Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

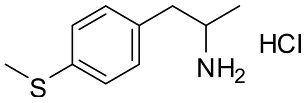
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D687b: (±)-4-Methylthioamphetamine hydrochloride

Report ID: D687b.2025.01 (Bottled 120515)

Chemical Formula: $C_{10}H_{16}ClNS$

Molecular Weight: 217.8 g/mol (HCl), 181.1 g/mol (base)



## Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|---|---|---|
| **11-D-17** | **94784-92-6(HCl)**<br>**14116-06-4 (base)** | **98.2 ± 1.4%** |

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

**IUPAC name:** 1-[4-(Methylsulfanyl)phenyl]-2-propanaminium chloride

**Expiration of certification:** The property values are valid till 23 October 2035, ten 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, certified for identity and purity by NMI Australia. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

**Intended use:** This certified reference material may be used for instrument calibration.

**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:** This material has demonstrated stability over a minimum period of ten years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

The long-term stability 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 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.
29 October 2025

This report supersedes any issued prior to 29 October 2025.

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.

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

$I_{ORG}$ = 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: Agilent 7890A/8890
Column: HP-1ms, 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: 180 °C
Detector Temp: 320 °C
Carrier: Helium
Split ratio: 20/1

Relative mass fraction of the main component:
Initial analysis: Mean = 98.2%, s = 0.3% (10 sub samples in duplicate, February 2012)
Re-analysis: Mean = 98.3%, s = 0.1% (5 sub samples in duplicate, January 2013)
Re-analysis: Mean = 98.2%, s = 0.5% (7 sub samples in duplicate, December 2015)
Re-analysis: Mean = 98.3%, s = 0.1% (5 sub samples in duplicate, October 2018)
Re-analysis: Mean = 98.6%, s = 0.1% (5 sub samples in duplicate, August 2021)
Re-analysis: Mean = 98.2%, s = 0.2% (5 sub samples in duplicate, October 2025)

GC-FID:
Instrument: Varian CP-3800 or Agilent 6890N
Column: 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: 180 °C
Detector Temp: 320 °C
Carrier: Helium
Split ratio: 20/1

Relative mass fraction of the main component:
Initial analysis: Mean = 98.3%, s = 0.2% (10 sub samples in duplicate, February 2012)

Karl Fischer analysis: Moisture content < 0.2% mass fraction (August 2021 and October 2025)

Thermogravimetric analysis: The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.
The non-volatile residue was < 0.2% mass fraction (February 2012)

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| ESI-MS: | Instrument: | Waters Acquity UPLC/TQD |
| | Operation: | Positive ion mode, direct infusion at 20 μL/min |
| | Ionisation: | ESI spray voltage at 3.0 kV positive ion |
| | Capillary voltage: | 3.0 kV |
| | Cone voltage: | 20 V |
| | Peak: | 182.10 ($M^+H^+$) *m/z* |
| GC-MS: | 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 300 °C (3 min) |
| | Injector: | 250 °C |
| | Transfer line temp: | 280 °C |
| | Carrier: | Helium, 1.0 mL/min |
| | Split ratio: | 20/1 |
| | The retention time of the free base 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. | |
| | (11.2 min): | 181 ($M^+$, 2), 164 (3), 138 (100), 122 (28), 91 (20), 78 (13) *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: | None detected |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. Methanol/ammonia (100/0.5)<br>Single spot observed, Rf = 0.3. Visualisation with UV at 254 nm vanillin stain. |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 $cm^{-1}$, KBr powder |
| | Peaks: | 3199, 2914, 2807, 2707, 2608, 2521, 2504, 2060, 1605, 1509, 1494, 1388, 1212, 1083, 795, 521, 460 $cm^{-1}$ |
| $^1H$ NMR: | Instrument: | Bruker AvanceIII-400 |
| | Field strength: | 400 MHz |
| | Solvent: | $D_2O$ (4.79 ppm) |
| | Spectral data: | δ 1.28 (3H, d, *J* = 6.6 Hz), 2.48 (3H, s), 2.89 (2H, d, *J* = 7.2 Hz), 3.59 (1H, m), 7.24 (2H, d, *J* = 8.4 Hz), 7.32 (2H, d, *J* = 8.4 Hz) ppm |
| $^{13}C$ NMR: | Instrument: | Bruker DMX-500 |
| | Field strength: | 126 MHz |
| | Solvent: | MeOH-$d_4$ (49.0 ppm) |
| | Spectral data: | δ 15.7, 18.3, 41.2, 50.2, 128.0, 130.9, 134.0, 139.2 ppm |
| Melting point: | | 183-187 °C |
| Microanalysis: | Found: | C = 55.2%; H = 7.5%; N = 6.4%; Cl = 16.2%; S = 14.6% (February, 2012) |
| | Calculated: | C = 55.2%; H = 7.4%; N = 6.4%; Cl = 16.3%; S = 14.7% (Calculated for $C_{10}H_{16}ClNS$) |