



### **B17 Infrared (IR) Spectroscopic Identification of GHB Free Acid, GHB Salts, and GBL**

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The IR spectroscopic identification of GHB free acid, GHB salts and GBL will be discussed. After attending this presentation, the participant will have an understanding of the molecular structural changes that result in the infrared (IR) spectral differences between GHB free acid and its salts. The participant will also have an understanding of the changes observed in the IR spectra which can be used to identify GHB free acid in the presence of GBL in solutions following interconversion.

Infrared (IR) spectroscopy has been applied successfully to identifying GHB salts as well as GBL, both as neat materials and in a variety of forensic matrices. While other analytical techniques are capable of differentiating between GHB and GBL, IR has the advantage in that it can differentiate between GHB salts, GHB free acid, and GBL. In the case of GHB free acid, the molecular structure is partly composed of a protonated carboxylic acid functional group. Currently, no reference standard of GHB free acid is available. Based on its molecular structure, a predominate feature observed in the infrared spectrum of GHB free acid is a strong carbonyl absorbance band. The position of the carbonyl absorbance band of GHB free acid in the IR should be at approximately  $1710\text{ cm}^{-1}$ . The molecular structure of a GHB salt exhibits a carboxylate functional group where the carbonyl functionality would normally be present in the molecular structure of the free acid. The IR spectrum of a GHB salt exhibits two absorbance bands at approximately  $1556\text{ cm}^{-1}$  and  $1409\text{ cm}^{-1}$  representing the O-C-O or "bond and a half" structure of a carboxylate functional group.

It has been previously shown that GBL will interconvert to GHB free acid in aqueous solutions over a period of time based in part on the pH of the solution. GBL solutions encountered in forensic casework have frequently been aqueous based. Determination of the presence of GHB free acid, as a result of interconversion, is made difficult in the IR due to the presence of an IR absorbance band associated with water ( $\text{H}_2\text{O}$ ) at  $1650\text{ cm}^{-1}$ . The presence of the  $\text{H}_2\text{O}$  band at  $1650\text{ cm}^{-1}$  and the strong absorbance band associated with GBL at  $1750\text{ cm}^{-1}$  result in overlapping bands. The carbonyl absorbance band associated with the GHB free acid is present underneath this set of overlapping bands.

The first series of experiments focused on determining which changes in the IR spectra could be used to make an in-situ identification of GHB free acid in the presence of GBL and water. Following this series of experiments, the focus shifted to the extraction and isolation of GHB free acid as a reference standard and to obtaining an IR spectrum of the GHB free acid.

The first experiments used a model system composed of different mixtures of GBL, water and a GHB free acid analog, beta-hydroxybutyric acid (BHB). BHB is readily available as a standard and exhibits a strong carbonyl absorbance band at approximately the same position in the IR as GHB free acid. A series of standard solutions of GBL and BHB were prepared within concentration ranges observed in forensic samples that contained both GBL and GHB free acid. These standard solutions were analyzed by LC-UV and IR spectroscopy. The IR profiles generated by these mixtures were then analyzed, and observed spectral differences were correlated with a change in concentration of GBL or BHB. The IR spectral changes observed in the model experiments with GBL and BHB were then used in the interpretation of IR spectra generated on real GBL forensic samples. The real samples analyzed were determined to contain both GBL and GHB free acid that was present as a result of interconversion. The IR identification results were compared to the LC-UV results generated for the same set of forensic samples.

Based on the knowledge gained in working with the model system of GBL and BHB, a series of interconversion experiments were conducted with GBL in  $\text{D}_2\text{O}$  instead of  $\text{H}_2\text{O}$  in order to more readily observe the carbonyl associated with GHB free acid. The OH bend normally observed in  $\text{H}_2\text{O}$  at  $1650\text{ cm}^{-1}$  is shifted to lower wave numbers in  $\text{D}_2\text{O}$ . Removal of the interfering water band permitted the observation of the carbonyl associated with the GHB free acid. Attempts to isolate the GHB free acid from the interconversion samples were unsuccessful. An alternative approach to generate GHB free acid was developed by reacting GHB sodium salt with a stoichiometric amount of HCl. The GHB free acid was then extracted and an IR spectrum was obtained.

Structurally the GHB salts, GHB free acid, and GBL are different and as a result generate different IR spectra. IR spectroscopy can be used effectively to identify the presence of GHB free acid, GHB salt, and/or GBL in forensic evidence/samples.

#### **GHB Free Acid, GBL, IR Spectroscopy**