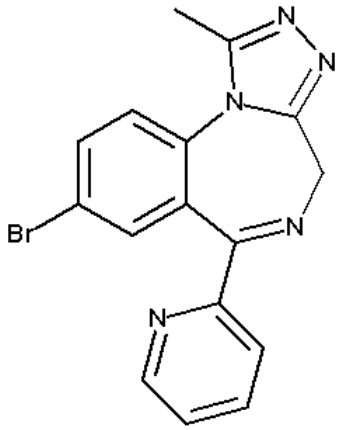
Certificate of Analysis

Reference Standard

Pyrazolam



Molecular Formula:	$C_{16}H_{12}BrN_5$
Molecular Weight:	354.20
CAS Number:	39243-02-2

Catalogue Number:	LGCFOR1441.00
Lot Number:	77200
Long-term Storage:	2 to 8 °C, dark
Appearance:	beige solid
Melting Point (DSC):	255 °C
Assay 'as is':	98.7 %

Date of shipment: **2017-September-05**

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

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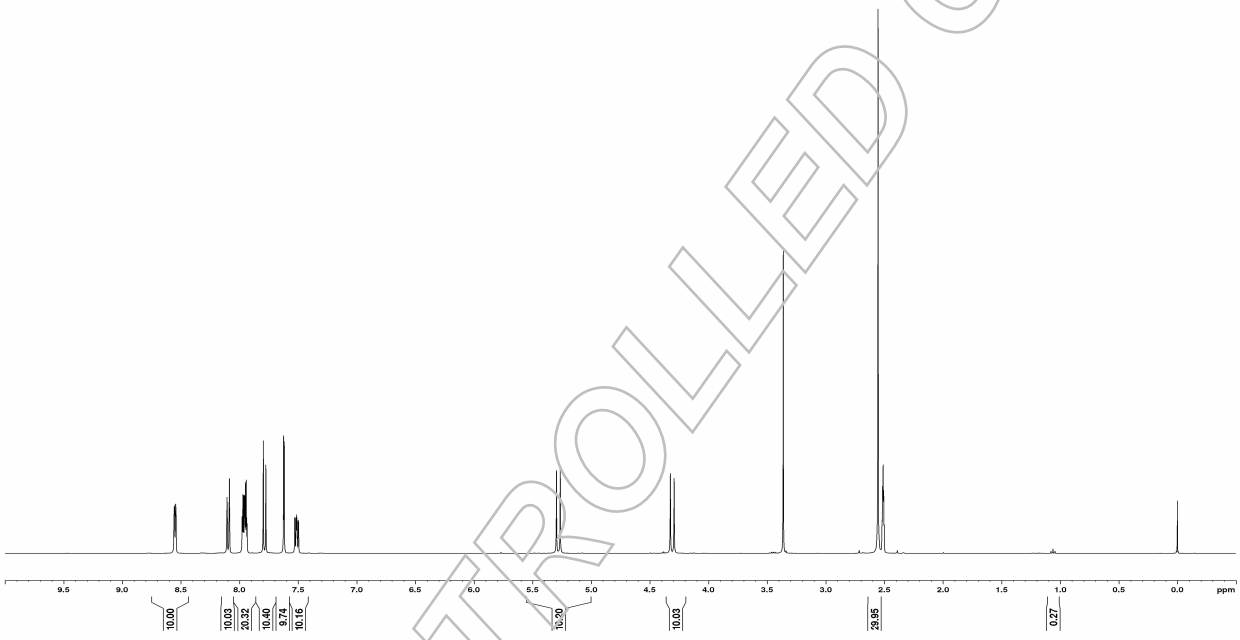
I. Identity

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.

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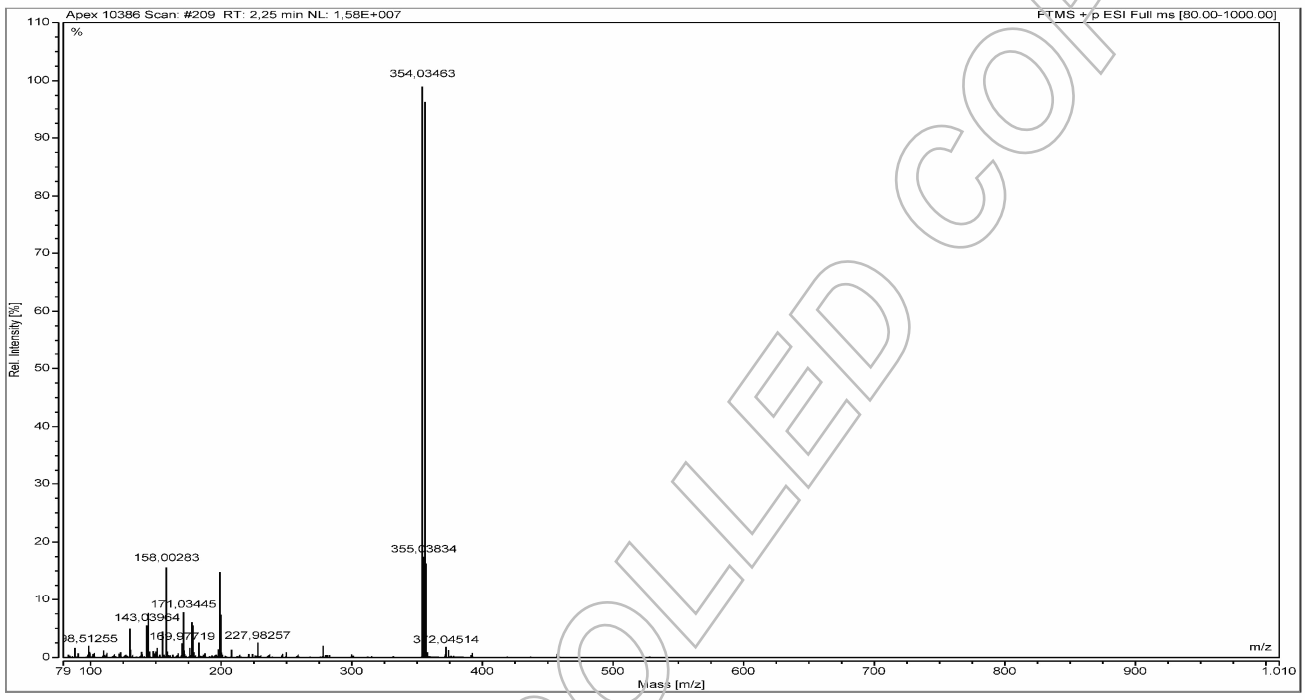
Ib. Mass Spectrum

Method: HRMS; 3.5 kV ESI+; capillary temperature: 269 °C



Theoretical value: 354.03488

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

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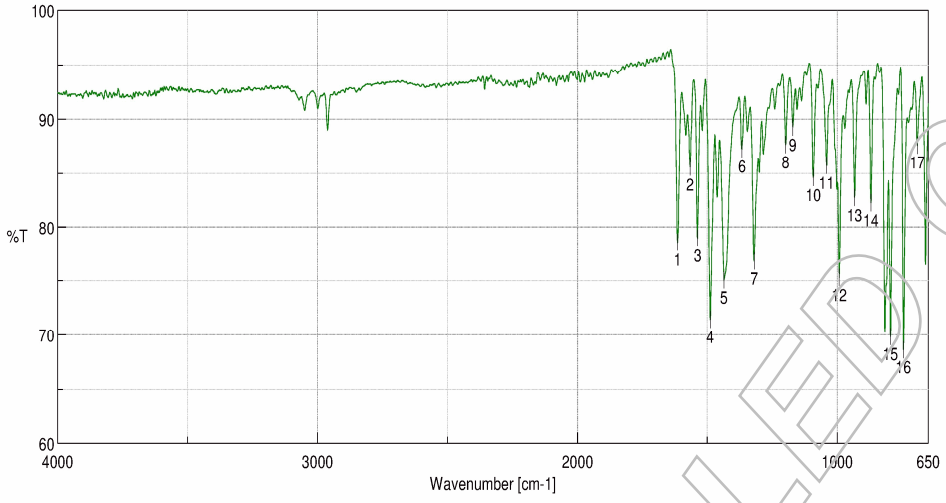
Ic. IR Spectrum

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



Results of Peak Find

No.	Position	Intensity
1	1615.09	78.5385
2	1566.88	85.529
3	1537.95	78.9228
4	1488.78	71.4194
5	1435.74	75.0614
6	1367.28	87.2229
7	1320.04	76.7783
8	1198.54	87.6208
9	1170.58	89.2591
10	1092.48	84.5757
11	1041.37	85.7046
12	992.196	75.2797
13	933.378	82.7219
14	870.703	82.2157
15	795.493	69.8094
16	745.352	68.609
17	692.32	87.577

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

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II. Purity

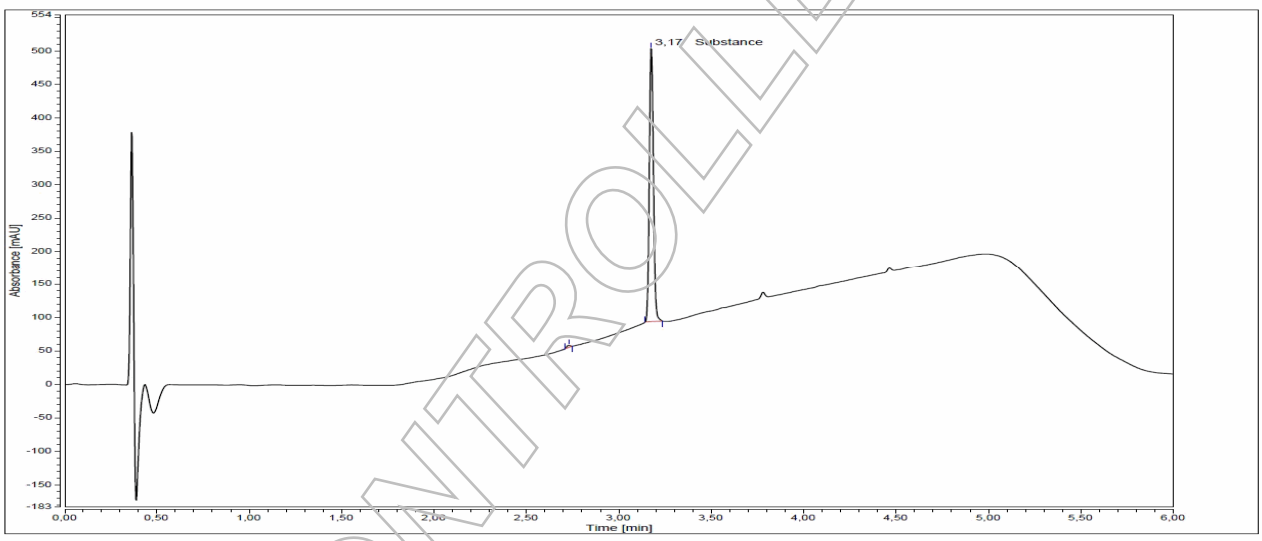
IIa. High Performance Liquid Chromatography (HPLC)

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

HPLC Conditions:

Column:	**Conditions:**	**Detector:**	**Injector:**
Cortecs UPLC C18 + 1.6 µm, 75 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 225 nm	Auto 2 µl; 0.050 mg/ml in Acetonitrile

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	2.730	0.0721	0.68
2	3.173	10.5823	99.32
Totals		**10.6544**	**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 %.

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Results:

Average	99.32 %
Number of results	n=3
Standard deviation	0.03 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average	0.48 %
Number of results	n=3
Standard deviation	0.05 %

IIc. Residual Solvents

Residual Solvent	**Average**	**Method**
Ethanol	0.12 %	^{1}H-NMR

III. Final Result

Chromatographic purity (HPLC)	99.32 %
Water content	0.48 %
Residual solvents	0.12 %
Assay (100 % method)[1]	98.72 %

The assay is assessed to be 98.7 % ‘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-14

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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