Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

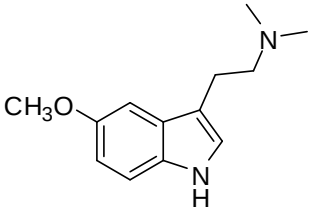
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D999: 5-Methoxy-N,N-dimethyltryptamine

Report ID: D999.2019.03

Chemical Formula: $C_{13}H_{18}N_2O$

Molecular Weight: 218.3 g/mol




## Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|---|---|---|
| **13-D-15** | **1019-45-0** | **98.9 ± 2.3%** |

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

**IUPAC name:** 2-(5-Methoxy-1H-indol-3-yl)-*N,N*-dimethylethanamine

**Expiration of certification:** The property values are valid till 15 April 2024, 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.

**Description:** Beige solid sourced from an external supplier, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

**Intended use:** This certified reference material is suitable for use as a primary calibrator.

**Instructions for use:** Equilibrate the bottled material to room temperature before opening.

**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.

**Metrological traceability:** The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

**Stability:** This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. The long-term stability of the compound in solution has not been examined.

**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.

**Safety:** Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
21 September 2022

This report supersedes any issued prior to 21 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

## Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and $^{1}$H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - $I_{ORG}$) x (100 % - $I_{VOL}$ – $I_{NVR}$) Equation 1

$I_{ORG}$ = Organic impurities of related structure, $I_{VOL}$ = volatile impurities, $I_{NVR}$ = non-volatile residue.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

| | | |
|---|---|---|
| GC-FID: | Instrument: | Varian CP-3800 |
| | Column: | VF-1ms, 30 m × 0.32 mm I.D. × 0.25 μm |
| | Program: | 100 °C (2 min), 10 °C/min to 260 °C (1 min), 30 °C/min to 300 °C (3 min) |
| | Injector: | 250 °C |
| | Detector Temp: | 320 °C |
| | Carrier: | Helium |
| | Split ratio: | 20/1 |
| | Relative mass fraction of the main component: | |
| | Initial analysis: | Mean = 99.6%, s = 0.05% (10 sub samples in duplicate, July 2013) |
| GC-FID: | Instrument: | Varian CP-3800 or Agilent 7890 |
| | Column: | HP-5, 30 m × 0.32 mm I.D. × 0.25 μm |
| | Program: | 100 °C (2 min), 10 °C/min to 260 °C (1 min), 30 °C/min to 300 °C (3 min) |
| | Injector: | 250 °C |
| | Detector Temp: | 320 °C |
| | Carrier: | Helium |
| | Split ratio: | 20/1 |
| | Relative mass fraction of the main component: | |
| | Initial analysis: | Mean = 99.7%, s = 0.05% (10 sub samples in duplicate, July 2013) |
| | Re-analysis: | Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, May 2014) |
| | Re-analysis: | Mean = 99.5%, s = 0.04% (5 sub samples in duplicate, June 2015) |
| | Re-analysis: | Mean = 99.5%, s = 0.005% (5 sub samples in duplicate, April 2016) |
| | Re-analysis: | Mean = 99.4%, s = 0.11% (5 sub samples in duplicate, April 2019) |
| Karl Fischer analysis: | | Moisture content 0.2% mass fraction (July 2013)<br>Moisture content 0.2% mass fraction (June 2014)<br>Moisture content 0.3% mass fraction (April 2015)<br>Moisture content 0.4% mass fraction (May 2016)<br>Moisture content 0.3% mass fraction (March 2019) |
| Thermogravimetric analysis: | | Non volatile residue < 0.2% mass fraction (July 2013). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures. |

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| GC-MS: | Parent compound: | |
| | Instrument: | Agilent 6890/5973 |
| | Column: | TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm |
| | Program: | 100 °C (2 min), 10 °C/min to 260 °C (1 min), 30 °C/min to 300 °C (3 min) |
| | Injector: | 250 °C |
| | Split ratio: | 20/1 |
| | Transfer line temp: | 300 °C |
| | Carrier: | Helium, 1.0 mL/min |
| | Scan range: | 50-550 *m/z* |
| | 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 (13.6 min): | 218 (17), 160 (10), 145 (6), 130 (4), 117 (7), 58 (100) *m/z* |
| ESI-MS: | Instrument: | Waters Acquity UPLC/TQD |
| | Operation: | Positive ion mode, direct infusion at 5 μL/min |
| | Ionisation: | ESI spray voltage at 3.0 kV positive ion |
| | Cone voltage: | 30 V |
| | Peak: | 219.2 ($M+H^+$) *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, mixed alkanes |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. $NH_3$/methanol (3/200)<br>Single spot observed, $R_f$ = 0.36 Visualisation with UV light (254 nm) |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 $cm^{-1}$, KBr powder |
| | Peaks: | 3129, 3036, 2940, 2859, 2781, 1626, 1588, 1465, 1442, 1218, 1065, 1050, 1037, 923, 828, 791, 704, 640, 424 $cm^{-1}$ |
| $^1$H NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 400 MHz |
| | Solvent: | $CDCl_3$ (7.26 ppm) |
| | Spectral data: | δ 2.36 (6H, s), 2.65 (2H, t, *J* = 8.4 Hz), 2.93 (2H, t, *J* = 8.4 Hz), 3.87 (3H, s), 6.85 (1H dd, *J* = 2.5, 8.8 Hz), 6.97 (1H, d, *J* = 2.4 Hz), 7.05 (1H, d, *J* = 2.8 Hz), 7.21 (1H, d, *J* = 9.0 Hz), 8.24 (1H, bs) ppm |
| | Ethyl acetate and hexane estimated at 0.2% and 0.1% mass fraction respectively were observed in the $^1$H NMR. | |
| $^{13}$C NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 101 MHz |
| | Solvent: | $CDCl_3$ (77.2 ppm) |
| | Spectral data: | δ 23.9, 45.6, 56.1, 60.4, 100.8, 112.0, 112.1, 114.1, 122.5, 127.9, 131.6, 153.9 ppm |
| Melting point: | | 65-67 °C |
| Microanalysis: | Found: | C = 71.5%; H = 8.5%; N = 12.9% (July 2013) |
| | Calculated: | C = 71.5%; H = 8.3%; N = 12.8% (Calculated for $C_{13}H_{18}N_2O$) |

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