Australian Government

National Measurement Institute

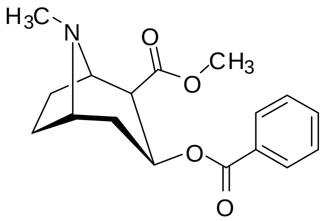
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D826b.2016.01

Compound Name: **Cocaine base**
Collection Number: D826b
Chemical Formula: $C_{17}H_{21}NO_4$
CAS Number: 50-36-2

Description: White powder
Batch Number: 09-D-29
Molecular Weight: 303.4
Release Date: 14th April 2010

Structure:



Synonyms:
- l-Cocaine
- Benzoylmethylecgonine
- Ecgonine methyl ester benzoate
- 2β-Carbomethoxy-3β-(benzoyloxy)tropane
- 3β-Hydroxy-2β-tropanecarboxylic acid methyl ester benzoate
- 3β-Hydroxy-1αH, 5αH-tropane-2β-carboxylic acid methyl ester benzoate
- [1*R*-(*exo*-*exo*)]-3-(Benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid methyl ester

Purity (mass fraction): 96.1 ± 2.6% (95% coverage interval)

Purity estimate obtained from quantitative nuclear magnetic resonance (qNMR). The purity estimate by qNMR was obtained using a certified internal standard of Potassium hydrogen maleate. Supporting evidence is provided by GC-FID, thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR and elemental microanalysis.

qNMR:
Instrument: Bruker Avance III-400
Field strength: 400 MHz Solvent: D_2O + DCl
Internal standard: Potassium hydrogen maleate
Purity estimate: Mean = 96.1%, s = 0.9% (5 sub samples, December 2009)

GC-FID:
Instrument: Agilent 6890N
Column: HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
Program 80 °C (1 min), 15 °C/min to 200 °C (10 min), 15 °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 = 98.6%, s = 0.20% (7 sub samples in duplicate, December 2009)
Re-analysis: Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, April 2012)
Re-analysis: Mean = 99.2%, s = 0.06% (9 sub samples in duplicate, November 2013)
Re-analysis: Mean = 99.1%, s = 0.01% (6 sub samples in duplicate, October 2016)

GC-FID:
Instrument: Varian CP-3800
Column: VF-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
Program: 80 °C (1 min), 15 °C/min to 200 °C (10 min), 15 °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 = 98.7 %, s = 0.1% (7 sub samples in duplicate, December 2009)

Thermogravimetric analysis: Non volatile residue < 0.1% Volatile content not determined due to volatility of the material

Karl Fischer analysis: Moisture content ≤ 0.1% mass fraction (November 2009, April 2012, October 2013 and September 2016)

Spectroscopic and other characterisation data

GC-MS: Instrument: Agilent 6890/5973
Column: VF-1ms, 15 m × 0.25 mm I.D. × 0.25 µm
Program: 120 °C (10 min), 15 °C/min to 300 °C (3 min)
Injector: 250 °C Transfer line temp: 300 °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.

16.7 min: 303 (M^+, 27) 272 (11), 198 (12), 182 (100), 122 (10), 105 (29), 94 (32), 82 (90), 77 (26), 42 (13) *m/z*

TLC: Conditions: Kieselgel $60F_{254}$. Methanol/conc NH_3 (200:3)
Single spot observed, $R_f = 0.7$
Visualization with UV light (254 nm)

IR: Instrument: BioRad FTS3000MX FT-IR
Range: 4000-400 cm^{-1}, KBr powder
Peaks: 2977, 2947, 2885, 2852, 2803, 1737, 1709, 1450, 1317, 1279, 1229, 1112, 1038, 776, 714 cm^{-1}

^1H NMR: Instrument: Bruker Avance III-400
Field strength: 400 MHz Solvent: $CDCl_3$ (7.26 ppm)
Spectral data: δ 1.72 (2H, m), 1.88 (1H, m), 2.05-2.19 (2H, m), 2.22 (3H, s), 2.43 (1H, ddd, J = 2.8, 11.8, 11.8 Hz), 3.01 (1H, m), 3.29 (1H, m), 3.56 (1H, m), 3.71 (3H, s), 5.24 (1H, ddd, J = 6.0, 8.8, 8.8 Hz), 7.41 (2 H, t, J = 7.7, 7.7 Hz), 7.53 (1 H, t, J = 7.4, 6.6 Hz), 8.01 (2H, d, J = 7.4 Hz) ppm
n-Hexane was observed in the ^1H NMR at 0.5-0.6 % mass fraction. (December 2009, April 2012 and November 2013)

^{13}C NMR: Instrument: Bruker Avance III-400
Field strength: 100 MHz Solvent: $CDCl_3$ (77.16 ppm)
Spectral data: δ 25.4, 25.6, 35.7, 41.3, 50.4, 51.6, 61.7, 65.0, 67.1, 128.5, 129.9, 130.5, 133.1, 166.4, 170.9 ppm

Melting point: 92-94 °C

Microanalysis: Found: C = 67.5 %; H = 7.0 %; N = 4.5 % (December 2009)
Calc: C = 67.3 %; H = 7.0 %; N = 4.6 % (Calculated for $C_{17}H_{21}NO_4$)

Expiration of certification

The property values are valid till 12th October 2019, i.e. three 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 three 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 seven 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.

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. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

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.

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: 10 February, 2017.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 17th October 2016.

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