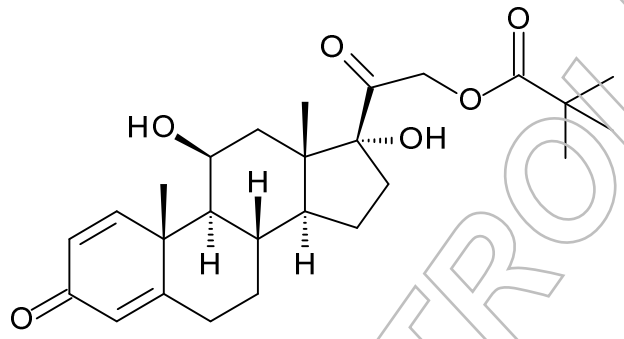
Certificate of Analysis

Reference Standard

Prednisolone Pivalate



Molecular Formula:	$C_{26}H_{36}O_6$
Molecular Weight:	444.56
CAS Number:	1107-99-9

Catalogue Number:	LGCFOR0304.00
Lot Number:	88340
Long-term Storage:	2 to 8 °C, dark
Appearance:	white solid
Melting Point (DSC):	234 °C
Assay 'as is':	98.9 %

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

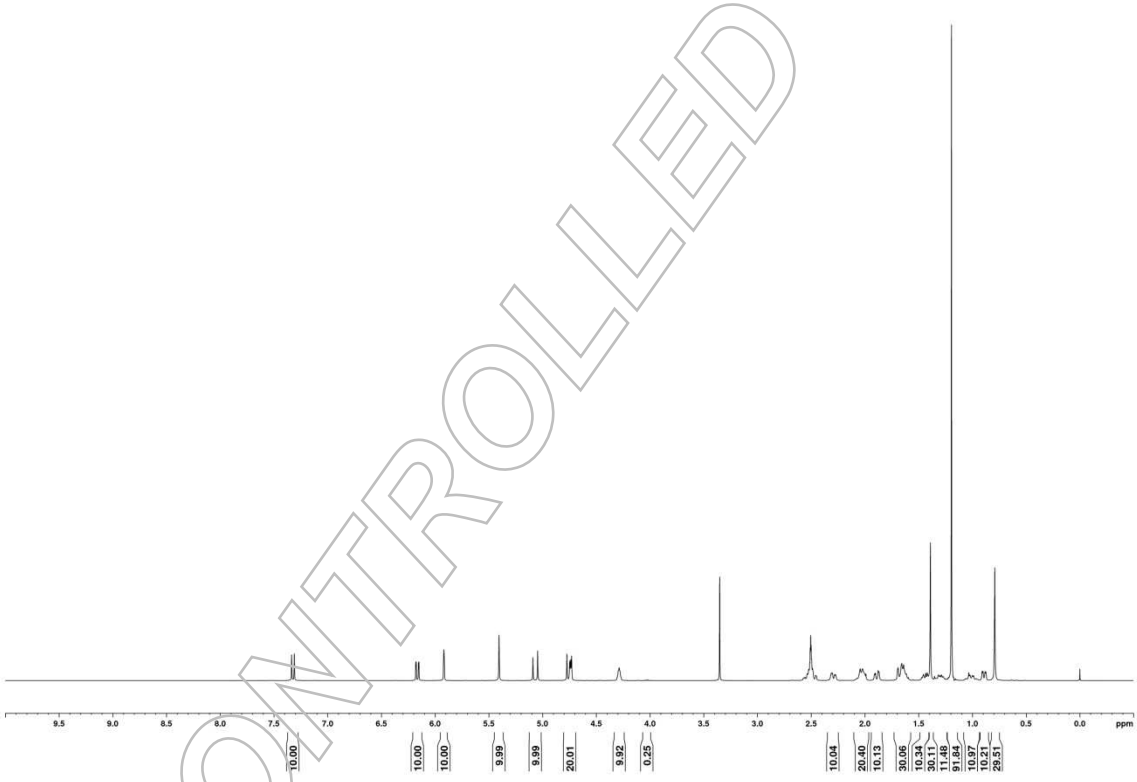
This certificate is valid for two years from the date of shipment provided the substance is stored under the recommended conditions unopened in the original container.

I. Identity

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

Ia. ^{1}H-NMR Spectrum

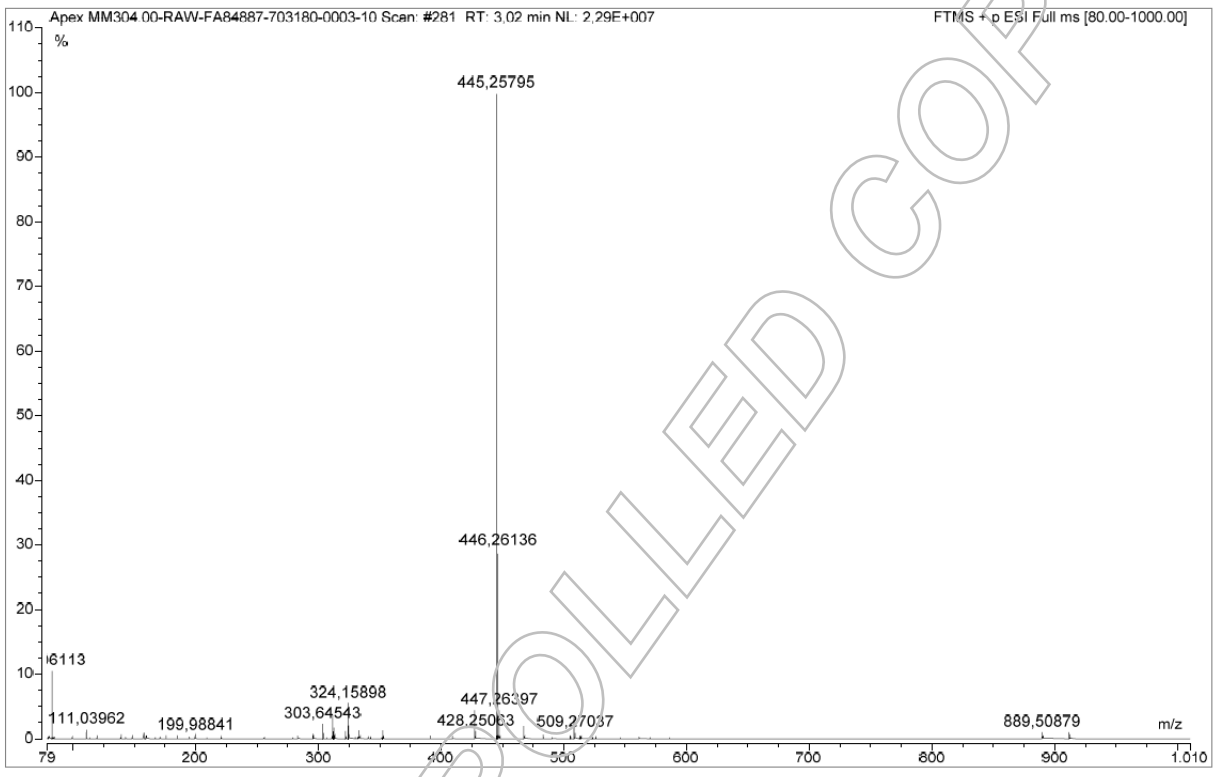
Conditions: 400 MHz, DMSO-d_6



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

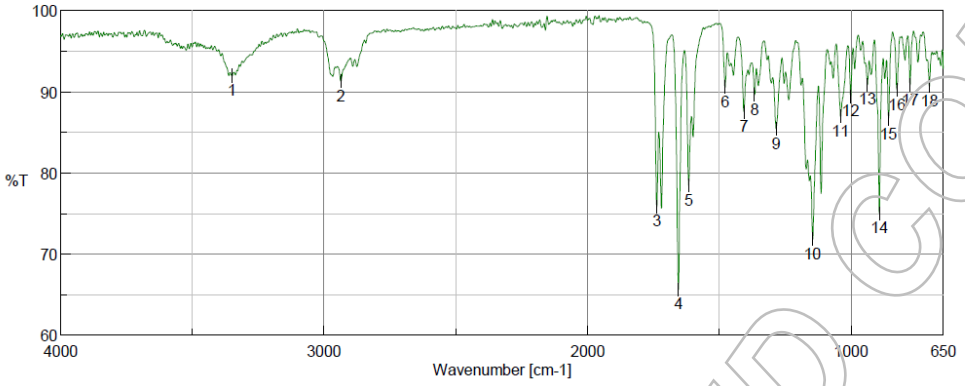


Theoretical value: 445.25847

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	3349.75	91.962
2	2937.06	91.3007
3	1737.55	75.8155
4	1655.59	65.6392
5	1616.06	78.445
6	1479.13	90.5236
7	1406.82	87.514
8	1366.32	89.6078
9	1284.36	85.4196
10	1146.47	71.8033
11	1040.41	86.9639
12	1001.84	89.4344
13	938.199	90.8746
14	892.88	75.0072
15	858.168	86.5663
16	826.348	90.2004
17	776.208	90.81
18	702.926	90.6744

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

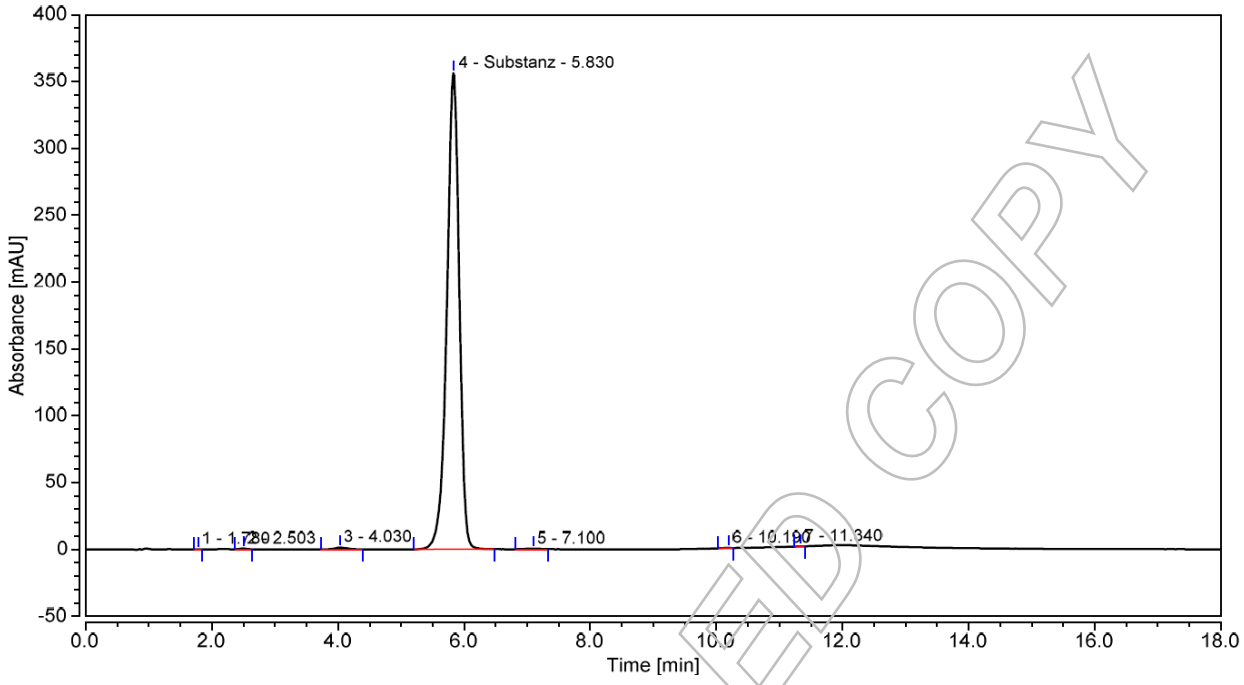
II. Purity

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:**
LiChrospher 60 RP-select B 5 µm, 125 x 4 mm	1.0 ml/min, 40 °C 0-7 min Water/Acetonitrile 50/50 7-10 min Water/Acetonitrile to 20/80 10-13 min Water/Acetonitrile to 50/50 13-18 min Water/Acetonitrile 50/50 (v/v); 0.1 % H_3PO_4	DAD 245 nm	Auto 10 µl; 0.0684 mg/ml in Acetonitrile



Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	1.780	0.005	0.01
2	2.503	0.071	0.09
3	4.030	0.322	0.40
4	5.830	81.045	99.31
5	7.100	0.114	0.14
6	10.190	0.036	0.04
7	11.340	0.013	0.02
Totals		81.606	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	99.32 %
Number of results	n=3
Standard deviation	0.03 %

IIb. Water Content

Method: Karl Fischer titration

Results:

Average	0.21 %
Number of results	n=3
Standard deviation	0.01 %

IIc. Residual Solvents

Method: ^{1}H-NMR

Result: 0.25 % Ethyl acetate

III. Final Result

Chromatographic purity (HPLC)	99.32 %
Water content	0.21 %
Residual solvents	0.25 %
Assay (100 % method)[1]	98.86 %

The assay is assessed to be 98.9 % 'as is'

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

Release Date:

Luckenwalde, 2015-10-28

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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