Technical Note

EPA Methods for Organic Extraction of SVOCs from Solid Matrices

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Solid matrix extraction

When setting out to analyze a sample from a complex sample matrix such as soil it is important to understand what analytes you are targeting and how to prepare your sample so it is suitable for your analysis. A common scope for analysis is to determine the quantity of semi volatile organics (SVOCs) in soil samples by gas chromatography (GC). There are different techniques for extraction from solid matrices, including methods from the EPA.

Soxhlet extraction

A traditional technique for extracting target analytes from a sample matrix is a Soxhlet extraction. Guidance for performing a Soxhlet extraction is outlined in EPA Method 3540. This method describes a procedure to extract nonvolatile and semi volatile organic compounds from solid matrices. Anhydrous sodium sulfate is mixed with the solid sample and then to be placed in an extraction thimble. The extraction is conducted in a Soxhlet extractor using an appropriate solvent.

After extraction the extract is dried and can be concentrated if necessary. The extract can then be put into a solvent which is suitable for cleanup and/or analysis Soxhlet extraction has the option to be automated as described in EPA Method 3541.

Alternative EPA extraction methods

The disadvantages of traditional techniques such as Soxhlet extraction is they are time consuming, require large volumes of solvent and specific apparatus. Other methods have been released by the EPA to offer an alternative to these traditional techniques which utilize other extraction techniques to reduce solvent consumption and extraction time.

EPA Methods 3545, 3546 and 3550 are alternative EPA methods which can be used for extraction of SVOCs from solid matrices with equivalent recovery to the Soxhlet extraction.

Method 3545 uses a commercially available and automated extraction system to extract target analytes from solid matrices using pressurized fluid extraction (PFE). This method utilizes temperatures of 100-180 °C and pressures of 1500-2000 psi. This method states that samples should be prepared for extraction by air drying and grinding, or by mixing the samples with anhydrous sodium sulfate or pelletized diatomaceous earth then loaded into an extraction cell. The extraction cell is then heated and pressurized with the appropriate solvent system. The solvent from the heated extraction vessel is collected and cooled. The extract can be concentrated if necessary before being put into a solvent which is suitable for clean up or analysis.

Method 3546 uses a commercially available extraction system to expose a sample to elevated temperature and pressure using microwave energy to perform a microwave extraction. Temperatures reach 100-115 °C and pressures of 50-175 psi in a closed vessel which contains your sample and an appropriate solvent. Samples are prepared by grinding into a powder, loading into an extraction vessel and then adding the appropriate solvent and sealing. The extraction vessel is heated and extraction begins. After extraction is complete and the contents cooled, it is filtered. The solid material is rinsed and solvent fractions combined. The extract can be concentrated if necessary before being put into a solvent which is suitable for clean up or analysis.

Method 3550 uses a procedure where an ultrasonic process is used to ensure maximum contact between the sample matrix and the extraction solvent in an ultrasonic extraction. The method details two procedures which differ due to analyte concentration. For the low concentration procedure, the sample is mixed with anhydrous sodium sulfate and is extracted three times with solvent using ultrasonic extraction. The extract is separated using vacuum filtration or centrifugation and then be concentrated, cleaned up and/or analyzed. For medium/high concentration samples, the sample is mixed with anhydrous sodium sulfate and is extracted from once with solvent using ultrasonic extraction. A portion of the extract is then collected for clean-up and/ or analysis.

QuEChERS

This solid phase extraction (SPE) sample preparation technique has grown in popularity as an alternative to traditional extraction techniques due to being time efficient, using minimal solvent and not needing specific laboratory glassware or extraction system.

There are two main steps to using QuEChERS: extraction and cleanup. Before extraction begins on solid samples with little or no water content, water must be added as appropriate for sample size. Sample extraction involves using a solvent and a salt mixture to separate the analytes into the organic phase whilst minimising matrix effects. Dispersive solid phase extraction (dSPE) cleanup removes any matrix interferences such as sugars and pigments from sample extract achieving a sample ready for analysis.

There are three widely used QuEChERS methods which are selected based on your sample, the original unbuffered method and then the two buffered methods, the AOAC method and European EN method. See our QuEChERS guide technical note for more information in SCION's knowledge centre.