

B145 The Combined Extraction and Analysis of Trace Organic, Inorganic, and Peroxide Explosives Residues

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Learning Overview: The goal of this presentation is to introduce attendees to a validated method that includes: (1) the combined extraction of inorganic, organic, and peroxide explosives residues; (2) the processing of samples using Solid-Phase Extraction (SPE) cartridges; and (3) the screening of processed samples using Gas Chromatography/Electron Capture Detection (GC/ECD) and Ion Chromatography/Conductivity Detection (IC/CD).

Impact on the Forensic Science Community: This presentation will impact the forensic science community by introducing a validated method for the combined recovery of inorganic and organic explosives residues, thus reducing the sample preparation typically required for the analysis of post-blast explosives residues.

Explosives residues can contain both inorganic and organic compounds that often require multiple extractions for analysis by different methods. These residues are usually extracted separately using various solvents. Currently, collection swabs containing suspected explosives residues are cut in half and processed through two syringe extractions: water for inorganic species and acetone for organic compounds. The resulting extracts are then screened by IC/CD and GC/ECD. The splitting of the collection matrix increases processing time and decreases the amount of explosives residue that will be extracted for analysis by each method and thus, may lead to negative findings if the levels fall below the instrument's Limit Of Detection (LOD).

This presentation will cover the validation of a method for the combined extraction of both inorganic and organic compounds from a single Cotton Ball (CB) or Cotton-tipped Applicator (CA). The organic explosives analyzed included nitramines, nitroesters, nitroalkanes, nitroaromatics, and peroxides. The anions analyzed were chlorate, chloride, nitrate, nitrite, perchlorate, sulfate, and thiocyanate. The cations analyzed were ammonium, barium, calcium, lithium, magnesium, potassium, sodium, and strontium. The method includes extraction in a water:acetone solvent followed by SPE, resulting in a load fraction and acetone eluate that are analyzed by IC/CD and GC/ECD, respectively. The validation experiments completed were the determination of LOD, the evaluation of sample stability at two concentrations, and the assessment of possible interferences from collection matrices, improvised explosive device components, or environmental factors.

In terms of organic explosives LODs, all were detectable at 500ppb for both matrices, with some present at lower concentrations. The peroxides were detectable at 5ppm for both matrices, with one at 1ppm. The anions spiked on CBs were all detectable at 1ppm, with some detectable at lower concentrations. For CAs, although some anions yielded detectable responses at lower concentrations, all were detected at 1ppm with one exception. Chloride failed due to a high matrix blank response. Cations were all detected on CB at 1ppm (with some at lower concentrations), but sodium and potassium failed with CAs due to a high matrix blank response.

For stability experiments, in the case of CB organics, 6 out of 12 organic explosives were considered stable up to 72 hours for the low concentration and 9 out of 12 explosives for the high concentration. In the case of CA organics, 3 out of 12 organic explosives were considered stable up to 72 hours for the low concentration and 8 out of 12 explosives for the high concentration. Hexamethylene Triperoxide Diamine (HMTD) CBs remained stable up to 32 hours for the low concentration and 16 hours for the high concentration. HMTD CAs remained stable up to 8 hours. Triacetone Peroxide (TATP), cations, and anions were considered stable up to 72 hours for CBs at all concentrations.

A large number of relevant matrices (65) were analyzed for their potential interference in the detection of analytes. It was determined that 28 of the samples interfered with organic and peroxide analytes. For the anions and cations, it was determined that 24 and 35 samples contained interferences, respectively. The analytes with the highest number of interferences were cyclotetramethylene tetranitramine (HMX), chloride, perchlorate, and potassium. Recovery experiments were also completed for swabs that were spiked with used motor oil (simulating contamination) and the current syringe protocol was compared with this validated SPE method.

In conclusion, a method for the combined extraction of inorganic and organic explosives residues was validated. The LODs and stability of analytes were determined, and 65 interference matrices were analyzed. The described method permits the processing of a single swab for analysis by IC/CD and GC/ECD for a streamlined approach to trace detection of explosives residues.

Explosives, Solid-Phase Extraction, Trace Analysis