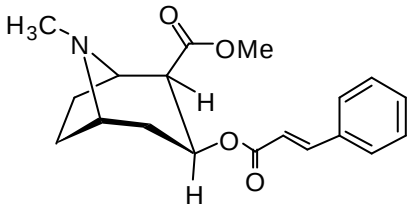
Australian Government

National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D755.2015.01

Compound Name: **trans-Cinnamoyl cocaine**
Collection Number: D755
Chemical Formula: $C_{19}H_{23}NO_4$
CAS Number: 50763-20-7
Structure:



Description: White powder
Batch Number: 02-D-31
Molecular Weight: 329.3
Release date: November 2002

Synonyms: (*E*)-[1R-(*exo*,*exo*)]-8-Methyl-3-[(1-oxo-3-phenyl-2-propenyl)oxy]-8-azabicyclo[3,2,1]octane-2-carboxylic acid methyl ester
Ecgonine cinnamate methyl ester
Cinnamoylecgoninmethyl ester
Cinnamoylmethylecgonine

Purity (mass fraction): 99.2 ± 0.3% (95% coverage interval)

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

GC-FID:	Instrument:	Agilent 6890	
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	180 °C (1 min), 10 °C/min to 260 °C (1 min), 30 °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 = 99.3%, s = 0.01% (10 samples in duplicate, September 2002)	
	Re-analysis:	Mean = 99.2%, s = 0.01% (3 sub samples in duplicate, October 2003)	
	Re-analysis:	Mean = 99.3%, s = 0.01% (3 sub samples in duplicate, October 2004)	
	Re-analysis:	Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, October 2007)	
	Re-analysis:	Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, January 2011)	
	Re-analysis:	Mean = 99.2%, s = 0.02% (5 sub samples in duplicate, November 2015)	
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (October 2007, January 2011 & November 2015)	

Spectroscopic and other characterisation data

GC-MS:	Parent compound:		
	Instrument:	Agilent 6890/5973	
	Column:	Zebron ZB-5, 30 m x 0.25 mm I.D. x 0.30 µm	
	Program:	180 °C 10 °C/min to 260 °C (4 min)	
	Injector:	280 °C	Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/mim	Split ratio: 20/1
	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 (12.8 min): 329 (M^+, 6), 238 (10), 131 (26), 103 (27), 96 (46), 94 (41), 82 (100) m/z		
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/conc. NH_3 (200/3) Single spot observed, R_f = 0.75 Visualisation with UV at 254 nm	
IR:	Instrument:	Biorad FTS3000MX FT-IR	
	Range:	4000-400 cm^{-1}, KBr powder	
	Peaks:	2959, 2856, 2804, 1749, 1699, 1630, 1319, 1179, 1037, 1008, 767, 683 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker DMX-300	
	Field strength:	300 MHz	Solvent: $CDCl_3$ (7.26 ppm)
	Key spectral data:	δ 2.21 (3H, s), 2.40 (1H, ddd), 3.71 (3H, s), 5.11 (1H, ddd), 6.44 (1H, d), 7.36 (3H, m), 7.51 (2H, m), 7.65 (1H, d) ppm	
^{13}C NMR:	Instrument:	Bruker Gyro-300	
	Field strength:	75.5 MHz	Solvent: $CDCl_3$ (77.16 ppm)
	Spectral data:	δ 25.2, 25.4, 35.5, 41.2, 50.1, 51.4, 61.6, 64.8, 66.6, 118.3, 128.1 (x 2), 128.8 (x 2), 130.2, 134.4, 144.9, 166.7, 170.8 ppm	
Melting point:		122-124 °C	
Microanalysis:		Found: C = 69.4%; H = 7.2%; N = 4.1% (October, 2002) Calc: C = 69.3%; H = 7.0%; N = 4.3% (Calculated for $C_{25}H_{34}O_8$)	

Expiration of certification
The property values are valid till 6th November 2020, 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 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 ten 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.

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.

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: 7th December 2015.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 7th December 2015.