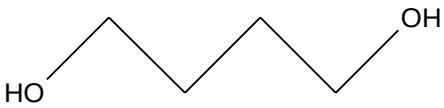
1,4-BUTANEDIOL

Latest revision: August 16, 2005



1. SYNONYMS

CFR: GHB analogs are treated as controlled substances under Federal law only if intended for human consumption. According to 21 U.S.C. ß 813, "a controlled substance analog(ue) shall, to the extent intended for human consumption, be treated, for the purposes of any Federal law as a controlled substance in Schedule I." Thus, authorities can prosecute drug offenses involving 1,4-butanediol in the same manner as offenses involving GHB. See 21 U.S.C. ß 802(32) for the definition of a controlled substance analog(ue).

CAS #: 110-63-4

Other Names:
1,4-Dihydroxybutane
Butylene glycol
1,4-Butylene glycol
1,4-Tetramethylene glycol
Tetramethylene 1,4-diol
Sucol-B

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)	Boiling Point (°C)
1,4-butanediol	$C_4H_{10}O_2$	90.1	16	230

2.2. SOLUBILITY

Form	A	C	E	H	M	W
1,4-butanediol	VS	S	S	I	VS	VS

A = acetone, C = chloroform, E = ether, H = hexane, M = methanol and W = water, VS = very soluble, FS = freely soluble, S = soluble, PS = sparingly soluble, SS = slightly soluble, VSS = very slightly soluble and I = insoluble

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

Most color tests are inadequate for 1,4-butanediol. There are currently no specific color tests for this compound and most common color tests give little response to 1,4-butanediol. Therefore, the results of both the Marquis and Mandelin's are subject to interpretation. (Morris, 2001)

COLOR TEST	RESULT
Marquis	Faint brown
Mandelin's	Brown

3.2. CRYSTAL TESTS

Currently, there are no reliable crystal tests.

3.3. GAS CHROMATOGRAPHY

Sample Preparation:
1,4-butanediol is soluble in methylene chloride and chloroform and may be extracted from aqueous solutions. It is also soluble in methanol.

Method - 1,4BD-GCS1

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 30 m x 0.33 mm x 0.25 µm film thickness
Carrier gas:	Hydrogen at 3.0 mL/min
Temperatures:	Injector: 260°C Detector: 300°C Oven program: 1) 100°C initial temperature for 1.0 min 2) Ramp to 270°C at 15°C/min 3) Hold final temperature for 3.7 min
Injection Parameters:	Split Ratio = 30:1, 1 µL injected

COMPOUND	RRT

1,2-butanediol	0.808
1,3-butanediol	0.845
GBL	0.964
1,4-butanediol	**1.00**

3.4. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method - 1,4BD-LCS1

1,4-Butanediol may be identified in aqueous solutions by LC-MS employing an ammonium acetate buffer (see the electrospray mass spectrum of 1,4-butanediol). The electrospray (+) mass spectrum displays a peak due to a protonated species of 1,4-butanediol (91 amu), as well as a peak for the cationic species (73 amu) that results from the loss of a hydroxide anion. The spectrum may also display a peak for the sodium ion complex with 1,4-butanediol(113 amu) if a sodium salt is present in the sample.

Standard Solution Preparation:
Prepare the standard solution of 1,4-butanediol (5-10 mg/mL) in methanol.

Instrument: High performance liquid chromatograph with atmospheric pressure ionization electrospray mass selective detector

Column: 5 μm Aqua C18, 100 mm x 4.6 mm

Detector: Scan mode, positive ion
Capillary voltage: 3000 V
Fragmentor: 30 eV
Nebulizer pressure: 60 psig
Drying gas flow: 13.0 L/min
Drying gas temperature: 350°C

Flow: 1.500 mL/min

Injection Volume: 5 μL

Buffer: 20 mM CH_3COONH_4 (~ pH 7.5)

Mobile Phase: 100% Buffer

COMPOUND	RRT
GHB	0.368

1,4-butanediol	**1.000**
GBL	1.188

Method - 1,4BD-LCS2

Instrument: High performance liquid chromatograph equipped with atmospheric pressure ionization electrospray mass selective detector

Column: 5 µm ODS hypersil, 4.6 mm x 10 0mm

Detector: Mass selective detector
Nebulizer gas temperature: 350°C
Nebulizer gas flow: 13 L/min
Nebulizer pressure: 55 psig
Capillary voltage: 5 kV

Flow: 0.75 mL/min

Injection Volume: 5 µL

Buffer: 1% acetic acid

Mobile Phase: Buffer: methanol 90:10
Samples are to be dissolved or diluted in mobile phase, then filtered with a 0.45-micron filter.

COMPOUND	RRT
1,4-Butanediol	**1.00**
GHB	1.01
GBL	1.14

3.5 NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

Sample preparation:
Use typically 5 to 10 mg of sample for proton NMR and 30 mg for carbon NMR. Dissolve sample in chloroform-d ($CDCl_3$) with the internal reference standard tetramethylsilane (TMS). *Filter all preparation solutions before analysis.

Method Test - NMRS1

Instrument: 400 MHz Nuclear magnetic resonance spectrometer

Probe 5 mm indirect detection gradient NMR probe

Parameters

^{1}H NMR:
Observed frequency: 400.1 MHz
Pulse angle: 30°
Acquisition time: 1.995 s
Acquisition delay: 1.000 s
Spectral window: 6410 Hz
Transmitter power: 57 dB
Variable temperature set @: 25°C
Number of transients: 16

^{13}C NMR:
Observed frequency: 100.6 MHz
Pulse angle: 45°
Acquisition time: 1.202 s
Acquisition delay: 1.000 s
Spectral window: 25062 Hz
Transmitter power: 61 dB
Decoupler: on
Decoupler modulation mode: Waltz
Decoupler modulation frequency: 10100 Hz
Variable temperature set @: 25°C
Number of transients: 1024

Water-d (D_2O) or methanol-d (CD_3OH) may be employed as the solvent using 2,2-dimethyl-2-silylpentane-5-sulfonate (DSS or DDS) as the internal reference standard.

4. SEPARATION TECHNIQUES

Relatively pure samples may be examined by infrared spectroscopy for the identification of 1,4-butanediol either directly or by solvent extraction. 1,4-butanediol is also readily separated from aqueous solutions by a liquid-liquid extraction using methylene chloride or chloroform. This is also possible for solutions with a matrix such as carbonated sodas.

Aqueous samples could alternatively be analyzed by allowing a portion of the sample to evaporate at low heat. Because 1,4-butanediol has a relatively high boiling point of 230°C, heating the sample on a surface up to 100°C is safe. Many of these samples contain sugars, dyes, and other components of the matrix. Therefore, a chloroform extraction is recommended to obtain a clean IR spectrum. (Garcia, Catterton, 2003)

5. QUANTITATIVE DATA

5.1 GAS CHROMATOGRAPHY

Method 7 - 1,4BDGC1

Internal Standard Stock Solution:
1.2 mg/mL dodecane in methanol*.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of 1,4-butanediol at approximately 2.0 mg/mL with methanol*.

Sample Preparation:
Accurately weigh the sample to target a final 1,4-butanediol concentration of approximately 2 mg/mL. Dilute to volume with methanol*.

*Note: Another solvent could be considered when gamma-hydroxybutyrate is present since it reacts with methanol to form the gamma-hydroxy methyl ester of GHB.

Instrument:	HP 5890 or 6890 gas chromatograph operated in split mode with FID
Column:	HP-1 cross-linked methyl siloxane, 15 m x 0.20 mm x 0.33 µm
Carrier gas:	Hydrogen at 2.0 mL/min
Temperatures:	Injector: 180°C Detector: 300°C Oven program: 80°C for 0.5 minute, ramp 40°C per minute to 100°C, hold 1.5 min
Injection Parameters:	Split Ratio = 30:1, 1 µL injected
Typical Retention Time:	1,4-Butanediol: 0.75 min Dodecane: 2.33 min

Linear Range: 1.0 – 4.0 mg/mL

Repeatability: RSD less than 2.5%

Correlation Coefficient: 0.999

Accuracy: Error less than 2%

COMPOUND	RRT	COMPOUND	RRT
1,2-butanediol	0.70	**1,4-butanediol**	**1.0 (0.75 min)**
1,3-butanediol	0.76	gamma-hydroxy methyl ester of GHB	1.14
GBL	0.85		

6. QUALITATIVE DATA

See spectra on the following pages for FT-IR, Mass Spectrometry, Nuclear Magnetic Resonance, and Vapor Phase IR.

7. REFERENCES

Federal Criminal Code and Rules, 2002, 21 U.S.C. ß 802

Federal Criminal Code and Rules, 2002, 21 U.S.C. ß 813

Garcia, A. D., Catterton, A. J., "1,4-Butanediol (BD) Forensic Profile," *Microgram Journal*, Vol. 1, January-June 2003.

Morris, J. A., "Analogs of GHB Part 1: Theoretical Perspective," *Journal of the Clandestine Laboratory Investigating Chemists Association*, Volume 10, Number 2, April 2000.

Morris, J. A., "Analogs of GHB Part 2: Analytical Perspective," *Journal of the Clandestine Laboratory Investigating Chemists Association*, Volume 11, Number 1, January 2001.

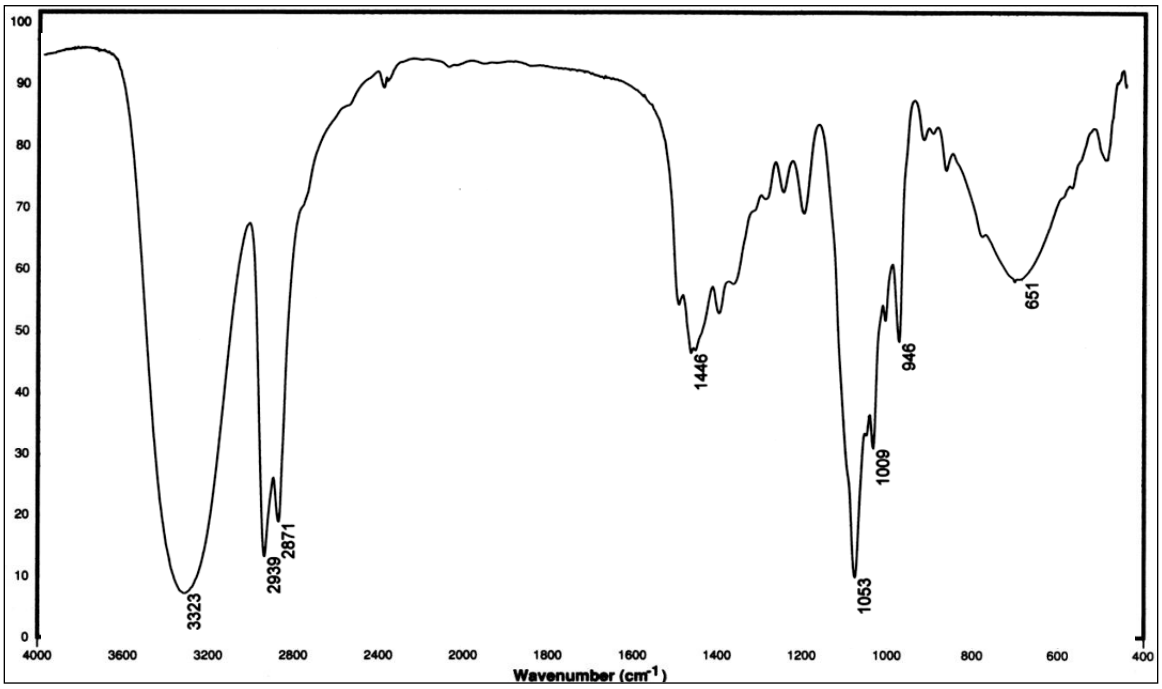
Walker, L., "Maple Syrup and 1,4-Butanediol," *Journal of the Clandestine Laboratory Investigating Chemists Association*, Volume 10, Number 3, July 2000.

Wesley, J. F., "Osmolality-A Novel and Sensitive Tool for Detection of Tampering of Beverages Adulterated with Ethanol, γ-Butyrolactone, and 1,4-Butanediol and for Detection of Dilution-Tampered Demerol Syringes." *Microgram*, Vol. 1, January-June 2003.

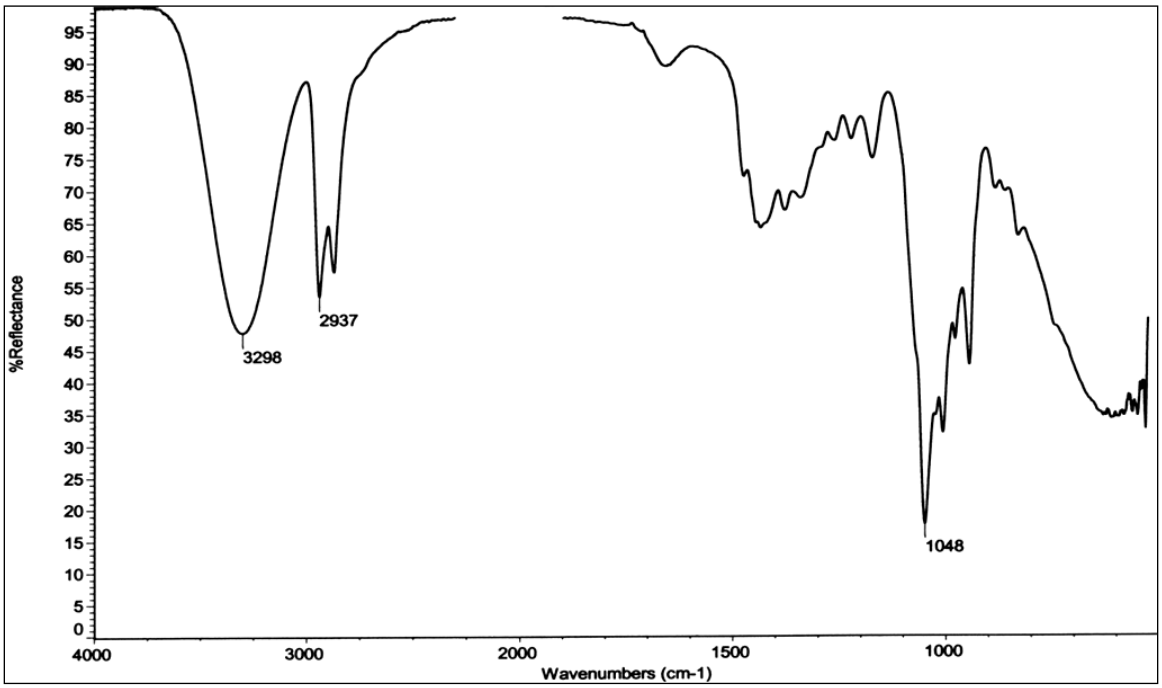
8. ADDITIONAL RESOURCES

Wikipedia

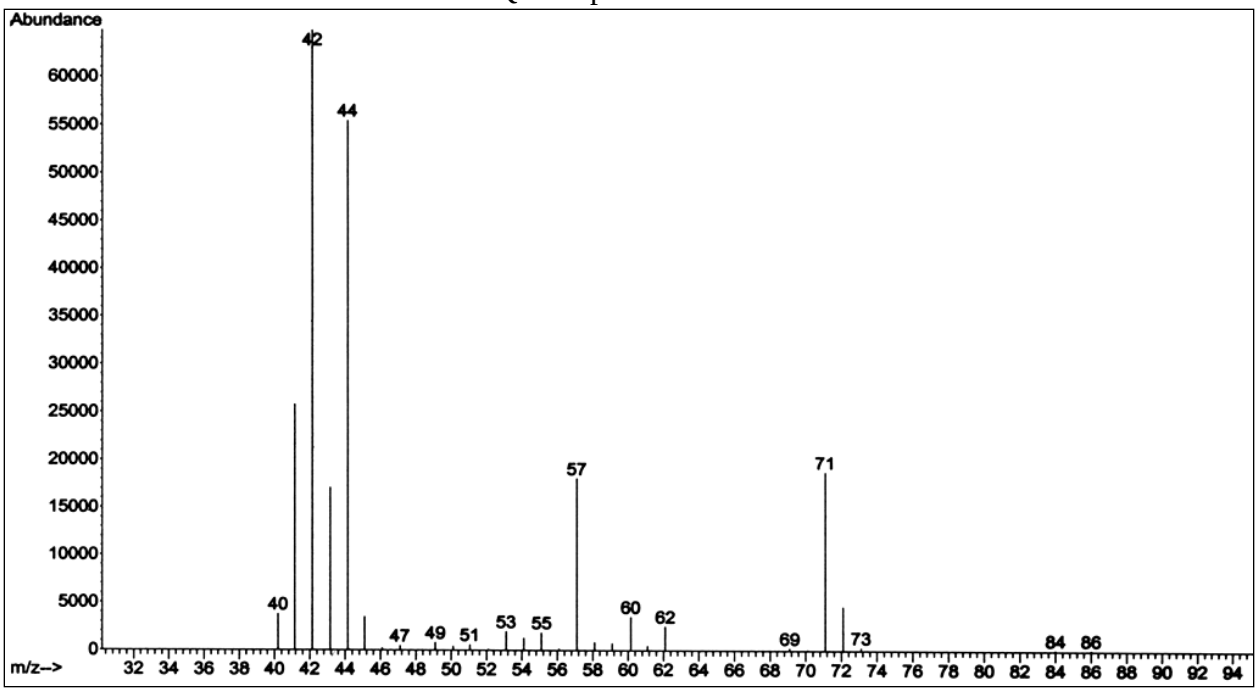
Transmission IR: 1,4-Butanediol, sample neat between KBr disks
16 scans, 4.0 cm^{-1} resolution



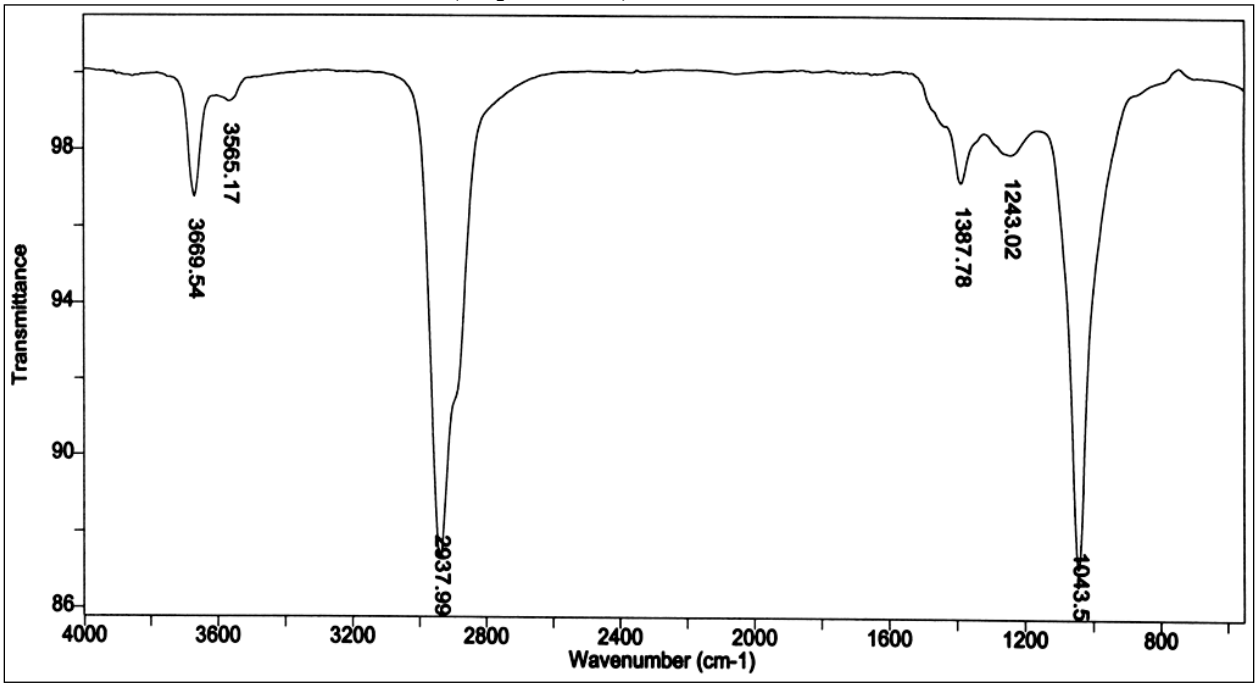
IR (ATR): 1,4-Butanediol, (3-bounce, diamond device)
16 scans, 4.0 cm^{-1} resolution



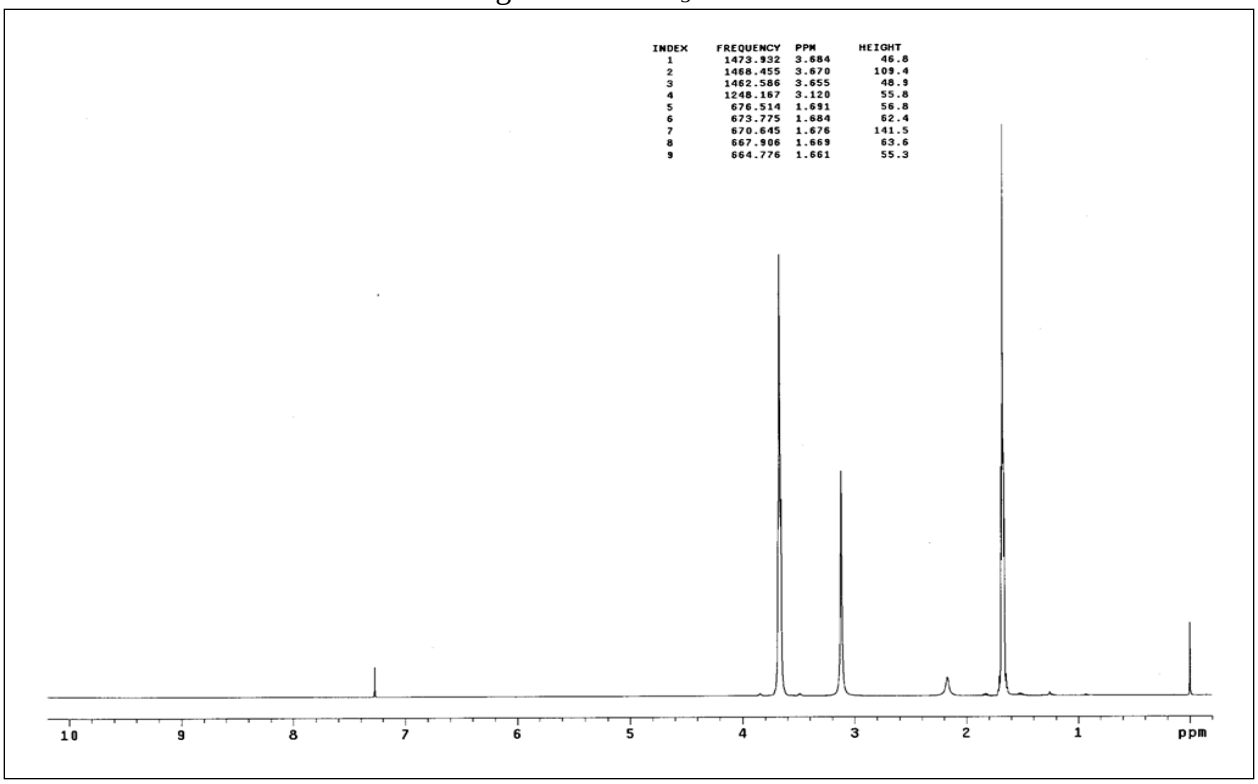
Electron Ionization MS: 1,4-Butanediol
Quadrupole Detector



IR (Vapor Phase): 1,4-Butanediol



Proton NMR: 1,4-Butanediol (400 MHz)
10 mg/mL in $CDCl_3$ with TMS



Carbon NMR: 1,4-Butanediol (100 MHz)
50 mg/mL in $CDCl_3$ with TMS