



Mikromol™



## Certificate of Analysis

ISO 9001

### Reference Material

#### Product name

6 $\alpha$ ,9-Difluoro-11 $\beta$ -hydroxy-16 $\alpha$ -methyl-3-oxo-17-(propanoylox  
y)androsta-1,4-diene-17 $\beta$ -carboxylic Acid

#### Product code

MM1244.01-0025

#### CAS number

65429-42-7

#### Molecular weight

452.49

#### Molecular formula

C<sub>24</sub>H<sub>30</sub>F<sub>2</sub>O<sub>6</sub>

#### Lot number

1026080

#### Appearance

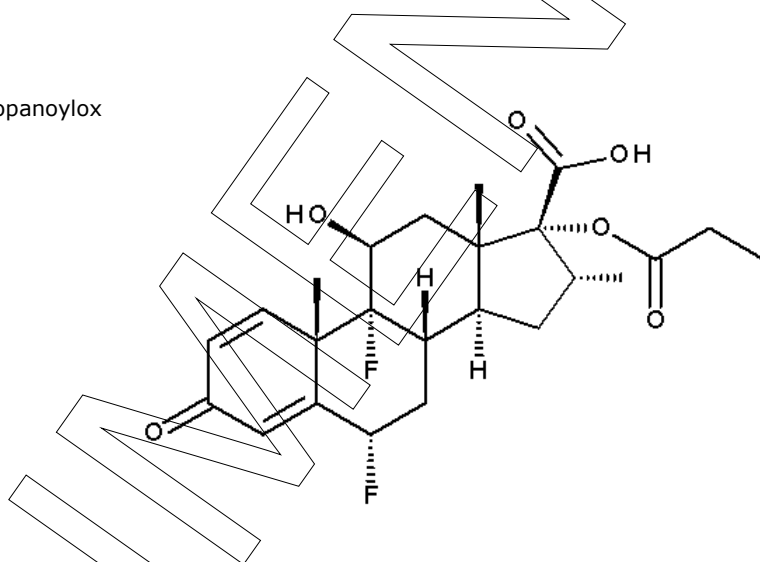
white solid

#### Melting point (DSC)

226 °C

#### Long-term storage

2 to 8 °C, dark



Assay "as is"  
94.8 %

Date of shipment:

13 Sep 2019

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, 06 Sep 2019 |  |                 |



**Mikromol<sup>TM</sup>**

## 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

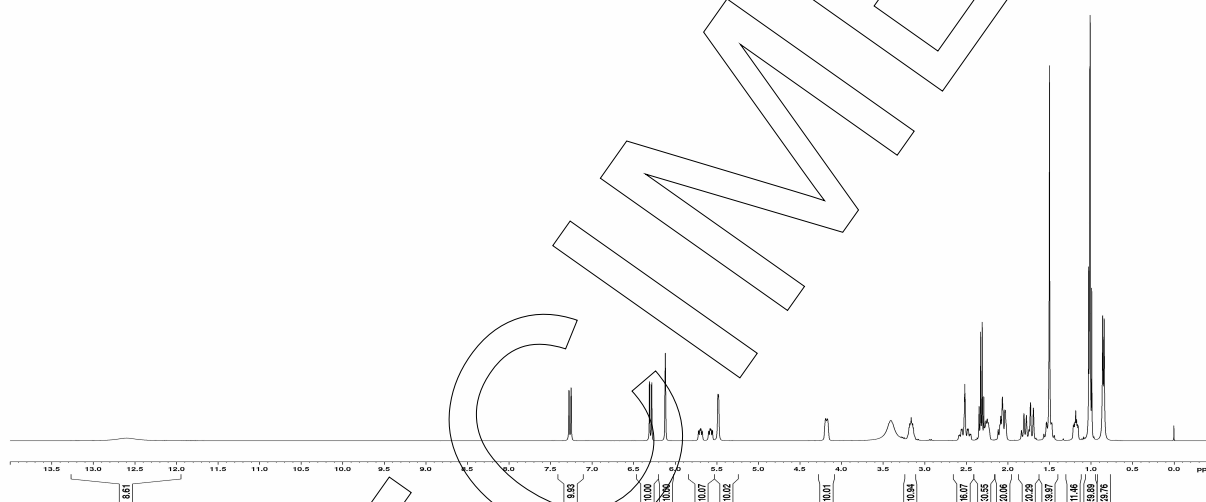
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## Identity

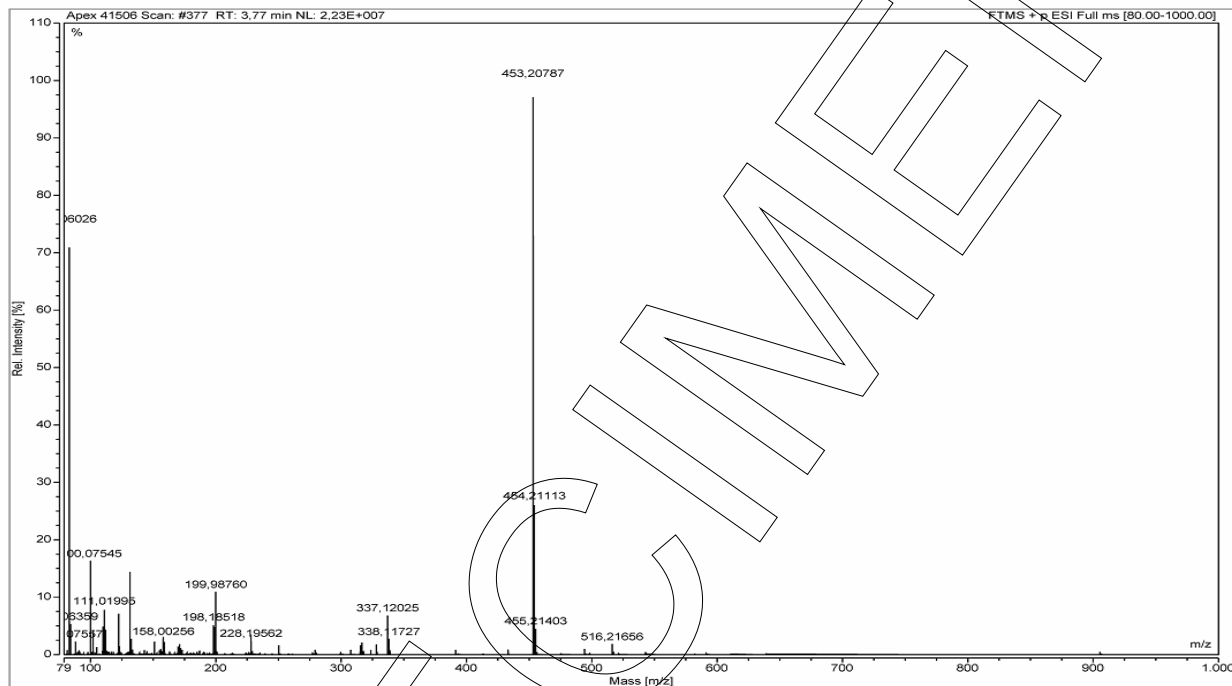
The identity of the reference material was established by following analyses.

| Method             | Conditions                   | Result              |
|--------------------|------------------------------|---------------------|
| <sup>1</sup> H-NMR | 400 MHz, DMSO-d <sub>6</sub> | Structure confirmed |



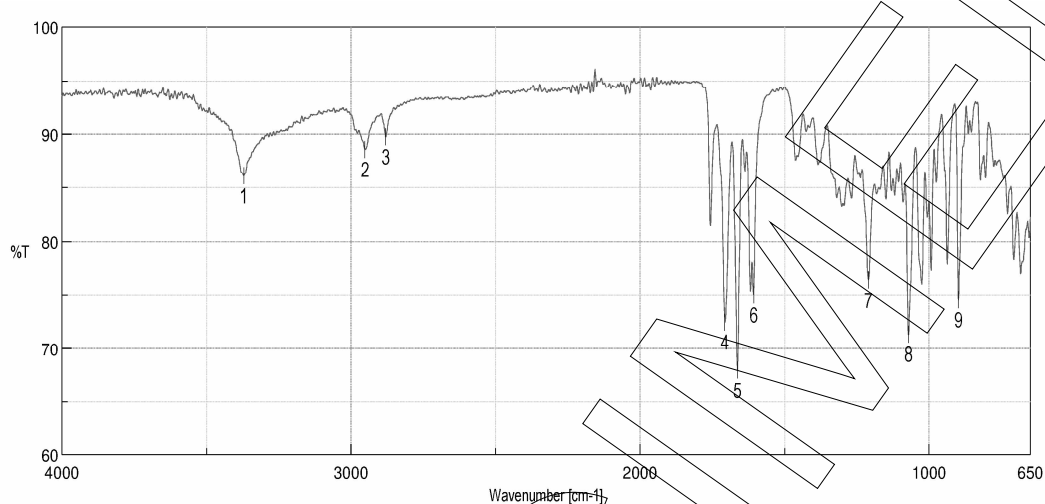


| Method | Conditions   | Result              |
|--------|--|---------------------|
| MS     | 3.5 kV ESI+; capillary temperature: 269 °C<br>Theoretical value: 453.20832 | Structure confirmed |





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



Results of Peak Find

| No. | Position | Intensity |
|-----|----------|-----------|
| 1   | 3370.96  | 86.0763   |
| 2   | 2951.52  | 88.5415   |
| 3   | 2880.17  | 89.73     |
| 4   | 1706.69  | 72.4552   |
| 5   | 1663.3   | 67.9299   |
| 6   | 1607.38  | 74.9961   |
| 7   | 1210.11  | 76.3635   |
| 8   | 1071.26  | 71.3043   |
| 9   | 898.666  | 74.5225   |



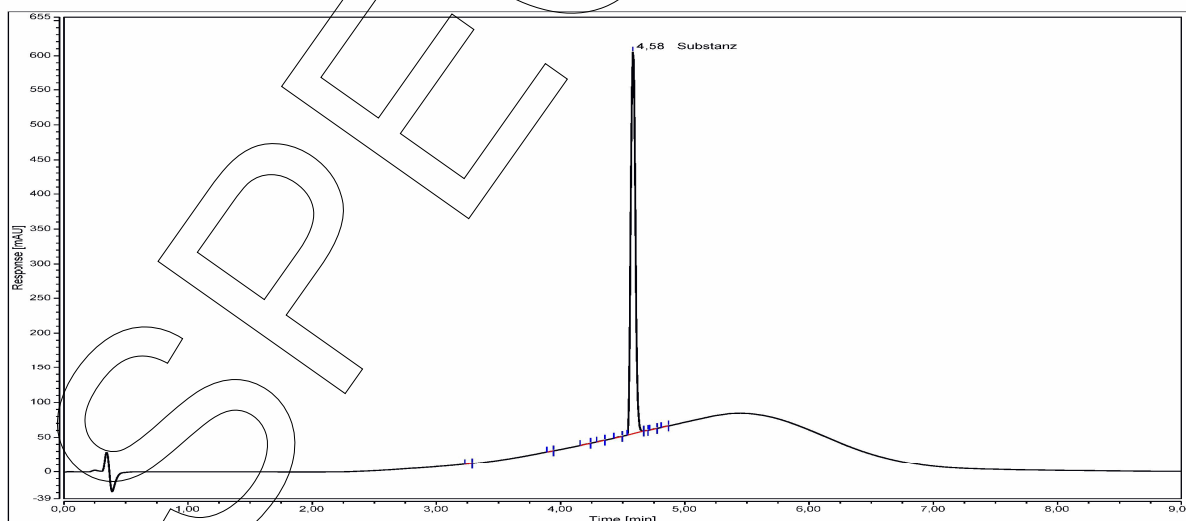
## Assay

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, 240 nm  |
| Injector           | Auto 5.00 µl; 0.077 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<br>1-4 min A/B to 2/98<br>4-5 min A/B to 98/2<br>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      | 3.289          | 0.0021  | 0.01   |
| 2      | 3.942          | 0.0020  | 0.01   |
| 3      | 4.242          | 0.0105  | 0.04   |
| 4      | 4.355          | 0.0131  | 0.05   |
| 5      | 4.495          | 0.0167  | 0.07   |
| 6      | 4.582          | 23.7892 | 99.69  |
| 7      | 4.702          | 0.0029  | 0.01   |
| 8      | 4.775          | 0.0140  | 0.06   |
| 9      | 4.869          | 0.0120  | 0.05   |
| Totals |                | 23.8625 | 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)** 99.70 %; SD = 0.01 %

## Volatile content

### Water content

**Method** Karl Fischer titration  
**Result (n = 3)** 4.44 %; SD = 0.21 %

### Residual solvents

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



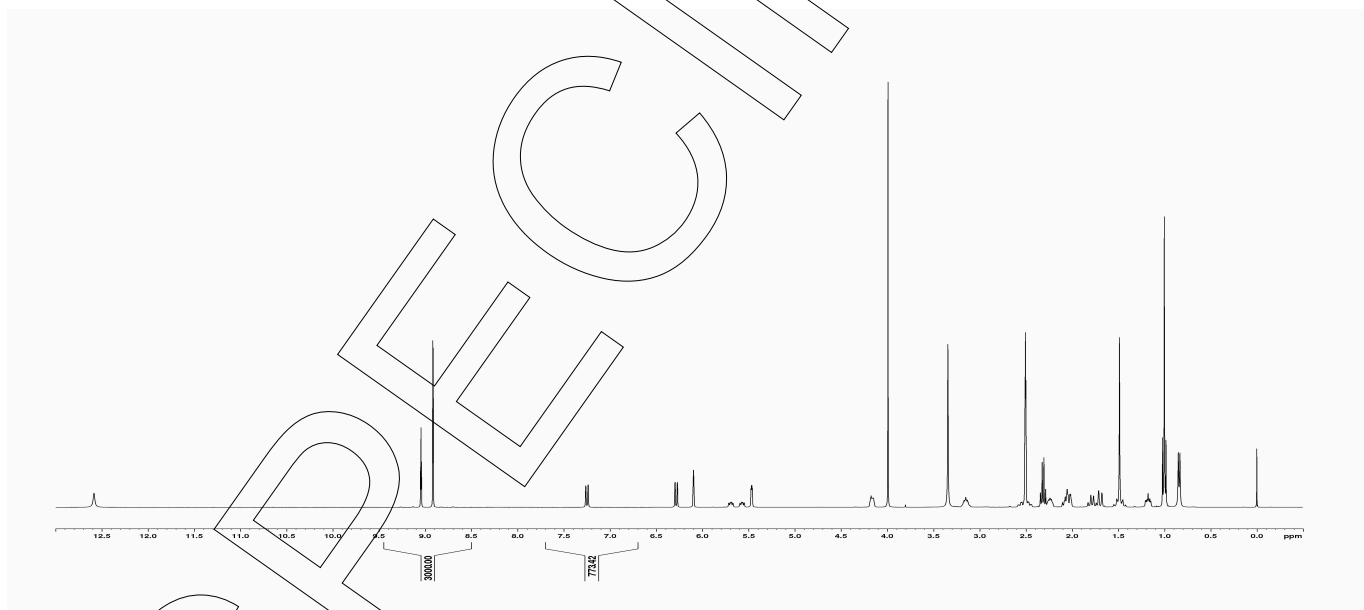
## Final result

**Assay "as is": 94.81 %**

The assay "as is" is assessed by quantitative NMR spectroscopy and is equivalent to the assay based on the not anhydrous and not dried substance respectively.

| Method: Value assigning technique - quantitative NMR spectroscopy |  |
|---|--|
| Conditions  | 400 MHz, DMSO-d <sub>6</sub>   |
| Internal standard   | Methyl 3,5-dinitrobenzoate (certified reference material), signal 8.5 - 9.5 ppm, 3 H |
| Result (mass fraction, n = 3)                                     | 94.81 %; SD = 0.15 %   |

Quantitative NMR spectrum







**Mikromol<sup>TM</sup>**

## Revision table

| Revision | Date        | Reason for revision                                      |
|----------|-------------|--|
| 00       | 06 Sep 2019 | Release of the Certificate of Analysis - initial version |

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

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