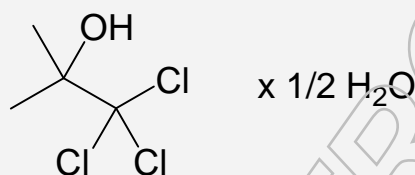


## Certificate of Analysis

### Reference Substance

#### Chlorobutanol Hemihydrate

Catalogue Number:	LGCFOR0535.00	Long-term Storage:	2 to 8 °C, dark
Lot Number:	37480	Appearance:	white solid
Molecular Formula:	C <sub>4</sub> H <sub>7</sub> Cl <sub>3</sub> O ½H <sub>2</sub> O	Melting Point:	84 °C
Molecular Weight:	186.47	Assay 'as is':	100 %
CAS Number:	[ 6001-64-5 ]		



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

This certificate is valid for two years from the date of shipment provided the substance is stored under the recommended conditions.

Release Date: 2013-09-13

LGC GmbH

Dr. Sabine Schröder  
Product Release

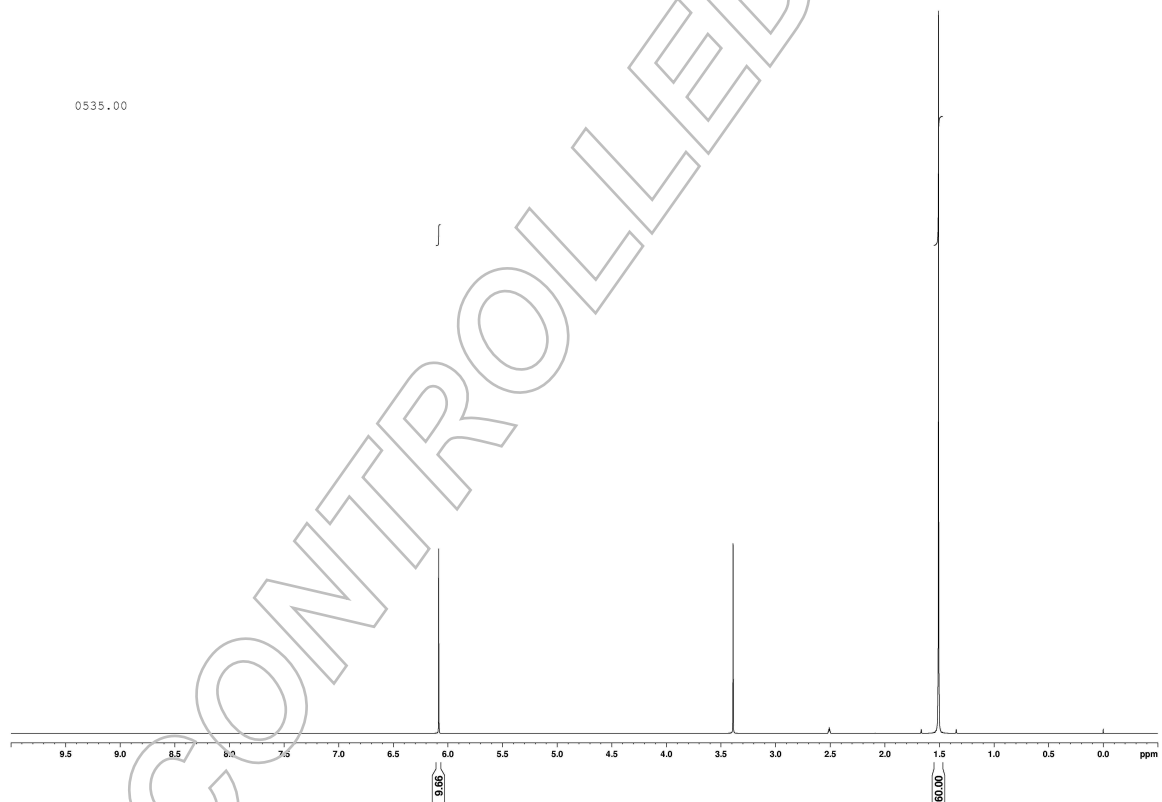
## I. Identity

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

### Ia. <sup>1</sup>H-NMR Spectrum

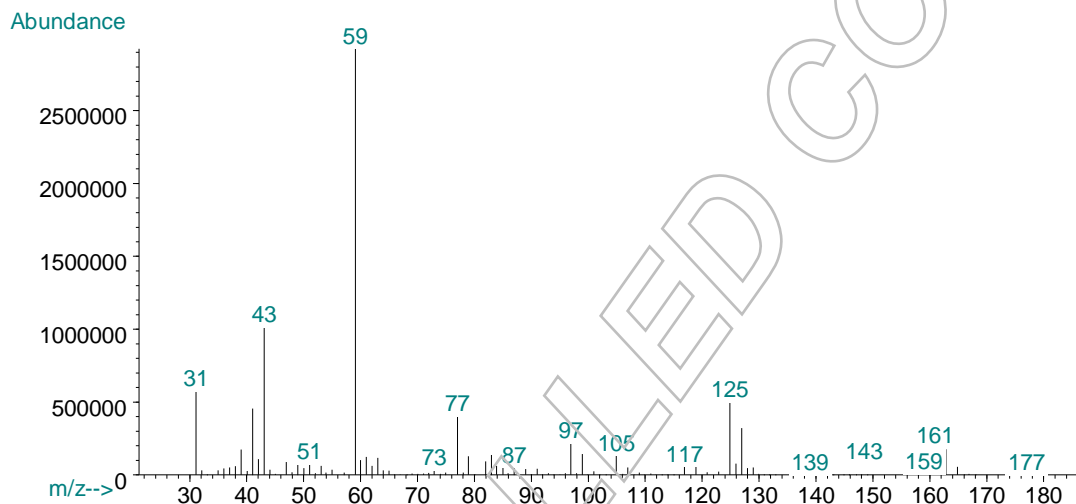
Conditions: 400 MHz, DMSO-d<sub>6</sub>

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



## Ib. Mass Spectrum

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

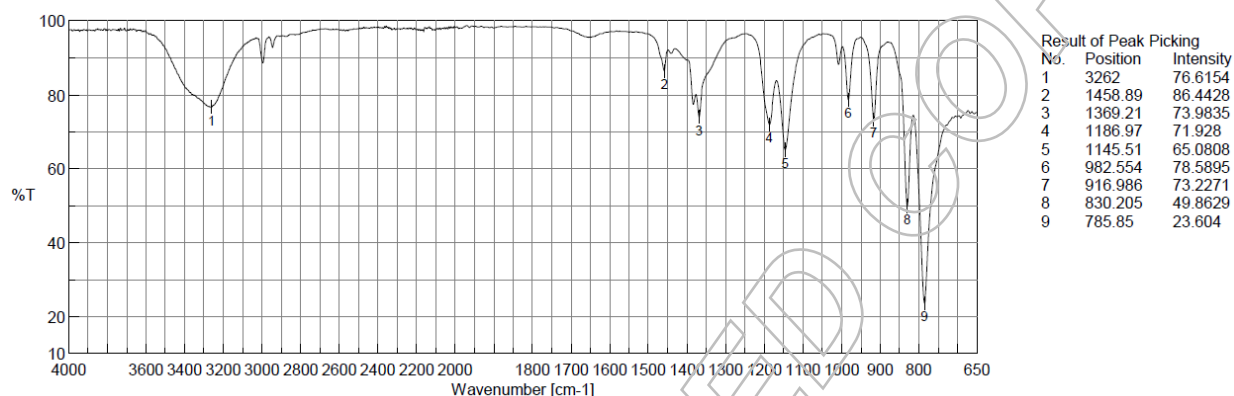


m/z	fragments (M = anhydrous base)
59	[ C <sub>3</sub> H <sub>7</sub> O ]

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



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

## II. Purity

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

### GC Conditions:

**Column:**

HP-5MS  
30 m x 0.25 mm x 0.25 µm

**Injector and Flow:**

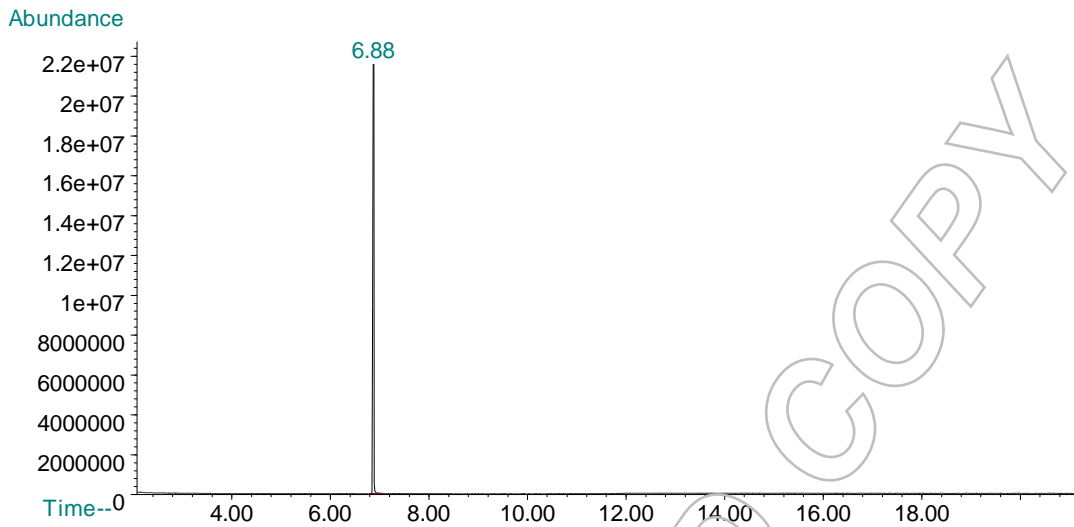
splitless Injection, 220 °C  
Helium 1.50 ml/min

**Oven Program:**

Initial Temp.: 50 °C for 5 min  
Heating Rate: 40 °C/min  
Final Temp.: 300 °C

**Detector:**

EI, 70 eV  
30 to 550 amu  
280 °C



### Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	6.878	395605508	100.00
Totals		395605508	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 %.

#### Results:

**Average** 100 %  
**Number of results** n=3  
**Standard deviation** < 0.01 %

### III. Water Content

Method: Karl Fischer titration

Determined value	4.82 %
Theoretical value	– 4.83 %
Content of excessive water	– 0.01 %

The result of the Karl Fischer titration confirms the theoretical value, taking the experimental error into account.

### IV. Residual Solvents

Method: <sup>1</sup>H-NMR

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

### V. Final Result

<b>Total impurities (GC)</b>	0.00 %
<b>Residual solvents</b>	n. d. (not detected)
<b>Assay (100 % method) <sup>1</sup></b>	100.00 %

The assay is assessed to be 100 % 'as is'

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

<sup>1</sup> The calculation of the 100 % method follows the formula:

$$\text{Assay (\%)} = (100 \% - \text{KF} - \text{RES}) \times \frac{\text{Purity GC (\%)}}{100 \%}$$

Water (KF) and Residual solvents (RES) are considered as absolute contributions, GC purity is considered as relative contribution.

LGCFOR0535.00 Lot Number 37480

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