Group. Greatly anticipated, it provides consistency to sampling and analysis procedures, as well as to the quality of analytical data. The guidelines are largely based on current reference methodologies and approved practices in Canada and the US, specifically the Ontario Ministry of Environment and Climate Change (OMOEECC), Environmental Protection Agency (EPA) and British Columbia (BC) Guidelines.

### Background

The following elements of the 2016 CCME guidance manual were adopted:

- Alberta, Saskatchewan and Manitoba adopted it fully.
- The British Columbia Ministry of Environment adopted the data quality objectives. However, local regulations and guidelines are to be followed.
- The Quebec MDDLCC and Atlantic provinces adopted it partially, particularly for field preservation with methanol for VOCs.
- Federal sites in Ontario use the guidance. For all other sites, the analytical methods used to support site assessments are governed by the Analytical Protocols document referenced in Ontario Regulation 153/04.

This technical bulletin highlights changes from the previous volume and summarizes key methods and practices.

### Parameter Groups

Under the 2016 CCME guidance, parameters have been consolidated and grouped in accordance with classes of contaminants, such as Petroleum Hydrocarbons and Volatile Organic Compounds. This is in contrast to the previous version published in 1993, which classified parameters based on applicable instrumentation or analytical methodologies.

Recommendation is provided towards analysis and reporting on the full list of parameters, regardless of suite, rather than individual parameters, to account for possible associated contaminants or degradation products. In addition, CCME recommends that a Qualified Person (QP) uses Open Characterization analyses when anthropogenic sources of impact are identified.

A notable addition to the list of parameters includes Perfluoro Alkyl Substances (PFAS) with 13 of the most commonly identified contaminants. It should be noted that no actual concentration guidelines are yet developed for the majority of these compounds, however this class of contaminants continues to receive attention due to its persistent and recalcitrant nature.

Maxxam has packaged PFC parameters to offer standard (2 or 17 parameters), extended (23 parameters) or full (25 parameters) lists.

### Sample Handling and Storage Requirements

CCME adopted the use of sample collection methodologies that have increased the ease of field procedures and improved parameter stability. On a general basis, samples should be submitted  $\leq 10^{\circ}\text{C}$ ; for samples submitted to the laboratory the same day as collected. Temperatures above

10°C are acceptable so long as cooling media has been added. This guideline is in specific reference to organic and other parameters that may be affected by volatilization or biodegradation.

### Sample Collection Procedures for BTEX, F1, VOCs and THMs in Soil

CCME specifies the use of either field preservation with methanol / sodium bisulphate or the use of hermetic sampling devices as the only suitable methods for the collection of volatile organic contaminants. A coring device may be used to extrude approximately 5g of soil into a pre-charged 40mL methanol vial. Alternatively, the coring device may be a hermetic sampler, which only requires capping and submission to the laboratory.

Care must be taken with regards to the applicable holding times as methanol extracts are considered stable for only 40 days from sample collection. CCME recommends separating the methanol extract from the soil material for prolonged storage. Hermetic sampling devices must be processed at the laboratory within 48 hours (i.e. extrusion in methanol, for extraction). Alternatively, hermetic devices may be frozen within 48 hours from collection to extend the holding time to 14 days.

### Physical Parameters and Inorganics in Soil

For stable parameters and the physical testing of soil, samples may be collected in polyethylene terephthalate (PET) or polypropylene (PP) plastic bags. This includes chloride, pH, conductivity, particle size, fraction organic carbon (FOC), total nitrogen, metals, sodium absorption ratio (SAR) and hot water soluble boron (HWSB). Please note that minimum volume requirements still apply, depending on the type or number of analyses performed from a bagged sample.

## Analytical Methods

### Analysis of PFAS

The use of Teflon lining for sample collection of PFAS parameters is prohibited. Polyethylene (PE)-lined lids must be used instead.

Soil samples must be extracted at the laboratory within 14 days. These extracts are stable for an additional 40 days from collection. Water samples must be analyzed within 14 days if preserved at the time of collection, or within 7 days if unpreserved.

### Analysis of Hydrocarbons

CCME prescribes using silica gel for soil sample extracts to remove polar materials that would contribute to quantified concentrations of hydrocarbons in the F2 to F4 (C<sup>10</sup>-C<sup>50</sup>) range. The guidance indicates that using silica is a single step performed either in-situ or ex-situ and is optional for water samples.

For samples that are identified to contain high concentrations of organic matter and, consequently, saturate the silica gel used in the clean-up step, the guidance provides the following acceptable strategies:

- Apply a second aliquot of the extract to be treated with a larger weight of silica than normally or originally used;
- Employ the use of GC/MS analysis to identify non-petrogenic components that may be contributing to the extractable PHC concentrations;
- Compare against background samples from quantitative and qualitative perspectives, for confirmation.

In addition, corrected concentration values for hydrocarbons in the F1, F2 and F3 ranges must be reported whenever BTEX and PAH compounds are also analyzed on the same sample: F1-BTEX, F2-naphthalene and F3-PAHs. PAHs in this context only refer to: phenanthrene, benz[a]anthracene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, fluoranthene, dibenz[a]anthracene, indeno[1,2,3-c,d]pyrene and pyrene.

### Analysis of Phenolic Compounds

CCME no longer identifies the 4-Aminoantipyrine (4-AAP) reagent (colorimetry) as an acceptable method. Due to the fact that the method only provides a total result and is inefficient at recovering many phenolic species, it has been replaced with a parameter-specific method by GC/MS. The list is comprised of a wide range of chlorinated and non-chlorinated phenols. The non-chlorinated phenols are grouped into two subsets: mono- & dihydric- phenols and non-chlorinated phenols.

## Required Quality Assurance and Quality Control

CCME clearly defines the reporting limits to eliminate confusion and promote consistency in terminology used across the industry. The Laboratory Reporting Limit (LRL) is the limit at which analytes in all matrices are consistently and confidently quantified. In contrast, the Method Detection Limit (MDL) is the limit at which the laboratory can consistently and

confidently identify the presence of the analyte. The LRL must be greater than or equal to the MDL and typically, the LRL is 3 to 10 times larger than the MDL.

Several other quality control related elements are prescribed in the guidance document, as indicated below.

#### Minimum Reporting

Laboratories must provide, in the standard Certificates of Analysis, information on:

1. Time markers;
2. Data reportable;
3. Quality control reportables;
4. Analysis reportables;
5. Remarks and comments; and
6. Subcontracted analyses.

#### Uncertainty of Measurement

Laboratories are required to calculate the estimated uncertainty of measurement for all analytes. This information is included on the Certificate of Analysis on request.

#### Quality Control (QC) Alert Limits

Guidance is provided for field and laboratory QC samples. Specific alert limits are provided for each laboratory QC parameter and specific to the various groups of parameters.

CCME indicates that it is generally accepted for field QC alert limits to be 1.5 to 2 times broader than the laboratory limits.

With regards to laboratory duplicates, as the measured result approaches the LRL, uncertainty associated with the Relative Percent Difference (RPD) increases dramatically. To account for this, duplicate acceptance criteria are either the tabulated RPD acceptance limits or an absolute difference of  $2 \times \text{LRL}$  (for low level data). For example, if the LRL is 10, duplicates of 15 and 30 are acceptable (difference of 15, acceptance  $2 \times \text{LRL} = 20$ ).

#### About Us

Maxxam is a leading North American provider of analytical services and solutions to the energy, environmental, food, Industrial Hygiene and DNA industries. We are a member of the Bureau Veritas Group of companies – a world leader in testing, inspection and certification services. We support critical decisions made by our customers through the application of rigorous science and the knowledge and expertise of over 2,500 employees.

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