A83 Trace Analysis of Urea Nitrate as an Ion-Pair

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After attending this presentation, attendees will understand the potential new methods for the analysis and detection of urea nitrate at trace levels.

This presentation will impact the forensic science community by providing a better understanding of the development of reliable procedures for the detection of this energetic salt. This presentation will review a number of different methods for the analysis of this energetic salt.

Urea nitrate is a fertilizer-based explosive that has been used by terrorists in various bombings all over the world. One example of its use is the World Trade Center bombing in New York City in 1993. One interesting property of urea nitrate is that it is easily decomposed into urea and nitrate in the presence of water. As these two components may be ubiquitous in certain environments and not necessarily characteristic of the explosive, new methods are needed to characterize the urea nitrate salt and differentiate it from low level background ions.

This project aims at developing a reliable method to extract urea nitrate as an ion-pair from a variety of surfaces and analyze it at trace levels using spectroscopic (UV-visible and fluorescence) and/or mass spectrometry (MS) detection. The ultimate goal of this project is to rapidly detect urea nitrate as a non aqueous ion pair on a variety of surfaces. Therefore, the analysis has to be quick and very sensitive while interferences with urea, nitrate, and other matrix components should be as low as possible.

Urea nitrate is only slightly absorbent in the UV-visible range so derivatization is needed for spectroscopic analyses. In this study, the derivatization was performed with fluorescent compound xanthydrol, which has already been used successfully for urea detection. After the derivatization, the samples were injected on reverse high performance liquid chromatography (HPLC) coupled to UV and fluorescence detection to separate the different products. A gradient composed of sodium acetate or ammonium acetate and acetonitrile mobile phases permitted separation of all compounds in less than 20 minutes. Different parameters such as reaction temperature, solvent and time, xanthydrol amount and wavelength were optimized to achieve accurate quantification over the widest range of concentrations possible. Then extraction from both porous and non porous surfaces was investigated. Finally, potential interferences were added to the sample to determine how they affect the analysis. Preliminary results indicated detection limits around 0.05 mM or 6 ppm and the ability to differentiate urea nitrate from urea and nitrate samples. Thanks to addition of an internal standard, a quantification limit very close to the detection limit was achieved.

In parallel, mass spectrometry was used in an attempt to characterize urea nitrate as an ion pair without any derivatization. Electrospray ionization (ESI) and time of flight mass spectrometry were used in order to determine the exact mass of urea nitrate potential adducts. In order to separate urea nitrate from other compounds present in the sample, liquid chromatography has been coupled to the ESI-MS system and HILIC (Hydrophilic Interaction Liquid Chromatography) columns have been tested since they allow a good separation with only a small amount of aqueous phase (which can break the ion-pair).

These two methods should permit the detection of urea nitrate with high accuracy and permit its detection on a wide variety of surfaces. **Urea Nitrate, Fluorescence, Mass Spectrometry**

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