

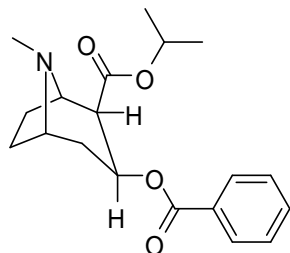


REFERENCE MATERIAL PRODUCT INFORMATION SHEET

Report ID: D936.2018.01

Compound Name: **Benzoylecgonine isopropyl ester**  
Collection Number: D936  
Chemical Formula: C<sub>19</sub>H<sub>25</sub>NO<sub>4</sub>  
CAS Number: 137819-55-7  
Structure:

Description: Off white solid  
Batch Number: 08-D-15  
Molecular Weight: 331.4  
Release date: 3<sup>rd</sup> June 2009



Synonyms: (1R,2R,3S,5S)-3-(Benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid-1-methylethyl ester,  
[1R-(exo,exo)]-3-(Benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid-methyl-1-methylethyl ester

Purity (mass fraction): 98.8 ± 0.3 % (95 % confidence interval)

The purity value was obtained from a combination of traditional analytical techniques by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and <sup>1</sup>H NMR spectroscopy. 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: 150 °C (1 min), 10 °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 = 98.8%, s = 0.06% (10 sub samples in duplicate, April 2009)  
Re-analysis: Mean = 99.2%, s = 0.08% (5 sub samples in duplicate, March 2013)  
Re-analysis: Mean = 99.2%, s = 0.07% (5 sub samples in duplicate, January 2018)

Thermogravimetric analysis: Non volatile residue < 0.2 % mass fraction (April 2009)  
Volatile content not determined due to volatility of the material.

Karl Fischer analysis: Moisture content < 0.1% mass fraction (April 2009 & 2010, March 2013, January 2018)

### Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1ms, 14.9 m × 0.25 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 15 °C/min to 200 °C, 40 °C/min to 300 °C (3 min).
	Injector:	250 °C
	Carrier:	Helium, 1.0 mL/min
		Transfer line temp: 280 °C
		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.	
	8.4 min: 331 (M <sup>+</sup> , 25), 272 (23), 226 (10), 210 (73), 168 (22), 122 (10), 105 (36), 94 (39), 82 (100) m/z	
ESI-MS:	Instrument:	Micromass Quattro Micro
	Operation:	Positive ion mode
	Injection:	Direct infusion of methanol / water (1:1) at 5.0 µL/min
	Ionisation	ESI spray voltage at 3.5 kV
	Cone voltage:	20 V
	Peak:	332 (M-H) m/z
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Methanol / Ammonium solution (28%) (100/1.5) Single spot observed, R <sub>f</sub> = 0.83. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR.
	Range:	4000-400cm <sup>-1</sup> , KBr powder.
	Peaks:	2977, 2885, 2801, 1736, 1717, 1453, 1372, 1283, 1226 1185, 1142, 1037, 717 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Spectral data:	Solvent: CDCl <sub>3</sub> (7.26 ppm) δ 1.20 (3H, d, J = 6.2 Hz), 1.24 (3H, d, J = 6.2 Hz), 1.65-1.78 (2H, m), 1.81-1.88 (1H, m), 2.03-2.22 (2H, m), 2.22 (3H, s), 2.45 (1H, dd, J = 3.0, 11.9 Hz), 2.94 (1H, dd, J = 3.0, 5.2 Hz), 3.28 (1H, m), 3.55 (1H, m), 5.10 (1H, s, J = 6.3 Hz), 5.22 (1H, m), 7.41 (2H, m), 7.53 (1H, m), 8.03 (2H, m) ppm. <sup>13</sup> C
NMR:	Instrument:	Bruker Avance-400
	Field strength:	100 MHz
	Spectral data:	Solvent: CDCl <sub>3</sub> (77.16 ppm) δ 22.0, 22.03, 25.5, 35.7, 41.3, 50.4, 61.7, 65.2, 67.0, 67.3, 128.4, 129.9, 130.6, 133.0, 166.4, 169.8 ppm.
Melting point:		60-62 °C
Microanalysis:	Found:	C = 68.8%; H = 7.8%; N = 4.2% (April 2009)
	Calc:	C = 68.9%; H = 7.6%; N = 4.2% (Calculated for C <sub>19</sub> H <sub>25</sub> NO <sub>4</sub> )

### Expiration of certification

The property values are valid till 4<sup>th</sup> January 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 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.

In the absence of stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last 10 years.

### 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 25 °C 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: 7 February 2018

Characterisation data and property values specified in this report supersede all reports issued prior to 7<sup>th</sup> February 2018.