B187 The Identification of Various Controlled Substances by Headspace Chemical Analysis Using Headspace Solid-Phase Microextraction (HS/SPME) and Gas Chromatography/Mass Spectrometry (GC/MS)

Justin Day, MS, PO Box 129, Mexia, TX 76667; Harry R. Ehmann, MS, Harris County Institute of Forensic Sciences, 1861 Old Spanish Trail, Houston, TX 77054; and Jorn Chi-Chung Yu, PhD, Sam Houston State University, Dept of Forensic Science, Box 2525, Huntsville, TX 77341*

After attending this presentation, attendees will be familiar with a solventless HS/SPME method with the option of simultaneous headspace derivatization for the analysis of a variety of controlled substances.

This presentation will impact the forensic science community by demonstrating the utility of this method for testing microgram sample levels, reducing the time and number of steps for extraction and/or derivatization, reducing the consumables required, and reducing or eliminating issues arising from "dirty" samples, such as edibles and botanicals.

Controlled substances come in a variety of forms that require a variety of extraction methods to properly isolate analytes of interest. Liquid-Liquid Extraction (LLE), Solid-Phase Extraction (SPE), and dilute and shoot extractions combined with chromatography and mass spectral analysis are the most common techniques for identifying controlled substances. Some samples, including edibles and botanicals, are notorious for causing instrument problems, even when cleanup procedures are utilized. Dirty samples can cause sample carryover during instrumental analysis, requiring additional blanks or cleanups before another sample can be analyzed. In some cases, instrument components may need to be replaced to maintain separation efficiency or eliminate contamination. Furthermore, some substances require derivatization for separation and stability. This often requires caustic reagents, heating blocks, additional chemical cleanup, and significant time. Each additional step in the process presents an opportunity for error, and retesting is not always possible.

Dried analytical controlled substance standards and seized case specimens were tested. Case specimens included crystals, powders, tablets (pharmaceutical and clandestine), liquids, botanicals, and edibles. Sample sizes ranged from micrograms of material to intact tablets. Samples were placed directly into headspace vials and sealed. Using an oil bath and a Polydimethylsiloxane (PDMS) SPME fiber, this study optimized incubation time, extraction temperature, and fiber exposure time for HS/SPME from a sample's headspace. Initial analysis was performed using a GC/Flame Ionization Detector (FID) with a modified injection port to establish retention times, recovery amount, and peak quality. The information from the GC/FID runs was used to program optimal parameters for GC/MS testing. A concurrent study of the viability of headspace derivatization was performed on samples whose analytes would benefit from the process, including the synthetic cathinones, psychedelic mushrooms, and marijuana. Derivatization was achieved by placing a small insert filled with a measured amount of solvated or dry derivatizing agent directly into a headspace vial with a sample. Physical contact of the sample with the agent was not allowed; that is, the entire derivatization process occurred in the headspace during the incubation period. Results from the new method to those from the samples extracted with standard procedures were compared.

It was found that a variety of controlled substances in a variety of forms can be extracted with HS/SPME. The incubation temperature proved to be the most significant variable. Most compounds extracted well at 150°C, but some appeared to need higher temperatures that are beyond the capability of an oil bath. Incubation times ranging from five to ten minutes, with one-minute fiber exposure, proved sufficient for extraction and derivatization. The technique, for the most part, provided similar results; however, sometimes additional compounds not found in standard extractions were able to be extracted. Though not always controlled substances, the presence of these other compounds could serve as unique identifiers for revealing formulation trends, synthesis methods, growing regions, and other information that could be of interest to multiregional agencies. This method also demonstrated that derivatization does occur in the headspace, turning it into one-step process. This technique is simple and, for the most part, non-destructive. No sample required cutting, crushing, scraping, or dissolving, and most exhibited little or no change after testing. The initial work is promising and further study for the optimization of this new technique will be discussed in this presentation.

Headspace/SPME, Solventless, Derivatization

*Presenting Author