B69 The Analysis of Gamma-Hydroxybutyric Acid (GHB) and Gamma-Butyrolactone (GBL) in Forensic Samples Using Gas Chromatography/Mass Spectrometry (GC/MS) and Proton Nuclear Magnetic Resonance (1H NMR)

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After attending this presentation, attendees will be presented information on the interconversion between GHB and GBL in solution at different pH's and time before analysis. Also, a method for the analysis of GHB will be presented which does not cause inadvertent conversion between GHB and GBL during sample preparation and analysis and still maintains the sensitivity, precision and linearity of other methods currently employed.

This presentation will facilitate a more thorough understanding of the relationship that exists between GHB and it's lactone, GBL, in solution. This understanding is necessary to ensure the full characterization of the sample and accurate interpretation of results. A method for analysis of GHB is presented which does not cause inadvertent conversion between GHB and GBL and still maintains the sensitivity, precision and linearity of other methods currently employed.

Gamma-Hydroxybutyric Acid (GHB) is an endogenous compound found in the central nervous system (CNS) and peripheral tissues. GHB, a CNS depressant, is abused recreationally for its purported euphoric and relaxation effects and for the purposes of drug facilitated sexual assault (DFSA) due to its sedative and amnesic effects. The dramatic increase in the abuse of GHB over the past decade has created the need for analytical methods to detect GHB in a variety of matrices. Specifically, the growing use of GHB for the purposes of drug facilitated sexual assault calls for the development of a method to determine if GHB is present in a drink that is suspected of having been spiked.

The analysis of GHB has presented some analytical difficulties in forensic laboratories due to the equilibrium that exists between GHB and its lactone, GBL, in solution. Many methods currently employed may inadvertently cause conversion between GHB and GBL during sample preparation and analysis. A thorough understanding of the relationship that exists between GHB and GBL under different conditions in solution can be used to guide analytical methodology so that unintentional sample manipulation does not occur.

In an effort to determine the effect of pH and time before analysis on the interconversion between GHB and GBL, a study was conducted using 1H NMR. Solutions of GHB and GBL were buffered to different pHs (2.2, 3.1, 4.5, 6, 7.1, 8, 10) using a 1M phosphate buffer prepared in deuterium oxide. Solutions of GHB and GBL in pure D2O were also analyzed. The samples were stored under ambient conditions and analyzed at time zero and at selected time intervals thereafter for several months using 1H NMR. Each solution was prepared and analyzed in triplicate. The area of resonance lines known to originate exclusively from GHB and GBL were ratioed to each other to determine the percentages of GHB and its lactone in solution at a particular pH and time. pH was found to have a significant effect on the interconversion between GHB and its lactone in solution. With this information in mind, a method was developed that avoids sample manipulations such as pH adjustment that could cause inadvertent conversion between GHB and GBL. In the method developed, solid phase microextraction (SPME) which is a fast, simple and solvent free method for the extraction of drugs directly from aqueous samples was used for extraction and preconcentration of GHB. Extracted GHB was then derivatized on-fiber using a silylating agent (BSTFA/TMCS). Derivatization offers several advantages: It imparts thermal stability so that conversion of GHB to GBL in the heated injection port of the gas chromatograph will not occur. Also, by derivatizing GHB, a less polar and more volatile compound with better chromatographic properties is analyzed. Gas chromatography-Mass spectrometry (GC/MS) was then used for the separation and detection of derivatized GHB.

The method detects GHB in aqueous based matrices with good sensitivity, high precision, excellent linearity from 0.01 mg/mL to 0.25 mg/mL and without the need for sample manipulation that could cause interconversion between GHB and its lactone. The method was successfully applied for detection of GHB in water as well as in several alcoholic and non-alcoholic beverages.

Gamma-Hydroxybutyric Acid, Interconversion, Solid Phase Microextraction

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