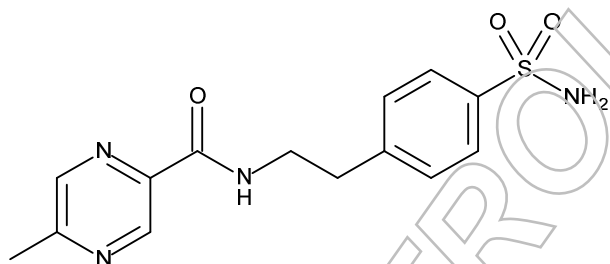




# Certificate of Analysis

## Reference Standard

5-Methyl-N-[2-(4-sulphamoylphenyl)ethyl]-  
pyrazine-2-carboxamide



Molecular Formula: C<sub>14</sub>H<sub>16</sub>N<sub>4</sub>O<sub>3</sub>S  
Molecular Weight: 320.37  
CAS Number: 33288-71-0

Catalogue Number: MM0942.01  
Lot Number: 130345  
Long-term Storage: 2 to 8 °C, dark  
Appearance: off-white solid  
Melting Point: 235 °C  
Assay 'as is': 99.2 %

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

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.

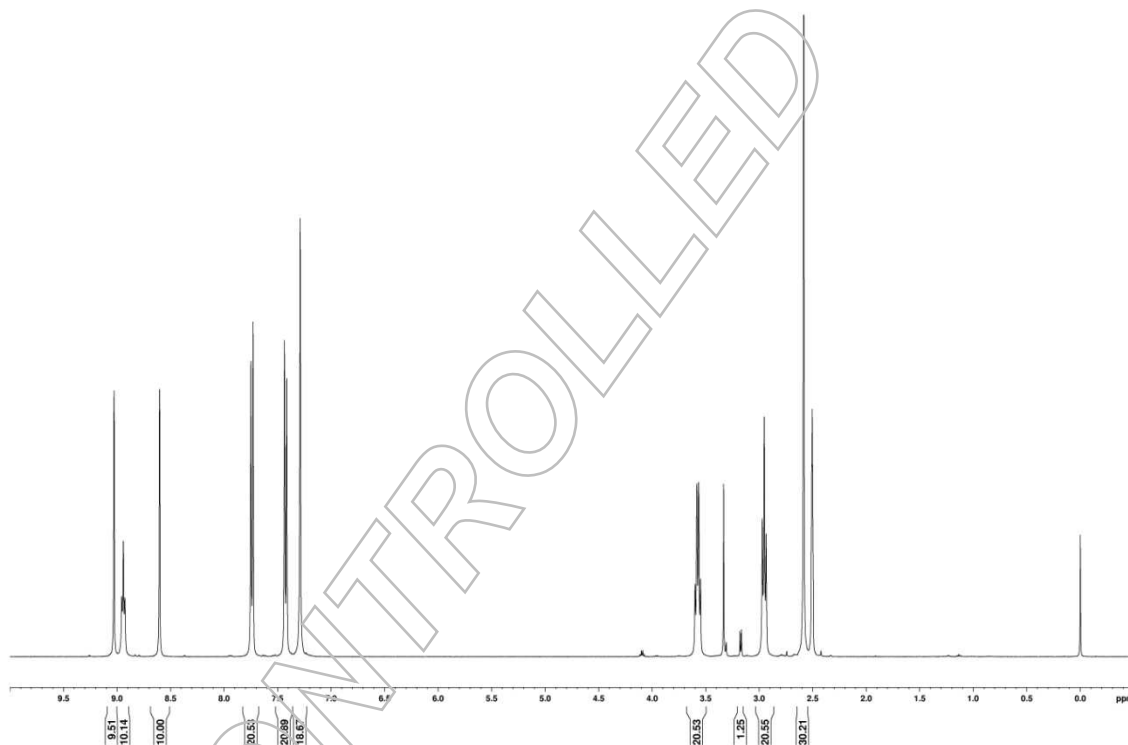


## I. Identity

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

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

Conditions: 400 MHz,  $\text{DMSO-d}_6$

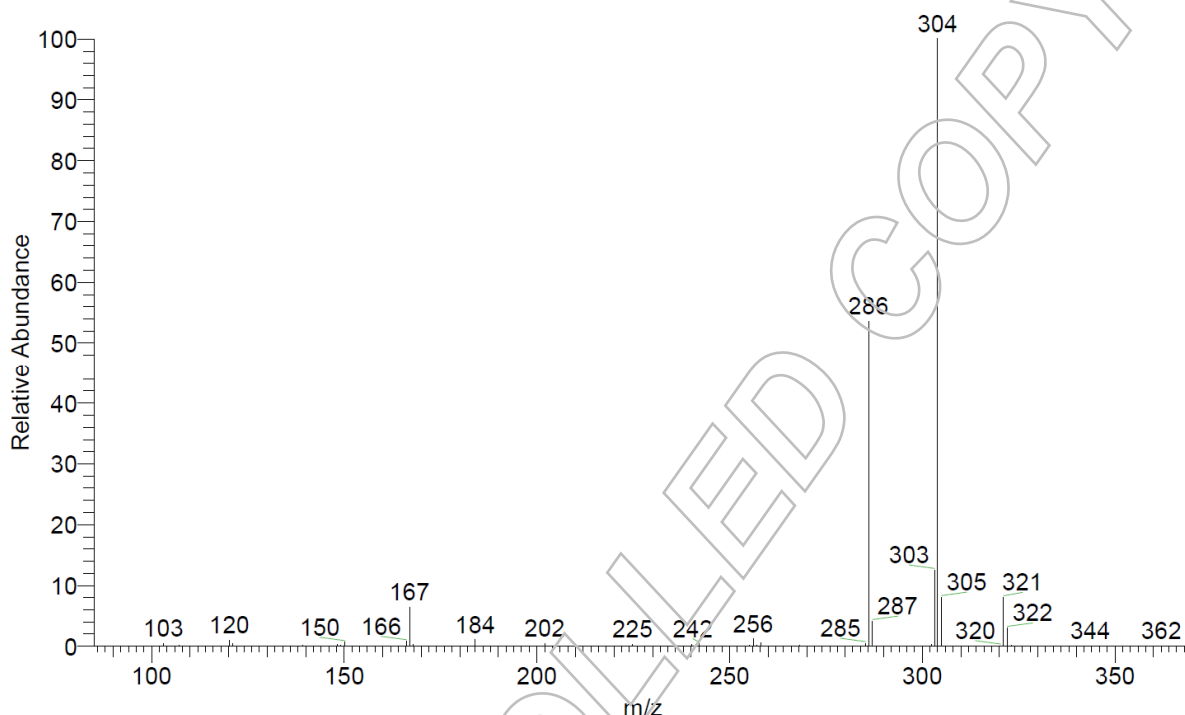


The structure is confirmed by the signals of the spectrum and their interpretation.



## Ib. Mass Spectrum

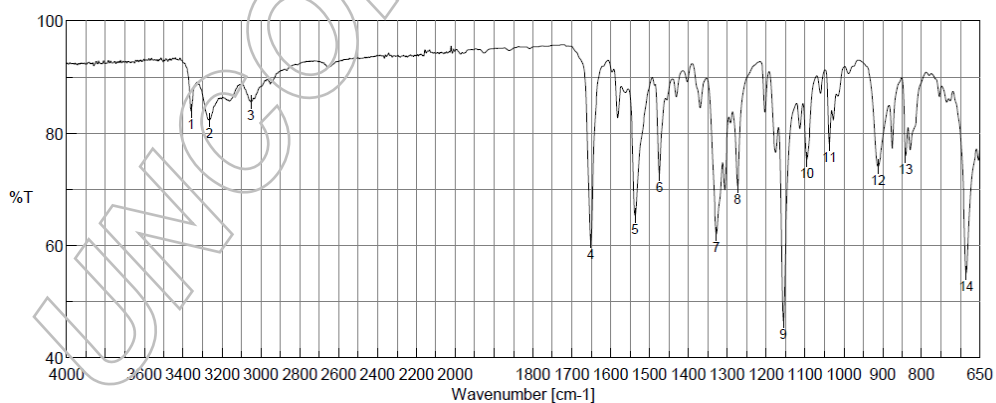
Method: 4.5 kV ESI+; vaporization temperature: 200 °C



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



Result of Peak Picking		
No.	Position	Intensity
1	3359.39	83.9627
2	3264.89	82.3079
3	3051.8	85.5544
4	1650.77	60.8176
5	1536.99	65.2971
6	1474.31	72.8289
7	1327.75	62.0168
8	1273.75	70.5804
9	1156.12	46.6025
10	1095.37	75.3213
11	1037.52	78.0443
12	912.165	74.046
13	840.812	75.9857
14	684.606	55.094

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  $\mu$ m, 150 x 4.6 mm

**Conditions:**

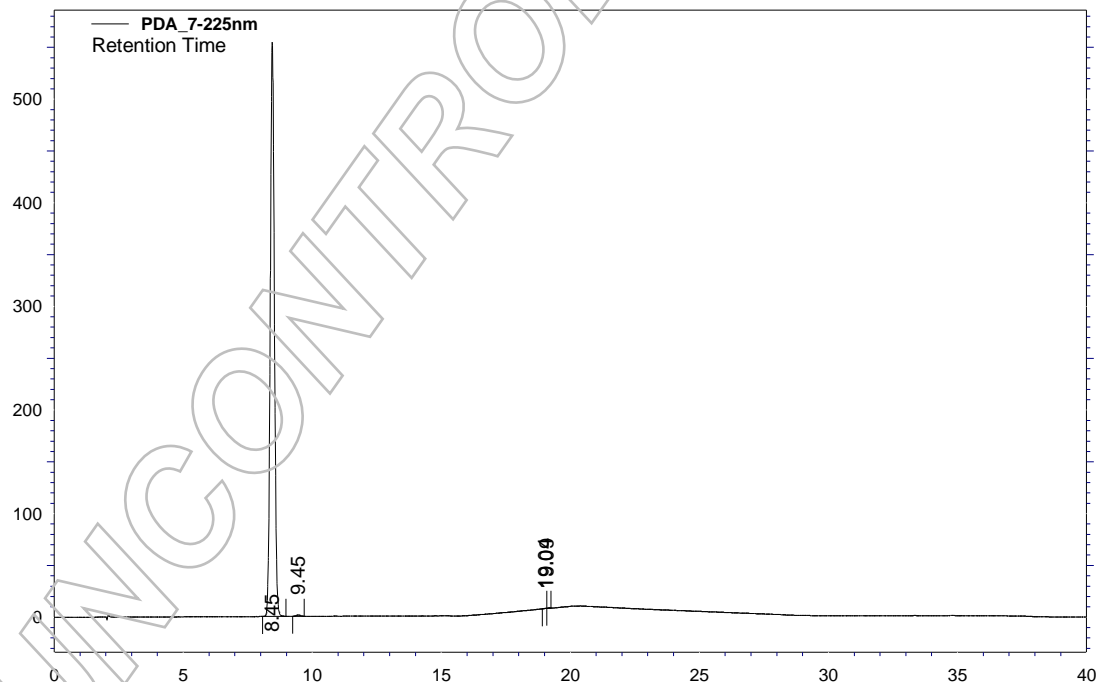
1.0 ml/min, 40 °C  
0-12 min Water/Acetonitrile 85/15  
12-17 min Water/Acetonitrile to 60/40  
17-25 min Water/Acetonitrile to 85/15  
25-40 min Water/Acetonitrile 85/15 (v/v),  
0.1 % H<sub>3</sub>PO<sub>4</sub>

**Detector:**

DAD  
225 nm

**Injector:**

Auto  
2  $\mu$ l; 0.1388 mg/ml in  
Water/Acetonitrile 50/50 (v/v)





### Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	8.45	6148498	99.71
2	9.45	14470	0.23
3	19.04	1104	0.02
4	19.09	2143	0.03
Totals		6166215	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 99.71 %  
Number of results n=3  
Standard deviation 0.01 %

#### IIb. Water Content

Method: Karl Fischer titration

#### Results:

Average 0.07 %  
Number of results n=3  
Standard deviation 0.01 %

#### IIc. Residual Solvents

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

Result: 0.42 % Methanol



### III. Final Result

<b>Chromatographic purity (HPLC)</b>	99.71 %
<b>Water content</b>	0.07 %
<b>Residual solvents</b>	0.42 %
<b>Assay (100 % method)<sup>1</sup></b>	99.22 %

The assay is assessed to be 99.2 % 'as is'

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

Release Date:

Luckenwalde, 2017-03-31

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.