B138 Comparison of HPLC and MECE for the Analysis of Organic Constituents in Smokeless Powder

Michael P. McCarriagher, BA, and J. Graham Rankin, PhD, Marshall University, Forensic Science Program, 1401 Forensic Science Dr., Huntington, WV 25701; and Wayne K. Morehead, MS, ForensicTrace, 7 Boxthorn, Rancho Santa Margarita, CA 92688*

After attending this presentation, attendees will come to appreciate the need for proper method validation for the quantitative analysis of the organic components of smokeless powders, as well as any limitations of these methods in regards to accuracy and precision.

This presentation will impact the forensic community and/or humanity by providing a better understanding of what knowledge can be gained from conducting validation studies on methodology used in the quantitative analysis of the organic components of smokeless powders.

Micellar electrokinetic capillary electrophoresis (MECE) and reverse phase high performance liquid chromatography (RP-HPLC) have both been shown to be viable analytical methods for the qualitative identification of the organic components found in smokeless powders which are used in arms ammunition and improvised explosive devices. The identification of these organic components from evidence collected at the scene of a crime can aid in the subsequent criminal investigation. Still, for these methods to be of greatest use to the forensic scientist, they must also yield reliable quantitative results for smokeless powder additives. MacCrehan et al (2002) have conducted an interlaboratory study, which compared the measurements of two powders, 1928-1 (the black particles of HiSkor 700X by the IMR Powder Company) and 1928-2 (231 distributed by Winchester). 1928-1 was shown to contain the propellant nitroglycerin (NG) and the stabilizer ethyl centralite (EC), while 1928-2 was shown to contain NG, the stabilizer diphenylamine (DPA) and DPA's nitrated derivative N-nitrosodiphenylamine (NnDPA). This study, however, yielded some inconsistencies in the quantitative identifications made by the participating laboratories. MacCrehan et al (2004) report the need for, and the introduction of, a reference material (RM 8107) for low explosives by the National Institute of Standards and Technology (NIST). This reference material contains NG, EC, DPA, and NnDPA. Quantitative determinations of RM 8107's components were carried out by NIST using both MECE and liquid chromatography. This reference material provides analytical laboratories with a means to validate their methods of organic additives analysis.

The purpose of the present research was to evaluate the performances of MECE and RP-HPLC in the quantitative identification of the additives present in the three powders mentioned above. Satisfactory performances in the analysis of these three materials will permit, and optimize, further research involving the characterization of smokeless powders in this laboratory. The goal of this ongoing project is to develop a database of analytical values for common organic constituents of smokeless powder in order to establish statistical criteria for determining the similarity between two different samples obtained in forensic evidence (i.e., bomb scene and suspect). At present the common procedure is comparison of physical size and shape of powder grains to determine brand (Wallace and Midkiff, 1993). Lot to lot variation within brand in chemical composition may provide additional probative value to the evidence.

Nitroglycerin measurements made by MECE analysis were found to fall within the expected ranges (MacCrehan et al, 2002) for 1928-1 and 1928-2. DPA and EC measurements for these powders were below the reported values. All measurements of additives present in RM 8107 were found to be consistent with NIST values. Intra-day reproducibility ranged from 1.83 to 2.08 (%RSD) for NG measurements made by MECE, while inter-day reproducibility was found to be 3.60 NG and 14.60 for DPA over a five day period. MECE provided resolutions around 3.548 for the closely eluting 2,4 DNT and 2,6 DNT. The capillary efficiency as determined by NG was ~60,300 theoretical plates. RP-HPLC analyses were below the reported range of values for all additives of all powders. Repeatability of NG was observed to be from 2.88 to 5.15 (%RSD) of the three powders within a day, while inter day reproducibility was found to be 1.02 for NG and 2.50 for DPA, over a five day period. RP-HPLC was unable to completely resolve 2,4 DNT and 2,6 DNT (resolution of 0.808) and displayed an efficiency of ~9,200 theoretical plates for NG.

Smokeless Powder, HPLC, Capillary Electrophoresis

 ** Presenting Author*