



A33 Analysis of Smokeless Powder Components by Capillary Electrochromatography - Time-of-Flight Mass Spectrometry

*Inge Corbin, BS**, Miami-Dade Police Department, 9105 Northwest 25th Street, Room 2149, Doral, FL 33172; *Maximilien Blas, PhD*, 11200 Southwest 8th Street, Miami, FL; and *Bruce R. McCord, PhD*, Department of Chemistry, Florida International University, University Park, Miami, FL 33199

After attending this presentation, attendees will learn how the components of commercial smokeless gunpowder can be detected and identified using capillary electrochromatography - mass spectrometry (CEC-TOF-MS).

This presentation will impact the forensic community by providing the details of a fast and robust analytical method with minimal sample preparation that avoids the sample instability and degradation that can occur with methods such as gas chromatography.

Unburned particles of smokeless powder found at a bombing scene can be analyzed to associate evidence found at the crime scene to a particular brand of powder. This identification may lead to a source of the powder and possibly generate investigative leads.

Individual standards of commonly found smokeless powder components were prepared by dissolving 1.0 mg of each standard in 1.0 ml of methylene chloride. A mixed standard was also prepared. An aliquot of each standard was put in a vial, the methylene chloride evaporated under ambient conditions, and reconstituted in acetonitrile and 5mM sodium phosphate buffer adjusted to pH 6.8. It was found that 85:15 was the optimum ratio. A 50 cm hexyl acrylate-based monolith was prepared and conditioned with the acetonitrile/buffer solution.

All standards were analyzed with an Agilent G3250AA LC/MSD TOF run in CEC mode. For each run the capillary was conditioned with buffer solution for five minutes, followed by electrokinetic injection of the sample at 30 kV for 10 seconds. The capillary was maintained at 30 kV and 5 bar pressure during the separation and TOF-MS detection.

The results proved the efficient and reproducible separation of ten compounds found in smokeless powders: diphenylamine, dimethylphthalate, diethylphthalate, dibutylphthalate, methyl centralite, ethyl centralite, 2-nitro- and 4-nitrodiphenylamine, and 2-nitroso- and 4-nitrosodiphenylamine.

The use of CEC-TOF-MS represents a promising analytical scheme for the detection and identification of smokeless powder components. It is a fast, reproducible technique for the discrimination of smokeless gunpowder that avoids the problems presented by the breakdown of thermally labile components of smokeless powder during GC-MS analysis. Further research will concentrate on post-blast analysis of both intact and burned smokeless powder components.

Smokeless Powder, Electrophoresis, Mass Spectrometry