B147 An Evaluation of Novel Headspace Extraction Methods for the Analysis of Ignitable Liquid Residues (ILRs)

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Learning Overview: After attending this presentation, attendees will better understand the performance capabilities of a novel headspace extraction devices for ignitable liquid residues.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by highlighting an emerging headspace sampling technique that is faster, more sensitive, and customizable for the analysis of fire debris.

Forensic analysts have an array of techniques to choose from when assessing fire debris for the presence of ignitable liquid residues (ILRs). In some instances, a technique like solvent extraction is used but is generally not favored due to solvent waste and destruction of the evidence. The techniques that see the most use in modern laboratories are passive headspace-based; adsorption on to an activated charcoal strip (ACS) or a solid-phase microextraction (SPME) fiber are the most popular choices.[1] These are non-destructive and do not require prior sample preparation. However, both techniques have notable disadvantages. Sampling with a charcoal strip necessitates the use of a toxic solvent such as carbon disulfide to desorb compounds and can be subject to the displacement of smaller molecules. The equilibrium-based SPME technique also suffers from this because it has a very limited extraction surface area. The overall sampling time for both techniques is also relatively long. Charcoal strips are normally exposed to the headspace between 2 -16 hours during extraction and SPME fibers require at least an hour of conditioning after every use to prevent carryover.[2]

In this study, the effectiveness of ACS and SPME for the analysis of ILRs is evaluated in comparison to capillary microextraction of volatiles (CMV). The CMV is a dynamic headspace sampling device which consists of a dual open-ended capillary tube filled with glass microfiber strips that are coated in a PDMS-incorporated sol-gel polymer. The sol-gel formulation has been modified with specific precursors to provide high adsorption affinity for a range of compounds and an increase in surface area over SPME of 5,000 times. Sampling is achieved in as little as 10 minutes, and the construction of the device allows the CMV to be thermally desorbed by direct introduction into a GC/MS inlet. Previous applications of the CMV include the headspace sampling of smokeless powders, marijuana plant material, and BTEX vapors.[3-5]

The three techniques are evaluated based on several points of comparison, including generated chromatographic patterns, the total length of time needed for equilibration and extraction, and percent mass recovered. Quantitative analysis is based on a representative '10-mix' of standard ILR compounds ranging from toluene to eicosane. The sampling system for the CMV has been previously reported and has been optimized for the extraction of ILRs.[5] The sampling conditions for ACS and SPME are in accordance with recommendations from their respective ASTM methods. Simulated charred debris samples are examined, including wood, cotton, and drywall substrates that have been doused with either gasoline or diesel in various weathered states. CMVs constructed with multiple functionalized sol-gel strips are also evaluated as an alternative to multi-phase SPME fibers.

The data presented here will demonstrate the potential of the CMV as an alternative sampling method for ILR extraction. These initial demonstrations of high sensitivity, fast analysis time, and general ease of use will hopefully encourage practicing fire debris analysts to explore the viability of the CMV in a routine laboratory setting.

Reference(s):

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Fire Debris Analysis, Headspace Sampling, Ignitable Liquid Residues