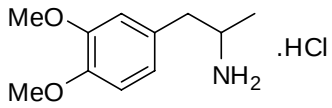
Australian Government

National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D453b.2011.02

Compound Name: **(±)-3,4-Dimethoxyamphetamine hydrochloride**
Description: Off white solid
Collection Number: D453b
Batch Number: 11-D-21
Chemical Formula: $C_{11}H_{17}NO_2.HCl$
Molecular Weight: 231.7 (HCl), 195.3 (base)
CAS Number: 13078-75-6
Batch Production Completed: November 2011
Structure:



Synonyms: 3,4-Dimethoxy-α-methylbenzeneethanamine hydrochloride
(±)-3,4-Dimethoxy-α-methyl-phenethylamine hydrochloride
(±)-3,4-Dimethoxyphenylisopropylamine hydrochloride
(±)-3,4-DMA HCl

Purity (mass fraction): 92.3 ± 1.9% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. Supporting evidence is provided by headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800	
Free base	Column:	VF-1MS, 29.58 m × 0.32 mm I.D. × 0.25 µm	
	Program:	100 °C (1 min), 10 °C/min to 170 °C, 20 °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 = 99.7%, s = 0.07% (10 sub samples in duplicate, November 2011)	

GC-FID:	Instrument:	Varian CP-3800	
Free base	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	100 °C (1 min), 10 °C/min to 170 °C, 20 °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 = 0.06% (10 sub samples in duplicate, November 2011)	

Thermogravimetric analysis: Volatile content 6.7% and non volatile residue < 0.2% mass fraction (November 2011)

Karl Fischer analysis: Moisture content 7.3% mass fraction (November 2011)

Spectroscopic and other characterisation data

GC-MS: Free base	Instrument:	Agilent 6890	
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm	
	Program:	90 °C (1 min), 10 °C/min to 180 °C (7 min), 30 °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 (10.1 min): 152 (60), 151 (10), 137 (7), 107 (6), 44 (100) m/z

LC/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	5 V
	Peak:	196.2 ($M+H^+$) 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:	Ethyl acetate, isopropanol	

TLC:	Conditions:	Kieselgel 60F_{254}. TBME/diethyl ether/diethylamine (9:9:1) Single spot observed, R_f = 0.15. Visualisation with UV at 254 nm

IR:	Instrument:	FT-IR, Biorad WIN FTS40	
	Range:	4000-500 cm^{-1}, KBr powder	
	Key Peaks:	1519, 1264, 1147, 1025 cm^{-1}	
	^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz	Solvent: CD_3OD (3.31 ppm)
	Spectral data:	δ 1.28 (3H, d, *J* = 6.6 Hz), 2.78 (1H, dd, *J* = 7.9, 13.7 Hz), 2.92 (1H, dd, *J* = 6.5, 13.7 Hz), 3.52 (1H, m), 3.81 (3H, s), 3.84 (3H, s), 6.81 (1H, dd, *J* = 2.0, 8.2 Hz), 6.88 (1H, d, *J* = 2.0 Hz), 6.93 (1H, d, *J* = 8.2 Hz) ppm	

Ethyl acetate and isopropanol estimated at 1.0% and 0.1% mass fraction respectively were observed in the ^{1}H NMR

^{13}C NMR:	Instrument:	Bruker DMX-600	
	Field strength:	151 MHz	Solvent: CD_3OD (49.0 ppm)
	Spectral data:	δ 18.4, 41.4, 50.4, 56.48, 56.47, 113.2, 114.0, 122.8, 130.1, 149.8, 150.7 ppm	
	Melting point:	144-147 °C	

Microanalysis:

Found: C = 53.0%; H = 8.2%; N = 5.6% (November, 2011)
Calc: C = 57.0%; H = 8.7%; N = 6.0% (Calculated for $C_{11}H_{17}NO_2$.HCl)
Calc: C = 52.9%; H = 8.1%; N = 5.6% (Calculated for $C_{11}H_{17}NO_2$.HCl + 7.3% H_2O)

Expiration of certification
The property values are valid till 2nd November 2014, i.e. three years from the date of 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.

This material has been given a shelf life of three years from the date of certification. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

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-FIDon ten randomly selected 1-2 mg sub samples of the material. The material was judged to be 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 20 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: 19 July, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 16th July 2012.