



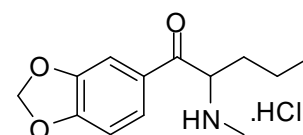
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D992: Pentylone hydrochloride

Report ID: D992.2019.03

Chemical Formula: C₁₃H₁₇NO₃.HCl

Molecular Weight: 271.7 g/mol (HCl), 235.3 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
13-D-02	17763-01-8	99.8 ± 0.5%

IUPAC name: 1-(1,3-Benzodioxol-5-yl)-2-(methylamino)-1-pentanone hydrochloride (1:1)

Expiration of certification: The property values are valid till 4 December 2024, 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 crystalline 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 should be used 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: 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 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.
19 October 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 by mass balance from a combination of traditional analytical techniques, including GC-FID, 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 elemental microanalysis.

GC-FID: Instrument: Agilent 6890 or Varian CP-3800
 Column: HP-1 or VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 130 °C (1 min), 10 °C/min to 190 °C (6 min), 30 °C/min to 300 °C (3 min)
 Injector: 250 °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 = 99.8%, s = 0.02% (10 sub samples in duplicate, March 2013)
 Re-analysis: Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, February 2014)
 Re-analysis: Mean = 99.8%, s = 0.18% (5 sub samples in duplicate, February 2015)
 Re-analysis: Mean = 99.8%, s = 0.03% (5 sub samples in duplicate, February 2016)
 Re-analysis: Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, December 2019)

GC-FID: Instrument: Varian CP-3800
 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 130 °C (1 min), 10 °C/min to 190 °C (6 min), 30 °C/min to 300 °C (3 min)
 Injector: 250 °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% (10 sub samples in duplicate, March 2013)

HPLC: Instrument: Thermo UltiMate 3000 RS pump, UltiMate 3000 RS
 Column: Atlantis C-18, 3.0 μm (4.6 mm x 150 mm)
 Column oven: 40 °C
 Mobile Phase: A: MilliQ water (0.05% formic acid), B: Acetonitrile (0.05% formic acid)
 Flow rate: 1.0 mL/min
 Gradient: 0-20 min 15%-40% B, 21-23 min 40% -15% B, 23-26 min 15% B
 Detector: Thermo UltiMate 3000 RS DAD operating at 321 nm

Relative peak area response of main component:

Initial analysis: Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, March 2013)

Karl Fischer analysis: Moisture content < 0.1% mass fraction (February 2013, 2014 and December 2019)
 Moisture content 0.1% mass fraction (February 2015 and 2016)

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

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	130 °C (1 min), 10 °C/min to 190 °C (6 min), 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 <i>m/z</i>
	The retention time of the parent compound 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):	149 (10), 121 (6), 86 (100), 44 (14) <i>m/z</i>
ESI-MS:	Instrument:	Waters Acquity UPLC TQD
	Operation:	Positive ion mode, direct infusion at 5 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	30 V
	Peak:	236.3 (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:	Ethanol, diethyl ether, butanal
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . NH ₃ /methanol (3/200) Single spot observed, R _f = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr powder
	Peaks:	3035, 2965, 2749, 2413, 1677, 1604, 1554, 1475, 1431, 1260, 1105, 1034, 929, 864, 826, 807, 781, 729, 714, 507 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Spectral data:	δ 0.83 (3H, t, <i>J</i> = 7.3 Hz), 1.21-1.29 (2H, m), 1.96 (2H, m), 2.73 (3H, s), 5.02 (1H, t, <i>J</i> = 5.5 Hz), 6.08 (1H, d, <i>J</i> = 1.0 Hz), 6.09 (1H, d, <i>J</i> = 1.0 Hz), 6.97 (1H, d, <i>J</i> = 8.7 Hz), 7.40 (1H, d, <i>J</i> = 1.5 Hz), 7.64 (1H, dd, <i>J</i> = 1.8, 8.3 Hz) ppm Ethanol estimated at 0.04% mass fraction was observed in the ¹ H NMR. Diethyl ether and butanal were not observed in the ¹ H NMR
¹³ C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	101 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 12.9, 17.0, 31.7, 32.3, 63.3, 102.6, 107.8, 108.5, 126.5, 127.6, 148.3, 153.5, 194.8 ppm
Melting point:	237-239 °C	
Microanalysis:	Found:	C = 57.8%; H = 6.7%; N = 5.3%; Cl% = 13.0% (March, 2013)
	Calculated:	C = 57.5%; H = 6.7%; N = 5.2%; Cl% = 13.1% (Calculated for C ₁₃ H ₁₇ NO ₃ .HCl)