



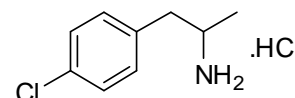
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1043: (\pm)-4-Chloroamphetamine hydrochloride

Report ID: D1043.2021.03

Chemical Formula: C₉H₁₂ClN.HCl

Molecular Weight: 206.1 g/mol (HCl), 169.7 g/mol (base)



Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|-----------|-----------------------------------|------------------------|
| 14-D-43 | 3706-38-5 (HCl) 64-12-0 (base) | 99.8 \pm 0.3% |

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

IUPAC name: 1-(4-Chlorophenyl)-2-propanamine hydrochloride.

Expiration of certification: The property values are valid till 16 April 2026, 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: 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 °C 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 5 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 seven 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 from a combination of traditional analytical techniques including GC-FID detection, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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

Supporting evidence is provided by quantitative NMR and elemental microanalysis. The certified purity value by qNMR was obtained using the two-proton multiplet at 2.88 ppm measured against a certified internal standard of maleic acid.

GC-FID: Instrument: Varian CP-3800 or Agilent 7890
 Column: HP-1 or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 80 °C (1 min), 10 °C/min to 180 °C, 30 °C/min to 300 °C (3 min)
 Injector: 200 °C Detector Temp: 320 °C
 Carrier: Helium Split ratio: 20/1

Relative mass fraction of the main component as the free base:
 Initial analysis: Mean = 99.9%, s = 0.01% (7 sub samples in duplicate, November 2014)
 Re-analysis: Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, May 2018)
 Re-analysis: Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, April 2021)

Karl Fischer analysis: Moisture content ≤ 0.2% mass fraction (November 2014, June 2018 and March 2021)

qNMR: Instrument: Bruker Avance-III-500
 Field strength: 500 MHz
 Solvent: D₂O (4.79 ppm)
 Internal standard: Maleic acid (98.7% mass fraction)
 Initial analysis: Mean (2.9 ppm) = 99.9%, s = 0.1% (3 sub samples, November 2014)

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: 60 °C (1 min), 10 °C/min to 300 °C (3 min) Injector: 250 °C Carrier: Helium, 1.0 mL/min Scan range: 50-550 <i>m/z</i> Transfer line temp: 280 °C Split ratio: 20/1 |
| | 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 (9.9 min): 168 (M+-H, 1), 156 (1), 154 (3), 127 (4), 125 (13), 117 (3), 115 (3), 91 (7), 89 (12), 75 (3), 63 (7), 44 (100) <i>m/z</i> |
| 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: 10 V Peak: 172.3 (M ^{Cl³⁷+H⁺}), 170.3 (M ^{Cl³⁵+H⁺}) <i>m/z</i> |
| IR: | Instrument: Bruker Alpha Platinum ATR Range: 4000-400 cm ⁻¹ , neat Peaks: 2987, 2899, 2808, 2741, 2708, 2604, 2519, 2502, 2065, 1506, 1491, 1464, 1387, 1215, 1105, 1086, 1014, 1001, 850, 797, 668, 520, 465, 409 cm ⁻¹ |
| ¹ H NMR: | Instrument: Bruker Avance III-500 Field strength: 500 MHz Solvent: DMSO- <i>d</i> ₆ (2.50 ppm) Spectral data: δ 1.11 (3H, d, <i>J</i> = 6.5 Hz), 2.69 (1H, dd, <i>J</i> = 8.8, 13.4 Hz), 3.03 (1H, dd, <i>J</i> = 5.4, 13.4 Hz), 3.39 (1H, m), 7.28 (2H, d, <i>J</i> = 8.4 Hz), 7.40 (2H, d, <i>J</i> = 8.4 Hz), 8.2 (3H, s) ppm Isopropanol estimated at 0.2% mass fraction was observed in the ¹ H NMR |
| ¹³ C NMR: | Instrument: Bruker Avance III-500 Field strength: 125 MHz Solvent: DMSO- <i>d</i> ₆ (39.5 ppm) Spectral data: δ 17.6, 39.2, 47.8, 128.5, 131.2, 131.5, 135.9 ppm |
| Melting point: | 161-163 °C |
| Microanalysis: | Found: C = 52.6%; H = 6.3%; N = 6.8%; Cl% = 34.7% (November, 2014) Calculated: C = 52.5%; H = 6.4%; N = 6.8%; Cl% = 34.4% (Calculated for C ₉ H ₁₂ ClN.HCl) |