B146 The Optimization of Gas Chromatography-Vacuum Ultraviolet (GC-VUV) Spectroscopy for the Analysis of Explosives and Their Post-Blast Residues

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Learning Overview: After attending this presentation, attendees will better understand the analysis of explosives via GC-VUV and the optimization of the system to improve detection levels for post-blast debris analysis.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by discussing a recently developed detector for GC (a VUV spectrometer) and its optimization for the analysis of post-blast debris.

The instrumentation employed in this research consisted of an Agilent® 7890B GC (50°C hold 0.5min, ramp 20°C/min to 280°C) with a multimode inlet (50°C to 280°C at 900°C/min), hydrogen carrier gas (3.2mL/min flow rate), a Restek® Rtx®-5MS column (15m x 0.32mm ID x 0.25um), and a VGA 101 VUV spectrometer. The VUV spectrometer was operated with nitrogen as the make-up gas (0.35psi), a transfer line/flow cell temperature range of 200°C to 300°C, and a spectral range of 125nm to 430nm. The optimization method utilized was a face centered central composite design, and the parameters that were optimized included the final inlet temperature (200°C, 250°C, 300°C), GC flow rate (1.9mL/min, 3.2mL/min, 4.5mL/min), and make-up gas pressure (0.00psi, 0.15psi, and 0.30psi). Flow cell temperature was optimized independently (200°C, 250°C, 300°C).

Various explosive compounds of interest were included in the optimization: Triacetone Triperoxide (TATP), 2,3-Dimethyl-2,3-Dinitrobutane (DMNB), Nitroglycerine (NG), diphenylamine, 2,4,6-TRInitrotoluene (TNT), Pentaerythritol Tetranitrate (PETN), and RDX. Nitrate ester and nitramine explosives thermally decompose in the transfer line/flow cell of the VUV detector at higher temperatures (>250°C) into nitric oxide, carbon monoxide, formaldehyde, oxygen, and water. Compounds that thermally decomposed (NG, PETN, and RDX) had higher optimized GC flow rates (4.5mL/min, 4.5mL/min, and 2.6mL/min, respectively), while the remaining compounds had an optimized flow rate of 1.9mL/min. All compounds had an optimized make-up gas pressure of 0.00psi and final inlet temperature of 200°C with the only exception being diphenylamine with an optimized temperature of 286°C.

To investigate the applicability to forensic post-blast debris analysis, a bomb squad assisted in obtaining realistic samples of post-blast residues of single-base (IMR 4064) and double-base (Alliant Red Dot) smokeless powder on galvanized steel and Polyvinyl Chloride (PVC) pipe fragments. The pipes were placed in a perforated steel box prior to initiation to allow for collection of the post-blast debris for analysis. These samples were analyzed using the original method and the optimized method for comparison.

Gas Chromatography, Vacuum Ultraviolet Spectroscopy, Explosive Analysis

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