

APICA *N*-(5-fluoropentyl) analog (7)

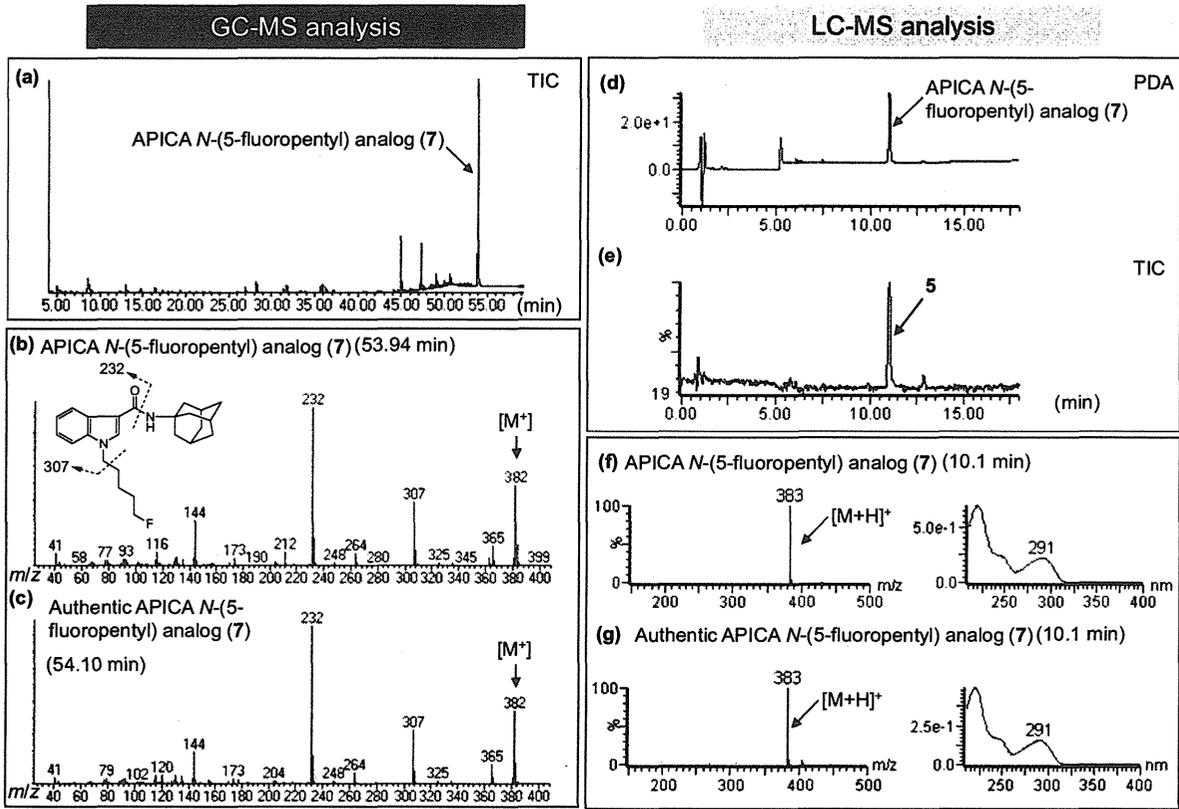


Fig. 11 GC-MS and LC-MS analyses of product D.

APINACA *N*-(5-fluoropentyl) analog (8)

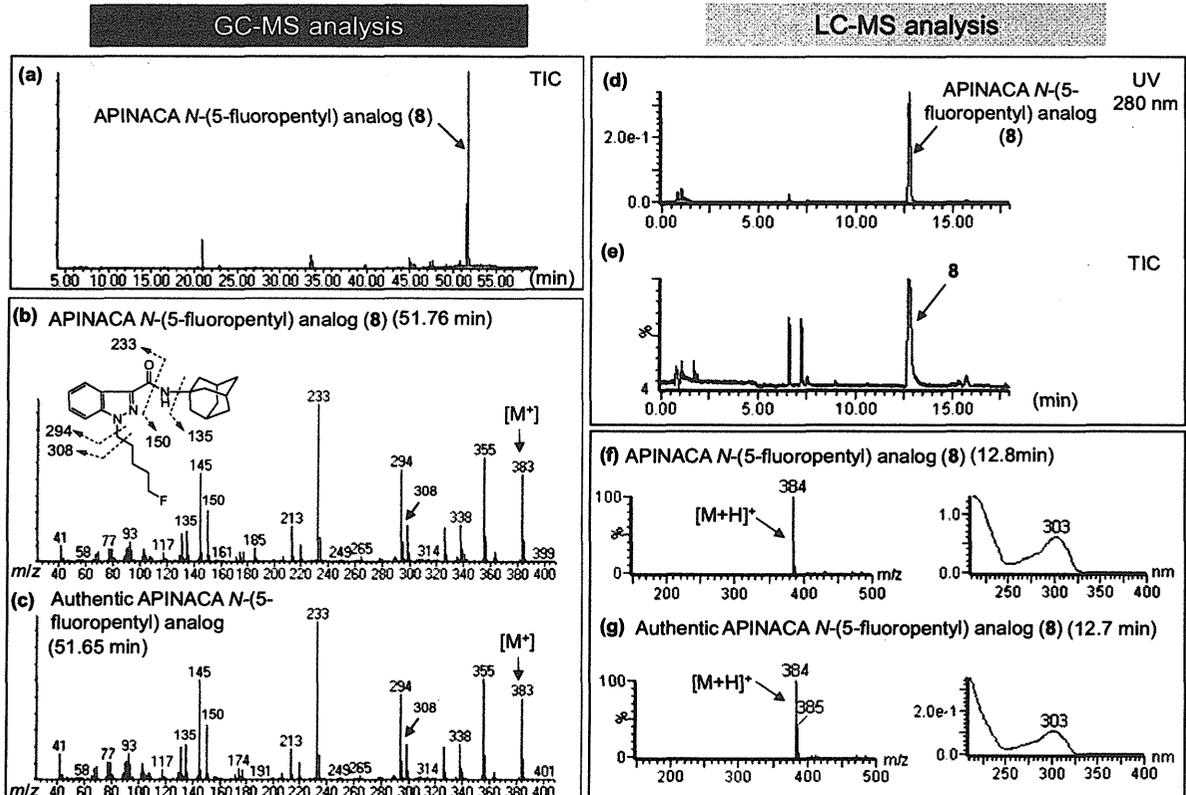


Fig. 12 GC-MS and LC-MS analyses of product E.

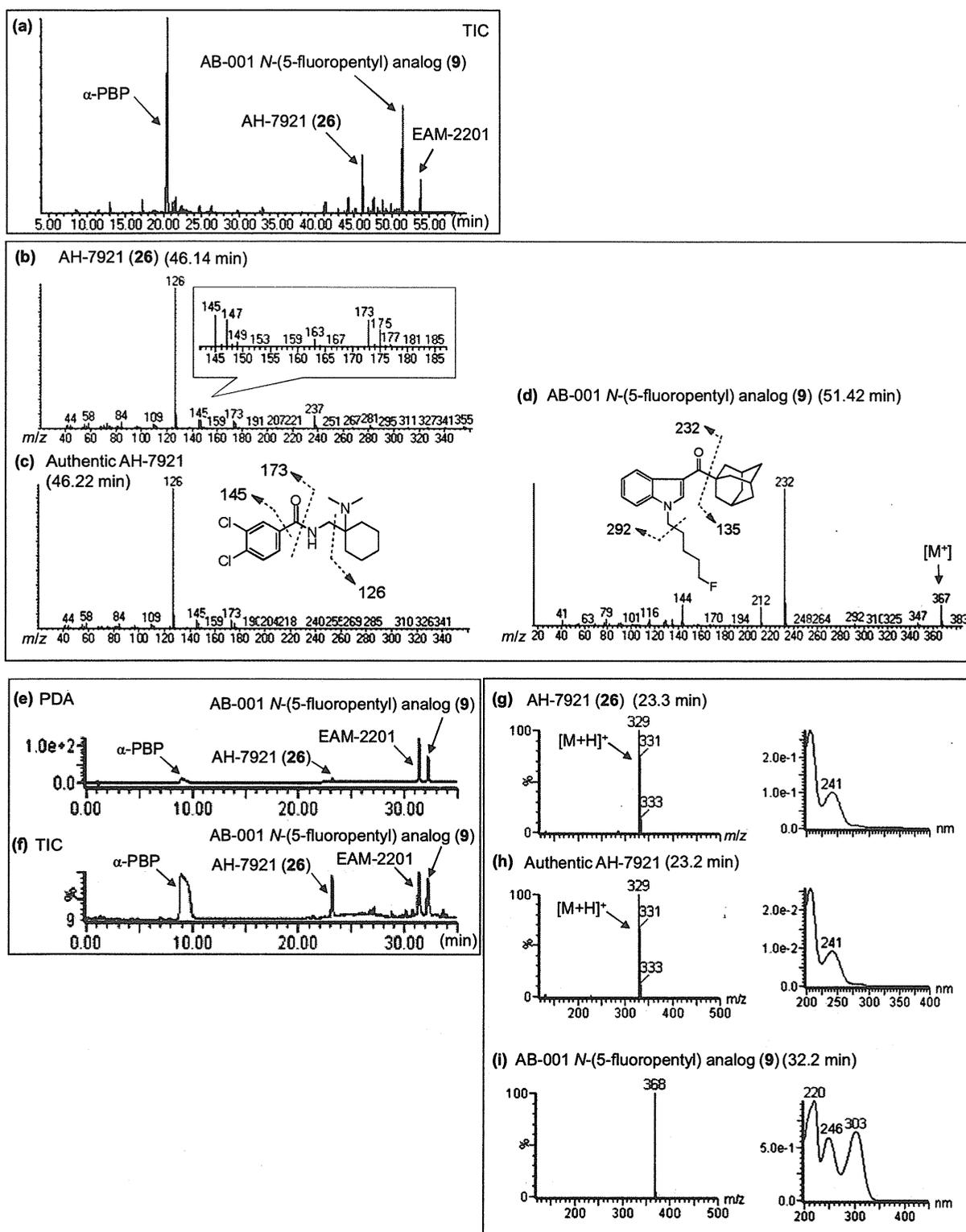


Fig. 13 GC-MS and LC-MS analyses of product F. TIC (a) and EI mass spectra of peaks 26 (b), 9 (d) and the authentic AH-7921 (c) obtained by GC-MS. LC-UV-PDA chromatogram (e) and TIC (f) using the elution program (2) obtained by LC-MS. UV and ESI mass spectra of peaks 26 (g), 9 (i) and the authentic AH-7921 (h).

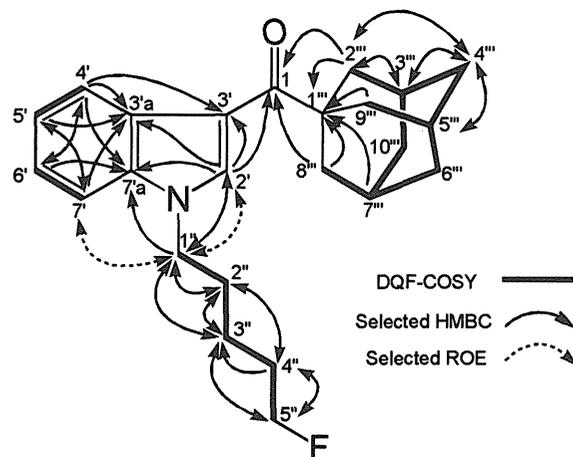


Fig. 14 DQF-COSY, HMBC selected and ROE correlations for AB-001 *N*-5-(fluoropentyl) analog (13).

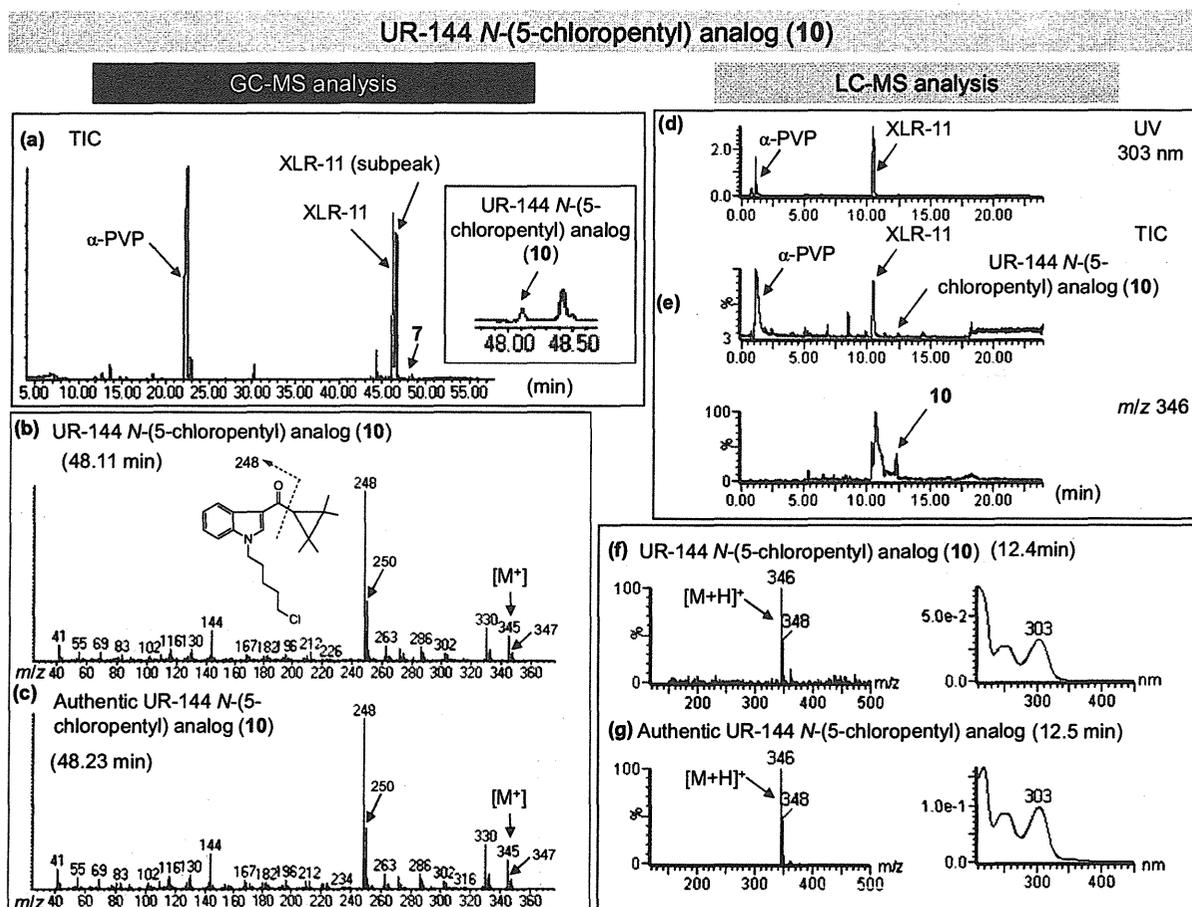


Fig. 15 GC-MS and LC-MS analyses of product G.

UR-144 *N*-(5-bromopentyl) analog (11)

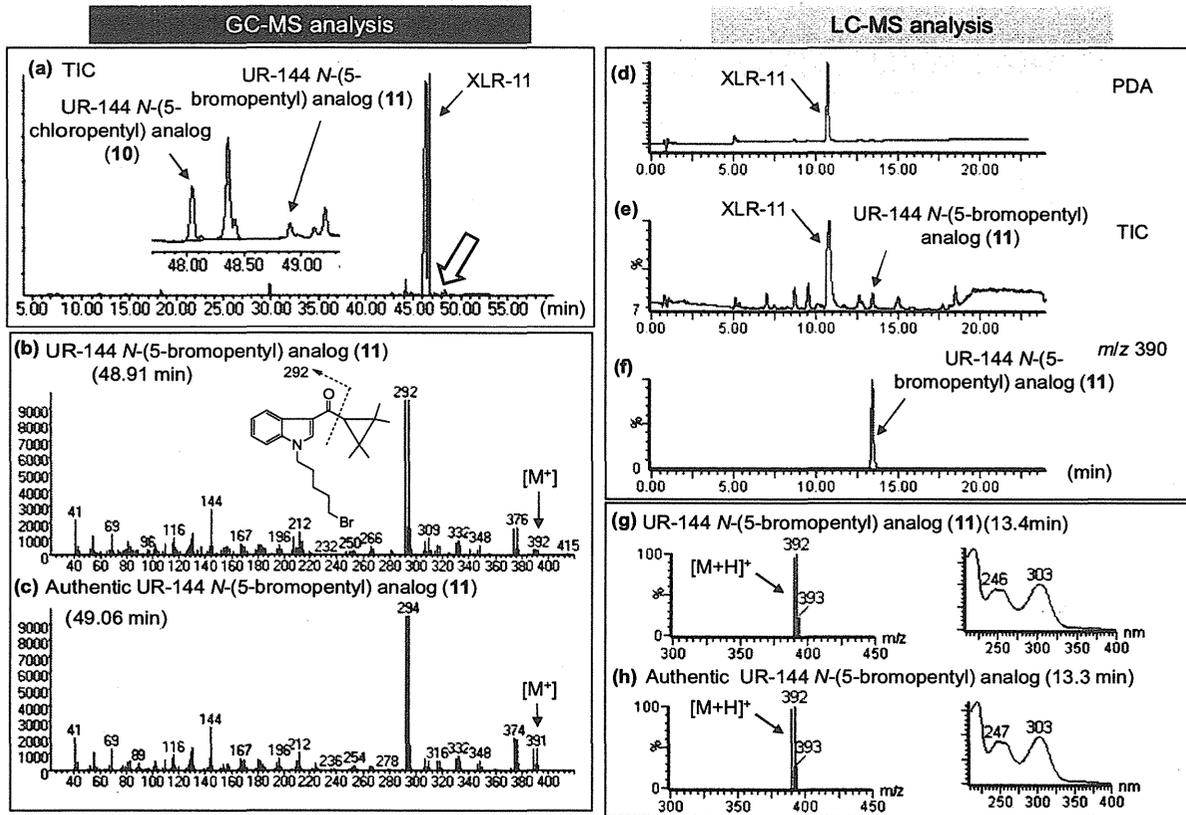


Fig. 16 GC-MS and LC-MS analyses of product H.

A-834735 (12)

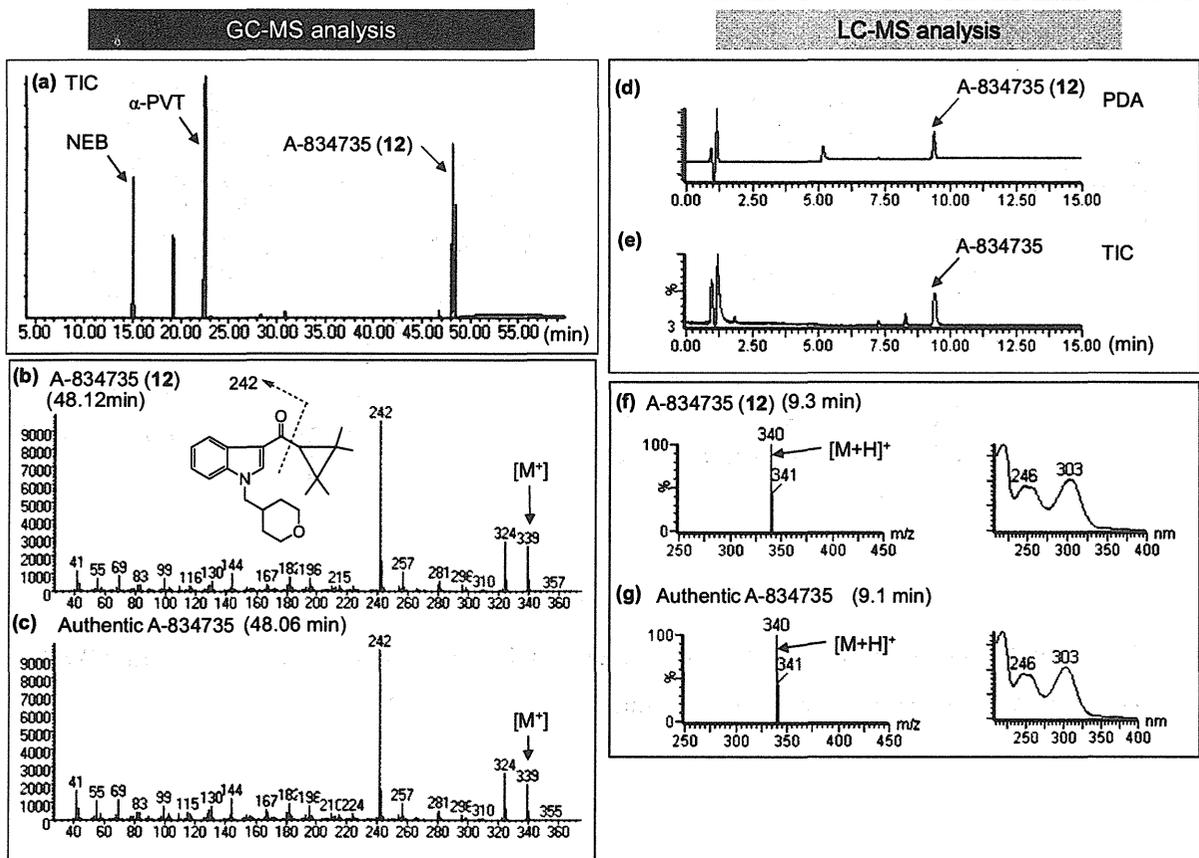


Fig. 17 GC-MS and LC-MS analyses of product I.

JWH-122 *N*-(5-chloropentyl) analog (13)

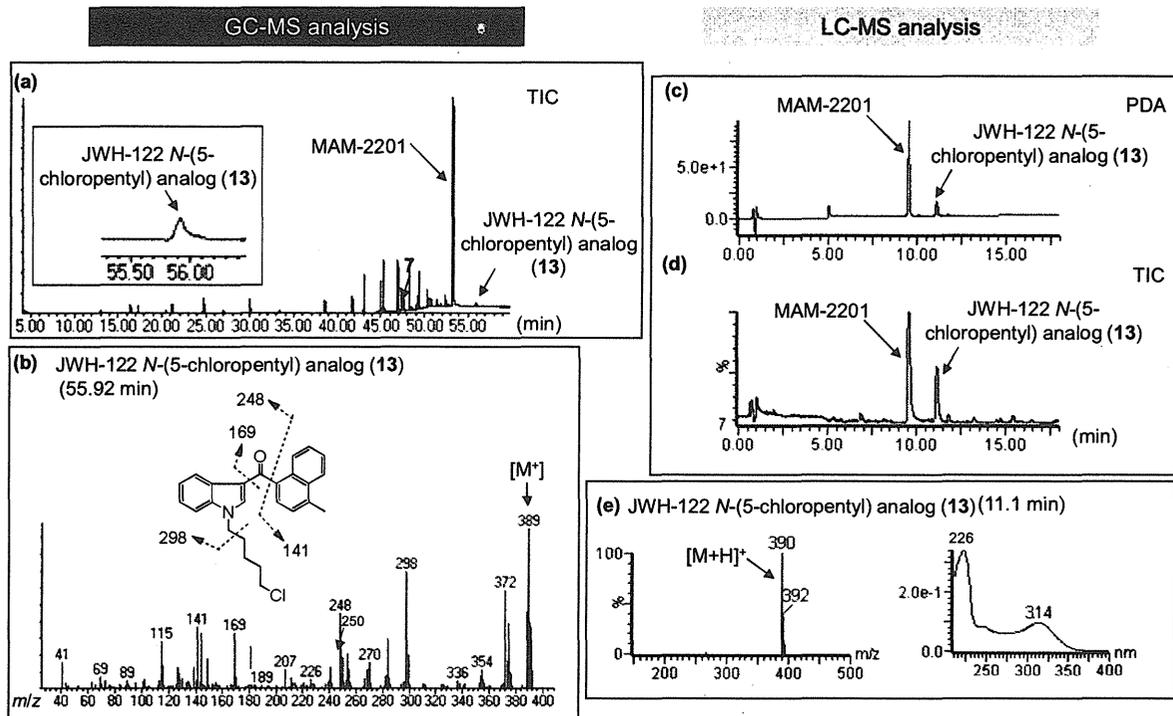


Fig. 18 GC-MS and LC-MS analyses of product J.

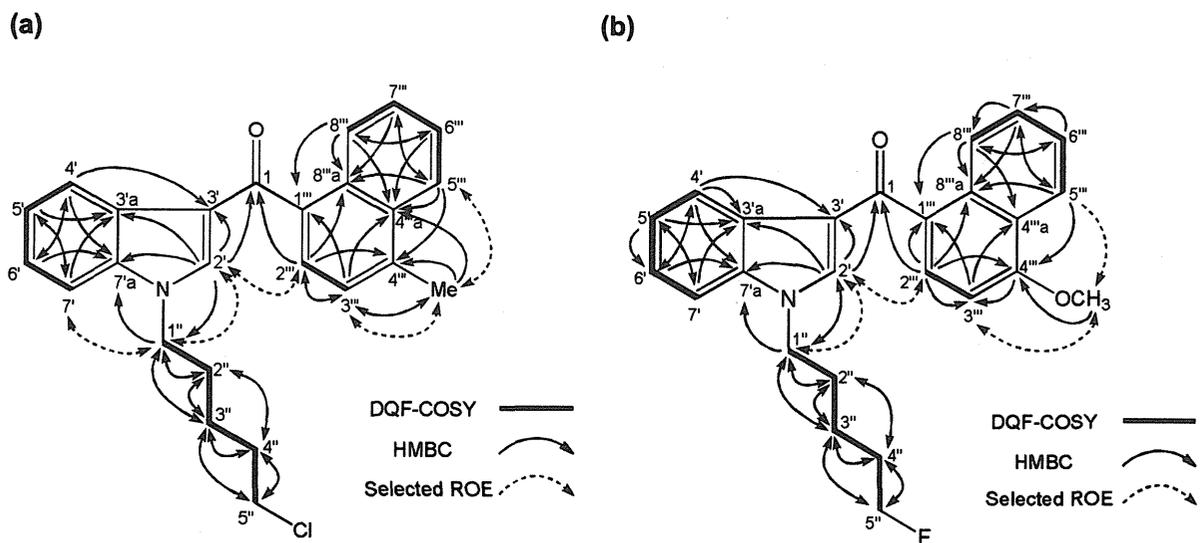


Fig. 19 DQF-COSY, selected HMBC and selected ROE correlations for compound 13 (JWH-122 *N*-(5-chloropentyl) analog, a) and compound 14 [4-methoxy (MeO)-AM-2201, b]

4-MeO-AM-2201 (14)

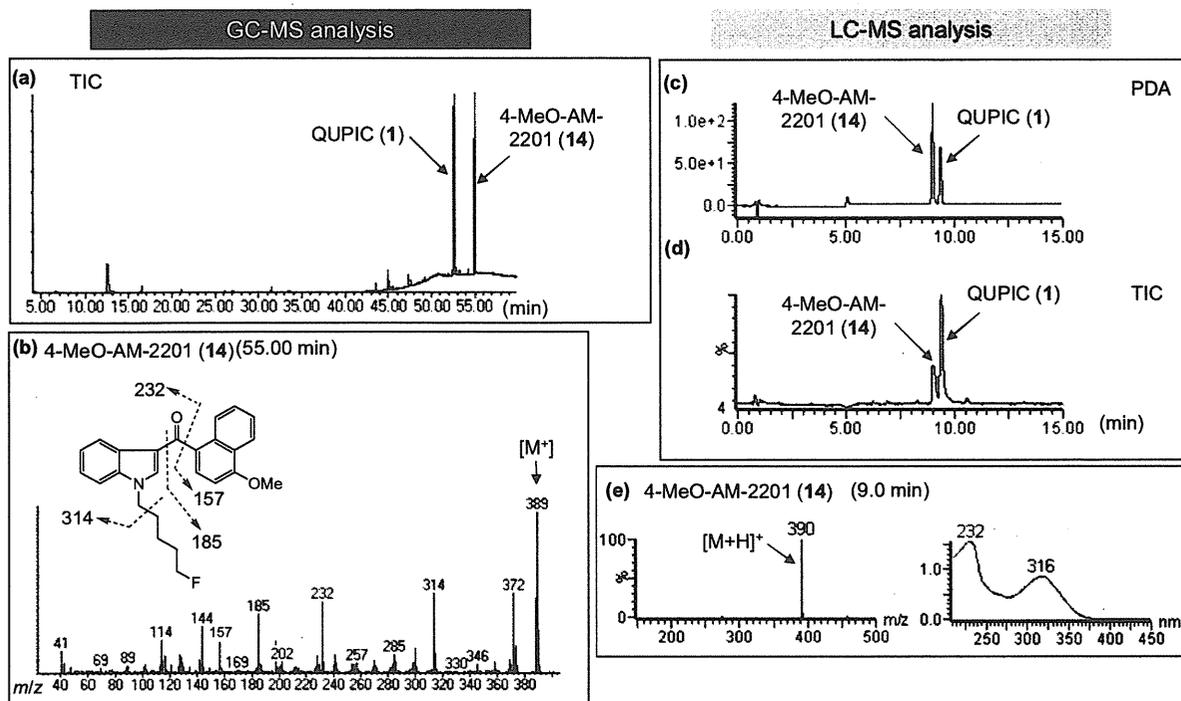


Fig. 20 GC-MS and LC-MS analyses of product K. TIC (a) and EI mass spectra of peak 14 (b) obtained by GC-MS analysis. LC-UV-PDA chromatogram (c) and TIC (d) using the elution program (1) obtained by LC-MS. UV and ESI mass spectra of peak 14 (e).

25B-NBOMe (15)

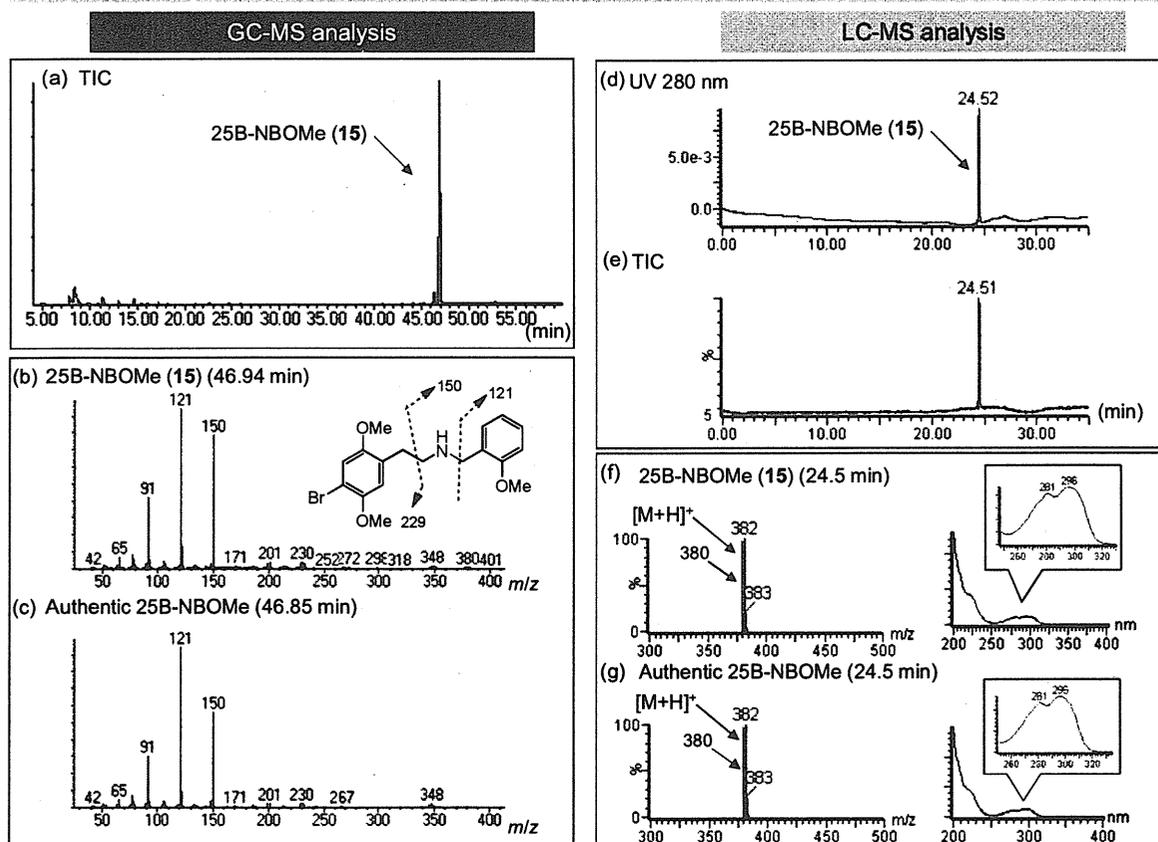


Fig. 21 GC-MS and LC-MS analyses of product L.

2-Methoxyamphetamine (16)

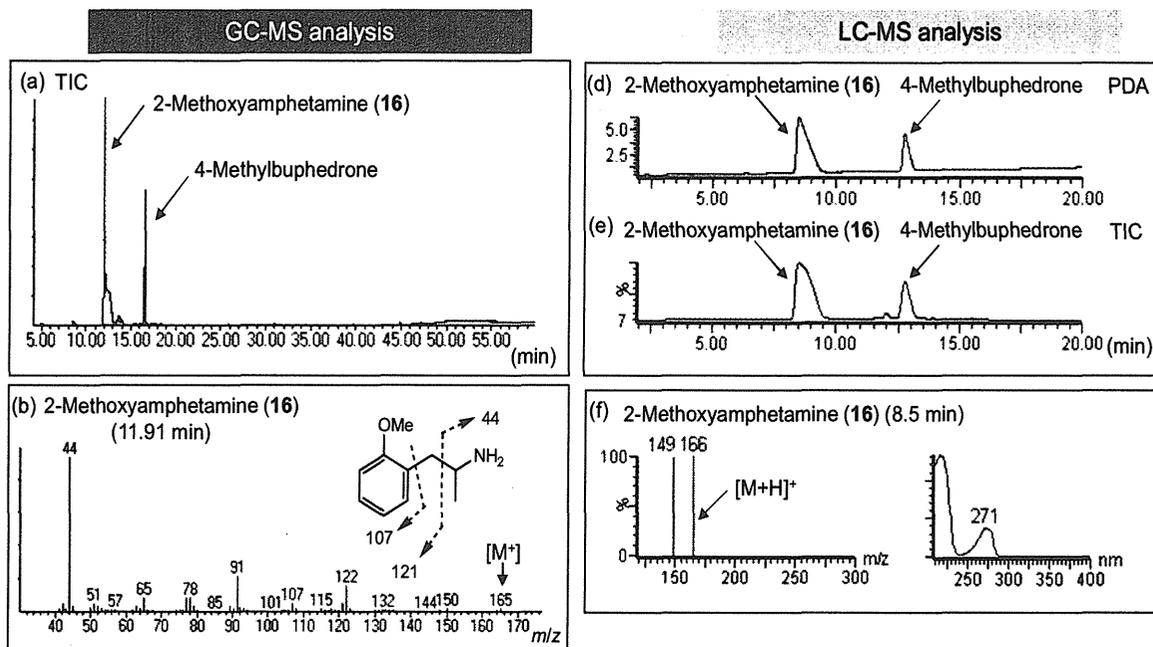


Fig. 22 GC-MS and LC-MS analyses of product M.

4-Methylbuphedrine (17)

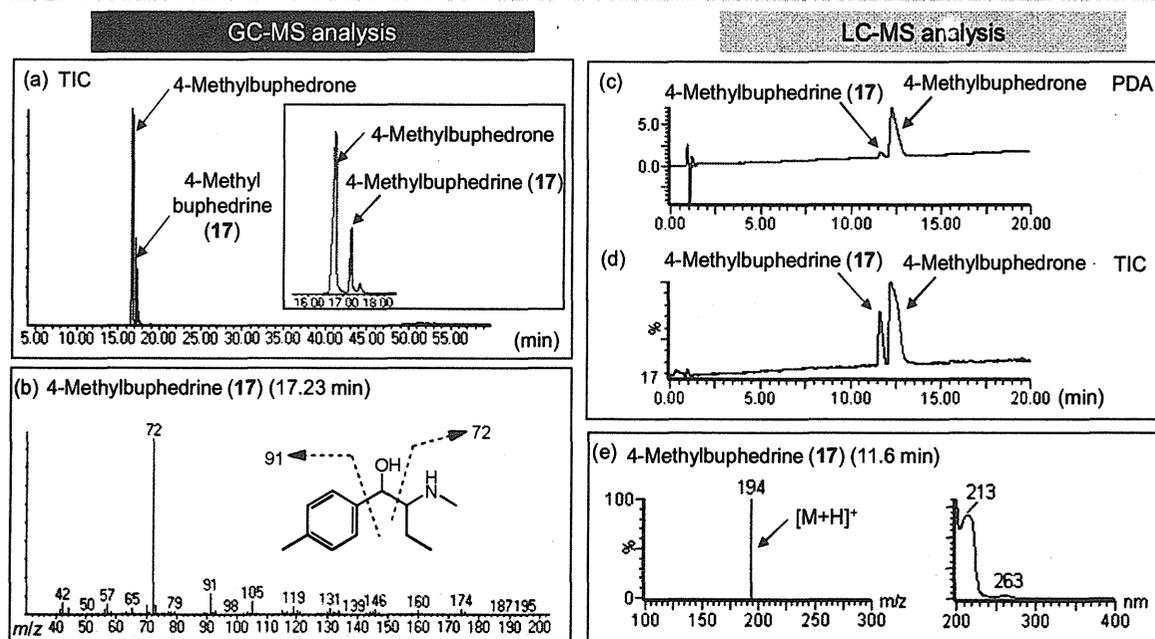


Fig. 23 GC-MS and LC-MS analyses of product N.

4-Methoxy- α -PVP (18)

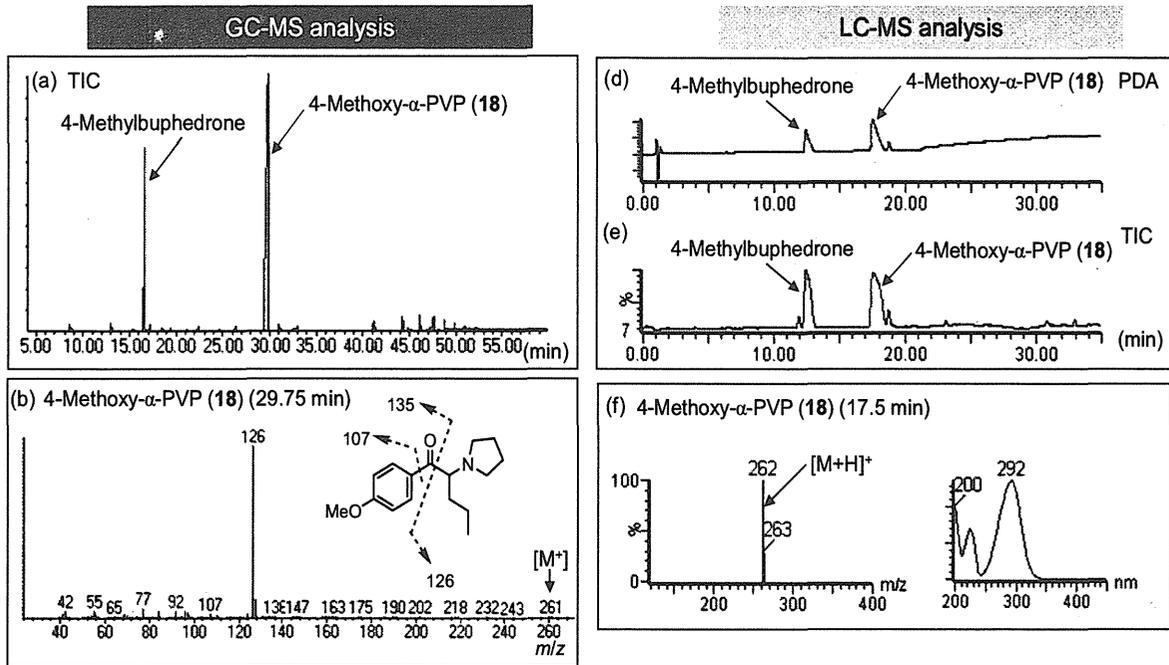


Fig. 24 GC-MS and LC-MS analyses of product O.

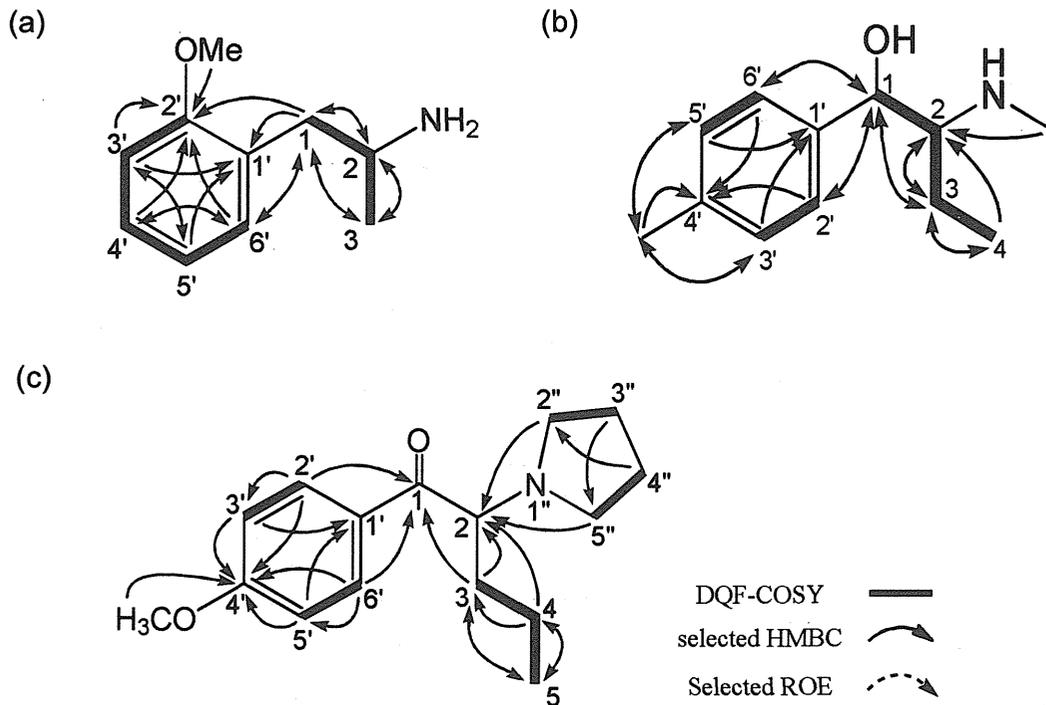


Fig. 25 DQF-COSY, HMBC correlations for 2-Methoxy-amphetamine (16, a) and 4-Methylbuphedrine (17, b) and 4-MeO- α -PVP (18, c).

MPBP (19)

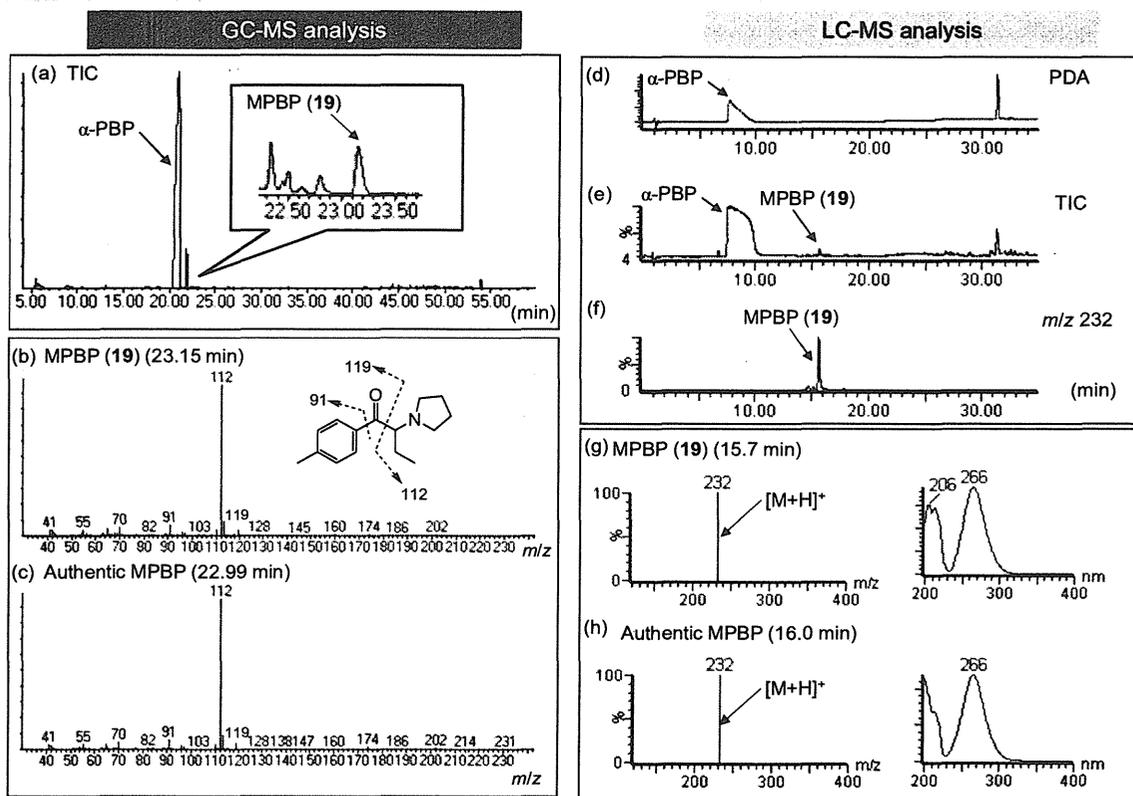


Fig. 26 GC-MS and LC-MS analyses of product P.

MPHP (20)

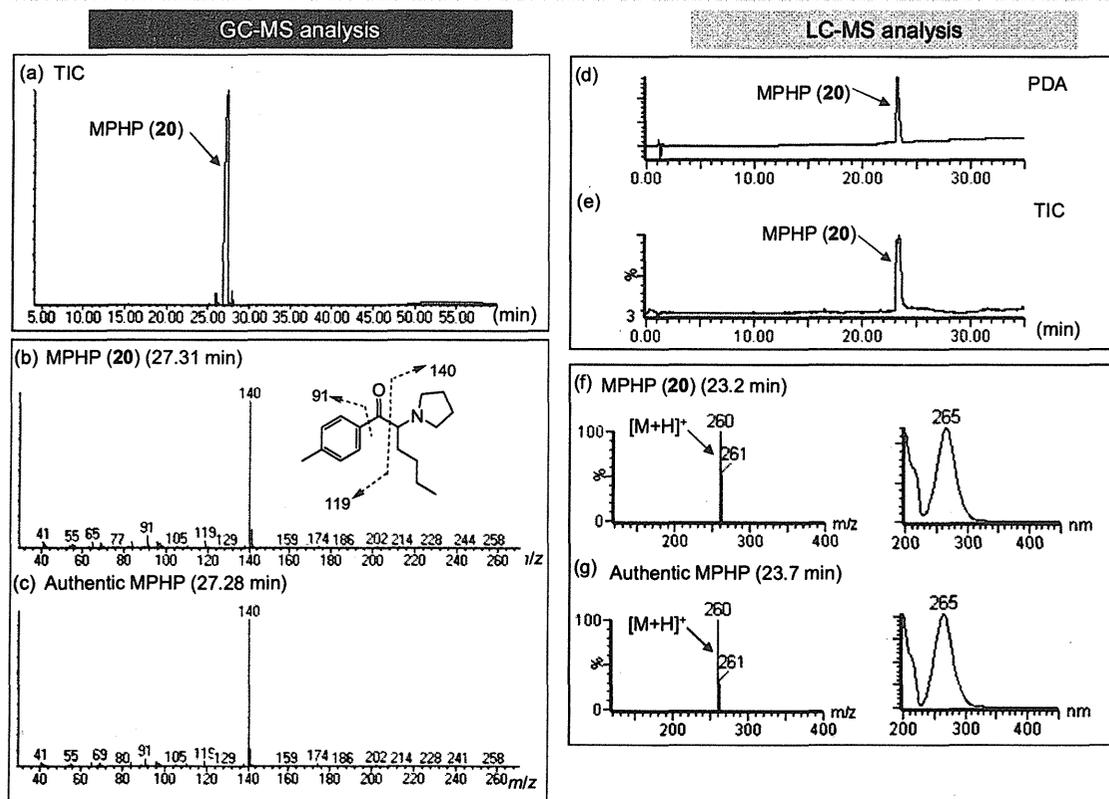


Fig. 27 GC-MS and LC-MS analyses of product Q.

MDPPP (21), MT-45 (27)

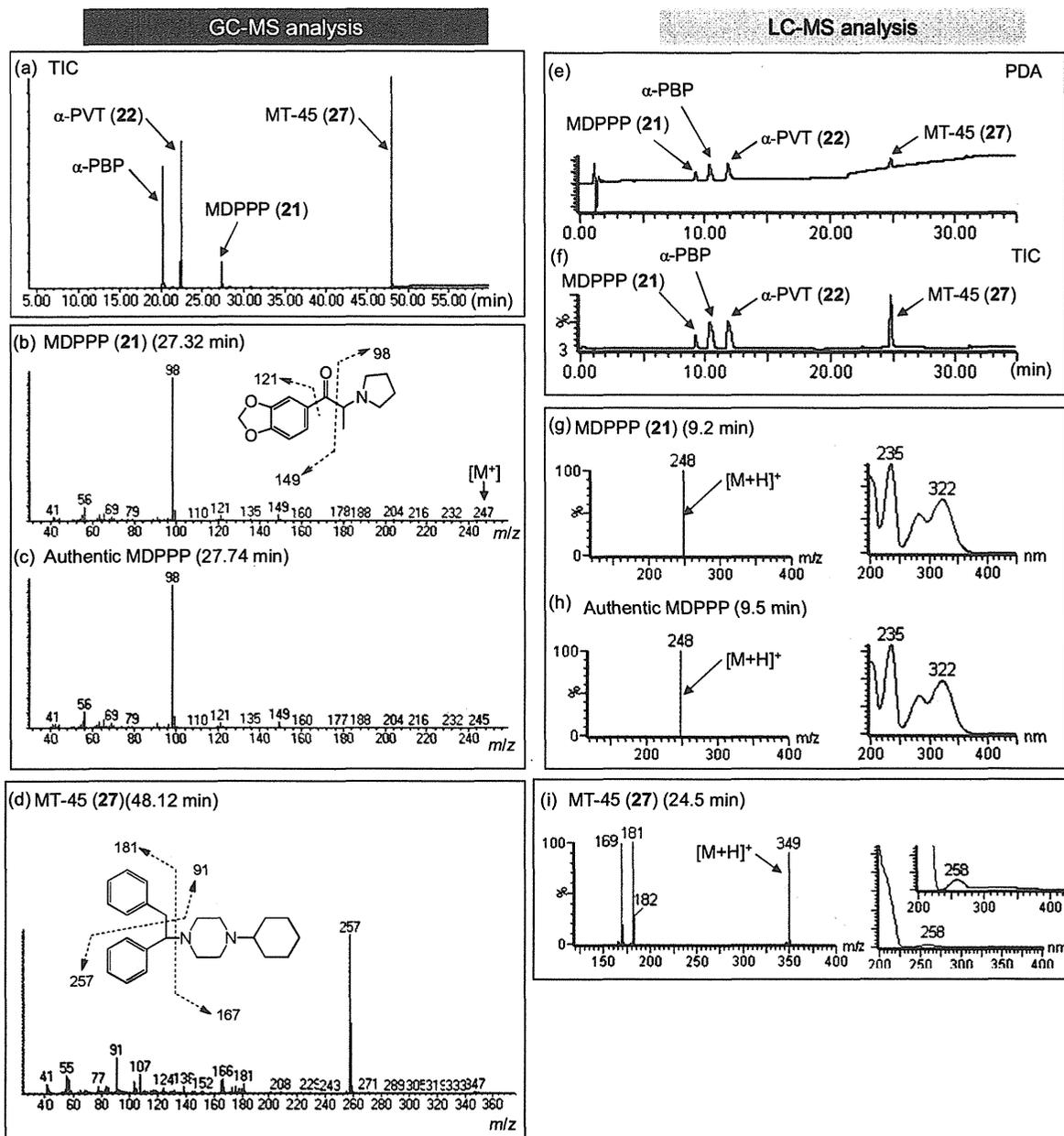


Fig. 28 GC-MS and LC-MS analyses of product R.

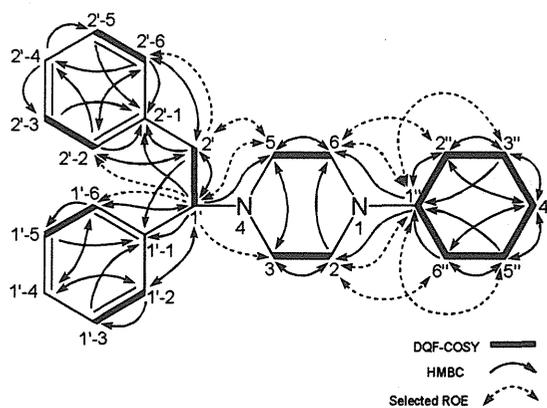


Fig. 29 DQF-COSY, HMBC correlations for MT-45(27).

α -PVT (22) and Bromo-substituted thiophene derivatives (23 – 25)

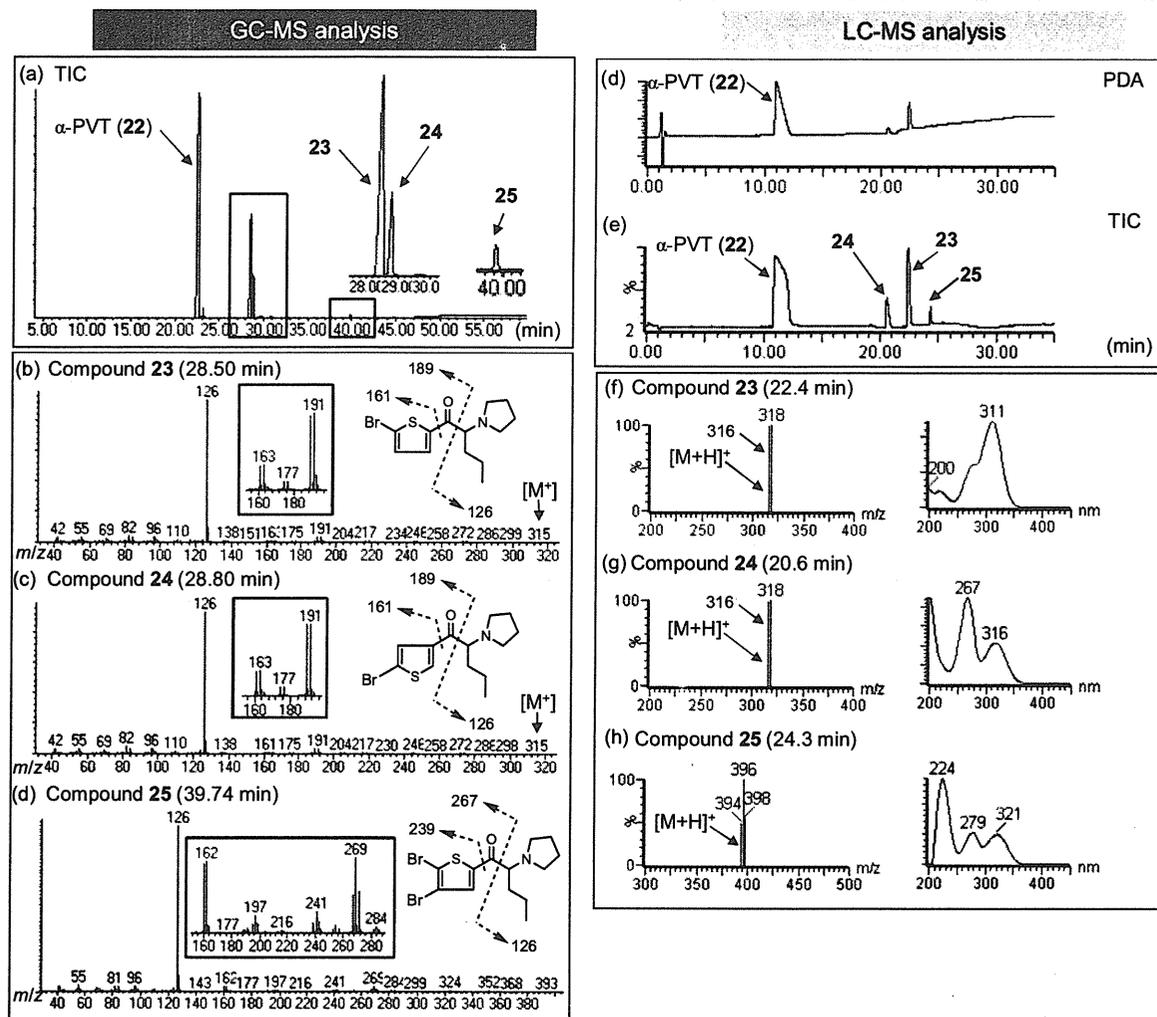


Fig.30 GC-MS and LC-MS analyses of product S.

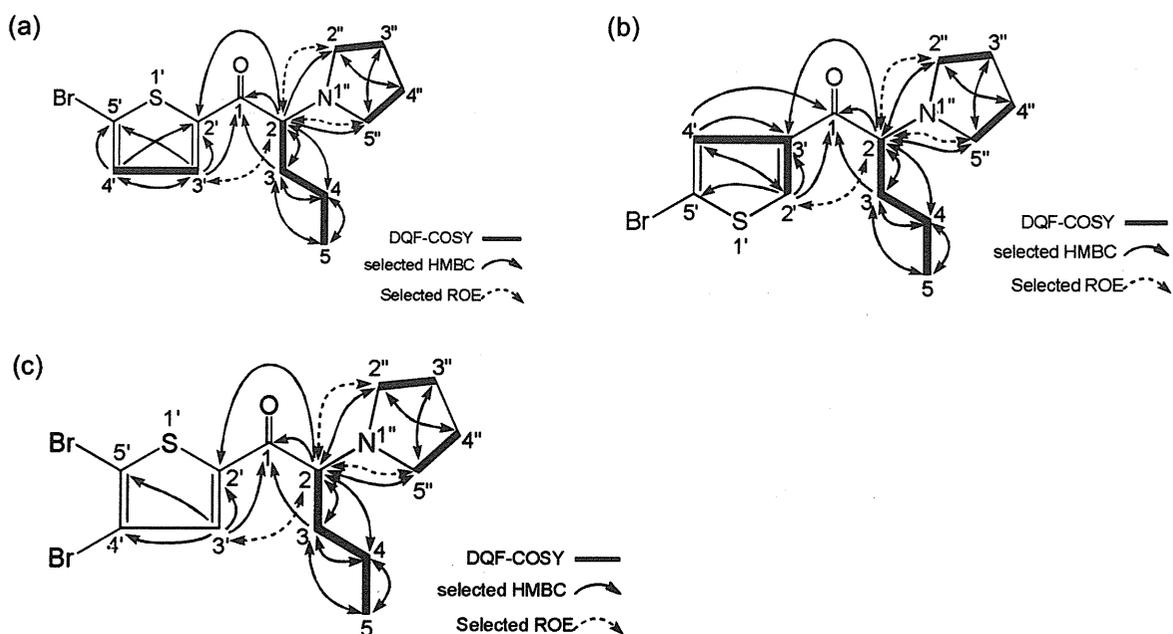


Fig. 31 DQF-COSY, HMBC correlations for compound 23(a) and compound 24 (b) and compound 25 (c)

Noopept (GVS-111, 28)

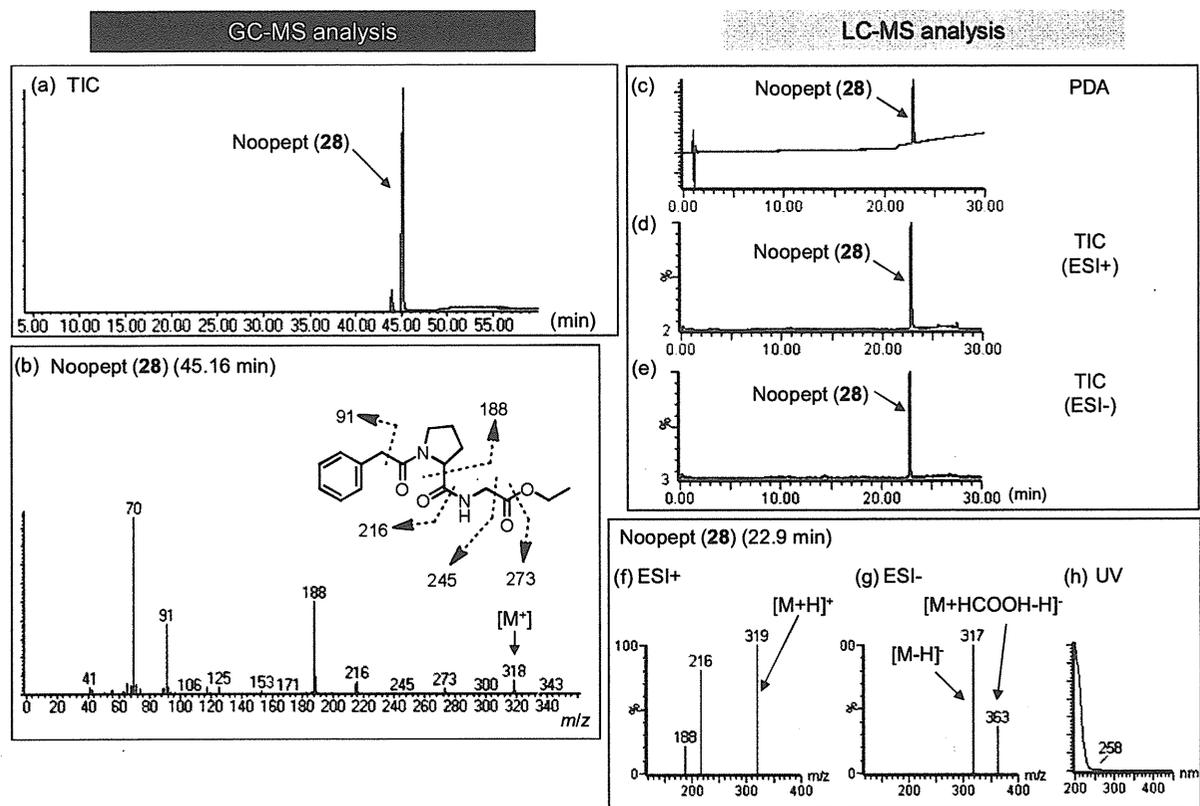


Fig. 32 GC-MS and LC-MS analyses of product T.

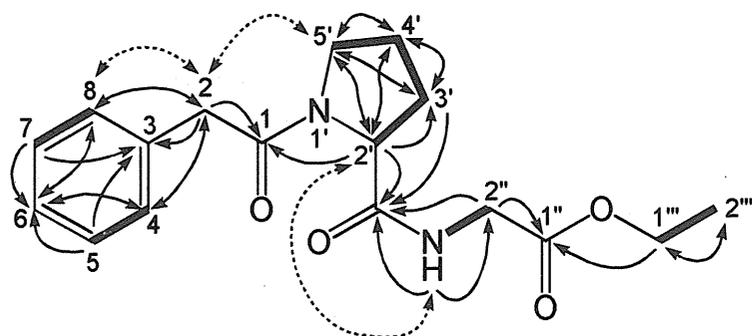
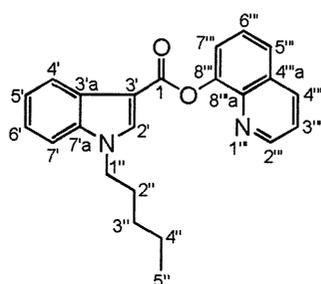
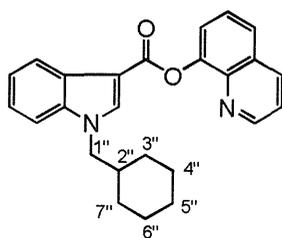


Fig. 33 DQF-COSY, HMBC selected and ROE correlations of compound 28.



QUPIC (1) (syn. PB-22)
C₂₃H₂₂N₂O₂: 358

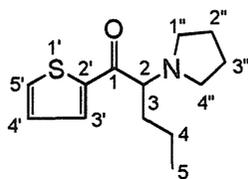


QUCHIC (2) (syn. BB-22)
C₂₅H₂₄N₂O₂: 384

Table 1 Nuclear magnetic resonance (NMR) data for QUPIC (1) and QUCHIC (2)^a

| No. | QUPIC (1) | | QUCHIC (2) | |
|---------|-----------------|---|-----------------|---|
| | ¹³ C | ¹ H | ¹³ C | ¹ H |
| 1 | 165.2 | – | 165.2 | – |
| 2' | 137.7 | 8.33, 1H, s | 138.2 | 8.28, 1H, s |
| 3' | 106.3 | – | 106.2 | – |
| 3'a | 128.6 | – | 128.5 | – |
| 4' | 122.4 | 8.13, 1H, brd, <i>J</i> =7.9 Hz | 122.3 | 8.13, 1H, d, <i>J</i> =7.9 Hz |
| 5' | 123.1 | 7.24, 1H, ddd, <i>J</i> =7.9, 6.9, 1.0 Hz | 123.0 | 7.23, 1H, td, <i>J</i> =7.9, 1.0 Hz |
| 6' | 124.0 | 7.31, 1H, ddd, <i>J</i> =7.9, 6.9, 1.0 Hz | 124.0 | 7.30, 1H, td, <i>J</i> =7.9, 1.0 Hz |
| 7' | 111.7 | 7.57, 1H, brd, <i>J</i> =7.9 Hz, overlapped | 111.9 | 7.55, 1H, d, <i>J</i> =7.9 Hz, overlapped |
| 7'a | 138.3 | – | 138.6 | – |
| 1'' | 48.0 | 4.33, 2H, t, <i>J</i> =7.2 Hz | 54.2 | 4.15, 2H, d, <i>J</i> =7.2 Hz |
| 2'' | 30.9 | 1.95, 2H, q, <i>J</i> =7.2 Hz | 39.9 | 1.97, 1H, m |
| 3'' | 30.1 | 1.37, 2H, m, overlapped | – | – |
| 4'' | 23.4 | 1.40, 2H, m, overlapped | – | – |
| 5'' | 14.3 | 0.92, 3H, t, <i>J</i> =7.2 Hz | 27.4 | 1.69, 2H, m, overlapped |
| 3''/7'' | – | – | 31.9 | 1.66, 2H, m, overlapped |
| | | | | 1.09, 2H, m |
| 4''/6'' | – | – | 26.8 | 1.28, 2H, m, overlapped |
| | | | | 1.75, 2H, m |
| 1''' | – | – | – | – |
| 2''' | 151.4 | 8.83, 1H, dd, <i>J</i> =4.1, 1.7 Hz | 151.4 | 8.83, 1H, dd, <i>J</i> =4.2, 1.7 Hz |
| 3''' | 123.1 | 7.56, 1H, m, overlapped | 123.1 | 7.52, 1H, dd, <i>J</i> =8.3, 4.2 Hz |
| 4''' | 138.1 | 8.41, 1H, dd, <i>J</i> =8.3, 1.4 Hz | 138.1 | 8.41, 1H, dd, <i>J</i> =8.3, 1.7 Hz |
| 4'''a | 131.2 | – | 131.2 | – |
| 5''' | 127.0 | 7.89, 1H, dd, <i>J</i> =7.6, 1.4 Hz | 127.0 | 7.89, 1H, brdd, <i>J</i> =7.6, 1.4 Hz |
| 6''' | 127.8 | 7.67, 1H, t, <i>J</i> =7.6 Hz | 127.8 | 7.66, 1H, t, <i>J</i> =7.6 Hz |
| 7''' | 123.7 | 7.63, 1H, dd, <i>J</i> =7.6, 1.4 Hz | 123.7 | 7.63, 1H, dd, <i>J</i> =7.6, 1.4 Hz |
| 8''' | 148.5 | – | 148.5 | – |
| 8'''a | 142.8 | – | 142.8 | – |

^a Recorded in CD₃OD at 600 MHz (¹H) and 150 MHz (¹³C), respectively; data in δ ppm (*J* in Hz)



α -Pyrrolidinovalerothiophenone

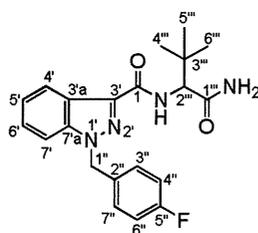
(α -PVT, **22**)

C₁₃H₁₉NOS: 237

Table 2 NMR data of α -PVT (**22**)^a

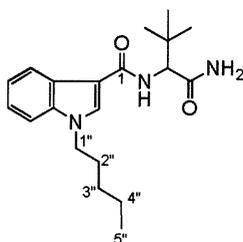
| No. | ¹³ C | ¹ H |
|-----|-----------------|--|
| 1 | 188.7 | – |
| 2 | 63.1 | 4.89, 1H, t, <i>J</i> =4.8 Hz |
| 3 | 32.9 | 2.31, 1H, m 2.03, 1H, m, overlapped |
| 4 | 19.8 | 1.47, 1H, m 1.30, 1H, m |
| 5 | 13.9 | 0.93, 3H, t, <i>J</i> =7.2 Hz |
| 1' | – | – |
| 2' | 143.4 | – |
| 3' | 134.5 | 7.88, 1H, d, <i>J</i> =4.8 Hz |
| 4' | 129.4 | 7.23, 1H, t, <i>J</i> =4.8 Hz |
| 5' | 137.7 | 7.85, 1H, d, <i>J</i> =4.8 Hz |
| 2'' | 52.9 | 3.77, 1H, m 2.82, 1H, m |
| 3'' | 23.9 | 2.15, 1H, m, overlapped 1.98, 1H, m, overlapped |
| 4'' | 23.6 | 2.18, 1H, m, overlapped 2.08, 1H, m, overlapped |
| 5'' | 48.6 | 3.89, 1H, m 3.63, 1H, m |

^a Recorded in CDCl₃ at 600 MHz (¹H) and 150 MHz (¹³C), respectively; data in δ ppm (*J* in Hz)



ADB-FUBINACA (5)

C₂₁H₂₃FN₄O₂: 382



ADBICA (6)

C₂₀H₂₉N₃O₂: 343

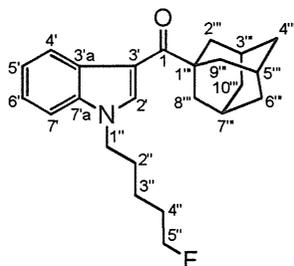
Table 3 NMR data for ADB-FUBINACA (5) and ADBICA (6)^a

| No. | AB-FUBINACA ^{b,d} | | ADB-FUBINACA (5) ^b | | ADBICA(6) ^c | |
|-------------------------|------------------------------|------------------------------|--|-----------------|-------------------------------|--|
| | ¹³ C | ¹³ C | ¹ H | ¹³ C | ¹ H | |
| 1 | 161.2 | 160.6 | – | 165.0 | – | |
| 2' | – | – | – | 131.5 | 7.70, 1H, s | |
| 3' | 137.1 | 136.9 | – | 110.3 | – | |
| 3'a | 122.3 | 122.2 | – | 125.4 | – | |
| 4' | 121.8 | 121.8 | 8.17, 1H, d, <i>J</i> =7.9 Hz | 120.2 | 7.99, 1H, m | |
| 5' | 122.8 | 122.8 | 7.29, 1H, t, <i>J</i> =7.9 Hz | 121.6 | 7.24, 1H, m, overlapped | |
| 6' | 127.0 | 127.1 | 7.46, 1H, brt, <i>J</i> =7.9 Hz | 122.5 | 7.26, 1H, m, overlapped | |
| 7' | 110.6 | 110.7 | 7.79, 1H, d, <i>J</i> =7.9 Hz | 110.3 | 7.36, 1H, m | |
| 7'a | 140.6 | 140.6 | – | 136.6 | – | |
| 1'' | 51.6 | 51.6 | 5.78, 2H, s | 46.9 | 4.10, 2H, t, <i>J</i> =7.2 Hz | |
| 2'' | 133.0, <i>J</i> =2.9 Hz | 133.0, <i>J</i> =2.9 Hz | – | 29.7 | 1.84, 2H, q, <i>J</i> =7.2 Hz | |
| 3''/7'' | 129.5, <i>J</i> =8.7 Hz | 129.5, <i>J</i> =8.7 Hz | 7.31, 2H, dd, <i>J</i> =7.2, 1.3 Hz, overlapped | – | – | |
| 4''/6'' | 115.5, <i>J</i> =21.7 Hz | 115.6, <i>J</i> =21.7 Hz | 7.16, 1H, brd, <i>J</i> =8.6 Hz, 7.15, 1H, brd, <i>J</i> =8.6 Hz | – | – | |
| 3'' | – | – | – | 29.0 | 1.29, 2H, m, overlapped | |
| 4'' | – | – | – | 22.3 | 1.32, 2H, m, overlapped | |
| 5'' | 161.6, d, <i>J</i> =242.8 Hz | 160.9, d, <i>J</i> =244.2 Hz | – | 13.9 | 0.87, 3H, t, <i>J</i> =6.9 Hz | |
| 1''' | 172.6 | 171.7 | – | 173.2 | – | |
| 2''' | 56.9 | 58.7 | 4.45, 1H, d, <i>J</i> =9.6 Hz | 59.7 | 4.68, 1H, d, <i>J</i> =9.3 Hz | |
| 3''' | 31.2 | 34.6 | – | 34.7 | – | |
| 4''' | 19.4 | – | – | – | – | |
| 5''' | 18.1 | – | – | – | – | |
| 4'''/5'''/6''' | – | 26.6 | 0.98, 9H, s | 26.8 | 1.13, 9H, s | |
| 1-CONH | – | – | 7.60, 1H, d, <i>J</i> =9.6 Hz | – | 6.7, 1H, d, <i>J</i> =8.9 Hz | |
| 1'''-CONH _{2a} | – | – | 7.27, 1H, brs, overlapped | – | 6.41, 1H, brs | |
| 1'''-CONH _{2b} | – | – | 7.72, 1H, brs | – | 5.63, 1H, brs | |

^a Recorded at 600 MHz (¹H) and 150 MHz (¹³C), respectively; data in δ ppm (*J* in Hz)

^b Recorded in dimethyl sulfoxide (DMSO)-*d*₆

^c Recorded in CDCl₃, ^d Ref. [9]



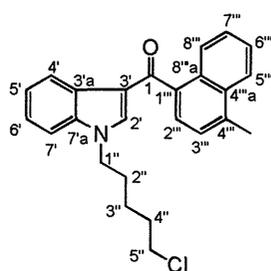
AB-001 *N*-5-(fluoropentyl) analog (**9**)

C₂₄H₃₀FNO: 367

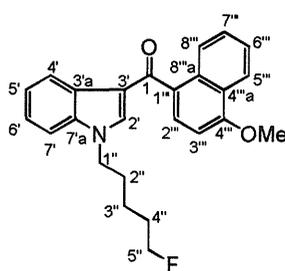
Table 4 NMR data of AB-001 *N*-(5-fluoropentyl) analog (**9**)

| No. | AB-001 ^{a, b)} | | AB-001 <i>N</i> -(5-fluoropentyl) analog (9) ^{d)} | |
|-----------------|-------------------------|-----------------------------|---|---------------------------------------|
| | ¹³ C | ¹³ C | ¹ H | HMBC ^{c)} |
| 1 | 202.4 | 202.0 | – | – |
| 2' | 133.2 | 133.0 | 7.90, 1H, s | 1, 3', 3'a, 7'a, 1'', |
| 3' | 113.0 | 112.9 | – | – |
| 3'a | 128.7 | 128.5 | – | – |
| 4' | 123.6 | 123.6 | 8.49, 1H, m | 3', 3'a, 5', 6', 7'a |
| 5' | 122.4 | 122.4 | 7.25, 1H, m, overlapped | 3'a, 7' |
| 6' | 123.1 | 123.1 | 7.26, 1H, m, overlapped | 4', 7', 7'a |
| 7' | 109.5 | 109.3 | 7.31, 1H, m | 3'a, 5', 6' |
| 7'a | 135.7 | 135.5 | – | – |
| 1'' | 47.1 | 46.9 | 4.18 2H, t, <i>J</i> =6.9 Hz | 2', 7'a, 2'', 3'' |
| 2'' | 29.8 | 29.6 | 1.93, 2H, quintet, <i>J</i> =7.3 Hz | 1'', 3'', 4'' |
| 3'' | 29.1 | 22.8 | 1.48, 2H, m | 2'', 4'', 5'' |
| 4'' | 22.3 | 29.9, d, <i>J</i> =19.3 Hz | 1.74, 1H, m | 2'', 3'', 5'' |
| | | | 1.70, 1H, m | |
| 5'' | 14.0 | 83.7, d, <i>J</i> =164.7 Hz | 4.45, 1H, t, <i>J</i> =6.0 Hz | 3'', 4'' |
| | | | 4.39, 1H, t, <i>J</i> =6.0 Hz | |
| 1''' | 47.0 | 46.9 | – | – |
| 2'''/8'''/9''' | 40.7 | 40.5 | 2.11, 6H, brs, overlapped | 1''', 3'''/5'''/7''', 4'''/6'''/10''' |
| 3'''/5'''/7''' | 28.7 | 28.5 | 2.09, 3H, m, overlapped | 1''' |
| 4'''/6'''/10''' | 37.1 | 36.9 | 1.80, 6H, brs | 3'''/5'''/7''' |
| | | | | 3'''/5'''/7''', 2'''/8'''/9''' |
| NH | – | – | – | – |

^a Recorded in CDCl₃ at 600MHz (¹H) and 150MHz (¹³C), respectively; data in δ ppm (*J* in Hz). ^b Jankovics P. et al., Forensic Sci. Int. 214(2012)27-32. ^c *J*=8 or 4 Hz, The proton signal correlated with the indicated carbons.



JWH-122 *N*-(5-chloropentyl)
analog (**13**)
C₂₅H₂₄ClNO: 389



4-MeO-AM-2201 (**14**)
C₂₅H₂₄FNO₂: 389

Table 5 NMR data for JWH-122 *N*-(5-chloropentyl) analog (**13**) and 4-MeO-AM-2201 (**14**)^a

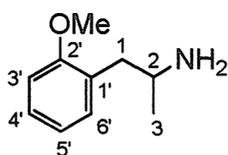
| No. | JWH-122 ^b | JWH-122 <i>N</i> -(5-chloropentyl) analog (13) ^c | | 4-MeO-AM-2201 (14) ^d | |
|----------|----------------------|---|---------------------------------|--|---|
| | ¹³ C | ¹³ C | ¹ H | ¹³ C | ¹ H |
| 1 | 192.2 | 191.5 | – | 190.5 | – |
| 2' | 137.8 | 137.1 | 6.96, 1H, s, overlapped | 138.9 | 7.83, 1H, s |
| 3' | 117.7 | 118.6 | – | 116.2 | – |
| 3'a | 127.0 | 127.0 | – | 126.6 | – |
| 4' | 122.9 | 123.9 | 9.13, 1H, d, <i>J</i> =7.9 Hz | 121.7 | 8.30, 1H, d, <i>J</i> =7.6 Hz |
| 5' | 122.7 | 123.2 | 7.34, 1H, t, <i>J</i> =7.9 Hz | 122.3 | 7.29, 1H, t, <i>J</i> =7.6 Hz, overlapped |
| 6' | 123.5 | 123.7 | 7.22, 1H, brt, <i>J</i> =7.9 Hz | 123.2 | 7.32, 1H, t, <i>J</i> =7.6 Hz, overlapped |
| 7' | 109.9 | 109.9 | 6.95, 1H, d, <i>J</i> =7.9 Hz | 110.9 | 7.64, 1H, d, <i>J</i> =7.6 Hz |
| 7'a | 137.0 | 137.2 | – | 136.7 | – |
| 1'' | 47.1 | 46.2 | 3.05, 2H, t, <i>J</i> =7.2 Hz | 46.0 | 4.23, 2H, t, <i>J</i> =6.9 Hz |
| 2'' | 29.5 | 28.8 | 0.93, 2H, m | 29.0 | 1.76, 2H, quintet, <i>J</i> =6.2 Hz |
| 3'' | 28.9 | 23.9 | 0.71, 2H, m | 21.9, d, <i>J</i> =5.8 Hz | 1.28, 2H, quintet, <i>J</i> =6.2 Hz |
| 4'' | 22.2 | 31.9 | 1.04, 2H, m | 29.3, d, <i>J</i> =18.8 Hz | 1.63, 1H, quintet, <i>J</i> =6.2 Hz, 1.58, 1H, quintet, <i>J</i> =6.2 Hz |
| 5'' | 13.9 | 44.3 | 2.85, 2H, t, <i>J</i> =6.5 Hz | 83.6, d, <i>J</i> =161.8 Hz | 4.40, 1H, t, <i>J</i> =6.2 Hz, 4.32, 1H, t, <i>J</i> =6.2 Hz |
| 1''' | 137.5 | 138.9 | – | 125.0 | – |
| 2''' | 125.8 | 125.8 | 7.54, 1H, d, <i>J</i> =6.9 Hz | 127.9 | 7.70, 1H, d, <i>J</i> =7.9 Hz |
| 3''' | 125.2 | 125.6 | 7.09, 1H, d, <i>J</i> =6.9 Hz | 103.1 | 7.06, 1H, d, <i>J</i> =7.9 Hz |
| 4''' | 136.6 | 136.3 | – | 156.1 | – |
| 4'''a | 132.8 | 133.3 | – | 130.8 | – |
| 5''' | 124.2 | 124.5 | 7.83, 1H, brd, <i>J</i> =8.4 Hz | 121.7 | 8.25, 1H, m |
| 6''' | 126.1 | 126.4 | 7.29, 1H, m | 125.7 | 7.55, 1H, m, overlapped |
| 7''' | 126.4 | 126.7 | 7.26, 1H, m | 127.2 | 7.54, 1H, m, overlapped |
| 8''' | 126.6 | 127.4 | 8.61, 1H, brd, <i>J</i> =7.6 Hz | 125.4 | 8.12, 1H, m |
| 8'''a | 130.9 | 131.7 | – | 131.5 | – |
| 4'''-Me | 19.8 | 19.6 | 2.42, 3H, s | – | – |
| 4'''-OMe | – | – | – | 55.9 | 4.06, 3H, s |

^a Recorded at 600MHz (¹H) and 150MHz (¹³C), respectively; data in δ ppm (*J* in Hz)

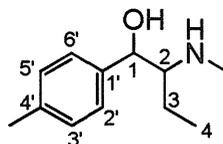
^b Recorded in CDCl₃ at 150MHz (¹³C)

^c Recorded in benzene-*d*₆

^d Recorded in DMSO-*d*₆



2-Methoxyamphetamine (**16**)
C₁₀H₁₅NO: 165



4-Methylbuphedrine (**17**)
C₁₂H₁₉NO: 193

Table 6 NMR data of 2-Methoxy-amphetamine(**16**)^a

| 2-Methoxy-amphetamine(16) in DMSO- <i>d</i> ₆ | | | |
|---|-----------------|--------------------------------------|-------------------|
| No. | ¹³ C | ¹ H | HMBC ^b |
| 1 | 34.9 | 2.95, 1H, dd, <i>J</i> =13.1, 5.2 Hz | 2, 3, 1', 2', 6' |
| | | 2.68, 1H, dd, <i>J</i> =13.1, 8.9 Hz | 2, 3, 1', 2', 6' |
| 2 | 46.7 | 3.38, 1H, m | 1, 3 |
| 3 | 17.8 | 1.07, 3H, d, <i>J</i> =6.5 Hz | 1, 2 |
| 1' | 124.4 | – | – |
| 2' | 157.3 | – | – |
| 3' | 110.9 | 6.99, 1H, d, <i>J</i> =8.3 Hz | 1', 2', 5' |
| 4' | 128.4 | 7.25, 1H, td, <i>J</i> =8.1, 1.7 Hz | 2', 6' |
| 5' | 120.4 | 6.90, 1H, t, <i>J</i> =7.2 Hz | 1', 3' |
| 6' | 130.9 | 7.15, 1H, dd, <i>J</i> =7.6 Hz | 1, 2', 4' |
| 2-NH ₂ | – | 8.06, 2H, brs | – |
| 2'-OCH ₃ | 55.3 | 3.78, 3H, s | 2' |

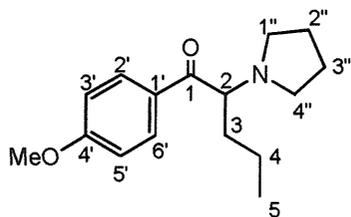
^a Recorded at 800 MHz (¹H) and 200 MHz (¹³C), respectively; data in δ ppm (*J* in Hz).

^b *J*=8 Hz, The proton signal correlated with the indicated carbons.

Table 7 NMR data of 4-methylbuphedrine (**17**)^a

| 4-methylbuphedrine (17) in CD ₃ OD | | | |
|--|-----------------|-------------------------------|-------------------|
| No. | ¹³ C | ¹ H | HMBC ^b |
| 1 | 70.8 | 5.11, 1H, d, <i>J</i> =3.1 Hz | 3, 2'/6' |
| 2 | 67.1 | 3.21, 1H, m | 3 |
| 3 | 19.9 | 1.55, 2H, m | 1, 2, 4 |
| 4 | 11.0 | 0.86, 3H, t, <i>J</i> =7.6 Hz | 2, 3 |
| 1' | 138.0 | – | – |
| 2'/6' | 127.1 | 7.31, 2H, d, <i>J</i> =7.9 Hz | 1, 4' |
| 3'/5' | 130.2 | 7.21, 2H, d, <i>J</i> =7.9 Hz | 1', 4'-Me |
| 4' | 139.0 | – | – |
| <i>N</i> -Me | 32.0 | 2.78, 3H, s | 2 |
| 4'-Me | 21.1 | 2.33, 3H, s | 3'/5', 4' |

^a Recorded at 600 MHz (¹H) and 150 MHz (¹³C), respectively; data in δ ppm (*J* in Hz). ^b *J*=8 and 4 Hz. The proton signal correlated with the indicated carbons.



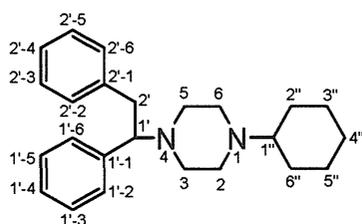
4-Methoxy- α -pyrrolidinovalerophenone
(4-MeO- α -PVP)

C₁₆H₂₃NO₂; 261

Table 8 NMR data of 4-MeO- α -PVP(18)^a

| No. | 4-MeO- α -PVP in pyridine- <i>d</i> ₅ | | HMBC ^b |
|---------|---|-------------------------------|-------------------|
| | ¹³ C | ¹ H | |
| 1 | 197.2 | – | – |
| 2 | 66.7 | 4.88, 1H, brs | ?? |
| 3 | 32.6 | 2.05, 2H, m | 1, 2, 5 |
| 4 | 19.8 | 1.36, 2H, m | 2, 3, 5 |
| 5 | 14.2 | 0.77, 3H, t, <i>J</i> =7.4 Hz | 3, 4 |
| 1' | 129.9 | – | – |
| 2'/6' | 131.7 | 8.35, 2H, d, <i>J</i> =8.7 Hz | 1, 4', 3'/5' |
| 3'/5' | 114.6 | 7.06, 2H, d, <i>J</i> =8.7 Hz | 1', 4' |
| 4' | 164.6 | – | – |
| 2''/5'' | 51.3 | 3.23, 2H, brs | 2 |
| | | 3.03, 2H, brs | 2 |
| 3''/4'' | 23.9 | 1.77, 4H, m | 2''/5'' |
| 4'-MeO | 55.6 | 3.72, 3H, s | 4' |

^a Recorded at 800 MHz (¹H) and 200 MHz (¹³C), respectively; data in δ ppm (*J* in Hz). ^b *J*=8 Hz, The proton signal correlated with the indicated carbons.



MT-45 (IC-6, 27)
 [1-cyclohexyl-4-(1,2-
 diphenylethyl)piperazine]
 $C_{24}H_{32}N_2$: 348

Table 9 NMR data for MT-45 (1-cyclohexyl-4-(1,2-diphenylethyl)piperazine)

| No. | MT-45 in $CDCl_3^a$ | |
|-----------|---------------------|--|
| | ^{13}C | 1H |
| 2 | 44.5 | 4.14, 1H, tdd, $J=12.7, 9.6, 3.0$ Hz 3.38, 1H, brd, $J=12.7$ Hz |
| 3 | 47.2 | 3.95, 1H, tdd, $J=12.7, 9.6, 3.0$ Hz 3.22, 1H, brd, $J=12.7$ Hz |
| 5 | 48.1 | 4.22, 1H, m, overlapped 4.04, 1H, brd, $J=12.7$ Hz |
| 6 | 44.5 | 4.49, 1H, tdd, $J=12.7, 9.6, 3.0$ Hz 3.55, 1H, brd, $J=12.7$ Hz |
| 1' | 74.4 | 4.20, 1H, m |
| 2' | 37.4 | 3.77, 1H, dd, $J=13.4, 4.5$ Hz 3.51, 1H, dd, $J=13.4, 10.7$ Hz |
| 1'-1 | 131.3 | – |
| 1'-2/1'-6 | 129.4 | 7.50, 2H, brs |
| 1'-3/1'-5 | 129.8 | 7.36, 2H, m, overlapped |
| 1'-4 | 130.5 | 7.36, 1H, m, overlapped |
| 2'-1 | 134.5 | – |
| 2'-2/2'-6 | 129.1 | 6.92, 2H, dd, $J=7.6, 2.4$ Hz |
| 2'-3/2'-5 | 128.8 | 7.13, 2H, m, overlapped |
| 2'-4 | 127.4 | 7.14, 1H, m, overlapped |
| 1'' | 64.5 | 3.11, 1H, tq, $J=12.4, 3.4$ Hz |
| 2''/6'' | 26.7 | 2.26, 2H, brd, $J=12.4$ Hz 1.52, 2H, m |
| 3''/5'' | 24.8 | 1.94, 2H, brd, $J=12.4$ Hz 1.29, 2H, brq, $J=12.4$ Hz |
| 4'' | 24.7 | 1.70, 1H, brd, $J=12.4$ Hz 1.14, 1H, qt, $J=12.4, 3.4$ Hz |

^a Recorded at 600MHz (1H) and 150MHz (^{13}C), respectively; data in δ ppm (J in Hz).