Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

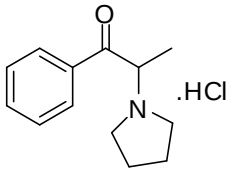
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D956: (±)-α-Pyrrolidinopropiophenone hydrochloride

Report ID: D956.2024.01

Chemical Formula: $C_{13}H_{17}NO.HCl$

Molecular Weight: 239.7 g/mol (HCl) 203.2 (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-09	**92040-10-3 (HCl)** **19134-50-0 (base)**	**99.6 ± 0.5%**

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

IUPAC name: 1-Phenyl-2-(1-pyrrolidinyl)-1-propanone hydrochloride

Expiration of certification: The property values are valid till 13 March 2029, 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, 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.

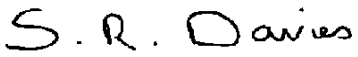
Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct 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 five years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials.

The material has been shown to decompose in solution and absorb water, up to 4% mass fraction, when exposed to the atmosphere.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on five 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.



Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
19 April 2024

This report supersedes any issued prior 19 April 2024.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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, HPLC with UV detection, qNMR and elemental microanalysis.

GC-FID:	Instrument:	Agilent 7890A or 8890
	Column:	HP-1 or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 200 °C, 30 °C/min to 300 °C (3 min)
	Injector:	250 °C or 200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, July 2012)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, June 2015)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, June 2018)
	Re-analysis:	Mean = 99.8%, s = 0.01% (7 sub samples in duplicate, May 2023)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, March 2024)
HPLC:	Instrument:	Shimadzu Model LC-20AB Binary pump, SIL-20A HT autosampler
	Column:	X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Acetonitrile/Milli Q water NH_4OAc buffer (60:40) The aqueous phase was buffered at pH 10.8 with 20mM NH_4OAc
	Flow rate:	1.0 mL/min
	Detector:	Shimadzu PDASPD-M20A operating at 244 nm
	Relative peak area of main component:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, August 2010)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, August 2011)
Thermogravimetric analysis:		Non volatile residue < 0.2 % mass fraction (November 2010). The volatile content (e.g., organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material.
Karl Fischer analysis:		Moisture content > 0.1% mass fraction (September 2010) Moisture content 0.3% mass fraction (July 2011 and 2012) Moisture content 0.2% mass fraction (May 2023) Moisture content 0.3% mass fraction (March 2024)
QNMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Potassium hydrogen maleate (98.8% mass fraction)
	Initial analysis:	Mean = 99.6%, s = 0.53% (5 sub samples, August 2010)

Spectroscopic and other characterisation data

LC/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:	204.1 $(M+H^+)$ *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
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Diethyl ether
TLC:	Conditions:	Kieselgel $60F_{254}$. Methanol/NH_3 (100/1.5) Single spot observed, R_f = 0.6. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3363, 3064, 3036, 2979, 2954, 2802, 2632, 2577, 2457, 1691, 1596, 1451, 1391, 1340, 1294, 1240, 1134, 977, 792, 698, 598, 458 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.66 (3H, d, *J* = 7.1 Hz), 1.90-2.40 (4H, bm), 3.13 (1H, bm), 3.42 (1H, bm), 3.73 (2H, bm), 5.29 (1H, q, *J* = 7.1 Hz), 7.65 (1H, t, *J* = 8.0 Hz), 7.81 (1H, t, *J* = 7.4 Hz), 8.05 (1H, d, *J* = 7.4 Hz) ppm
		Diethyl ether estimated at 0.02% mass fraction was observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	150 MHz
	Solvent:	D_2O
	Spectral data:	δ 15.4, 22.5, 51.7 (b), 53.9 (b), 65.0, 128.5, 128.9, 131.9, 135.2, 197.2 ppm
Melting point:		202-206 °C (decomposition)
Microanalysis:	Found:	C = 65.1%; H = 7.9%; N = 5.9%; Cl = 14.7% (August 2010)
	Calculated:	C = 65.1%; H = 7.6%; N = 5.8%; Cl = 14.8% (Calculated for $C_{13}H_{17}NO.HCl$)