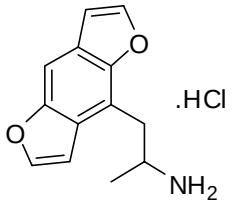
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1058: (±)-α -Methyl-benzo[1,2-b:4,5-b']difuran-4-ethanamine hydrochloride



Report ID: D1058.2023.01

Chemical Formula: $C_{13}H_{13}NO_2$.HCl

Molecular Weight: 251.7 g/mol (HCl), 215.3 g/mol (base)

Certified value

Batch No.	CAS No.	Purity (Mass fraction)
15-D-14	**260809-94-7 (base)**	**93.4 ± 1.0%**

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

IUPAC name: (±)-α-Methyl-benzo[1,2-b:4,5-b']difuran-4-ethanamine hydrochloride (1:1).

Expiration of certification: The property values are valid till 21 March 2026, i.e. three 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 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 ºC 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.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
24 March 2023

This report supersedes any issued prior to 24 March 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 certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection, 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 GC-FID and elemental microanalysis.

Warning: This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.

GC-FID:

Instrument:	Varian CP-3800
Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
Program:	120 °C (1 min), 10 °C/min to 230 °C, 30 °C/min to 300 °C (3 min)
Injector:	200 °C
Detector Temp:	320 °C
Carrier:	Helium
Split ratio:	20/1

Relative mass fraction of the main component as the free base:

Initial analysis:	Mean = 97.7%, s = 0.07% (7 sub samples in duplicate, July 2015)

GC-FID:

Instrument:	Varian CP-3800
Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
Program:	120 °C (1 min), 10 °C/min to 230 °C, 30 °C/min to 300 °C (3 min)
Injector:	200 °C
Detector Temp:	320 °C
Carrier:	Helium
Split ratio:	20/1

Relative mass fraction of the main component as the free base:

Initial analysis:	Mean = 97.8%, s = 0.14% (7 sub samples in duplicate, July 2015)

HPLC:

Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler or 2695 Separation module
Column:	Waters X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
Column oven:	40 °C
Mobile Phase:	A = MilliQ water; B = Acetonitrile 0-15 min 30% B, 15-16 min 30-80% B, 16-20 min 80% B, 20-21 min 80-30% B, 21-35 min 30% B The aqueous phase was buffered at pH 10.8 using 20 mM NH_4OAc and NH_4OH
Flow rate:	1.0 mL/min
Detector:	Shimadzu SPD-M20A or Waters 2998 PDA operating at 280 nm

Relative mass fraction of the main component:

Initial analysis:	Mean = 97.7%, s = 0.04% (5 sub samples in duplicate, June 2016)
Re-analysis:	Mean = 97.2%, s = 0.02% (5 sub samples in duplicate, June 2017)
Re-analysis:	Mean = 96.4%, s = 0.05% (5 sub samples in duplicate, May 2020)
Re-analysis:	Mean = 96.5%, s = 0.1% (5 sub samples in duplicate, March 2023)

Karl Fischer analysis:
Moisture content 0.7% mass fraction (May 2015)
Moisture content 0.8% mass fraction (June 2016)
Moisture content 1.0% mass fraction (June 2017 and May 2020)
Moisture content 0.9% mass fraction (March 2023)

Thermogravimetric analysis: Volatiles content 0.3% and non-volatile residue 0.2% mass fraction (May 2015)

Spectroscopic and other characterisation data

GC-MS:	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 300 °C (5 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention times of the free base compound and *N*-acetyl derivative are 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 (15.3 min):	215 (M^+, 2), 172 (56), 171 (31), 115 (14), 89 (4), 63 (5), 44 (100) *m/z*
	N- Acetyl (18.7 min):	257 (M^+, 13), 198 (100), 171 (49), 115 (18), 89 (6), 86 (20), 44 (78) *m/z*
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:	15 V
	Peak:	216.1 ($M+H^+$) *m/z*
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2926, 1580, 1551, 1516, 1416, 1381, 1212, 1147, 1021, 761, 702, 536 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 1.13 (3H, d, *J* = 6.5 Hz), 3.30 (1H, dd, *J* = 9.5, 13.0 Hz), 3.52 (1H, dd, *J* = 4.8, 13.1 Hz), 3.56 (1H, m), 7.02 (1H, d, *J* = 2.2 Hz), 7.29 (1H, d, *J* = 1.5 Hz), 7.73 (1H, s), 8.03 (1H, d, *J* = 2.3 Hz), 8.04 (1H, d, *J* = 2.3 Hz), 8.4 (3H, bs) ppm
		Isopropanol (1.0%) and diethyl ether (0.9%) estimated mass fraction was observed in the ^{1}H NMR (2020).
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 17.8, 31.9, 47.1, 101.1, 105.6, 107.3, 111.3, 125.1, 125.4, 146.7, 150.1, 151.3 ppm
Melting point:		274-278 °C
Microanalysis:	Found:	C = 61.7%; H = 5.7%; N = 5.4%; Cl = 13.9% (May, 2015)
	Calculated:	C = 62.0%; H = 5.6%; N = 5.6%; Cl = 14.1% (Calculated for $C_{13}H_{13}NO_2$.HCl)