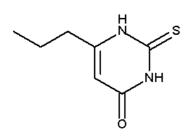


# **Certificate of Analysis**

### **Reference Standard**

Propylthiouracil



Molecular Formula:  $C_7H_{10}N_2OS$ Molecular Weight: 170.23 CAS Number: 51-52-5 Catalogue Number: LGCFOR3284.00

Lot Number: 69051

Long-term Storage: 2 to 8 °C, dark Appearance: white solid Melting Point (DSC): 218 °C

Assay 'as is': 99.2 %

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.

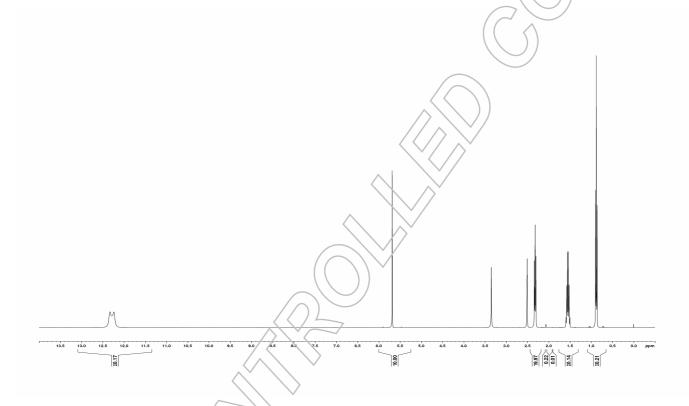


# I. Identity

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

# Ia. <sup>1</sup>H-NMR Spectrum

Conditions: 400 MHz, DMSO-d<sub>6</sub>



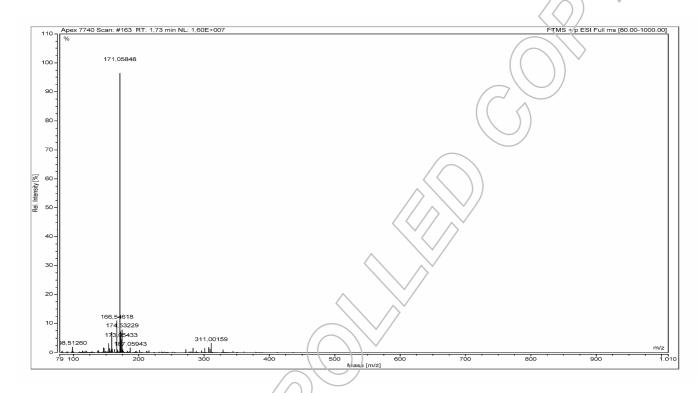
The structure is confirmed by the signals of the spectrum and their interpretation.





# **Ib.** Mass Spectrum

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



Theoretical value: 171.05866

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



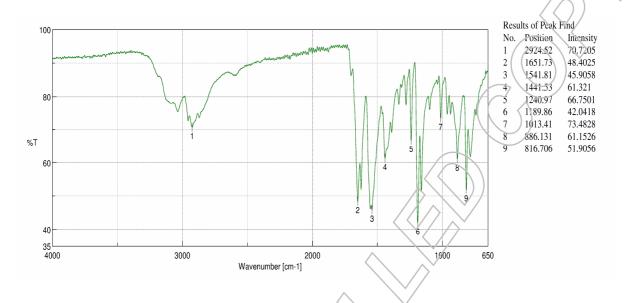


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# Ic. IR Spectrum

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



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



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## **II.** Purity

# **High Performance Liquid Chromatography (HPLC)**

The purity of the reference substance was analysed by high performance liquid chromategraphy (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 A/B 98/2 min 1-4 min A/B to 2/98 4-5 A/B 98/2 min to 98/2 A/B min 5-6

Detector:

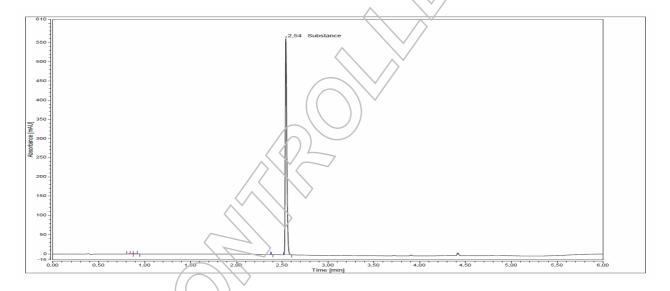
DAD 275 nm

(v/v)

Auto

injector:

1 µl; 42.000 mg/ml in Acetonitrile/Water 50/50







#### **Area Percent Report - Sorted by Signal**

Pk#	Retention Time	Area	Area %
1	0.842	0.0330	0.30
2	0.919	0.0334	0.30
3	2.384	0.0012	0.01
4	2.541	10.9661	99.39
Totals		11.0337	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 99.39 % Number of results n=3 Standard deviation 0.01 %

#### **IIb.** Water Content

Method: Karl Fischer titration

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

### IIc. Residual Solvents

Residual Solvent	Average	Method
Acetic acid	0.02 %	<sup>1</sup> H-NMR
Acetonitrile	0.17 %	<sup>1</sup> H-NMR

LGCFOR3284.00 lot number 69051





#### **III. Final Result**

Chromatographic purity (HPLC) 99.39 %

Water content No significant amounts of water were detected (< 0.05 %)

**Residual solvents** 0.19 % **Assay (100 % method)**<sup>1</sup> 99.20 %

The assay is assessed to be 99.2 % '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-March-11

Signed:

Dr. Sabine Schröder Product Release

Assay (%) = (100 % - volatile contents) \* Purity (%)

Volatile contents are considered as absolute contributions, purity is considered as relative contribution

LGCFOR3284.00

lot number 69051

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<sup>&</sup>lt;sup>1</sup> The calculation of the 100 % method follows the formula: