

# Certificate of Analysis

ISO 9001

## Reference Material

### Product name

Imiquimod N-Oxide

### Product code

MM1158.01-0025

### CAS number

1807607-22-2

### Molecular weight

256.303

### Molecular formula

C<sub>14</sub>H<sub>16</sub>N<sub>4</sub>O

### Lot number

1023035

### Appearance

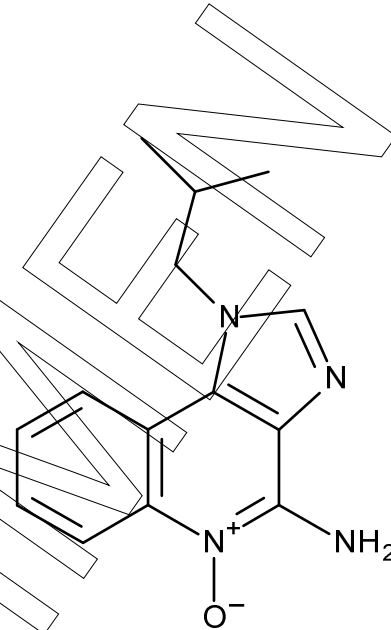
beige solid

### Melting point

> 260 °C (dec.)

### Long-term storage

2 to 8 °C, dark  
hygroscopic



Assay "as is"  
92.8 %

Date of shipment: **02 Feb 2021**

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.

<b>Release by:</b>	<b>Date of Release:</b>		Product Release
Dr. Sabine Schröder	Luckenwalde, 29 Jan 2021		



## 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

Assay

Final result

Revision table

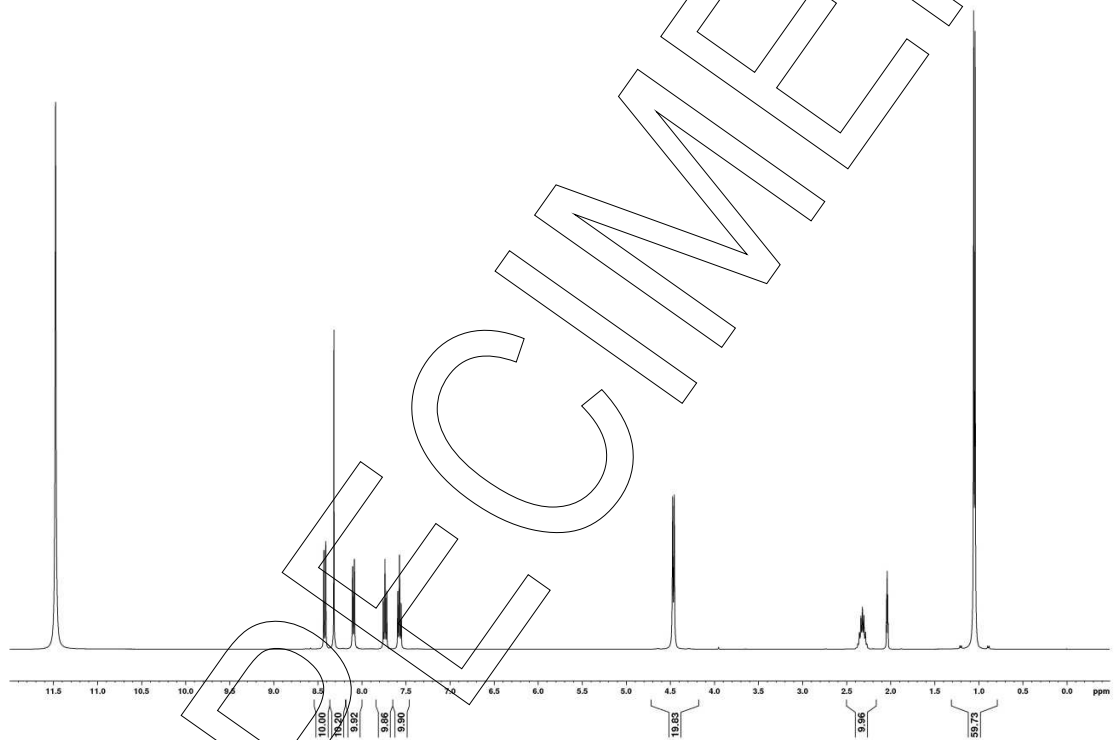
SPECIMEN



## Identity

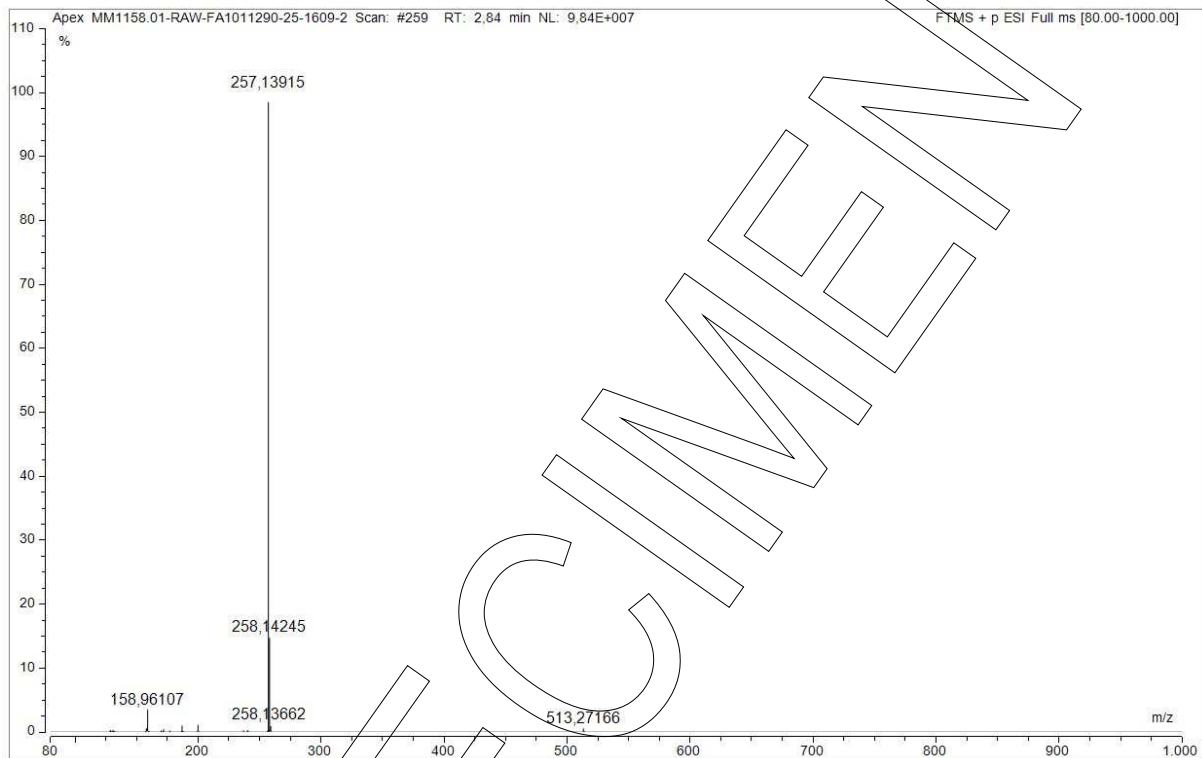
The identity of the reference material was established by following analyses.

Method	Conditions	Result
<sup>1</sup> H-NMR	400 MHz, CD <sub>3</sub> COOD	Structure confirmed





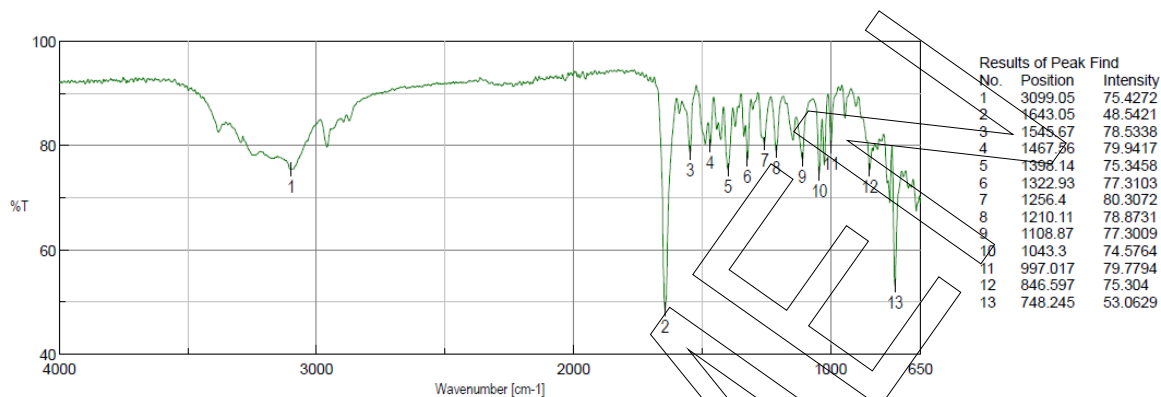
Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 257.13969	Structure confirmed



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



## Assay

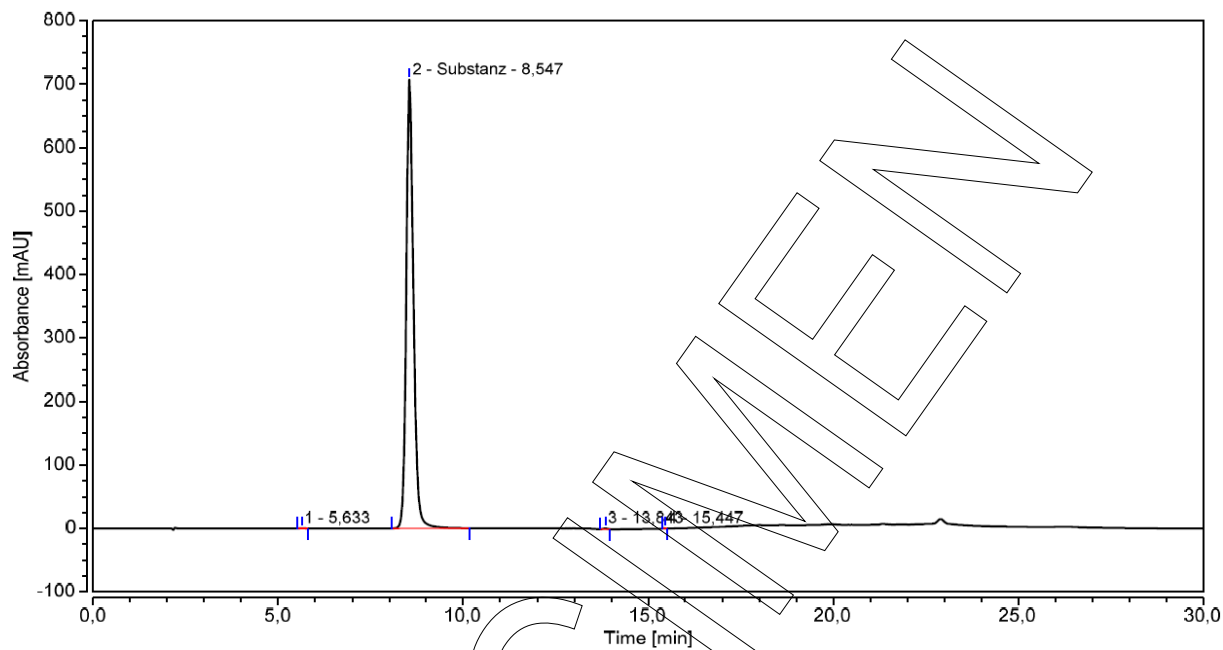
The assay of the reference material was assessed by following analyses.

## Purity by High Performance Liquid Chromatography (HPLC)

HPLC conditions:	
Column	Hypersil Gold C18; 5 µm, 150 x 4.6 mm
Column temperature	40 °C
Detector	DAD, 245 nm
Injector	Auto 2 µl; 0.112 mg/ml in Water/Acetonitrile 50/50 (v/v)
Flow rate	1.0 ml/min
Phase A	Water, 0.1 % H <sub>3</sub> PO <sub>4</sub>
Phase B	Acetonitrile, 0.1 % H <sub>3</sub> PO <sub>4</sub>
Gradient program	0-10 min A/B 82/18 10-15 min A/B to 30/70 15-20 min A/B 30/70 20-23 min A/B to 82/18 23-30 min A/B 82/18 (v/v)



## HPLC chromatogram and peak table



Area percent report - sorted by signal			
Pk #	Retention time	Area	Area %
1	5.633	0.037	0.02
2	8.547	158.656	99.85
3	13.843	0.173	0.11
4	15.447	0.033	0.02
<b>Totals</b>		<b>158.898</b>	<b>100.00</b>

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.

<b>Result (n = 3)</b>	99.86 %; SD = 0.01 %
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## Volatile content

Water content	
<b>Method</b>	Karl Fischer titration
<b>Result</b> (n = 3)	7.08 %; SD = 0.11 %

Residual solvents	
<b>Method</b>	<sup>1</sup> H-NMR
<b>Result</b> (n = 1)	No significant amounts of residual solvents were detected (< 0.05 %).

## Final result

**Assay "as is": 92.79 %**

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	29 Jan 2021	Release of the Certificate of Analysis – initial version

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