UNIVERSITY OF CHEMISTRY AND TECHNOLOGY PRAGUE

Central laboratories – Laboratory of mass spectrometry

Analysis of Gamma butyrolactone for registration according REACH

Report no. JK 75/17

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Description of samples and testing

For analysis sample of Gamma butyrolactone was delivered in 100 mL polyethylene bottle. Delivered sample was analyzed by three different analytical methods for registration according REACH. Used analytical methods were: nuclear magnetic resonance (NMR) spectroscopy, infrared (IR) spectroscopy and gas chromatography with mass spectrometry detection (GC-MSD).

Figure 1: Picture of delivered sample.

NMR spectroscopy

Sample name: Gama butyro lactone (GBL)

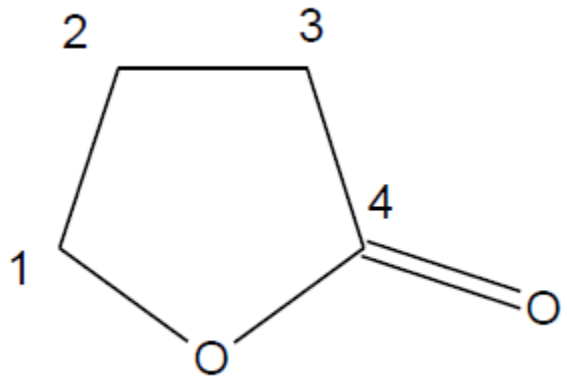
Sample preparation: 40 mg of GBL was dissolved in 0.5 ml CDCl3 (99.9 % D, Sigma-Aldrich)

Experimental conditions:

1H-experiment: Bruker DRX 500, resonance frequency 500.13 MHz, sweep width 6053.269 Hz, pulse length 12.8 µs, 16 scans, chemical shifts were referenced to TMS (0 ppm)

13C-experiment: Bruker DRX 500, resonance frequency 125.773 MHz, sweep width 34090.910 Hz, pulse length 10.5 µs, 1000 scans, broad band decoupling of 1H (Waltz 16), chemical shifts were referenced to TMS (0 ppm)

Results:



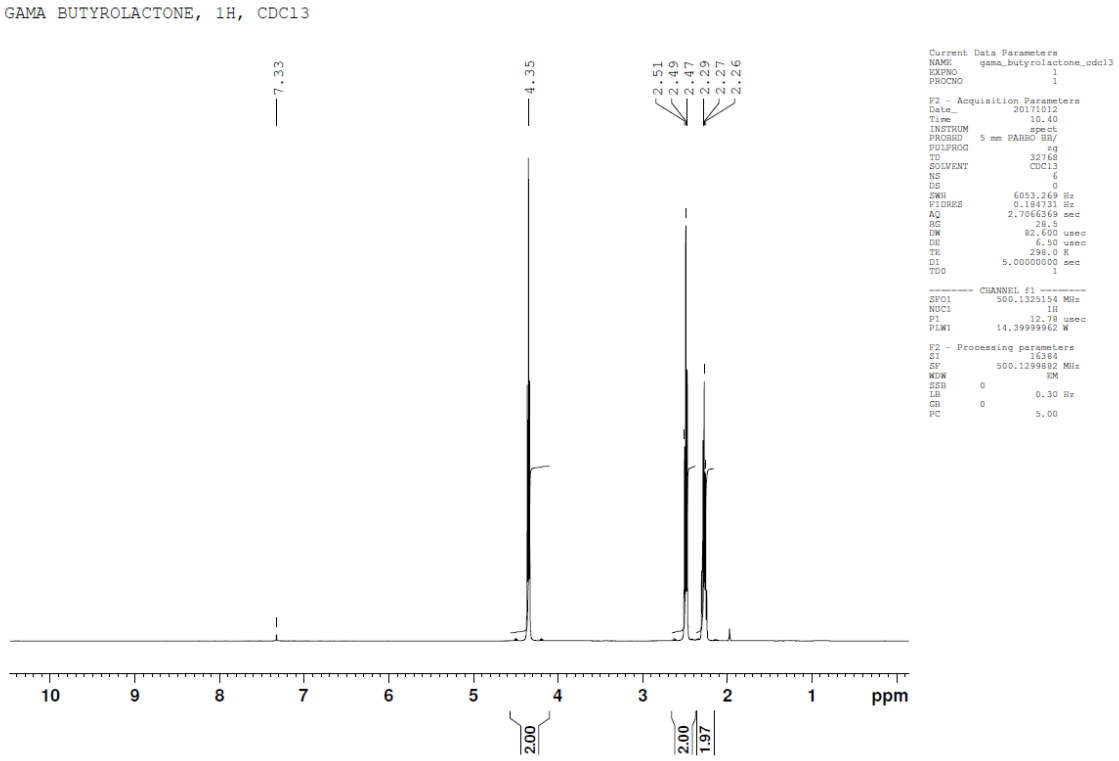


Figure 2: 1H spectrum: 2.27 ppm, tt, 2H (H2), 2.49 ppm, t, 2H (H3), 4.35 ppm, t, 2H (H1)

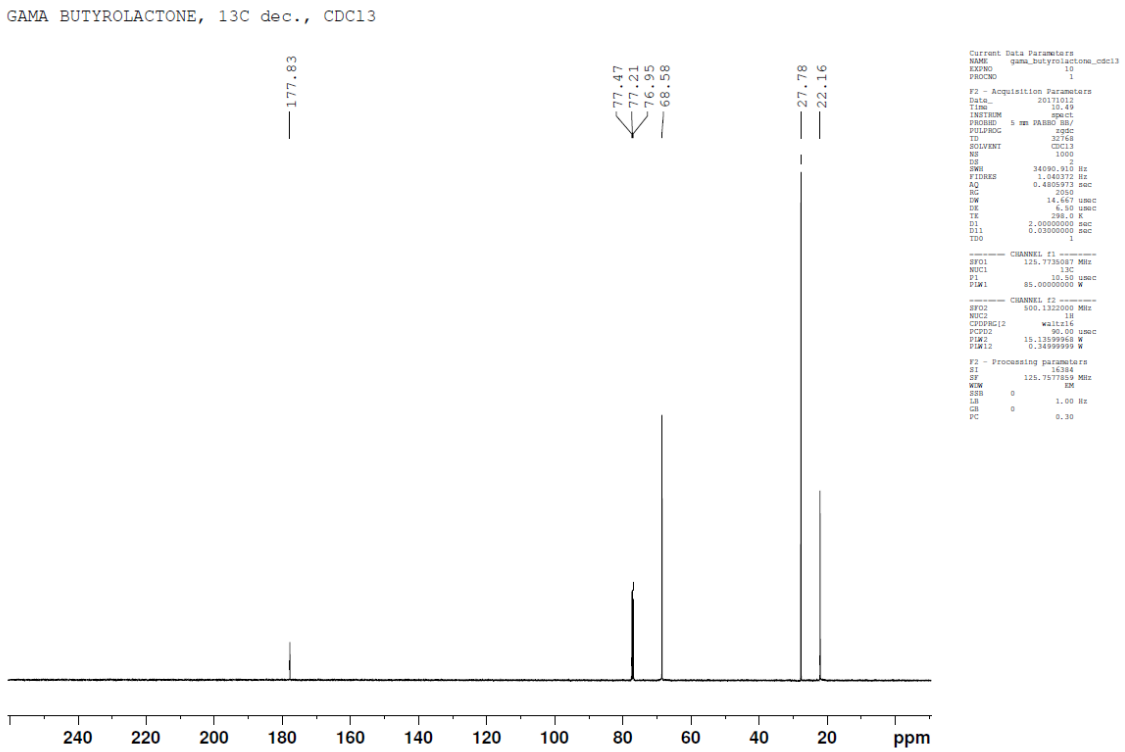


Figure 3: ^{13}C spectrum: 22.16 ppm, (C2), 27.78 ppm, (C3), 68.58 ppm, (C1), 177.83 ppm (C4)

Conclusion:

NMR spectra confirm the identity of the delivered Gama butyrolactone.

IR spectroscopy

Sample name: Gama butyro lactone (GBL)

Experimental conditions:

DATA COLLECTION INFORMATION
Number of sample scans: 64
Collection length: 94,47 sec
Resolution: 4,000
Levels of zero filling: 0
Number of scan points: 16672
Number of FFT points: 16384
Laser frequency: 15798,3 cm-1
Interferogram peak position: 8192
Apodization: Happ-Genzel
Phase correction: Mertz
Number of background scans: 64
Background gain: 4,0

DATA DESCRIPTION
Number of points: 1868
X-axis: Wavenumbers (cm-1)
Y-axis: Absorbance
First X value: 399,1989
Last X value: 3999,7031
Data spacing: 1,928497

SPECTROMETER DESCRIPTION
Spectrometer: Nicolet 6700
Source: IR
Detector: DTGS KBr
Smart Accessory ID: Unknown
Beamsplitter: KBr
Sample spacing: 1,0000
Digitizer bits: 24
Optical velocity: 0,4747
Aperture: 150,00
Sample gain: 4,0
High pass filter: 20,0000
Low pass filter: 11000,0000

COLLECTION ERRORS
Errors During Sample Collection
Number of Rejected Scans:0
Error Types:
None
Errors During Background Collection
Number of Rejected Scans:0
Error Types:
None

DATA PROCESSING HISTORY
Collect Sample
Background collected on Wed Oct 25 10:46:34 2017 (GMT+02:00)
Final format: %Reflectance
Resolution: 4,000 from 399,1989 to 3999,7031
Bench Serial Number:AHR0700926
Sample compartment: Main

Title changed on Wed Oct 25 10:55:57 2017 (GMT+02:00)
Previous Title: Wed Oct 25 10:51:30 2017 (GMT+02:00)
Converted to absorbance y-axis units on Mon Oct 30 17:12:48 2017 (GMT+01:00)
Straight Line on Mon Oct 30 17:13:18 2017 (GMT+01:00)
Data format: Absorbance
From 3748,9851 to 3675,9048

Straight Line on Mon Oct 30 17:13:31 2017 (GMT+01:00)
Data format: Absorbance
From 3679,0820 to 3590,1147

Straight Line on Mon Oct 30 17:13:41 2017 (GMT+01:00)
Data format: Absorbance
From 2404,9426 to 1937,8641

ATR Correction on Mon Oct 30 17:15:44 2017 (GMT+01:00)

Title changed on Mon Oct 30 17:19:59 2017 (GMT+01:00)
Previous Title: *Gamma butyrolactone, GladiATR
Title changed on Mon Oct 30 17:20:11 2017 (GMT+01:00)
Previous Title: Gamma butyrolactone, GladiATR
SYSTEM STATUS

Instrument Status	Pass	Date Tested:	Wed; 25 Oct 2017
Scheduled Maintenance	Disabled		
System Suitability	Disabled		

CURRENT DIGITAL SIGNATURE STATUS
Not currently signed.

EXPERIMENT INFORMATION:
Experiment filename: c:\my documents\omnic\param\GladiATR.exp
Experiment title: GladiATR
Experiment accessory:

SPECTRAL QUALITY RESULTS:
Background and spectrum resolution test passed
Background and spectrum range test passed

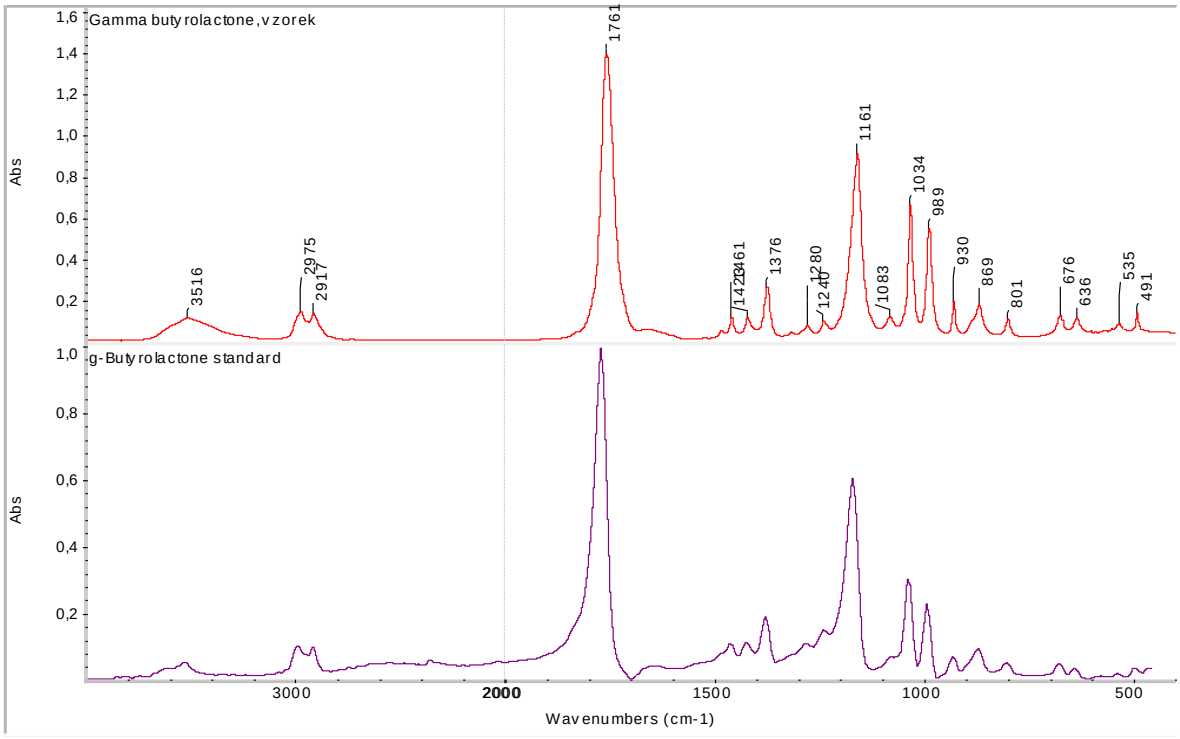


Figure 4: Comparison of measured IR Spectrum and Standard IR spectrum from the Library

The spectrum in Figure 4 is compared to the standard spectrum in the Hummel polymers and additives library (Dieter O. Hummel, Institute for Physical Chemistry of Universität Koln, and is identical to the standard.) Fine displacements of the positions of the absorbent tops of the absorbent bands and the intensity ratio of the absorbent bands between the standard and the sample are due to a measurement error due to a different measurement technique.

Conclusion:

IR spectroscopy confirmed the composition of the sample and did not find any other sample component except gamma butyrolactone.

GC-MSD

Sample name: Gama butyro lactone (GBL)

Sample preparation: 4 μl of GBL was dissolved in 4 ml of acetonitrile (LC-MS grade, Honeywell)

Experimental conditions:

Instruments:
GC: Trace GC Ultra (Thermo Scientific, USA)
MS: single quadrupole ISQ (Thermo Scientific, USA)
GC column: DB-5MS UI, 60m x 0.32mm x 1.0μm (J&W Agilent, USA)

GC-MS Conditions:

GC: Injection port: split/splitless, operated in split mode, split ratio 1:30
Temperature of injection port: 250^{o}C
Carrier gas: Helium (purity 5.5), constant flow @ 1.5mL/min
Thermal program of separation: 40^{o}C (5min) –> 15^{o}C/min –> 290^{o}C (1 min)

MS: Ionization: EI @ +70 eV
Temperature of Ion Source 200^{o}C
Scan: 20-450 a.m.u. @ 5 scans/s

Results:

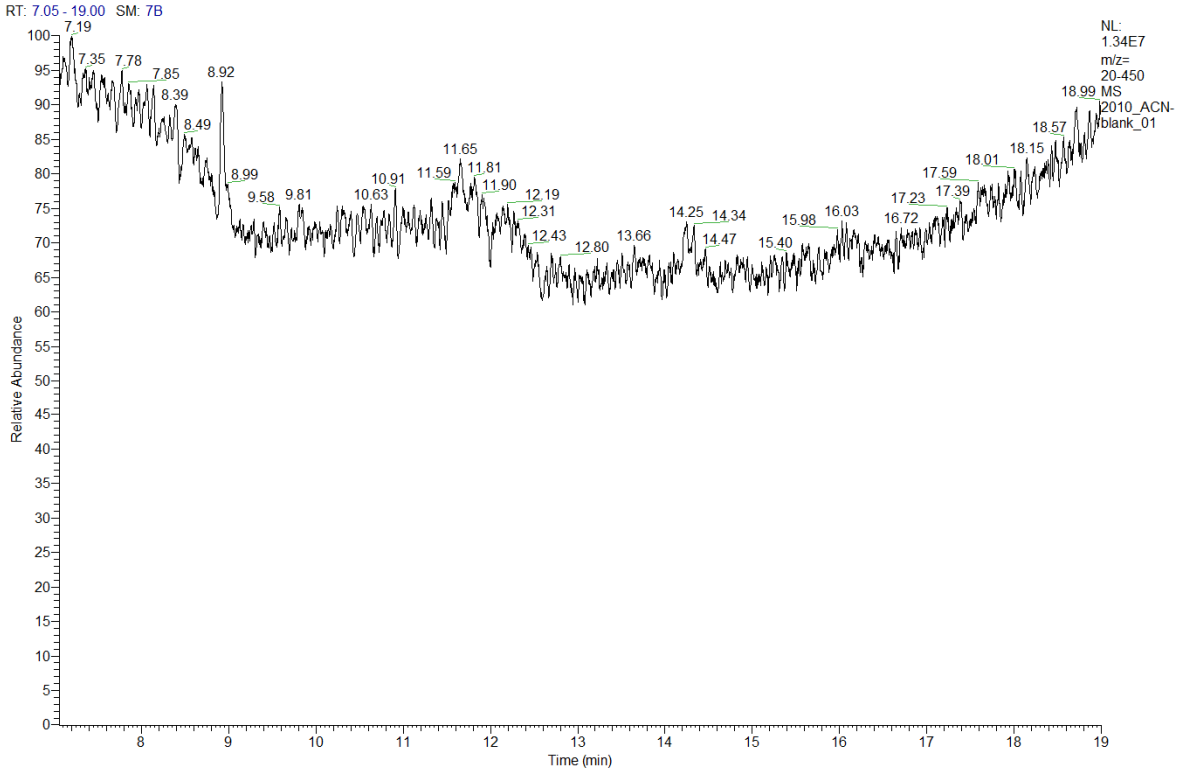


Figure 5. TIC chromatogram of GC-MS analysis of blank (acetonitril)

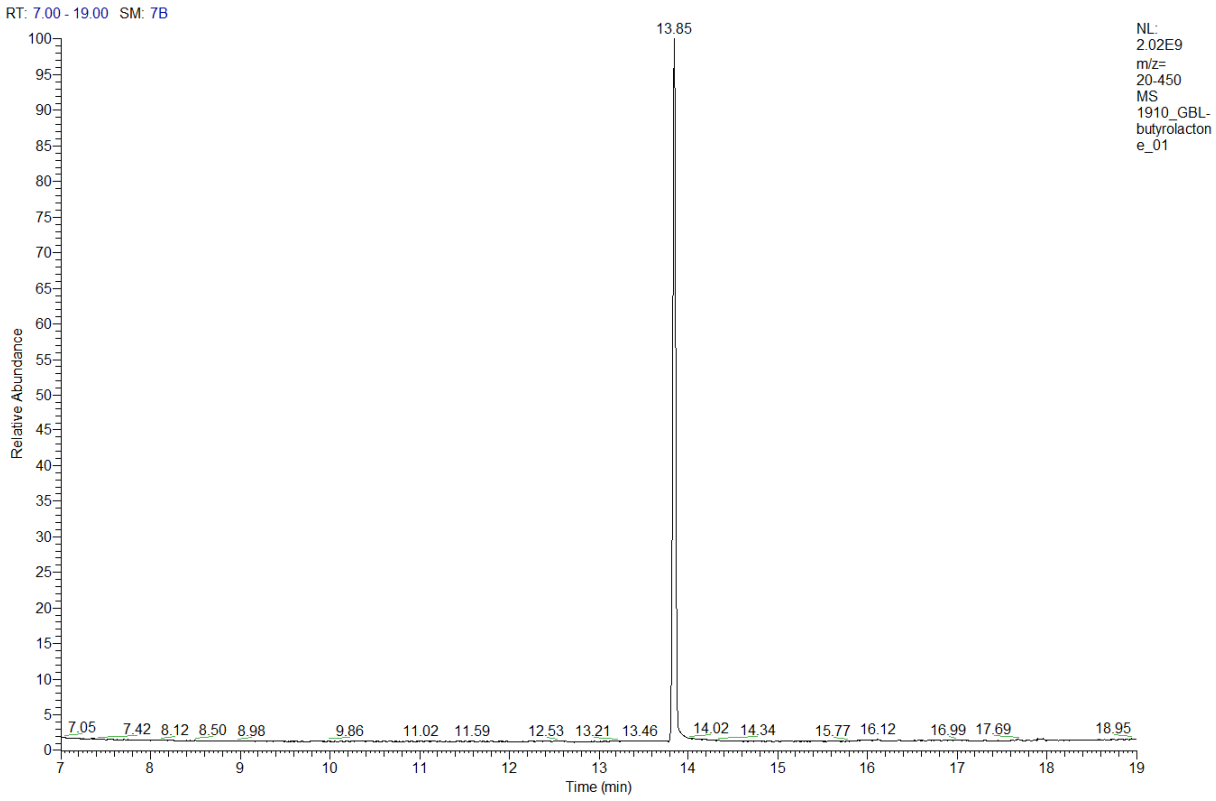


Figure 6. TIC chromatogram of GC-MS analysis of gamma butyrolactone. Peak at RT 13.85 min gamma butyrolactone.

A measured MS spectrum of determined compound was compared with Mass Spectral Library NIST 14.

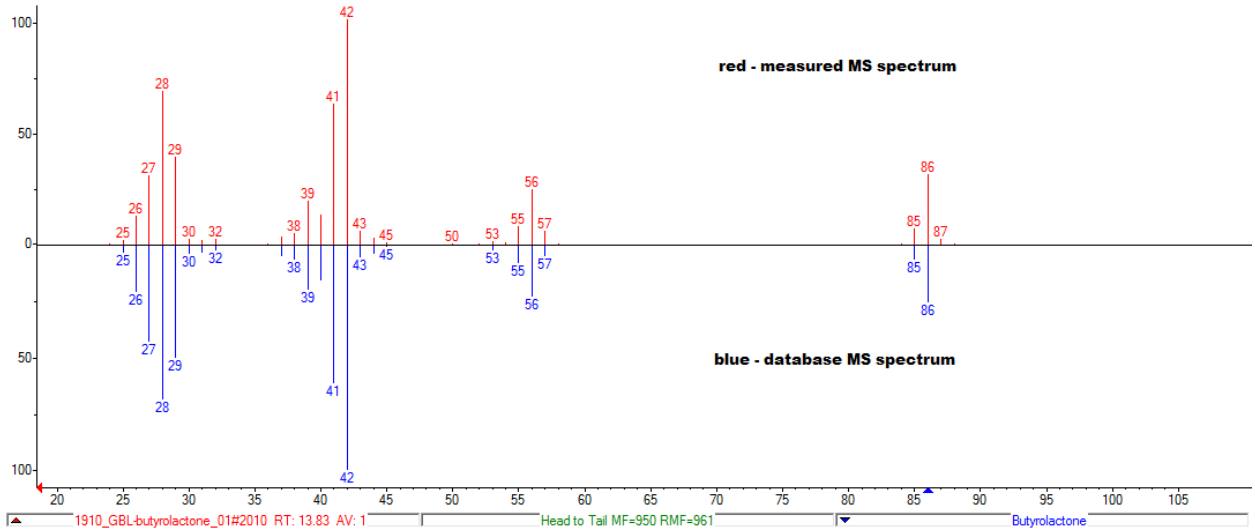


Figure 7. Comparison of measured MS spectrum at RT 13.85 min and database MS spectrum of gamma butyrolactone.

Table 1. List of identified compounds in sample of gamma butyrolactone

RT [min]	Compound	Minimal content [%]	Maximal content [%]	Typical content [%]	Molecular formula	CAS number
13.85	Gamma butyrolactone	98	100	> 99	$C_4H_6O_2$	96-48-0

Conclusion:

GC-MS analysis confirms the identity of the delivered gamma butyrolactone.

Prague, 31.10.2017

MSc. Jiří Kosina

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