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

## Abstract

Semi-Volatile Organic Compounds (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 herbacides. Prolonged exposure to these compounds, especially indoors, raises a public health concern. For example, many of these compounds have been 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.

Soil is one of the most common matrices in which these compounds are present and the extraction of these compounds from soil can be a lengthy and tedious process. The CEM EDGE™ is a revolutionarily simple system for the rapid extraction of semi-volatile organic compounds (SVOCs) from soil that is more than six times faster than other automated techniques. With its patent pending Q-Cup Technology™ the EDGE™ makes the extraction process fast and simple and meets the requirements of EPA 3545.

## Introduction

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 soil matrix from which the SVOCs are to be extracted is often complex with multiple components adding to the complexity of extraction. The CEM 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 3545 is a method for the extraction of water-insoluble or slightly water-

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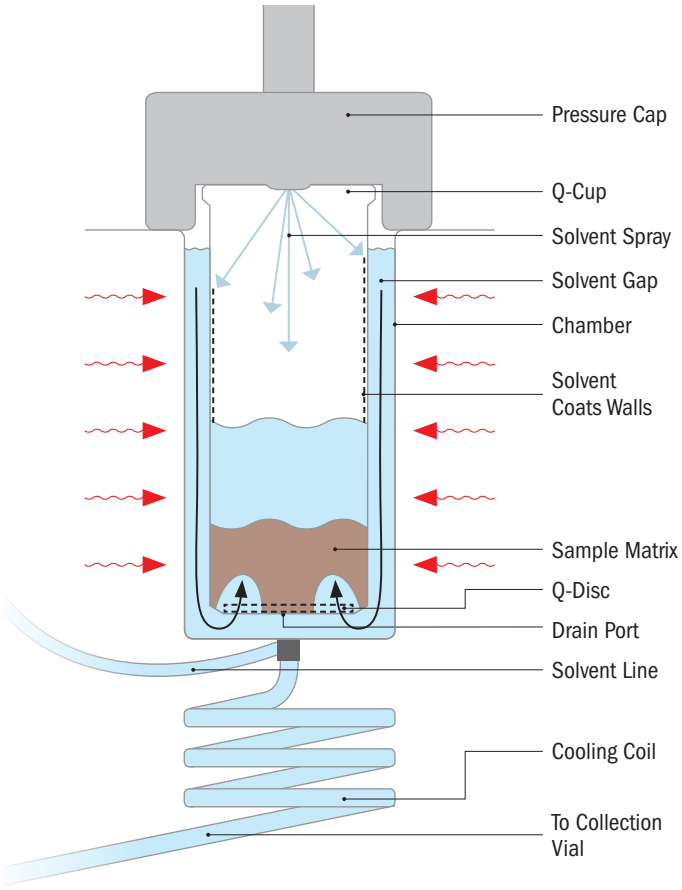
soluble volatile and semi-volatile compounds in soils, clays, sediments, sludges, and waste solids. EDGE is meets the requirements of EPA 3545 and is preprogrammed with the methodology.

## Instrumentation

The EDGE uses Q-Cup Technology that combines the process of Pressurized Fluid Extraction and Dispersive Solid Phase Extraction in one instrument that yields rapid and efficient extraction. The easy-to-assemble Q-Cup sample holder offers a unique open cell concept that creates a dispersive effect and promotes rapid extraction and filtration. The result is fast, simple and efficient extractions.

Sample preparation in the EDGE could not be easier. Simply place a Q-Disc™ into the Q-Cup base and screw the two parts together. Soil samples from 1 g to 30 g can be extracted and $NaSO_4$ can be added to dry the sample if needed. The EDGE will use only 40 mL of solvent per extraction which includes solvent for diffusive extraction, sample rinse, and cleaning of the system. The rapid heating of the extraction chamber in combination with diffusive action allows a temperature of up to 180°C to be achieved in less than 2 minutes.

**Figure 1**: The EDGE Process




### Sample is Loaded

The Q-Cup is automatically loaded into the chamber by the auto sampler. The pressure cap then creates a pressurized seal on the top of the Q-Cup.

### Solvent is Added

Solvent is first added through the bottom to fill the gap between the chamber and Q-Cup, this aids in heat transfer. Then, solvent is added through the top of the Q-Cup to wet the sample.

### Chamber is Heated

As the chamber walls are heated, the pressure in the gap increases. This overcomes the pressure inside the Q-Cup, forcing the solvent to disperse into the sample.

### Extract is Collected

Once the sample reaches temperature, the solvent is dispensed through the Q-Disc, the cooling coil, and into a collection vial.

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## Procedure and Method

10 g of sand, loam, or clay spiked with 250 µl of spike solution was weighed into an assembled Q-Cup containing a Q-Disc. 10 g of CRM 110-100 from Sigma Aldrich was also prepared. The Q-Cups were placed in the EDGE removable rack each with a collection vial and the rack was slid into place on the EDGE. The One Touch Method™ for EPA 3545 was used. The extracts were 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.

## Samples

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. CRM 110-110 was purchased from Sigma Aldrich. CRM and Spiked samples were extracted via the EDGE and soxhlet. A 50/50 mixture of acetone/hexane was used as the extraction and rinse solvent. The system was washed with hexane and acetone.

## 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, solvent exchange or concentration was necessary. The extracts were directly injected into the GCMS for analysis. The recoveries for all three types of spiked soil were comparable to soxhlet. **Table 1** is a table of the % recoveries as compared to soxhlet from spiked soil of some difficult to extract semi-volatile organic compounds. The extraction of CRM 110-100 via EDGE was also comparable to soxhlet as seen in **Table 2**.

**Table 1:** % recovery data as compared to soxhlet for spiked sand, loam, and clay

| Analyte | Sand (% Soxhlet) | Clay (% Soxhlet) | Soil (% Soxhlet) |
|---|---|---|---|
| 1,4 dichlorobenzene | 94 | 98 | 93 |
| hexachloroethane | 91 | 94 | 88 |
| 4-methylphenol | 82 | 91 | 85 |
| nitrobenzene | 86 | 101 | 95 |
| hexachlorobutadiene | 94 | 97 | 89 |
| 2,4,5-trichlorophenol | 87 | 76 | 81 |
| 2,4,6-trichlorophenol | 92 | 73 | 86 |
| 2,4-dinitrotoluene | 85 | 85 | 86 |
| hexachlorobenzene | 86 | 84 | 82 |

**Table 2:** % recovery data as compared to soxhlet for CRM 110-100

| Compound | % Soxhlet |
|---|---|
| 2-nitroanaline | 94 |
| 2,4-dinitrotoluene | 114 |
| dibenzofuran | 92 |
| fluorene | 105 |

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