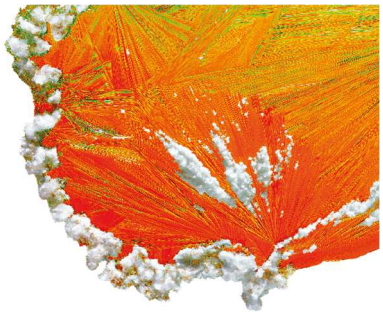


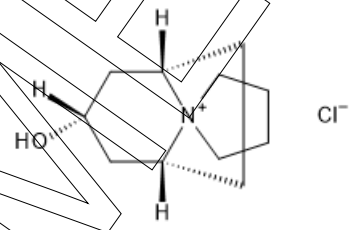
Certificate of Analysis

Characterisation methods are accredited according to ISO 17025

Reference Material

Product name

(1R,3r,5S)-3-Hydroxyspiro[8-azoniabicyclo[3.2.1]octane-8,1'-pyrrolidinium] Chloride



Product code MM1266.03-0025	**Lot number** W1144608
CAS number 3464-71-9	**Appearance** beige solid
Molecular weight 217.74	**Melting point (DSC)** 238 °C
Molecular formula $C_{11}H_{20}NO$ Cl	**Long-term storage** 2 to 8 °C, dark

Assay[1] "as is"	Uncertainty[2] U
99.7 %	**0.6 %**

Intended Use: Use for identification and quantification. The assay is verified by a second testing method.

Date of shipment: **20 Jul 2021**

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

Release by:	**Date of Release:**		
Dr. Sabine Schröder	Luckenwalde, 11 Jun 2021		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.

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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

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Assigned value

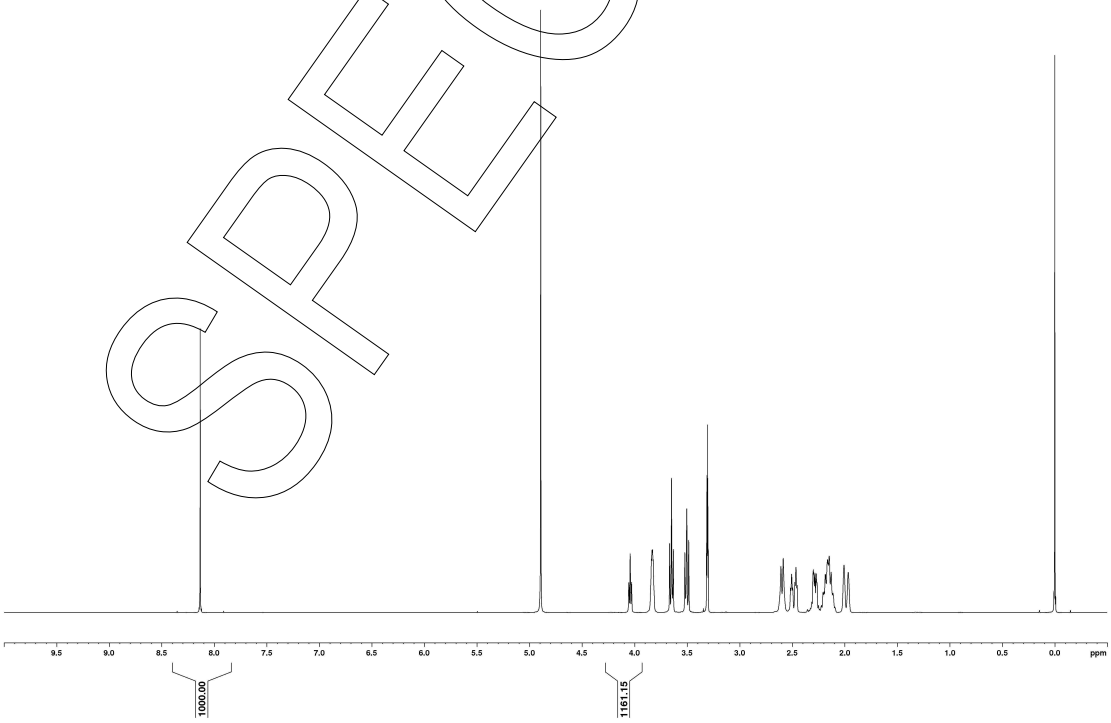
Assay "as is": **99.67 %; U = 0.58 %**

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 carbon titration of elemental analysis. 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 "as is basis". The uncertainty of the assay can be used for estimation/calculation of measurement uncertainty.

Method 1: Value assigning technique - quantitative NMR spectroscopy	
Conditions	400 MHz, CD_3OD
Internal standard	2,3,5,6-Tetrachloro-1-nitrobenzene (certified reference material), signal 7.8 – 8.4 ppm, 1 H
Result (mass fraction, **n = 6**)	99.67 %; U = 0.58 %

Quantitative NMR spectrum



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Method 2: Value verifying technique - carbon titration of elemental analysis	
Method	percentage carbon found in relation to percentage carbon as calculated for molecular formula
Result (mass fraction, **n = 3**)	100.18 %

Purity

Volatile content

Water content	
Method	Karl Fischer titration
Result (n = 3)	0.05 %*; SD < 0.01 %

*not accredited testing method

Residual solvents	
Method	^{1}H-NMR
Result (n = 1)	Sum: 0.22 %* 0.11 % Methanol; 0.11 % Methylene chloride

*not accredited testing method

Inorganic residues

Method: Elementary analysis

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

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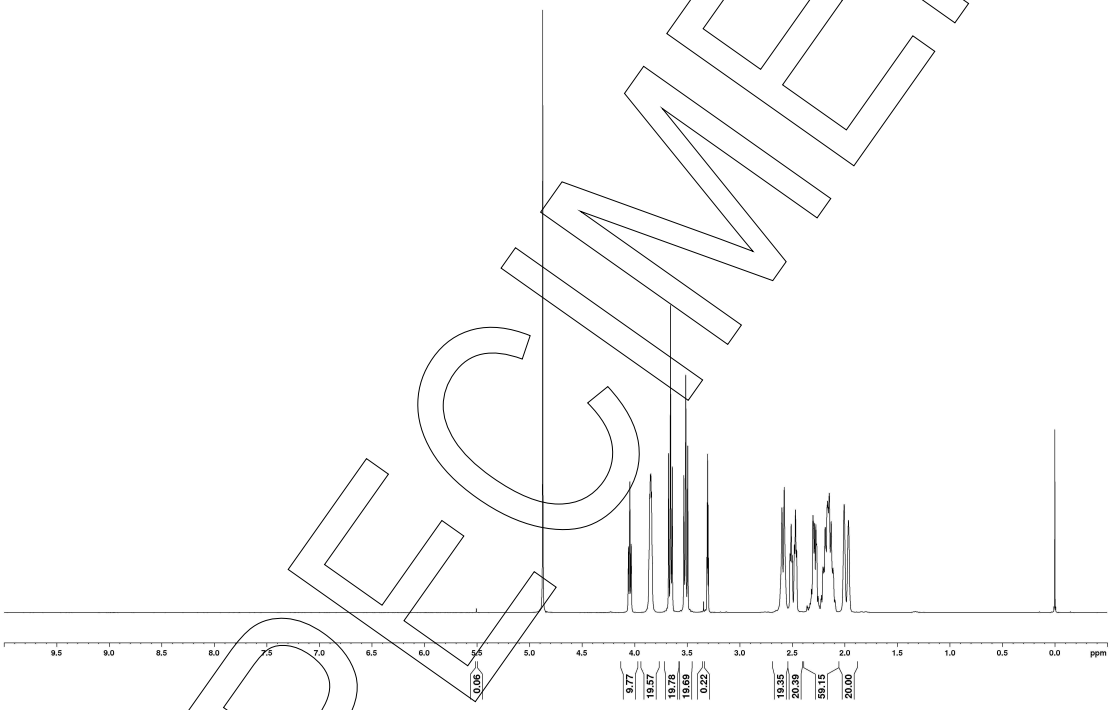
Identity

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

Method	Conditions	Result
^{1}H-NMR	400 MHz, CD_3OD	Structure confirmed



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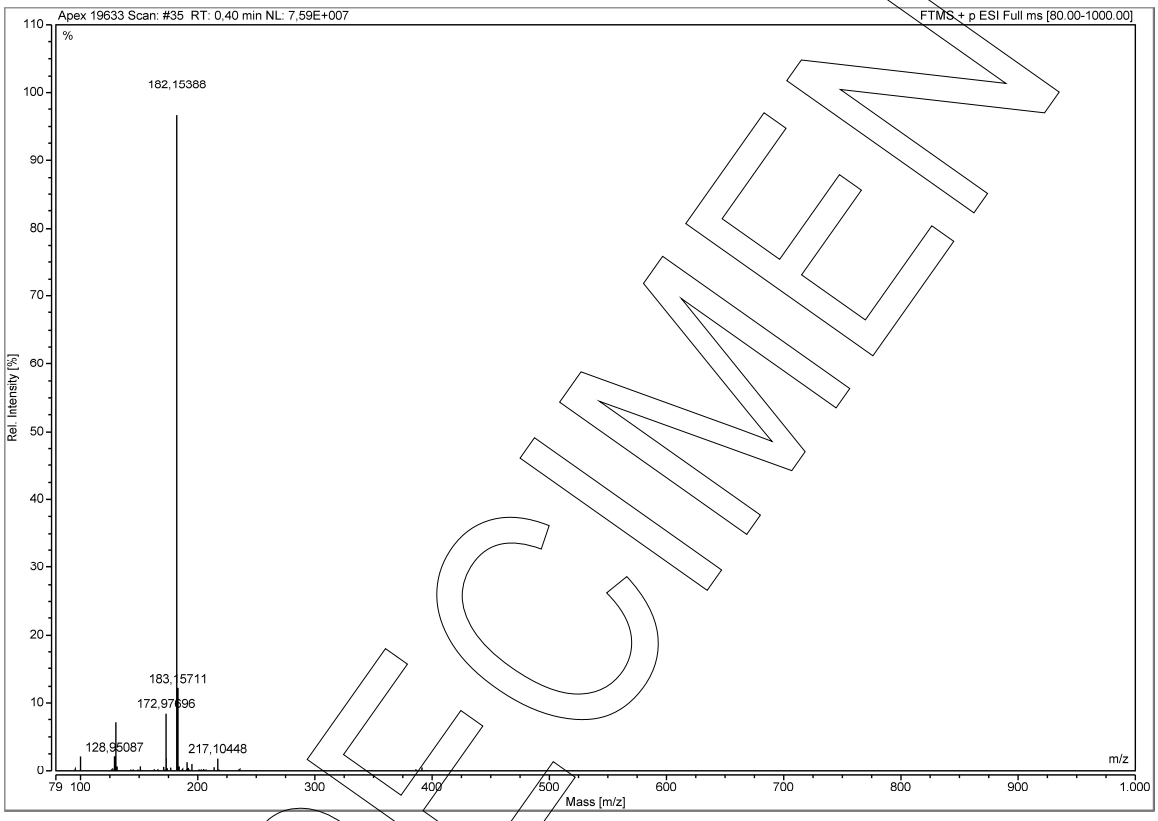
Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 182.15394	Structure confirmed



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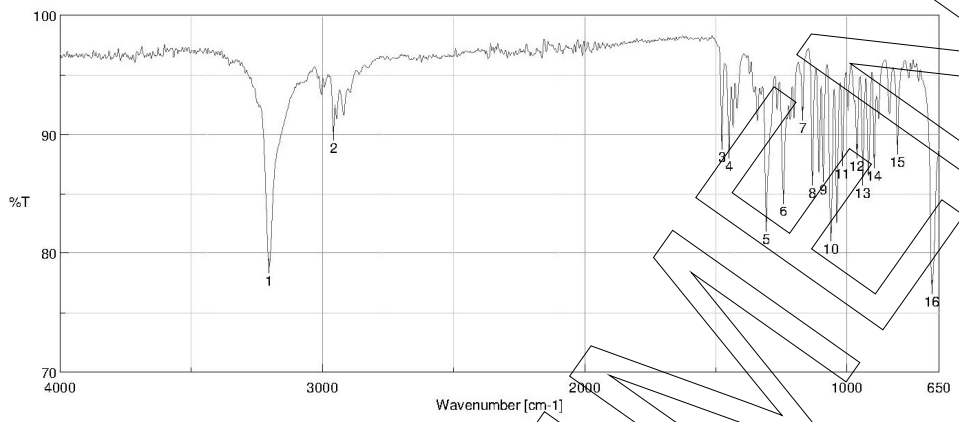
Method	Conditions	Result
IR	Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy	Structure confirmed



Results of Peak Find

No.	Position	Intensity
1	3203.18	78.8691
2	2958.27	90.0857
3	1477.21	89.3186
4	1449.24	88.5768
5	1308.46	82.3652
6	1242.9	84.7773
7	1169.62	91.7907
8	1132.01	86.3271
9	1090.55	86.6052
10	1060.66	81.5358
11	1017.27	87.952
12	962.305	88.5707
13	940.128	86.3363
14	896.737	87.7966
15	808.028	88.9171
16	676.892	77.1686

Revision table

Revision	Date	Reason for revision
00	11 Jun 2021	Release of the Certificate of Analysis – initial version

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

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