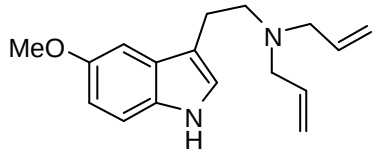
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D954.2016.01

Compound Name: **5-Methoxy-*N*,*N*-diallyltryptamine**
Collection Number: D954
Chemical Formula: $C_{17}H_{22}N_2O$
CAS Number: 928822-98-4
Description: White powder
Batch Number: 10-D-06
Molecular Weight: 270.4
Release date: 30th September 2010

Structure:



Synonyms: 5-MeO-DALT
(2-(5-Methoxyindol-3-yl)ethyl)diprop-2-enylamine
5-Methoxy-*N*,*N*-di-2-propen-1-yl-indole-3-ethanamine
N,*N*-Diallyl-5-methoxytryptamine
3-(2-(Diallylamino)ethyl)-5-methoxyindole

Purity (mass fraction): 99.6 ± 1.5% (95% coverage interval)

The purity estimate was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR analysis. Supporting evidence is provided by HPLC with UV detection, QNMR analysis using a certified internal standard of dimethylterephthalate, headspace GC-MS analysis of occluded solvent and elemental microanalysis.

GC-FID:	Instrument:	Varian 3800	
	Column:	VF-1ms, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	200 °C (15 min), 20 °C/min to 300 °C (5 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 = 0.02% (10 sub samples in duplicate, July 2010)	
	Re-analysis:	Mean = 99.9%, s = 0.005% (5 sub samples in duplicate, June 2011)	
	Re-analysis:	Mean = 99.9%, s = 0.007% (5 sub samples in duplicate, April 2012)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, April 2013)	
	Re-analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, February 2016)	
GC-FID:	Instrument:	Varian 3800	
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	200 °C (17 min), 20 °C/min to 300 °C (5 min)	
	Injector:	200 °C	Detector Temp: 300 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.8%, s = 0.15% (10 sub samples in duplicate, July 2010)	
GC-FID:	Instrument:	Agilent 6890	
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	200 °C (15 min), 20 °C/min to 300 °C (5 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% (10 sub samples in duplicate, July 2010)	

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/MilliQ water (75:25)
	Flow rate:	1.0 mL/min
	Detector:	Waters PDA 996 operating at Max plot
	Retention time:	8.47 min
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.03% (10 sub samples in duplicate, September 2010)
Thermogravimetric analysis:		Non volatile residue < 0.1% mass fraction (July 2010). 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 0.3% mass fraction (February 2016)
QNMR:	Instrument:	Bruker Avance-600
	Field strength:	600 MHz Solvent: $CDCl_3$ (7.26 ppm) + 1 drop D_2O
	Internal standard:	Dimethylterephthalate (100% mass fraction)
	Initial analysis:	Mean (5.9 ppm) = 99.4%, s = 0.13% (3 sub samples, September 2010)

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:	120 °C (1 min), 10 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Transfer line temp: 300 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 30/1
	The retention time of the parent compound is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.		
	Parent (14.3 min): 270 (M^+, 2), 241 (5), 160 (8), 110 (100), 41 (12) m/z		
ESI-MS:	Instrument:	Micromass Quatro Micro	
	Operation:	Positive ion mode, direct infusion at 5 μL/min	
	Ionisation:	ESI spray voltage at 3.5 kV positive ion	
	EM voltage:	650 V	
	Cone voltage:	20 V	
	Peak:	293 $(M+Na)^+$, 271 $(M+H)^+$ m/z	
ESI-MS:	Instrument:	Micromass Quatro Micro	
	Operation:	Negative ion mode, direct infusion at 5 μL/min	
	Ionisation:	ESI spray voltage at 3.0 kV negative ion	
	EM voltage:	650 V	
	Cone voltage:	20 V	
	Peak:	269 $(M-H)^+$ 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:	Isopropanol	
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/methanol (9/1) Single spot observed, R_f = 0.64. Visualisation with UV at 254 nm	
IR:	Instrument:	Biorad FTS300MX FT-IR	
	Range:	4000-500 cm^{-1}, KBr powder	
	Peaks:	3124, 3044, 3001, 2943, 2879, 2840, 2597, 1586, 1490, 1473, 1456, 1447, 1239, 1220, 1110, 1059, 1031, 1006, 934, 920, 792, 640 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker Avance 400	
	Field strength:	400 MHz	Solvent: $CDCl_3$ (7.26 ppm)
	Spectral data:	δ 2.78–2.85 (2H, m), 2.87–2.94 (2H, m), 3.25 (4H, d, *J* = 6.5 Hz), 3.87 (3H, s), 5.18 (2H, dm, *J* = 10.2 Hz), 5.26 (2H, dm, *J* = 17.1 Hz), 5.94 (2H, m), 6.85 (1H, dd, *J* = 2.4, 8.8 Hz), 6.98 (1H, d, *J* = 2.2 Hz), 7.04 (1H, d, *J* = 2.4 Hz), 7.23 (1H, d, *J* = 8.8 Hz), 7.95 (1H, s) ppm	
	Isopropanol estimated at 0.1% mass fraction was observed in the ^{1}H NMR		
^{13}C NMR:	Instrument:	Bruker Avance 400	
	Field strength:	100 MHz	Solvent: $CDCl_3$ (77.16 ppm)
	Spectral data:	δ 23.0, 53.9, 56.0, 57.1, 100.9, 111.9, 112.2, 114.4, 117.6, 122.4, 128.1, 131.5, 135.9, 154.0 ppm	
Melting point:		103-104 °C	
Microanalysis:		Found: C = 75.7%; H = 8.4%; N = 10.5% (July 2010) Calc: C = 75.5%; H = 8.2%; N = 10.4% (Calculated for $C_{17}H_{22}N_2O$)	

Expiration of certification
The property values are valid till 26th February 2021, 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.

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

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.

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 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. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

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 March, 2016.

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