Australian Government

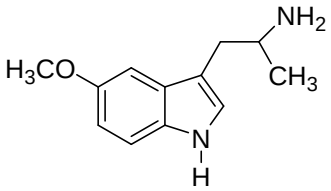
National Measurement Institute

ACCREDITED FOR TECHNICAL COMPETENCE

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D899.2012.03

Compound Name: **5-Methoxy-α-methyltryptamine**
Collection No: D899
Chemical Formula: $C_{12}H_{16}N_2O$
CAS No: 1137-04-8
Structure:



Description: Tan coloured solid
Batch No: 05-D-015
Molecular Weight: 204.3
Release date: 30th October 2005

Synonyms: 5-Methoxy-α-methyl-3-ethanamine-indole
3-(1-Amino-1-methylethyl)-5-methoxy-indole
3-(2-Aminopropyl)-5-methoxy-indole

Purity (mass fraction): 98.1 ± 1.4% (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, Karl Fischer and ^{1}H NMR. Supporting evidence provided by elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 6890N	
	Column:	VF-1MS or HP-1, 30.0 m × 0.32 mm I.D. × 0.25 μm	
	Program:	180 °C (9 min), 25 °C/min to 300 °C (6 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.9%, s = 0.01% (7 sub samples in duplicate, October 2005)	
	Re-analysis	Mean = 99.9%, s = 0.001% (5 sub samples in duplicate, February 2008)	
	Re-analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, March 2009)	
	Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, February 2012)	

Thermogravimetric analysis: Volatile content < 0.1% mass fraction. Non volatile residue 0.7% mass fraction (October 2005 & February 2007).

Karl Fischer analysis: Moisture content 0.9% mass fraction. (2 sub samples, March 2009)
Moisture content 0.6% mass fraction. (2 sub samples, February 2012)

GC-MS:	Instrument:	HP 6890 / 5973	
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.20 μm	
	Program:	160 °C (1 min), 25 °C/min to 250 °C (2 min), 40 °C/min to 300 °C (4 min)	
	Injector:	180°C	Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 5/1

The retention time of the parent compound 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.

5.3 min: 204 (M^+, 4), 161 (100), 160 (48), 146 (21), 117 (14), 91 (33) m/z

TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/Methanol /Diethylamine (95/5/0.4). Single spot observed, R_f = 0.39. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR.
	Range:	4000-400cm^{-1}, KBr pellet.
	Peaks:	3361, 3293, 3134, 1620, 1578, 1479, 1442, 1219, 1079, 924, 795 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DPX2-300
	Field strength:	300 MHz Solvent: $CDCl_3$
	Spectral data:	δ 1.18 (3H, d, *J* = 6.4 Hz), 2.63 (1H, dd, *J* = 8.3, 13.9 Hz), 2.85 (1H, dd, *J* = 4.9, 14.3 Hz), 3.29 (1H, m), 3.86 (3H, s), 6.86 (1H, dd, *J* = 2.3, 8.7 Hz), 7.00 (1H, d, *J* = 2.3 Hz), 7.05(1H, d, *J* = 2.3 Hz), 7.24 (1H, d, *J* = 9.1 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DPX2-300
	Field strength:	75 MHz Solvent: $CDCl_3$
	Spectral data:	δ 23.8, 36.0, 47.2, 55.9, 100.9, 111.8, 112.0, 113.4, 123.3, 128.1, 131.5, 153.9 ppm.
Melting point:		98-100 °C (Lit 100-101 °C)
Microanalysis:		Found: C = 69.9%; H = 8.1%; N = 13.7% Calc: C = 70.6%; H = 7.9%; N = 13.7% (Calculated for $C_{12}H_{16}N_2O$)

Expiration of certification
The property values are valid till 17th February 2017, 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/ampoules 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 demonstrated stability over a minimum period of 5 years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

Homogeneity assessment
The homogeneity of the material was assessed using purity assay by GCFID on 5 randomly selected 1.5 mg 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.

Metrological Traceability
The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis.

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: 14 November, 2014.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 14th November 2014.