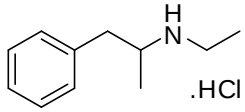
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D753c.2017.01 (Bottled 160901)
This batch of bottles was prepared from the bulk material on 1st September 2016.

Compound Name: **(±)-Ethylamphetamine hydrochloride**
Collection Number: D753c
Chemical Formula: $C_{11}H_{17}N.HCl$
CAS Number: 16105-78-5
Description: White solid
Batch Number: 14-D-26
Molecular Weight: 199.7 (HCl), 163.3 (base)
Release Date: 15th October 2014
Structure:



Synonyms: (±)-*N*-Ethyl-α-methylbenzeneethanamine hydrochloride
N-Ethyl-α-methyl-phenethylamine hydrochloride
(±)-*N*-Ethylamphetamine hydrochloride
dl-*N*-Ethylamphetamine hydrochloride
N-Ethylamphetamine hydrochloride
Ethylamphetamine hydrochloride

Purity (mass fraction): 99.5 ± 2.0% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID detection, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. The purity estimate by qNMR was obtained using one proton multiplet at 3.53 ppm against a certified internal standard of maleic acid. Supporting evidence is provided by headspace GC-MS analysis of occluded solvent and elemental microanalysis.

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 peak area response of main component:		
	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 peak area response of main component:		
	Initial analysis:	Mean = 99.5%, s = 0.09% (5 sub samples in duplicate, 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.	
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (September 2014) Moisture content 0.2% mass fraction (March 2017)	
QNMR:	Instrument:	Bruker Avance-III 500	
	Field strength:	500 MHz	Solvent: D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% m/m)	
	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	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 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	Transfer line temp: 280 °C
	Carrier:	Helium, 1.2 mL/min	Split ratio: 50/1
	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) Calc: C = 66.2%; H = 9.1%; N = 7.0%; Cl = 17.8 (Calculated for $C_{11}H_{18}ClN$)	

Expiration of certification
The property values are valid till 23rd March 2020, 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.

The long-term stability of the compound in solution has not been examined.

In the absence of 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 10 years.

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.

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.

Intended use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 12 April, 2017.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 12th April 2017.