



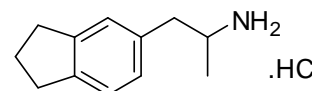
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D1060: ( $\pm$ )-5-(2-Aminopropyl)-2,3-dihydro-1H-indene hydrochloride

Report ID: D1060.2015.04

Chemical Formula: C<sub>12</sub>H<sub>17</sub>N.HCl

Molecular Weight: 211.7 g/mol (HCl), 175.3g/mol (base)



### Property value

Batch No.	CAS No.	Purity estimate
15-D-16	152623-95-5 (HCl) 13396-94-6 (base)	98.8 ± 1.6%

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

**IUPAC name:** 1-(2,3-Dihydro-1H-inden-5-yl)-2-propanamine hydrochloride (1:1).

**Expiration of certification:** The property values are valid till 21 May 2018, i.e. three years from the date of 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 solid 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 measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated 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 nine 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.  
12 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.

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**Characterisation Report:**

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

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

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

Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Varian CP-3800  
Column: VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm  
Program: 100 °C (1 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)  
Injector: 200 °C  
Detector Temp: 320 °C  
Carrier: Helium  
Split ratio: 20/1

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

Initial analysis: Mean = 99.0%, s = 0.2% (9 sub samples in duplicate, May 2015)

GC-FID: Instrument: Varian CP-3800  
Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm  
Program: 100 °C (1 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)  
Injector: 200 °C  
Detector Temp: 320 °C  
Carrier: Helium  
Split ratio: 20/1

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

Initial analysis: Mean = 99.1%, s = 0.04% (9 sub samples in duplicate, May 2015)

Karl Fischer analysis: Moisture content ≤ 0.1% mass fraction (May 2015)

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 using thermogravimetric analysis.

### Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)
	Injector:	180 °C,
	Split ratio:	20/1
	Transfer line temp:	280 °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 (8.3 min):	175 ( <i>M</i> <sup>+</sup> , 2), 132 (14), 131 (18), 129 (13), 128 (15), 117 (26), 115 (24), 91 (15), 44 (100) <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:	20 V
	Peak:	176.3 ( <i>M</i> + <i>H</i> <sup>+</sup> ) <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:	Isopropanol, diethyl ether
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Methanol/ammonia (200:3) Single spot observed, <i>R</i> <sub>f</sub> = 0.2
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm <sup>-1</sup> , neat
	Peaks:	2905, 1600, 1529, 1491, 1094, 806 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D <sub>2</sub> O (4.79 ppm)
	Spectral data:	δ 1.27 (3H, d, <i>J</i> = 6.6 Hz), 2.02 (2H, quintet, <i>J</i> = 7.4 Hz), 2.83-2.92 (6H, m), 3.56 (1H, sextet, <i>J</i> = 6.8 Hz), 7.05 (1H, d, <i>J</i> = 7.6 Hz), 7.18 (1H, s), 7.27 (1H, d, <i>J</i> = 7.6 Hz) ppm Diethyl ether and isopropanol estimated at 0.2% and 0.1% mass fraction respectively, were observed in the <sup>1</sup> H NMR
<sup>13</sup> C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D <sub>2</sub> O
	Spectral data:	δ 17.3, 25.2, 31.9, 32.2, 39.9, 49.2, 124.7, 125.4, 127.2, 133.9, 143.7, 145.5 ppm
Melting point:	214-215 °C	
Microanalysis:	Found:	C = 68.2%; H = 8.8%; N = 6.7%; Cl = 16.6% (June, 2015)
	Calculated:	C = 68.1%; H = 8.6%; N = 6.6%; Cl = 16.7% (Calculated for C <sub>12</sub> H <sub>17</sub> N.HCl)