



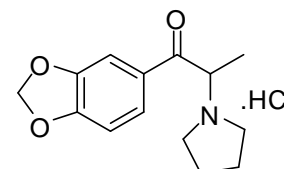
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D960: (\pm)-3, 4-Methylenedioxy- α -pyrrolidinopropiophenone hydrochloride

Report ID: D960.2017.04

Chemical Formula: C₁₄H₁₈ClNO₃

Molecular Weight: 283.8 g/mol (HCl), 247.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-013	24698-57-5 (HCl) 783241-66-7 (base)	98.7 \pm 3.1%

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

IUPAC name: 1-(1,3-Benzodioxol-5-yl)-2-(1-pyrrolidinyl)-1-propanone hydrochloride

Expiration of certification: The property values are valid till 1 August 2020, 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. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: White crystalline solid 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 °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: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten 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 HPLC with UV detection 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.

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 and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, HPLC with UV detection, thermogravimetric analysis, 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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument: Column: Program: Injector: Detector Temp: Carrier: Split ratio: Relative mass fraction of the main component: Initial analysis: Re-analysis: Re-analysis:	Agilent 6890N HP-1, 30 m × 0.32 mm I.D. × 0.25 μm 120 °C (1 min), 15 °C/min to 300 °C (3 min) 200 °C 320 °C Helium 20/1 Mean = 99.6%, s = 0.07% (5 sub samples in duplicate, October 2013) Mean = 99.2%, s = 0.06% (5 sub samples in duplicate, September 2016) Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, August 2017)
HPLC:	Instrument: Column: Column oven: Mobile Phase: Flow rate: Detector: Relative mass fraction of the main component: Initial analysis: Re-analysis: Re-analysis:	Shimadzu Model LC-20AB Binary pump, SIL-20A HT autosampler Or Waters Model 1525 Binary pump, 717 plus autosampler X-Bridge C-18, 5 μm (4.6 mm x 150 mm) Ambient or 40°C Acetonitrile/MilliQ water (35:65 v/v) The aqueous phase was buffered at pH 10.8 using 20 mM NH ₄ OAc and NH ₃ 1.0 mL/min Shimadzu SPD-M20A PDA or Waters PDA 2998 operating at 227 nm Mean = 100.0%, s = 0.0% (10 sub samples in duplicate, November 2010) Mean = 100.0%, s = 0.0% (5 sub samples in duplicate, October 2011) Mean = 100.0%, s = 0.0% (5 sub samples in duplicate, November 2012)
Karl Fischer analysis:		Moisture content 0.4% mass fraction (October 2010) Moisture content 0.7% mass fraction (November 2011) Moisture content 2.1% mass fraction (November 2012) Moisture content 1.1% mass fraction (October 2013) Moisture content 0.3% mass fraction (August 2016) Moisture content 0.2% mass fraction (August 2017)
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.
qNMR:	Instrument: Field strength: Solvent: Internal standard: Initial analysis:	Bruker Avance III-600 600 MHz D ₂ O (4.79 ppm) Potassium hydrogen maleate (98.8% mass fraction) Mean = 99.0%, s = 0.58% (5 sub samples, October 2010)

Spectroscopic and other characterisation data

LC/ESI -MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	21 V
	Peak:	248.3 (M+H ⁺) <i>m/z</i>
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μ m
	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:	Acetone and ethanol
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/NH ₃ (100/1.5) Single spot observed, R _f = 0.60. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr powder
	Peaks:	3342, 2982, 2956, 2899, 2802, 2637, 2588, 2509, 2459, 1849, 1680, 1603, 1509, 1489, 1449, 1255, 1107, 1036, 927, 882, 744, 574 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance-600
	Field strength:	600 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Spectral data:	δ 1.64 (3H, d, <i>J</i> = 7.1 Hz), 2.10 (2H, m), 2.15 (2H, brs), 3.52 (4H, brs), 5.16 (1H, q, <i>J</i> = 7.1 Hz), 6.12 (2H, d, <i>J</i> = 2.9 Hz), 7.01 (1H, d, <i>J</i> = 8.2 Hz), 7.42 (1H, d, <i>J</i> = 1.6 Hz), 7.67 (1H, dd, <i>J</i> = 1.7, 8.3 Hz) ppm Ethanol estimated at ~ 0.3% mass fraction was observed in the ¹ H NMR. Acetone was not detected.
¹³ C NMR:	Instrument:	Bruker Avance-600
	Field strength:	150 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 16.3, 22.9, 53.3, 65.3, 102.7, 107.9, 108.6, 126.7, 126.9, 148.4, 153.7, 195.4 ppm
Melting point:		230-235 °C (Stage microscope)
Microanalysis:	Found:	C = 59.3%; H = 6.5%; N = 4.7%; Cl = 12.5% (October, 2010)
	Calc:	C = 59.3%; H = 6.4%; N = 4.9%; Cl = 12.5% (Calculated for C ₁₄ H ₁₈ ClNO ₃)