Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

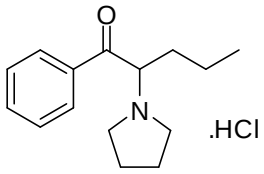
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D986b: (±)-α-Pyrrolidinopentiophenone hydrochloride

Report ID: D986b.2021.02

Chemical Formula: $C_{15}H_{21}NO.HCl$

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% ± 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 19 August 2024, i.e. three 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 ºC 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.
20 September 2022

This report supersedes any issued prior to 20 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. 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 ºC) 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)
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)
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_2O (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 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:	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 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	232.2 ($M+H^+$) *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
	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_{254}. Dichloromethane/methanol/diethylamine (95:5:0.1) Single spot observed, R_f = 0.35. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 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 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III 600
	Field strength:	600 MHz
	Solvent:	D_2O + DCl (4.79 ppm)
	Spectral data:	δ 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 ^{1}H NMR	
^{13}C NMR:	Instrument:	Bruker Ascend 500
	Field strength:	126 MHz
	Solvent:	D_2O + DCl
	Spectral data:	δ 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
^{13}C NMR:	Instrument:	Bruker Avance-400
	Field strength:	101 MHz
	Solvent:	d_6-DMSO (39.5 ppm)
	Spectral data:	δ 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 $[C_{15}H_{23}NO.HCl]_5$ + $[C_{15}H_{23}NO.HBr]$ + $[H_2O]_4$)