

Liquid-Solid Partitioning (Leachability) of VOCs – Prescriptive

Parameter Leaching potential of volatile organic compounds (VOCs) in soil samples.

Analytical Method Extraction of as-received solid material with a dilute buffered aqueous solution of glacial acetic acid and sodium hydroxide (pH 4.93 ± 0.05) using the Zero Headspace Extraction technique from the EPA Method 1311 Toxicity Characteristic Leaching Procedure.

Introduction This method is intended to be used as part of an environmental leaching assessment for the evaluation of disposal, beneficial use, treatment effectiveness, and site remediation options.

This method is a leaching characterization method that is used to provide values for intrinsic material parameters that control leaching of selected volatile organic species under equilibrium conditions. This test method is intended as a means for obtaining an extract of a solid material (i.e., the eluate), which may be used to estimate the liquid-solid partitioning (e.g., solubility and release) of constituents under the laboratory conditions described.

The Zero Headspace Extraction (ZHE) portion of EPA Method 1311 must be used as-written, except for modifications as noted herein.

This method is not applicable for characterizing the leachability of metals, inorganics, or semi-volatile organic substances.

This method is not for use under the Hazardous Waste Regulation for the classification of hazardous wastes (see EPA 1311) or for determining suitability for disposal in a secure landfill (see MLEP).

Method Summary This method follows the EPA Method 1311 TCLP procedure for Zero Headspace Extraction (ZHE) of volatile organic compounds from soil samples. The EPA 1311 Method is followed exactly, except that no particle size reduction is done, and alternative hold times, sample storage requirements, and QC requirements are used as described herein.

The EPA 1311 ZHE procedure extracts 25 gram samples of soils in a zero headspace extraction device for 18 ± 2 hours, tumbled in a rotary extraction box at 30 ± 2 rpm at 23 ± 2 °C, using a 20:1 fluid to soil (dry weight) ratio with extraction fluid #1 from the TCLP method. Extraction fluid #1 is a dilute solution of glacial acetic acid and sodium hydroxide with pH of 4.93 ± 0.05. Following the leaching procedure, extracts are pressure filtered prior to preservation and analysis for VOCs. Instrumental analysis for parameters of interest may be conducted using any appropriate BC MOE approved instrumental analysis technique (normally Headspace or Purge and Trap with GCMS detection, which provides low detection limits with few interferences).

The leachate concentrations of constituents of potential concern (COPCs) are determined and reported.

MDL(s) and EMS Analyte Code(s)

Analytes	CAS Number	Approx MDL (ug/L)	EMS Analyte Code
Benzene	71-43-2	1.0	n/a
Ethylbenzene	100-41-4	1.0	n/a
Methanol	67-56-1	10 - 50	n/a
Naphthalene	91-20-3	1.0	n/a
Tetrachloroethylene	127-18-4	1.0	n/a
Toluene	108-88-3	1.0	n/a
Trichloroethylene	79-01-06	1.0	n/a
Xylenes, Total	1330-20-7	2.0	n/a

Leachability of other prescribed substances from CSR Schedule 3.1 Parts 2 and 3 may also be assessed by this procedure if permitted under Protocols 2 or 13 or if acceptable to the Director.

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

Matrix	Soil
Interferences and Precautions	Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents may be necessary. Refer to each analytical method to be used for specific guidance on instrumental QC procedures.
Sample Handling and Preservation	<p>Containers: Glass soil jars, packed tightly to minimize headspace. 125 – 250 mL recommended.</p> <p>Sample Collection: Samples should be collected in such a way as to exclude particle sizes >9.5 mm, where possible. Sampling should be representative and reproducible. Pack soil jars as fully as possible to minimize headspace / pore space. Pack samples on ice as quickly as possible (to at least $\leq 10^{\circ}\text{C}$, ideally to $\leq 6^{\circ}\text{C}$ if possible). Transport as soon as possible to the laboratory for commencement of testing, or freeze to $\leq -10^{\circ}\text{C}$. If samples will be frozen, leave an air gap prior to freezing of ~5-10% of the container volume to allow for expansion, to minimize the chance of breakage.</p> <p>Preservation:</p> <p>Soil Samples: No chemical preservation is used. Zero headspace (or minimized headspace) and cold storage minimizes loss of volatiles prior to analysis.</p> <p>Leachates: Preserve leachates with solid sodium bisulfate (NaHSO_4) to $\text{pH} < 2$. Preserve only after filtration has been completed.</p>
Stability	<p>Holding Times: Soil samples must be leached within 48 hours from time of sampling, or within 14 days if frozen within 48 hours of sampling (to $\leq -10^{\circ}\text{C}$). Field methanol preservation is not possible for this test, so hold times must be minimized. The EPA 1311 hold times for VOCs are not used for this method, to maximize consistency with standard BC MOE protocols for VOCs in soils. Leachates preserved with NaHSO_4 must be analyzed within 14 days of preservation.</p> <p>Storage:</p> <p>Soil Samples: Store soils refrigerated at $\leq 6^{\circ}\text{C}$ (or frozen at $\leq -10^{\circ}\text{C}$) at the laboratory, and at $\leq 10^{\circ}\text{C}$ during transport to the laboratory. If samples have been frozen for preservation purposes, thaw in a refrigerator at $\leq 6^{\circ}\text{C}$ as close as possible to the time the test will be initiated (no more than 24 hours). If samples will be frozen, leave an air gap prior to freezing of ~5-10% of the container volume to allow for expansion, to minimize the chance of breakage. After initial sub-sampling it is recommended to freeze remaining soil immediately (if within hold time) to permit re-analysis.</p> <p>Leachates: Store leachates refrigerated at $\leq 6^{\circ}\text{C}$.</p>
Procedure	<p>Follow the Zero Headspace Extraction procedure from the US EPA 1311 TCLP Method without modification, except for differences noted and prescribed in this document.</p> <p>Particle size reduction as described in EPA 1311 is not required or recommended. Particle size reduction within the 1311 method may cause significant losses of COPCs and is intended for heterogeneous waste samples, whereas this method is intended only for use with soils.</p>

Quality Control

QC Component	Minimum Frequency	Minimum Data Quality Objectives
Method Blank (MB)	One per batch (max 20 samples)	Less than reported DL
Lab Control Sample (LCS); spiked post-extraction	One per batch (max 20 samples)	60-140%
Lab Duplicates (DUP); includes leaching procedure	One per batch (max 20 samples)	<50% RPD [or within 2x reported DL for low level results]
Matrix Spike (MS); spiked post-extraction	Optional	None

If DQOs are not met, repeat testing or report qualified test results.

References US EPA SW 846 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, US EPA Office of Solid Waste.

Revision History Sept 15, 2017 First version. Method was designed by the CSAP Leachate Testing Procedure Working Group, with translation into a working method by BCELTA.