Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

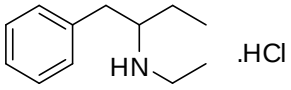
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D997: (±)-N-Ethyl-1-phenyl-2-butylamine hydrochloride

Report ID: D997.2022.01 (Bottled 160825)

Chemical Formula: $C_{12}H_{19}N.HCl$

Molecular Weight: 213.8 g/mol (HCl), 177.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
13-D-13	29805-52-5 (HCl) 119486-07-6 (base)	92.1 ± 1.0%

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

IUPAC name: (±)-*N*-Ethyl-1-phenyl-2-butylamine hydrochloride.

Expiration of certification: The property values are valid till 12 May 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 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of five 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 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.
20 July 2022

This report supersedes any issued prior to 19 July 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. The certified 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 and quantitative nuclear magnetic resonance (qNMR).

GC-FID:	Instrument:	Agilent 6890, 7890, 8890 or Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	80 °C (1 min), 5 °C/min to 120 °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 = 99.6%, s = 0.03% (10 sub samples in duplicate, June 2013)
	Re-analysis:	Mean = 99.7%, s = 0.05% (5 sub samples in duplicate, April 2014)
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, April 2015)
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, September 2016)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, July 2017)
	Re-analysis:	Mean = 99.8%, s = 0.06% (5 sub samples in duplicate, July 2018)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, May 2022)
Karl Fischer analysis:		Moisture content 2.8% mass fraction (June 2013) Moisture content 4.4% mass fraction (September 2013) Moisture content 4.3% mass fraction (April 2014) Moisture content 6.9% mass fraction (March 2015) Moisture content 5.6% mass fraction (September 2015) Moisture content 6.8% mass fraction (August 2016) Moisture content 7.4% mass fraction (August 2017) Moisture content 8.0% mass fraction (June 2018) Moisture content 7.9% mass fraction (July 2019) Moisture content 7.6% mass fraction (May 2022)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (June 2013). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material or degradation at elevated temperatures.
QNMR:	Instrument:	Bruker Avance-III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (7.30 ppm) = 97.7%, s = 0.02% (5 sub samples, June 2013)

Spectroscopic and other characterisation data

GC-MS:	Free base:	
	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	80 °C (1 min), 5 °C/min to 170 °C, 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the free base 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 (11.1 min):	148 (9), 91 (16), 86 (100), 58 (7) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive/Negative 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:	178.2 ($M+H^+$) *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:	Isopropanol, diethyl ether
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate/diethyl amine (15/3/0.2) Single spot observed, R_f = 0.4
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3360, 3318, 2965, 2791, 2724, 2484, 2391, 1601, 1498, 1456, 1305, 737, 699 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 0.99 (3H, t, *J* = 7.6 Hz), 1.33 (3H, t, *J* = 7.2 Hz), 1.61-1.78 (2H, m), 2.93 (1H, dd, *J* = 8.8, 13.6 Hz), 3.11 (2H, q, *J* = 7.2 Hz), 3.13 (1H, dd, *J* = 5.0, 13.9 Hz), 3.44 (1H, m), 7.26-7.38 (5H, m) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 9.2, 11.7, 23.6, 37.0, 41.8, 61.4, 128.3, 130.0, 130.4, 137.4 ppm
Melting point:		137-140 °C
Microanalysis:	Found:	C = 64.0%; H = 9.8%; N = 6.3%; Cl% = 15.7% (June, 2015)
	Calculated:	C = 67.4%; H = 9.4%; N = 6.6%; Cl% = 16.6% (Calculated for $C_{12}H_{19}N.HCl$)