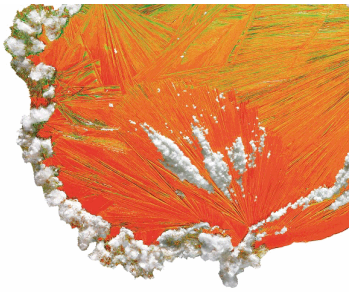


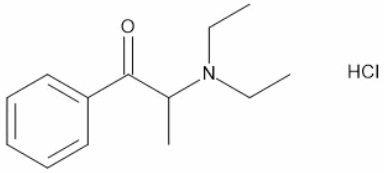
Certificate of Analysis

Characterisation methods are accredited according to **ISO 17025**

Reference Material

Product name

Amfepramone Hydrochloride (Diethylpropion Hydrochloride)



Product code MM1390.00-0250	**Lot number** W1300348
CAS number 134-80-5	**Appearance** white solid
Molecular weight 241.76	**Melting point (DSC)** 177 °C
Molecular formula $C_{13}H_{19}NO$ HCl	**Long-term storage** 2 to 8 °C, dark very hygroscopic

Assay[1] "as is"	Uncertainty[2] U
97.3 %	**0.4 %**

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

Date of shipment: **31 May 2024**

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, 31 May 2024	Sabine	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.

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).

Health and safety information

All chemical reference materials should be consider potentially hazardous and should be used only by qualified laboratory personnel only. Please refer to the Safety Data Sheet for detailed information about the nature of any hazard and appropriate precautions to be taken.

Further content

Assigned value

Purity

Identity

Revision table

Assigned value

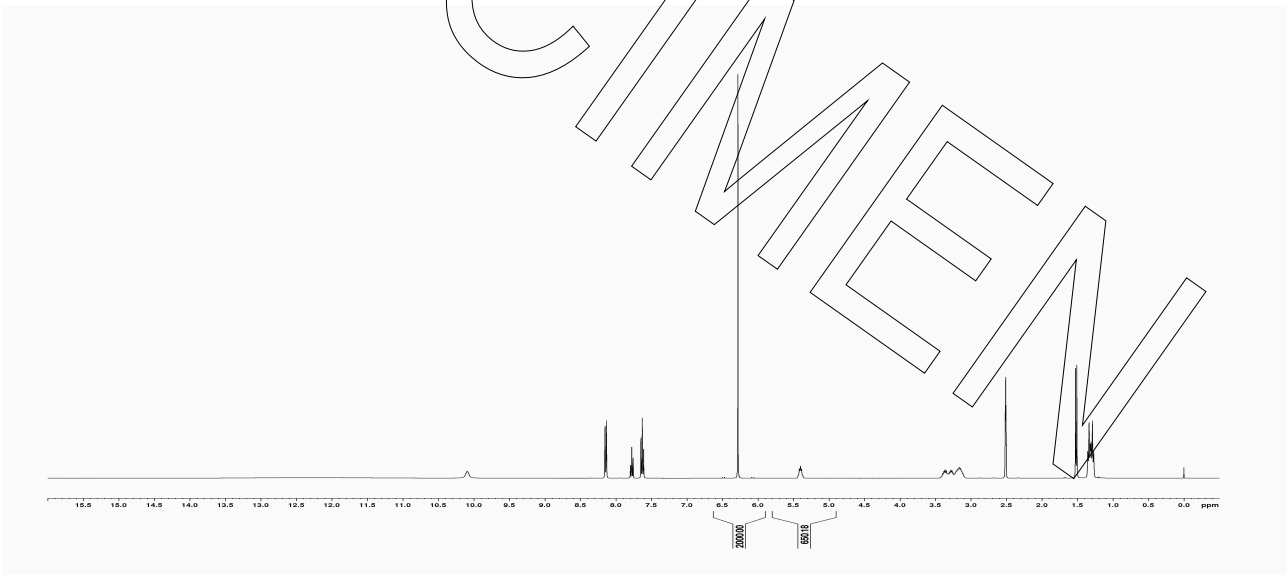
Assay "as is": **97.29 %; U = 0.42 %**

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, DMSO-d_6
Internal Standard	Maleic acid (certified reference material), signal 5.9 - 6.6 ppm, 2 H
Result (mass fraction, **n = 6**)	97.29 %; U = 0.42 %

Quantitative NMR spectrum



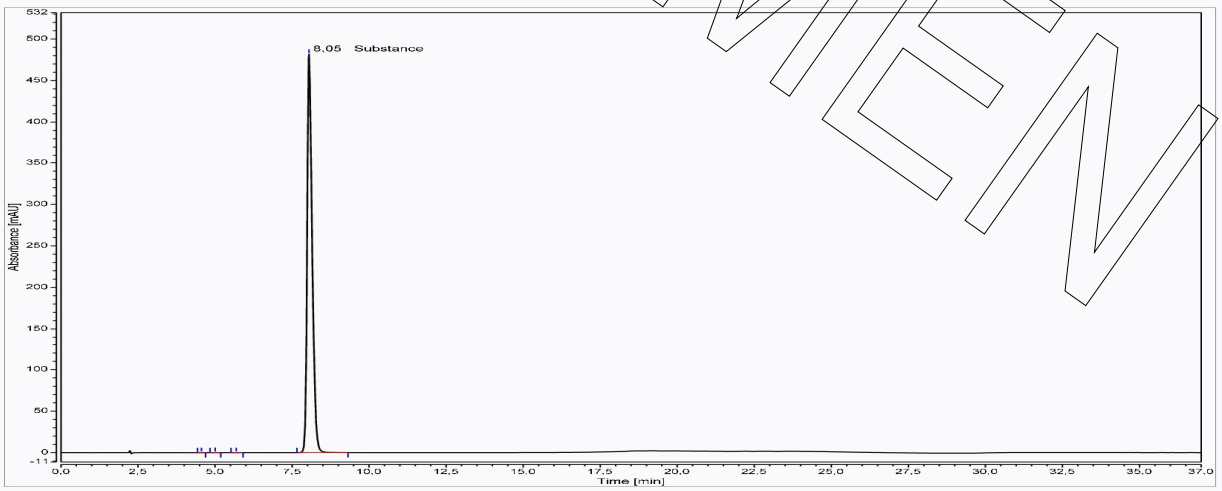
Method 2: Value verifying technique - carbon titration of elemental analysis	
Method	percentage carbon found in relation to percentage carbon as calculated for molecular formula
Result (mass fraction, **n = 3**)	97.68 %

Purity

Purity by high performance liquid chromatography (HPLC)

HPLC Conditions:	
Column	Hypersil Gold C18; 5 µm, 150 x 4.6 mm
Column temperature	40 °C
Detector	DAD, 255 nm
Injector	Auto 2 µl; 0.1656 mg/ml in Acetonitrile/Water 50/50 (v/v)
Flow rate	1.0 ml/min
Phase A	Water, 0.1 % H_3PO_4
Phase B	Acetonitrile, 0.1 % H_3PO_4
Gradient program	0-12 min A/B 90/10 12-17 min A/B to 50/50 17-22 min A/B 50/50 22-27 min A/B to 90/10 27-37 min A/B 90/10 (v/v)

HPLC chromatogram and peak table



Area percent report - sorted by signal				
Pk #	**Retention time**	**Area**	**Area %**	
1	4.562	0.0066	0.01	
2	5.005	0.0230	0.03	
3	5.685	0.0114	0.01	
4	8.048	91.0750	99.95	
Totals		**91.116**	**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 = 6)	99.96 %; U = 0.19 %

Volatile content

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

Residual solvents	
Method	GC headspace
Result (n = 3)	No significant amounts of residual solvents were detected (< 0.05 %).

Inorganic residues

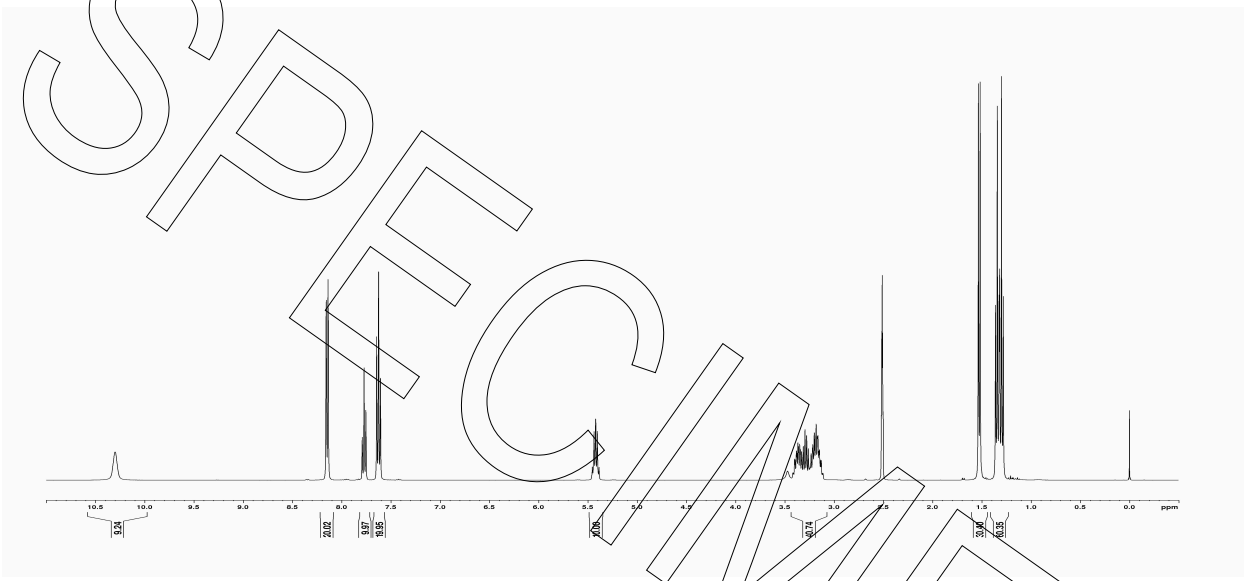
Sulphated Ash	
Method	EP 10.3, chapter 2.4.14
Result (n = 1)	2.78 %*

*not accredited testing method

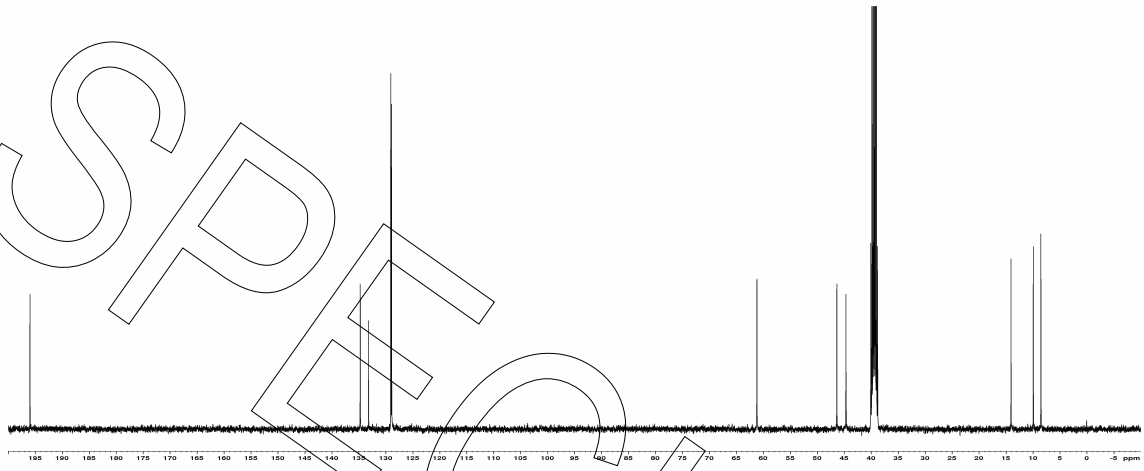
Identity

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

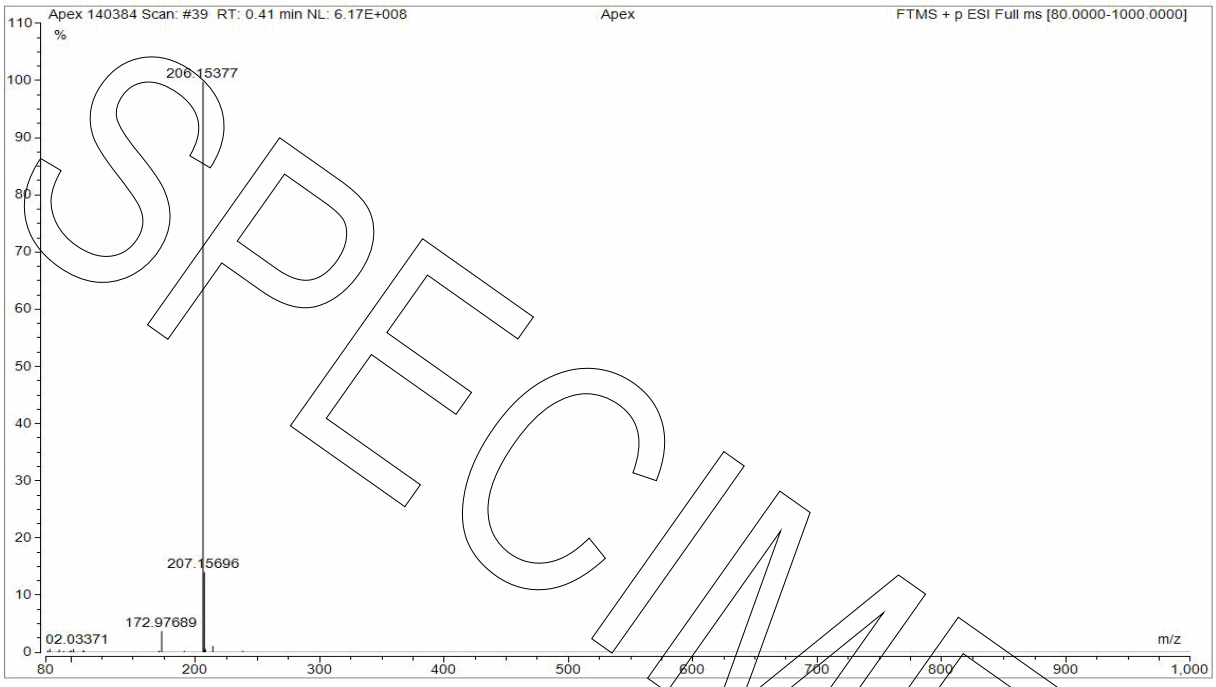
Method	Conditions	Result
^{1}H-NMR	400 MHz, DMSO-d_6	Structure confirmed



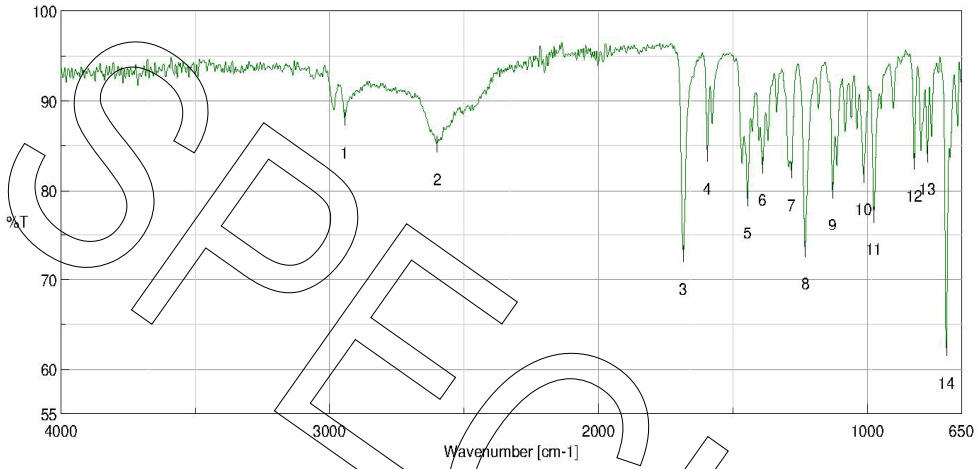
Method	Conditions	Result
^{13}C-NMR	100 MHz, DMSO-d_6	Structure confirmed



Method	Conditions	Result
MS	3.5 kV ESI+; capillary temperature: 269 °C Theoretical value: 206.15394	Structure confirmed



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



Results of Peak Find

No.	Position	Intensity
1	2943.8	88.1278
2	2600.54	85.1563
3	1684.52	72.8343
4	1594.84	84.0808
5	1445.39	79.1048
6	1389.46	82.8251
7	1281.47	82.2425
8	1231.33	73.4078
9	1129.12	80.0153
10	1013.41	81.7904
11	974.84	77.2605
12	825.384	83.2655
13	776.208	84.0639
14	705.819	62.3536

Revision table

Revision	Date	Reason for revision
00	31 May 2024	Release of the Certificate of Analysis - initial version

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