

Certificate of Analysis

Characterisation methods are accredited according to

ISO 17025

Reference Material

Product name

L-Valine Methyl Ester Hydrochloride

Product code

MM0487.06-0025

CAS number

6306-52-1

Molecular weight

167.63

Molecular formula

C₆H₁₃NO₂ HCl

Lot number

W1023064

Appearance

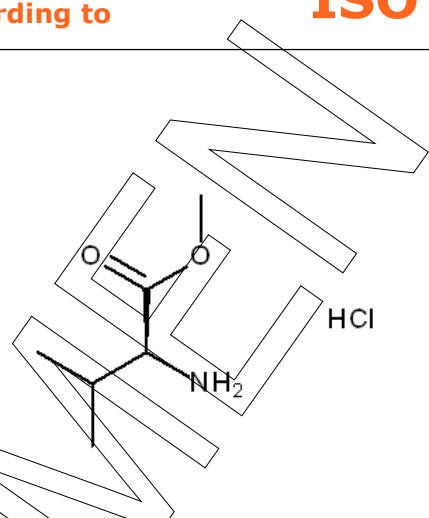
white solid

Melting point (DSC)

172 °C

Long-term storage

2 to 8 °C, dark



Assay¹ "as is"
99.4 %

Uncertainty² U
0.4 %

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

Date of shipment: **06 Apr 2020**

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: | | Product Release |
| Dr. Sabine Schröder | Luckenwalde, 27 Mar 2020 | | |

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.



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

SPECIMEN



Assigned value

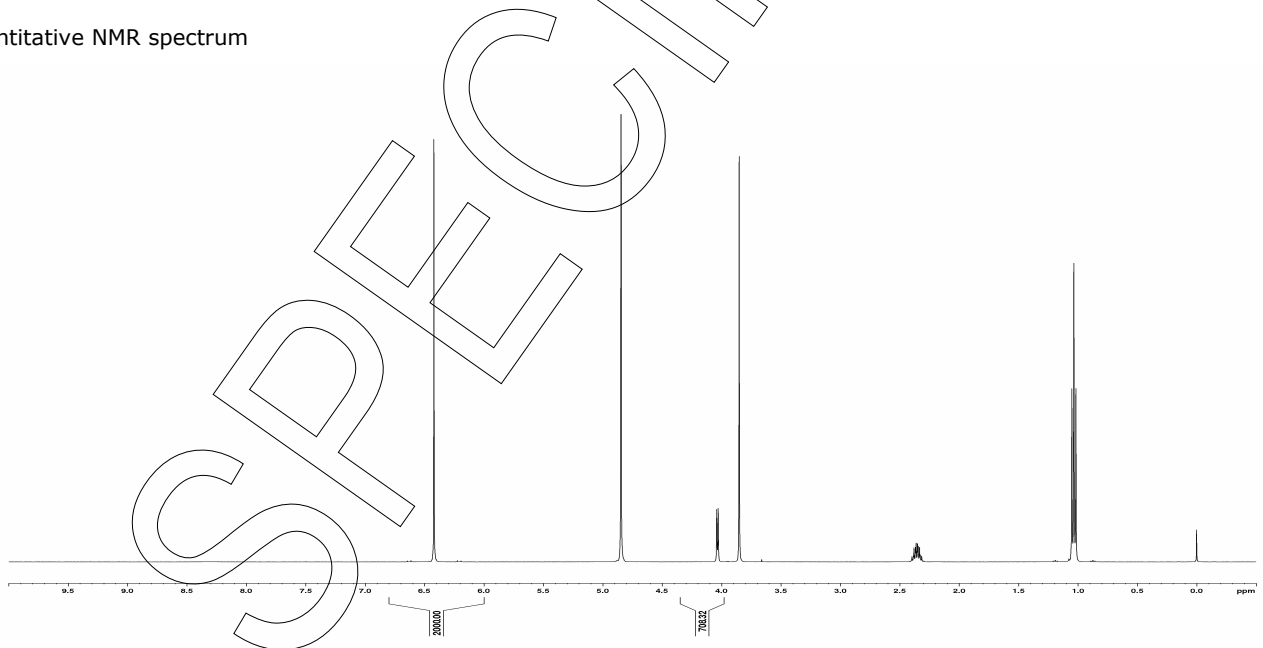
Assay "as is": 99.38 %; U = 0.42 %

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, D ₂ O |
| Internal Standard | Maleic acid (certified reference material), signal 6.0 - 6.8 ppm, 2 H |
| Result (mass fraction, n = 6) | 99.38 %; U = 0.42 % |

Quantitative NMR spectrum





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) | 99.85 % |

SPECIMEN



Purity

Volatile content

Water content

| | |
|-----------------------|------------------------|
| Method | Karl Fischer titration |
| Result (n = 3) | 0.10 %*; SD = 0.02 % |

*not accredited testing method

Residual solvents

| | |
|-----------------------|--|
| Method | ¹ H-NMR |
| Result (n = 1) | No significant amounts of residual solvents were detected (< 0.05 %).* |

*not accredited testing method

Inorganic residues

Method: Elementary analysis

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

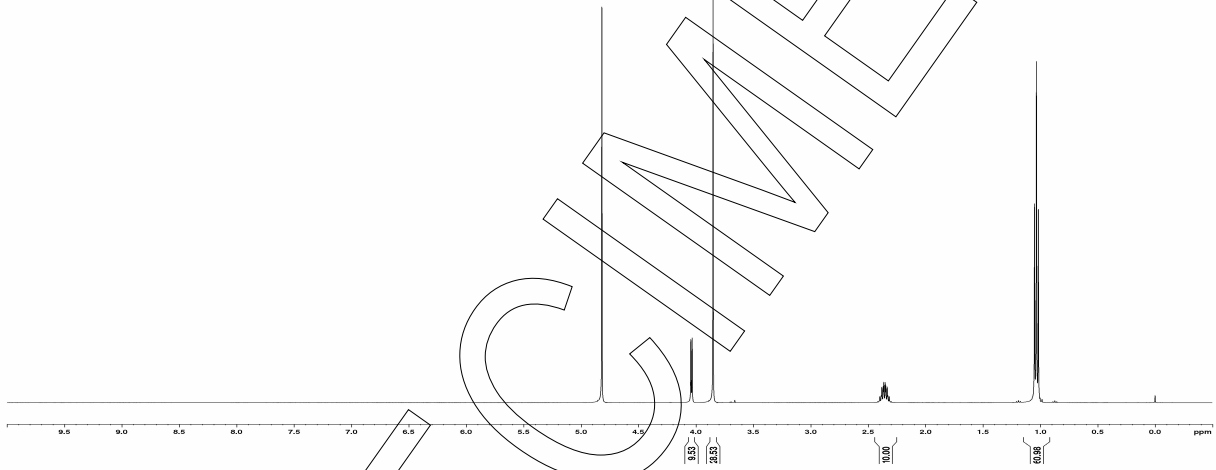
SPECIMEN



Identity

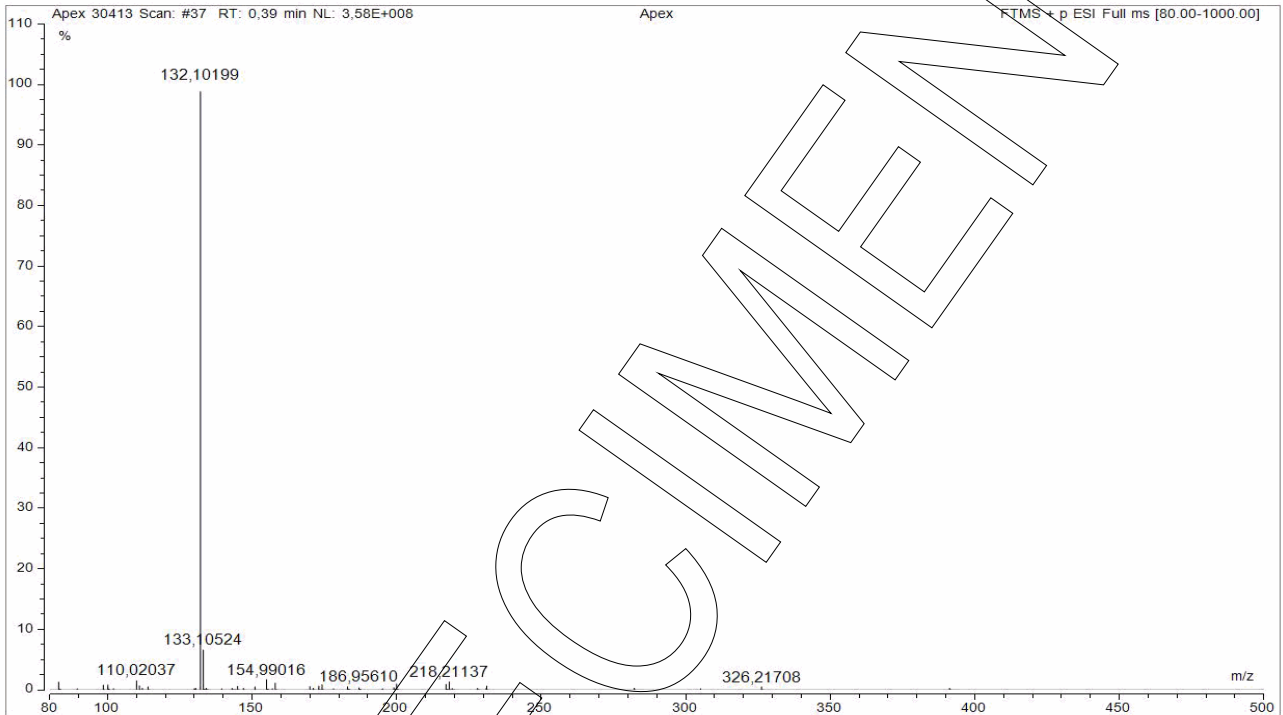
The identity is assessed by ISO/IEC 17025 accredited testing methods.

| Method | Conditions | Result |
|--------------------|---------------------------|---------------------|
| ¹ H-NMR | 400 MHz, D ₂ O | Structure confirmed |





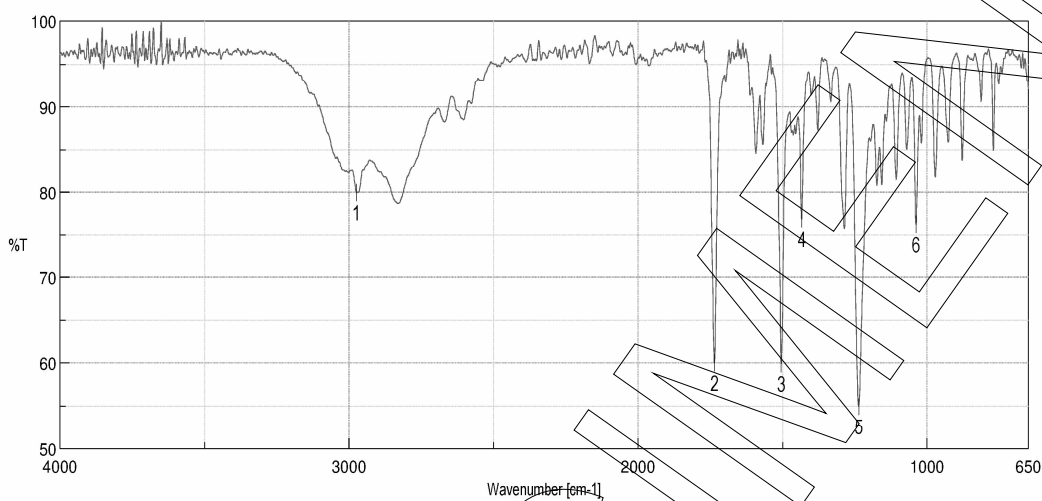
| Method | Conditions | Result |
|--------|--|---------------------|
| MS | 3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 132.10191 | Structure confirmed |



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



| No. | Position | Intensity |
|-----|----------|-----------|
| 1 | 2973.7 | 79.8831 |
| 2 | 1735.62 | 59.9504 |
| 3 | 1505.17 | 59.8501 |
| 4 | 1433.82 | 76.846 |
| 5 | 1237.11 | 54.9321 |
| 6 | 1038.48 | 76.1751 |

Revision table

| Revision | Date | Reason for revision |
|----------|-------------|--|
| 00 | 27 Mar 2020 | Release of the Certificate of Analysis - initial version |

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