Certificate of Analysis

Reference Material - Primary Standard

Product Name: γ–Butyrolactone (GBL) 1.0 mg/ml in Acetonitrile

Catalogue Number: LGCAMP1275.51-11

Lot Number: 11647

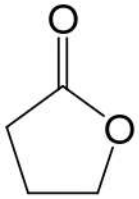
CAS Number: 96-48-0

Molecular Formula: $C_4H_6O_2$

Molecular Weight: 86.09

Solvent: Acetonitrile

Volume per Ampoule: Not less than 1 ml [1]



Long-term Storage: 2 to 8 °C, dark

Expiry Date: February-2017

Intended Use: The primary aim of this material is for identification, calibration and quantification.

Component	Concentration ("as is")	Uncertainty
see product name	1.000 mg/ml [2]	U = 0.004 mg/ml [3]
Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and Guide 34 at the about 95 % level of confidence using a coverage factor of k = 2 and has been calculated by statistical analysis of our production system and incorporates uncertainty of the purity, material density and balance and weighing technique. Concentration based on material weighings and material purity factor (assay of the neat material).		

The solution's concentration and homogeneity are verified by independent method.

LGC certifies that this standard meets the specification stated in this certificate and warrants this product to meet the stated acceptance criteria through the retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.

Release Date:
Luckenwalde, April 2012

Signed:

Dr. Sabine Schröder
Unit for Reference Materials

[1] Ampoules are overfilled to ensure a minimum 1 ml volume fill. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.

[2] The value is based on the results of analytical techniques, which calibration and verification was carried out with standards traceable to SI-units. The value is expressed on an "as is" basis.
The concentration with its uncertainty is valid in the range between 19 °C and 25 °C.
The identity is verified by data from international scientific literature.
Gravimetrically prepared using qualified balances calibrated annually by accredited calibration service. Calibration verification performed daily prior to use utilizing weights traceable to SI via other mass standards.

[3] The uncertainty "U" is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM). It is corresponding to a level of confidence of about 95 %. Standard uncertainties are indicated with "u".

Verification of Concentration and Homogeneity				
Lot Number	Verified Concentration (mg/ml)		% RSD - Homogeneity	
	Result	*Acceptance Criteria*	*Result*	*Acceptance Criteria*
11647	0.987	± 3 %	1.844	≤ 3 %
Concentration verified by GCMS				

Solution Standard Assay Parameters				**External Calibration (100 % amount)**	
Analysis Method	GCMS				
Column:	HP-5MS, 30 m x 0.25 mm x 0.25 µm			Number of Measurements:	6
Injector:	220 °C				
Flow:	1.5 ml/min				
Oven Program:	Rate	°C	Time		
		50	5 min		
	40 °C/min	300	10 min (run time 21.25 min)		
Detector:	280 °C				

Neat Material Data	
Product Name:	γ–Butyrolactone (GBL)
CAS Number:	96-48-0
Molecular Formula:	$C_4H_6O_2$
Molecular Weight:	86.09
Compound Lot:	6555

Test	**Method**	**Result**
^{1}H-NMR Spectrum*	SOP 06-053	conform / complies to structure
IR Spectrum*	SOP 06-036	conform / complies to structure
Mass Spectrum (EI)*	SOP 06-022	conform / complies to structure
Assay by quantitative NMR (as is)*	**Quant. NMR**	**99.58 %**
The expanded uncertainty according to the assay is U = 0.41 % (about 95 % level of confidence using a coverage factor of k = 2).		

*: Validated method performed by ISO/IEC 17025 accredited testing lab

The assay of the neat material is verified by the 100 % method using GC, corrected with water (KFT) and residual solvents.

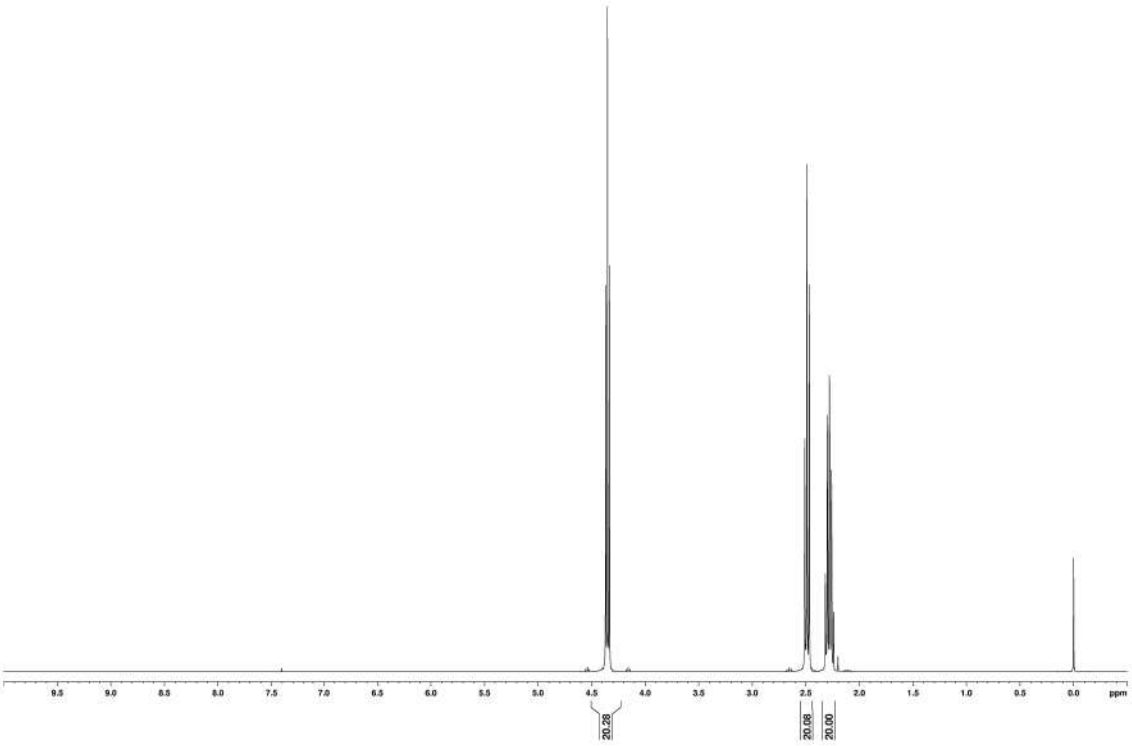
I. Identity

The identity of the reference substance (neat material) was established by the following analyses.

Ia. ^{1}H-NMR Spectrum

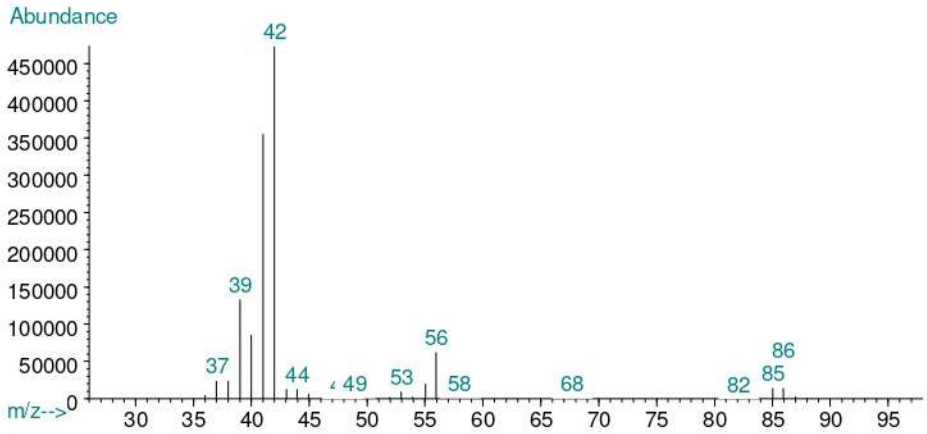
Conditions: 400 MHz, $CDCl_3$

The structure is confirmed with the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

Method: EI, 70eV, detector temperature: 280 °C

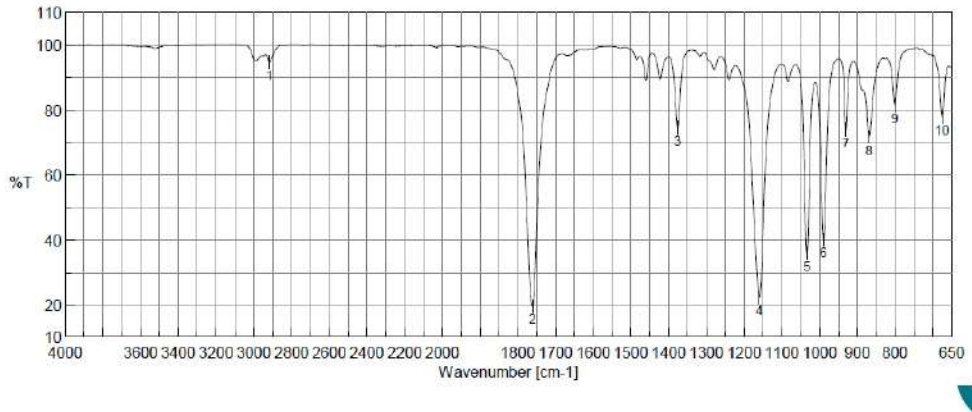


m/z	fragments
86	[M]
56	[M – CH_2O]
42	[M – CO_2]

The signals of the mass spectrum and their interpretation are consistent with the structural formula.

Ic. IR Spectrum

Method: attenuated total reflection fourier transform infrared (ATR-FTIR) spectroscopy



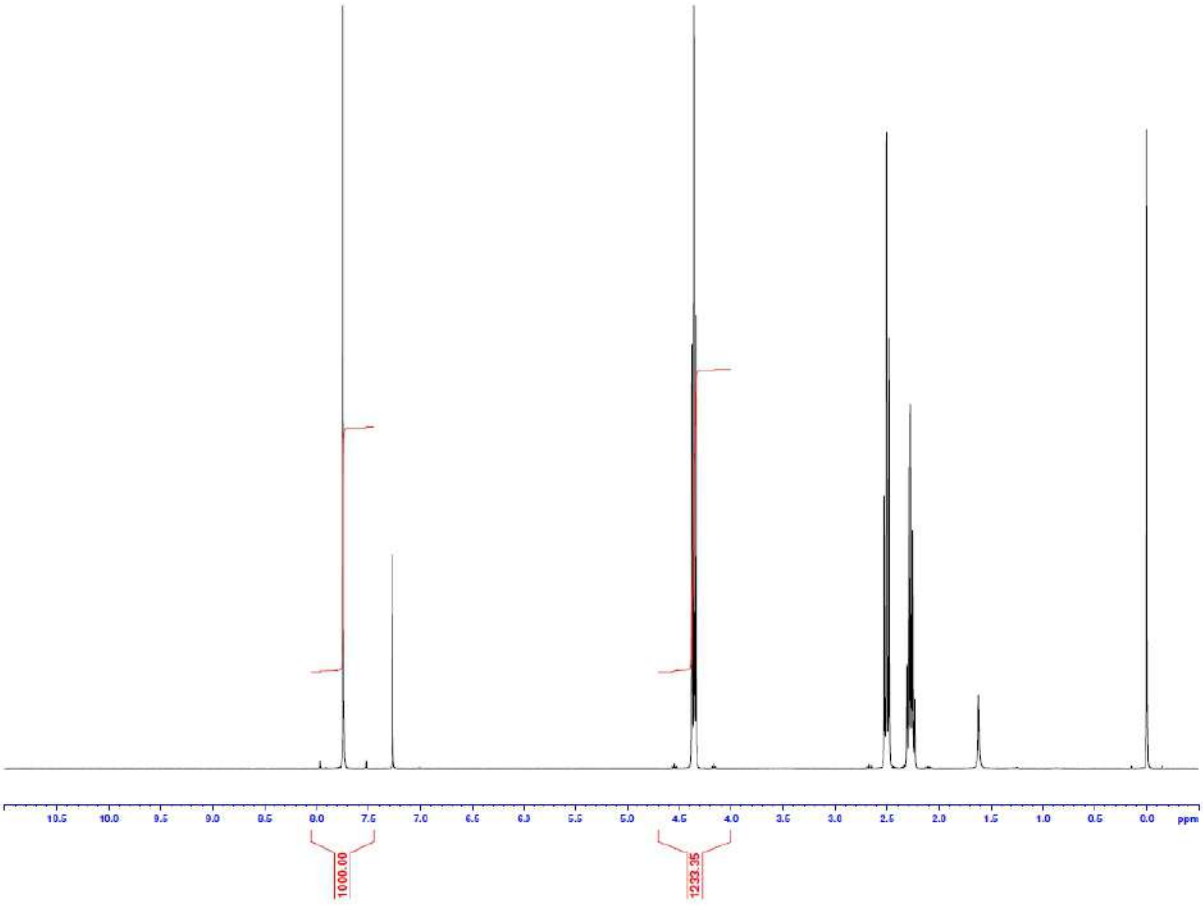
Result of Peak Picking

No.	Position	Intensity
1	2915.84	94.6627
2	1761.65	19.4674
3	1375.96	74.4384
4	1159.97	22.1719
5	1033.66	35.7651
6	989.304	39.9664
7	929.521	73.8955
8	868.774	71.5687
9	800.314	81.2811
10	674.963	77.7589

The signals of the IR spectrum and their interpretation are consistent with the structural formula.

II. Assay by quantitative NMR spectroscopy

The assay of the reference substance was established by quantitative NMR spectroscopy using $CDCl_3$ as the solvent and with 2,3,5,6-Tetrachloro-1-nitrobenzene (certified reference material, signal 7.45 – 8.05 ppm, 1 H) as internal standard.



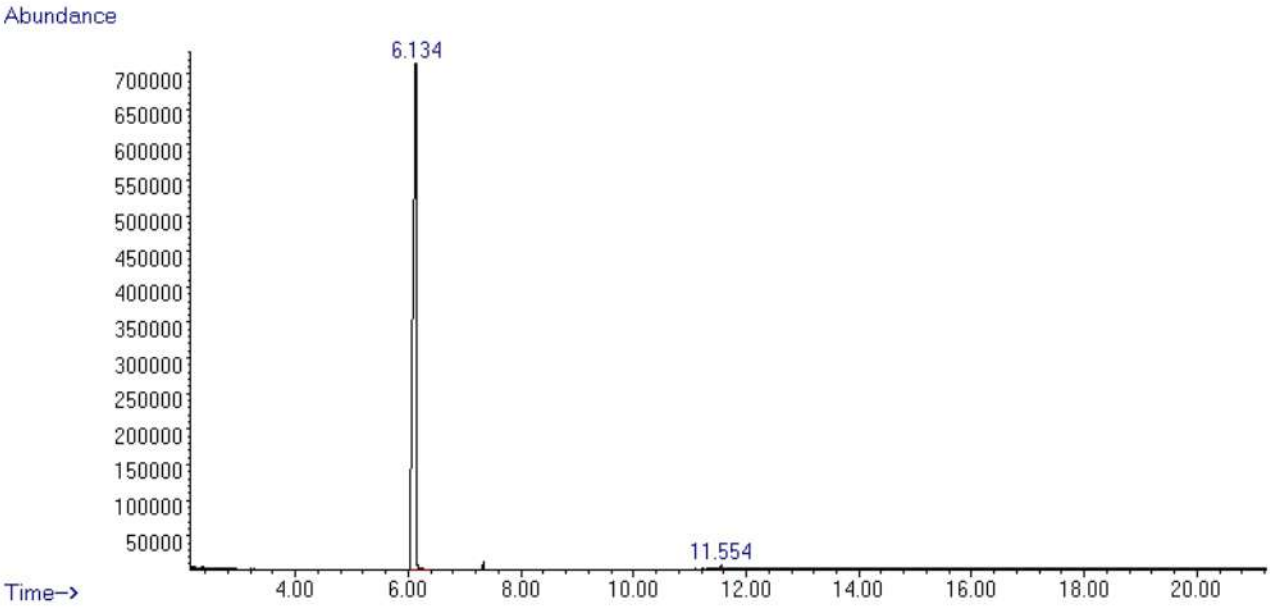
Results:

Average	99.58 %
Number of results	n=6
Uncertainty U (expanded)	0.41 %

III. Purity

IIIa. Gas Chromatography (GC)

The purity of the reference substance (neat material) was analysed by gas chromatography (GC).



Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	6.134	27565953	99.876
2	11.554	34335	0.124
Totals		27600288	100.00

For the calculation the air peaks were ignored. The content of the analyte was determined as ratio of the peak area of the analyte and the cumulative areas of the purities, added up to 100 %.

GC Conditions:

Column:	Injector and Flow:	Oven Program:	Detector:
HP-5MS	Split 20:1, 220 °C	Initial Temp.: 50 °C for 5 min	EI, 70 eV
30 m x 0.25 mm x 0.25 µm	Helium 1.50 ml/min	Heating Rate: 40 °C/min	35 to 550 amu
		Final Temp.: 300 °C for 10 min	280 °C

Results:

Arithmetic mean (n=3) 99.88 %

IIIb. Water Content

Method: coulometric Karl Fischer titration

Results:

Arithmetic mean (n=3) 0.18 % (mass fraction)

IIIc. Residual Solvents

Method: ^{1}H-NMR

No significant amounts of residual solvents were detected (< 0.05 %).

IV. Stability and Homogeneity

Accelerated stability studies indicate no significant instability. The given validity period is based on this data. This is backed up by historical data over the range of several years for the neat substance. Homogeneity assured by validated process of preparation (incl. ampoulation), verified by homogeneity testing (GCMS).

V. Further Information

General

For laboratory use only. Not suitable for human or animal consumption.

This material conforms to the characteristics of a primary standard as described within ISO Guide 30 (Terms and definitions used in connection with reference materials).
The certified values quoted in this certificate are LGC's best estimate of the true values within the stated uncertainties and based on the techniques described in this certificate.

Handling of the RM

Before usage of the RM, it should be allowed to warm to room temperature. The concentration with its uncertainty is guaranteed in the range between 19 °C and 25 °C. The uncertainty accounts for the temperature-dependent density in this range.

Quality Control Assessment

The product quality is controlled by regularly performed quality control tests (retests).

Revision	Date	Reason for Revision
00	April 2012	Release of the Lot – initial version
01	June 2013	Copyright added