



B49 Forensic Analysis of Organic and Inorganic Components of Black Powder Substitutes by Ion Chromatography (IC) and Capillary Electrophoresis (CE)

Megan N. Bottegal, BS, and Bruce R. McCord, PhD, Florida International University, Chemistry Department, 11200 SW 8th Street, Miami, FL 33199*

After attending this presentation, attendees will be introduced to two newly developed methods for the detection of ascorbic acid in black powder substitutes.

This presentation will impact the forensic community and/or humanity by demonstrating how the information gained from using these methods for the analysis of black powder substitutes can serve as an investigative lead for law enforcement personnel in the event these types of powders are used in the commission of an illegal act, such as the construction of an Improvised Explosive Device (IED).

Black powder substitutes are alternative propellants that have several advantages over traditional black powder: they are not classified as explosives, making them easier to purchase; they generate less smoke when fired; and they are less corrosive to the gun barrel. These powders come in a variety of formulations and grain sizes, but generally contain inorganic oxidizers and organic fuels. Depending on the formulation, the oxidizer may consist of KNO_3 and/or KClO_4 . Typically, charcoal is selected as the fuel component, but in recent years, several manufacturers have instead turned to the fruit sugar ascorbic acid. This compound has the advantages of water solubility and a reduced smoke output when burned. These characteristics of ascorbic acid result in less fouling of the gun barrel, leading to a faster reloading time.

Previously, no published methods existed whereby both ascorbic acid and the inorganic oxidizer anions ClO_4^- and ClO_3^- could be visualized during the same run on a capillary zone electrophoresis (CZE) or an ion chromatography (IC) system. To that end, methods were developed for the analysis of ascorbic acid, perchlorate, chlorate, and chloride by capillary electrophoresis and ion chromatography. Concurrent use of these two methods resulted in a presumptive identification of the compounds of interest.

A CZE method was developed that used dual-indirect photometric detection to visualize both the organic and inorganic components. Separation was carried out on fused-silica capillaries with $75\mu\text{m}$ i.d. x 40 cm total length. Samples were injected in the hydrodynamic mode for 7s. The applied voltage was held constant at -12kV. The running buffer was 5 mM benzoic acid at an optimum pH of 7; benzoic acid served as the background electrolyte. The wavelength of detection was optimized to be 225 nm and 240 nm. The total run time was approximately fifteen minutes. The analytical method for the ion chromatography system utilized an isocratic elution with indirect photometric detection and conductivity detection in series. A weak anion exchange resin was used to separate sample components. An isophthalic acid buffer served as the UV-absorbing eluent; indirect photometric detection occurred at 280 nm.

The two methods described above were applied to the analysis of several black powder substitutes that contain ascorbic acid as the fuel component. Both burned and unburned powder samples were analyzed. Both the organic and inorganic components of interest were successfully isolated using the optimized instrumental methods. While the IC and CZE methods were only applied to powders known to contain ascorbic acid in this experiment, they are also applicable to powders that contain charcoal in place of fruit sugars.

Ion Chromatography, Capillary Electrophoresis, Ascorbic Acid