B154 Development of a Sample Clean-Up Procedure for the Recovery of Trace Quantities of Organic Explosives in Soil and Sand

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After attending this presentation, attendees will be informed about techniques for the extraction and detection of trace amounts of organic explosives residue from soil, sand, and oil-contaminated soil matrices utilizing Solid Phase Extraction (SPE).

This presentation will impact the forensic science community by providing an improved clean-up method for detecting trace organic explosives residue in complex matrices. In addition, this presentation will compare and contrast three SPE cartridges (Bond Elut NEXUS®, Empore™ SDB-XC, and Oasis® HLB) based on the recovery of organic explosives residue from soil/sand and matrix rejection.

The forensic science community is constantly searching to improve currently established protocols in lieu of older alternatives. A fast and simple option often used for samples is to use syringe filtration of the extract prior to sample analysis. Although samples are filtered, the sample typically contains matrix interferences that are not removed and are injected with the sample into the instrument. In this research, a Gas Chromatograph coupled to an Electron Capture Detector (GC/ECD) was utilized for analysis. The matrix of filtered samples may lead to signal suppression, increased background noise, and extra (or missing) peaks. If this occurs, instrument maintenance must be performed, which can be costly both in time and materials. The research to be presented focuses on finding an efficient extraction process that can separate the analyte(s) from the contaminants, facilitating sample throughput and reducing maintenance costs associated with instrument malfunction due to damage from matrix interferences.

Twelve organic explosives were investigated in this study: ethyleneglycol dinitrate (EGDN); dimethyl dinitrobutane (DMDNB); 4-nitrotoluene (4-NT); nitroglycerin (NG); 2,4-dinitrotoluene (DNT); 2,4,6-trinitrotoluene (TNT); pentaerythritol tetranitrate (PETN); trimethylene trinitramine (RDX); 2,4,6-trinitrophenylmethylnitramine (Tetryl); tetramethylene tetranitramine (HMX); erythritol tetranitrate (ETN); and cyclotrimethylene trinitrosoamine (R-salt). A mixture of these explosives was spiked onto samples of soil, sand, and soil contaminated with used motor oil. Acetonitrile was used to extract the explosives from the matrices. As a comparison of a simplified extraction procedure, syringe filtration was used prior to GC/ECD analysis for one sample set. This involved filtering the extracted liquid, a dry-down step to reduce sample volume, and injection via syringe for analysis by GC/ECD. In this research, the extraction liquid was cleaned up using Solid Phase Extraction (SPE) prior to GC/ECD analysis. This method has the advantage of bypassing the time-consuming dry-down step and of removing residual impurities contributed by the complex matrix.

In conclusion, the experiments reflected that it was possible to extract organic explosives residue from soil and sand samples using acetonitrile. Using SPE, it was possible to clean up the samples for GC/ECD analysis. Preliminary results indicate that the SPE cartridges provided the recovery of all 12 explosives, purified the oil-contaminated samples well, and generally did not require an extensive processing time. The results also indicate that the Oasis® HLB cartridges provided the highest percent recoveries of the explosives and are the most cost effective.

Explosives, Trace Analysis, Solid Phase Extraction

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