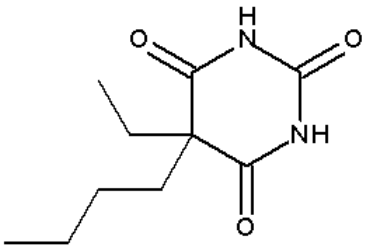
Certificate of Analysis

Reference Standard

Butobarbital



Molecular Formula:	$C_{10}H_{16}N_2O_3$
Molecular Weight:	212.25
CAS Number:	77-28-1

Catalogue Number:	LGCFOR1625.00
Lot Number:	78572
Long-term Storage:	2 to 8 °C, dark
Appearance:	white solid
Melting Point:	123 °C
Assay 'as is':	99.6 %

Date of shipment: **2016-May-20**

This certificate is valid one year from the date of shipment provided the substance is stored under the recommended conditions unopened in the original container.

LGC GmbH, Im Biotechnologiepark, TGZ II, D-14943 Luckenwalde, Germany

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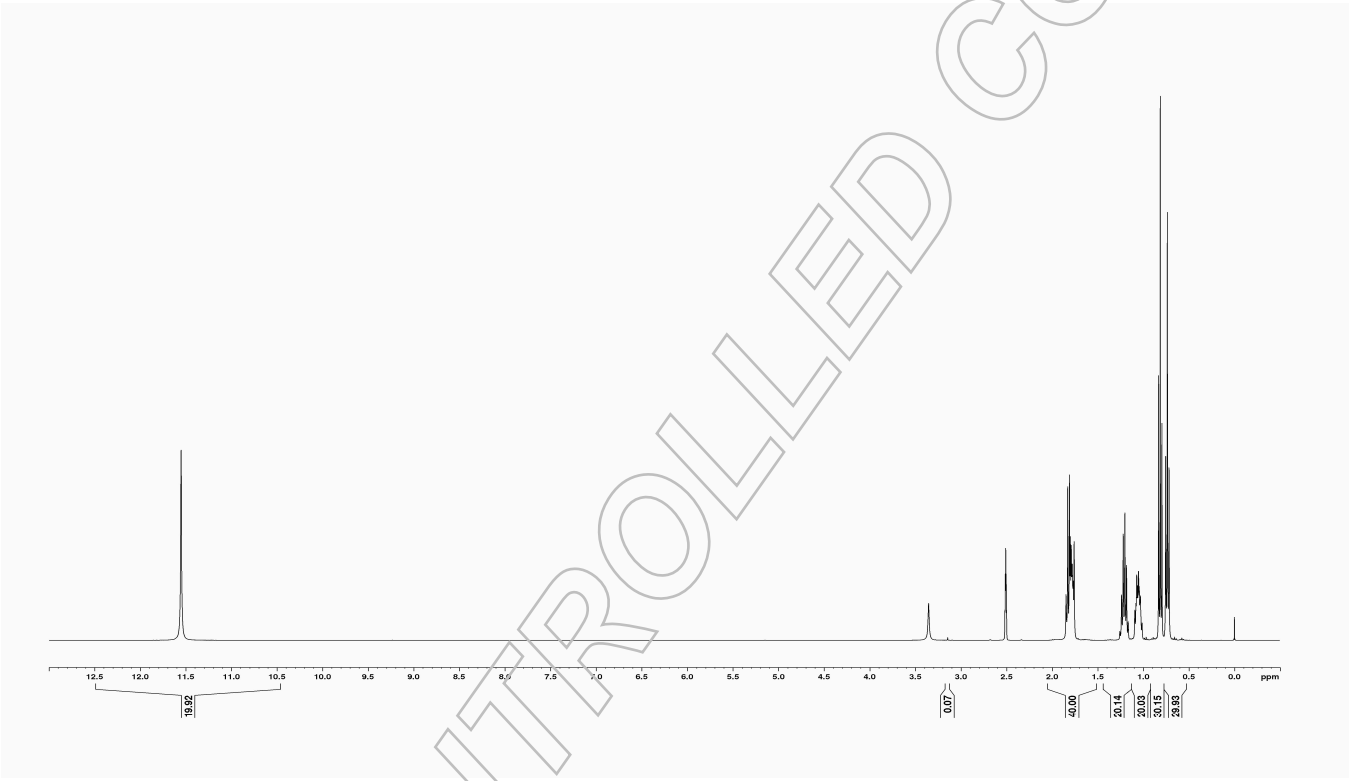
I. Identity

The identity of the reference substance was established by following analyses.

Ia. ^{1}H-NMR Spectrum

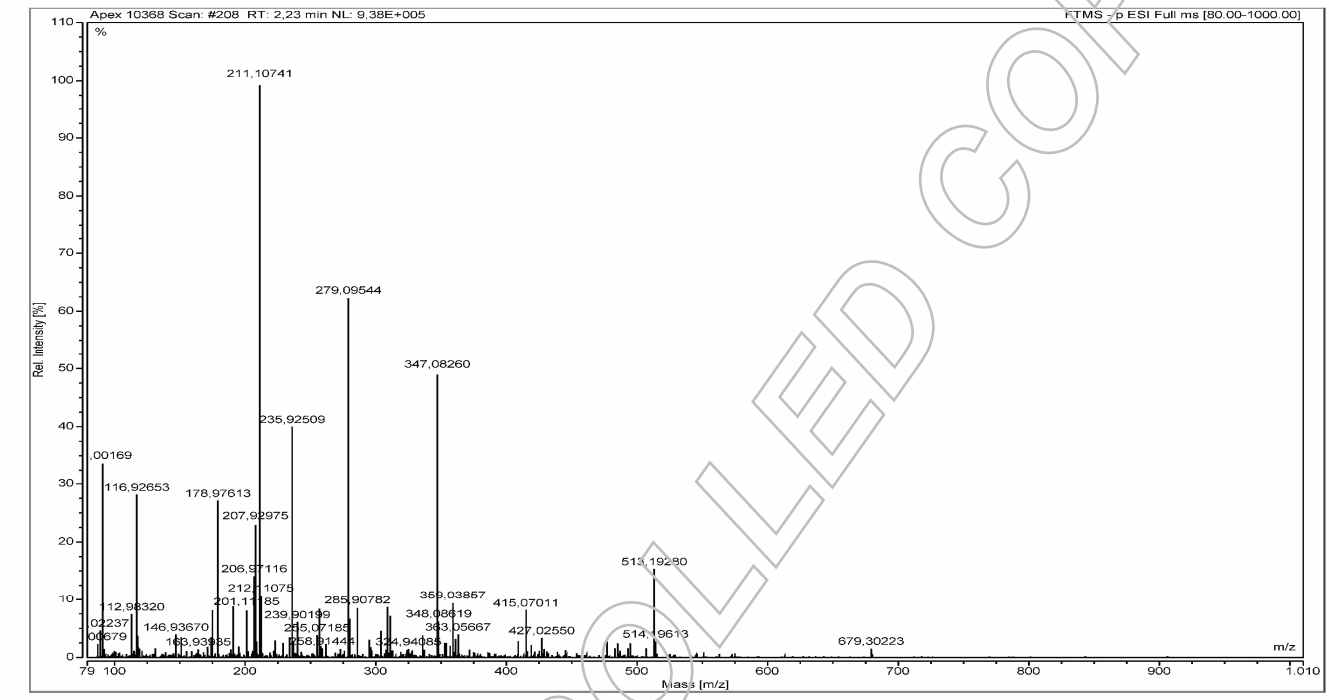
Conditions: 400 MHz, DMSO-d_6



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

Ib. Mass Spectrum

Method: HRMS; 3.2 kV ESI-; capillary temperature: 269 °C

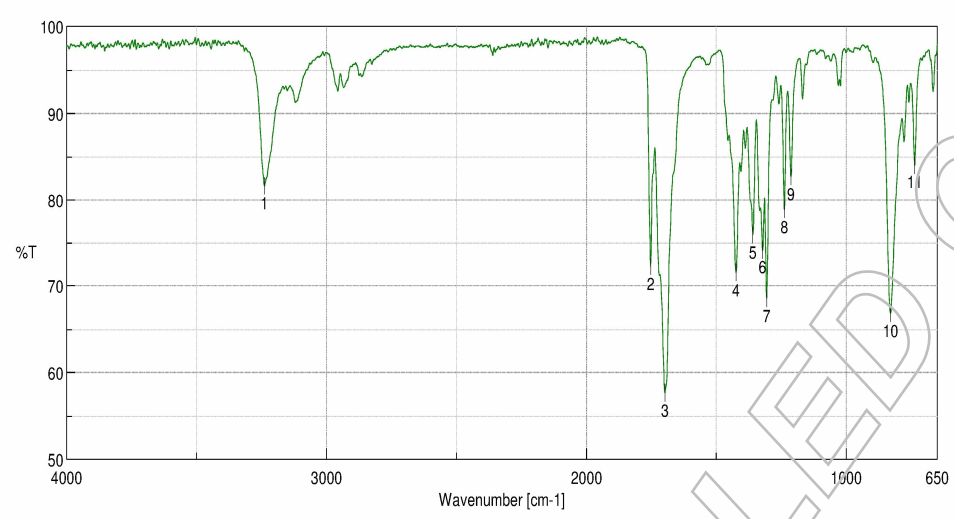


Theoretical value: 211.10882

The signal of the MS spectrum is consistent with the theoretical value and its interpretation is consistent with the structural formula.

Ic. IR Spectrum

Method: Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy



Results of Peak Find

No.	Position	Intensity
1	3238.86	81.5713
2	1752.98	72.1742
3	1698.02	57.6399
4	1424.17	71.4889
5	1359.57	75.9158
6	1321.96	74.1428
7	1306.54	68.5333
8	1238.08	78.864
9	1213.01	82.6432
10	829.241	66.806
11	736.674	84.0212

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

II. Purity

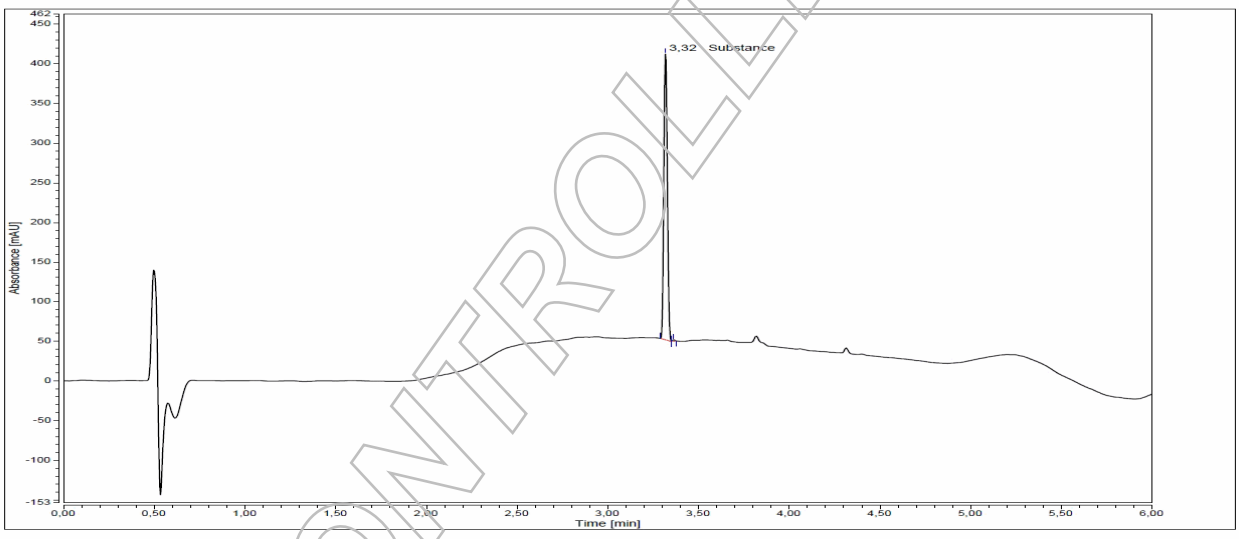
IIa. High Performance Liquid Chromatography (HPLC)

The purity of the reference substance was analysed by high performance liquid chromatography (HPLC).

HPLC Conditions:

Column:	**Conditions:**	**Detector:**	**Injector:**
Kinetex Phenyl-Hexyl 1.7 µm, 100 x 2.1 mm	0.50 ml/min, 40 °C mob. Phase A: Water, 0.1 % HCOOH mob. Phase B: Acetonitrile, 0.1 % HCOOH	DAD 210 nm	Auto 3 µl; 0.042 mg/ml in Acetonitrile/Water 50/50 (v/v)

0-1	min	A/B		98/2	
1-4	min	A/B	to	2/98	
4-5	min	A/B	to	98/2	
5-6	min	A/B		98/2	(v/v)



Area Percent Report - Sorted by Signal

Pk#	Retention Time	Area	Area %
1	3.317	8.1204	99.68
2	3.363	0.0260	0.32
Totals		**8.1464**	**100.00**

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

Results:	
Average	99.67 %
Number of results	n=3
Standard deviation	0.01 %

IIb. Water Content

Method: Karl Fischer titration

Results:	
Average	0.06 %
Number of results	n=3
Standard deviation	0.01 %

IIc. Residual Solvents

Method: ^{1}H-NMR

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

III. Final Result

Chromatographic purity (HPLC)	99.67 %
Water content	0.06 %
Residual solvents	No significant amounts of residual solvents were detected (< 0.05 %)
Assay (100 % method)[1]	99.61 %

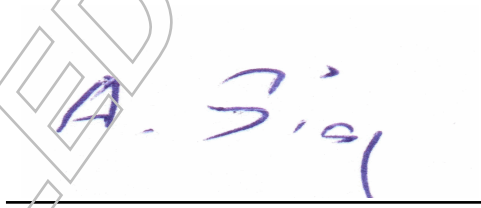
The assay is assessed to be 99.6 % ‘as is’

The assay 'as is' is equivalent to the assay based on the not anhydrous and not dried substance respectively.

Release Date:

Luckenwalde, 2015-July-21

Signed:



Dr. Andreas Sieg

Product Release

[1] The calculation of the 100 % method follows the formula:

$$\text{Assay (\%)} = (100\ \% - \text{volatile contents}) * \frac{\text{Purity (\%)}}{100\%}$$

Volatile contents are considered as absolute contributions, purity is considered as relative contribution

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