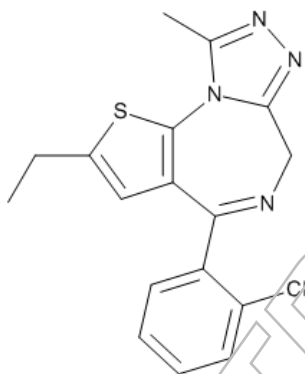




# Certificate of Analysis

## Reference Standard

Etizolam



Molecular Formula:  $C_{17}H_{15}ClN_4S$   
Molecular Weight: 342.85  
CAS Number: 40054-69-1

Catalogue Number: LGCFOR1386.00  
Lot Number: 79345  
Long-term Storage: 2 to 8 °C, dark  
Appearance: white solid  
Melting Point: 148 °C  
Assay 'as is': 99.95 %

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 unopened in the original container.

**LGC Quality** | ISO 9001:2008  
DQS 102448 QM08

**LoGiCal®**  
produced by LGC

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

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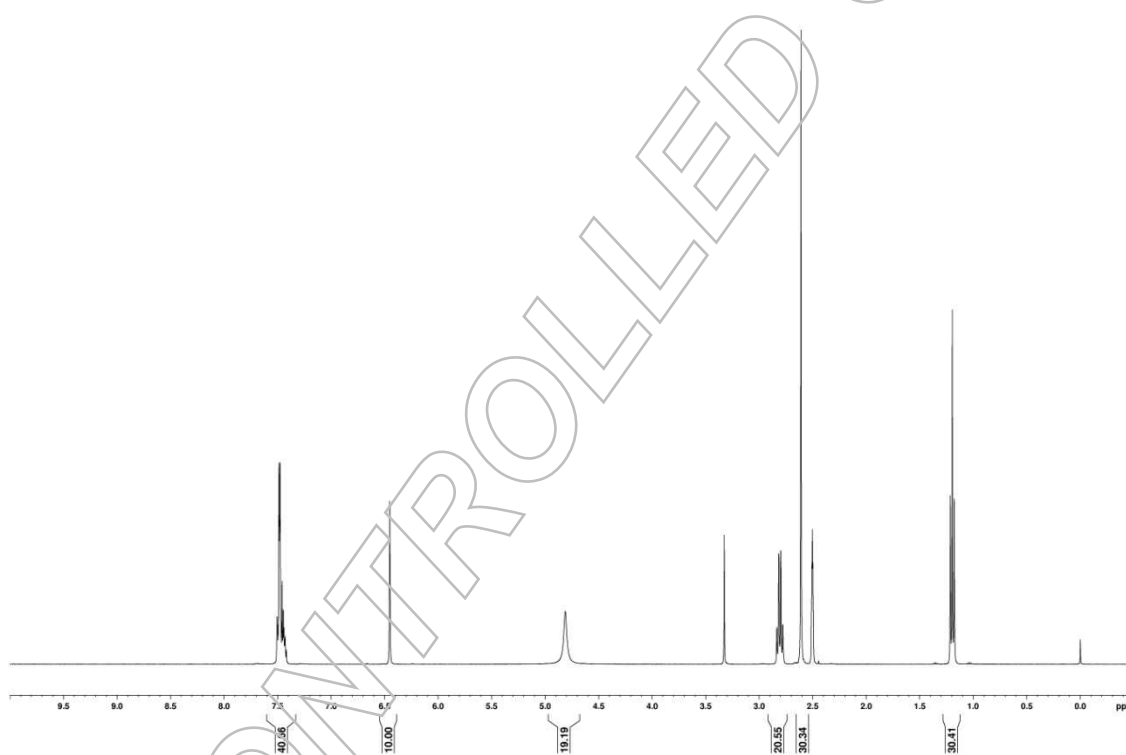


## I. Identity

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

### Ia. $^1\text{H}$ -NMR Spectrum

Conditions: 400 MHz, DMSO- $d_6$



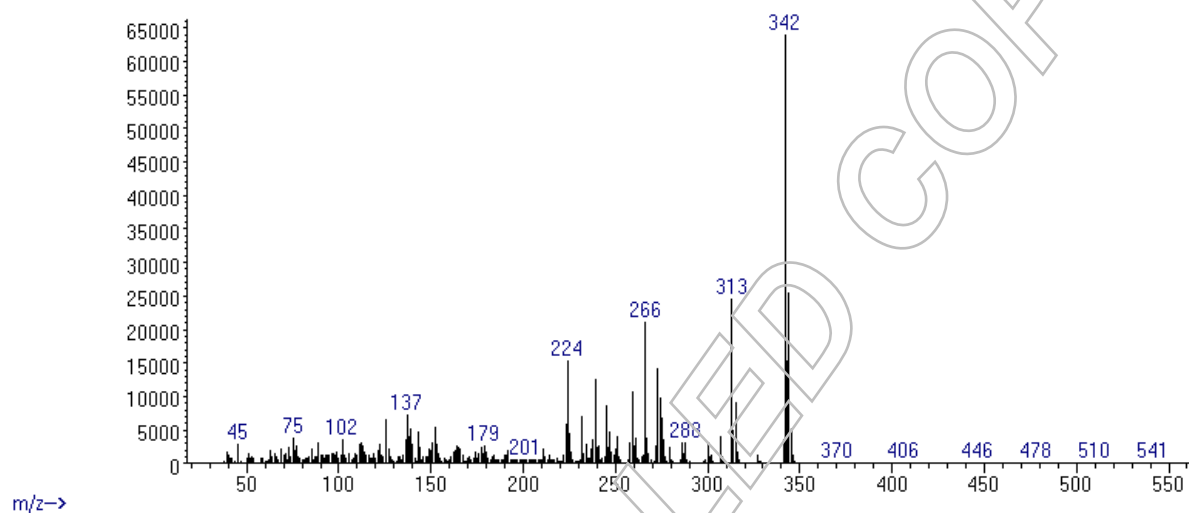
The structure is confirmed by the signals of the spectrum and their interpretation.



## lb. Mass Spectrum

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

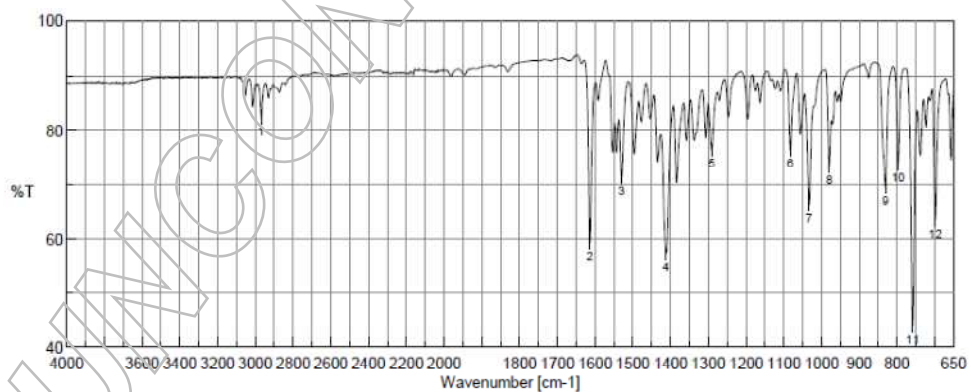
Abundance



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

## lc. IR Spectrum

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



Result of Peak Picking		
No.	Position	Intensity
1	2966.95	82.3578
2	1614.13	59.2664
3	1529.27	71.2869
4	1411.64	57.3158
5	1291.11	76.2974
6	1081.87	76.3098
7	1033.66	66.3175
8	979.661	73.1928
9	830.205	69.4506
10	797.421	73.621
11	758.852	43.9326
12	699.069	63.4392

The signals of the IR spectrum and their interpretation are consistent with the 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:**

Hypersil Gold C18  
5 µm, 150 x 4.6 mm

**Conditions:**

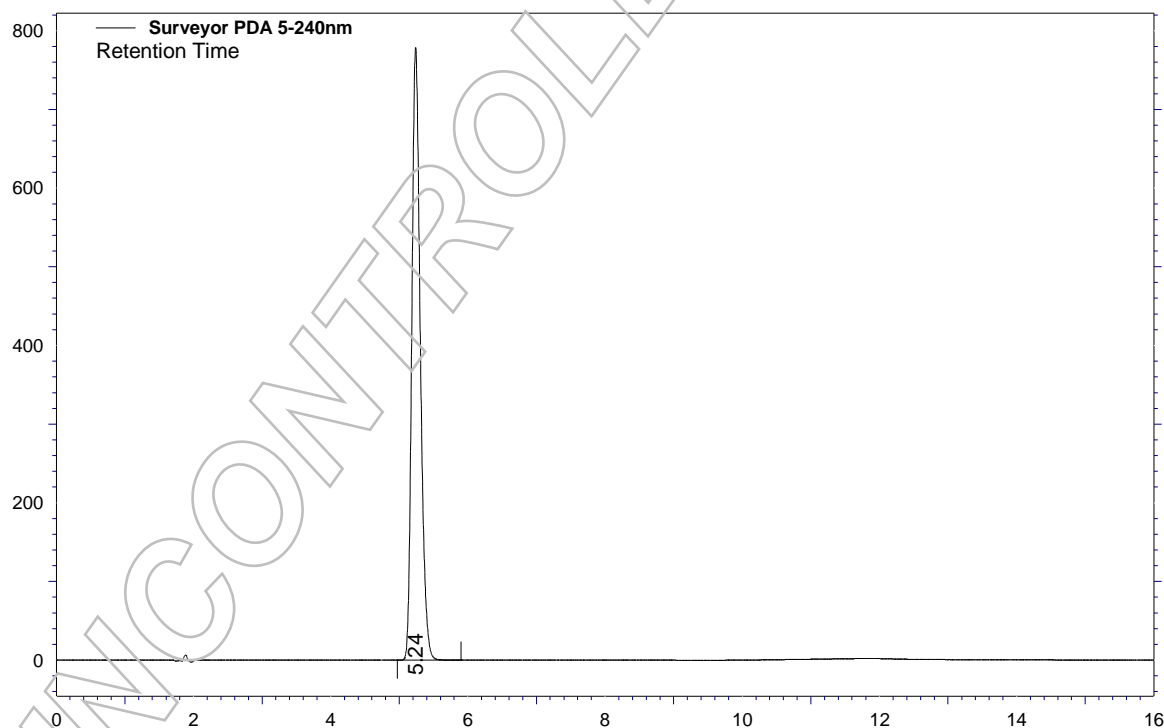
1.0 ml/min, 40 °C  
0-6 min Water/Acetonitrile 60/40  
6-9 min Water/Acetonitrile to 20/80  
9-11 min Water/Acetonitrile to 60/40  
11-16 min Water/Acetonitrile 60/40 (v/v);  
0.1 % H<sub>3</sub>PO<sub>4</sub>

**Detector:**

DAD  
240 nm

**Injector:**

Auto  
0.5 µl; 1.028 mg/ml in  
Methanol





### Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	5.24	6712674	100.00
Totals		6712674	100.00

For the calculation the system 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 %

#### IIb. Water Content

Method: Karl Fischer titration

#### Results:

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

#### IIc. Residual Solvents

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

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



### III. Final Result

<b>Chromatographic purity (HPLC)</b>	100 %
<b>Water content</b>	0.05 %
<b>Residual solvents</b>	No significant amounts of residual solvents were detected (< 0.05 %).
<b>Assay (100 % method)<sup>1</sup></b>	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, 2015-06-18

Dr. Sabine Schröder  
Product Release

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<sup>1</sup> 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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