B14 The Analysis of Gamma-Hydroxybutyric Acid (GHB) and GammaHydroxybutyrolactone (GBL) in Forensic Samples Using GC/MS and ^{1}H NMR

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This presentation will describe a fast, simple, and solvent free method for detection of Gamma-Hydroxybutyric acid (GHB) without conversion to its corresponding lactone, (GBL). Distribution constants for the conversion between GHB and GBL at different pHs over time will also be presented.

Gamma-Hydroxybutyric acid (GHB), a CNS depressant, has been used repeatedly over the past decade to commit drug-facilitated sexual assault. The growing use of GHB for the purpose of date rape calls for the development of a method to determine if GHB is present in a drink that is suspected of having been spiked or if GHB is present in a urine sample of a person who believes they may have been drugged and assaulted. This study proposes to develop a method for detection of GHB in such samples. Limits of detection and linear range for the method developed will be determined and compared to existing methods.

Method development is complicated by the equilibrium that exists between GHB and its corresponding lactone (GBL) in solution. This study proposes to determine the equilibrium reached between GHB and GBL at different pHs over time using ^{1}H NMR.

Solid phase microextraction (SPME) will be used for the extraction and pre-concentration of GHB. Solid phase microextraction is a simple, effective adsorption/desorption technique, which eliminates the need for solvents or complicated apparatus for concentrating compounds in liquid samples or headspace. Many methods of extraction currently being implemented for GHB analysis either intentionally or inadvertently convert GHB to GBL or vice-versa. The method proposed for extraction, SPME, does not cause conversion between GHB and GBL, which is important due to the legal distinction between the two compounds. GHB will then be derivatized on-fiber using BSTFA/TMCS (99:1) in order to impart thermal stability so that conversion from GHB to its lactone does not occur in the heated injection port of the gas chromatograph. The best fiber for extraction and desorption of GHB will be determined based on extraction efficiency, carryover percentage and background produced by the fiber. Derivatization of GHB will be optimized by varying the amount of derivatizing agent used and the time the fiber is placed in the headspace of the derivatizing agent. Gas Chromatography-Mass Spectrometry will then be used for the separation and detection of derivatized GHB. A Varian 3400 Gas Chromatography instrument is used for separation while a Varian Saturn 2000 Ion Trap Mass Spectrometer is used as a detector.

Distribution constants between GHB and its corresponding lactone will be determined at different pHs over time in order to understand the effect of the sample medium on GHB detection. For instance, GHB is often spiked into alcoholic beverages with low pHs. It is therefore important to know how pH will affect the ratio of GHB to GBL so proper interpretation of results can be made. To determine equilibrium constants at various pHs, samples of GHB will be dissolved in deuterated water and buffered at pHs ranging from 2 to 12. The samples will be analyzed at selected time intervals using a 400 MHz NMR in proton mode over a period of several months. These same procedures will be followed for samples of GBL. Once stable conditions are reached, equilibrium ratios will be determined based on integration of peaks known to come from either GHB or GBL exclusively.

Gamma-Hydroxybutyric Acid, Solid Phase Microextraction, Equilibrium Constant

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