

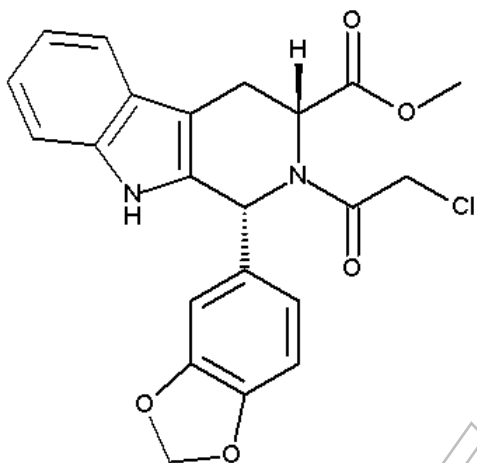


Certificate of Analysis

Reference Standard

Chloropretadafil (Methyl

(1R,3R)-1-(1,3-benzodioxol-5-yl)-2-(chloroacetyl)-2,3,4,9-tetrahydro-1H-pyrido-[3,4-b]indole-3-carboxylate)



Molecular Formula: C₂₂H₁₉ClN₂O₅
Molecular Weight: 426.85
CAS Number: 171489-59-1

Catalogue Number: LGCFOR0806.11
Lot Number: 64565
Long-term Storage: -18 °C, dark
Appearance: light yellow solid
Melting Point: 222 °C (dec.)
Assay 'as is': 99.9 %

Date of shipment: **2016-May-20**

This certificate is valid one year from the date of shipment provided the substance is stored under the recommended conditions unopened in the original container.

LGC Quality ISO 9001:2008
DQS 102448 QM08

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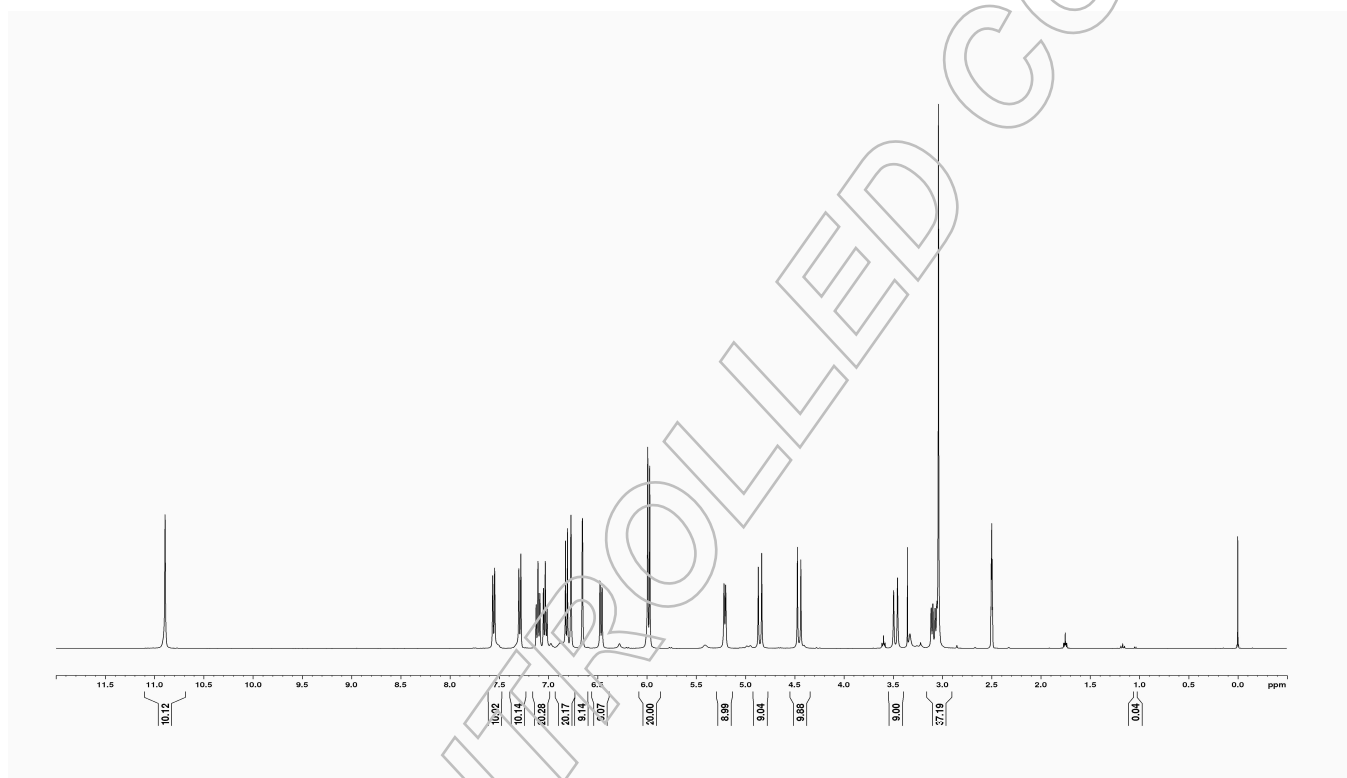


I. Identity

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

1a. ¹H-NMR Spectrum

Conditions: 400 MHz, DMSO-d₆



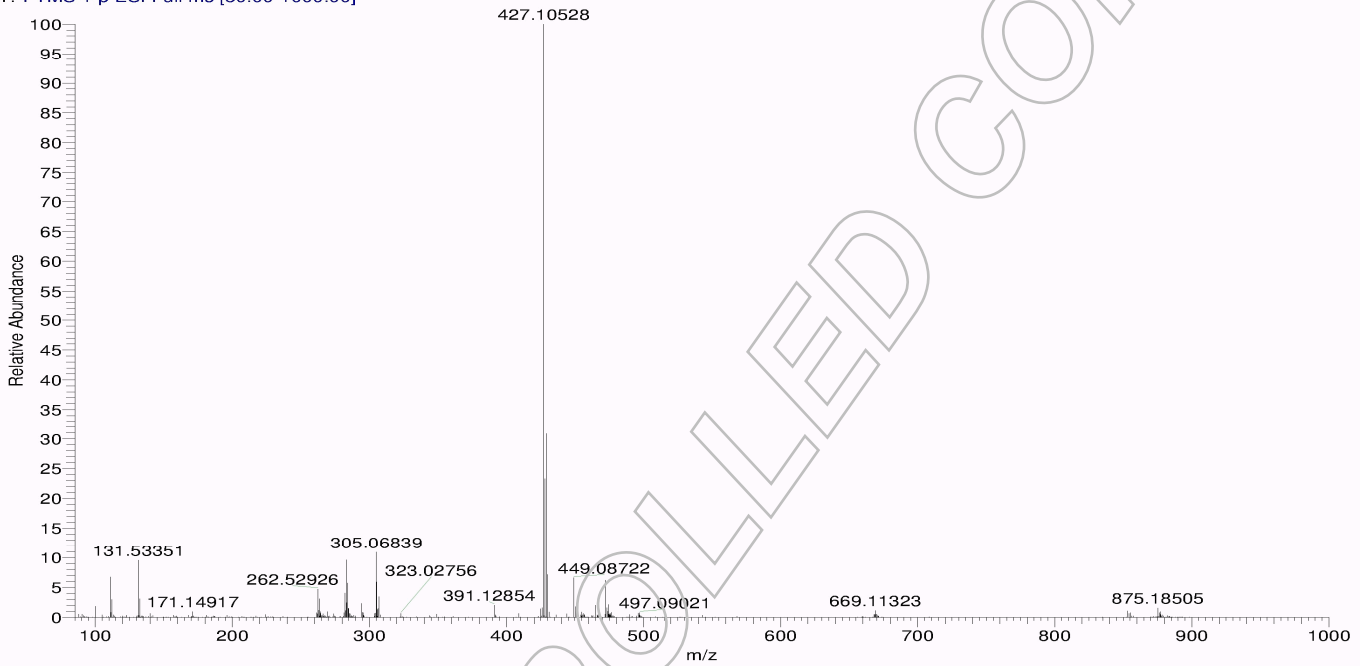
The structure is confirmed by the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

Method: HRMS; 3.5 kV ESI+; capillary temperature: 269 °C

6791 #291 RT: 2.95 AV: 1 SB: 286 0.44-6.02 , 0.01-0.38 NL: 5.19E7
T: FTMS + p ESI Full ms [80.00-1000.00]



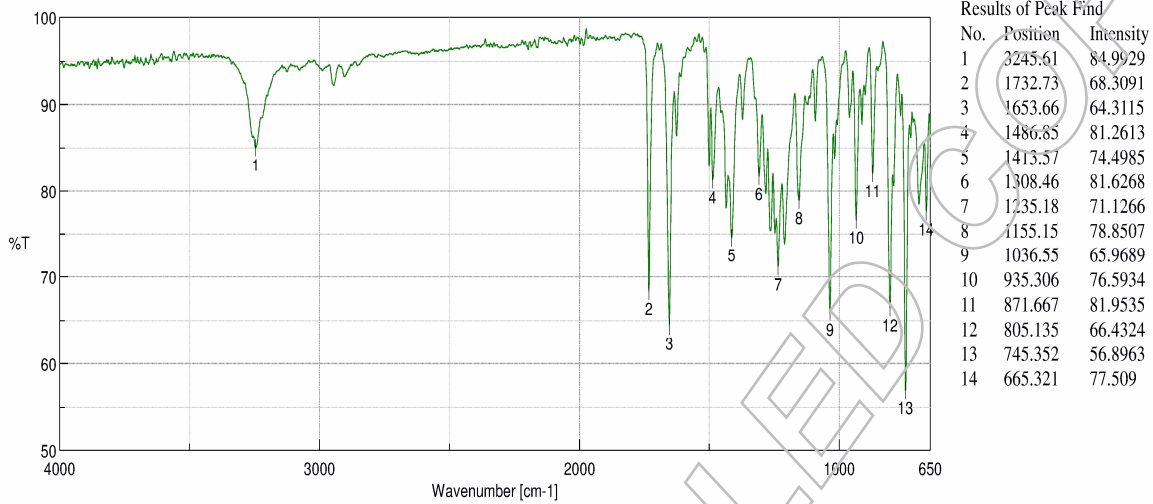
Theoretical value: 427.10552

The signal of the MS spectrum is consistent with the theoretical value and its interpretation is consistent with the structural formula.



Ic. IR Spectrum

Method: Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy



The signals of the IR spectrum and their interpretation are consistent with structural formula.



II. Purity

Ila. High Performance Liquid Chromatography (HPLC)

The purity of the reference substance was analysed by high performance liquid chromatography (HPLC).

HPLC Conditions:

Column:

Cortecs UPLC C18 +
1.6 µm, 75 x 2.1 mm

Conditions:

0.50 ml/min, 40 °C

mob. Phase A: Water, 0.1 % HCOOH

mob. Phase B: Acetonitrile, 0.1 % HCOOH

0-1 min A/B 98/2

1-4 min A/B to 2/98

4-5 min A/B to 98/2

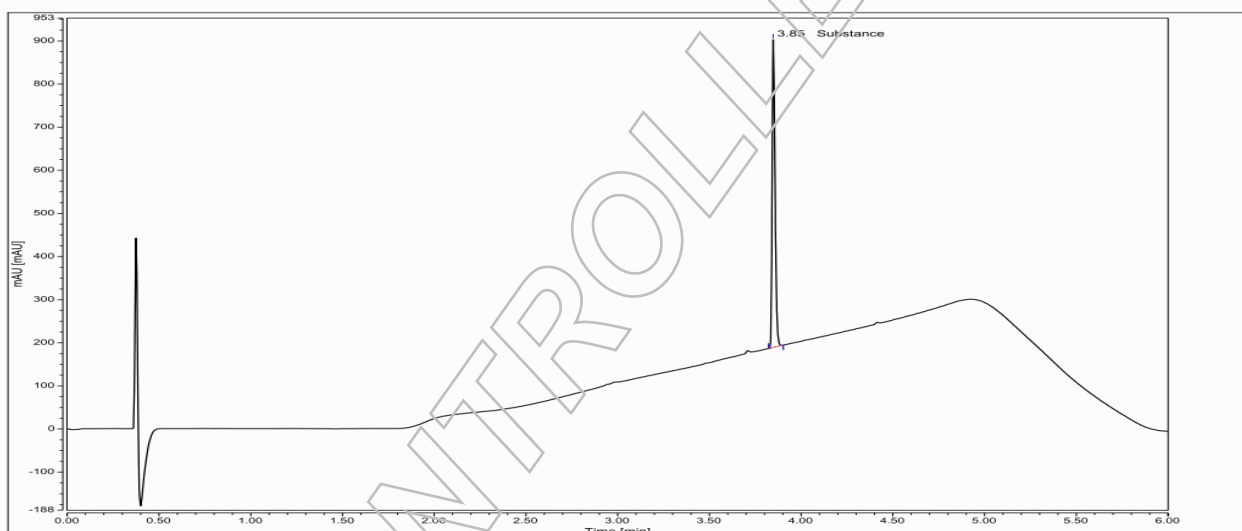
5-6 min A/B 98/2 (v/v)

Detector:

DAD
230 nm

Injector:

Auto
2 µl; 0.040 mg/ml in
Acetonitrile



Area Percent Report - Sorted by Signal

PK#	Retention Time	Area	Area %
1	3.848	13.0073	100.00
Totals		13.0073	100.00

For the calculation the system peaks were ignored. The content of the analyte was determined as the ratio of the peak area of the analyte and the cumulative areas of the purities, added up to 100 %.



Results:

Average	100.00 %
Number of results	n=3
Standard deviation	< 0.01 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average	0.06 %
Number of results	n=3
Standard deviation	< 0.01 %

IIc. Residual Solvents

Method: ¹H-NMR

No significant amounts of residual solvents were detected (< 0.05 %).

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III. Final Result

Chromatographic purity (HPLC)	100.00 %
Water content	0.06 %
Residual solvents	No significant amounts of residual solvents were detected (< 0.05 %)
Assay (100 % method)¹	99.94 %

The assay is assessed to be 99.9 % 'as is'

The assay 'as is' is equivalent to the assay based on the not anhydrous and not dried substance respectively.

Release Date:

Luckenwalde, 2015-February-06

Signed:

Dr. Sabine Schröder
Product Release

¹ 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, purity is considered as relative contribution