



Australian Government Department of Industry,

Science and Resources

National Measurement Institute



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA M299: Heptaminol hydrochloride

Report ID: M299.2025.01 (Bottled 200825)

Chemical Formula: C₈H₁₉NO.HCl

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

Certified value

| | .HCI | |
|--------------------|-------------|--|
| H ₂ N ∖ | $\sim \sim$ | |
| | ÓН | |

| Batch No. | CAS No. | Purity (mass fraction) |
|-----------|-----------------------------------|------------------------|
| 05-D-02 | 543-15-7 (HCI) 372-66-7 (base) | 99.3 ± 0.7% |

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 7 April 2030, 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 NMI Australia. 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 °C in a closed container in a dry, dark area.

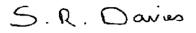
Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%

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.

Report ID: M299.2025.01 (Bottled 200825) Product release date: 15 July 2005 NMIA M299 Heptaminol hydrochloride



Dr Stephen R. Davies, Team Leader, Chemical Reference Materials, NMI. 12 May 2025

This report supersedes any issued prior to 12 May 2025.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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

Heptaminol hydrochloride

NMIA M299

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 ¹H 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: Column: Program: Injector: Detector Temp: Carrier: Split ratio: | Agilent 6890N HP-1, 30 m × 0.32 mm I.D. × 0.25 μm 60 °C (1 min), 10 °C/min to 100 °C, 15°C/min to 250 °C (2 min) 180 °C 320 °C Helium 20/1 |
|-----------------------------|--|--|
| | Relative mass fraction of Initial analysis: Re-analysis: | of the main component as the free base: Mean = 98.9%, s= 0.14% (7 sub samples in duplicate, March 2005) Mean = 99.2%, s= 0.15% (5 sub samples in duplicate, August 2008) |
| GC-FID: | Instrument: Column: Program: Injector: Detector Temp: Carrier: Split ratio: | Agilent 7890 HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm 60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min) 180 °C 320 °C Helium 20/1 |
| | Relative mass fraction of Initial analysis: Re-analysis: Re-analysis: Re-analysis: Re-analysis: | of the main component as the free base: Mean = 99.1%, s = 0.09% (4 sub samples in duplicate, October 2011) Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, September 2014) Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, June 2017) Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, May 2020) Mean = 99.4%, s = 0.01% (5 sub samples in duplicate, April 2025) |
| Karl Fischer and | alysis: | Moisture content is ≤ 0.2% mass fraction (July 2008, August 2011, August 2014, July 2017, May 2020 & April 2025) |
| Thermogravimetric analysis: | | Volatile residue < 0.1% and non volatile content < 0.2% mass fraction (April 2005, June 2006 and July 2007) |

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Spectroscopic and other characterisation data

| GC-MS: | Instrument: Column: Program: Injector: Split ratio: Transfer line temp: Carrier: Scan range: | HP6890/5973 ZB-5, 30 m × 0.25 mm l.D. × 0.20 μ m 60 °C (1 min), 10 °C/min to 300 °C (3 min) 100 °C 20/1 280 °C Helium 50-550 <i>m/z</i> | |
|----------------------|---|--|--|
| | 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) <i>m/z</i> | |
| TLC: | Conditions: | Kieselgel 60F ₂₅₄ . Conc NH ₃ /MeOH (1.5/100) Single spot observed, $R_f = 0.18$ Visualisation with ninhydrin | |
| IR: | Instrument: Range: Peaks: | Biorad FTS300MX FT-IR 4000-400 cm ⁻¹ , KBr 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: Field strength: Solvent: Spectral data: | Bruker DMX-600 600 MHz MeOH- <i>d</i> ₄ (3.31 ppm) δ 1.20 (6H, s), 1.30 (3H, d, <i>J</i> = 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: Field strength: Solvent: Spectral data: | Bruker DMX-600 151 MHz MeOH- <i>d</i> ₄ (49 ppm) δ 18.7, 21.3, 29.2 (d), 36.4, 44.1, 49.0, 71.2 ppm | |
| Melting point: | | 161-166 °C | |
| Microanalysis: | Found: Calculated: | C = 53.1 %, H = 11.0 %, N = 7.6% (April 2005) C = 52.9 %, H = 11.1 %, N = 7.7% (Calculated for C ₈ H ₁₉ NO.HCl) | |

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