

## Alcohols in Soil and Water - PBM

**Parameter** Alcohols in soil and water.

**Analytical Method** Direct injection or Headspace 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** **Direct injection:** A portion of the water or aqueous soil extract is transferred to an autosampler vial, and is injected into the GC inlet (on-column or splitless), for direct analysis by GC/FID, using a polar stationary phase (e.g. DB-Wax or DB-624 or equivalent). This method yields detection limits of approximately 1 mg/L and 1 mg/kg. If lower reporting levels are required (e.g. for methanol), GC/FID and headspace is recommended (US EPA method 5021A).

**Headspace:** a portion of the water sample or aqueous soil extract is transferred to a headspace vial containing salt as matrix modifier. The vial is sealed and heated. When equilibrated, a portion of the headspace above the liquid is introduced into the GC/FID.

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.

MDL(s) and EMS Analyte Codes	<u>Analyte</u>	<u>CAS No.</u>	<u>Approx.</u>	<u>Approx.</u>	<u>EMS</u>
			<u>Soil MDL</u> ( $\mu\text{g/g}$ )	<u>Water MDL</u> ( $\mu\text{g/L}$ )	<u>Analyte</u> <u>Code</u>
	Methanol	67-56-1	0.5 – 5	500 – 1,000	M020
	Ethanol	64-17-5	0.5 – 5	500 – 1,000	N/A
	n-Propanol	71-23-8	0.5 – 5	500 – 1,000	N/A
	2-Propanol (Isopropanol)	67-63-0	0.5 – 5	500 – 1,000	N/A
	n-Butanol	71-36-3	0.5 – 5	500 – 1,000	N/A
	2-Butanol	78-92-2	0.5 – 5	500 – 1,000	N/A
	2-Methyl-1-propanol (Isobutanol)	78-83-1	0.5 – 5	500 – 1,000	N/A
	2-Methyl-2-propanol (t-Butanol)	75-65-0	0.5 – 5	500 – 1,000	N/A

\*\*\*Refer to [EMS Parameter Dictionary](#) on the ministry website for all current EMS codes.

**Matrix** **Soil:** Soil, Sediment, Sludges, Solid wastes.

**Water:** Freshwater, Seawater, Groundwater, Wastewater

**Interferences and Precautions**

- Alcohols by GC-FID may be subject to interferences by any substance with similar boiling points and chromatographic characteristics that may co-elute under the conditions of the analysis (e.g. by alkanolamines or petroleum hydrocarbons). Matrix spikes and/or GC/MS confirmation is recommended to assist with correct identification of target compounds in samples if co-eluting interferences are suspected.
- Hydrocarbon interferences may be removed with a hexane pre-extraction cleanup step, using brief physical agitation.
- Isobutane interferes with methanol on a dimethyl polysiloxane column (e.g. DB-1). Use of a polar column phase is recommended to resolve this interference (e.g. DB-624).
- 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** **Sample Containers:**

**Soil:** Glass jar with Teflon (or foil) lined lid, 125 – 250 mL recommended.

**Water:** 40-60 mL glass vials with Teflon septa and zero headspace (2 vials per sample recommended).

**Preservation:**

**Soil:** Chemical preservation is not required. Collect samples with minimized headspace.

**Water:** Samples may be preserved with NaHSO<sub>4</sub> to pH <2 to extend hold times (200 mg solid NaHSO<sub>4</sub> per 40 mL sample is recommended).

**Storage:** Store samples at ≤ 10°C during shipment to the laboratory and at ≤ 6°C at the laboratory. Avoid freezing to prevent sample breakage.

**Stability**

**Holding Time:**

**Soil:** Analyze soil samples within 14 days after collection.

**Water:** Analyze unpreserved water samples within 7 days after collection. Acid preservation extends hold times to 14 days from sampling.

Where holding times are exceeded, test results must be qualified.

**Procedure**

**Soils:** 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 of water to field-moist soil. Soil samples are physically agitated using a mechanical shaker for a minimum of 30 minutes. Suspended solids may be removed by filtration, or sample extracts may be centrifuged until clear. Moisture content analysis is performed on a separate aliquot. Test results are corrected for moisture content and are reported on a dry-weight basis. Include sample moisture content in the aqueous extract volume for data calculation purposes.

**Waters:** Water samples are analyzed by direct aqueous injection by gas chromatography with flame ionization detection (GC/FID). Suspended solids may be removed by filtration, or samples may be centrifuged until clear.

Detailed instrumental analysis procedures are not provided for this method. The procedures described in EPA Method 8015D (see references) 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 80-120% for Lab Control Samples or Reference Materials at concentrations above ten times the MDL.

**Precision Requirement:** Laboratories must demonstrate method precision equal to or better than 15% relative standard deviation for Lab Control Samples 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 <sup>nd</sup> Source	One per initial calibration	85 – 115%

Continuing Calibration Verification (CCV) – mid-level	At least every 12 hours (max 20 samples), and at end of each batch.	80 – 120%
Laboratory Control Sample (LCS)	One per batch (max 20 samples)	70 – 130%
Laboratory Duplicate (DUP)	One per batch (max 20 samples)	50% RPD (soils) 30% RPD (waters) [or within 2x reported DL for low level results]
Matrix Spike (MS) or Reference Material (RM)	One per batch (max 20 samples)	60 – 140%
Surrogate Compounds	Recommended	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.		

### Prescribed Elements

The following components of this method are mandatory:

1. Calibrations must consist of a minimum of 4 calibration standards.
2. For soil extractions, soil moisture content must be accounted for in the extraction solvent volume if using extraction by water or a water-miscible solvent.
3. For FID analysis, at least 80% baseline chromatographic separation must be achieved for all target parameters in calibration standards (valley height of peak overlap must not exceed 20% of maximum peak height).
4. Where sample extracts require filtration or pre-extraction cleanup with hexane, QC samples must be processed in the same manner.
5. All stated performance and quality control requirements must be met.
6. Sample container materials, preservation, storage, and hold time requirements may not be modified. Samples analyzed beyond the stated holding time must be qualified. Refer to latest version of “BC MOE Sample Preservation and Hold Time Requirements” for updates.

Apart from these limitations, and provided performance requirements are met, laboratories may introduce modifications to this method in order to improve quality or efficiency.

### References

EPA 8015D. Nonhalogenated Organics Using GC/FID, US EPA, Revision 4, June 2003  
EPA 5021A. Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis, Revision 2, July 2014.

### Revision History

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