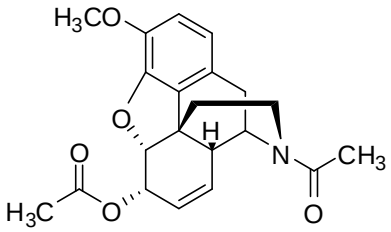
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D699: *O*^{6},*N*-Diacetylnorcodeine



Report ID: D699.2021.02

Chemical Formula: $C_{21}H_{23}NO_5$

Molecular Weight: 369.4 g/mol

Property value

Batch No.	CAS No.	Purity estimate
01-D-007	**89493-70-9**	**97.6 ± 1.8%**

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

IUPAC Name: (5α,6α)-6,17-diacetyl-7,8-didehydro-4,5-epoxy-3-methoxymorphinan

Expiration of certification: The property values are valid till 30 September 2031, i.e. ten 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 micro-crystalline 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 is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

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 4 ºC in a closed container in a dry, dark area.

Stability: 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 five randomly selected 1-2 mg sub samples of the material. The material was judged to be 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 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.
14 September 2022

This report supersedes any issued prior to 14 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 ^{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 elemental microanalysis.

GC-FID:	Instrument:	HP5890
	Column:	ZB-1 Capillary, 30 m × 0.32 mm I.D. × 0.5 μm
	Program:	220 °C (1min) 10 °C /min to 280 °C (5 min)
	Injector:	250 °C.
	Detector Temp:	340 °C
	Carrier:	Helium
	Split ratio:	15/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 98.7 %, s = 0.02% (8 samples in duplicate, August 2001)
	Re-analysis:	Mean = 98.3 %, s = 0.07% (3 samples in duplicate, September. 2003)
	Re-analysis:	Mean = 98.4 %, s = 0.03% (3 samples in duplicate, September. 2004
	Re-analysis:	Mean = 98.3 %, s = 0.04% (5 samples in duplicate, September. 2005)
	Re-analysis:	Mean = 98.1 %, s = 0.01% (5 samples in duplicate, December. 2006)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	220 °C (1 min), 10 °C/min to 280 °C (8min), 20 °C/min to 300 °C (3min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 98.0%, s = 0.04% (5 sub samples in duplicate, December 2011)
	Re-analysis:	Mean = 98.2 %, s = 0.03% (5 sub samples in duplicate, September 2021)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile content < 0.2% mass fraction (June 2001 and November 2005) Volatile content 0.6% and non-volatile residue < 0.2% mass fraction (December 2011)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (December 2011 & September 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890 / 5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.3 µm
	Program:	200 °C (1 min), 12 °C /min to 300 °C, hold 6 min.
	Injector:	250 °C
	Transfer line temp:	280 °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 (6.2 min):	369 (M^+, 53), 250 (17), 223 (100), 208 (36), 195 (23), 87 (44) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/acetone (60:40) Single spot observed, R_f = 0.45 (3 replicates)
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet.
	Peaks:	2997, 2944, 2835, 1734, 1635, 1502, 1427, 1230, 1045, 804 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.94 (2H, m), 2.12 (3H, s), 2.14 (3H, s), 2.21 (1H, br s), 2.64 (1H, d, *J* = 18.8 Hz), 2.75 (1H, d, *J* = 16.7 Hz), 2.87 (1H, dd, *J* = 6.4, 18.8 Hz), 3.25 (1H, br m), 3.68 (1H, br d, *J* = 13.1 Hz), 3.84 (3H, s), 4.45 (1H, br s), 4.57 (1H, br d, *J* = 11.1 Hz), 5.07 (1H, d, *J* = 6.6 Hz), 5.15 (OH), 5.44 (2H, m), 5.68 (1H, m), 6.54 (1H, d, *J* = 8.2 Hz), 6.69 (1H, d, *J* = 8.2 Hz) ppm
	Dichloromethane and diethyl ether were observed at 0.3 and 0.8% mass fraction respectively.	
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 20.7, 21.8, 29.0, 35.5, 39.3, 40.2, 43.0, 47.4, 53.5, 56.6, 67.6, 87.6, 114.4, 119.7, 125.5, 128.3, 129.3, 142.5, 146.8, 168.9, 170.4 ppm
Melting point:		194–195 °C
Microanalysis:	Found:	C = 68.0%; H = 6.5%; N = 3.9% (June, 2001)
	Calculated:	C = 68.3%; H = 6.3%; N = 3.8% (Calculated for $C_{21}H_{23}NO_5$)