Y14 Analysis of Drugs Used in Crimes Using Solid Phase Extraction (SPE) and Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

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Learning Overview: The goal of this presentation is for attendees to learn of an alternative to detecting drugs in very low concentrations that are used in the facilitation of crime.

Impact on the Forensic Science Community: This presentation will impact the forensic science community as this project aims to develop a more efficient and sensitive method to detect drugs that are commonly used to facilitate crime. The optimized method in this project has lower Limits Of Detection (LOD) than methods currently used in forensic laboratories.

Benzodiazepines are a class of antianxiety drugs that are also known as predator or date-rape drugs used in Drug-Facilitated Crimes (DFCs)—crimes that include robbery and the maltreatment of the elderly and children, as well as rape and other sexual assaults. Identification of these drugs or their metabolites in biological specimens such as urine, blood, saliva and hair of victims is commonly proof of exposure to the drug.

Like other psychoactive drugs used in DFCs, benzodiazepines are highly potent and have short half-lives. The combination of potency and a short half-life makes the time window for when the drug is still detectable in biological samples very small. Victims of DFCs usually experience short-term memory loss and often report the crime days after they happened, which adds to the challenge of detecting the drugs in the biological samples by the currently used analytical methods in forensic toxicology laboratories. The goal of the current project is to develop a method for the detection, identification, and quantification of benzodiazepines, including 7-aminoflunitrazepam, alpha-hydroxyalprazolam, alprazolam, bromazepam, chlordiazepoxide, clonazepam, diazepam, flunitrazepam, lorazepam, and nitrazepam, at very low concentrations in aqueous solutions, which can be adopted for the analysis of these drugs and their metabolites in biological samples such as urine and blood.

An SPE coupled to an LC-MS/MS method was successfully optimized. A Supel™- Select HLB 54183-U 200mg cartridge was selected for SPE. Washing and condition of the packing was completed using 12mL of methanol and 2mL of ultra-pure water, respectively. After sample introduction, the analytes of interest were eluted using 5mL of a 50:50 mixture of methanol and acetonitrile. The elutant was evaporated using rotary evaporation and, upon dryness, was reconstituted using 1mL of 0.01M ammonium acetate. The reconstituted sample was then subjected to LC using a C18 HPLC column (particle size: 1.9µm; length: 20cm) with gradient elution with a mixture of acetonitrile and 0.01M ammonium acetate buffer solution. The elution started with100% 0.01M ammonium acetate and was gradually decreased to 10%, while acetonitrile was increased to 90% and held for one-half minute. Then 0.01M ammonium acetate was increased back to 100% and held for 4min. For mass spectrometry detection, a Selected Ion Monitoring (SIM) method was started at minute 3.00 of analysis until minute 8.00. Using the optimized SPE and LC-MS/MS method, this study achieved the LOD for ten analytes and their metabolites to be in the range of pg/mL to ng/mL levels, which is much lower than LODs for the common methods used in forensic labs.

DFC, Liquid Chromatography, Solid Phase Extraction