Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

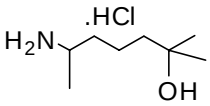
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA M299: Heptaminol hydrochloride

Report ID: M299.2020.01

Chemical Formula: $C_8H_{19}NO.HCl$

Molecular Weight: 181.7 g/mol (HCl), 145.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
05-D-02	**543-15-7 (HCl)** **372-66-7 (base)**	**99.8 ± 0.3%**

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: 6-Amino-2-methyl-2-heptanol hydrochloride.

Expiration of certification: The property values are valid till 13 May 2025, i.e. five years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body. The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: White powder sourced from an external supplier, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use this material should be stored at or below 25 ºC in a closed container in a dry, dark area.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. The long-term stability of the compound in solution has not been examined.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on seven randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Safety: Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
18 May 2020

This report supersedes any issued prior to 18 May 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15°C/min to 250 °C (2 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the free base:	
	Initial analysis:	Mean = 98.9%, s= 0.14% (7 sub samples in duplicate, March 2005)
	Re-analysis:	Mean = 99.2%, s= 0.15% (5 sub samples in duplicate, August 2008)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the free base:	
	Initial analysis:	Mean = 99.1%, s = 0.09% (4 sub samples in duplicate, October 2011)
	Re-analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, September 2014)
	Re-analysis:	Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, June 2017)
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, May 2020)
Karl Fischer analysis:		Moisture content is ≤ 0.2% mass fraction (July 2008, August 2011 and 2014) Moisture content < 0.1% mass fraction (July 2017 & May 2020)
Thermogravimetric analysis:		Volatile residue < 0.1% and non volatile content < 0.2% mass fraction (April 2005, June 2006 and July 2007)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.20 μm
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	100 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the parent compoundis reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (6.97 min):	113 (7), 110 (4), 95 (5), 69 (7), 59 (12), 56 (10), 44 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Conc NH_3/MeOH (1.5/100) Single spot observed, R_f = 0.18 Visualisation with ninhydrin
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr
	Peaks:	3322, 2974, 2948, 2892, 2740, 2647, 2560, 2107, 1623, 1537, 1473, 1396, 1375, 1267, 1219, 1186, 1155, 1123, 899, 646 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 1.20 (6H, s), 1.30 (3H, d, *J* = 6.5 Hz), 1.43-1.51 (4H, m), 1.51-1.58 (1H, m), 1.62-1.68 (1H, m), 3.28 (1H, m) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 18.7, 21.3, 29.2 (d), 36.4, 44.1, 49.0, 71.2 ppm
Melting point:		161-166 °C
Microanalysis:	Found:	C = 53.1 %, H = 11.0 %, N = 7.6% (April 2005)
	Calculated:	C = 52.9 %, H = 11.1 %, N = 7.7% (Calculated for $C_8H_{19}NO.HCl$)