B137 Identification of Smokeless Powders Through Capillary Electrophoresis Using Highly Sulfated Beta Cyclodextrin

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The goal of this presentation is to determine the usefulness of highly sulfated beta cyclodextrin (HS-â-CD) in capillary electrophoresis (CE) for smokeless powder discrimination. This particular additive has been used for CE-MS analysis with electrospray mass spectrometry[1].

This presentation will impact the forensic community by demonstrating how HS-β-CD can be used as a pseudostationary phase in CE to separate and identify different components in smokeless powders and eventually discriminate between powders pre- and post-blast.

Smokeless powder evidence is frequently used in forensic casework to associate post-blast or gunpowder residue to a particular brand of powder. Such identification can eventually lead to potential suppliers who could be interrogated for further insight on past purchases and possible identification of a suspect. Samples were prepared by placing 1.0 mg of each powder into 1.0 mL of methyl chloride and extracting overnight. The supernatant is then air dried to remove the methyl chloride. Buffer is then added to the sample and mixed thoroughly. After which, the mixture is injected onto a CE instrument equipped with photodiode array detection for the separation and identification of the individual components. This information can then be used to help determine the manufacturer of the powder.

All standards and samples were analyzed using Beckman-Coulter P/ACE MDQ capillary electrophoresis system. Each run entailed a 2 minute 0.1M sodium hydroxide flush, followed by a 2-minute wash with the buffer being used. Initial testing was conducted with SDS in a borate buffer to provide plausible data for comparison.[2] Then a series of low pH buffers with varying concentrations of HS-β-CD, were examined to test the efficacy of the reagent. The varying concentrations were examined to develop a more time-efficient procedure. The initial buffer used was 50 mM phosphate and 10mM HS-β-CD at a pH of 2.5 which had proven to be an optimal concentration for separating amphetamines.[3] Further optimization was then performed to improve run time, increase resolution, and enhance compatibility with electrospray mass spectrometry.

The separation efficiency of cyclodextrin is based on its use as a pseudostationary phase in the CE buffer. The circular shape of the molecule includes a hydrophobic and optically active interior that forms inclusion complexes with host molecules. Considering that many components of smokeless powders are neutral, negatively charged sulfated cyclodextrins permit separations based on differential migration within the applied electric field. Following development of the buffer, samples of unburned and burned smokeless powders were compared to a standard mixture of common compounds found in known powders, including ethyl centralite, 2-nitrotoluene, 4-nitrotoluene, 2,4- dinitrotoluene, 2,6-dinitrotoluene, nitroglycerine, 2-nitrodiphenylamine, 4-nitrodiphenylamine, diphenylamine, dimethyl phthalate, and diethyl phthalate. The results demonstrated an efficient and reproducible separation of powder components.

Overall, the use of HS-â-CD in CE represents an attractive alternative to micellar mobile phases incorporating SDS. Future analysis through the incorporation of capillary electrophoresis coupled to electrospray mass spectroscopy will permit direct identification of each component in the powder.

References:

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2 David Northrop, Daniel E. Martire, William A. MacCrehan. *Separation and identification of organic gunshot and explosive constituents by micellar electrokinetic capillary electrophoresis.* Analytical Chemistry 1991 63, 1038-1042

3 Yuko T. Iwata, Agnes Garcia, Tatsuyuki Kanamori, Hiroyuki Inoue, Tohru Kishi, Ira S. Lurie, *The use of a highly sulfated cyclodextrin for the simultaneous chiral separation of amphetamine-type stimulants by capillary electrophoresis.* Electrophoresis. 2002 23(9), 1770 – 1776.

Capillary Electrophoresis, Cyclodextrin, Smokeless Powders

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