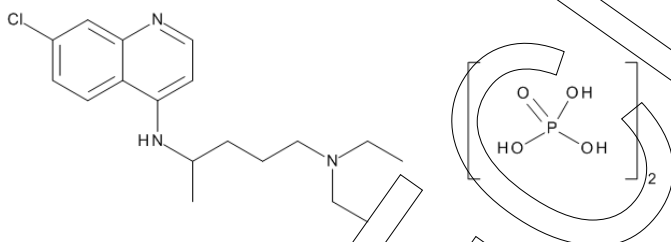




Certificate of Analysis

Reference Standard

Chloroquine Phosphate



Molecular Formula: $C_{18}H_{26}ClN_3 \cdot 2H_3PO_4$
Molecular Weight: 515.86
CAS Number: 50-63-5

Catalogue Number: LGCFOR1055.00
Lot Number: 1048793
Long-term Storage: 2 to 8 °C, dark
Appearance: white solid
hygroscopic
Melting Point: 210 °C (dec.)
Assay 'as is': 94.7 %

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

LoGiCal[®]
produced by LGC

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

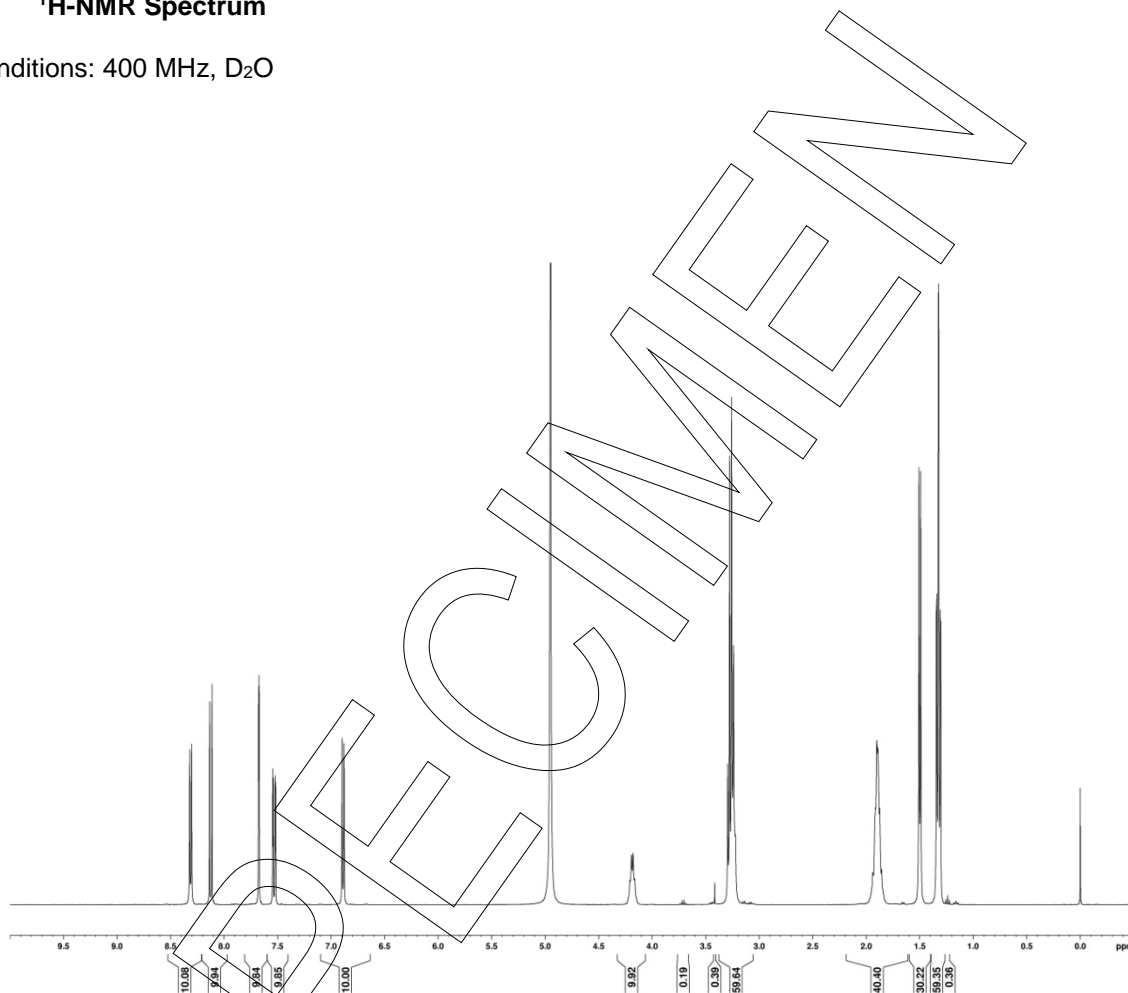


I. Identity

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

Ia. $^1\text{H-NMR}$ Spectrum

Conditions: 400 MHz, D_2O

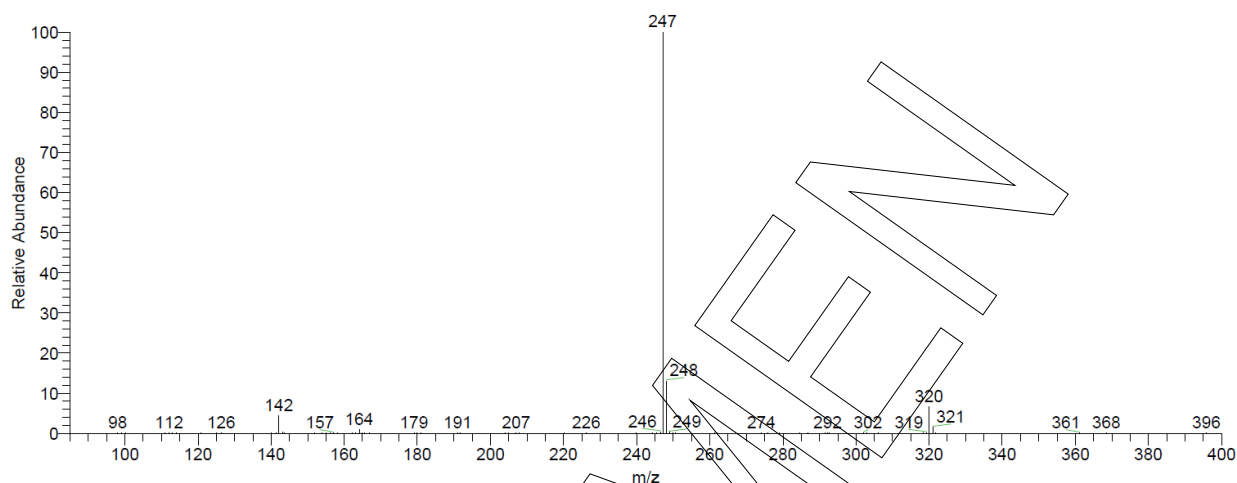


The structure is confirmed by the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

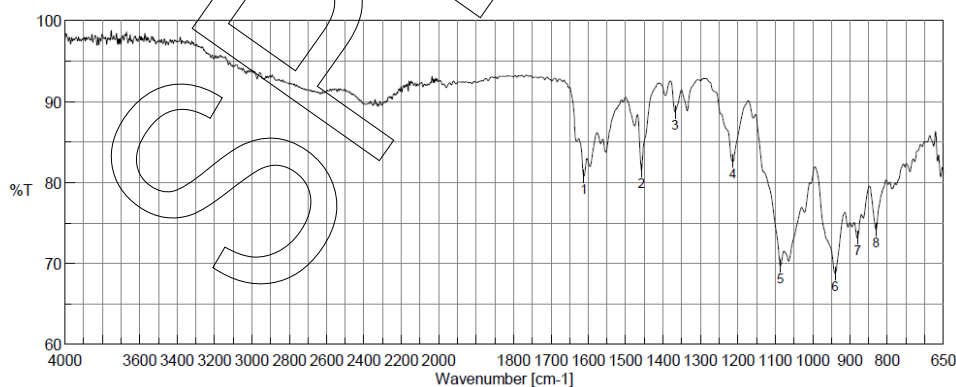
Method: 4.5 kV ESI+; vaporization temperature: 200 °C



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

Ic. IR Spectrum

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



Result of Peak Picking		
No.	Position	Intensity
1	1612.2	80.7415
2	1457.92	81.4221
3	1368.25	88.7131
4	1213.97	82.5845
5	1085.73	69.7099
6	940.128	68.7149
7	880.345	73.1307
8	830.205	74.0974

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:

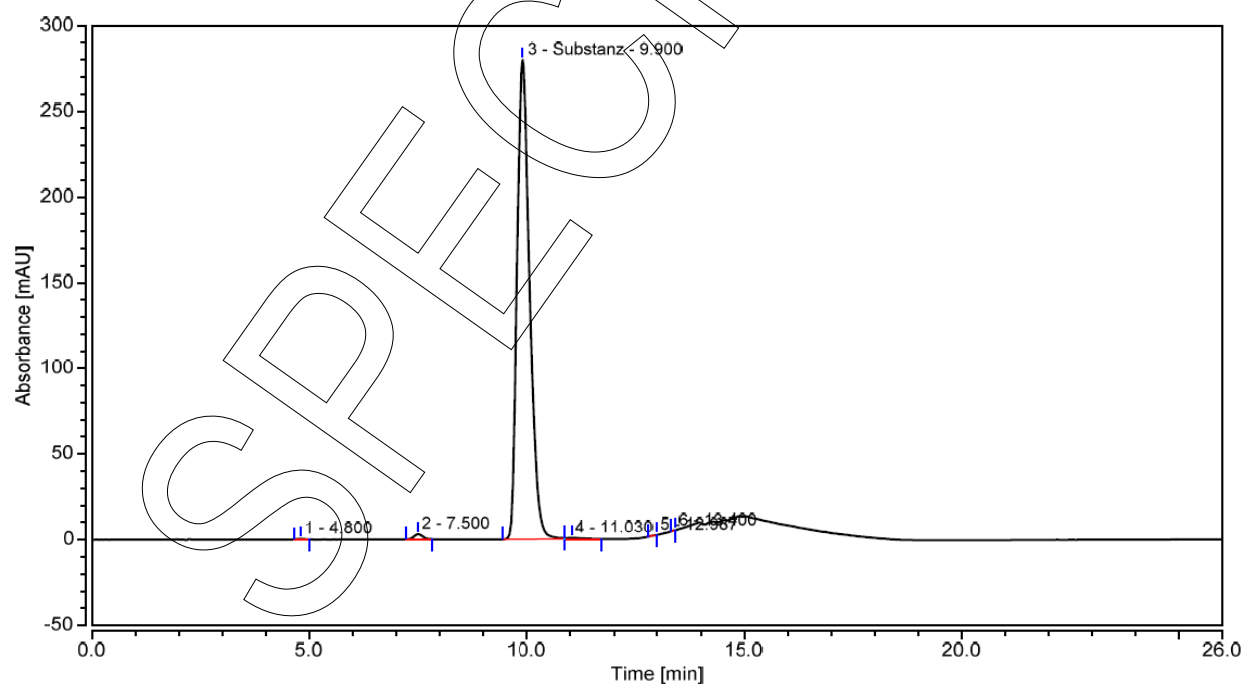
1.0 ml/min, 40 °C
0-9 min Water/Acetonitrile 92/8
9-12 min Water/Acetonitrile to 60/40
12-15 min Water/Acetonitrile to 92/8
15-26 min Water/Acetonitrile 92/8 (v/v);
0.1 % H₃PO₄

Detector:

DAD
220 nm

Injector:

Auto
1.5 µl; 0.176 mg/ml in
Water/Acetonitrile 50/50 (v/v)





Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	4.800	0.066	0.07
2	7.500	0.698	0.74
3	9.900	93.118	98.84
4	11.030	0.306	0.32
5	12.987	0.014	0.02
6	13.400	0.012	0.01
Totals		94.214	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 98.84 %
Number of results n=3
Standard deviation 0.02 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average 3.99 %
Number of results n=3
Standard deviation 0.14 %

IIc. Residual Solvents

Method: ¹H-NMR

Result: 0.10 % Ethanol
0.08 % Methanol



III. Final Result


Chromatographic purity (HPLC)	98.84 %
Water content	3.99 %
Residual solvents	0.18 %
Assay (100 % method)¹	94.72 %

The assay is assessed to be 94.7 % 'as is'

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

Release Date:

Luckenwalde, 2019-12-06



Dr. Sabine Schröder
Product Release

SPECIMEN

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.