Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

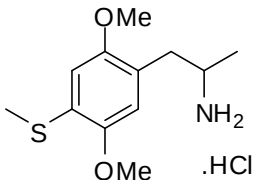
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D1015: 2,5-Dimethoxy-4-methylthioamphetamine hydrochloride

Report ID: D1015.2020.03

Chemical Formula: $C_{12}H_{19}NO_2S.HCl$

Molecular Weight: 277.8 g/mol (HCl), 214.4 g/mol (base)




## Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|---|---|---|
| **14-D-10** | **61638-08-2 (HCl)**<br>**61638-07-1 (base)** | **99.5 ± 0.7%** |

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

**IUPAC name:** 1-[2,5-Dimethoxy-4-(methylsulfanyl)phenyl]-2-propanamine hydrochloride

**Expiration of certification:** The property values are valid till 1 July 2025, 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:** Off-white powder prepared by synthesis, 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%. 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).

**Stability:** In the absence of long term 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 ten years. 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 nine 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.

**Legal notice:** Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

## 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 or HP-1, 30 m × 0.32 mm I.D. × 0.25 μm |
| | Program: | 60 °C (1 min), 10 °C/min to 100 °C, 15 °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 componentas the free base: | |
| | Initial analysis: | Mean = 99.8%, s = 0.03% (10 sub samples in duplicate, June 2014) |
| | Re-analysis: | Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, June 2015) |
| | Re-analysis: | Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, May 2016) |
| | Re-analysis: | Mean = 99.8%, s = 0.05% (5 sub samples in duplicate, July 2020) |
| Karl Fischer analysis: | | Moisture content 0.2% mass fraction (June 2014)<br>Moisture content 0.3% mass fraction (June 2015)<br>Moisture content 0.2% mass fraction (May 2016)<br>Moisture content < 0.2% mass fraction (July 2020) |
| Thermogravimetric analysis: | | Non volatile residue < 0.2% mass fraction (June 2015). 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: | Free base: | |
| | Instrument: | Agilent 6890/5973 |
| | Column: | TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm |
| | Program: | 60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (2 min) |
| | Injector: | 250 °C |
| | Split ratio: | 20/1 |
| | Transfer line temp: | 300 °C |
| | Carrier: | Helium, 1 mL/min |
| | Scan range: | 50-550 *m/z* |
| | The retention time of the free base 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. | |
| | Free base (13.7 min): | 241 ($M^+$, 3), 198 (100), 183 (25), 167 (8), 152 (6), 137 (3), 109 (4) 44 (88) *m/z* |
| 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: | 6 V |
| | Peak: | 242.2 ($M^{Cl35}+H^+$) *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: | Ethanol, diethyl ether, acetone |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. ammonia/methanol (1.5/100)<br>Single spot observed, $R_f$ = 0.45. Visualisation with UV at 254 nm and vanillin reagent |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 $cm^{-1}$, KBr powder |
| | Peaks: | 2995, 2908, 2743, 2572, 2502, 2033, 1592, 1497, 1393, 1211, 1037, 861, 832, 804, 741, 634 $cm^{-1}$ |
| $^1$H NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 400 MHz |
| | Solvent: | $D_2O$ (4.79 ppm) |
| | Spectral data: | δ 1.23 (3H, d, *J* = 6.6 Hz), 2.40 (3H, s), 2.83 (2H, d, *J* = 6.8 Hz), 3.57 (1H, dt, *J* = 6.7, 6.7 Hz), 3.78 (6H, s), 6.81 (1H, s), 6.82 (1H, s) ppm |
| | Ethanol estimated at 0.3% mass fraction was observed in the $^1$H NMR | |
| $^1$H NMR: | Instrument: | Bruker Ascend-500 |
| | Field strength: | 400 MHz |
| | Solvent: | MeOH-$d_4$ (3.31 ppm) |
| | Spectral data: | δ 1.27 (3H, d, *J* = 6.7 Hz), 2.42 (3H, s), 2.84 (1H, dd, *J* = 7.1, 13.5 Hz), 2.94 (1H, dd, *J* = 6.6, 13.5 Hz), 3.55 (1H, ddq, *J* = 6.6, 6.7, 7.1 Hz), 3.82 (3H, s), 3.85 (3H, s), 6.80 (1H, s), 6.83 (1H, s) ppm |
| $^{13}$C NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 100 MHz |
| | Solvent: | $D_2O$ |
| | Spectral data: | δ 15.3, 18.9, 36.0, 49.6, 57.4, 57.8, 111.3, 116.0, 123.3, 127.0, 151.1, 153.5 ppm |
| Melting point: | | 205-206 °C |
| Microanalysis: | Found: | C = 52.0%; H = 7.3%; N = 5.0%; S = 11.4%; Cl = 12.7% (May, 2014) |
| | Calculated: | C = 51.9%; H = 7.3%; N = 5.0%; S = 11.5%; Cl = 12.8%<br>(Calculated for $C_{12}H_{19}NO_2S.HCl$) |