

Glycols in Soil and Water - PBM

Parameter	Glycols 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 glycols 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 using a polar stationary phase. Quantitation is performed through linear, external-standard calibration. Target compounds are identified by retention time. This method yields reporting limits that are suitable to meet BC CSR standards. If lower reporting limits are required, samples may be analyzed by HPLC/MS (US EPA Method 8321B) with a large volume injection.</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>Ethylene glycol</td> <td style="text-align: center;">5</td> <td style="text-align: center;">2000</td> <td style="text-align: center;">E020</td> </tr> <tr> <td>Propylene glycol, 1,2-</td> <td style="text-align: center;">5</td> <td style="text-align: center;">2000</td> <td style="text-align: center;">P020</td> </tr> <tr> <td>Diethylene glycol</td> <td style="text-align: center;">5</td> <td style="text-align: center;">2000</td> <td style="text-align: center;">E220</td> </tr> <tr> <td>Triethylene glycol</td> <td style="text-align: center;">5</td> <td style="text-align: center;">2000</td> <td style="text-align: center;">E320</td> </tr> </tbody> </table> <p><i>Note: This method may also be applicable to the analysis of other BC CSR substances such as the glycol monoalkyl ethers.</i></p>	<u>Analyte</u>	<u>Approx. Soil MDL, µg/g</u>	<u>Approx. Water MDL, µg/L</u>	<u>EMS Analyte Code</u>	Ethylene glycol	5	2000	E020	Propylene glycol, 1,2-	5	2000	P020	Diethylene glycol	5	2000	E220	Triethylene glycol	5	2000	E320
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EMS Method Code	<p>GLYC (Glycols in Soil and Water by GC/FID)</p> <p>***Refer to EMS Parameter Dictionary on the ministry website for all current EMS codes.</p>																				
Matrix	<p>Soil: Soil, Sediment, Sludges, Solid wastes.</p> <p>Water: Freshwater, Seawater, Groundwater, Wastewater, and Drinking Water.</p>																				
Interferences and Precautions	<ol style="list-style-type: none"> 1. Propylene glycol and ethylene glycol can be difficult to separate when one analyte is found in significantly higher concentrations than the other. 2. Glycols by the direct injection technique are prone to interferences (e.g. by alkanolamines). 3. Other oxygenated compounds and hydrocarbons may interfere. Suggestion to use matrix spikes to correctly identify the compounds. Hydrocarbon interferences could be removed by adding hexane and short physical agitation. 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>Soil: 125 mL glass jar with Teflon (or foil) lined lid.</p> <p>Water: 40-60 mL glass vial with Teflon lined lid.</p> <p>Preservation:</p> <p>Soil: Samples are not preserved.</p>																				

Water: Samples are not preserved.

Stability

Holding Time:

Soil: Analyze soil samples within 14 days after collection.

Water: Analyze water samples within 14 days after collection.

Where holding times are exceeded, data must be qualified.

Storage: Store samples at $\leq 10^{\circ}\text{C}$ during shipment to the laboratory and at $\leq 6^{\circ}\text{C}$ 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 of water to soil. 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. A separate aliquot of the sample is retained for moisture determination. 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 large amounts 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 70-130% 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 20% 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%
Lab Control Sample (LCS)	One per batch (max 20 samples)	70-130% for solids 80-120% for water
Lab Duplicates (DUP)	One per batch (max 20 samples)	50% RPD for solids 30% RPD for water [or within 2x reported DL for low level results]
Matrix Spike (MS) or Reference Material (RM)	One per batch (max 20 samples)	60-140% for solids 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. For waters, the LCS is equivalent to the ICV or the CCV as there is no extraction

Matrix Spike: Required.

Surrogate Compounds: Optional, e.g. tetramethylene glycol

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 requirements 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

Jan 13, 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.