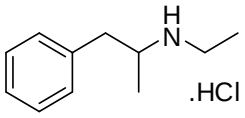
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D753c: (±)-Ethylamphetamine hydrochloride



Report ID: D753c.2020.03 (Bottled 160901)

Chemical Formula: $C_{11}H_{17}N.HCl$

Molecular Weight: 199.7 g/mol (HCl), 163.3 g/mol (base)

Property value

Batch No.	CAS No.	Purity estimate
14-D-26	**16105-78-5 (HCl)** **457-87-4 (base)**	**99.6 ± 0.7%**

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-propanamine hydrochloride.

Expiration of certification: The property values are valid till 25 March 2025, 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: White 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 ºC in a closed container in a dry, dark area.

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 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.
15 September 2022

This report supersedes any issued prior to 15 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. Impurities of related structure were assessed by GC-FID. The purity value was obtained from a combination of traditional analytical techniques The purity estimate by traditional analytical techniques 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 qualitative headspace GC-MS analysis of occluded solvents, elemental microanalysis, and quantitative nuclear magnetic resonance (qNMR). The purity estimate by qNMR was obtained using one proton multiplet at 3.53 ppm against a certified internal standard of maleic acid.

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	80 °C (1 min), 10 °C/min to 120 °C (3 min), 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 = 99.8%, s = 0.01% (7 sub samples in duplicate, September 2014)
GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60°C (1 min),10°C/min to 100 C, 15°C/min to 250°C, 30°C/min to 300°C (3 min)
	Injector:	250 °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 = 99.5%, s = 0.09% (5 sub samples in duplicate, March 2017)
	Re-analysis	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, March 2020)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (September 2014 and March 2020) Moisture content 0.2% mass fraction (March 2017)
Thermogravimetric analysis:		Non-volatile residue < 0.2 % mass fraction (September 2014). The volatile content (e.g. organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material.
QNMR:	Instrument:	Bruker Avance-III 500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (3.53 ppm) = 99.4%, s = 0.3% (5 sub samples, October 2014)

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
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	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 (8.7 min):	162 (M^+-H, 1), 148 (3), 117 (3), 91 (20), 72 (100), 44 (21) *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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Isopropanol
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate/diethylamine (15:3:0.2) Single spot observed, R_f = 0.24. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2972, 2785, 2484, 2390, 1588, 1495, 1456, 1388, 1372, 1171, 1096, 1035, 751 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.22 (3H, d, *J* = 6.6 Hz), 1.24 (3H, t, *J* = 7.3 Hz), 2.83 (1H, dd, *J* = 8.7, 13.7 Hz), 3.04-3.18 (3H, m), 3.55 (1H, m), 7.28-7.30 (2H, m), 7.32-7.35 (1H, m), 7.38-7.41 (2H, m) ppm Isopropanol estimated at 0.3% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	101 MHz
	Solvent:	D_2O
	Spectral data:	δ 10.6, 15.2, 38.8, 40.1, 54.9, 127.4, 129.0, 129.5, 135.9 ppm
Melting point:		147-150 °C
Microanalysis:	Found:	C = 66.3%; H = 9.3%; N = 7.1%; Cl = 17.8 (September 2014)
	Calculated:	C = 66.2%; H = 9.1%; N = 7.0%; Cl = 17.8 (Calculated for $C_{11}H_{18}ClN$)