Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

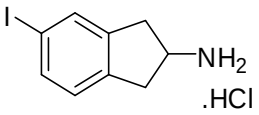
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D980: 5-Iodo-2-aminoindane hydrochloride

Report ID: D980.2022.01

Chemical Formula: $C_9H_{10}IN.HCl$

Molecular Weight: 295.6 g/mol (HCl), 259.1 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
12-D-07	1782044-60-3 (HCl) 132367-76-1 (base)	90.1 ± 2.9%

IUPAC name: 5-Iodo-2-indanamine hydrochloride (1:1).

Expiration of certification: The property values are valid till 20 October 2027, 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 sourced from an external supplier, 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 is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

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.

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.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on five 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.
31 October 2022

This report supersedes any issued prior to 31 October 2022.

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

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 estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR spectroscopy, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

Note: Di-iodo-aminoindane hydrochloride and an iodo, chloro-aminoindane hydrochloride estimated at 7% and 1% mass fraction respectively are present in this material.

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	120 °C (1 min), 10 °C/min to 300 °C (5 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 = 90.6%, s = 0.2% (10 sub samples in duplicate, October 2012)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	120 °C (1 min), 10 °C/min to 300 °C (5 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 = 91.3%, s = 0.2% (10 sub samples in duplicate, October 2012)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	120 °C (1 min), 10 °C/min to 300 °C (5 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 = 91.2%, s = 0.04% (5 sub samples in duplicate, July 2013)
	Re-analysis:	Mean = 91.0%, s = 0.08% (5 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 90.6%, s = 0.19% (5 sub samples in duplicate, July 2015)
	Re-analysis:	Mean = 91.0%, s = 0.02% (5 sub samples in duplicate, October 2022)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (October 2012). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.
Karl Fischer analysis:		Moisture content 0.2% mass fraction (October 2012) Moisture content 0.4% mass fraction (July 2013, June 2014, June 2015, October 2022)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	150 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	10/1
	The retention time of the free base is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (5.49 min):	259 (M^+, 100), 242 (19), 130 (15), 115 (33), 105 (16), 77 (9) *m/z*
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:	*tert*-Butyl methyl ether and isopropanol
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate/diethylamine (64:32:4) Single spot observed, R_f = 0.2. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3205, 2928, 2842, 2760, 2684, 2591, 2506, 2034, 1619, 1599, 1520, 1472, 1437, 1408, 1388, 1258, 1162, 1059, 1045, 973, 858, 797, 635, 417 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 2.98 (2H, dt, *J* = 5.4, 17.1 Hz), 3.23 (2H, dt, *J* = 7.6, 16.6 Hz), 3.94 (1H, m), 7.10 (1H, d, *J* = 8.0 Hz), 7.53 (1H, d, *J* = 7.8 Hz), 7.65 (1H, s), 8.45 (3H, br s) ppm
		tert-Butyl methyl ether and isopropanol estimated at 0.03% and 0.05% mass fraction respectively were observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance-400
	Field strength:	101 MHz
	Solvent:	DMSO-d_6 (39.5 ppm)
	Spectral data:	δ 36.9, 37.0, 50.3, 92.5, 127.0, 133.4, 135.5, 139.8, 143.0 ppm
Microanalysis:	Found:	C = 35.9%; H = 3.4%; N = 4.5%; Cl = 11.9%; I = 43.6% (September 2012)
	Calculated:	C = 36.6%; H = 3.8%; N = 4.7%; Cl = 12.0%; I = 42.9%; (Calculated for $C_9H_{11}N.HCl$)