Extraction of Semi-Volatile Organic Compounds from Soil in Accordance with EPA 3545A

Summary

Soil is one of the most common matrices in which semi-volatile organic compounds (SVOCs) are present. The extraction of these compounds from soil can be a lengthy and tedious process. The EDGE® is a revolutionary simple system for the rapid extraction of SVOCs from soil that is more than six times faster than other automated techniques. The Q-Cup Technology™ combines the process of Pressurized Fluid Extraction (PFE) and Dispersive Solid Phase Extraction (dSPE) in a single instrument that yields a fast, simple, and efficient extract. While maintaining all properties of PFE and adhering to EPA 3545A, the EDGE also offers the benefits of the easy-to-assemble Q-Cup® sample holder. The Q-Cup, with its unique open cell concept, creates a dispersive effect and promotes rapid extraction and filtration.

Introduction

SVOCs are a subgroup of Volatile Organic Compounds (VOCs) that have a high molecular weight and high boiling points. They are primarily composed of pesticides and herbicides. Prolonged exposure to these compounds, especially indoors, raises a public health concern, including several listed by the US EPA as hazardous air pollutants (HAPs). This classification applies to pollutants that can cause serious health effects, such as allergies, asthma, endocrine and thyroid disruption, reproductive toxicity, fetal and child development delays, and even cancer.

Semi-volatile organic compounds consist of substances with a broad set of chemical properties and structural features. These differences make it challenging to efficiently extract all analytes of interest with one method. Furthermore, the complexity of the soil matrix from which the SVOCs are to be extracted often includes multiple components, adding to the complexity of extraction. The EDGE with Q-Cup Technology can effectively extract a difficult set of analytes from complex matrices with one simple method.

Traditional methods, such as Soxhlet, are very time consuming and use a large amount of solvent. Automated methods often require tedious sample preparation with complex sample holders. EDGE is the fastest exaction system available and uses a minimal amount of solvent. The Q-Cup sample holder is comprised of two easy-to-assemble pieces, allowing the sample to be prepared in seconds.

Due to their persistent nature, SVOCs continue to accumulate and concentrate in our environment. To ensure our safety, these compounds need to be extracted, identified, and quantified. The accuracy of SVOC analysis is dependent on an efficient extraction. EDGE yields an efficient extract that is filtered, cooled, and ready for analysis in under 5 minutes. EPA 3545A is a method for the extraction of water-insoluble or slightly water-soluble volatile and semi-volatile compounds in soils, clays, sediments, sludges, and waste solids. EDGE meets the requirements of EPA 3545A and is preprogrammed for that method.

Materials and Method

Reagents

Sand, loam, and clay, purchased from Sigma Aldrich were spiked with SPEX CertiPrep TCLP Base/Neutral/Acid Extractable Spike Solution in Methylene Chloride, Part#: TCLP-BNA. The spike mix was also used to construct calibration curves. CRM 110-100 was purchased from Sigma Aldrich. A 50/50 mixture of acetone/hexane was used as the extraction and rinse solvent. The system was washed with hexane and acetone.

Sample Preparation

15 g of sand, loam, or clay, spiked with 250 µl of spike solution was weighed into an assembled Q-Cup containing a C1 Q-Disc® . 15 g of CRM 110-100 from Sigma Aldrich was also weighed into an assembled Q-Cup containing a C1 Q-Disc. The Q-Cups were placed in the EDGE removable rack, each with a collection vial and the rack was positioned on the EDGE. The CEM approved method for EPA 3545A was started.

EDGE 3545A Method

Q-Disc:	C1
Solvent:	50/50 acetone/hexane
Top Add:	15 mL
Bottom Add:	10 mL
Rinse:	10 mL
Temperature:	120 °C
Hold Time:	1 min
Wash 1:	10 mL acetone
Wash 2:	10 mL hexane

Analysis

The extract samples were concentrated to a volume of 10 mL. An aliquot of each extract was injected into the Agilent 7890A with a 5975C MSD for analysis, adhering to EPA 8270. A Phenomenex ZB-5MSplus 30 m, 0.25 mm column was used.

Results and Discussion

The EDGE extracted sand, loam, and clay samples in under 5 minutes, including filtration, cooling, and system washing. No post clean up or solvent exchange was necessary. The extracts were concentrated and injected into the GCMS for analysis. **Figure 1** is a representative GCMS chromatogram of a clay extraction showing clean separation of the SVOCs. The SVOCs analyzed are known to be difficult to extract and the EDGE was able to achieve good extraction of all compounds in different soil types with a single method. The recovery data was determined via a 6-point calibration curve for each SVOC. The absolute recoveries for the SVOCs spiked in all three types of soil, sand, loam, and clay, were acceptable. **Table 1** shows the percent recoveries from spiked soil of some difficult to extract semi-volatile organic compounds. The extraction of CRM 110-100 via EDGE was comparable to Soxhlet as seen in **Table 2**.

Figure 1: GCMS Chromatogram of the SVOCs Extracted from Clay

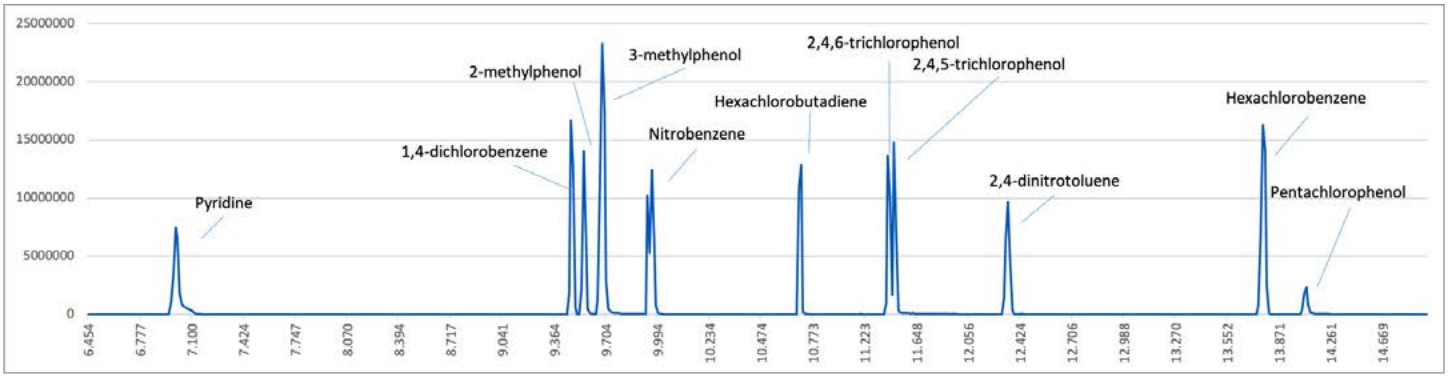

Table 1: Percent Recovery Data as Compared for 15 g Spiked Sand, Loam, and Clay

Compound	Sand	Clay	Soil
Pyridine	100	88	93
1,4 dichlorobenzene	88	88	96
2-methylphenol	84	95	115
3-methylphenol	90	102	104
Hexachlorobutadiene	86	92	97
2,4,6-trichlorophenol	90	105	103
2,4,5-trichlorophenol	89	113	99
2,4-dinitrotoluene	90	102	99
Hexachlorobenzene	86	86	81

Table 2: Percent Recovery Data as Compared to Soxhlet for 15 g CRM 110-100

Compound	% Soxhlet
2-nitroanaline	94
2,4-dinitrotoluene	114
Dibenzofuran	92
Fluorene	105
Bis-2-ethylhexyl-phthalate	97

Conclusion

To be in the acceptable recovery range, the percent recovery data for the extraction of SVOCs from soil should be within 80-120%. The EDGE with Q-Cup Technology yielded acceptable recoveries for the extraction of SVOCs from three different types of soil and a CRM. Furthermore, the EDGE method used less solvent and time than traditional extraction techniques such as Soxhlet. Lastly, since the EDGE is an automated system, it eliminates the human error, often existing with other extraction techniques. EDGE was able to efficiently and accurately extract SVOCs from soil.

United States (Headquarters)
800-726-3331
704-821-7015
Fax: 704-821-7894
info@cem.com

France
33 (01) 69 35 57 80
Fax: 33 (01) 60 19 64 91
info.fr@cem.com

Germany, Austria, Switzerland
(49) 2842-9644-0
Fax: (49) 2842-9644-11
info@cem.de

Ireland
+353 (0) 1 885 1752
Fax: +353 (0) 1 885 1601
info.ireland@cem.com

Italy
(39) 35-896224
Fax: (39) 35-891661
info.srl@cem.com

Japan
+81-3-5793-8542
Fax: +81-3-5793-8543
info@cemjapan.co.jp

United Kingdom
(44) 1280-822873
Fax: (44) 1280-822873
info.uk@cem.com

www.cem.com