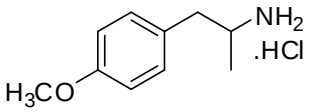
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D756b.2016.01 (Bottled 160531)
This batch of bottles was prepared from the bulk material on 31st May 2016

Compound Name: **(±)-4-Methoxyamphetamine hydrochloride**
Description: White powder
Collection Number: D756b
Batch Number: 15-D-20
Chemical Formula: $C_{10}H_{15}NO.HCl$
Molecular Weight: 201.7 (HCl), 165.2 (base)
CAS Registry Number: 3706-26-1 (HCl), 64-13-1 (base)
Release date: 16th September 2015.
Structure:



Synonyms: (±)-PMA hydrochloride
(±)-4-Methoxy-α-methylbenzeneethanamine hydrochloride
(±)-*p*-Methoxy-α-methylphenethylamine hydrochloride

Purity (mass fraction): 99.5 ± 0.6% (95% coverage interval)

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

GC-FID:	Instrument:	Agilent 6890N	
	Column:	HP-1 Capillary, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	60 °C (1 min), 10 °C/min to 150 °C, 20 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Initial analysis:	Mean = 99.9%, s = 0.02% (10 samples in duplicate, August 2015)	
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, July 2016)	
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (August 2015)	
Karl Fischer analysis:		Moisture content 0.1% mass fraction (August 2015 and July 2016)	
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 (2.9 ppm) = 99.7%, s = 0.2% (4 sub samples, August 2015)	
	Initial analysis:	Mean (3.6 & 3.8 ppm) = 100.1%, s = 0.2% (4 sub samples, August 2015)	
	Initial analysis:	Mean (7.2 ppm) = 99.8%, s = 0.2% (4 sub samples, August 2015)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5971
	Column:	30 m x 0.25 mm I.D. x 0.25 µm
	Program:	60 °C, 10 °C/min to 100 °C, 15 °C/min to 250 °C
	Injector:	180 °C Transfer line temp: 340 °C
	Carrier:	Helium, 1.0 mL/min Split ratio: 20/1
	The retention time of the free base is reported with the peaks in the mass spectrum. The latter are reported as mass/charge ratio and (in brackets) as a percentage relative to the base peak.	
	9.15 min:	165 (M^+, 3), 150 (3), 134 (4), 122 (100), 121 (42), 107 (9), 91 (13), 78 (22) m/z
TLC:	Conditions:	Kieselgel 60F_{254} (Ethyl acetate/diethylamine, 20:1) Single spot observed, R_f = 0.2 (3 replicates)
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2913, 1612, 1506, 1251, 1178, 1031, 807 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz Solvent: MeOH-d_4 (3.31ppm) δ 1.29 (3H, d, *J* = 6.6 Hz), 2.75 (1H, dd, *J* = 8.3, 13.8 Hz), 2.94 (1H, dd, *J* = 6.1, 13.8 Hz), 3.47 (1H, m), 3.78 (3H, s), 6.91 (2H, d, *J* = 8.7 Hz), 7.18 (2H, d, *J* = 8.7 Hz) ppm Isopropanol and diethyl ether estimated at 0.2% and 0.1% mass fraction respectively were observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	126 MHz Solvent: CD_3OD (49.0 ppm)
	Spectral data:	δ 18.2, 40.9, 50.4, 55.7, 115.3, 129.2, 131.4, 160.4 ppm
Melting point:		211-212 °C
Microanalysis:		Found: C = 59.6%; H = 8.2%; N = 7.0% (August 2015) Calc.: C = 59.6%; H = 8.0%; N = 6.9% (Calculated for $C_{10}H_{15}NO.HCl$)

Expiration of certification
The property values are valid till 29th July 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.

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 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.

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: 9 August, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 9th August 2016.