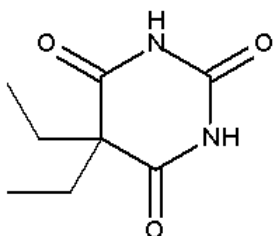




Certificate of Analysis

Reference Standard

Barbital



Molecular Formula: $C_8H_{12}N_2O_3$
Molecular Weight: 184.19
CAS Number: 57-44-3

Catalogue Number: LGCFOR1721.00
Lot Number: 87365
Long-term Storage: 2 to 8 °C, dark
Appearance: white solid
Melting Point (DSC): 189 °C
Assay 'as is': 99.95 %

Date of shipment: **2017-January-25**

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 Quality | ISO 9001:2008
DQS 102448 QM08

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

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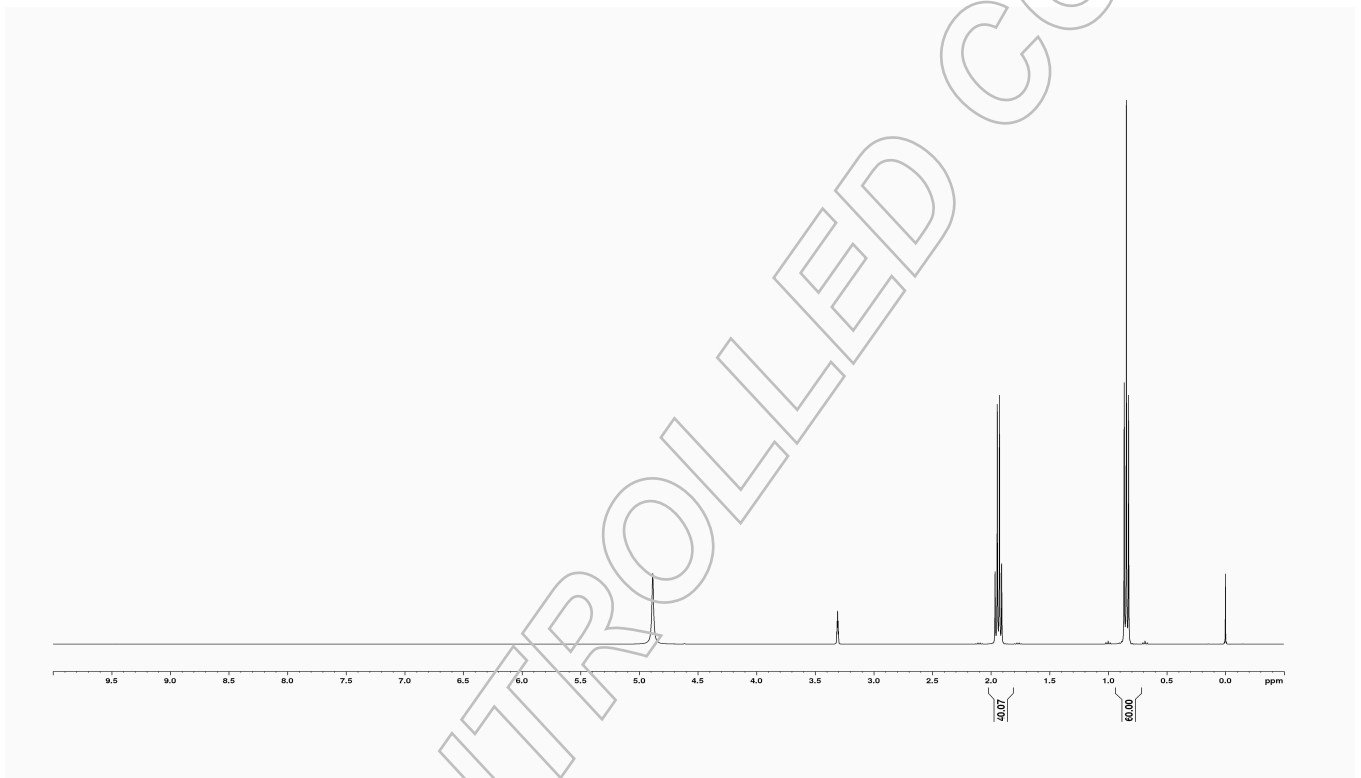


I. Identity

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

1a. ¹H-NMR Spectrum

Conditions: 400 MHz, CD₃OD

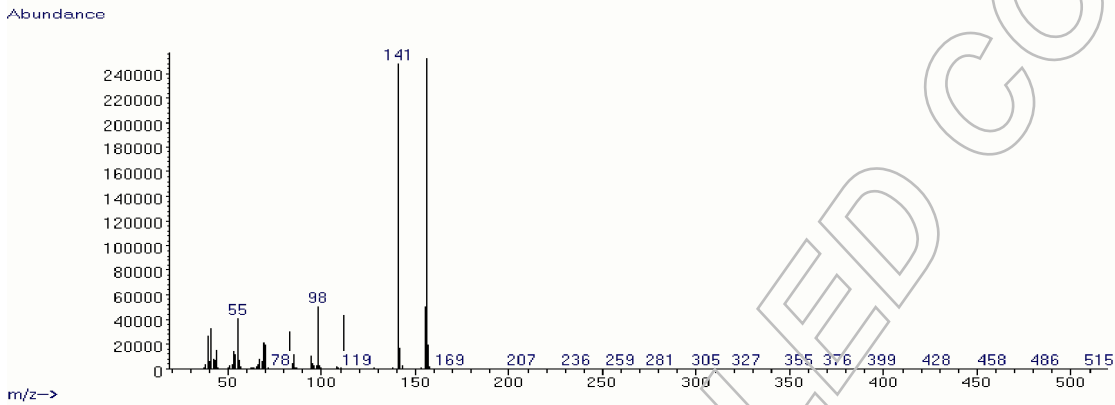


The structure is confirmed by the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

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

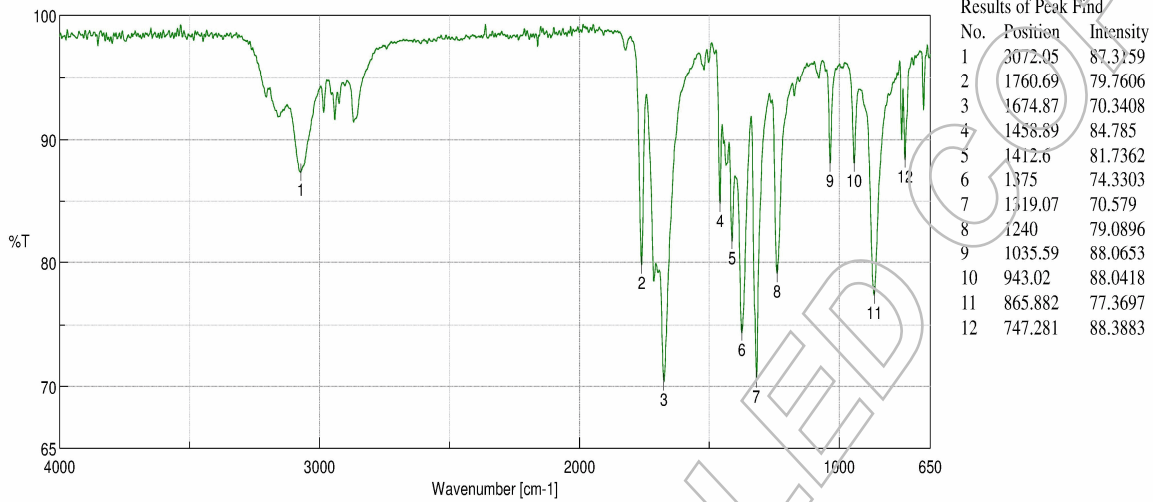


The signals of the MS spectrum and their interpretation are consistent with the structural formula.



Ic. IR Spectrum

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



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



II. Purity

Ila. High Performance Liquid Chromatography (HPLC)

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

HPLC Conditions:

Column:

Cortecs UPLC C18 +
1.6 µm, 75 x 2.1 mm

Conditions:

0.50 ml/min, 40 °C

mob. Phase A: Water, 0.1 % HCOOH

mob. Phase B: Acetonitrile, 0.1 % HCOOH

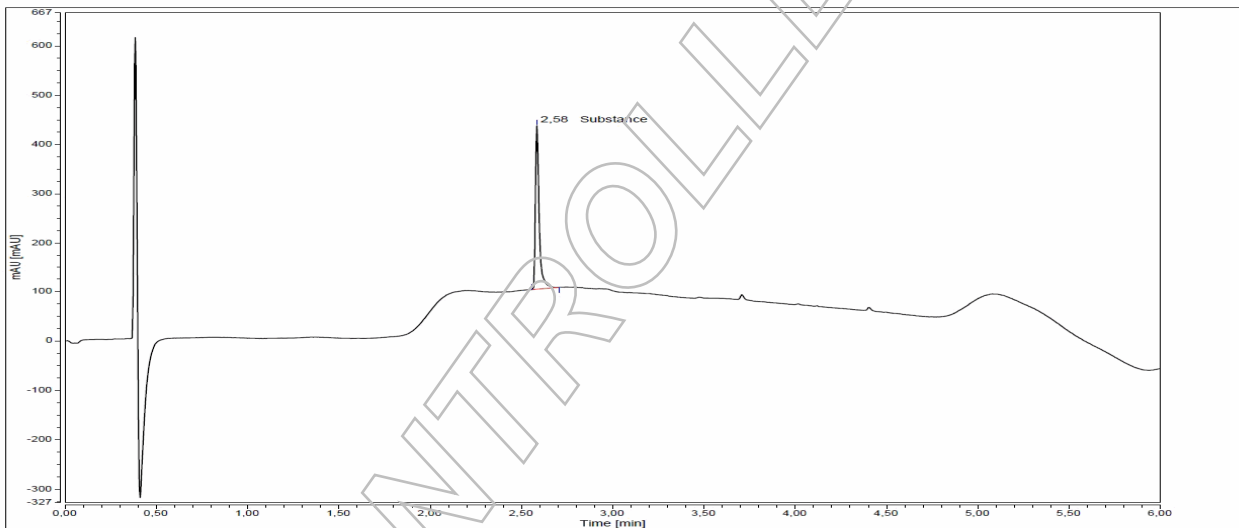
| | | | | |
|-----|-----|-----|----|------------|
| 0-1 | min | A/B | | 98/2 |
| 1-4 | min | A/B | to | 2/98 |
| 4-5 | min | A/B | to | 98/2 |
| 5-6 | min | A/B | | 98/2 (v/v) |

Detector:

DAD
210 nm

Injector:

Auto
1 µl; 0.032 mg/ml in
Acetonitrile



Area Percent Report - Sorted by Signal

| PK# | Retention Time | Area | Area % |
|--------|----------------|---------------|---------------|
| 1 | 2.584 | 7.8832 | 100.00 |
| Totals | | 7.8832 | 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 | 100.00 % |
| Number of results | n=3 |
| Standard deviation | < 0.01 % |

IIb. Water Content

Method: Karl Fischer titration

Results:

| | |
|---------------------------|--------|
| Average | 0.05 % |
| Number of results | n=3 |
| Standard deviation | 0.01 % |

IIc. Residual Solvents

Method: ¹H-NMR

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

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III. Final Result

| | |
|---|--|
| Chromatographic purity (HPLC) | 100.00 % |
| Water content | 0.05 % |
| Residual solvents | No significant amounts of residual solvents were detected (< 0.05 %) |
| Assay (100 % method)¹ | 99.95 % |

The assay is assessed to be 99.95 % 'as is'

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

Release Date:

Luckenwalde, 2016-January-27

Signed:

Dr. Sabine Schröder
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