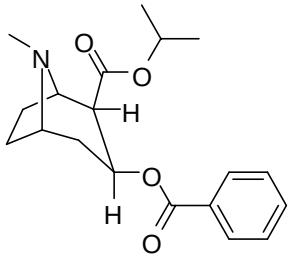
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D936: Benzoylecgonine isopropyl ester



Report ID: D936.2022.01

Chemical Formula: $C_{19}H_{25}NO_4$

Molecular Weight: 331.4 g/mol

Property value

Batch No.	CAS No.	Purity estimate
08-D-15	137819-55-7	99.2 ± 0.5%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: Isopropyl (1R,2R,3S,5S)-3-(benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylate

Expiration of certification: The property values are valid till 25 August 2032, 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: Off-white solid 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 25 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of three 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.
30 August 2022

This report supersedes any issued prior to 30 August 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, thermogravimetric analysis, 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 or 7890
	Column:	HP-1 or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	150°C(1 min),10°C/min to 210°C (0 min), 3°C/min to 245°C (3 min), 30°C/min to 300°C (6 min)
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the 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)
	Re-analysis	Mean = 99.2%, s = 0.04% (5 sub samples in duplicate, August 2022)
	Karl Fischer analysis:	Moisture content < 0.1% mass fraction (April 2009 & 2010, March 2013, January 2018, August 2022)
Thermogravimetric analysis:		Non-volatile residue < 0.2 % mass fraction (April 2009). Volatile content not determined due to volatility of the material.

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
	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 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 (8.4 min):	331 (M^+, 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
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	332 (M-H) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol / Ammonium solution (28%) (100:1.5) Single spot observed, R_f = 0.83. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR.
	Range:	4000-400cm^{-1}, KBr powder.
	Peaks:	2977, 2885, 2801, 1736, 1717, 1453, 1372, 1283, 1226 1185, 1142, 1037, 717 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	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.
^{13}C NMR:	Instrument:	Bruker Avance-400
	Field strength:	100 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	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)
	Calculated:	C = 68.9%; H = 7.6%; N = 4.2% (Calculated for $C_{19}H_{25}NO_4$)