

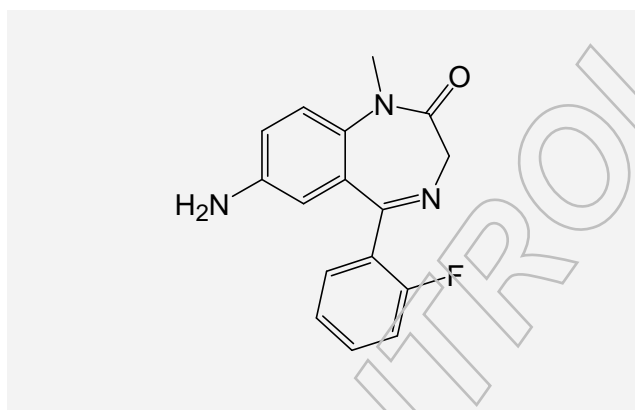
## Certificate of Analysis

### Reference Substance

#### 7-Aminoflunitrazepam

Catalogue Number: LGCFOR0069.07  
Lot Number: 18493  
Molecular Formula: C<sub>16</sub>H<sub>14</sub>FN<sub>3</sub>O  
Molecular Weight: 283.30  
CAS Number: [ 34084-50-9 ]

Long-term Storage: 2 to 8 °C, dark  
Appearance: pale yellow solid  
Melting Point: 207 °C (dec.)  
Assay 'as is': 99.7 %



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: 2012-09-10

LGC GmbH

Dr. Sabine Schröder  
Product Release

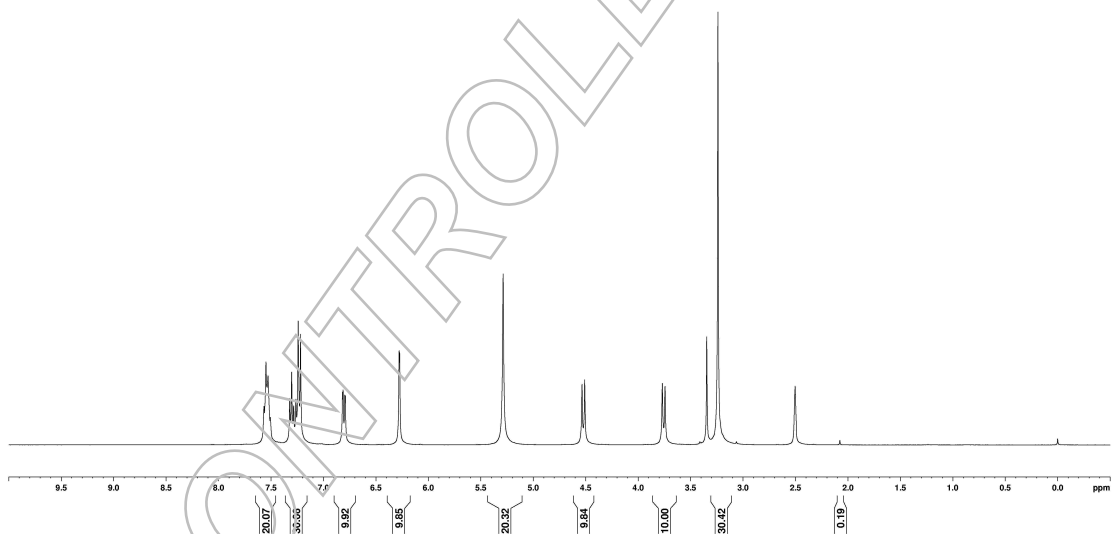
## I. Identity

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

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

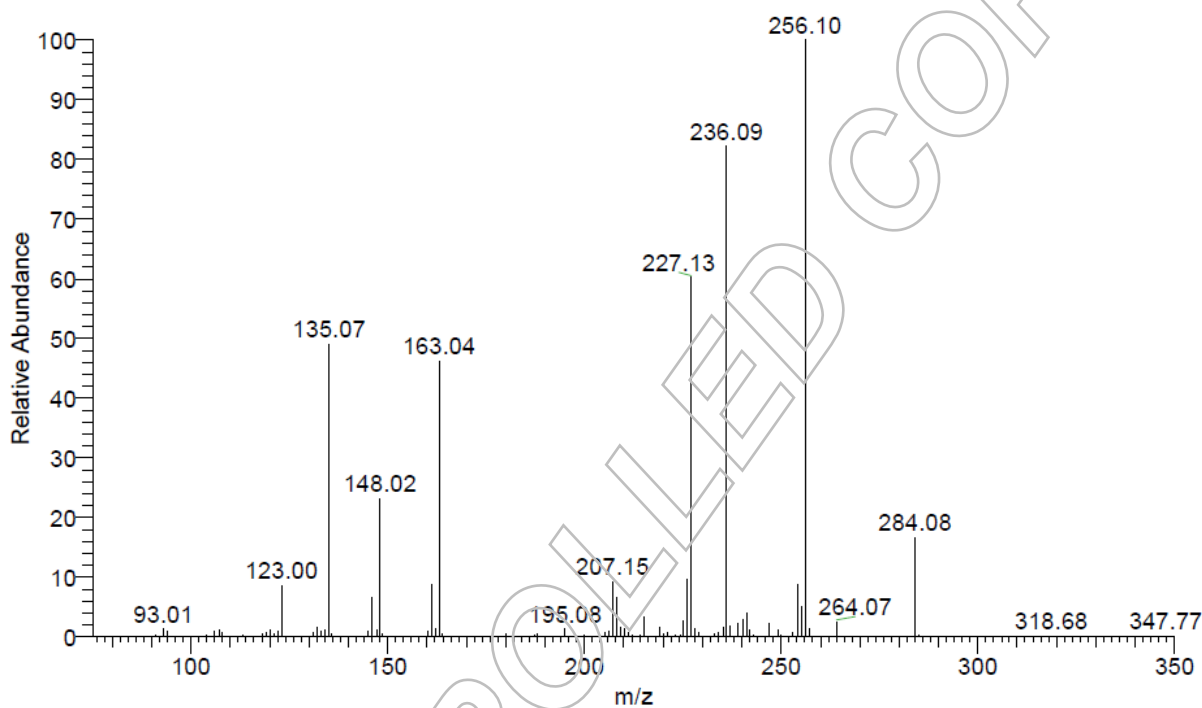
Conditions: 400 MHz, DMSO-d<sub>6</sub>

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



## 1b. Mass Spectrum

Method: 4.5 kV ESI; vaporization temperature: 200 °C, direct inlet

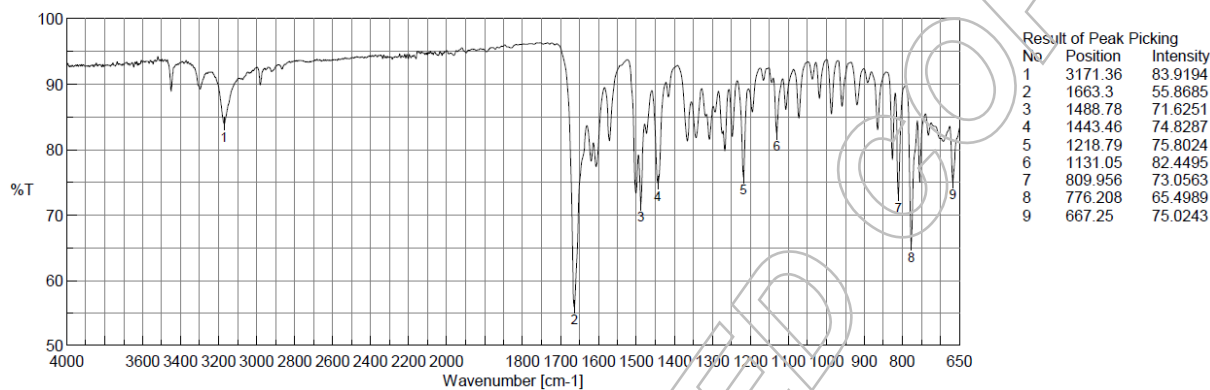


m/z	fragments
284	[ MH ]

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 high performance liquid chromatography (HPLC).

### HPLC Conditions:

#### Column:

Hypersil Gold (C18)  
5 µm, 150 x 4.6 mm

#### Conditions:

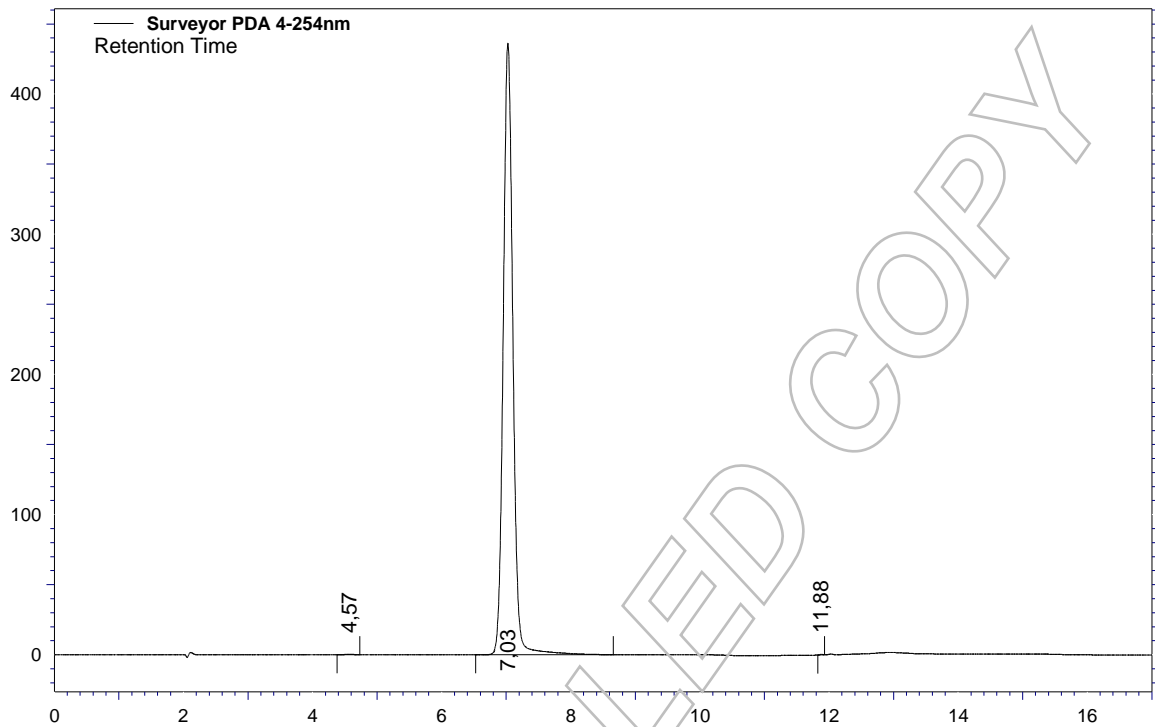
1.0 ml/min, 40 °C  
0 – 7 min Water/Acetonitrile 90/10  
7 – 10 min Water/Acetonitrile to 50/50  
10 – 12 min Water/Acetonitrile to 90/10  
12 – 17 min Water/Acetonitrile 90/10 (v/v);  
0.1 % H<sub>3</sub>PO<sub>4</sub>

#### Detector:

DAD  
254 nm

#### Injector:

Auto  
4 µl; 0.0572 mg/ml in  
Water/Acetonitrile 50/50 (v/v)



**Area Percent Report - Sorted by Signal**

Pk #	Retention Time	Area	Area %
1	4.57	2720	0.06
2	7.03	4539138	99.92
3	11.88	1078	0.02
Totals		4542936	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** 99.92 %  
**Number of results** n=3  
**Standard deviation** < 0.01 %

### III. Water Content

Method: Karl Fischer titration

**Results:**

<b>Average</b>	0.09 %
<b>Number of results</b>	n=3
<b>Standard deviation</b>	0.01 %

### IV. Residual Solvents

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

Result: 0.09 % Acetonitrile

### V. Final Result

<b>Total impurities (HPLC)</b>	0.08 %
<b>Water content</b>	0.09 %
<b>Residual solvents</b>	0.09 %
<b>Assay (100 % method) <sup>1</sup></b>	99.74 %

The assay is assessed to be 99.7 % 'as is'

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

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

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

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

LGCFOR0069.07 Lot Number 18493

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