26.0 GAMMA-HYDROXYBUTYRATE (GHB) AND GAMMA-BUTYROLACTONE (GBL)

26.1 Testing Procedures

26.1.1 Samples in water

A color test is performed by placing a drop of liquid sample in a spot plate and adding 3 drops of GHB test reagent. A positive test will turn a greenish color and eventually will turn a pale blue. A negative test and a blank will stay bright orange.

The liquid sample can be prepared for FTIR analysis by placing an aliquot of the suspected sample in a mortar and drying it. Let the mortar cool completely before proceeding to FTIR analysis.

If an aqueous sample contains the lactone (GBL) instead of GHB, it will dry down to an oily residue instead of a powder. This oily residue may be analyzed on the FTIR and used to identify the GBL.

26.1.2 Samples in other aqueous matrices

Derivitization procedure:

⚠ *Personal protective equipment is required when working with derivatizing agents such as Bis-(Trimethylsilyl) Trifuoracetamide (BSTFA) and Trimethylchlorosilane (TCMS). All evaporations should be performed under a fume hood.*

Extract 1 to 5 mL of the unknown sample three times with excess $CHCl_3$. Extract once more with an equal amount of $CHCl_3$. This will remove any residual GBL from the sample. Analyze the last $CHCl_3$ layer on a GC-MS to show that all the GBL has been removed. If GBL is still present, re-extract and analyze the $CHCl_3$ until no GBL is present.

If possible, dry down the extracted sample in a small (~10 mL) beaker. This can be accomplished by using a hair dryer; however, do not exceed 150°C or the GHB may convert to GBL.

Crystallize with acetone by adding 10 mL of acetone to the beaker containing the sample and heating it on a hot plate at about 70°C (the boiling point of acetone is 57°C) with stirring until it boils. This should take no longer than five minutes. Immediately pour the hot solvent into another small beaker and dry off the acetone using a hair dryer on low heat.

Add 2 mL of acetonitrile, 500 μL of BSTFA, and 800 μL of TCMS to the beaker containing the dry sample. Cover the beaker and heat the solution on a hot plate between 60°C and 80°C for 10-15 minutes. Do not exceed 80°C, and do not let the solution boil.

Analyze the product on a GC-MS. To identify GHB-TMS, ions 117, 133, 459, 204, and 233 must be present. Ions common to TCMS derivatives include 73, 147, and 243.

Water inhibits the derivatizing agents. It is imperative that the sample is as dry as possible.

26.1.3 Samples of powdered GHB

A color test may be performed on suspected GHB powder. A neat FTIR analysis on a sample of the powder may be performed by preparing a KBr pellet or by utilizing an ATR accessory.

26.1.4 Samples of liquid GHB

An aliquot of suspected GHB liquid may be mixed with an equal portion of MEOH and injected on the GC/MS using the appropriate program. If GHB is present, it will convert to the lactone (GBL) in the heated injection port and present a peak with a base peak of 42 and a molecular ion peak of 86. A moderately concentrated sample must be used in order to see the lactone. This result only indicates that GHB is possibly present or the sample contains GBL. Therefore, a non-derivatized GC-MS is not a confirmatory test for GHB or GBL.

26.2 References

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