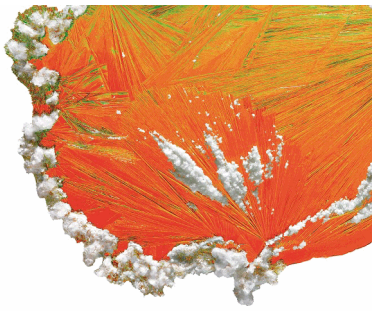
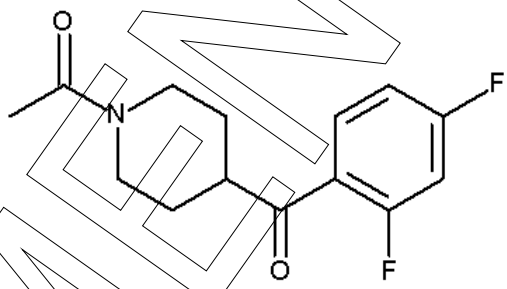


Certificate of Analysis

Reference Material

Product name

1-[4-(2,4-Difluorobenzoyl)piperidin-1-yl]ethanone



Product code
MM0491.24-0025

Lot number
1143951

CAS number
84162-82-3

Appearance
white solid

Molecular weight
267.27

Melting point (DSC)
100 °C

Molecular formula
$C_{14}H_{15}F_2NO_2$

Long-term storage
2 to 8 °C, dark

Assay "as is"
98.3 %

Date of shipment: **30 Aug 2022**

Producer confirms that this reference material (RM) meets the specification detailed on this Certificate of Analysis for **one year** from the date of shipment, provided the substance is stored under the recommended conditions unopened in the original container.

Release by:	**Date of Release:**		
Dr. Sabine Schröder	Luckenwalde, 24 Jun 2021		Product Release

Product information

For laboratory use only. Not suitable for human or animal consumption.

Before usage of the RM, it should be allowed to warm to room temperature. No drying required, as the certified value is already corrected for the content of water and other volatile materials.

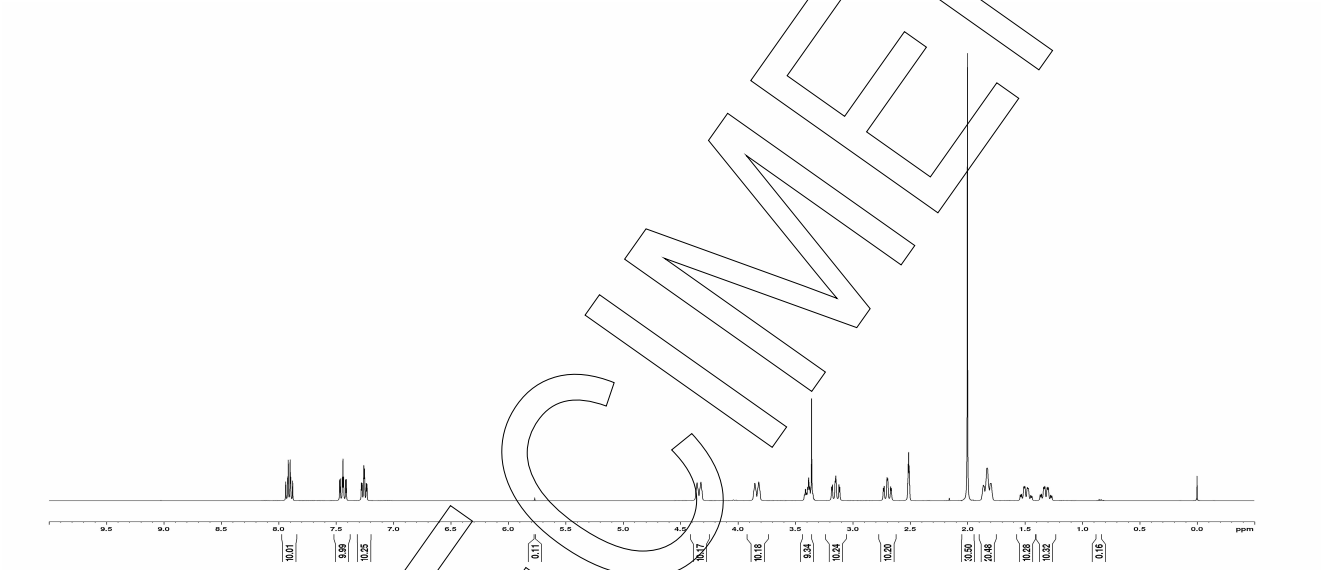
The product quality is controlled by regularly performed quality control tests (retests).

Further content

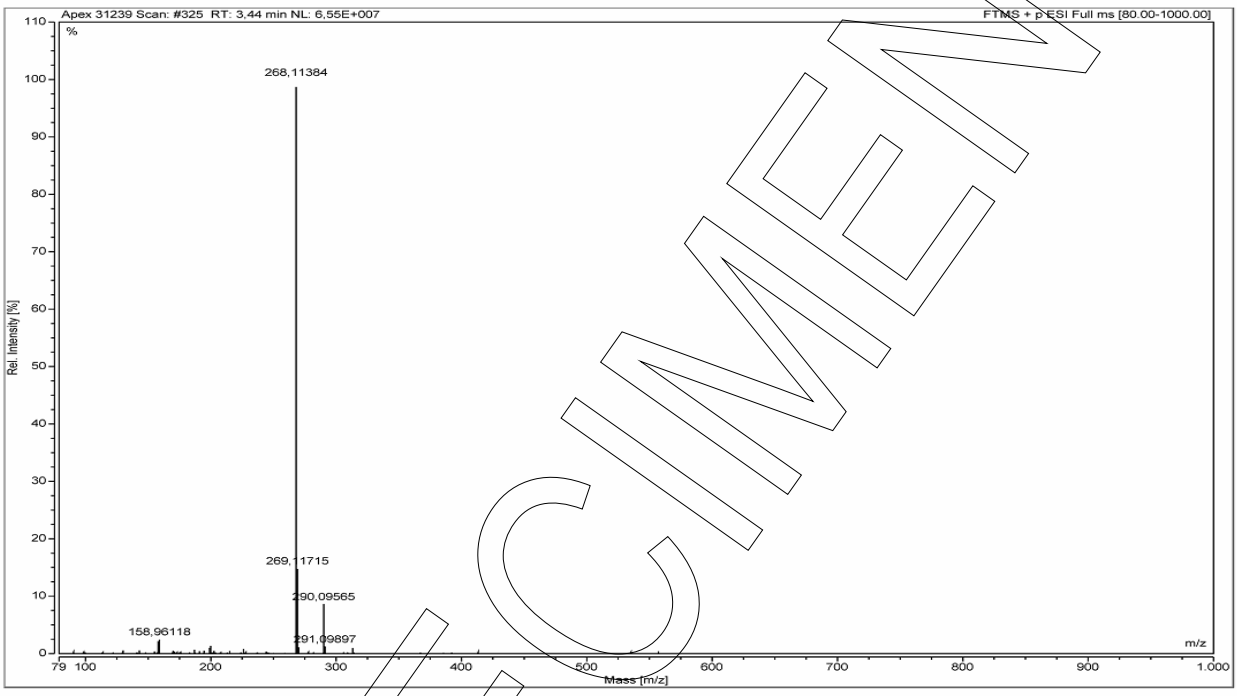
Identity

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

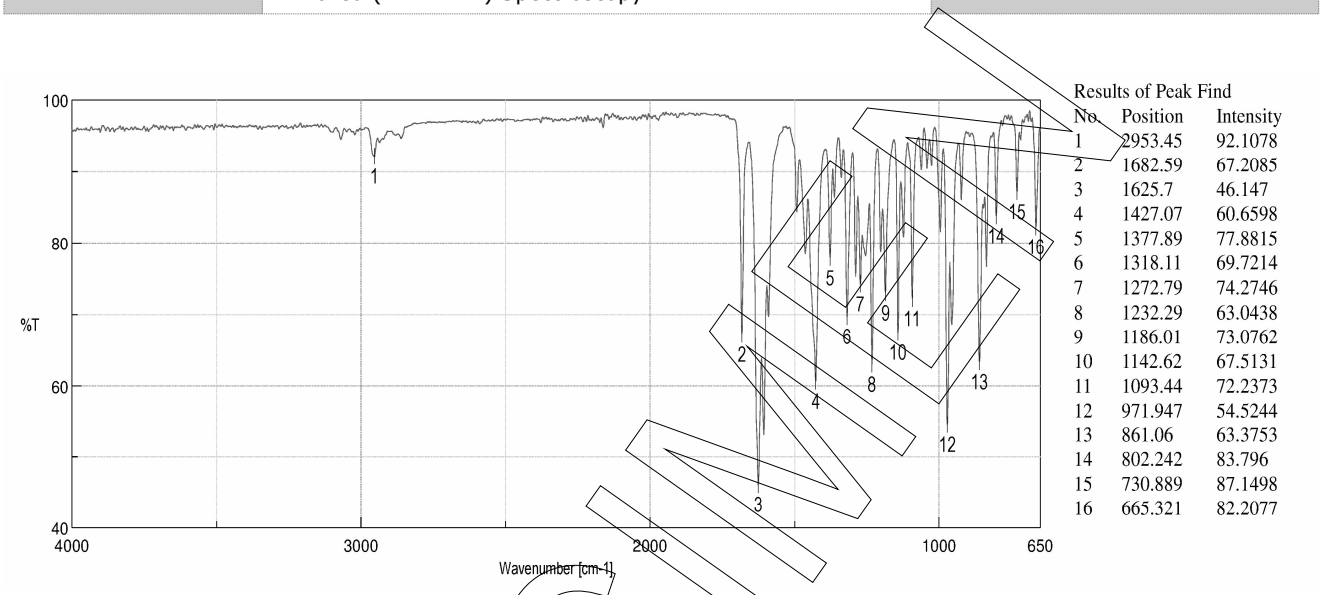
Method	Conditions	Result
^{1}H-NMR	400 MHz, DMSO-d_6	Structure confirmed



Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 268.11436	Structure confirmed



Method	Conditions	Result
IR	Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy	Structure confirmed



Results of Peak Find

No	Position	Intensity
1	2953.45	92.1078
2	1682.59	67.2085
3	1625.7	46.147
4	1427.07	60.6598
5	1377.89	77.8815
6	1318.11	69.7214
7	1272.79	74.2746
8	1232.29	63.0438
9	1186.01	73.0762
10	1142.62	67.5131
11	1093.44	72.2373
12	971.947	54.5244
13	861.06	63.3753
14	802.242	83.796
15	730.889	87.1498
16	665.321	82.2077

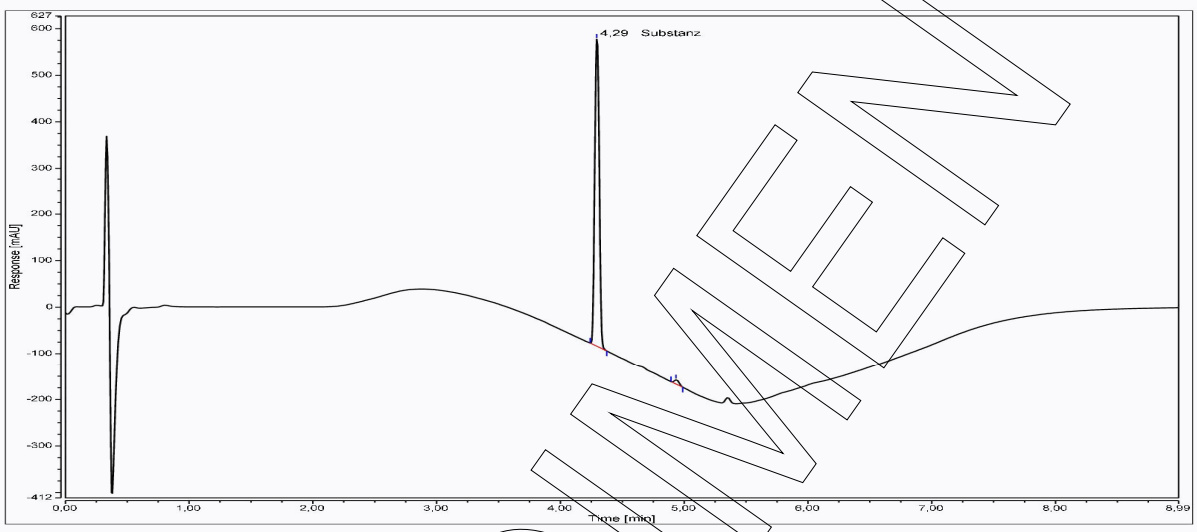
Assay

The assay of the reference material was assessed by following analyses.

Purity by High Performance Liquid Chromatography (HPLC)

HPLC Conditions:	
Column	Cortecs UPLC C18 +; 1.6 µm, 75 x 2.1 mm
Column temperature	40 °C
Detector	DAD, 200 nm
Injector	Auto 3 µl; 0.050 mg/ml in Acetonitrile
Flow rate	0.5 ml/min
Phase A	Water, 0.1 % HCOOH
Phase B	Acetonitrile, 0.1 % HCOOH
Gradient program	0-1 min A/B 98/2 1-4 min A/B to 2/98 4-5 min A/B to 98/2 5-9 min A/B 98/2 (v/v)

HPLC chromatogram and peak table



Area percent report - sorted by signal				
Pk #	**Retention time**	**Area**	**Area %**	
1	4.294	28.1527	98.57	
2	4.934	0.4096	1.43	
Totals		**28.5623**	**100.00**	

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 %. System peaks were ignored in calculation.

Result (n = 3)	98.57 %; SD = 0.01 %

Volatile content

Water content	
Method	Karl Fischer titration
Result (n = 3)	No significant amounts of water were detected (< 0.05 %).

Residual solvents	
Method	^{1}H-NMR
Result (n = 1)	Sum: 0.26 % 0.09 % n-Hexane; 0.17 % Methylene chloride

Final result

Assay "as is": 98.31 %

The assay "as is" is assessed by 100% method (mass balance) and is equivalent to the assay based on the not anhydrous and not dried substance respectively.

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 and purity is considered as relative contribution. Inorganic residues are excluded by additional tests.

Revision table

Revision	Date	Reason for revision
00	24 Jun 2021	Release of the Certificate of Analysis - initial version

Product warranties for the RM are set out in the terms and conditions of purchase.