K13 Application of Ion Mobility Spectrometry to the Analysis of Gamma-Hydroxybutyrate and Gamma-Hydroxyvalerate in Toxicological Matrices

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After attending this presentation, attendees will learn about a rapid, portable, screening technique for the simultaneous analysis of GHB, GHV, and analogs in urine. The physical extraction of the hydrophilic analytes from urine will be discussed, as will the benefits of ion mobility spectrometry in forensic analyses.

This presentation will impact the forensic community and/or humanity by introducing a physical extraction with ion mobility spectrometry as a rapid, portable screening technique suitable for the detection of GHB, GHV, and analogs in urine.

The predator drug, gamma-hydroxybutyrate (GHB), the lactone precursor (gamma-butyrolactone, GBL), and the diol precursor (1,4- butanediol, BD) continue to present significant analytical challenges to forensic toxicologists and chemists. The five-carbon analog (gamma- hydroxyvalerate, GHV) and the corresponding lactone (gamma- valerolactone, GVL) are emerging as substitutes for GHB, adding further complications.

A rapid and reliable screening test for detection of GHB and GHV would be useful for toxicologists as well as forensic chemists working with solid dose samples. This lab has previously reported a microcrystal test effective for aqueous solutions, but felt the development of a rapid, simple instrumental test effective for screening urine required development. In addition, GHB and GHV are extremely hydroscopic and hydrophilic, negating the possibility of rapid and simple extractions that might be necessary for quick screening. Thus, any successful GHB/GHV screening methodology must either be matrix independent or insensitive or capable of rapid and semi-quantitative extraction from the matrix. The latter issue has been the limiting factor to date.

Ion mobility spectrometry (IMS) was investigated as a method of screening urine for the presence of these drugs and their degradation products. In the present study, a high-performance split/splitless injector and autosampler were utilized to effect a physical separation of GHB and GHV from aqueous matrices (including urine) based on differences in relative volatility. This was achieved by a timed period of solvent evaporation followed by rapid temperature increase and thermal desorption of the residuals. The injection method in effect replaces problematic solvent extraction methods with a physical extraction, an efficient method in the present case considering the hydrophilic nature of GHB. Sample was introduced directly into a detection system

without any chromatography, resulting in rapid analysis times. The negative ion mode showed the greatest sensitivity with detection limits in the low parts-per-million range for GHB and GHV. Since GHB is often delivered in alcoholic beverages, ethanol and acetaldehyde, along with potential interfering compounds methanol, isopropanol, acetone, were also analyzed. None were found to interfere. The thermally- induced ring opening prevented differentiation of GHB and GBL using direct injection/ thermal desorption protocol, but IMS does show promise as a rapid, simple, and affordable screening technique for GHB and related compounds.

Reduced mobilities of GHB, GHV, GBL, GVL, and BD were determined by analysis of vapor generated from neat samples. Resulting Ko's are shown in Table 1. GHB, GBL, and BD were indistinguishable based on Ko's and standard IMS alarm variability (standard is ±50us in the drift times). Very slight difference in the reduced mobilities of GHB and GBL were noted, consistent with earlier results.

Table 1

Analyte	GHB	GHV	GBL	GVL	BD
Reduced Mobility (K_o)	1.7097	1.6190	1.7105	1.6380	1.7103

To gauge applicability in toxicology, GHB and GHV were dissolved in saturated synthetic urine solutions followed by serial dilutions as described previously. The synthetic urine was found to have no interfering peaks and LOD was estimated to be in the low ppm range by serial dilution methods. Furthermore, GHB and GHV are distinguishable in synthetic urine. Although the urine matrix contributes additional background peaks, the analytical peaks remain discernible.

Ion Mobility Spectrometry, GHB, GHV

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