



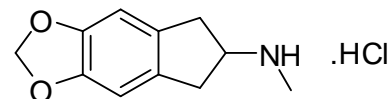
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1050: 5,6-Methylenedioxy-N-methyl-2-aminoindane hydrochloride

Report ID: D1050.2020.03

Chemical Formula: C₁₁H₁₃NO₂.HCl

Molecular Weight: 227.7 g/mol (HCl), 191.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
15-D-02	132741-82-3 (base)	98.7 ± 2.1%

IUPAC name: *N*-Methyl 6,7-dihydro-5*H*-indeno[5,6-*d*]-1,3-dioxol-6-amine hydrochloride (1:1).

Expiration of certification: The property values are valid till 2 July 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: Off-white powder prepared by synthesis, 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 reference material should be used for qualitative analysis only.

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.

Stability: In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years. 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 ten 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.
19 October 2022

This report supersedes any issued prior to 28 September 2022.

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.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. Impurities of related structure were assessed by GC-FID. The 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.

$$\text{Purity} = (100 \% - I_{\text{ORG}}) \times (100 \% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

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

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvent and elemental microanalysis.

GC-FID: Instrument: Varian CP-3800
 Column: VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 60 °C (1 min), 10 °C/min to 100 °C (2 min), 15 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1

Relative peak area of the main component as the free base:

Initial analysis: Mean = 99.4%, s = 0.04% (10 sub samples in duplicate, May 2015)

Re-analysis: Mean = 99.5%, s = 0.02% (10 sub samples in duplicate, July 2020)

Karl Fischer analysis: Moisture content 0.3% mass fraction (May 2015)
 Moisture content 0.2% mass fraction (July 2020)

Thermogravimetric analysis: Non volatile residue < 0.2% mass fraction (May 2015). The volatile content (e.g. organic solvents and/or water) could not be determined by thermogravimetric analysis.

Spectroscopic and other characterisation data

GC-MS:	Parent compound: Instrument: Agilent 6890/5973 Column: HP-1MS, 30 m x 0.25 mm I.D. x 0.25 µm Program: 60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 300 °C (3 min) Injector: 250 °C, Split ratio: 20/1 Transfer line temp: 300 °C Carrier: Helium, 1.0 mL/min Scan range: 50-550 <i>m/z</i>
	The retention time of the free base compound is 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. Free base (11.9 min): 191 (M ⁺ , 93), 160 (68), 150 (100), 135 (39), 102 (17) <i>m/z</i>
ESI-MS:	Instrument: Micromass Quatro LC Micro Operation: Positive ion mode, direct infusion at 10 µL/min Ionisation: ESI spray voltage at 3.5 kV positive ion EM voltage: 650 V Cone voltage: 10 V Peak: 192.2(M+H ⁺) <i>m/z</i>
HS-GC-MS:	Instrument: Agilent 6890/5973/G1888 Column: DB-624, 30 m x 0.25 mm I.D. x 1.4 µm Program: 50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min) Injector: 150 °C Transfer line temp: 280 °C Carrier: Helium, 1.2 mL/min Split ratio: 50/1 Solvents detected: Propan-2-ol
TLC:	Conditions: Kieselgel 60F ₂₅₄ . Methanol/NH ₃ (200/3) Single spot observed, R _f = 0.3
IR:	Instrument: Bruker Alpha Platinum ATR Range: 4000-400 cm ⁻¹ , neat Peaks: 2915, 2744, 2698, 2446, 1467, 1309, 1237, 1147, 1035, 938, 862, 826, 415 cm ⁻¹
¹ H NMR:	Instrument: Bruker Avance III-500 Field strength: 500 MHz Solvent: D ₂ O (4.79 ppm) Spectral data: δ 2.72 (3H, s), 2.98 (2H, dd, <i>J</i> = 4.5, 16.4 Hz), 3.29 (2H, dd, <i>J</i> = 7.4, 16.4 Hz), 4.02 (1H, m), 5.915 (1H, d, <i>J</i> = 3.7 Hz), 5.923 (1H, d, <i>J</i> = 3.7 Hz), 6.80 (2H, s) ppm
¹³ C NMR:	Instrument: Bruker Avance III-500 Field strength: 126 MHz Solvent: D ₂ O Spectral data: δ 30.9, 35.3, 60.1, 101.1, 105.2, 131.7, 146.7 ppm
Melting point:	227-230 °C
Microanalysis:	Found: C = 58.0%; H = 6.2%; N = 6.1%; Cl = 15.6% (May, 2015) Calculated: C = 58.0%; H = 6.2%; N = 6.2%; Cl = 15.6% (Calculated for C ₁₁ H ₁₃ NO ₂ .HCl)