

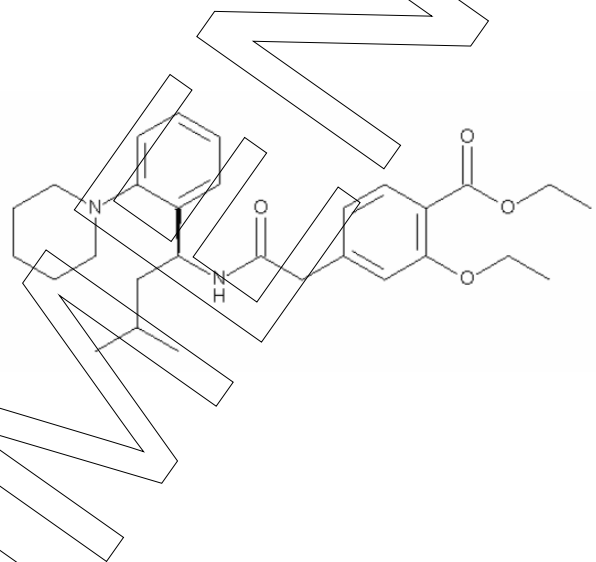
Certificate of Analysis

ISO 9001

Reference Material

Product name

Ethyl
2-Ethoxy-4-[2-[[[(1S)-3-methyl-1-[2-(piperidin-1-yl)phenyl]butyl]amino]-2-oxoethyl]benzoate (Repaglinide Ethyl Ester)



Product code

MM0944.05

Lot number

1028713

CAS number

147770-06-7

Appearance

white solid

Molecular weight

480.64

Melting point (DSC)

125 °C

Molecular formula

C₂₉H₄₀N₂O₄

Long-term storage

2 to 8 °C, dark

Assay "as is"
97.7 %

Date of shipment:

02 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.

Release by:	Date of Release:		Product Release
Dr. Sabine Schröder	Luckenwalde, 20 Aug 2019		



Mikromol™

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

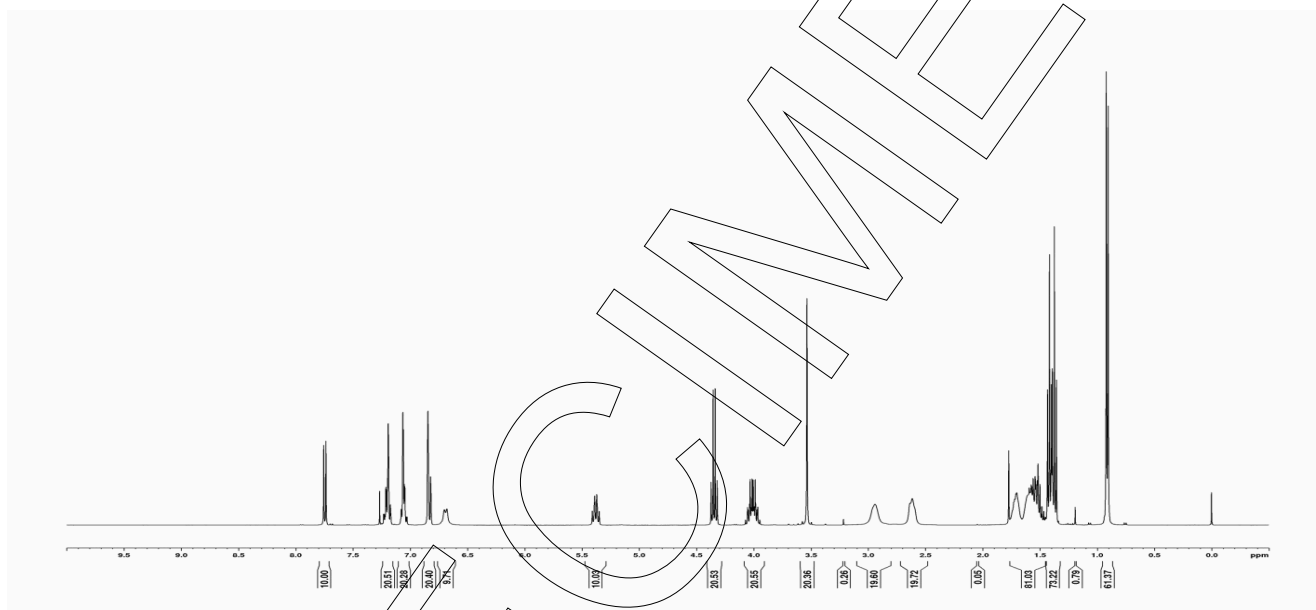
SPECIMEN



Identity

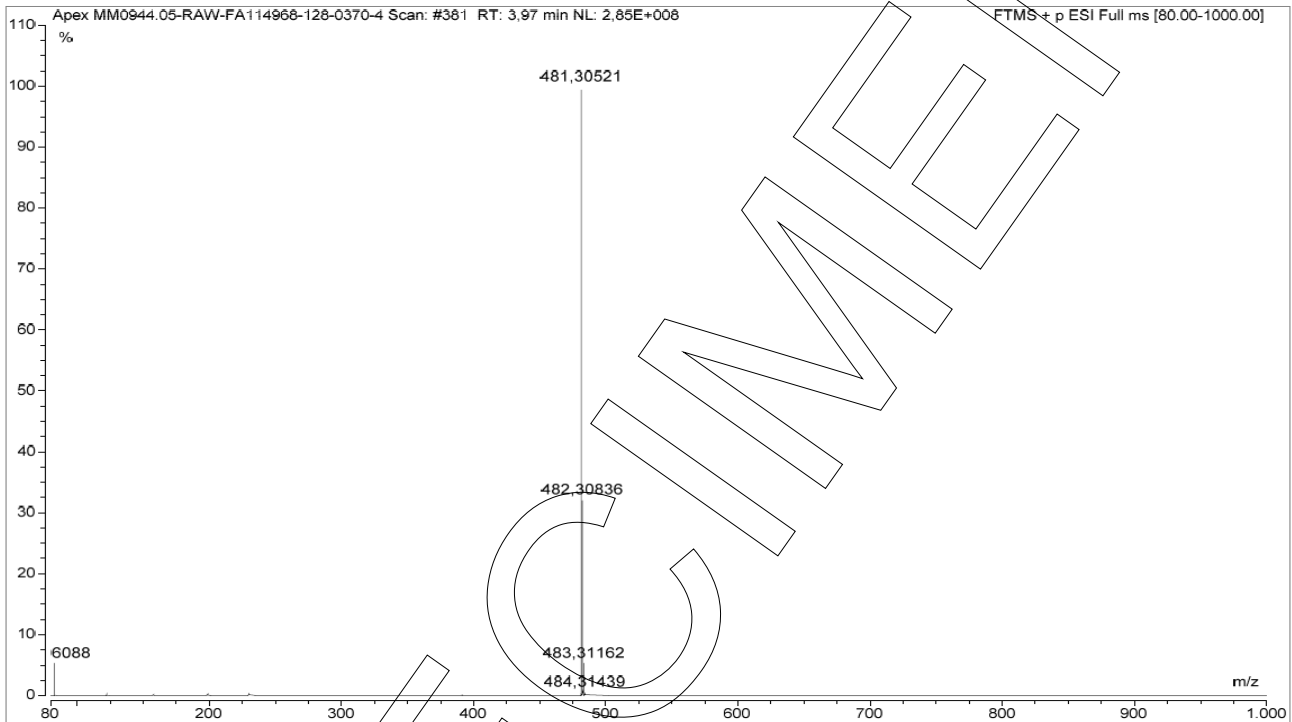
The identity of the reference material was established by following analyses.

Method	Conditions	Result
¹ H-NMR	400 MHz, CDCl ₃	Structure confirmed





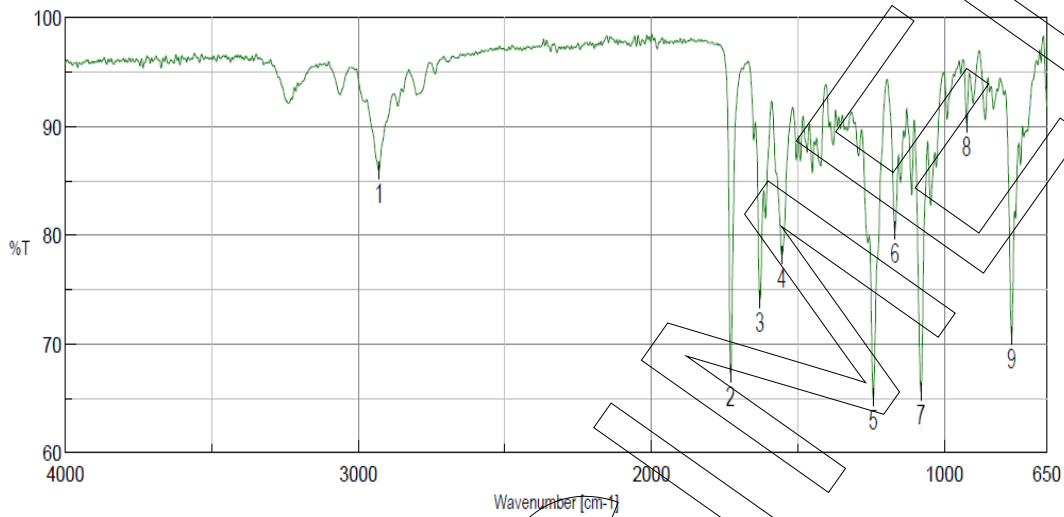
Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 481.30608	Structure confirmed



SPLEIN



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



No.	Position	Intensity
1	2929.34	85.8849
2	1728.87	67.2671
3	1628.59	74.0429
4	1554.34	78.1639
5	1241.93	65.0868
6	1168.65	80.4401
7	1078.98	65.5885
8	922.771	90.1888
9	770.423	70.7022



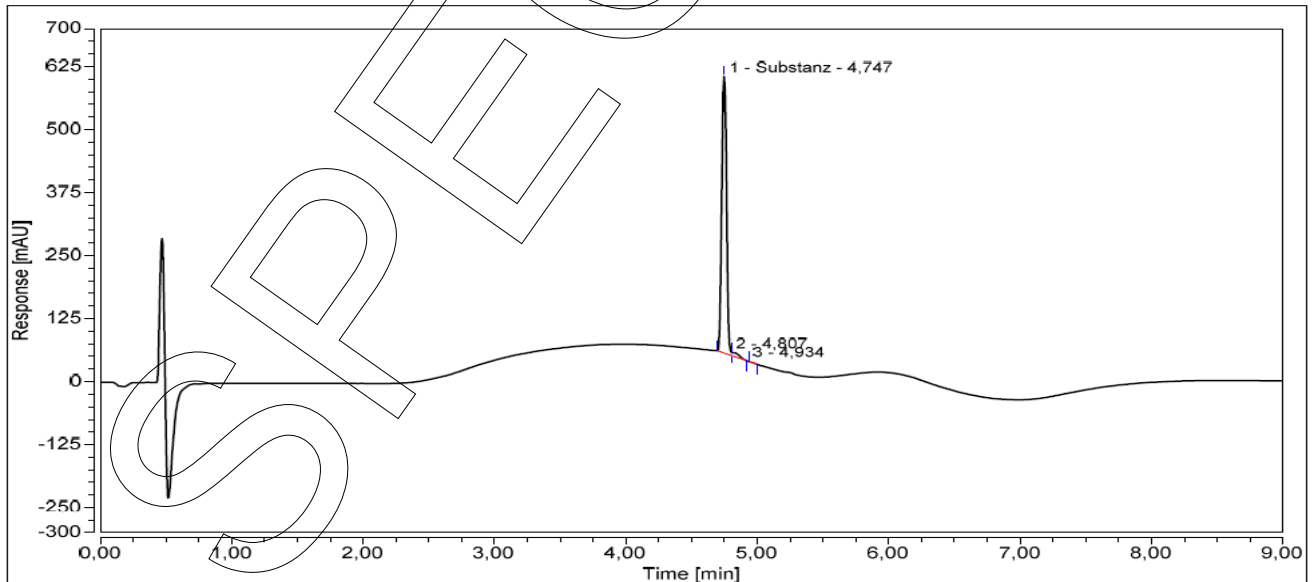
Assay

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, 254 nm
Injector	Auto 3.00 µl; 0.051 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	4.747	23.378	97.91
2	4.807	0.479	2.01
3	4.934	0.021	0.09
Totals		23.878	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) 97.91 %; SD = 0.01 %

Volatile content

Water content

Method Karl Fischer titration
Result (n = 3) 0.08 %; SD = 0.01 %

Residual solvents

Method ¹H-NMR
Result (n = 1) Sum: 0.19 %
0.16 % tert-Butyl methyl ether; 0.03 % Ethyl acetate



Final result

Assay "as is": 97.65 %

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	20 Aug 2019	Release of the Certificate of Analysis - initial version

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