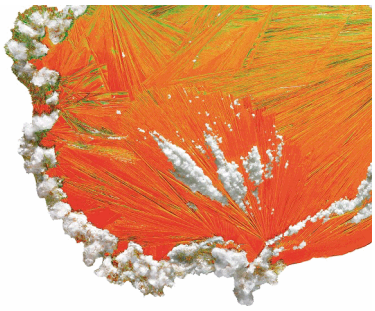


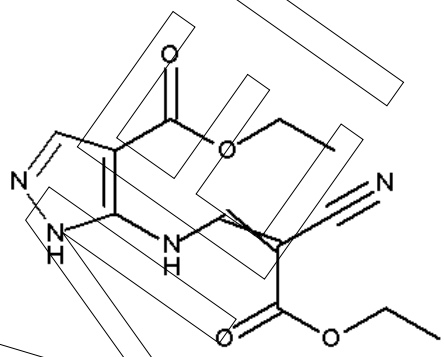
Certificate of Analysis

ISO 9001

Reference Material

Product name

Ethyl
(E/Z)-3-(2-Carbethoxy-2-cyanoethenyl)amino-1H-pyrazole-4-carboxylate



Product code	**Lot number**
MM0034.10-0025	1012414
CAS number	**Appearance**
321571-07-7	beige solid
Molecular weight	**Melting point**
278.26	247 °C (dec)
Molecular formula	**Long-term storage**
$C_{12}H_{14}N_4O_4$	2 to 8 °C, dark

Assay "as is"
98.0 %

Date of shipment: **02 Sep 2019**

Producer confirms that this reference material (RM) meets the specification detailed on this Certificate of Analysis for **two years** 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, 30 Jul 2019		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.

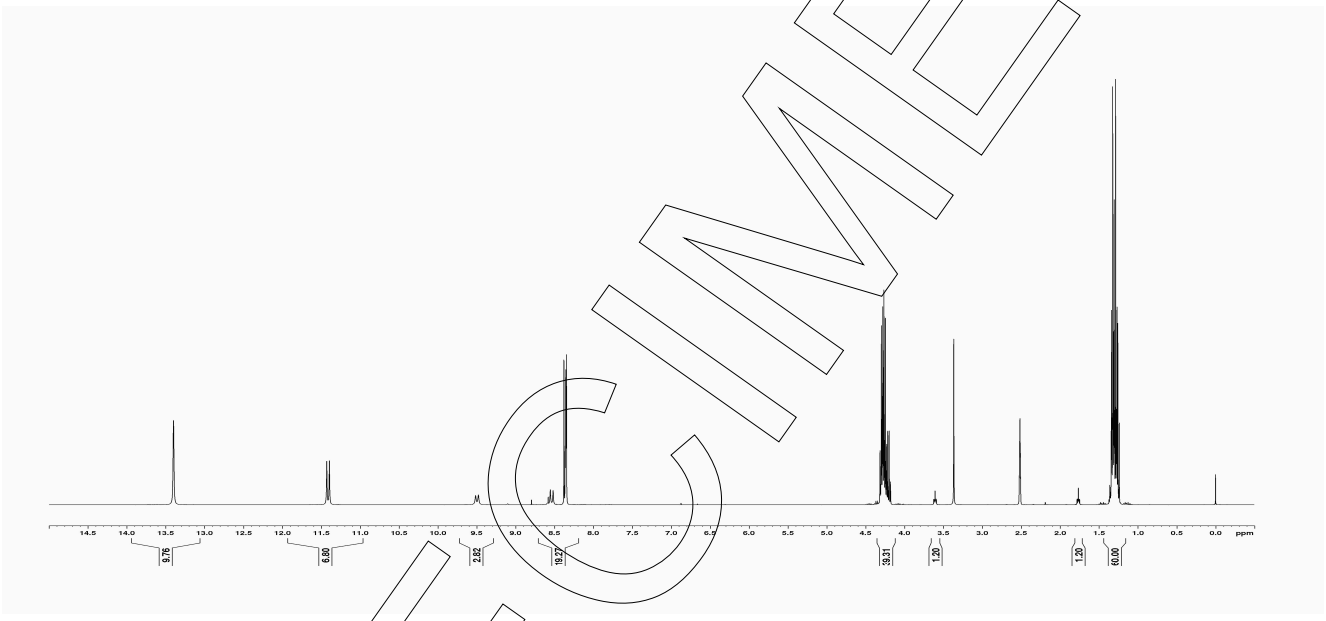
The product quality is controlled by regularly performed quality control tests (retests).

Further content

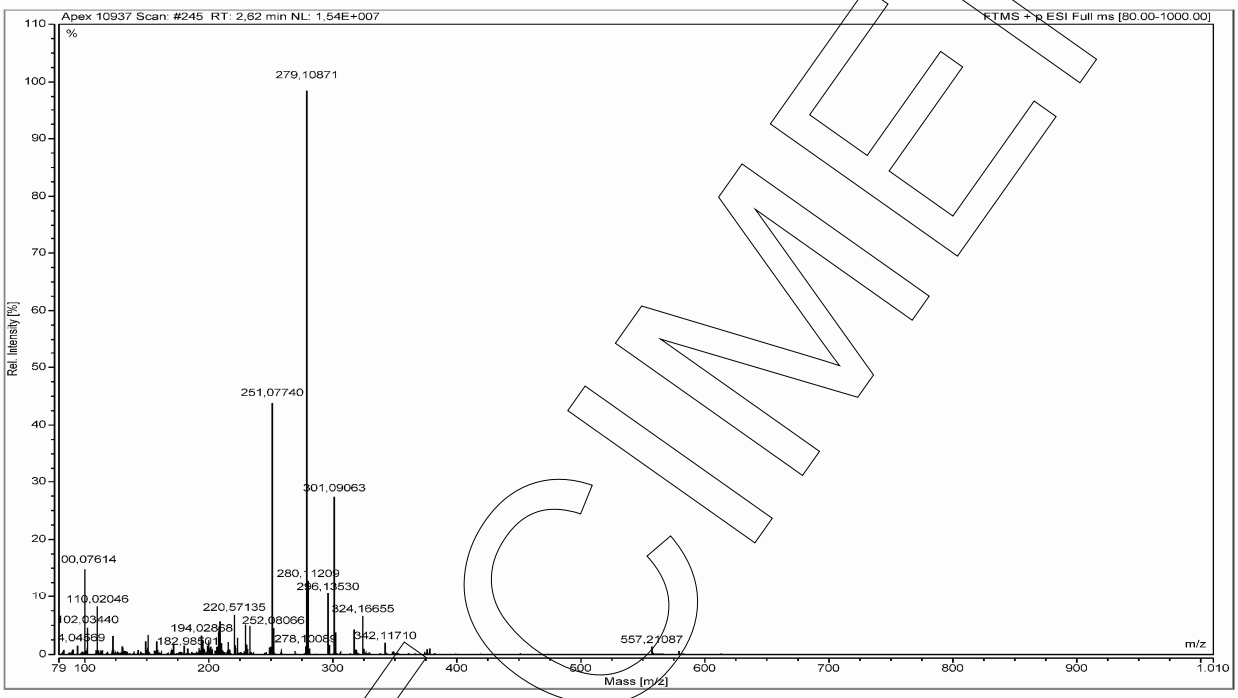
Identity

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

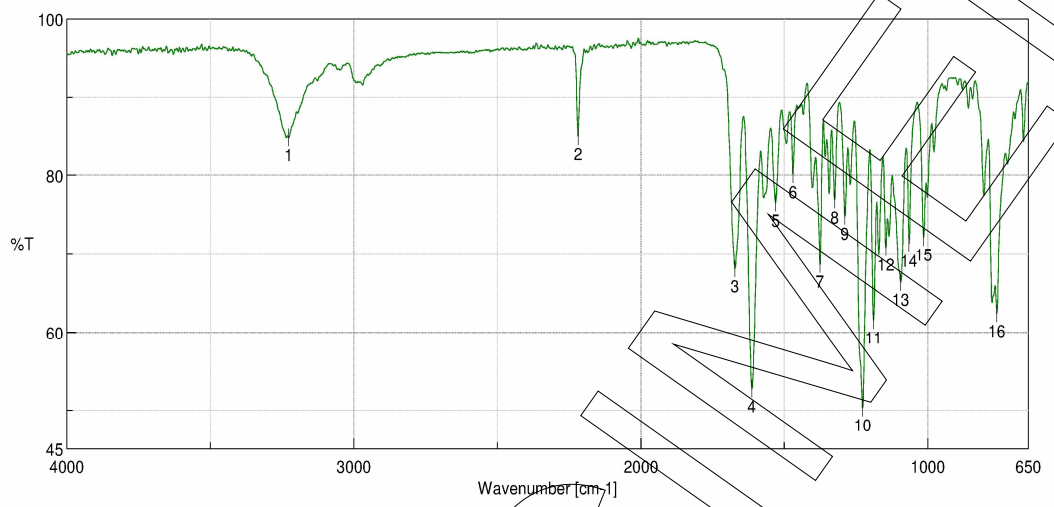
Method	Conditions	Result
^{1}H-NMR	400 MHz, DMSO-d_6	Structure confirmed



Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 279.10878	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	3226.33	84.8341
2	2218.7	85.0074
3	1671.98	68.0984
4	1613.16	52.7036
5	1530.24	76.508
6	1470.46	80.0608
7	1375.96	68.6538
8	1324.86	76.878
9	1289.18	74.8158
10	1227.47	50.2874
11	1188.9	61.4925
12	1146.47	70.8477
13	1094.4	66.3649
14	1065.48	71.3297
15	1014.37	72.1535
16	759.816	62.3983

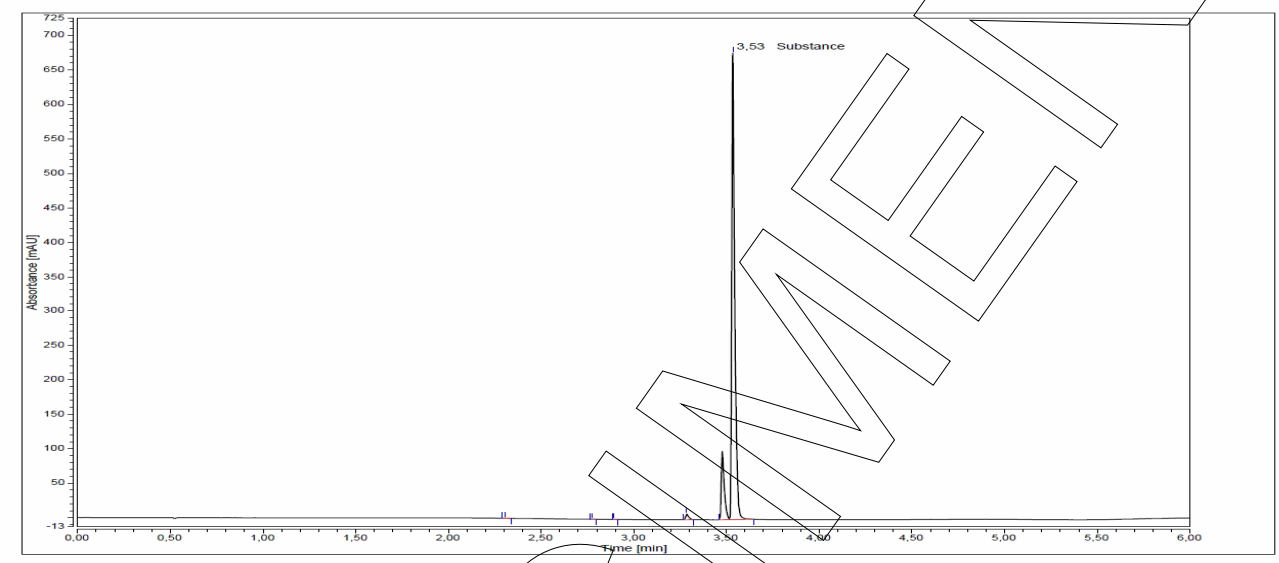
Assay

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

Purity by High Performance Liquid Chromatography (HPLC)

HPLC Conditions:	
Column	Kinetex Phenyl-Hexyl; 1.7 µm, 100 x 2.1 mm
Column temperature	40 °C
Detector	DAD, 315 nm
Injector	Auto 1 µl; 0.036 mg/ml in Acetonitrile/Water 50/50 (v/v)
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 5-6 min A/B 98/2 (v/v)

HPLC chromatogram and peak table



Area percent report - sorted by signal				
Pk #	**Retention time**	**Area**	**Area %**	
1	2.306	0.0047	0.03	
2	2.774	0.0028	0.02	
3	2.893	0.0031	0.02	
4	3.286	0.1486	1.03	
5	3.534	14.2704	98.90	
Totals		**14.4296**	**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)	98.90 %; SD = 0.01 %

Volatile content

Water content	
Method	Karl Fischer titration
Result (n = 3)	0.15 %; SD = 0.01 %

Residual solvents	
Method	^{1}H-NMR
Result (n = 1)	Sum: 0.77 % 0.77 % Tetrahydrofuran

Final result

Assay "as is": **97.99 %**

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	30 Jul 2019	Release of the Certificate of Analysis - initial version

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