B19 The Detection of Gamma-Hydroxybutyric Acid (GHB) in Water and Mixed Drinks Without Sample Preparation Using Total Vaporization-Solid Phase Microextraction (TV-SPME) With On-Fiber Derivatization

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After attending this presentation, attendees will be aware of a new technique by which GHB can be effectively identified by Gas Chromatography/Mass Spectrometry (GC/MS) in aqueous or mixed drink samples with no sample preparation required.

This presentation will impact the forensic science community by introducing a new procedure for the detection and identification of GHB in drink samples using GC/MS without extensive sample preparation. The method is simple and effective and could be used to acquire evidence of drug-facilitated sexual assault involving the use of GHB.

GHB is often used in drug-facilitated sexual assaults because it incapacitates victims, induces memory loss, and does not persist in the human body.[1] The drug can be surreptitiously administered to a victim's drink, in which case a forensic scientist would receive a beverage suspected of containing GHB. The "workhorse" technique of forensic science laboratories is GC/MS. GHB is difficult to analyze by GC/MS due in part to its acidity (pKa 4.7), high polarity, and high solubility in aqueous solution. It is also well known that GHB readily dehydrates to Gamma-Butyrolactone (GBL) in the high heat of the GC inlet, further complicating its analysis.[2] One method used to make compounds more amenable to GC/MS analysis is derivatization, in which labile hydrogens are replaced with more stable groups, resulting in a product that has increased volatility and stability.[3] Derivatization in solutions followed by liquid injection GC/MS is already in use in forensic science laboratories, but this practice requires separate steps for solvent extraction, derivatization, and analysis.

Solid Phase Microextraction (SPME) is a technique in which the analytes are pre-concentrated onto a thin fiber coated in absorptive or adsorptive material. In TV-SPME, a small aliquot of analyte in solution is placed in a vial and heated until the sample completely vaporizes, resulting in a two-phase system. An SPME fiber is then introduced and the sample is absorbed onto the fiber coating. The maximum volume for total vaporization of a given solvent can be easily calculated given the solvent vapor pressure, molecular weight, vial volume, and temperature.[4] For example, the calculated maximum volume of methanol for total vaporization in a 20mL vial at 60°C is 24µL. The use of TV-SPME for sampling can streamline the derivatization process by allowing the derivatization to be done on-fiber. In this process, an SPME fiber is exposed to the headspace of a vial containing derivatization agent, then exposed to the heated headspace of a vial containing the sample. The reaction between the analyte and the derivatization agent then takes place directly on the SPME fiber. After sufficient time for reaction, the fiber is moved to the inlet of the GC for desorption. The use of a robotic autosampler can make this a fully automated process for which no sample preparation is necessary.

This method is ideal for the analysis of GHB in drink samples. GHB has been successfully identified in aqueous solution and in simulated mixed drinks by TV-SPME with on-fiber derivatization using N, O-Bis trifluoroacetamide with 1% trimethylchlorosilane (BSTFA + 1% TMCS).

Reference(s):

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Solid Phase Microextraction, Derivatization, GHB

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