

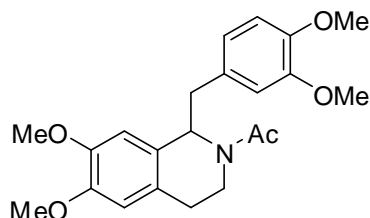


CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D697.2018.01

Compound Name: *N*-Acetylnorlaudanosine
Collection Number: D697
Chemical Formula: C₂₂H₂₇NO₅
CAS Number: 860-23-1
Structure:

Description: Off-white powder
Batch Number: 01-D-005
Molecular Weight: 385.5
Release date: 2nd August 2001



Synonyms: 1,2,3,4-tetrahydro-6,7-dimethoxy-2-acetyl-1-veratrylisoquinolin
(*R,S*)-1-[(3,4-Dimethoxyphenyl)methyl]-1,2,3,4-tetrahydro-6,7-dimethoxy-2-acetylisquinoline

Purity (mass fraction): 99.2 ± 1.0% (95% coverage interval)

The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR analysis. Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890N
Column: HP-1 Capillary, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 200 °C (1 min), 10 °C/min to 270 °C (3.5 min), 20 °C/min to 300 °C (3 min)
Injector: 250 °C Detector Temp: 320 °C
Carrier: Helium Split ratio: 20/1
Relative peak area response of main component:
Initial analysis: Mean = 99.6%, s = 0.02% (8 sub samples in duplicate, June 2001)
Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, September 2008)
Re-analysis: Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, July 2013)
Re-analysis: Mean = 99.5%, s = 0.02% (3 sub samples in duplicate, June 2018)

Thermogravimetric analysis: Volatiles content < 0.1% and non-volatile content < 0.2% mass fraction
(April 2001, November 2005 and September 2008)

Karl Fischer analysis: Moisture content < 0.1% mass fraction (2 sub samples, September 2008)
Moisture content ≤ 0.2% mass fraction (2 sub samples, July 2013)
Moisture content ≤ 0.2% mass fraction (2 sub samples, June 2018)

GC-MS:	Instrument: HP6890/5973 Column: ZB-5, 30 m × 0.25 mm I.D. × 0.3 μm Program: 230 °C (1 min), 10 °C /min to 300 °C (5 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 (7.1 min): 234 (M ⁺ -CH ₂ C ₆ H ₃ (MeO) ₂ , 100), 192 (80), 176 (12) <i>m/z</i>
TLC:	Conditions: Kieselgel 60F ₂₅₄ . Chloroform/acetone (80:20) Single spot observed, R _f = 0.42 (3 replicates)
IR:	Instrument: FT-IR, Biorad WIN FTS40 Range: 4000-400 cm ⁻¹ , KBr pellet Peaks: 3078, 2940, 2834, 1635, 1516, 1444, 1159, 1022, 821 cm ⁻¹
¹ H NMR:	Instrument: Bruker DMX-600 Field strength: 600 MHz Solvent: d ₅ -pyridine (7.445 ppm) Spectral data: δ 1.63 (3H, s), 2.01 (3H, s), 2.47 (1H, ddd, <i>J</i> = 4.3, 15.8), 2.54 (1H, br dd, <i>J</i> = 16.1), 2.61 (1H, ddd, <i>J</i> = 5.6, 9.9, 15.8), 2.79 (1H, ddd, <i>J</i> = 5.9, 11.8, 16.1), 3.07-3.18 (5H, m), 3.34 (1H, ddd, <i>J</i> = 4.6, 9.9, 13.5), 3.49-3.53 (1H, m), 3.52 (3H, s), 3.57 (3H, s), 3.58 (3H, s), 3.61 (3H, s), 3.63 (3H, s), 3.64 (3H, s), 3.65 (3H, s), 3.67 (3H, s), 4.95 (1H, dd, <i>J</i> = 5.3, 13.2), 5.04 (1H, dd, <i>J</i> = 4.3, 9.9), 5.98 (1H, dd, <i>J</i> = 6.9), 6.48 (1H, s), 6.62 (1H, s), 6.64 (1H, s), 6.71-6.75 (2H, m), 6.78-6.82 (2H, m), 6.86 (1H, s), 6.91 (1H, s), 6.92 (1H, s) ppm. Solvents signals (DCM 0.1% and diethyl ether 0.14%) were observed in the proton NMR.
¹³ C NMR:	Instrument: Bruker DMX-600 Field strength: 151 MHz Solvent: d ₅ -pyridine (150.0 ppm) Spectral data: δ 21.4, 22.1, 28.4, 28.8, 35.1, 41.4, 42.2, 42.6, 54.0, 55.9, 55.99, 56.02, 56.04, 56.1, 56.2, 59.4, 111.7, 112.0, 112.4, 112.5, 112.6, 112.9, 114.38, 114.42, 122.47, 122.53, 126.8, 127.2, 129.5, 129.6, 131.7, 132.0, 148.2, 148.5, 148.7, 148.8, 149.0, 149.1, 169.1, 169.3 ppm.
Microanalysis:	Found: C = 68.4%; H = 7.2%; N = 3.6% (June 2001) Calc: C = 68.6%; H = 7.1%; N = 3.6% (Calculated for C ₂₂ H ₂₇ NO ₅)
Melting point:	112–114 °C

Expiration of certification

The property values are valid till 22nd June 2023, 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 bottle 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 five 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 eight randomly selected 1-2 mg 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.

Metrological traceability

The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis.

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.

Intended use

This certified reference material may be used for instrument calibration.

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: 27 June, 2018.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 27 June, 2018.