B137 The Chemistry of Phosphorus-Containing Reducing Agents and the Significance of Phosphate, Phosphite, and Hypophosphite in Clandestine Laboratory Casework

David M. Northrop, PhD, Washington State Patrol Crime Laboratory, 2700 116th Street, NE, Suite P, Marysville, WA 98271; Eric C. Person, PhD, California State University, Fresno, Department of Chemistry, 2555 East San Ramon Avenue, SB 70, Fresno, CA 93740-8034; Lori A. Knops, BS, North Carolina State Bureau of Investigation, PO Box 2408, Skyland, NC, 28776-2408; and Robert A. Heegel, BA, Washington State Patrol Crime Laboratory, 143302 East Law Lane, Kennewick, WA 99337*

After attending this presentation, attendees will understand the signif- icance of the anions of phosphate, phosphite, and hypophosphite in the various phosphorus – iodine methods of methamphetamine manufacture and how these anions can be identified using capillary electrophoresis.

Due to the similarities of the various phosphorus – iodine methods of manufacture, this presentation will impact the forensic community and/or humanity by providing a better understanding of the phosphorus chemistry involved in each method as well as assisting in the recognition of the man- ufacturing process through the use of inorganic anion analysis by capillary electrophoresis.

Since the early 1980s, the use of red phosphorus with iodine or hydriodic acid has been a common means of reducing ephedrine and/or pseudoephedrine to methamphetamine. Other phosphorus-containing reducing agents, such as hypophosphorous acid, phosphorous acid, phos- phorus triiodide and white phosphorus, can be used as a substitute for red phosphorus to convert iodine to hydriodic acid. Although these other phos- phorus-containing reducing agents are common in some regions of the world, they do not have the popularity that the red phosphorus method cur- rently has in the United States.

Identification of phosphorus-containing reducing agents used in sus- pected methamphetamine laboratories is an integral part of the evaluation of samples collected from these sites. Samples may include unused starting materials, reaction mixtures, and waste byproducts. Visual inspection of samples prior to analysis provides only limited value in assessing possible materials present. Chemical analysis is required to identify materials present in collected samples. In the absence of readily identified starting materials, evaluation of reaction mixtures and or waste products is nec- essary to identify the manufacturing method and make production capacity estimates.

Six experiments were carried out using different phosphorus-con- taining reducing agents (1 – red phosphorus, 3 – hypophosphorous acid, 1 – phosphorous acid, and 1 – phosphorus triiodide) with iodine to convert ephedrine to methamphetamine. Samples were collected throughout each experiment to provide a means of monitoring the reaction progression and the chemical species generated during the reaction process.

This paper will discuss the use of anion analysis by capillary elec- trophoresis (CE) for the identification of iodine and phosphorus-containing anions (hypophosphite, phosphite, and phosphate) and compare those results to organic analysis data obtained using gas chromatography / mass spectrometry (GC/MS). Phosphorus chemistry and reaction mechanisms will be reviewed for the purpose of determining the significance of finding various phosphorus anions in samples that have been collected. Equations have been developed, using phosphorus chemistry and reaction mecha- nisms for the various phosphorus-containing reducing agents in combi- nation with iodine, that can be used to predict the ratios of phosphorus anions that should be present based on the type and quantity of starting material. Using these predictive measures, anionic species identified in samples from clandestine laboratories can be used to determine what starting materials were used and the relative ratios of those starting materials. Results from the examination of actual case samples will be presented.

Methamphetamine, Phosphorus, CE

 ** Presenting Author*