K10 The Detection of Various Drugs in Human Urine Samples Via Total Vaporization-Solid Phase Microextraction (TV-SPME)

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Learning Overview: After attending this presentation, attendees will understand: (1) TV-SPME and how it may be used to detect drug analytes, and (2) the ability of Gas Chromatography/Mass Spectrometry (GC/MS) and TV-SPME to detect drugs in toxicological samples.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by showing how TV-SPME may be implemented in laboratories.

While GC/MS is a frequently used technique in forensic science laboratories, there are limitations, such as the need for compounds to be thermally stable as well as volatile. Some compounds must undergo derivatization prior to being injected into the gas chromatogram to satisfy these requirements. SPME is a technique in which analytes are absorbed onto a fiber that is then placed inside the GC inlet for desorption. TV-SPME utilizes the same technique as standard SPME but vaporizes a sample extract that is then absorbed onto the fiber. The Polydimethylsiloxane-Divinylbenzene (PDMS-DVB) fiber is first exposed to a vial containing the derivatization agent. The SPME fiber is then exposed to a new vial containing the analyte of interest. A study was conducted to determine the optimal time needed for the fiber to absorb the derivatization agent so that both the solvent (e.g., water) and the analyte were fully derivatized with no underivatized compounds detected. This study indicated that 50 minutes was the optimal time needed to adequately saturate the fiber. TV-SPME is beneficial because it allows for the analyte to be derivatized during the extraction process, which reduces analysis time. TV-SPME requires little sample preparation and small sample sizes.

It was hypothesized that TV-SPME could be used to analyze drugs and drug metabolites in human urine samples. The GC temperature program was set to an initial temperature of 60°C with a 15°C/min oven ramp. The inlet temperature was 250°C using splitless mode. The MS scan range was 40m/z–550m/z. Drugs of interest included methamphetamine and amphetamine. Each drug was spiked into a human urine sample known to be free of any illegal substances. Concentrations were at ng/mL levels. Each sample was then analyzed using a TV-SPME GC/MS method after being exposed to the proper derivatization agent for a sufficient amount of time. After exposing the SPME fiber to Trifluoroacetic Anhydride (TFAA) for 50 minutes, methamphetamine (28.6ng/mL) and amphetamine (19.0ng/mL) derivatives were both successfully detected in a urine sample.

Currently, this method shows that methamphetamine and amphetamine can be detected at ng/mL levels in human urine with no prior sample preparation aside from placing the sample into a headspace vial. Future work will include a recovery analysis and future compounds may include benzoylecgonine, ɣ-Hydroxybutyric Acid (GHB), ɣ-Butyrolactone (GBL), and 11-Nor-9-Carboxy-Δ^9-Tetrahydrocannabinol (THC-COOH). There are currently numerous methods and techniques available for the analysis of controlled substances. However, these methods often require sample preparation such as an extraction. This TV-SPME method requires little-to-no sample preparation and utilizes a simple GC/MS method.

Gas Chromatography, Solid-Phase Microextraction (SPME), Drugs