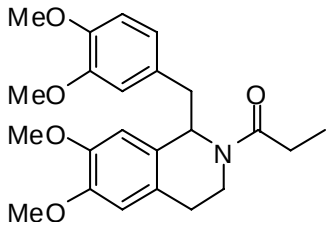
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D859.2010.02

Compound Name: (±) ***N*-Propionylnorlaudanosine**
Collection Number: D859
Chemical Formula: $C_{23}H_{29}NO_5$
CAS Registry No: Not available
Structure:

Description: Off-white powder
Batch Number: 04-D-07
Molecular Weight: 399.5
Completed on March 2004



Synonyms: 1,2,3,4-Tetrahydro-6,7-dimethoxy-2-propionyl-1-veratryl-isoquinoline; (R,S)-1-[(3,4-dimethoxyphenyl)methyl]-1,2,3,4-tetrahydro-6,7-dimethoxy-2-propionylisoquinoline

Purity (mass fraction): 99.3 ± 1.3% (95% coverage interval)

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 Temp: 250 °C — Detector Temp: 300 °C
Carrier: Helium, 2mL/min — Split ratio: 20/1
Relative peak area response of 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 Temp: 250 °C — Detector Temp: 300 °C
Carrier: Helium — Split ratio: 20/1
Relative peak area response of 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)

Thermogravimetric analysis: Volatile content ca. 0.1% mass fraction
Non volatile content < 0.2% mass fraction (July 2004)

Karl Fischer analysis: Moisture content 0.3 % mass fraction (September 2007)
Moisture content 0.2 % mass fraction (October 2010

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 film thickness	
	Program:	230 °C, 10 °C/min to 280 °C (18 min)	
	Injector Temp:	250 °C	Transfer line Temp: 300 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 40/1

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.
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
	Solvent:	MeOH/H_2O at 10 ppm
	Ionisation:	ESI capillary at 3.5 kV for positive ion mode
	Desolvation temp:	200 °C
	Multiplier:	650 V
	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: $CDCl_3$
	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: $CDCl_3$
	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) Calc: C = 69.2%, H = 7.3%, N = 3.5% (Calculated for $C_{23}H_{29}NO_5$)

Expiration of certification
The property values are valid till 18th 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.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of 3 years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

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.

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.

Intended Use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 19 July, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 19th July 2012.