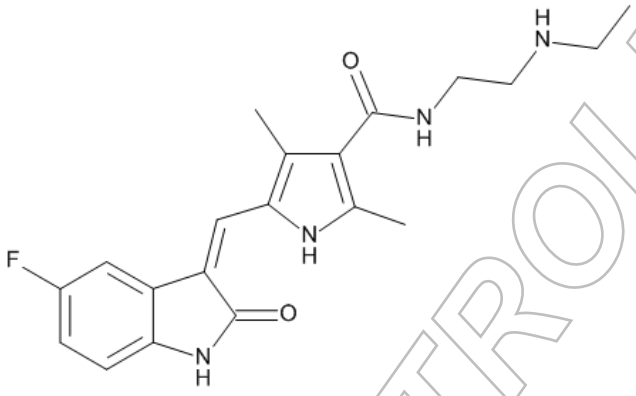
Certificate of Analysis

Reference Standard

N-Desethylsunitinib



Molecular Formula:	$C_{20}H_{23}FN_4O_2$
Molecular Weight:	370.42
CAS Number:	356068-97-8

Catalogue Number:	LGCFOR3568.06
Lot Number:	91501
Long-term Storage:	2 to 8 °C, dark
Appearance:	orange solid
Melting Point:	> 200 °C (dec.)
Assay ‘as is’:	99.1 %

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

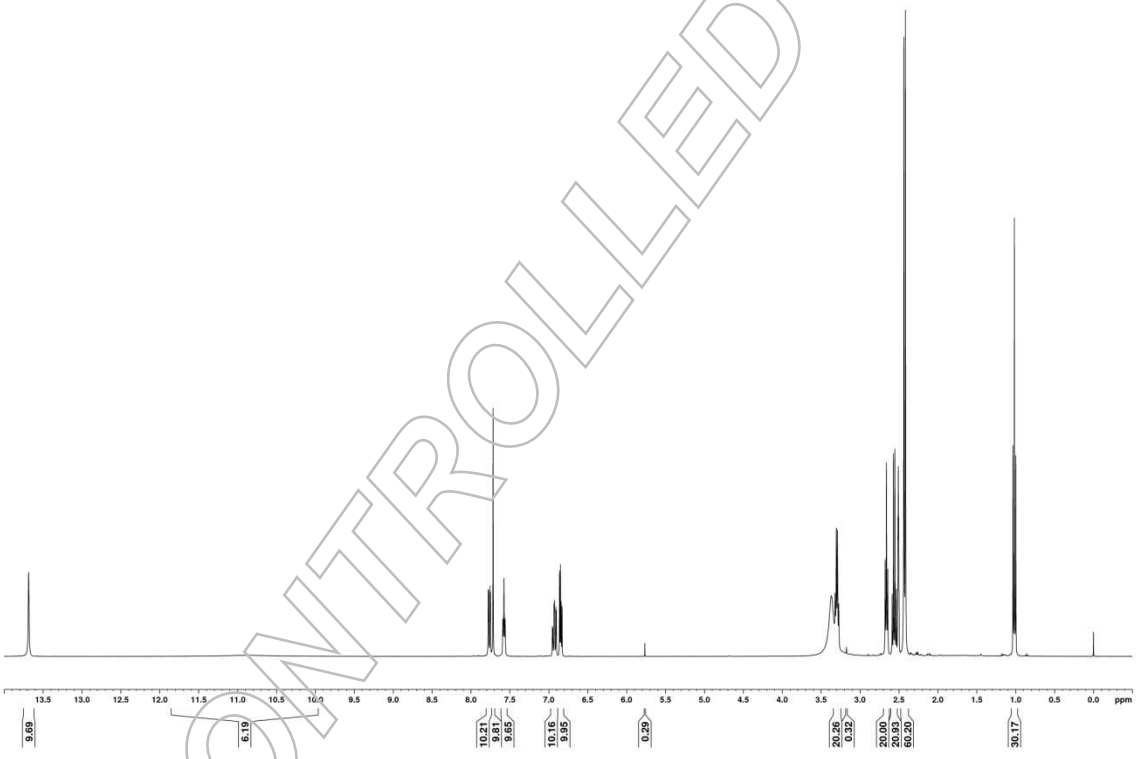
This certificate is valid for one year from the date of shipment provided the substance is stored under the recommended conditions unopened in the original container.

I. Identity

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

Ia. ^{1}H-NMR Spectrum

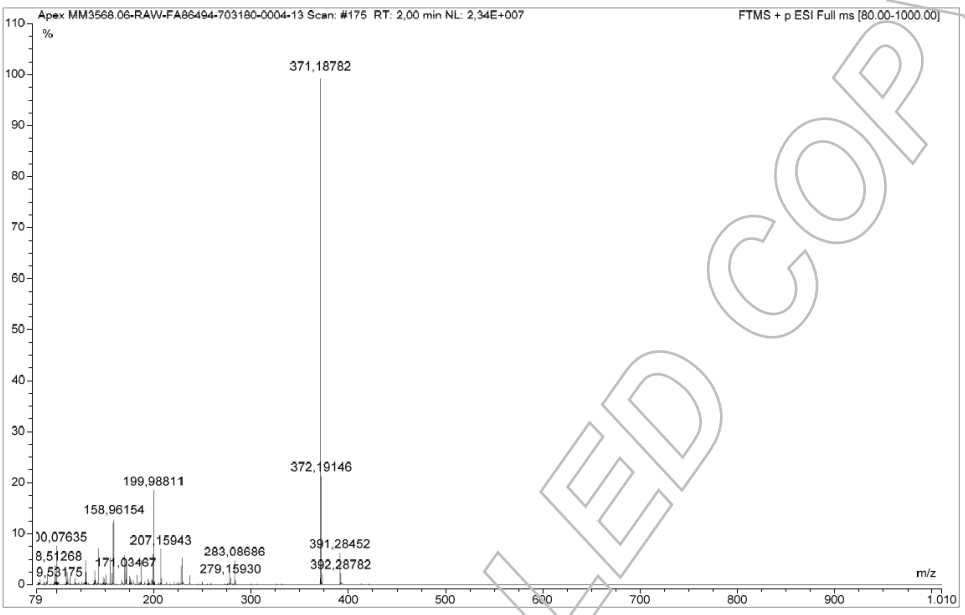
Conditions: 400 MHz, DMSO-d_6



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

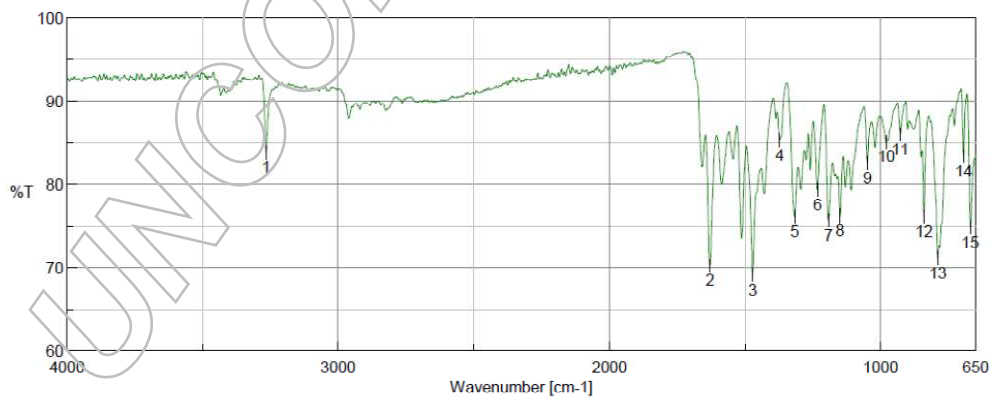


Theoretical value: 371.18778

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	3265.86	83.7877
2	1629.55	70.229
3	1472.38	69.1196
4	1373.07	85.2656
5	1317.14	76.1169
6	1233.25	79.3618
7	1192.76	75.6975
8	1149.37	76.2064
9	1049.09	82.5768
10	979.661	85.0806
11	927.593	85.9566
12	840.812	76.109
13	789.707	71.1032
14	694.248	83.3898
15	669.178	74.8227

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

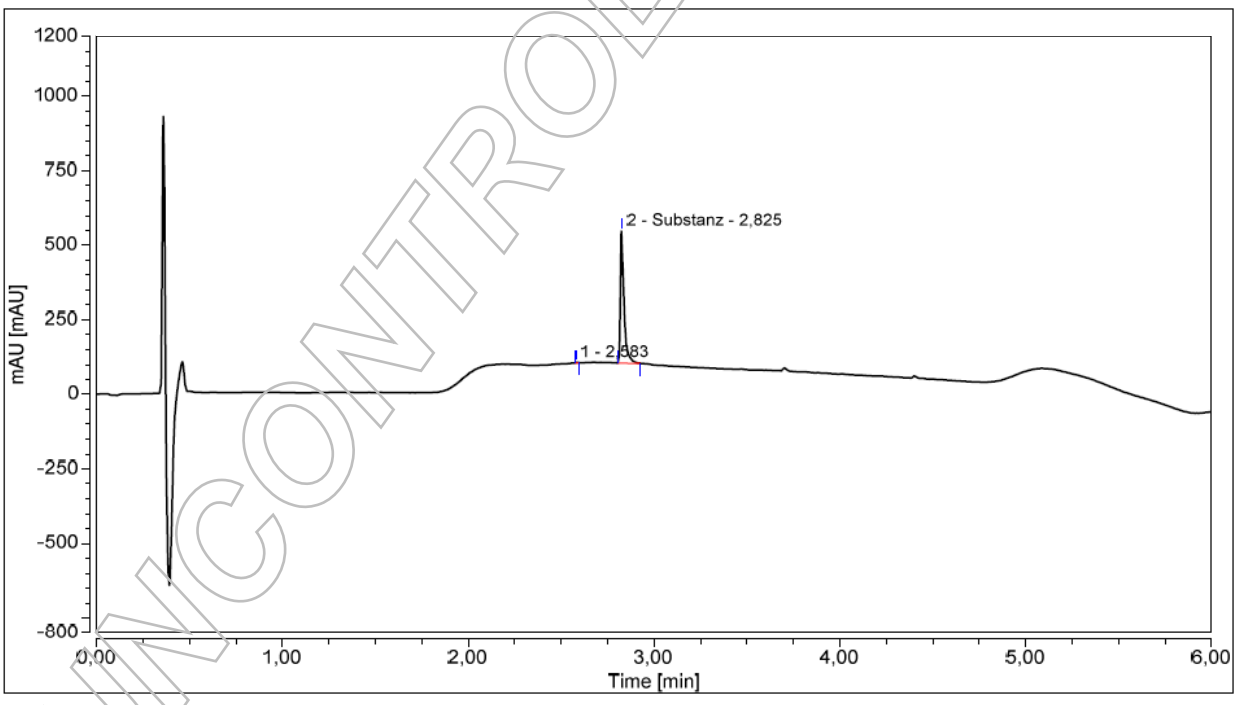
II. Purity

IIa. High Performance Liquid Chromatography (HPLC)

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

HPLC Conditions:

Column:	Conditions:	Detector:	Injector:
Cortecs UPLC C18+ 1.6 µm, 75 x 2.1 mm	0.5 ml/min, 40 °C 0-1 min Water/Acetonitrile 98/2 1-4 min Water/Acetonitrile to 2/98 4-5 min Water/Acetonitrile to 98/2 5-6 min Water/Acetonitrile 98/2 (v/v); 0.1 % HCOOH	DAD 210 nm	Auto 1 µl; 0.048 mg/ml in Methanol



Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	2.583	0.029	0.29
2	2.825	9.919	99.71
Totals		9.949	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	99.70 %
Number of results	n=3
Standard deviation	0.01 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average	0.20 %
Number of results	n=3
Standard deviation	0.01 %

IIc. Residual Solvents

Method: ^{1}H-NMR

Result: 0.33 % Dichloromethane
0.09 % Methanol

III. Final Result

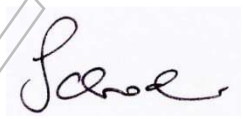
Chromatographic purity (HPLC)	99.70 %
Water content	0.20 %
Residual solvents	0.42 %
Assay (100 % method)[1]	99.08 %

The assay is assessed to be 99.1 % ‘as is’

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

Release Date:

Luckenwalde, 2015-12-01



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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