



## 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 CDCl<sub>3</sub> (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  $\mu$ s, 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  $\mu$ s, 1000 scans, broad band decoupling of 1H (Waltz 16), chemical shifts were referenced to TMS (0 ppm)

# Results:

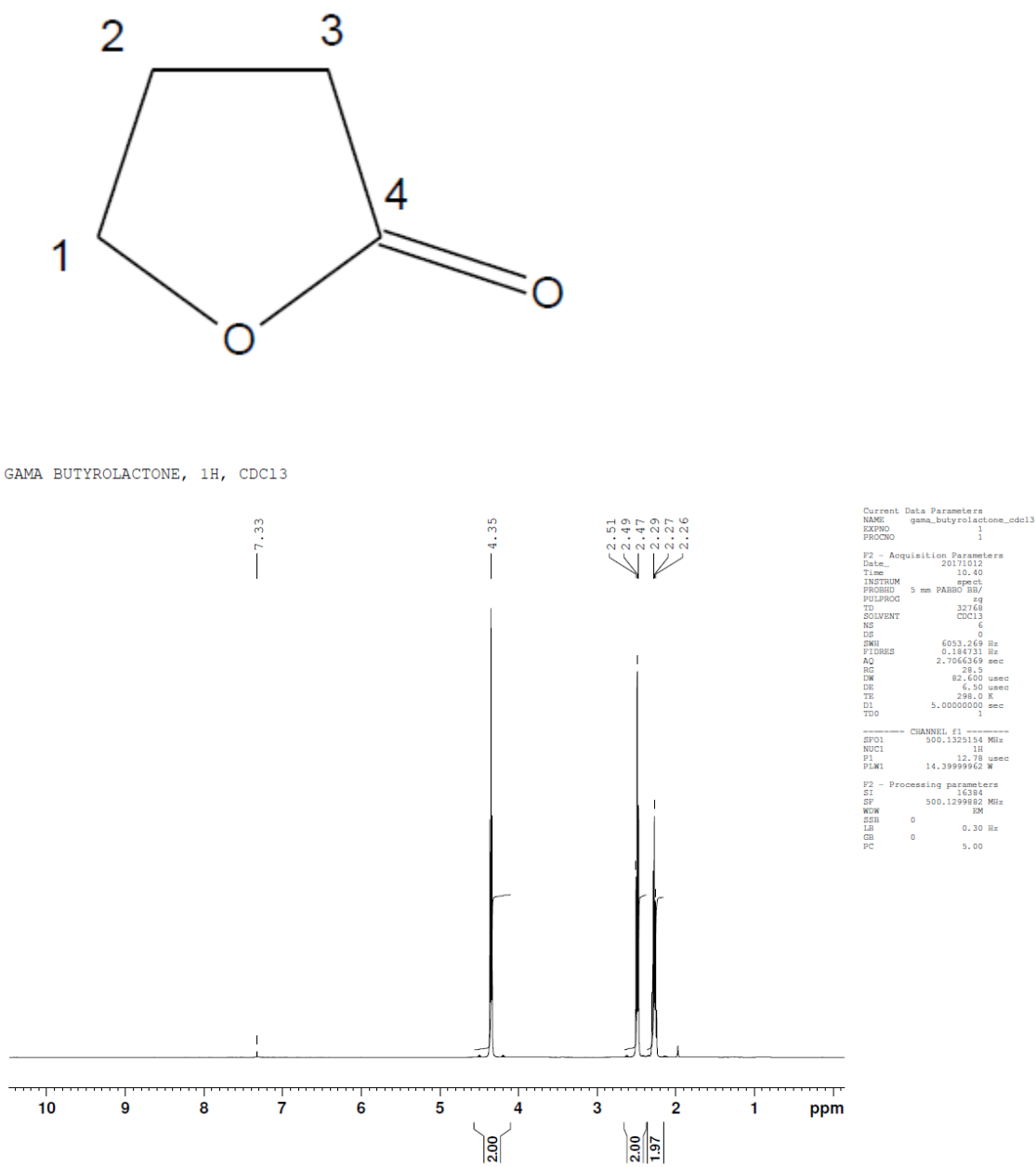
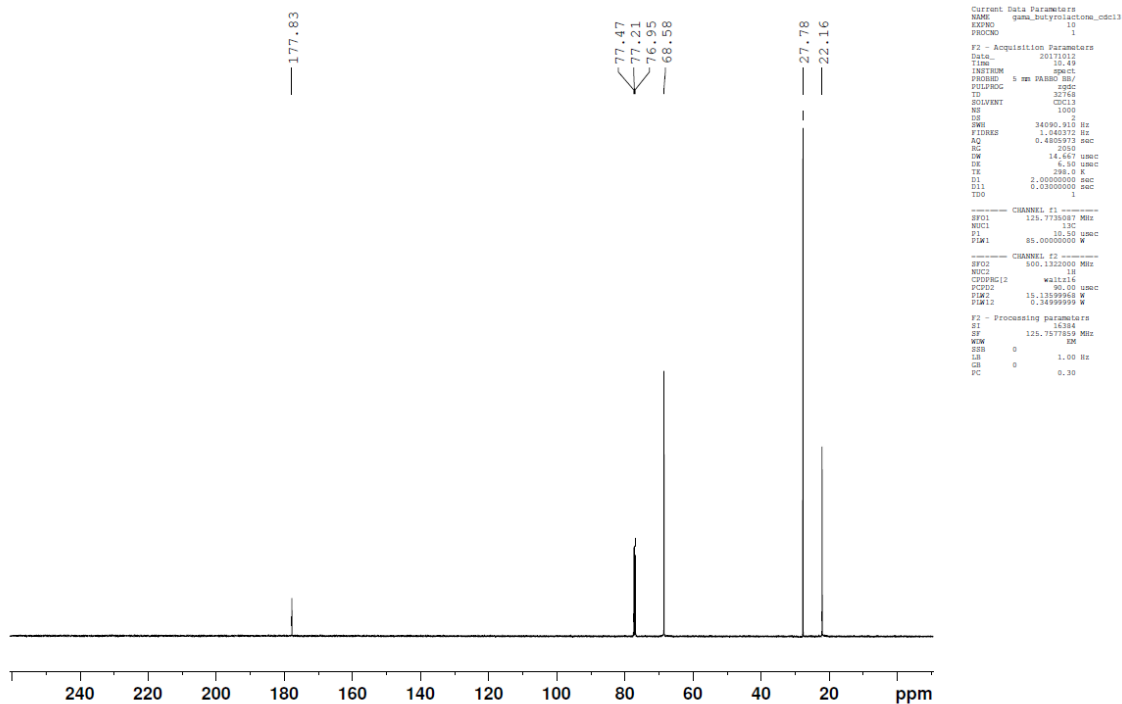


Figure 2: <sup>1</sup>H spectrum: 2.27 ppm, tt, 2H (H2), 2.49 ppm, t, 2H (H3), 4.35 ppm, t, 2H (H1)

GAMA BUTYROLACTONE, 13C dec., CDC13



**Figure 3: <sup>13</sup>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<sup>-1</sup>
- 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

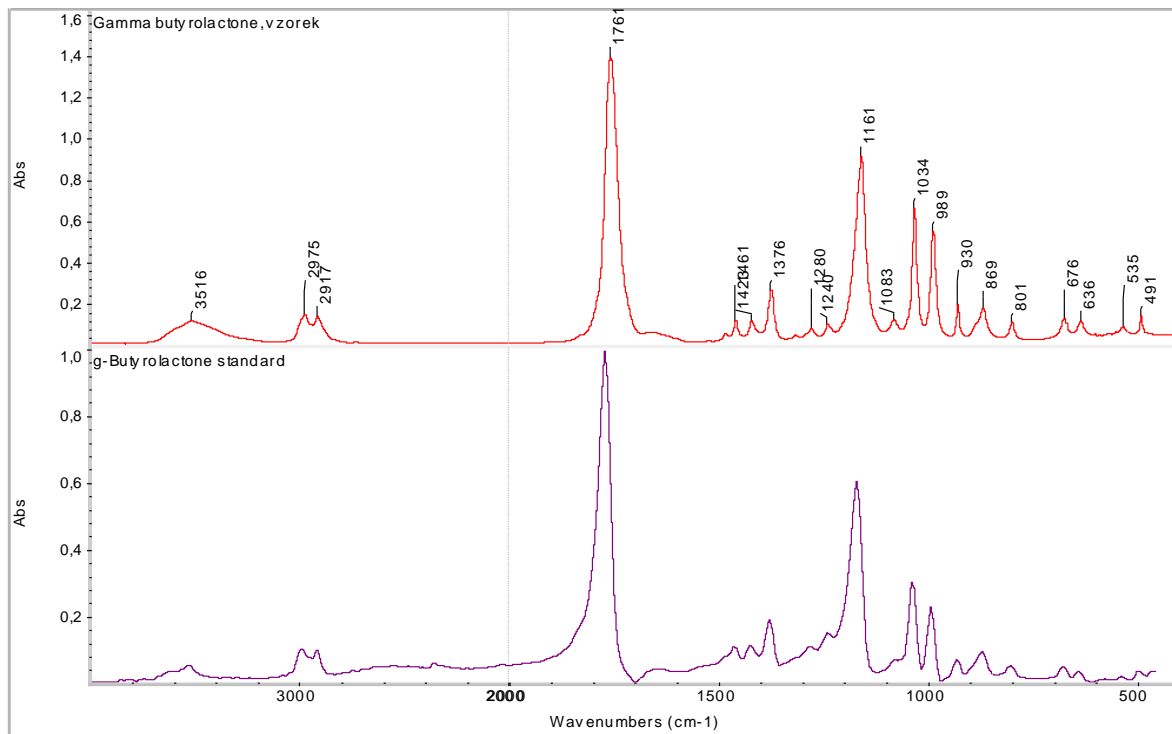


Figure 4: Comparison of measured IR Spectrum and Standard IR spectrum from the Library

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  $\mu$ l 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 1.0 $\mu$ m (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°C

Carrier gas: Helium (purity 5.5), constant flow @ 1.5mL/min

Thermal program of separation: 40°C (5min)  $\rightarrow$  15°C/min  $\rightarrow$  290°C (1 min)

MS: Ionization: EI @ +70 eV

Temperature of Ion Source 200°C

Scan: 20-450 a.m.u. @ 5 scans/s

## Results:

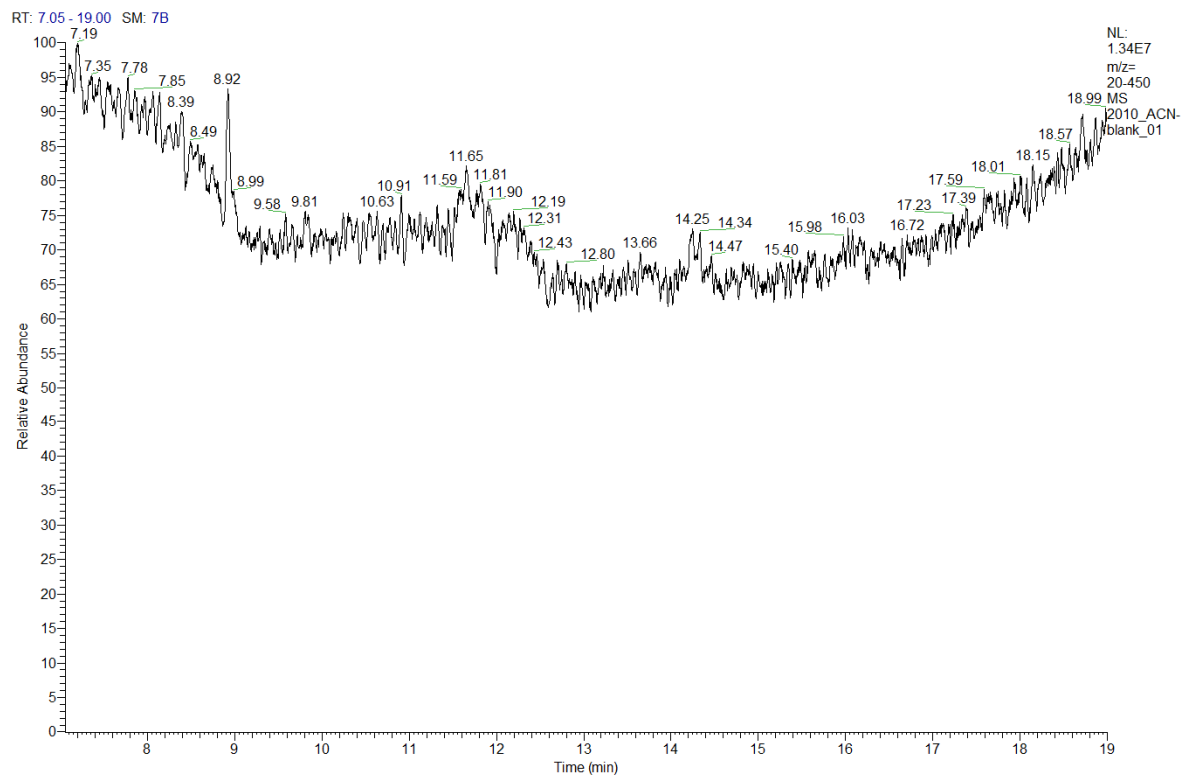


Figure 5. TIC chromatogram of GC-MS analysis of blank (acetonitril)

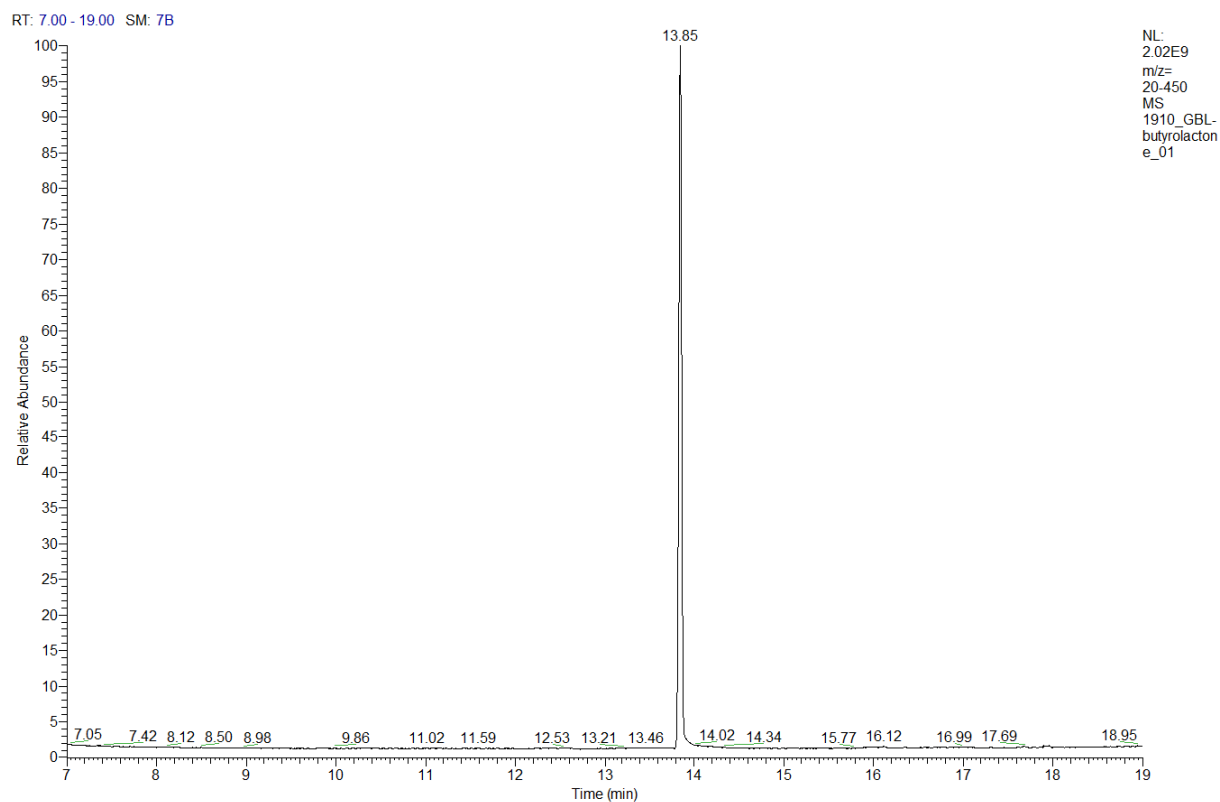


Figure 6. TIC chromatogram of GC-MS analysis of gamma butyrolactone. Peak at RT 13.85 min gamma butyrolactone.



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

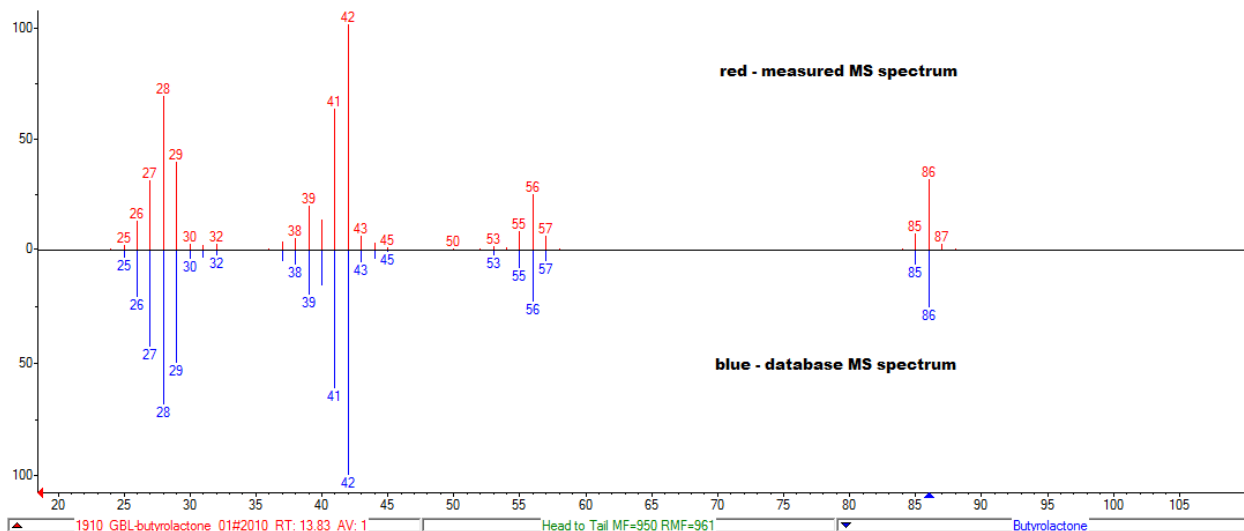


Figure 7. Comparison of measured MS spectrum at RT 13.85 min and database MS spectrum of gamma butyrolactone.

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 <sub>4</sub> H <sub>6</sub> O <sub>2</sub>	96-48-0

### Conclusion:

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

Prague, 31.10.2017

*Jiří Kosina*

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