

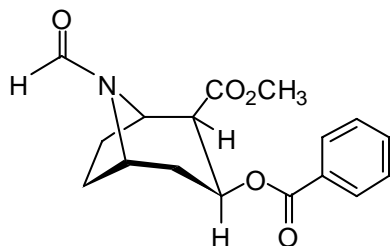


REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D857.2010.03

Compound Name: *N*-Formylcocaine  
Collection No: D857  
Chemical Formula: C<sub>17</sub>H<sub>19</sub>NO<sub>5</sub>  
CAS Registry No: 137360-14-6  
Structure:

Description: Viscous oil  
Batch No: 04-D-04  
Molecular Weight: 317.3  
Batch production completed: May 2004



Synonyms: [1R-(exo, exo)]-3-(Benzoyloxy)-8-formyl-8-azabicyclo [3.2.1]octane-2-carboxylic acid methyl ester  
*N*-Formylcocaine

Purity (mass fraction): 96.3 ± 0.8% (95% confidence interval)

Purity estimate obtained by subtraction from 100% of total impurities by GC-FID, Karl Fischer and <sup>1</sup>H NMR.

GC-FID Instrument: HP 6890N  
Column: HP-1 29 74m × 0.32mm × 0.25 μm  
Program: 200 °C (2 min), 10 °C/min to 300 °C (3 min)  
Injector: 240 °C Detector Temp: 320 °C  
Carrier: Helium Split ratio: 20/1  
Relative peak area response of main component:  
Initial analysis: Mean = 97.9%, s = 0.1 (7 sub samples in duplicate, March 2004)  
Current analysis: Mean = 98.0%, s = 0.03 (2 sub samples in triplicate, October 2010)

GC-MS: Instrument: Agilent 6890 / 5973  
Column: Zebron ZB-5, 30 m x 0.25 mm I.D. x 0.30 μm  
Program: 150 °C, (1 min), 10 °C/min to 300 °C (2 min)  
Injector: 180 °C Transfer line temp: 280 °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 (m/z) and as a percentage relative to the base peak.

15.7 min: 289 (33), 195 (34), 168 (96), 136 (36), 105 (100), 77 (68), 68 (54) m/z

TLC: Conditions: Kieselgel 60F<sub>254</sub>. 100% Ethyl acetate.  
Single spot observed, R<sub>f</sub> = 0.23. Visualization with UV light (254 nm).

IR: Instrument: BioRad FTS3000MX FT-IR  
Range: 4000-400 cm<sup>-1</sup>, KBr powder  
Peaks: 3464, 2954, 1739, 1718, 1669, 1436, 1389, 1276, 1113, 1035, 714 cm<sup>-1</sup>

<sup>1</sup>H NMR: Instrument: Bruker DMX-600  
Field strength: 600 MHz Solvent: CDCl<sub>3</sub>  
Double resonances observed due to restricted rotation about the amide bond.  
Rotamers present in ca. 1:1 ratio at room temperature.  
Key spectral data: **Rotamer A** δ 2.37 (1H, ddd), 3.25 (1H, bdd), 3.65 (3H, s), 4.30 (1H, bd), 4.81 (1H, m), 5.53 (1H, ddd), 8.02 (0.5H, s) ppm  
**Rotamer B** δ 2.53 (1H, ddd), 3.17 (1H, bdd), 3.68 (3H, s), 4.27 (1H, m), 4.95 (1H, bd), 5.49 (1H, ddd), 8.16 (1H, s) ppm

<sup>13</sup>C NMR: Instrument: Bruker DMX-600  
Field strength: 151MHz Solvent: CDCl<sub>3</sub>  
Spectral data: δ 26.9, 27.4, 27.9, 28.4, 33.4, 35.4, 48.5, 48.7, 49.3, 51.1, 51.9, 52.0, 53.6, 55.5, 66.2, 66.3, 128.4, 128.4, 129.6, 129.7, 133.3, 133.3, 157.8, 158.0, 165.6, 165.7, 169.6, 170.0 ppm

Microanalysis: Found: C = 62.7%; H = 5.9%; N = 4.2% (April 2004)  
Calc: C = 64.3%; H = 6.0%; N = 4.4% (Calculated for C<sub>17</sub>H<sub>19</sub>NO<sub>5</sub>)

Thermogravimetric analysis: Volatile content 1.4% and non-volatile residue < 0.2 % mass fraction (May 2004, October 2006 & October 2007).

Karl Fisher titration: Moisture content 1.7% mass fraction (October 2007)

### Expiration of certification

The property values are valid till 15<sup>th</sup> October 2015, 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 3 years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials.

### Recommended storage

When not in use this material should be stored at or below 20 °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: 19 July, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 19<sup>th</sup> July 2012



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