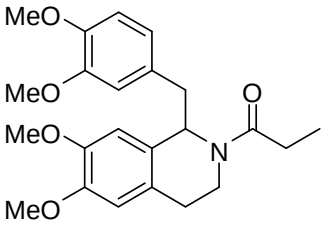
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D859: (±) N-Propionylnorlaudanosine



Report ID: D859.2010.05

Chemical Formula: $C_{23}H_{29}NO_5$

Molecular Weight: 399.5 g/mol

Certified value

Batch No.	CAS No.	Purity (mass fraction)
04-D-07	1391561-58-2 (1S isomer)	99.3 ± 1.3 %

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

IUPAC name: 1-[(1-(3,4-Dimethoxybenzyl)-6,7-dimethoxy-3,4-dihydro-2(1H)-isoquinolinyl]propanone.

Expiration of certification: The property values are valid till 18 October 2015, 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: Off-white powder 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 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 20 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 approach all impurities are quantified as a mass fraction and subtracted from 100%.

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 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 September 2022

This report supersedes any issued prior to 19 September 2022.

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 elemental microanalysis.

GC-FID:	Instrument	Varian CP3800
	Column:	HP-5, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	230 °C (1 min), 10 °C/min to 280 °C (12 min), 20 °C/min 300°C (3 min)
	Injector:	250 °C
	Detector Temp:	300 °C
	Carrier:	Helium, 2.0 mL/min
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, October 2010)
GC-FID:	Instrument	Agilent 6890N
	Column:	HP-1, 29.74 m x 0.32 mm I.D. x 0.25 μm
	Program:	230 °C (1 min), 10 °C/min to 280 °C (12 min)
	Injector:	250 °C
	Detector Temp:	300 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.5%, s = 0.01% (7 sub samples in duplicate, July 2004)
	Re-analysis:	Mean = 99.5%, s = 0.04% (5 sub samples in duplicate, August 2006)
	Re-analysis:	Mean = 99.5%, s = 0.02% (5 sub samples in duplicate, September 2007)
Karl Fischer analysis:		Moisture content 0.3 % mass fraction (September 2007) Moisture content 0.2 % mass fraction (October 2010)
Thermogravimetric analysis:		Volatile content ca. 0.1% mass fraction. Non-volatile content < 0.2% mass fraction (July 2004)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890 / 5973
	Column:	DB-5, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	230 °C, 10 °C/min to 280 °C (18 min)
	Injector:	250 °C
	Split ratio:	40/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	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.	
	Parent (15.1 min):	281 (5), 249 (14), 248 (90), 207 (10), 193 (14), 192 (100), 177 (5), 176 (12), 151 (9), 148 (6), 107 (5), 57 (9) *m/z*
ESI-MS:	Instrument:	Micromass Quattro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI capillary at 3.5 kV for positive ion mode
	EM voltage:	650 V
	Cone voltage:	25 V
	Solvent:	MeOH/H_2O at 10 ppm
	Desolvation temp:	200 °C
	Peak:	422.4 ($M+Na^+$, 22), 400.4 ($M+H^+$, 100) *m/z*
IR:	Instrument:	Biorad FTS 3000 MXFT-IR
	Range:	4000-400cm^{-1}, KBr.
	Peaks:	3241, 2994, 2934, 2834, 2597, 2045, 2016, 1627, 1515, 1458, 1254, 1237, 1160, 1117, 1028, 865, 807, 788 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.89 (3H, t, *J* = 7.1 Hz), 1.16 (3H, t, *J* = 7.3 Hz), 3.60 (3H, s), 3.80 (3H, s), 3.83 (3H, s), 3.84 (3H, s), 3.86 (3H, s), 4.84 (1H, m), 5.60 (1H, dd, *J* = 5.0 and 7.5 Hz), 6.15 (1H, s), 6.46 (1H, s), 6.53 (1H, d, *J* = 7.8 Hz), 6.58 (1H, s), 6.61 (1H, s), 6.68 (1H, d, *J* = 7.8 Hz), 6.71 (1H, d, *J* = 8.0 Hz), 6.81 (1H, d, *J* = 8.0 Hz) ppm
	^{1}H NMR in the original certification showed the presence of 0.6% mass fraction of ethyl acetate. Re-analysis of the material in August 2006 showed no detectable ethyl acetate.	
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	150 MHz
	Solvent:	$CDCl_3$ (77.0 ppm)
	Spectral data:	δ 9.5, 25.9, 26.9, 27.9, 28.5, 35.3, 41.0, 41.9, 42.6, 54.4, 55.7, 55.8, 55.8, 56.0, 58.2, 110.0, 110.8, 111.4, 111.5, 112.7, 112.8, 112.9, 121.7, 121.9, 125.6, 126.7, 127.8, 128.2, 130.2, 130.6, 146.9, 147.2, 147.6, 148.0, 148.6, 148.9, 174.0 ppm
Melting point:		135–137 °C
Microanalysis:	Found:	C = 69.2%, H = 7.3%, N = 3.4% (April 2004)
	Calculated:	C = 69.2%, H = 7.3%, N = 3.5% (Calculated for $C_{23}H_{29}NO_5$)