

Date of shipment:

04 Nov 2019

Producer confirms that this reference material (RM) meets the specification detailed on this Certificate of Analysis for **two years** from the date of shipment, provided the substance is stored under the recommended conditions unopened in the original container.

Release by: Date of Release:	P	Draduct Dalages
Dr. Sabine Schröder Luckenwalde, 25 Oct 2019	Jarok	Product Release

¹ Calibration and verification were carried out using standards traceable to SI-units. The value is expressed on an "as is" basis.

² The uncertainty "U" is the expanded uncertainty of the testing method for the assigned value estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM). It corresponds to a level of confidence of about 95%. Coverage factor k =2.

Organisation certified to ISO 9001 | DQS 102448 and GMP (EXCIPACTTM) Test methods used for characterisation are accredited to ISO/IEC 17025 | DAkkS D-PL-14176-01-00

Producer: LGC GmbH Louis-Pasteur-Str. 30 D-14943 Luckenwalde Germany www.lgcstandards.com Page 1/9



Product information

This RM is intended for laboratory use only and is not suitable for human or animal consumption. This RM conforms to the characteristics of a primary standard as described in the ICH Guidelines. The values quoted in this Certificate of Analysis are the producer's best estimate of the true values within the stated uncertainties and based on the techniques described in this Certificate of Analysis. The characterisation of this material was undertaken in accordance with the requirements of ISO/IEC 17025. The identity is verified by data from international scientific literature.

Storage and handling

Before usage of the RM, it should be allowed to warm to room temperature. No drying is required, as assigned values are already corrected for the content of water and other volatile materials.

Reference Material quality is controlled by regularly performed quality control tests (retests).

Further content

Assigned value Purity

Identity

Revision table



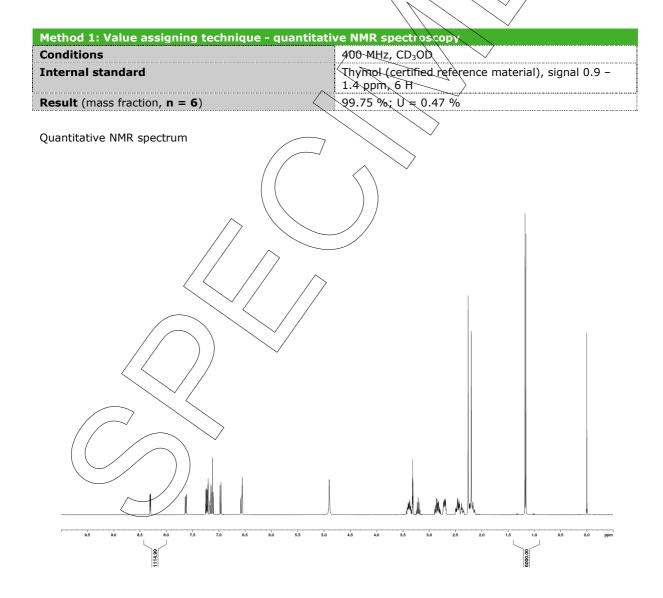
Assigned value

Assay "as is":

99.75 %; U = 0.47 %

The assay "as is" is assessed by quantitative NMR spectroscopy and is equivalent to the assay based on the not-anhydrous and not-dried substance. The assay is verified by 100% method (mass balance). The verified result lies inside our acceptance criteria, i.e. less than 1.0 % difference to assay assigning technique.

For quantitative applications, use the assay as a calculation value on the ^kas is basis^m. The uncertainty of the assay can be used for estimation/calculation of measurement uncertainty.





Method 2: Value verifying technique - 100% me	thod	
100% method (mass balance) with chromatographic purity by HPLC		
Result	99.91 %	
The calculation of the 100% method follows the form	ıla:	

Purity (%)

100 %

Assay (%) = (100 % - volatile contents (%))

Volatile contents are considered as absolute contributions and purity is considered as relative contribution. Inorganic residues are excluded by additional tests.

Purity

Purity by High Performance Liquid Chromatography (HPLC)

HPLC conditions:	\sim
Column	Hypersil Gold C18; 5 μm, 150 x 4.6 mm
Column temperature	40 9C
Detector	DAD,/210 nm
Injector	Auto 1 μ l; 0.191 mg/ml in Water/Acetonitrile 50/50 (v/v)
Flow rate	1.0 ml/min
Phase A	Water, 0.1 % H ₃ PO ₄
Phase B	Acetonitrile, 0.1 % H ₃ PO ₄
Gradient program	0 min A/B 98/2 0-20 min A/B to 3/97 20-22 min A/B 3/97 22-25 min A/B to 98/2 25-30 min A/B 98/2 (v/v)



HPLC chromatogram and peak table 1224 7,44 Substance 1100 1000 900 800 700 Absorbance [mAU] 600 500 400 300 200 100 -16 12 2,5 5,0 27,5 20,0 22,5 25,0 17,5 10,0 12,5

Area percent report - sorted by signal				
Pk #	Retention time	Area	Area %	
1	/7.443))	79.6762	99.90	
2	7.995	0.0070	0.01	
3	8.530	0.0716	0.09	
Totals		79.7548	100.00	

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 %. System peaks were ignored in calculation.

Result (n = 3)

99.91 %; U = 0.19 %



Volatile content

Water content		
Method	Karl Fischer titration	
Result (n = 3)	No significant amounts of water were detected (×0.05 %).

*not accredited testing method

Residual solvents		$\langle \rangle$	\leq /		
Method	¹ H-NMR		\smallsetminus \checkmark /	/	
Result (n = 1)	No significant amounts of residual				// *

*not accredited testing method

Inorganic residues

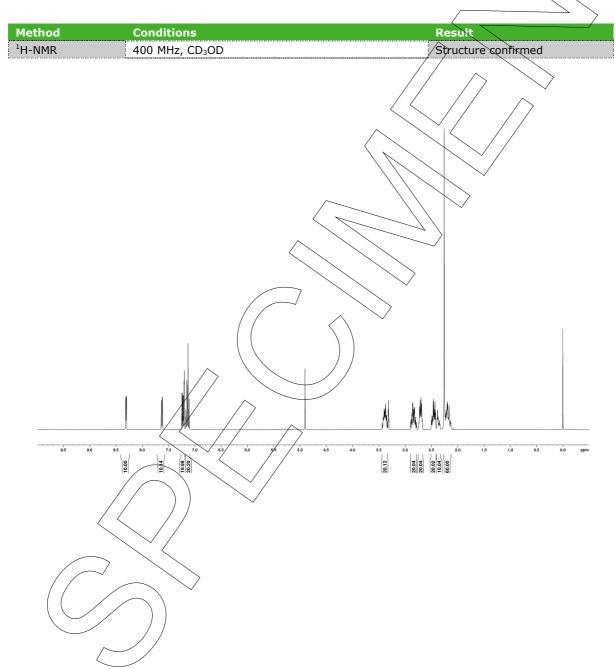
Method: Elementary analysis

Inorganic residues can be excluded by elementary analysis (CHN).



Identity

The identity is assessed by ISO/IEC 17025 accredited testing methods.





S 3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 325,14650 Aeex 4331 Sam 455 BT 0.38 mm NL 108E-008 Aeex 40 193077560 1930777560 1930777560 193077757757775775777577577577577757775777	Method	Conditions	Result
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Method Result Conditions IR Attenuated Total Reflection Fourier Transform Structure confirmed Infrared (ATR-FTIR) Spectroscopy Results of Peak Find 100 Position Intensity No. 2892.7 82.3933 1557.24 1421.28 84.8182 72.8817 90 1279.54 81.0341 M 130.08 71.2318 989.304 80.6329 876.488 82.0675 829.241 68.7514 %T 80 782.958 64.1342 70 60└─ 4000 3000 2000 1000 650 Wavenumber [cm-1] **Revision table** Revision Date Reason for revision 00 25 Oct 2019 Release of the Certificate of Analysis - initial version

Product warranties for the RM are set out in the terms and conditions of purchase.