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Recommended methods for the **Identification and Analysis of Cocaine in Seized Materials**

MANUAL FOR USE BY NATIONAL DRUG ANALYSIS LABORATORIES

Laboratory and Scientific Section
UNITED NATIONS OFFICE ON DRUGS AND CRIME
Vienna

Recommended Methods for the Identification and Analysis of Cocaine in Seized Materials

(Revised and updated)

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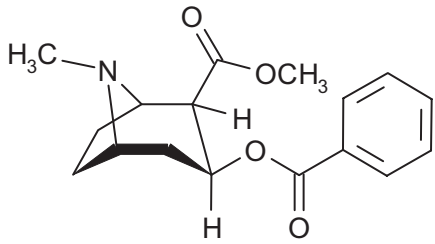
UNITED NATIONS
New York, 2012

3. Description of the pure compounds

The compounds listed below include cocaine, major components (> 1% by weight) and minor components (usually < 1% by weight). Trace components (usually < 0.1% by weight and typically requiring an extraction step) are not described here.

Cocaine



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

3β-Hydroxy-1αH,5αH-tropane-2β-carboxylic acid methyl ester benzoate

Ecgonine methyl ester benzoate

l-Cocaine

β-Cocaine

Benzoylmethylecgonine

$C_{17}H_{21}NO_4$
Molecular Weight = 303.4 (base), 339.8 (hydrochloride)
Melting point: 98° C (base), 195° C (hydrochloride)

Solubilities (1g/ml):	*Base*	*Hydrochlorid*e
Water	slightly soluble (1 in 600)	soluble (1 in 0.4)
Ethanol	soluble (1 in 6.5)	soluble (1 in 3.2)
Diethyl ether	soluble (1 in 3.5)	practically insoluble
Chloroform	soluble (1 in 0.7)	soluble (1 in 12.5)

GC-MS data (percentage abundance):

303 (M^+, 17), 182 (71), 105 (29), 96 (24), 94 (36), 82 (100), 77 (35) m/z

NMR data (hydrochloride):

^{1}H NMR 600 MHz; (D_2O): δ 2.90 (3H, s), 3.63 (3H, s), 3.65 (1H, dd,), 4.10 (1H, bm), 4.24 (1H, bm), 5.59 (1H, ddd), 7.54, (1H, t), 7.70 (1H, t), 7.96 (1H, d) ppm

^{13}C NMR (151 MHz; D_2O): δ 22.8, 23.9, 32.8, 39.1, 46.3, 53.6, 63.4, 64.1, 64.7, 128.7, 129.2, 129.8, 134.7, 167.5, 173.6 ppm

Infrared data:

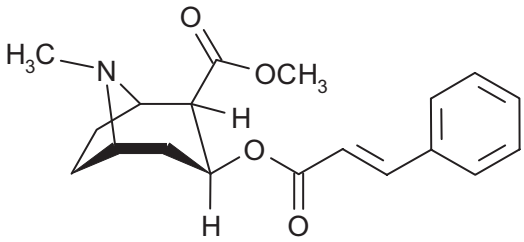
Principal peaks at wavenumbers 1710, 1738, 1275, 1110, 712, 1037 cm^{-1} (KBr disk).

UV Data:

Aqueous acid—233 nm (A¦ = 430), 275 nm

Major and minor components

Cinnamoylcocaine



Synonyms: [1R-(*exo,exo*)]-8-Methyl-3-[(1-oxo-3-phenyl-2-propenyl)oxy]-8-azabicyclo[3.2.1]octane-2-carboxylic acid methyl ester

Ecgonine cinnamate methyl ester

Cinnamoylecgoninmethyl ester

Cinnamoylmethylecgonine

Cinnamylcocaine

$C_{19}H_{23}NO_4$

Molecular Weight: 329.4

Melting point: 121° C (base)

Solubilities (1g/ml):	*Base*	*Hydrochloride*
Water	almost insoluble	soluble
Ethanol	soluble	soluble
Diethyl ether	soluble	soluble
Chloroform	soluble	slightly

GC-MS data (percentage abundance):

329 (M^+, 15), 238 (14), 182 (72), 131 (33), 103 (24), 96 (59), 94 (35), 82 (100), 42 (27) m/z

NMR data (hydrochloride):

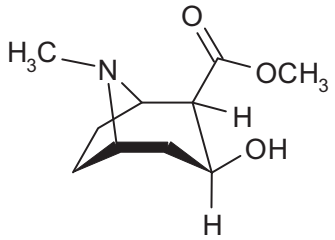
^{1}H NMR (300 MHz; $CDCl_3$): (Key spectral data): δ 2.21 (3H, s), 2.40 (1H, ddd), 3.71 (3H, s), 5.11 (1H, ddd), 6.44 (1H, d), 7.36 (3H, m), 7.51 (2H, m), 7.65 (1H, d) ppm

^{13}C NMR (75.5 MHz; $CDCl_3$): 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

Infrared data:

Principal peaks at wavenumbers 2959, 2856, 2804, 1749, 1699, 1630, 1319, 1179, 1037, 1008, 767, 683 cm^{-1} (KBr disk)

Methylecgonine



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

Ecgonine methyl ester

3β-Hydroxy-1αH,5αH-tropane-2β-carboxylic acid methyl ester

$C_{10}H_{17}NO_3$

Molecular Weight: 199.3 (base), 235.7 (hydrochloride)

Melting point: oil (base), 215° C (hydrochloride)

GC-MS data (percentage abundance):

199 (M^+, 30), 168 (18), 112 (12), 96 (78), 94 (38), 82 (100), 68 (8), 42 (32) m/z

NMR data (hydrochloride):

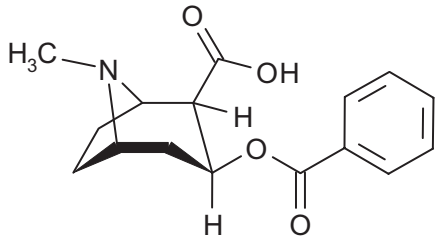
^{1}H NMR (500 MHz; D_2O): δ 2.03-2.14 (3H, m), 2.20-2.24 (1H, m), 2.30-2.48 (2H, m), 2.83 (3H, s), 3.31 (1H, dd, *J* = 2.2, 7.2 Hz), 3.80 (3H, s) 3.99 (1H, m), 4.15 (1H, bd, *J* = 7.0 Hz), 4.43-4.48 (1H, m) ppm

^{13}C NMR (125 MHz; D_2O): δ 22.5, 23.5, 34.8, 38.4, 48.8, 52.8, 60.3, 63.1, 63.8, 174.2 ppm

Infrared data:

Principal peaks at wavenumbers 3269, 2963, 2132, 1704, 1481, 1428, 1350, 1215, 1140, 1049, 1013, 968, 777, 616 cm^{-1} (KBr disk).

Benzoylecgonine



Synonyms: 3-(Benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid
3β-Hydroxy-1αH,5αH-tropane-2β-carboxylic acid benzoate

Ecgonine benzoate

$C_{16}H_{19}NO_4$

Molecular Weight: 289.3

Melting point: 195° C (anhydrous) (decomposes), 86-92° C (tetrahydrate), 200° C hydrochloride

Solubilities (1g/ml):	*Base*	*Hydrochloride*
Water, boiling	soluble	soluble
Ethanol	soluble	soluble

GC-MS data (percentage abundance):

289 (M^+, 5), 168 (26), 124 (100), 105 (31), 96 (19), 94 (26), 82 (61), 77 (40), 67 (11) m/z

NMR data (hydrochloride):

^{1}H NMR (300 MHz; D_2O): (Key spectral data): δ 2.61-2.17 (6H, m) 2.88 (3H, s), 3.22 (1H, dd), 4.07 (2H, bd), 5.54 (1H, m), 7.59 (2H, dd), 7.76 (1H, dd), 8.06 (2H, d) ppm

^{13}C NMR (75.5 MHz; D_2O): δ 23.2, 32.6, 37.6, 48.8, 62.3, 64.8, 128.7, 128.9, 129.5, 133.9, 167.2, 176.9 ppm

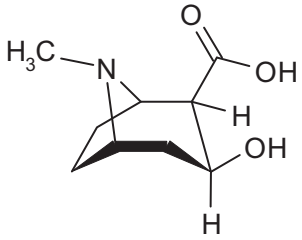
Infrared data:

Principal peaks at wavenumbers 1275, 1720, 1618, 717, 1116, 1316 cm^{-1}

UV Data:

Aqueous acid—234 nm (A¦ = 376), 274 nm

Ecgonine



Synonyms: [1R-(*exo,exo*)]-3-Hydroxy-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylic acid

3β-Hydroxy-1α*H*,5α*H*-tropane-2β-carboxylic acid

$C_9H_{15}NO_3$

Molecular Weight: 185.2 (base), 221.7 (hydrochloride)

Melting point: 198° C (base), 246° C (hydrochloride)

Solubilities (1g/ml):	*Base*	*Hydrochloride*
Water	soluble	soluble
Ethanol	slightly soluble	slightly soluble
Diethyl ether	sparingly soluble	
Chloroform	sparingly soluble	

GC-MS data (percentage abundance):

185 (M^+, 9), 124 (33), 96 (82), 82 (100), 57 (54), 42 (89) m/z

NMR data (hydrochloride):

^{1}H NMR (600 MHz; D_2O): δ 1.98-2.19 (4H, m), 2.25-2.41 (2H, m), 2.78 (3H, s), 3.18 (1H, dd, *J* = 2.3, 7.1 Hz), 3.92 (1H, m), 4.10 (1H, d, *J* = 7.3 Hz), 4.41 (1H, m) ppm

^{13}C NMR (150 MHz; D_2O): δ 23.2, 24.0, 35.5, 38.9, 49.5, 60.8, 63.6, 64.6, 176.4 ppm

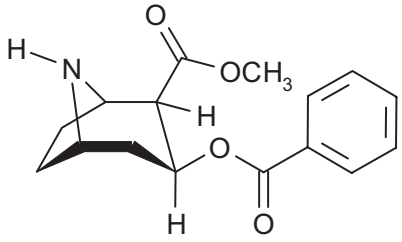
Infrared data:

Principle peaks at wavenumbers 1688, 1210, 1200, 1223, 1134, 1179 cm^{-1} (ecgonine hydrochloride, KBr disk)

UV Data:

Ethanol—275 nm

Norcocaine



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

$C_{16}H_{19}NO_4$

Molecular Weight: 289.3 (base), 325.8 (hydrochloride)

Melting point: 115-116° C (hydrochloride)

Solubilities (1g/ml):	*Hydrochloride*
Water	soluble
Ethanol	slightly soluble

GC-MS data (percentage abundance):

289 (M^+, 11), 168 (100), 136 (37), 108 (25), 105 (23), 82 (13), 80 (23), 77 (33), 68 (41) m/z

NMR data (hydrochloride):

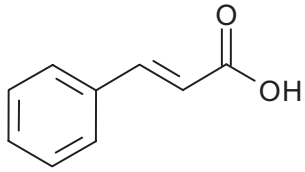
^{1}H NMR (500 MHz; D_2O): (Key spectral data): δ 3.59 (1H, dd), 3.64 (3H, s), 4.38 (1H, bd), 5.56 (1H, ddd), 7.54 (2H, t), 7.71 (1H, t), 7.95 (1H, d) ppm

^{13}C NMR (75.5 MHz; $CDCl_3$): δ 24.4, 25.1, 31.1, 44.9, 53.0, 54.3, 55.4, 65.0, 128.4, 128.8, 129.4, 134.3, 167.0, 173.0 ppm

Infrared data:

Principle peaks at wavenumbers 3597, 3408, 3152, 2951, 2772, 2744, 2527, 1721, 1440, 1350, 1275, 717 cm^{-1}

*Cinnamic acid (*trans-*)*



Synonyms: 3-phenyl-2-propenoic acid

β-phenylacrylic acid

$C_9H_8O_2$

Molecular Weight: 148.2

Melting point: 133° C (base)

Solubilities (1g/ml):	*Base*
Water	slightly soluble
Ethanol	soluble
Diethyl ether	soluble
Chloroform	soluble

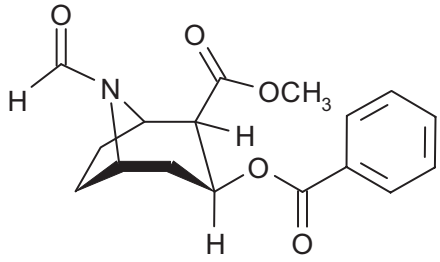
GC-MS data (percentage abundance):

148 (M^+, 74), 148 (100), 147 (100), 131 (22), 103 (61), 77 (47), 51 (40) m/z

UV Data:

Ethanol—273 nm

N-*Formylnorcocaine*



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

N-Formylcocaine

$C_{17}H_{19}NO_5$

Molecular Weight: 317.3

GC-MS data (percentage abundance):

289 (38), 195 (39), 168 (100), 136 (42), 105 (94), 77 (58), 68 (48) m/z.

NMR data:

^{1}H NMR (600 MHz; $CDCl_3$): 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

^{13}C NMR ($CDCl_3$): 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, 129.6, 129.7, 133.3, 157.8, 158.0, 165.6, 165.7, 169.6, 170.0 ppm

For additional details related to the substances, the reader is referred to the *Multilingual Dictionary of Narcotic Drugs and Psychotropic Substances Under International Control* (http://www.unodc.org/unodc/en/scientists/multilingual-dictionary-of-narcotic-drugs-and-psychotropic-substances-under-international-control.html.html), the widely used Merck Index [1] and *Clarke's Analysis of Drugs and Poisons* [2].