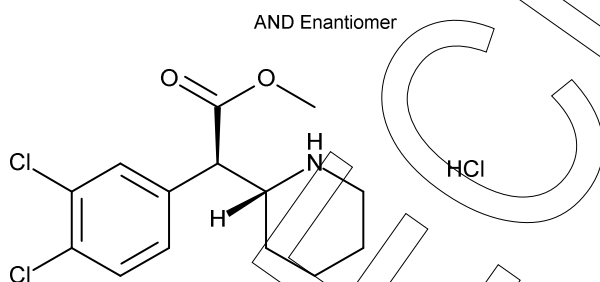




Certificate of Analysis

Reference Standard

3,4-Dichloromethylphenidate Hydrochloride
(3,4-CTMP HCl; Methyl (2RS)-3,4-Dichloro-
phenyl[(2RS)-piperidin-2-yl]acetate
Hydrochloride)



Molecular Formula: $C_{14}H_{17}Cl_2NO_2 \cdot HCl$
Molecular Weight: 338.66
CAS Number: 214149-42-5

Catalogue Number: LGCFOR1031.13
Lot Number: 451878
Long-term Storage: 2 to 8 °C, dark
Appearance: white solid
Melting Point (DSC): 223 °C
Assay 'as is': 99.8 %

Date of shipment: **2020-November-30**

This certificate is valid for two years 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

LGC GmbH, Louis-Pasteur-Str. 30, D-14943 Luckenwalde, Germany

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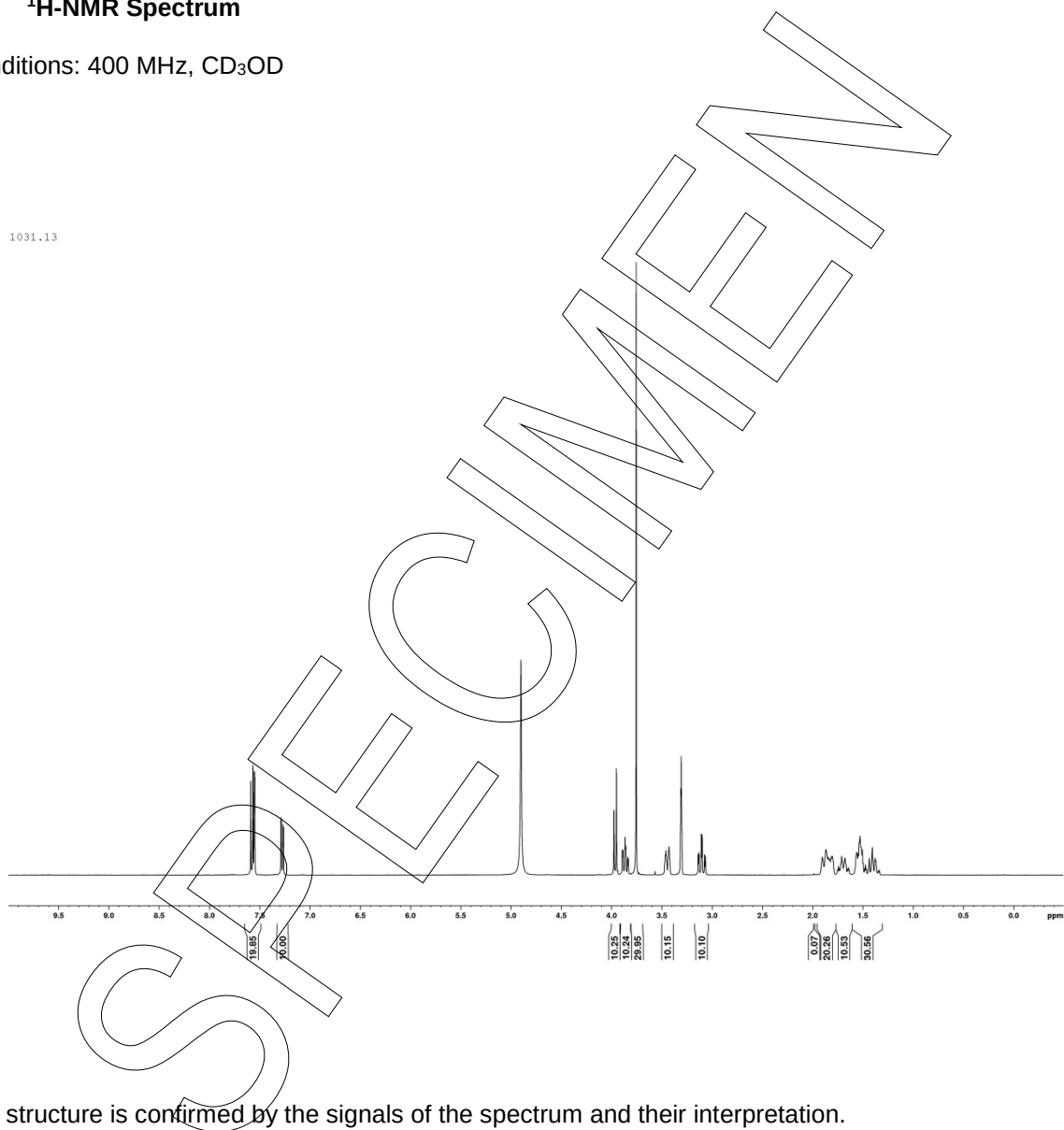


I. Identity

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

Ia. ^1H -NMR Spectrum

Conditions: 400 MHz, CD_3OD

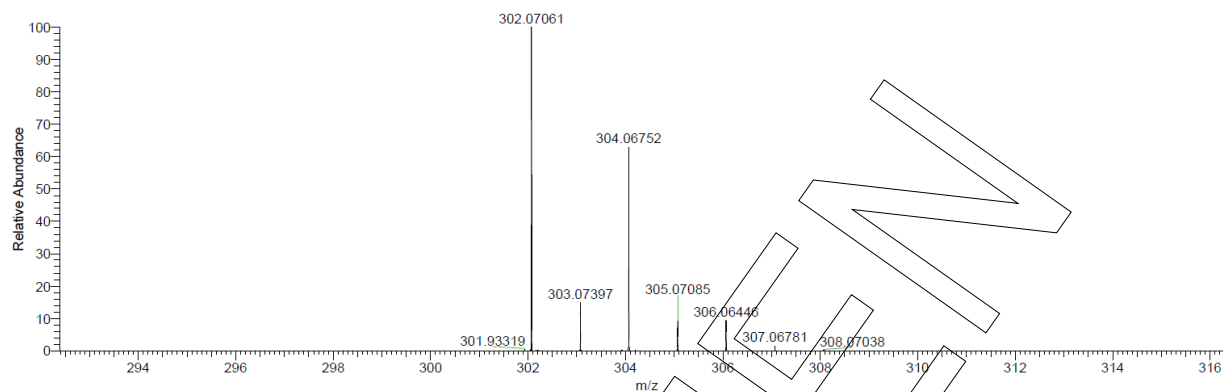


The structure is confirmed by the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

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

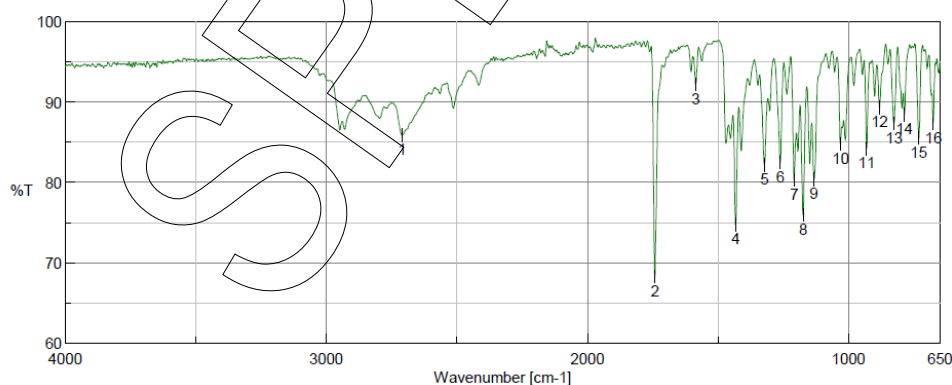


Theoretical value: 302.07091

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



Results of Peak Find		
No.	Position	Intensity
1	2709.5	85.8204
2	1741.41	68.3107
3	1585.2	92.1483
4	1431.89	74.7006
5	1321.96	82.1645
6	1261.22	82.4335
7	1208.18	80.2682
8	1174.44	75.8991
9	1132.01	80.3565
10	1030.77	84.7333
11	930.485	84.1896
12	881.309	89.2868
13	826.348	87.3055
14	785.85	88.4268
15	730.889	85.5124
16	675.928	87.3725

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



II. Purity

IIa. High Performance Liquid Chromatography (HPLC)

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

HPLC Conditions:

Column:

Hypersil Gold C18
5 µm, 150 x 4.6 mm

Conditions:

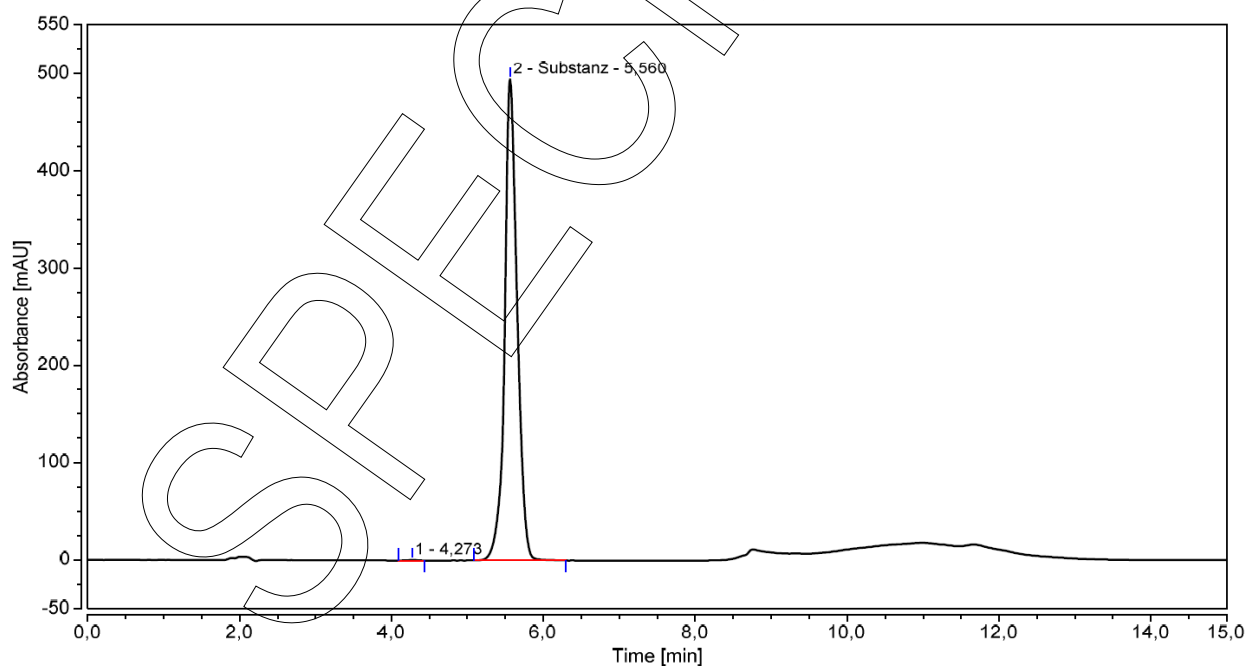
1.0 ml/min, 40 °C
0-5 min Water/Acetonitrile 70/30
5-8 min Water/Acetonitrile to 20/80
8-10 min Water/Acetonitrile to 70/30
10-15 min Water/Acetonitrile 70/30 (v/v);
0.1 % H₃PO₄

Detector:

DAD
215 nm

Injector:

Auto
3 µl; 0.232 mg/ml in
Methanol





Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	4.273	0.079	0.08
2	5.560	93.791	99.92
Totals		93.870	100.00

For the calculation the system peaks were ignored. 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 %.

Results:

Average 99.92 %
Number of results n=3
Standard deviation 0.01 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average 0.08 %
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 %).



III. Final Result

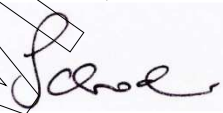
Chromatographic purity (HPLC)	99.92 %
Water content	0.08 %
Residual solvents	No significant amounts of residual solvents were detected (< 0.05 %).
Assay (100 % method)¹	99.84 %

The assay is assessed to be 99.8 % 'as is'

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

Release Date:

Luckenwalde, 2018-05-16



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.

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