Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

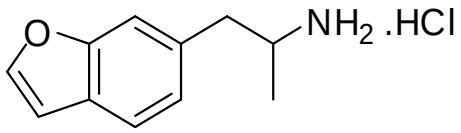
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D990: 6-(2-Aminopropyl)benzofuran hydrochloride

Report ID: D990.2023.01

Chemical Formula: $C_{11}H_{13}NO.HCl$

Molecular Weight: 211.7 (HCl), 175.2 (base) g/mol



Property value

Batch No.	CAS No.	Purity estimate
12-D-20	**286834-84-2**	**93.9 ± 2.0%**

IUPAC name: 1-(1-Benzofuran-6-yl)-2-propanamine hydrochloride (1:1)

Expiration of certification: The property values are valid till 16 February 2028, 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, 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 ºC 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.
24 February 2023

This report supersedes any issued prior to 24 February 2023.

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

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 ^{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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm or HP-1 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (15 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of main component as the free base:	
	Initial analysis:	Mean = 94.6%, s = 0.16% (10 samples in duplicate, October 2012)
	Re-analysis:	Mean = 94.5%, s = 0.07% (6 sub samples in duplicate, September 2013)
	Re-analysis:	Mean = 94.6%, s = 0.12% (5 sub samples in duplicate, February 2015)
	Re-analysis:	Mean = 94.9%, s = 0.09% (5 sub samples in duplicate, March 2016)
	Re-analysis:	Mean = 94.8%, s = 0.21% (5 sub samples in duplicate, March 2019)
	Re-analysis:	Mean = 94.9%, s = 0.12% (5 sub samples in duplicate, April 2020)
	Re-analysis:	Mean = 95.0%, s = 0.04% (5 sub samples in duplicate, February 2023)
GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	130 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of main component as the free base:	
	Initial analysis:	Mean = 94.9%, s = 0.03% (10 sub samples in duplicate, October 2012)
Thermogravimetric analysis:		The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.
		Non volatile residue < 0.1% mass fraction (September 2012)
Karl Fischer analysis:		Moisture content 1.8% mass fraction (September 2012) Moisture content 3.5% mass fraction (September 2013) Moisture content 2.2% mass fraction (February 2015) Moisture content 2.4% mass fraction (February 2016) Moisture content 3.2% mass fraction (March 2019) Moisture content 1.5% mass fraction (May 2020) Moisture content 0.9% mass fraction (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:	60 °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:	20/1
		The retention time of the free base is reported along with the major peaks in the mass spectrum. The latter is reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.
		Free base (11.9 min): 175 (M^+, 3), 131 (28), 102 (5), 77 (14), 44 (100) *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:	Toluene, acetone
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/methanol (19/1)
		Single spot observed, R_f = 0.61. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3102, 2984, 2943, 2805, 2741, 2704, 2609, 2498, 2058, 1620, 1533, 1504, 1435, 1389, 1262, 1210, 1148, 1029, 945, 877, 799, 737, 641, 412 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ1.31 (3H, d, *J* = 6.6 Hz), 3.01 (1H, dd, *J* = 7.6 14.0 Hz), 3.06 (1H, dd, *J* = 6.9, 14.0 Hz) , 3.66 (1H, sextet, *J* = 6.8 Hz), 6.89 (1H, dd, *J* = 0.9, 2.2 Hz), 7.18 (1H, dd, *J* = 1.4, 8.0 Hz), 7.47 (1H, s), 7.66 (1H, d, *J* = 8.0 Hz), 7.75 (1H, d, *J* = 2.2 Hz) ppm
		Toluene (0.1%) and acetone (0.1%) mass fractions were determined from ^{1}H NMR in CD_3OD
^{13}C NMR:	Instrument:	Bruker Avance 400
	Field strength:	100 MHz
	Solvent:	D_2O
	Spectral data:	δ 17.5, 40.1, 49.3, 106.5, 112.0, 121.7, 124.2, 126.5, 132.5, 145.9, 154.9 ppm
Melting point:		155-162 °C
Microanalysis:	Found:	C = 55.8%; H = 6.6%; N = 7.8%; Cl = 20.5% (September 2012)
	Calc:	C = 62.4%; H = 6.7%; N = 6.6%; Cl = 16.8% (Calculated for $C_{11}H_{14}ClNO$)