



A194 Further Studies Investigating Zeolites for the Recovery of Oxygenated Compounds From Fire Debris Samples

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After attending this presentation, attendees will have gained knowledge of further studies into the use of zeolites for improved recovery of oxygenated compounds from fire debris samples which may supplement the activated carbon strip technique for recovering petroleum-based products.

This presentation will impact the forensic science community by demonstrating the feasibility for implementing zeolites into forensic casework as a viable technique in fire debris analysis for the recovery of oxygenated ignitable liquids.

Heated passive headspace concentration is presently the most commonly utilized technique for the extraction of ignitable liquid residues from fire debris evidence. This process, introduced by William Dietz in 1991, typically involves suspending an activated charcoal strip within an airtight container such as a metal can and incubating the sample for a period of time. ASTM Standard Practice E 1412-07 advises heating the sample for 2 to 24 hours at a temperature of 50 to 80° Celsius. Subsequently, the compounds are easily eluted from the adsorbent with a suitable solvent, often carbon disulfide, and analyzed using gas chromatography/mass spectrometry (GC/MS) for the potential identification of any ignitable liquid residues. It is a simple, sensitive, and nondestructive method, and can often be performed within the original sample packaging. The activated charcoal strip, which does not interact with water or nitrogen, is advantageous in its affinity for hydrocarbons and resistance to oxidation. The technique is highly efficient for recovering petroleum-based ignitable liquids, however, it has had limited success with adsorbing and concentrating oxygenated species.

In an effort to improve the recovery of ignitable liquids containing oxygenated compounds, previous studies have suggested zeolites are a suitable adsorbent for the recovery of acetone through heated passive headspace concentration. Zeolites are inorganic, microcrystalline materials that have a well-defined internal structure and uniform pore size. Most frequently aluminosilicate with cations dispersed internally, zeolite beads attract small organic molecules including alcohols. Their high thermal and chemical stability make them ideal adsorbents for heated passive headspace applications. An additional advantage to utilizing zeolite beads involves selective adsorption of small organic molecules due to their pore size. Molecular modeling has shown that the 13X zeolites were effective for recovering analytes smaller than 10 Å, such as acetone (6.3 Å). Any compound with a molecular diameter greater than the pore size may not gain access to the internal zeolite channels, and thus may not be adsorbed.

The primary aim of this study was to further optimize the conditions for implementing zeolites as a viable extraction technique within fire debris casework, complementing the activated charcoal strip method. Extraction time and temperature, desorption solvent, and GC parameters were all examined to provide for the most efficient recovery of oxygenated volatile compounds, including (but not limited to) ethanol, 1-propanol, 1-butanol and isopropanol. For example, approximately a 900% increase in recovery was observed for 1-butanol by the use of zeolites in comparison to an activated charcoal strip. This is in accordance with previous studies that reported a 300% improvement in acetone recovery by utilizing zeolites. In an effort to evaluate the ability of zeolites to selectively adsorb oxygenated volatile compounds, comparative recoveries of mixtures of petroleum and alcohol-based ignitable liquids were studied utilizing both activated charcoal strips and zeolites. In the presence of both adsorption media within the same can, 100% of three major components of gasoline (toluene, 1,2,4-trimethylbenzene, and naphthalene) selectively adsorbed to an activated charcoal strip, while approximately 90% of isopropanol adsorbed to zeolites. This phenomenon may be attributed to the size exclusion properties and polarity of the zeolites. An ideal technique for the analysis of ignitable liquids would allow for the efficient recovery of both petroleum-based and oxygenated products in a single concentration procedure.

Zeolites, Volatiles, Fire Debris Analysis