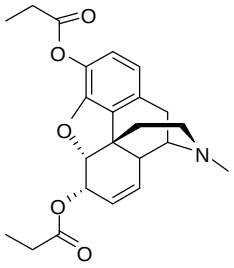
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D950: Dipropionylmorphine



Report ID: D950.2012.05

Chemical Formula: $C_{23}H_{27}NO_5$

Molecular Weight: 397.5 g/mol

Property value

Batch No.	CAS No.	Purity estimate
10-D-01	10589-79-4	99.0 ± 2.4%

IUPAC name: (5α,6α)-17-Methyl-7,8-didehydro-4,5-epoxymorphinan-3,6-diyl dipropanoate

Expiration of certification: The property values are valid till 14th April 2015, i.e. three years from the date of 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. This material has been given a shelf life of three years from the date of re-certification. 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: Off-white powder prepared by synthesis or sourced from an external supplier, 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 20 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. 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 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.

Caution: 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.
13 October 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. Impurities of related structure were assessed by GC-FID. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR spectroscopy, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 29.58 m × 0.32 mm I.D. × 0.25 μm
	Program	200 °C (1 min), 20 °C/min to 240 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	300 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.6 %, s = 0.02% (10 sub samples in duplicate, June 2011)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-5, 30.0 m × 0.32 mm I.D. × 0.25 μm
	Program	200 °C (1 min), 20 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	300 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.4%, s = 0.1% (10 sub samples in duplicate, June 2011)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, May 2012)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (June 2011) Moisture content <0.1% mass fraction (May 2012)
Thermogravimetric analysis:		The volatile content could not be determined on this material using thermogravimetric analysis. The non volatile residue < 0.2% mass fraction (June 2011).

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	150 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention time of the parent compound 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.	
	Parent (11.8 min):	397 (M^+, 46), 341 (100), 324 (42), 268 (54), 218 (20), 215 (20), 57 (20) *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}. Chloroform/Methanol (10/1) Single spot observed, R_f = 0.43. Visualization with UV light (254 nm)
IR:	Instrument:	BioRad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2982, 2934, 2908, 2842, 2798, 1767, 1734, 1618, 1493, 1448, 1368, 1352, 1247, 1238, 1193, 1141, 1081, 1037, 981, 946, 891, 834, 807, 797, 784, 766, 737, 681, 571 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.19 (3H, t, *J* = 7.6 Hz), 1.24 (3H, t, *J* = 7.6 Hz), 1.88 (1H, bm), 2.0 (1H, ddd, *J* = 5, 12.5, 12.5 Hz), 2.28-2.44 (4H, m), 2.43 (3H, s), 2.55 (2H, quartet, *J* = 7.6 Hz), 2.56 (1H, bdd, *J* = 4.2, 12.4 Hz), , 2.74 (1H, broad quintet, 2.5 Hz), 3.05 (1H, d, *J* = 18.7 Hz), 3.35 (1H, dd, *J* = 3.3, 5.7 Hz), 5.12-5.17 (2H, m), 5.43 (1H, bdt, *J* = 2.3, 2.3, 10 Hz), 5.62 (1H, bdm, *J* = 10 Hz), 6.57 (1H, d, *J* = 8.2 Hz), 6.75 (1H, d, *J* = 8.2 Hz). Diethyl ether (0.3%) mass fraction was estimated by ^{1}H NMR spectrum.
^{13}C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	151 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 9.22, 9.25, 20.8, 27.46, 27.54, 35.4, 40.8, 43.0, 43.2, 46.7, 59.1, 68.1, 88.8, 119.4, 122.0, 128.7, 129.6, 131.6, 132.0, 132.3, 149.6, 172.0, 174.0 ppm
Melting point:		100-101 °C
Microanalysis:	Found:	C = 69.7 %; H = 6.8 %; N = 3.5 % (June 2011)
	Calc:	C = 69.5 %; H = 6.9 %; N = 3.5 % (Calculated for $C_{23}H_{27}NO_5$)