

Alcohols in Soil and Water - PBM

Parameter	Alcohols, including methanol, in soil and water.																																				
Analytical Method	Direct injection – Gas chromatography with Flame Ionization Detection (GC/FID) - PBM.																																				
Introduction	This method is applicable to the quantitative determination of alcohols in soil and water.																																				
Method Summary	<p>Direct injection: A portion of the water or aqueous extract is transferred to an autosampler vial, and is injected into the GC inlet (on-column), for direct analysis by GC/FID. This method yields detection limits of approximately 1 mg/L and 1 mg/kg, which is well below regulatory guidelines. If lower reporting levels are required, samples may be analyzed by GC/FID and headspace (US EPA method 5021a) (e.g. for methanol).</p> <p>Headspace: a portion of the water sample or aqueous soil extract is transferred to a headspace vial containing salt. The vial is sealed and heated. When equilibrated, a portion of the headspace above the liquid is introduced into the GC/FID.</p> <p>This method is performance-based. Laboratories may adopt alternative options to improve performance or efficiency provided that all stated performance requirements and prescribed (mandatory) elements are met.</p>																																				
MDL(s) and EMS Analyte Codes	<table border="0" style="width: 100%;"> <thead> <tr> <th style="text-align: left;"><u>Analyte</u></th> <th style="text-align: center;"><u>Approx. Soil MDL (µg/g)</u></th> <th style="text-align: center;"><u>Approx. Water MDL (µg/L)</u></th> <th style="text-align: center;"><u>EMS Analyte Code</u></th> </tr> </thead> <tbody> <tr> <td>Methanol</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">M020</td> </tr> <tr> <td>Ethanol</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>n-Propanol</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>2-Propanol (Isopropanol)</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>n-Butanol</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>2-Butanol</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>2-Methyl-1-propanol (Isobutanol)</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> <tr> <td>2-Methyl-2-propanol (t-Butanol)</td> <td style="text-align: center;">0.5 – 5.0</td> <td style="text-align: center;">500 – 1000</td> <td style="text-align: center;">N/A</td> </tr> </tbody> </table>	<u>Analyte</u>	<u>Approx. Soil MDL (µg/g)</u>	<u>Approx. Water MDL (µg/L)</u>	<u>EMS Analyte Code</u>	Methanol	0.5 – 5.0	500 – 1000	M020	Ethanol	0.5 – 5.0	500 – 1000	N/A	n-Propanol	0.5 – 5.0	500 – 1000	N/A	2-Propanol (Isopropanol)	0.5 – 5.0	500 – 1000	N/A	n-Butanol	0.5 – 5.0	500 – 1000	N/A	2-Butanol	0.5 – 5.0	500 – 1000	N/A	2-Methyl-1-propanol (Isobutanol)	0.5 – 5.0	500 – 1000	N/A	2-Methyl-2-propanol (t-Butanol)	0.5 – 5.0	500 – 1000	N/A
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EMS Method Codes	***Refer to EMS Parameter Dictionary on the ministry website for all current EMS codes.																																				
Matrix	<p>Soil: Soil, Sediment, Sludges, Solid wastes.</p> <p>Water: Freshwater, Seawater, Groundwater, Wastewater,</p>																																				
Interferences and Precautions	<ol style="list-style-type: none"> 1. Interferences by other oxygenated compounds and petroleum hydrocarbons. Matrix spikes may be used to correctly identify the compounds. 2. Hydrocarbon interferences may be removed by adding hexane and short physical agitation. 3. Isobutane interferes with methanol on a DB1 column. A more polar column is suggested (e.g. DB-624). 4. Contamination of the analytical system can occur after high level samples are analyzed. Analysts should be aware of the degree of carry-over that occurs on their instrument and should take appropriate steps to prevent the occurrence of false positives. 																																				
Sample Handling and Preservation	<p>Container:</p> <p style="margin-left: 20px;">Soil: 125 mL glass jar with Teflon (or foil) lined lid</p> <p style="margin-left: 20px;">Water: (minimum of) 2 40-60 mL septum capped glass vials</p> <p>Preservation:</p> <p style="margin-left: 20px;">Soil: Samples are not preserved. When the analysis includes methanol, or to meet low RLs, it is suggested to collect the sample in 2 vials of aqueous NaHSO₄ (2:1</p>																																				

water to soil ratio). A hermetic container could also be used, but this has a hold time of only 48 h.

Water: NaHSO₄ to pH <2

Stability

Holding Time:

Soil: Analyze soil samples within 14 days after collection.

Water: Analyze preserved water samples within 14 days.

Where holding times are exceeded, data must be qualified.

Storage: Store samples at ≤ 10°C during shipment to the laboratory and at ≤ 6°C at the laboratory.

Procedure

Soil samples are subjected to aqueous extraction prior to direct injection of the aqueous extract. Soils are extracted in water using a 2:1 ratio. Soil samples are physically agitated using a mechanical shaker for approximately 1 hour. Suspended particles may be filtered through a 0.45 µm membrane, or centrifuged until clear. Moisture is performed on a separate aliquot and results are reported on a dry-weight basis.

Water samples are analyzed by direct aqueous injection by gas chromatography-flame ionization detector (GC/FID). When a large amount of suspended material is present, the sample may be filtered through a 0.45 µm membrane.

Detailed instrumental procedures are not provided for this method. The procedures described in reference 1 are suitable for general guidance.

Performance Requirements

Any analytical method options selected for this analysis must meet or exceed the performance requirements specified below.

Accuracy and Precision requirements are distinct from daily QC requirements, and apply to measures of long term method performance (averages and standard deviations). Achievement of these requirements is to be demonstrated during initial and ongoing method re-validation studies. For Initial Validations, averages of at least 8 Lab Control Samples or RMs must be assessed. Ongoing Re-validations (performance reviews) should assess QC data encompassing longer timeframes (e.g. 6 months to 1 year). A minimum frequency of 2 years is recommended for Ongoing Re-validations.

Accuracy Requirement: Laboratories must demonstrate method accuracy (measured as average recovery) of 60% or better for Lab Control Samples or Certified Reference Materials at concentrations above ten times the MDL.

Precision Requirement: Laboratories must demonstrate method precision equal to or better than 30% relative standard deviation for clean matrix spikes at concentrations above ten times the MDL.

Sensitivity Requirement: Where possible, the method should support Reporting Limits (and MDLs) that are less than 1/5 of applicable numerical standards. The method is not fit-for-purpose if an MDL exceeds a guideline, standard, or regulatory criteria against which it will be used for evaluation of compliance.

Quality Control

Summary of QC Requirements		
QC Component	Minimum Frequency	Minimum Data Quality Objectives
Method Blank (MB) - matrix specific	One per batch (max 20 samples)	Less than reported DL
Calibration Verification Standard (CVS) – 2 nd Source	One per initial calibration	85 – 115%
Continuing Calibration verification (CCV). midpoint	At the beginning of the batch, after every 20 samples and at the end of the batch	+/- 20% (beginning and mid) +/- 30% at end of batch of initial calibration
Lab Control Sample (LCS) or Reference Material (RM)	One per batch (max 20 samples)	70 – 130% for soil 80 – 120% for water

Lab Duplicates (DUP)	One per batch (max 20 samples)	50% RPD for soil 30% RPD for water [or within 2x reported DL for low level results]
Matrix Spike (MS)	One per batch (max 20 samples)	60-140% for soil 70-130% for water
Surrogate Compounds	Optional	Not specified
If DQOs are not met, repeat testing or report qualified test results. DQOs do not apply to MS results where sample background exceeds spike amount.		

Method Blank: Required. Minimum one per batch.

Lab Duplicates: Required.

Reference Material or Method Spike: Required.

Matrix Spike: Required.

Surrogate Compounds: Optional.

Prescribed Elements The following components of this method are mandatory:

1. Ensure calibration stability is monitored.
2. Calibrations must consist of a minimum of 4 calibration standards.
3. Samples that exceed the calibration range must be diluted and re-analyzed, or reported as estimated, or minimum values.
4. All stated performance and quality control requirements must be met.

Apart from these limitations, and provided performance requirements are met, laboratories may introduce modifications to this method in order to improve quality or efficiency. Laboratories must disclose to their clients where modified or alternative methods are employed.

- References**
1. US EPA method 8015D, Nonhalogenated organics using GC/FID (June 2003).
 2. BC MOE sample preservation & holding time requirements (Nov 06, 2015)

Revision History Mar 15, 2017 New method added to the BC Lab Manual to correspond with updates to the BC CSR. Effective date for this method is November 1, 2017.