



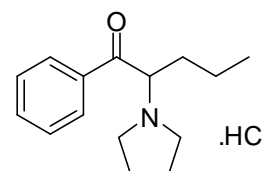
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D986b: ( $\pm$ )- $\alpha$ -Pyrrolidinopentiophenone hydrochloride

Report ID: D986b.2023.01

Chemical Formula: C<sub>15</sub>H<sub>21</sub>NO.HCl

Molecular Weight: 267.8 g/mol (HCl), 231.3 g/mol (base)



### Property value

Batch No.	CAS No.	Purity estimate
13-D-24	5485-65-4	79.7% $\pm$ 2.4%

**IUPAC name:** 1-Phenyl-2-(1-pyrrolidinyl)-1-pentanone hydrochloride (1:1).

The material exists predominately as the hydrochloride salt mixed with the hydrogen bromide salt. The stated purity is the mass fraction of the free base in the sample.

**Expiration of certification:** The property values are valid till 13 September 2028, 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 solid 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 reference material is recommended for qualitative analysis only.

**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.

**Stability:** In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years. This material has demonstrated stability over a minimum period of three 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 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 2023

This report supersedes any issued prior to 21 September 2023.

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.

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## Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by quantitative NMR analysis using a certified internal standard of potassium hydrogen maleate.

Supporting evidence is provided by GC-FID, thermogravimetric analysis, Karl Fischer analysis, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

**Warning:** This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 °C) into a GC instrument.

GC-FID: Instrument: Agilent 6890 or Varian CP-3800  
 Agilent 8890 (2021)  
 Column: HP-1 or VF-1MS or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm  
 Program: 100 °C (1 min), 10 °C/min to 180 °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 peak area of main component as the free base:

Initial analysis: Mean = 99.4%, s = 0.2% (10 sub samples in duplicate, November 2013)  
 Re-analysis: Mean = 99.3%, s = 0.08% (7 sub samples in duplicate, October 2014)  
 Re-analysis: Mean = 99.5%, s = 0.06% (7 sub samples in duplicate, September 2015)  
 Re-analysis: Mean = 99.5%, s = 0.07% (5 sub samples in duplicate, October 2018)  
 Re-analysis: Mean = 99.5%, s = 0.06% (5 sub samples in duplicate, October 2021)  
 Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, September 2023)

Karl Fischer analysis: Moisture content 4.3% mass fraction (December 2013)  
 Moisture content 5.0% mass fraction (October 2014)  
 Moisture content 4.6% mass fraction (September 2015)  
 Moisture content 4.2% mass fraction (July 2018)  
 Moisture content 4.8% mass fraction (August 2021)  
 Moisture content 5.0% mass fraction (August 2023)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (November 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.

QNMR: Instrument: Bruker Avance-III-400  
 Field strength: 600 MHz  
 Solvent: D<sub>2</sub>O (4.79 ppm)  
 Internal standard: Potassium hydrogen maleate (100% mass fraction)  
 Initial analysis: Mean (7.2-8.1 ppm) = 80.3%, s = 0.03% (4 sub samples, May 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  $\mu$ m  
 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: 280 °C  
 Carrier: Helium, 1.0 mL/min  
 Scan range: 50-550 *m/z*

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.

Free base (12.8 min): 126 (100), 105 (7), 77 (9), 55 (4) *m/z*

ESI-MS: Instrument: Micromass Quatro LC Micro  
 Operation: Positive ion mode, direct infusion at 10  $\mu$ L/min  
 Ionisation: ESI spray voltage at 3.5 kV positive ion  
 EM voltage: 650 V  
 Cone voltage: 20 V  
 Peak: 232.2 (M+H<sup>+</sup>) *m/z*

HS-GC-MS: Instrument: Agilent 6890/5973/G1888  
 Column: DB-624, 30 m x 0.25 mm I.D. x 1.4  $\mu$ m  
 Program: 50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)  
 Injector: 150 °C  
 Split ratio: 50/1  
 Transfer line temp: 280 °C  
 Carrier: Helium, 1.2 mL/min  
 Solvents detected: Diethyl ether, dichloromethane, butanal

TLC: Conditions: Kieselgel 60F<sub>254</sub>. Dichloromethane/methanol/diethylamine (95:5:0.1)  
 Single spot observed, R<sub>f</sub> = 0.4. Visualisation with UV at 254 nm

IR: Instrument: Biorad FTS3000MX FT-IR  
 Range: 4000-400  $\text{cm}^{-1}$ , KBr powder  
 Peaks: 3504, 3454, 3402, 3245, 2963, 2932, 2878, 2795, 2618, 2472, 2094, 1682, 1595, 1451, 1376, 1338, 1235, 1136, 1105, 1038, 1005, 771, 720, 694  $\text{cm}^{-1}$

<sup>1</sup>H NMR: Instrument: Bruker Avance III 600  
 Field strength: 600 MHz  
 Solvent: D<sub>2</sub>O + DCI (4.79 ppm)  
 Spectral data:  $\delta$  0.42 (3H, t, *J* = 7.3 Hz), 0.75 (1H, m), 0.86 (1H, m), 1.56-1.80 (5H, m), 1.85 (1H, m), 2.70 (1H, m), 2.98 (1H, m), 3.32 (1H, m), 3.39 (1H, m), 4.95 (1H, dd, *J* = 4.6, 5.9 Hz), 7.25 (2H, dd, *J* = 7.6, 8.2 Hz), 7.41 (1H, t, *J* = 7.5 Hz), 7.67 (2H, dd, *J* = 1.0, 8.5 Hz) ppm  
 Diethyl ether estimated at 0.1% mass fraction was observed in the <sup>1</sup>H NMR

<sup>13</sup>C NMR: Instrument: Bruker Ascend 500  
 Field strength: 126 MHz  
 Solvent: D<sub>2</sub>O + DCI  
 Spectral data:  $\delta$  12.9, 16.7, 22.6, 22.7, 31.7, 51.9, 55.1, 69.2, 128.8, 129.2, 13.3, 135.5, 197.6 ppm

<sup>13</sup>C NMR: Instrument: Bruker Avance-400  
 Field strength: 101 MHz  
 Solvent: d<sub>6</sub>-DMSO (39.5 ppm)  
 Spectral data:  $\delta$  13.6, 17.4, 22.8, 31.6, 51.8, 53.5, 67.2, 128.7, 129.1, 134.5, 134.8, 196.6 ppm

Melting point: 174-176 °C

Microanalysis: Found: C = 62.9%; H = 8.1%; N = 4.8%, Cl = 12.3%, Br = 4.7% (December 2013)  
 Calculated: C = 62.7%; H = 8.2%; N = 4.9%, Cl = 10.3%, Br = 4.6% (Calculated for [C<sub>15</sub>H<sub>23</sub>NO.HCl]<sub>5</sub> + [C<sub>15</sub>H<sub>23</sub>NO.HBr] + [H<sub>2</sub>O]<sub>4</sub>)