



### K19 Comparative Analysis of GHB and GHV II

Jennifer Wiseman Mercer\*, BS; Lucy S. Oldfield, MS; Dina M. Shakleya, PhD; and Suzanne C. Bell, PhD, Bennett Department of Chemistry, West Virginia University, 217 Clark Hall, Morgantown, WV 26506; and Patrick S. Callery, PhD, School of Pharmacy, West Virginia University, Morgantown, WV 26506

After attending this presentation, attendees will learn about an emerging drug threat, gamma-hydroxy valerate (4-hydroxypentanoic acid, GHV), and a rapid, sensitive, quantitative confirmatory technique for the simultaneous analysis of GHV and its analog gamma-valerolactone (GVL). In the body, GVL and PD are metabolized to GHV with the associated effects. In addition, the synthesis of GHV from gamma-valerolactone (GVL) will be discussed, as GHV is not available for purchase from classic chemical retailers. This presentation will focus on comparative analysis via liquid chromatography with ultraviolet detection (LC-UV) and gas chromatography with mass spectrometric detection (GC-MS) and is meant to complement the related presentation entitled "Comparative Analysis of GHB and GHV I."

GHV has been shown to have effects similar to GHB but requires a higher dosage increasing the threat of toxicity and lethality. GHV is anecdotally reported to have a longer duration of action. Given that GHB and its precursors are controlled, drug abusers may switch to GHV. A recent comprehensive internet search revealed that commercial GHV products are sold on many websites. In addition, the effects of GHV also make it suitable for use in drug-facilitated sexual assault. The forensic community needs to be aware of this new drug and prepare to combat its use.

Gamma-hydroxy valerate (GHV) is the 4-methyl-substituted form of gamma-hydroxy butyrate (GHB). As GHV is not available as an analytical standard, a synthesis was developed which involves the hydrolysis of GVL. Thus, the similarities between the two should be exploited to formulate accurate and sensitive confirmatory tests for GHV and its precursors. Liquid chromatography with ultraviolet detection (LC-UV) is a suitable technique for the separation and confirmation of GHB and its analogs. Gas chromatography analysis is more difficult than liquid chromatography because generally GHB must be extracted from complicated matrices and subjected to derivatization due to the small size of the molecule. Thus, developed methods for GC-MS (with *in situ* derivatization) and LC-UV will be compared to determine the advantages and disadvantages of each method.

Since GHB and GHV are small polar molecules, LC provides a more desirable analysis than the more commonly used GC technique. Suspect solutions may be injected directly onto the column, thereby eliminating the need for extraction and derivatization steps required for GC analysis. The LC used in this study was a Shimadzu with the following components: SCL-10A controller, SPD-10A UV-Vis detector, SIL-10AD auto injector, LC-10AD liquid chromatograph (2), DGU-14A degasser, and CTO-10AS column oven. The software used was EZStart 7.2.1. The column used was a  $\mu$ Bondapak™ C18 3.9 x 300 mm column. The method is made quantitative by the addition of (S)-(+)-carvone as an internal standard. Data obtained from LC analysis is shown in **Table 1**.

Table 1 – LC Results			
Analyte	LOD	LOQ	Range
GHB	5 ppm	50 ppm	50 – 2000 ppm
GHV GBL	5 ppm	50 ppm	50 – 2700 ppm
GVL	100 ppm	200 ppm	200 – 2700 ppm
	25 ppm	100 ppm	100 – 2700 ppm

The GC was an Agilent model 6890 coupled with an Agilent mass selective detector model 5973. An HP-5 capillary column (30 m x 0.25 mm i.d., 0.25  $\mu$ m film thickness) was used for chromatographic analysis. The method was made quantitative by the use of 1,5-pentanediol as an internal standard. Data obtained from GC analysis is shown in **Table 2**. Future studies will test the methods' suitability for beverage and urine analysis.

Table 2 – GC Results				
Analyte	LOD	LOQ	Range	Quantitative
GHB	0.1 ppm	5	5 – 100	ions 233
GHV	0.1 ppm	ppm	ppm	75
		5	5 – 100	

#### GHV, GHB, LC-MS