



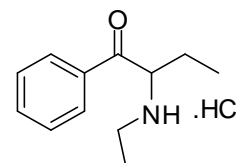
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1013: 2-(Ethylamino)-1-phenyl-1-butanone hydrochloride

Report ID: D1013.2016.04

Chemical Formula: C₁₂H₁₇NO.HCl

Molecular Weight: 227.7 g/mol (HCl), 191.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-05	1354631-28-9 (free base)	96.5 ± 1.9%

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

IUPAC name: 2-(Ethylamino)-1-phenyl-1-butanone hydrochloride (1:1).

Expiration of certification: The property values are valid till 22 March 2019, 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: White crystalline 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 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 °C 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: 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 HPLC with UV detection 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.
21 September 2022

This report supersedes any issued prior to 21 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.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained from a combination of traditional analytical techniques including HPLC with UV detection thermogravimetric analysis, Karl Fischer analysis and ¹H 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_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue

Supporting evidence is provided by qualitative head space GC-MS analysis and elemental microanalysis.

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

HPLC:	Instrument: Shimadzu Binary pump LC-20AB, SIL-20A HT autosampler Column: X-Bridge C-18, 5.0 μm (4.6 mm x 150 mm) Column oven: 40 °C Mobile Phase: Milli Q water (A) and acetonitrile (B) 0-14 min 35% B, 14-15 min 35%-80% B, 15-19 min 80% B, 19-20 min 80%-35% B, 20-25 min 35% B. The aqueous phase was buffered at pH 10.8 using 20 mM NH ₄ OAc and NH ₄ OH Flow rate: 1 mL/min Detector: SPD-M20A PDA operating at 242 nm Relative mass fraction of the main component: Initial analysis: Mean = 99.7%, s = 0.06% (5 sub samples in duplicate, March 2016)
HPLC:	Instrument: Shimadzu Binary pump LC-20AB, SIL-20A HT autosampler Column: X-Bridge C-18, 5.0 μm (4.6 mm x 150 mm) Column oven: 40 °C Mobile Phase: Acetonitrile/MilliQ water (30:70) The aqueous phase was buffered at pH 10.8 using 20 mM NH ₄ OAc and NH ₄ OH Flow rate: 1.5 mL/min Detector: SPD-M20A PDA operating at 225 nm Relative mass fraction of the main component: Initial analysis: Mean = 99.9%, s = 0.004% (7 sub samples in duplicate, March 2014) Re-analysis: Mean = 99.9%, s = 0.002% (5 sub samples in duplicate, March 2015)
Thermogravimetric analysis:	Non-volatile residue < 0.2% mass fraction (March 2014). 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.
Karl Fischer analysis:	Moisture content 2.4% mass fraction (March 2014) Moisture content 2.9% mass fraction (March 2015) Moisture content 3.1% mass fraction (March 2016)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	100 $^{\circ}$ C (1 min), 5 $^{\circ}$ C /min to 120 $^{\circ}$ C (2 min), 30 $^{\circ}$ C/min to 300 $^{\circ}$ C (3 min)
	Injector:	200 $^{\circ}$ C
	Carrier:	Helium, 1.0 mL/min
	Transfer line temp:	300 $^{\circ}$ C
	Split ratio:	20/1
	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.	
	(9.4 min):	162 (3), 105 (12), 86 (100), 84 (12), 77 (23), 58 (18), 41 (12) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	192.2 (M+H ⁺) <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 μ m
	Program:	50 $^{\circ}$ C (5 min), 7 $^{\circ}$ C /min to 120 $^{\circ}$ C, 15 $^{\circ}$ C/min to 220 $^{\circ}$ C (8.3 min)
	Injector:	150 $^{\circ}$ C
	Carrier:	Helium, 1.2 mL/min
	Transfer line temp:	280 $^{\circ}$ C
	Split ratio:	50/1
	Solvents detected:	Propanal
TLC:	Conditions:	Kieselgel 60F254. NH ₃ /methanol (3/200)
		Single spot observed, R _f = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3373, 2933, 2798, 2739, 2518, 1691, 1597, 1444, 1313, 1231, 1122, 963, 806, 702, 556 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance-III-600
	Field strength:	600 MHz
	Solvent:	DMSO- <i>d</i> ₆ (2.50 ppm)
	Spectral data:	δ 0.76 (3H, t, <i>J</i> = 7.6 Hz), 1.28 (3H, t, <i>J</i> = 7.3 Hz), 1.93 (1H, m), 2.09 (1H, m), 2.91 (1H, m), 3.01 (1H, m), 5.29 (1H, m), 7.60 (1H, m), 7.75 (1H, m), 8.08 (1H, m), 9.16 (1H, bs), 9.76 (1H, bs) ppm
	Ethylamine hydrochloride at 0.2% mass fraction was observed in the ¹ H NMR	
¹³ C NMR:	Instrument:	Bruker Avance-III-600
	Field strength:	151 MHz
	Solvent:	DMSO- <i>d</i> ₆ (39.5 ppm)
	Spectral data:	δ 8.3, 11.1, 22.8, 41.1, 61.2, 128.8, 129.2, 134.0, 134.7, 196.2 ppm
	Melting point:	220-221 $^{\circ}$ C
Microanalysis:	Found:	C = 62.1%; H = 8.2%; N = 6.0%; Cl = 15.3% (March 2014)
	Calculated:	C = 63.3%; H = 8.0%; N = 6.2%; Cl = 15.6% (Calculated for C ₁₂ H ₁₇ NO.HCl)