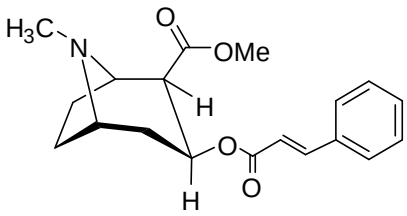
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D755: trans-Cinnamoyl cocaine



Report ID: D755.2020.03

Chemical Formula: $C_{19}H_{23}NO_4$

Molecular Weight: 329.3 g/mol

Property value

Batch No.	CAS No.	Purity estimate
02-D-31	50763-20-7	99.2 ± 0.3%

IUPAC name: Methyl (1R,2R,3S,5S)-8-methyl-3-{[(2E)-3-phenyl-2-propenoyl]oxy}-8-azabicyclo[3.2.1]octane-2-carboxylate.

Expiration of certification: The property values are valid till 4 September 2030, 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: 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 reference material is recommended for qualitative analysis only.

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: In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years. This material has demonstrated stability over a minimum period of five 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 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.

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.
13 October 2022

This report supersedes any issued prior to 15 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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, 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:	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 of the 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)
	Re-analysis:	Mean = 99.2%, s = 0.02% (5 sub samples in duplicate, September 2020)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (October 2007, January 2011, November 2015 and July 2020)

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
	Split ratio:	20/1
	Transfer line temp:	280 °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 (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)
	Spectral data:	δ 2.21 (3H, s), 2.40 (1H, ddd, *J* = 3.0, 12.0, 12.0 Hz), 3.71 (3H, s), 5.11 (1H, ddd, *J* = 6.0, 6.0, 12.1 Hz), 6.44 (1H, d, *J* = 15.8 Hz), 7.36 (3H, m), 7.51 (2H, m), 7.65 (1H, d, *J* = 16.2 Hz) 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)
	Calculated:	C = 69.3%; H = 7.0%; N = 4.3% (Calculated for $C_{25}H_{34}O_8$)