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Supporting Information

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Asymmetric Brønsted Acid Catalysis: First Enantioselective Nucleophilic Substitutions and 1,4-Additions

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General: Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator and visualised by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.040-0.063 mm). Solvent mixtures are understood as volume/volume.
^{1}H-NMR and ^{13}C-NMR were recorded on a Bruker AM 250, a Bruker AV 300 or a Bruker AV 400 spectrometer in $CDCl_3$. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br s (broadened singlet), s (singlet), d (doublet), t (triplet), m (multiplet)); coupling constants (*J*) are in Hertz (Hz). Mass spectra (MS-EI, 70 eV) were conducted on GC-MS Shimadzu QP2010 (column: Equity®-5, length × I.D. 30 m × 0.25 mm, d_f 0.25 µm, lot # 28089-U, Supelco). IR spectra were recorded on a Jasco FT/IR-420 spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Optical rotations were measured on a Perkin Elmer 241 polarimeter. The enantiomeric excesses were determined by HPLC analysis using a chiral stationary phase column (column: CHIRALCEL OD-H or CHIRALPAK AS-H or CHIRALPAK AD-H; eluent: hexane/2-propanol). The HPLC spectra of enantio-enriched compounds were calibrated with the corresponding racemic mixtures. Chemical yields refer to pure isolated substances. The yields and enantiomeric excesses are given in the corresponding tables.

Preperation of methyl 4-(1-methyl-1H-indol-3-yl)-2-oxo-4-phenylbutanoate - General Procedure for the 1,4-addition of N-Methylindole to (E)-methyl 2-oxo-4-phenylbut-3-enoate[1,2] : In a typical experiment (E)-methyl 2-oxo-4-phenylbut-3-enoate (0.07 mmol) and Catalyst **6a** (0.0035 mmol) were dissolved in 1 mL of dry dichloromethane and cooled down to -78°C. Now 1-Methylindol was added (1.5 mmol) and the yellow solution was stirred for 15h at -78 °C. After the reaction was completed the mixture was purified by column chromatography (hexane / ethylacetate 5:1) to afford 19 mg (62%) of a white solid.
^{1}H-NMR (250 MHz, $CDCl_3$): 7.42-7.30 (m, 1H), 7.30-7.15 (m, 5H), 7.15-7.06 (m, 2H), 6.99-6.90 (m, 1H), 6.80 (s, 1H), 4.84 (t, *J* = 7.5 Hz, 1H), 3.69 (s, 3H), 3.66 (s, 3H), 3.62-3.45 (m, 2H); ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 192.7, 161.4, 143.4, 137.4, 128.6, 127.8, 126.9, 126.6, 126.4, 121.9, 119.5, 119.1, 116.9, 109.3, 52.9, 45.8, 37.7, 32.8; IR (KBr): $\tilde{\nu}$ = 3448, 3054, 2952, 1731, 1488, 1255, 1089, 744 cm^{-1}; MS-EI: *m/z*(%): 321.1 (19) $[M]^{+\bullet}$, 322.1 (4) $[M]^{+\bullet}$, 220.1 (100) $[M-C_4H_5O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+20,8 (c=0.5 in $CHCl_3$) for S-Enantiomer; HPLC conditions: AD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=29.3 min; minor enantiomer: t_R=28.1 min.

ethyl 4-(1-methyl-1H-indol-3-yl)-2-oxo-4-phenylbutanoate (Table 3 – Entry 2)

pale yellow oil
^{1}H-NMR (250 MHz, $CDCl_3$): 7.40-7.31 (m, 1H), 7.30-7.15 (m, 5H), 7.15-7.07 (m, 2H), 6.98-6.90 (m, 1H), 6.80 (s, 1H), 4.84 (t, *J* = 7.55 Hz, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.66 (s, 3H), 3.63-3.48 (m, 2H), 1.20 (t, *J* = 7.15 Hz, 1H); ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 193.1, 161.0, 143.4, 137.3, 128.6, 127.8, 126.9, 126.6, 126.4, 121.9, 119.5, 119.0, 116.9, 109.3, 105.0, 62.5, 45.8, 37.8, 32.8, 13.9; IR (KBr): $\tilde{\nu}$ = 3421, 3058, 2950, 1729,

1614, 1471, 1257, 1062, 740, 700 cm^{-1}; MS-EI: m/z(%):335.2 (18) $[M]^{+\bullet}$, 336.1 (4) $[M]^{+\bullet}$, 220.1 (100) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+15.6 (c=0.5 in $CHCl_3$); HPLC conditions: OD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min-1, major enantiomer: t_R=62.8 min; minor enantiomer: t_R=72.0 min.

2 methyl 4-(5-bromo-1-methyl-1H-indol-3-yl)-2-oxo-4-phenylbutanoate (Table 3 – Entry 3)

pale yellow oil which solidifies upon standing

^{1}H-NMR (250 MHz, $CDCl_3$): 7.48-7.44 (m, 1H), 7.26-7.08 (m, 6H), 7.04 (d, *J* = 8.7 Hz, 1H), 6.80 (s ,1H), 4.77 (t, *J* = 7.6 Hz, 1H), 3.72 (s, 3H), 3.64 (s, 3H), 3.60-3.43 (m, 2H); ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 192.4, 164.5, 161.3, 142.9, 136,0 128.7, 128.5, 127.7, 127.5, 126.8, 124.8, 121.9, 116.5, 112.5, 110.8, 53.0, 45.8, 37.5, 33.0; IR (KBr): $\tilde{\nu}$=3446, 2923, 1731, 1481, 1280, 1066, 696 cm^{-1}

MS-EI: *m/z*(%): 399.0 (20) $[M]^{+\bullet}$, 400.0 (4)$[M]^{+\bullet}$, 401.0 (19), 402.0 (4)$[M+H]^{+\bullet}$, 298.0 (100) $[M-C_5H_8O_3]^{+\bullet}$, 299.0 (19) $[M-C_5H_8O_3]^{+\bullet}$ 300.0 (99) $[M-C_5H_8O_3]^{+\bullet}$, 300.9 (18) $[M-C_5H_8O_3]^{+\bullet}$ $[\alpha]_D^{20}$=+9.8 (c=0.4 in $CHCl_3$); HPLC conditions: AS-H column, n-hexane/2-propanol = 75/25, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=18.2 min; minor enantiomer: t_R=17.5 min.

methyl 4-(1,7-dimethyl-1H-indol-3-yl)-2-oxo-4-phenylbutanoate (Table 3 – Entry 4)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$): 7.29-7.13 (m, 5H), 7.13-7.04 (m, 1H), 6.83-6.76 (m, 2H), 6.67 (s, 1H), 4.79 (t, *J* = 7.55 Hz, 1H), 3.91 (s, 3H), 3.69 (s, 3H), 3.63-3.42 (m, 2H), 2.64 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 192.6, 161.4, 143.4, 136.1, 128.6, 128.0, 127.9, 127.8, 126.6, 124.6, 121.3, 119.4, 117.5, 116.5, 52.9, 45.8, 37.6, 36.8, 19.8; IR (KBr): $\tilde{\nu}$= 3448, 2927, 1729,

1461, 1299, 1068, 746,3 cm^{-1}; MS-EI: *m/z*(%): 335.2 (30) $[M]^{+\bullet}$, 336.2 (6) $[M]^{+\bullet}$, 234.2 (100) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+25.8 (c=0.5 in $CHCl_3$); HPLC conditions: AS-H column, n-hexane/2-propanol = 97/3, flow rate = 0.5 mL min^{-1}, major enantiomer: t_R=44.2 min; minor enantiomer: t_R=39.0 min.

methyl 4-(4-chlorophenyl)-4-(1-methyl-1H-indol-3-yl)-2-oxobutanoate (Table 3 – Entry 5)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.33-7.26 (m, 1H), 7.23-7.08 (m, 6H), 6.98-6.91 (m, 1H), 6.80-6.79 (m, 1H), 4.81 (t, *J* = 7.51 Hz, 1H), 3.71 (s, 3H), 3.67 (s, 3H), 3.63-3.44 (m, 2H) ^{13}C-NMR (75 MHz, $CDCl_3$): δ 192.3, 161.2, 141.9, 137.4, 132.3, 129.2, 128.7, 126.6, 126.3, 122.0, 119.3, 119.1, 116.3, 109.3, 53.0, 45.6, 37.1, 32.8; IR (KBr): $\tilde{\nu}$= 3450, 3025, 2950, 1727, 1257, 1062, 740, 698 cm^{-1} MS-EI: *m/z*(%):355.1 (19) $[M]^{+\bullet}$, 356.1 (4) $[M]^{+\bullet}$, 357.1 (7) $[M]^{+\bullet}$, 254.0 (100)$[M-C_5H_8O_3]^{+\bullet}$, 255.0 (18) $[M-C_5H_8O_3]^{+\bullet}$, 256.0 (33) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+14.4 (c=0.5 in $CHCl_3$); HPLC conditions: AD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=35.9 min; minor enantiomer: t_R=30.0 min.

methyl 4-(4-bromophenyl)-4-(1-methyl-1H-indol-3-yl)-2-oxobutanoate (Table 3 – Entry 6)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$) δ = 7.34-7.27 (m, 3H), 7.23-7.08 (m, 4H), 7.00-6.89 (m, 1H), 6.80 (s, 1H), 4.79 (t, *J* = 7.5 Hz, 1H), 3.71 (s, 3H), 3.67 (s, 3H), 3.64-3.42 (m, 2H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 192.2, 161.2, 142.5, 137.4, 131.6, 129.5, 126.6, 126.3, 122.0, 120.4, 119.3, 119.2, 116.2, 109.3, 53.0, 45.5, 37.1, 32.8; IR (KBr): $\tilde{\nu}$= 3446, 2923, 1731, 1486, 1257,

1072, 740 cm^{-1}; MS-EI: *m/z*(%):399.0 (20) $[M]^{+\bullet}$, 400.0 (5) $[M]^{+\bullet}$, 401.0 (20) $[M]^{+\bullet}$, 402.0 (4) $[M]^{+\bullet}$, 298.0 (100)$[M-C_5H_8O_3]^{+\bullet}$, 299.0 (20)$[M-C_5H_8O_3]^{+\bullet}$, 300.0 (100)$[M-C_5H_8O_3]^{+\bullet}$, 301.0 (17)$[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+10.2 (c=0.5 in $CHCl_3$); HPLC conditions: AS-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=43.7 min; minor enantiomer: t_R=38.6 min.

methyl 4-(1-methyl-1H-indol-3-yl)-2-oxo-4-p-tolylbutanoate (Table 3 – Entry 7)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$) δ = 7.39-7.32 (m, 1H), 7.21-7.06 (m, 4H), 7.02-6.90 (m, 3H), 6.78 (s, 1H), 4.80 (t, *J* = 7.6 Hz, 1H), 3.68 (s, 3H), 3.65 (s, 3H), 3.62-3.45 (m, 2H), 2.21 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 192.7, 161.4, 140.3, 137.3, 136.0, 129.4, 129.2, 127.6, 126.8, 126.6, 126.3, 121.8, 119.5, 119.0, 117.1, 109.2, 52.8, 45.9, 37.3, 32.7, 21.0; IR (KBr): $\tilde{\nu}$= 3446, 3023, 2950, 1729, 1612, 1471, 1255, 1081, 742 cm^{-1}; MS-EI: *m/z*(%):335.1 (18) $[M]^{+\bullet}$, 336.1 (4) $[M]^{+\bullet}$, 234.1 (100) $[M-C_5H_8O_3]^{+\bullet}$, 235.1 (19) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+11.2 (c=0.5 in $CHCl_3$); HPLC conditions: AS-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=29.7 min; minor enantiomer: t_R=26.0 min.

methyl 4-(5-bromo-1-methyl-1H-indol-3-yl)-2-oxo-4-p-tolylbutanoate ((Table 3 – Entry 8)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$) δ = 7.50-7.44 (m, 1H), 7.21-7.08 (m, 3H), 7.05-6.99 (m, 3H), 6.79 (s, 1H), 4.73 (t, *J* = 7.6 Hz, 1H), 3.71 (s, 3H), 3.63 (s, 3H), 3.59-3.42 (m, 2H), 2.22 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 192.4, 161.3, 139.9, 136.3, 136.0, 129.3, 128.5, 127.5, 127.4, 124.7, 121.9, 116.7, 112.5,

110.7, 52.9, 45.8, 37.1, 32.9, 21.0; IR (KBr): $\tilde{\nu}$ = 3432, 1729, 1608, 1475, 1072, 792 cm^{-1}; MALDO-TOF: *m/z*(%):436.4 $[M+Na]^+$, 438.8 $[M+Na]^+$; $[\alpha]_D^{20}$=–27.8 (c=0.5 in $CHCl_3$); HPLC conditions: AD-H column, n-hexane/2-propanol = 98/2, flow rate = 0.4 mL min^{-1}, major enantiomer: t_R=83.6 min; minor enantiomer: t_R=86.4 min.

methyl 4-(4-methoxyphenyl)-4-(1-methyl-1H-indol-3-yl)-2-oxobutanoate (Table 3 – Entry 9)

colorless oil

^{1}H-NMR (300 MHz, $CDCl_3$..) δ = 7.38-7.29 (m, 1H), 7.19-7.06 (m, 4H), 6.98-6.90 (m, 1H), 6.78-6.69 (m, 3H), 4.79 (t, *J* = 7.6 Hz, 1H), 3.69 (s, 3H), 3.67 (s, 3H), 3.65 (s, 3H), 3.61-3.43 (m, 2H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 192.7, 161.4, 158.2, 137.4, 135.5, 128.7, 126.8, 126.2, 121.8, 119.5, 119.0, 117.2, 113.9, 109.2, 55.2, 52.9, 45.9, 37.0, 32.7; IR (KBr): $\tilde{\nu}$ = 3446, 2952, 1729, 1612, 1511, 1247, 1078, 1033 833, 744 cm^{-1}; MS-EI: *m/z*(%): 351.0 (17) $[M]^{+\bullet}$, 352.1 (4) $[M]^{+\bullet}$, 250.0 (100) $[M-C_5H_8O_3]^{+\bullet}$, 251.1 (19) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+19.4 (c=0.5 in $CHCl_3$); HPLC conditions: AS-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=56.4 min; minor enantiomer: t_R=51.1 min.

methyl 4-(1-methyl-1H-indol-3-yl)-4-(naphthalen-2-yl)-2-oxobutanoate (Table 3 – Entry 10)

pale yellow oil

^{1}H-NMR (300 MHz, $CDCl_3$) δ = 7.77-7.65 (m, 4H), 7.41-7.32 (m, 4H), 7.20-7.16 (m, 1H), 7.13-7.07 (m, 1H), 6.95-6.88 (m, 1H), 6.83 (s, 1H), 5.01 (t, *J* = 7.5 Hz, 1H), 3.67-3.63 (m, 8H); ; ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 192.6, 161.3, 140.8, 137.4, 133.5, 132.4, 128.3, 127.8, 127.6, 126.9, 126.5, 126.4, 125.9, 125.9, 125.6, 121.9, 119.5, 119.1, 116.7, 109.2, 52.9, 45.6, 37.8,

32.8; IR (KBr): $\tilde{\nu}$ = 3446, 3052, 2923, 1731, 1255, 1076, 742 cm^{-1} MS-EI: *m/z*(%):371.1 (20) $[M]^{+\bullet}$, 372.1 (6) $[M]^{+\bullet}$, 270.1 (100) $[M-C_5H_8O_3]^{+\bullet}$, 271.1 (22) $[M-C_5H_8O_3]^{+\bullet}$; $[\alpha]_D^{20}$=+6.2 (c=0.5 in $CHCl_3$); HPLC conditions: OD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=88.6 min; minor enantiomer: t_R=100.5 min.

(E)-methyl 2,2-bis(1-methyl-1H-indol-3-yl)-4-phenylbut-3-enoate (3a)

colorless crystals; In the NMR we suggest that two different conformers are visible.

^{1}H-NMR (300 MHz, $CDCl_3$) δ = 7.57-7.51 (m, 1H, major isomer), 7.44-7.38 (m,1H), 7.36-7.31 (m,3H), 7.28-7.06 (m,8H), 6.98-6.89 (m,2H), 7.06-6.98 (m, 2H), 6.78-6.73 (m,2H, major isomer), 6.72-6.64 (m,2H, minor isomer), 6.16 (d, *J* = 16.11 Hz,1C, minor isomer), 5.65 (d, *J* = 10.49 Hz,1H, major isomer), 3.75 (s,3H, major isomer), 3.68-3.60 (m,15C, both isomers; ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 174.3, 168.9, 143.6, 138.3, 137.6, 137.5, 131.2, 129.1, 129.1, 128.5, 128.4, 128.2, 128.2, 128.2, 127.1, 127.1, 126.8, 126.7, 126.5, 126.4, 126.3, 125.3, 122.0, 121.6, 121.6, 121.4, 120.1, 119.9, 119.9, 119.1, 118.9, 116.9, 115.3, 112.4, 109.5, 109.2, 109.2, 53.3, 52.5, 51.8, 42.8, 32.8, 32.8, 32.7, 21.5,; IR (KBr): $\tilde{\nu}$ = 3446, 3051, 2946, 1716, 1473, 1384, 1218, 736 cm^{-1}; MS-EI: *m/z*(%):434.2 (70) $[M]^{+\bullet}$, 435.2 (23) $[M]^{+\bullet}$, 436.2 (4) $[M]^{+\bullet}$, 375.2 (100) $[M-C_2H_3O_2]^{+\bullet}$, 376.2 (30) $[M-C_2H_3O_2]^{+\bullet}$, 377.2 (5) $[M-C_2H_3O_2]^{+\bullet}$; HPLC conditions: OD-H column, n-hexane/2-propanol = 95/5, flow rate = 0.6 mL min^{-1}, major enantiomer: t_R=48.8 min; minor enantiomer: t_R=36.2 min.

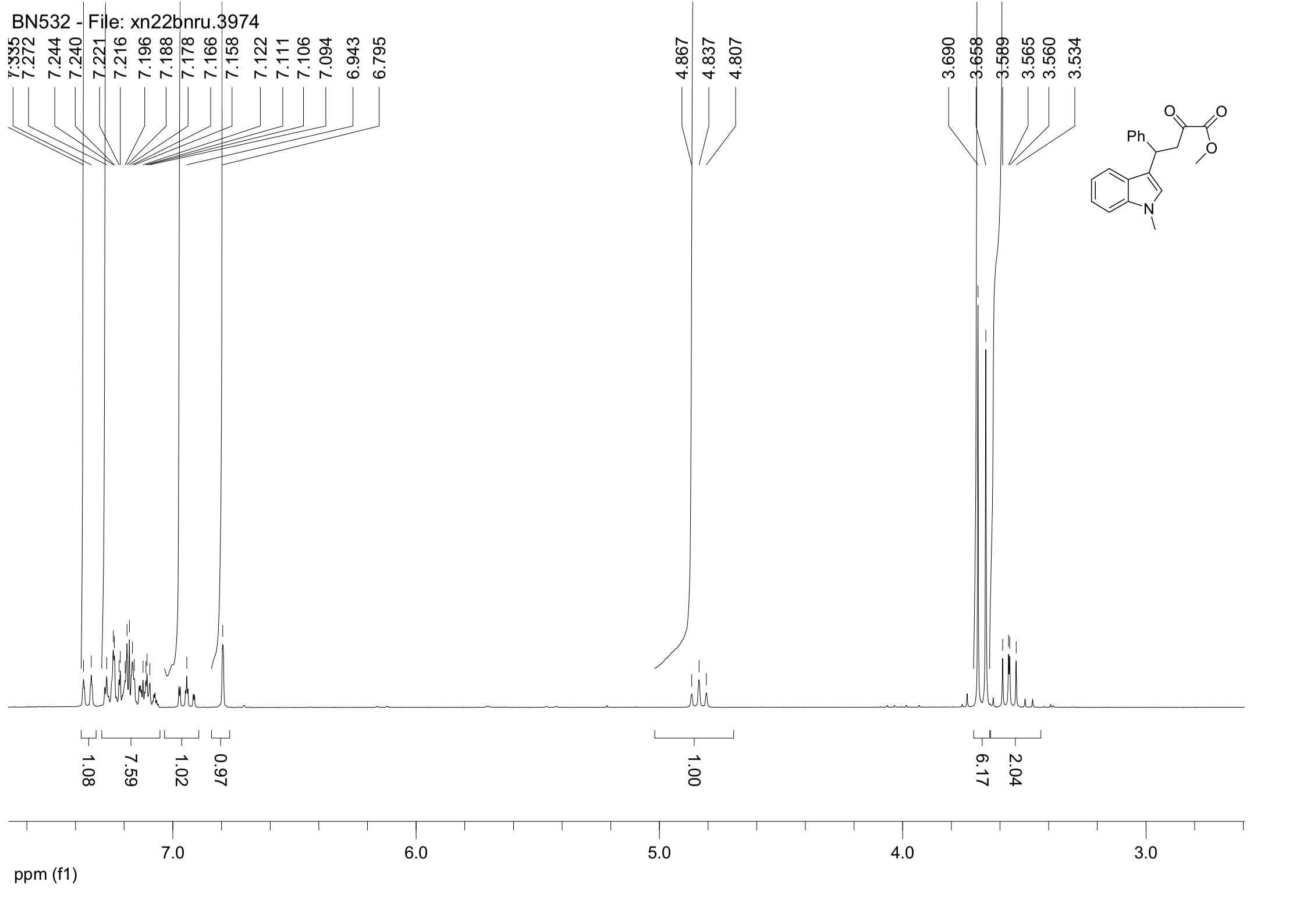
(4R)-methyl 4-(4-methoxyphenyl)-2-(4-methoxyphenylamino)-4-(1-methyl-1H-indol-3-yl)butanoate (8)

pale yellow oil – Mixture of both diastereoisomers 1:1.3 ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.22-7.14 (m, 8H), 7.37-7.31 (m, 3H), 7.13-7.07 (m, 3H), 6.97-6.88 (m, 3H), 6.81-6.73 (m, 7H), 6.68-6.54 (m, 5H), 6.44-6.31 (m, 5H), 4.45 (m, 1H, minor diastereomer), 4.36 (t, *J* = Hz, 1H, major diastereomer), 3.95-3.88 (m, 1H, major diastereomer), 3.84-3.76 (m, 1H, minor diastereomer), 3.73-3.43 (m, 12H, both diastereomers), 2.64-2.52 (m, 1H, major diastereomer), 2.34 (m, 2H, minor diastereomer) 2.32-2.21 (m, 1H, major diastereomer); ^{13}C-NMR (75 MHz, $CDCl_3$) δ = 129.2, 128.9, 126.2, 125.8, 121.7, 121.7, 119.7, 119.6, 118.9, 118.9, 115.6, 115.4, 114.8, 114.8, 113.9, 113.8, 109.2, 109.1, 56.6, 56.4, 55.7, 55.3, 55.3, 52.1, 52.0, 40.2, 39.7, 38.5, 38.3, 32.8, 32.8, MALDI-TOF: *m/z*:459.6 $[M]^+$

References

[1] Jørgensen, K. A. *Synthesis* **2003**, 1117-1125.

[2] Jensen, K. B.; Torhauge, J.; Hazell, R. G.; Jørgensen, K. A. *Angew. Chem.* **2001**, *113*,164-167.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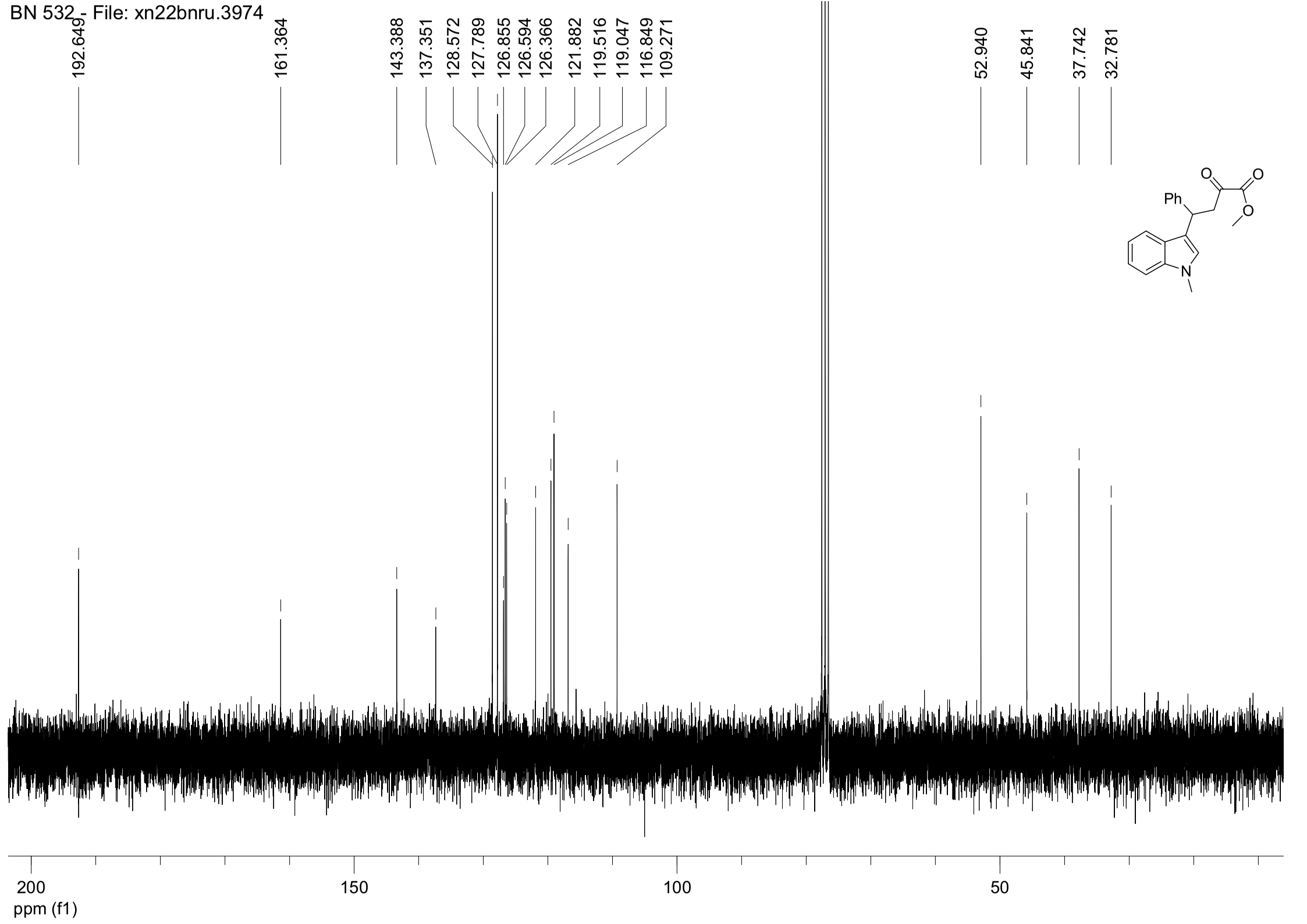
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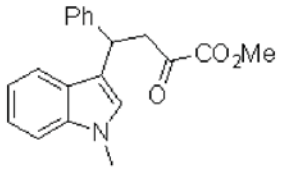

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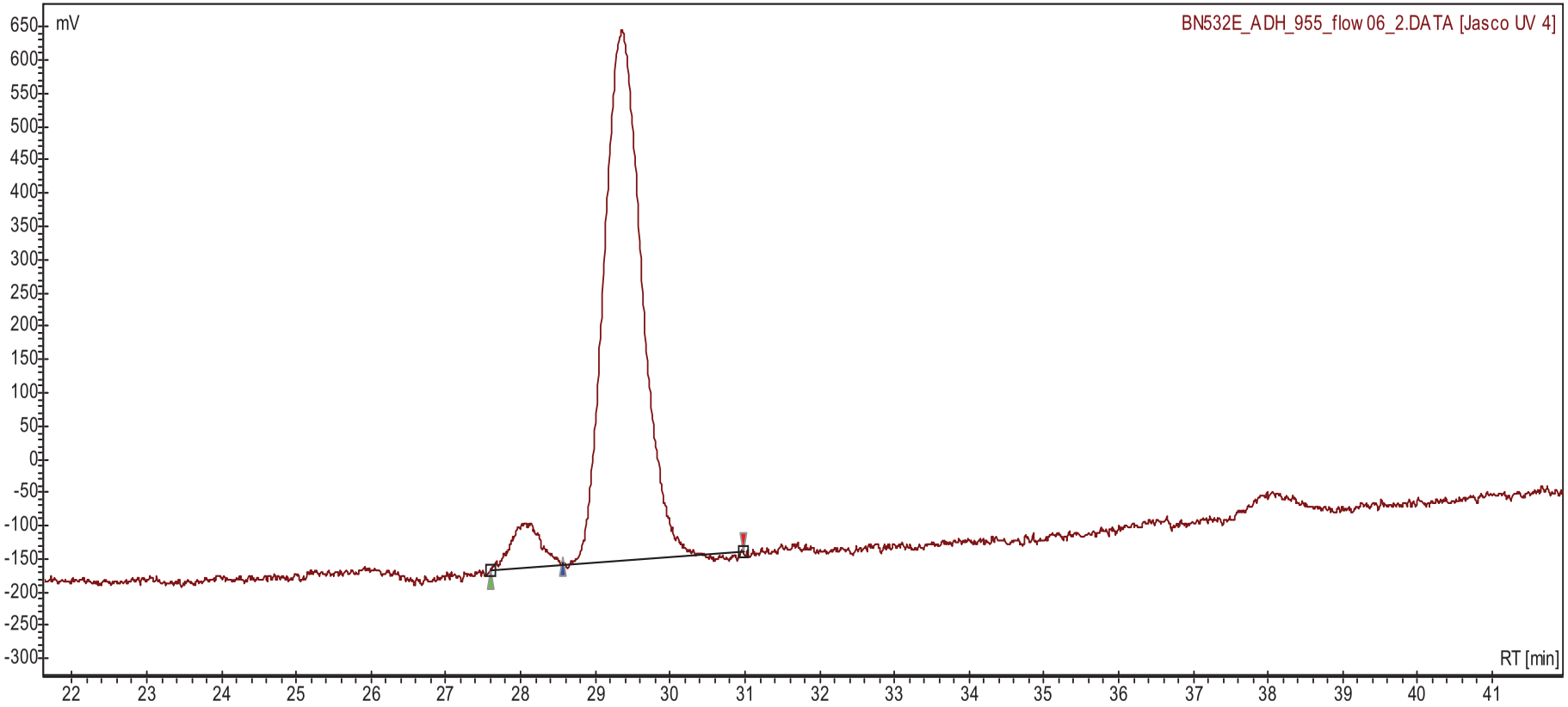

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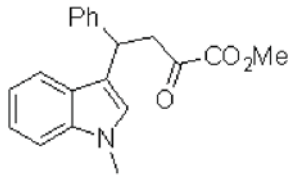

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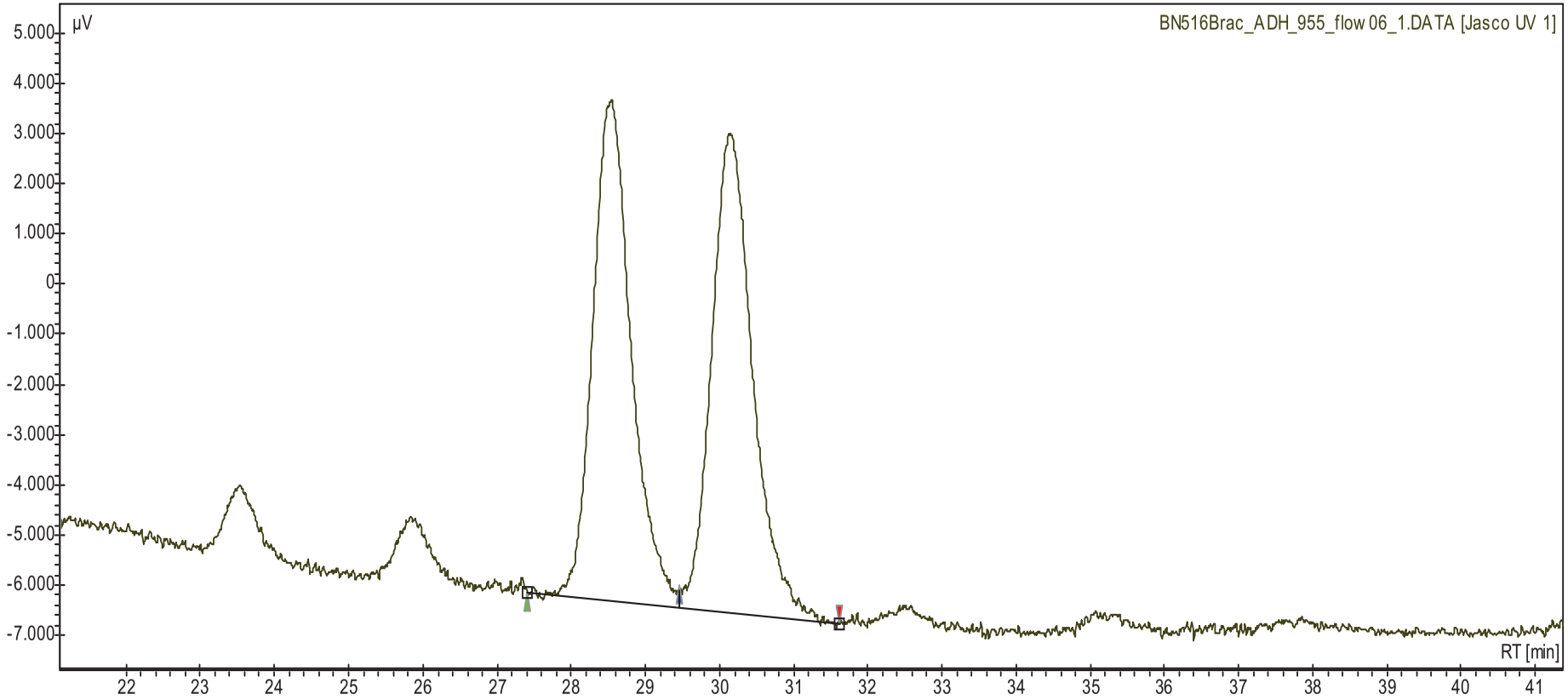
Date: 27.02.2007 22:03:02



BN516Brac_ADH_955_flow06_1.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	27,408	28,533	29,460	49,479
2	29,460	30,150	31,617	50,521
Total				100,000

Sample Information

Analyzed : 28.03.2007 12:56:46
Sample Name : BN532E

Method

====== Analytical Line 1 ======

[GC-2010]

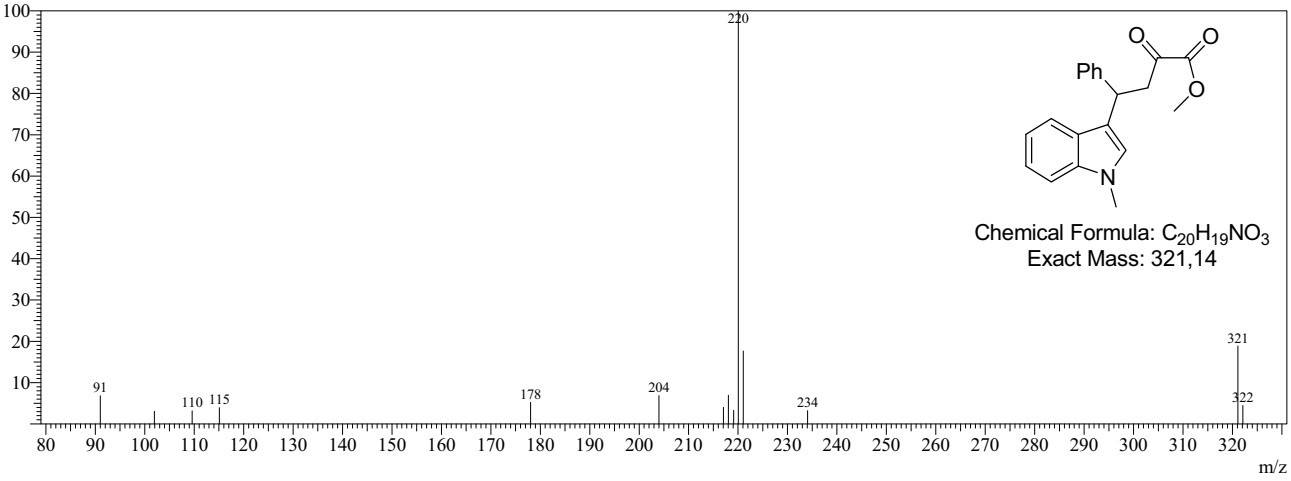
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

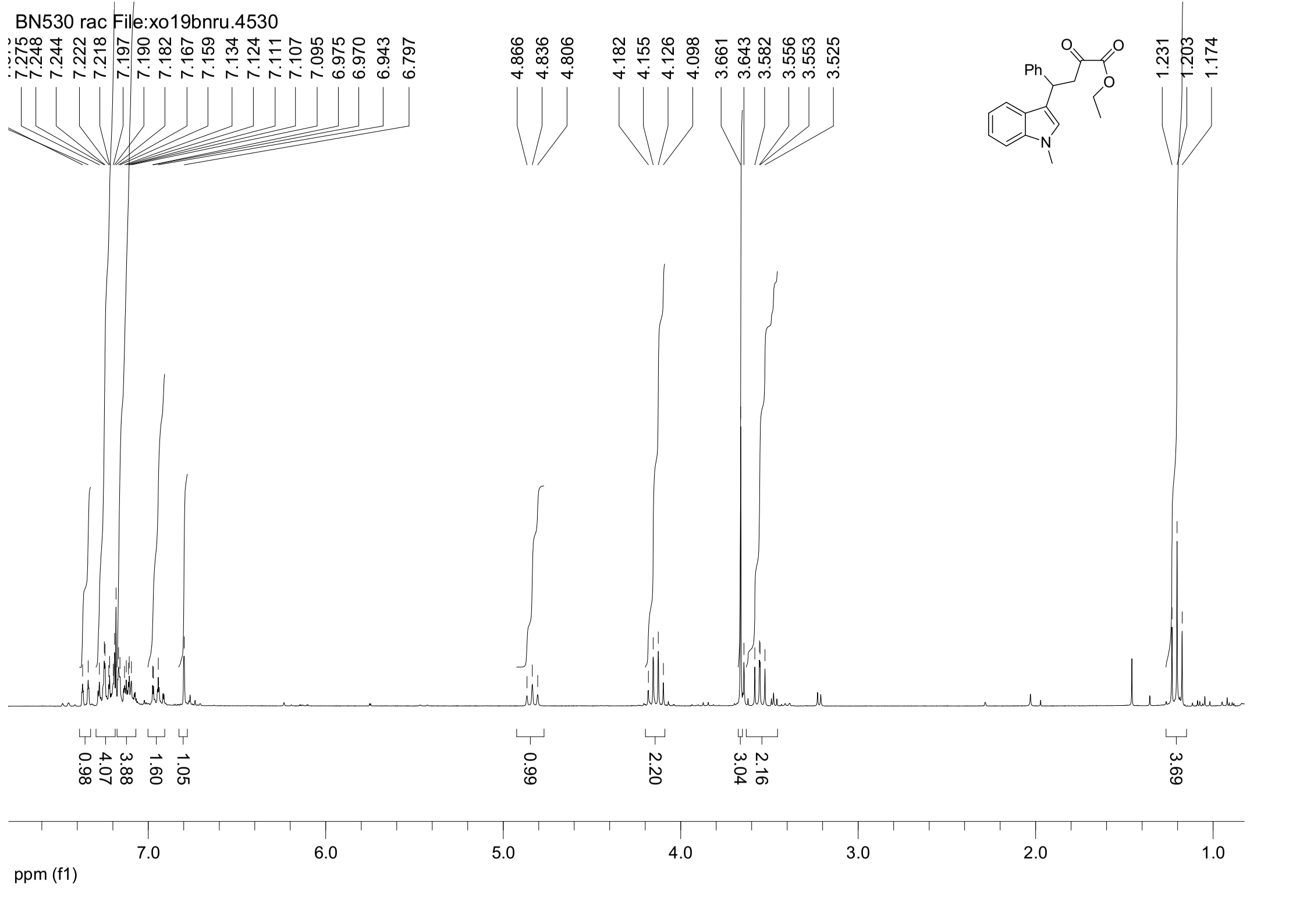
Line#:1 R.Time:12.0(Scan#:1080)
MassPeaks:14 BasePeak:220(696110)
RawMode:Averaged 11.9-12.0(1074-1085)
BG Mode:None



Mass Table

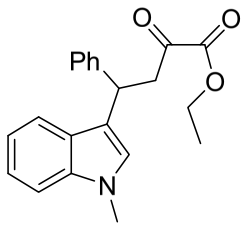
Line#:1 R.Time:12.0(Scan#:1080)
MassPeaks:14 BasePeak:220(696110)
RawMode:Averaged 11.9-12.0(1074-1085)
BG Mode:None

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	91.00	6.82	5	178.05	5.21	9	219.10	3.29	13	321.10	18.84
2	101.95	3.05	6	204.00	6.87	10	220.05	100.00	14	322.10	4.44
3	109.60	3.17	7	217.05	4.02	11	221.05	17.65			
4	115.10	3.96	8	218.05	6.95	12	234.05	3.22			


BN530 rac File:xo19bnru.4530
7.275
7.248
7.244
7.222
7.218
7.197
7.190
7.182
7.167
7.159
7.134
7.124
7.111
7.107
7.095
6.975
6.970
6.943
6.797
4.866
4.836
4.806
4.182
4.155
4.126
4.098
3.661
3.643
3.582
3.556
3.553
3.525
1.231
1.203
1.174
Ph
O
O
O
N
0.98
4.07
3.88
1.60
1.05
0.99
2.20
3.04
2.16
3.69
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

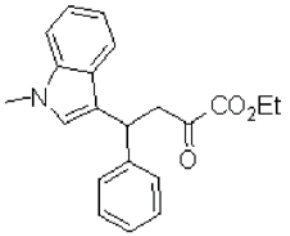
BN530rac - File:xo19bnru.4530

193.115
161.023
143.437
137.337
128.562
127.801
126.872
126.572
126.393
121.866
119.520
119.033
116.881
109.257
62.463
45.788
37.804
32.785
13.960

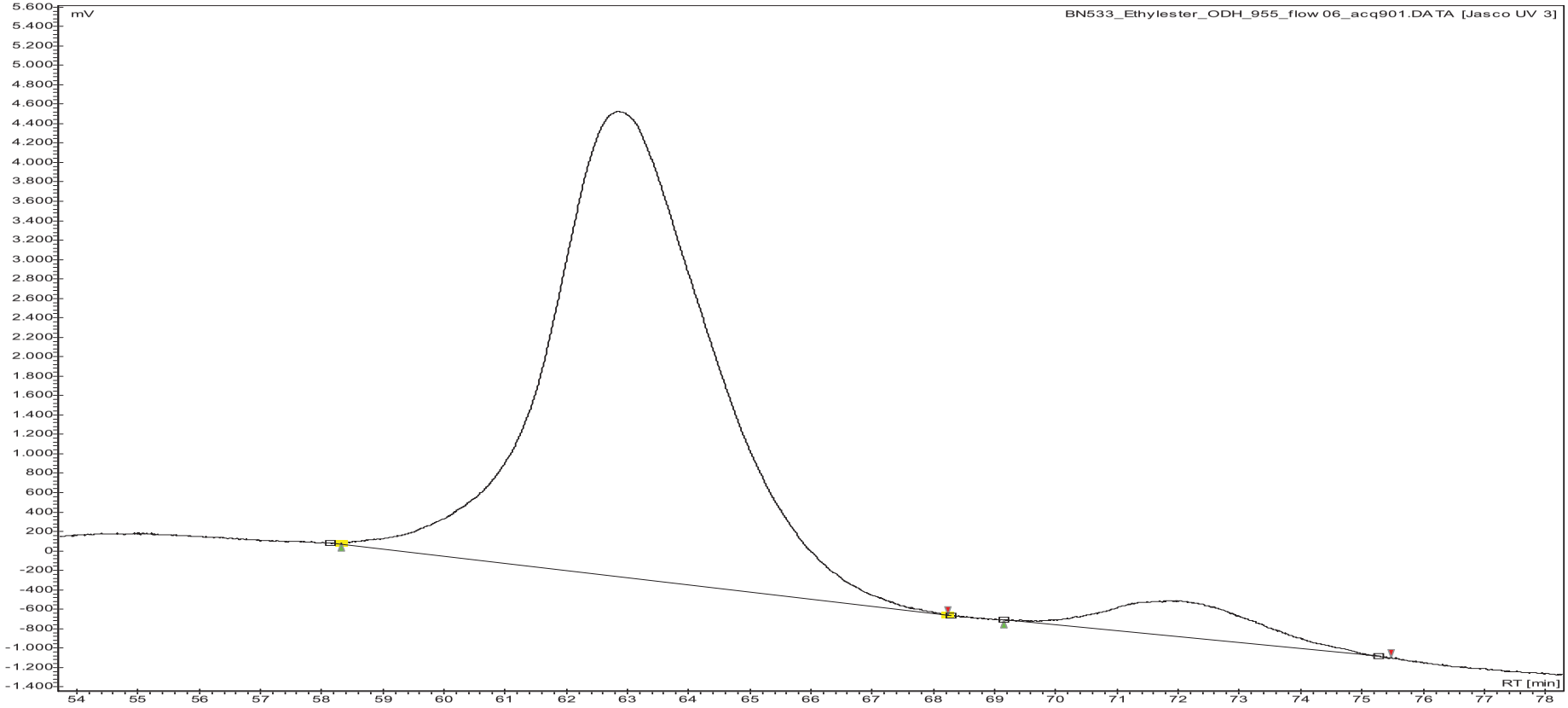


250
200
150
100
50
0
ppm (f1)

Chromatogram : BN533_Ethylester_ODH_955_flow06_acq901



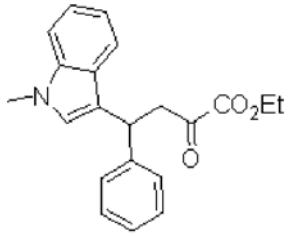
Data file: BN533_Ethylester_ODH_955_flow06_acq901.DATA
Method: HPLC2_ODH_955_flow06_acq90
Date: 20.03.2007 14:56:10



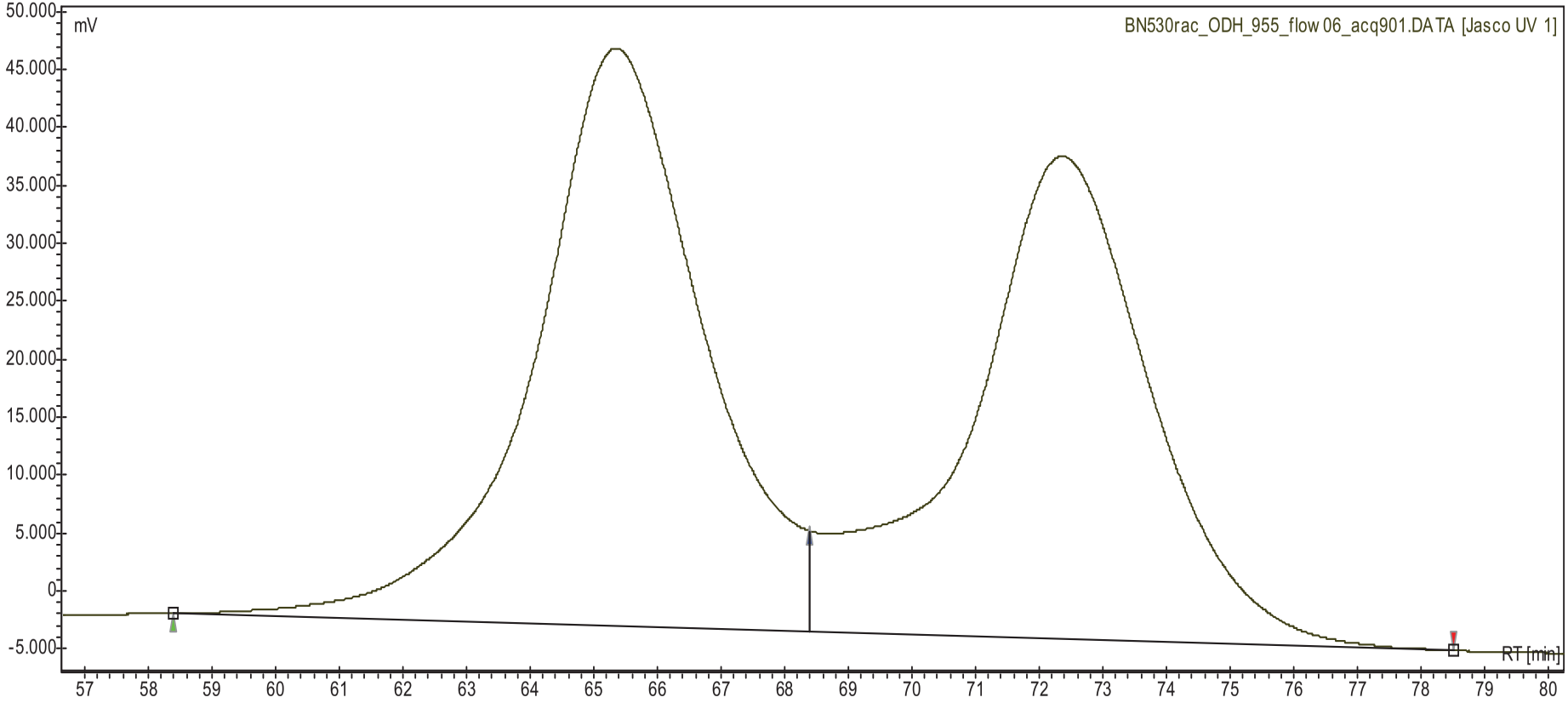
BN533_Ethylester_ODH_955_flow06_acq901.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	58,314	62,867	68,242	0,000	93,36	4790,9	14776,2	93,359
2	UNKNOWN	69,155	72,033	75,469	0,000	6,64	372,7	1051,1	6,641
Total						100,00	5163,6	15827,3	100,000

Chromatogram : BN530rac_ODH_955_flow06_acq901



Data file: BN530rac_ODH_955_flow06_acq901.DATA
Method: HPLC2_ODH_955_flow06_acq90
Date: 19.03.2007 15:43:58



BN530rac_ODH_955_flow06_acq901.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	58,378	65,342	68,381	52,475
2	68,381	72,350	78,505	47,525
Total				100,000

Sample Information

Analyzed : 21.05.2007 12:48:20
Sample Name : BN533

Method

====== Analytical Line 1 ======

[GC-2010]

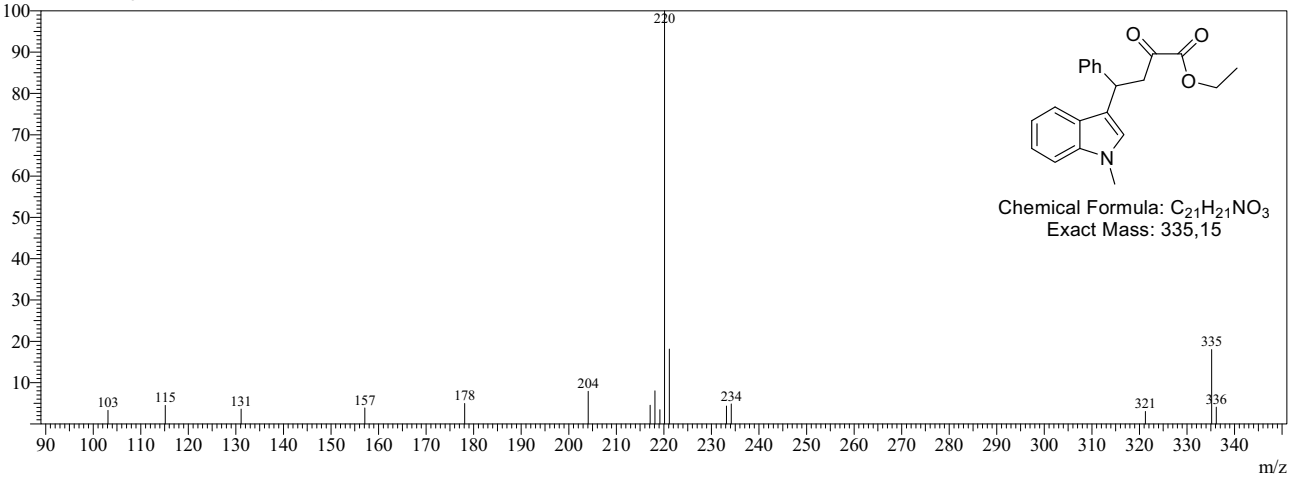
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:10.3 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:10.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	320.0	10.00

Spectrum

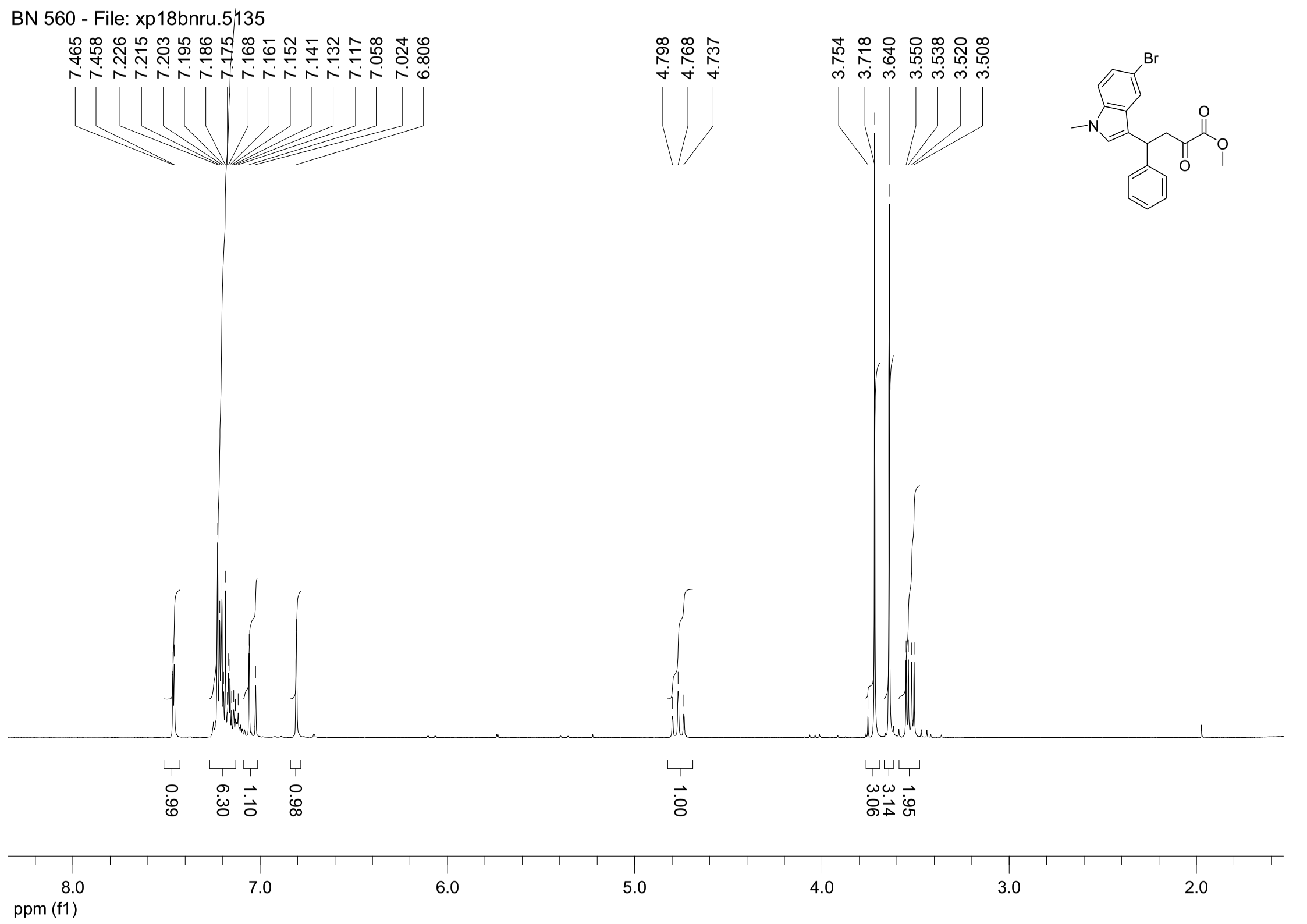
Line#:1 R.Time:12.2(Scan#:1100)
MassPeaks:16 BasePeak:220(113831)
RawMode:Averaged 12.1-12.2(1096-1108)
BG Mode:Averaged 12.6-13.4(1147-1249)



Mass Table
Line#:1 R.Time:12.2(Scan#:1100)
MassPeaks:16 BasePeak:220(113831)
RawMode:Averaged 12.1-12.2(1096-1108)
BG Mode:Averaged 12.6-13.4(1147-1249)

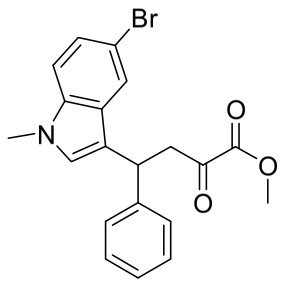
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	103.10	3.29	5	178.10	4.95	9	219.15	3.43	13	234.15	4.86
2	115.15	4.49	6	204.10	7.92	10	220.15	100.00	14	321.25	3.01
3	131.10	3.60	7	217.10	4.54	11	221.15	18.11	15	335.20	18.00
4	157.10	3.87	8	218.10	8.02	12	233.15	4.36	16	336.15	4.05

BN 560 - File: xp18bnru.5135



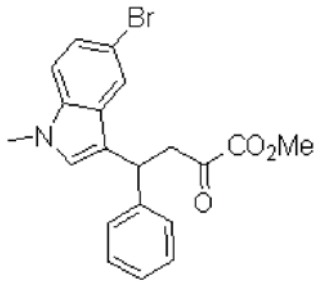
BN 560 - File:xp18bnru.5135

192.359
164.528
161.274
142.935
135.990
128.705
128.493
127.655
127.474
126.797
124.796
121.950
116.488
112.545
110.808
53.024
45.790
37.456
32.979

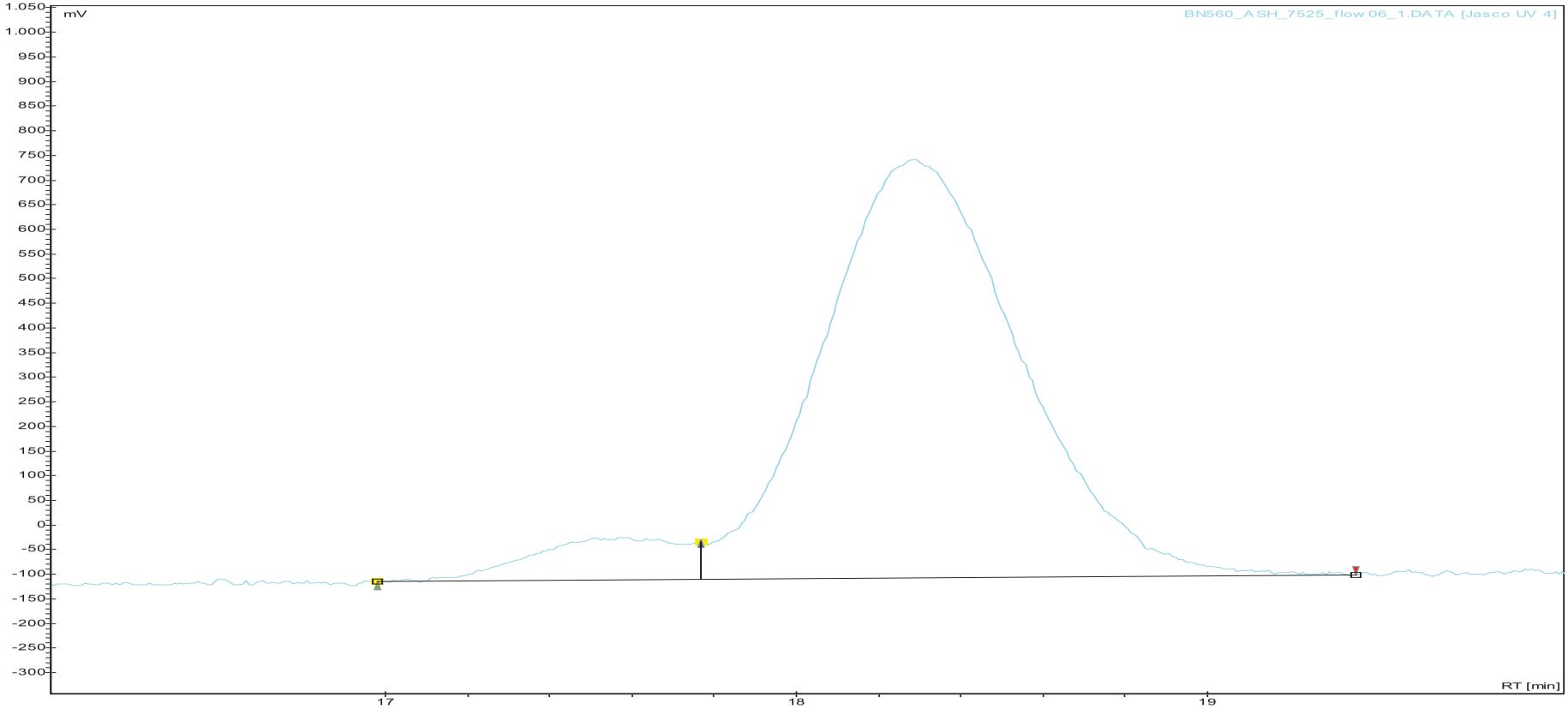


200
150
100
50
ppm (f1)

Chromatogram : BN560_ASH_7525_flow06_1



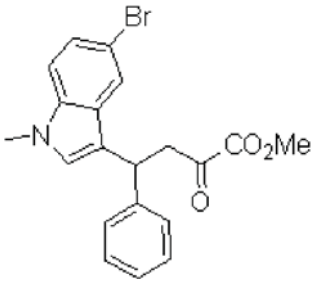
Data file: BN560_ASH_7525_flow06_1.DATA
Method: HPLC2_ASH_7525_flow06_acq30
Date: 27.04.2007 10:01:01



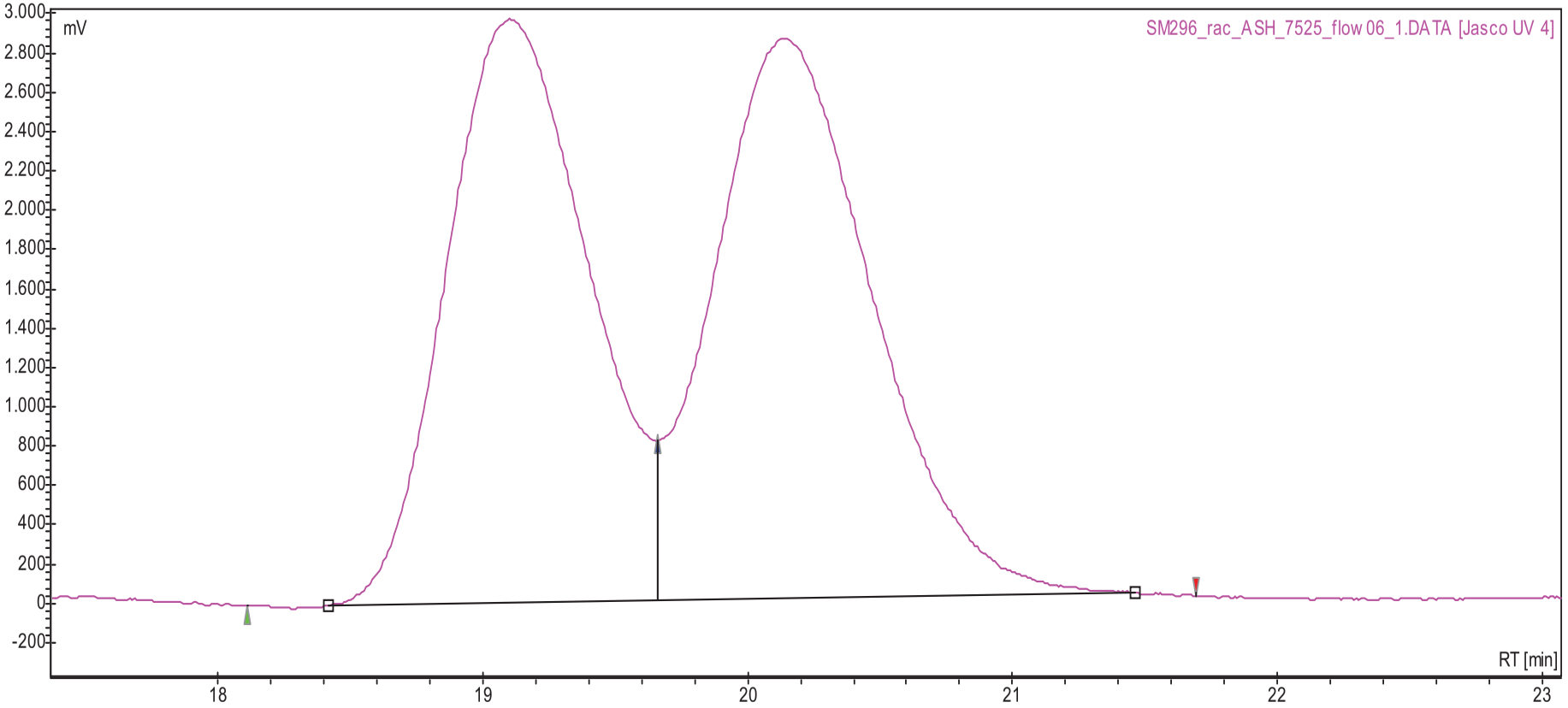
BN560_ASH_7525_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	16,980	17,558	17,766	0,000	7,16	81,2	36,5	7,158
2	UNKNOWN	17,766	18,283	19,365	0,000	92,84	848,1	472,8	92,842
Total						100,00	929,3	509,3	100,000

Chromatogram : SM296_rac_ASH_7525_flow06_1



Data file: SM296_rac_ASH_7525_flow06_1.DATA
Method: HPLC2_ASH_7525_flow06_acq30
Date: 20.04.2007 11:06:46



SM296_rac_ASH_7525_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	18,110	19,100	19,660	0,000	48,14	2968,3	1844,8	48,139
2	UNKNOWN	19,660	20,142	21,694	0,000	51,86	2846,0	1987,5	51,861
Total						100,00	5814,3	3832,3	100,000

Sample Information

Analyzed : 26.04.2007 15:22:31
Sample Name : BN560

Method

====== Analytical Line 1 ======

[GC-2010]

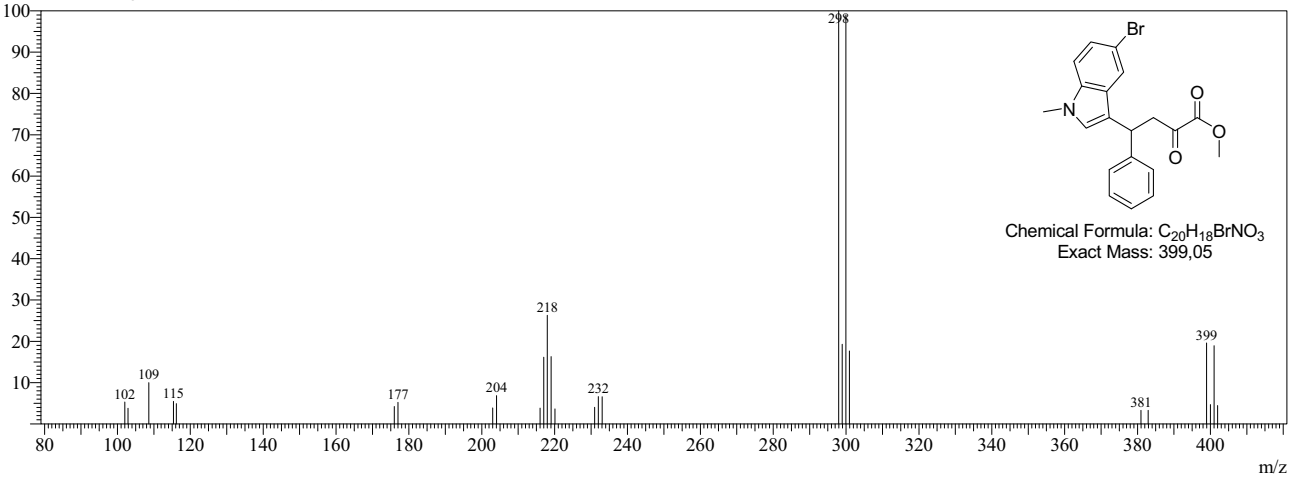
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

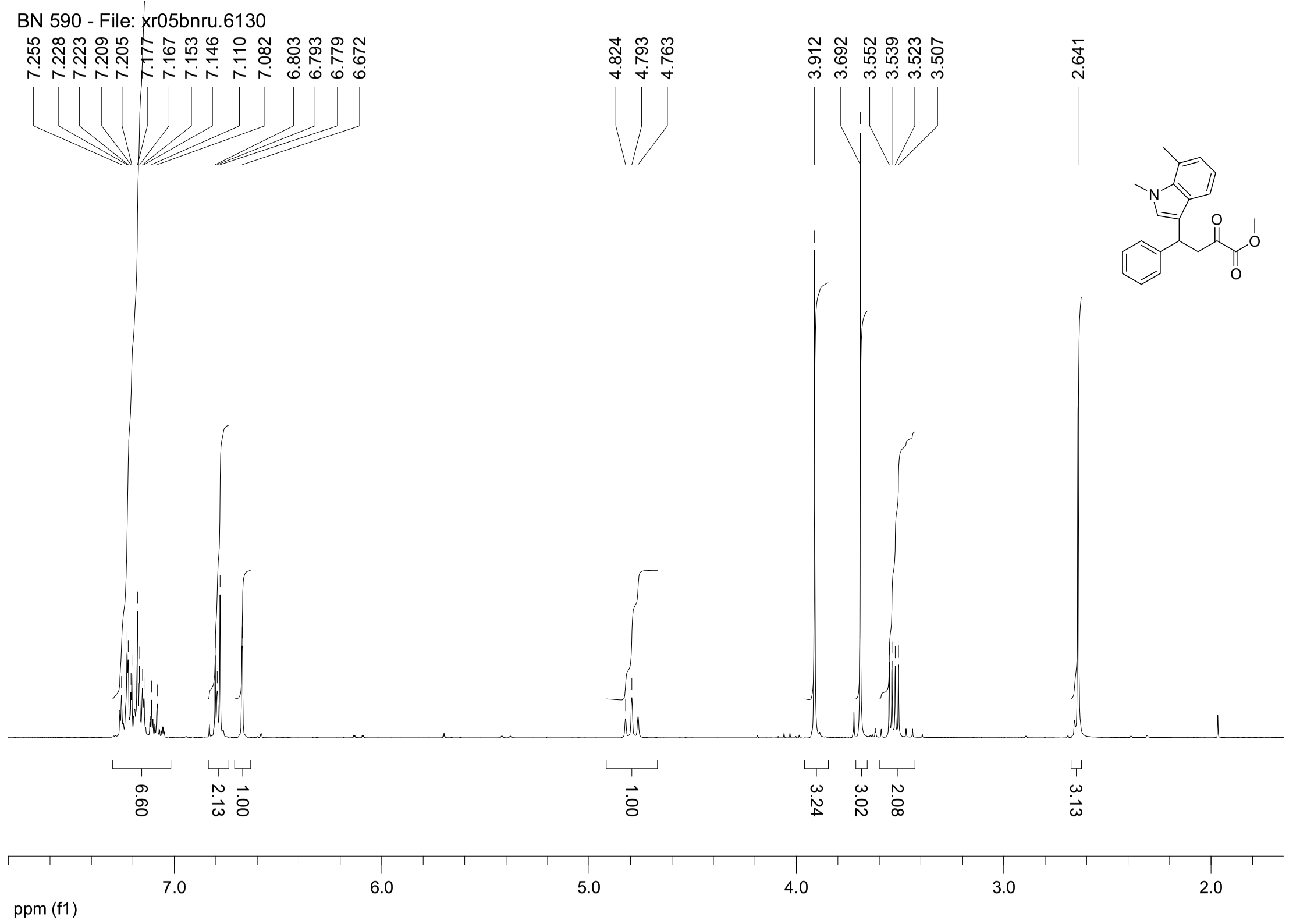
Line#:1 R.Time:13.7(Scan#:1280)
MassPeaks:27 BasePeak:298(18795)
RawMode:Averaged 13.5-13.8(1261-1297)
BG Mode:Averaged 14.3-15.9(1357-1549)



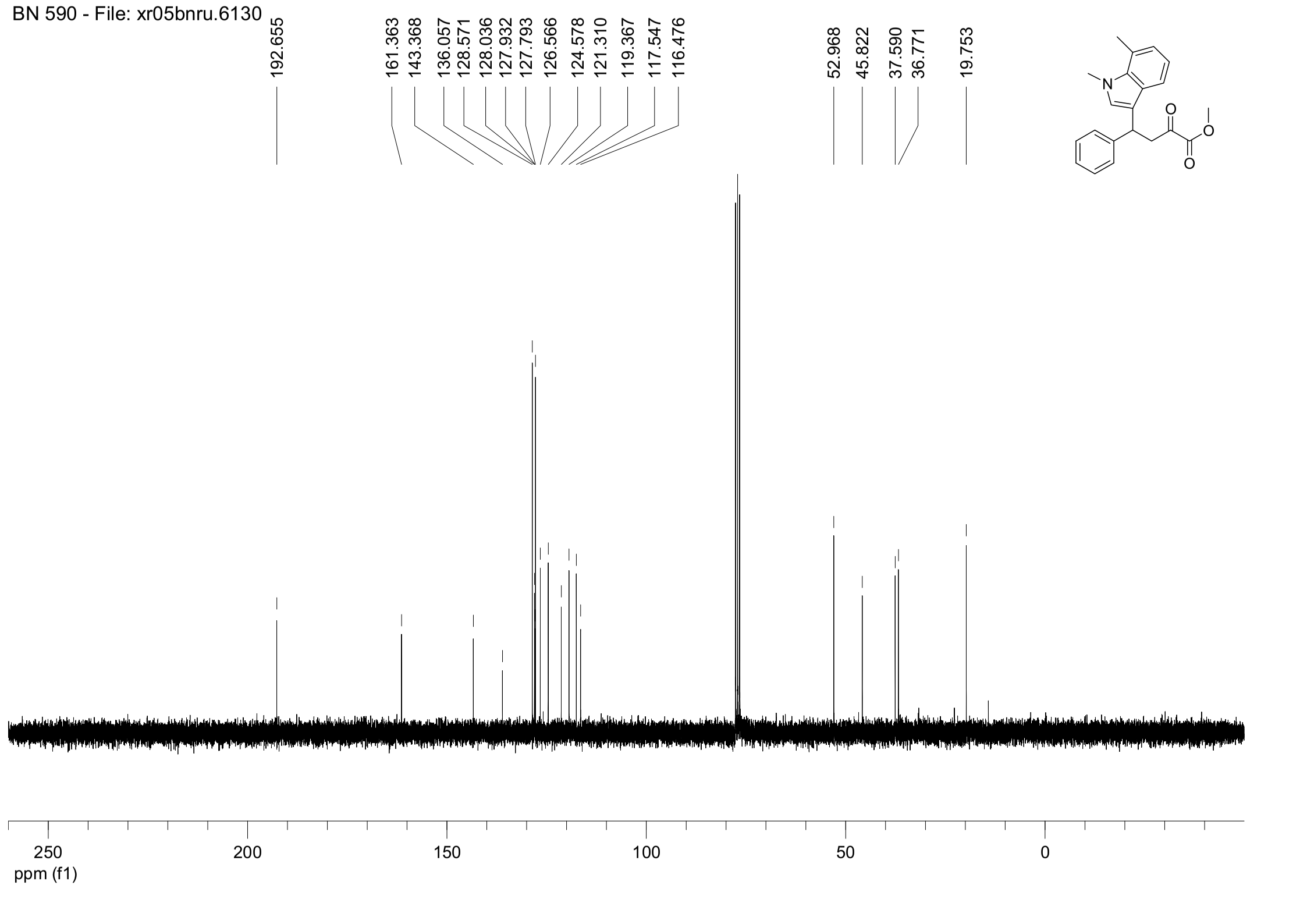
Mass Table

Line#:1 R.Time:13.7(Scan#:1280)
MassPeaks:27 BasePeak:298(18795)
RawMode:Averaged 13.5-13.8(1261-1297)
BG Mode:Averaged 14.3-15.9(1357-1549)

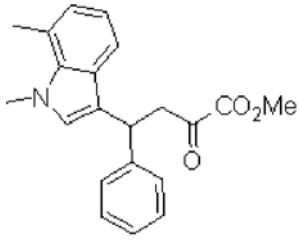
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	102.00	5.32	8	203.00	3.87	15	231.00	4.03	22	380.95	3.30
2	102.90	3.78	9	204.05	6.84	16	232.00	6.63	23	382.90	3.30
3	108.60	10.00	10	216.00	3.85	17	233.05	6.59	24	398.95	19.60
4	115.35	5.48	11	217.00	16.16	18	297.95	100.00	25	400.00	4.69
5	116.15	4.93	12	218.00	26.32	19	298.95	19.29	26	401.00	18.95
6	176.00	4.24	13	219.05	16.29	20	299.95	99.02	27	402.00	4.46
7	177.00	5.25	14	220.10	3.64	21	300.90	17.65			


BN 590 - File: xr05bnru.6130
7.255
7.228
7.223
7.209
7.205
7.177
7.167
7.153
7.146
7.110
7.082
6.803
6.793
6.779
6.672
4.824
4.793
4.763
3.912
3.692
3.552
3.539
3.523
3.507
2.641
N
O
O
O
6.60
2.13
1.00
1.00
3.24
3.02
2.08
3.13
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)

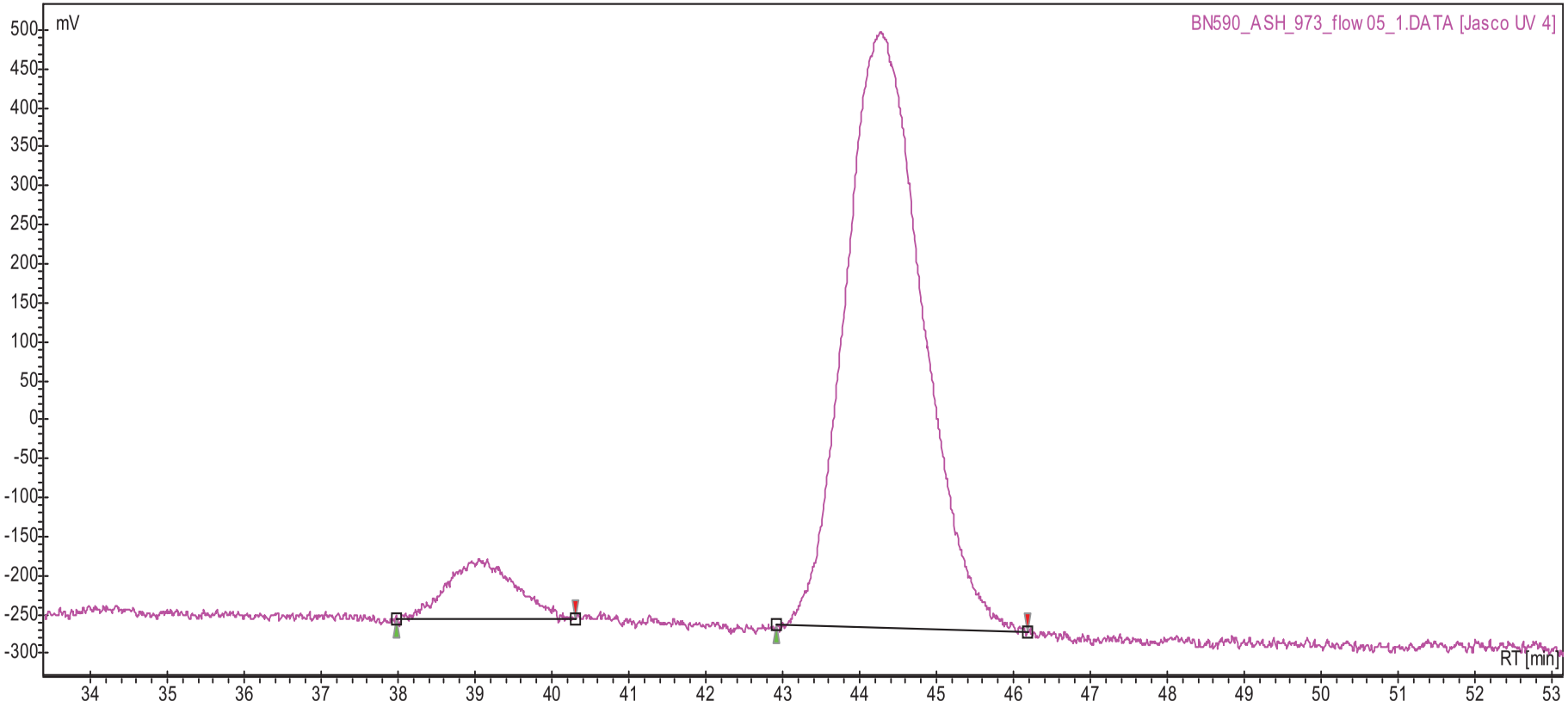
BN 590 - File: xr05bnru.6130



Chromatogram : BN590_ASH_973_flow05_1



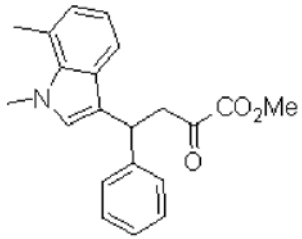
Data file: BN590_ASH_973_flow05_1.DATA
Method: HPLC2_ASH_973flow05_acq80
Date: 05.06.2007 16:05:20



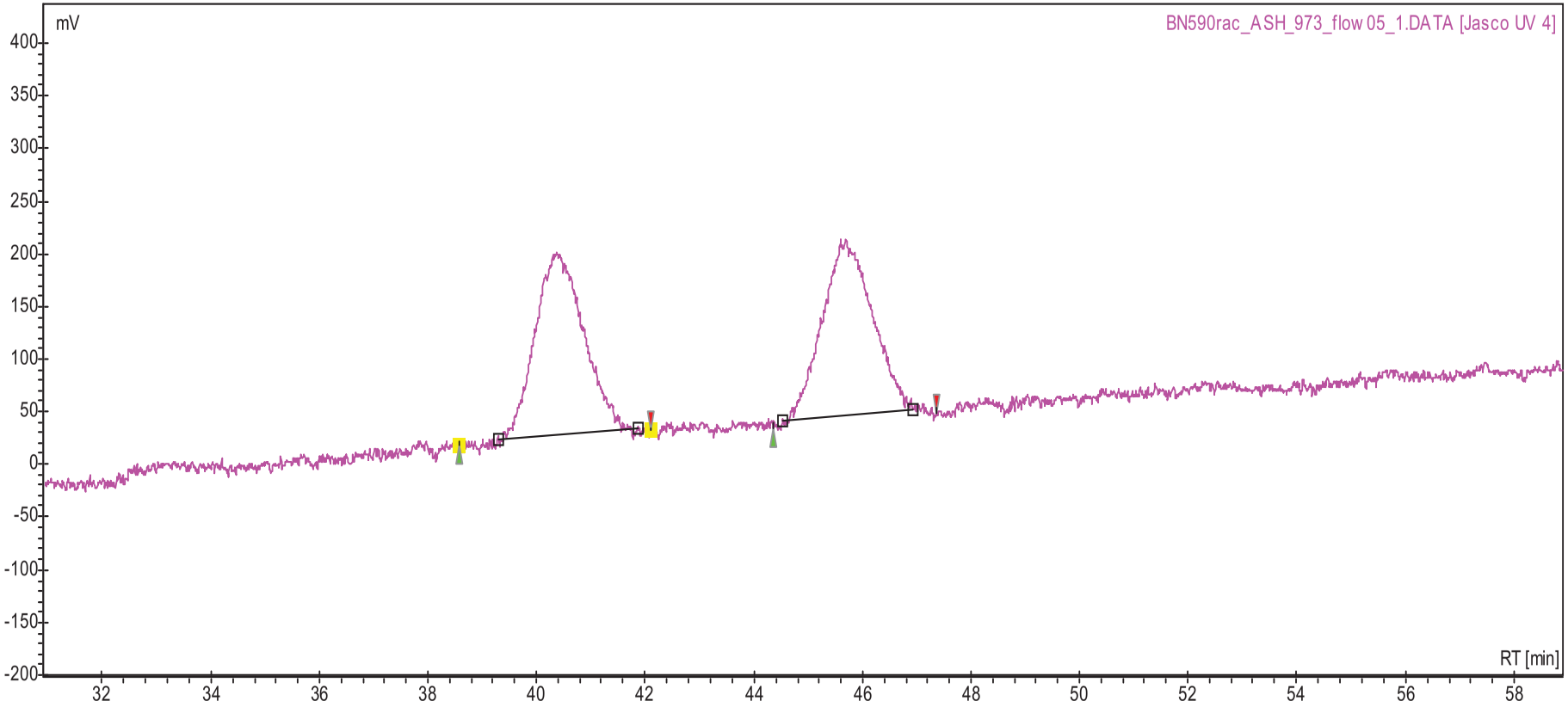
BN590_ASH_973_flow05_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	37,990	39,075	40,311	0,000	8,03	73,9	77,4	8,033
2	UNKNOWN	42,925	44,267	46,175	0,000	91,97	765,3	886,2	91,967
Total						100,00	839,2	963,6	100,000

Chromatogram : BN590rac_ASH_973_flow05_1



Data file: BN590rac_ASH_973_flow05_1.DATA
Method: HPLC2_ASH_973flow05_acq80
Date: 04.06.2007 12:51:44



BN590rac_ASH_973_flow05_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	38,586	40,375	42,110	0,000	49,64	173,2	174,0	49,639
2	UNKNOWN	44,358	45,608	47,372	0,000	50,36	165,2	176,6	50,361
Total						100,00	338,3	350,6	100,000

Sample Information

Analyzed : 11.06.2007 18:21:53
Sample Name : BN590

Method

====== Analytical Line 1 ======

[GC-2010]

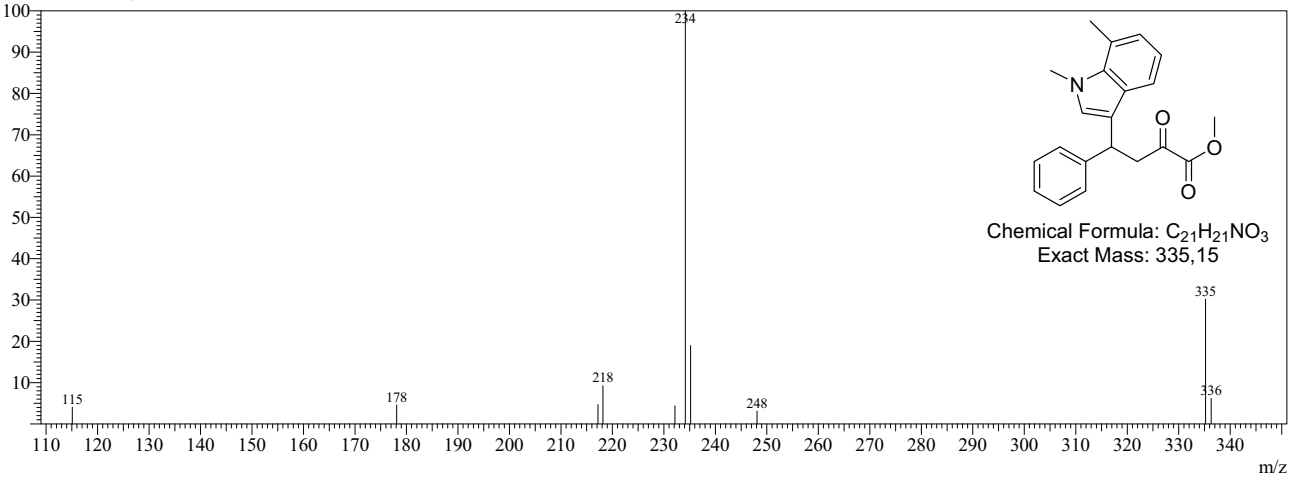
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:12.7(Scan#:1160)
MassPeaks:10 BasePeak:234(10176)
RawMode:Averaged 12.6-12.7(1154-1168)
BG Mode:Averaged 13.6-15.3(1272-1477)

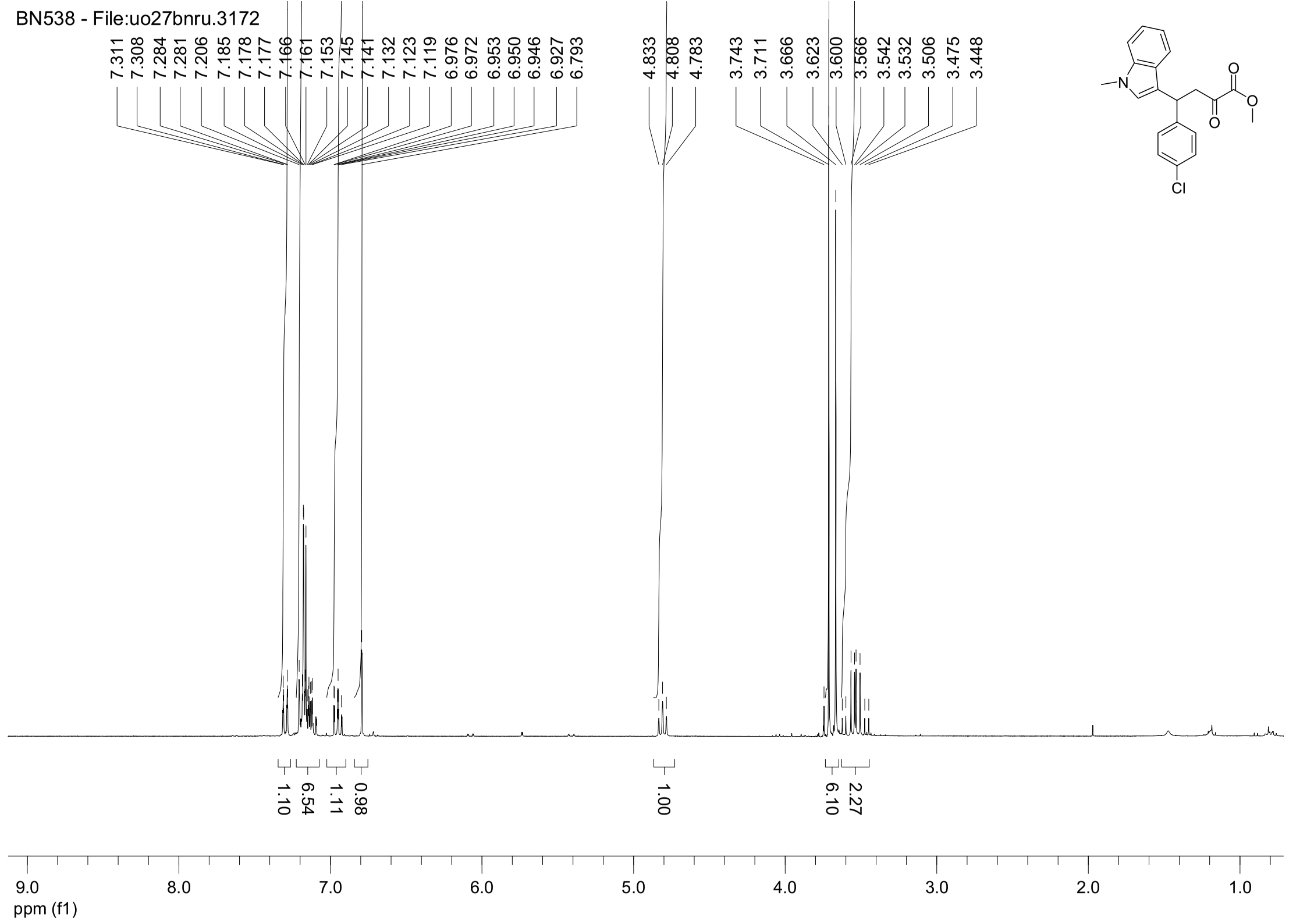


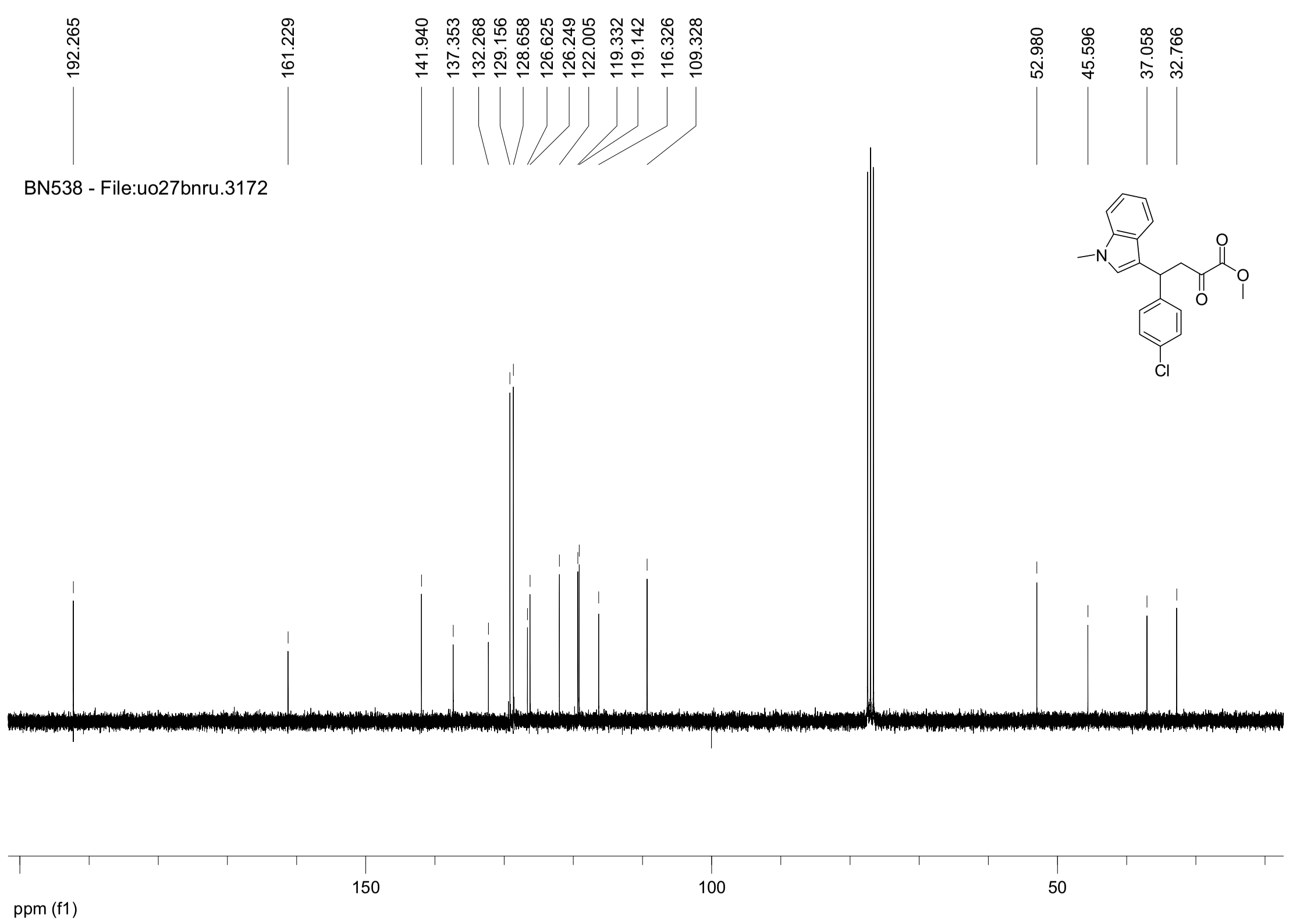
Mass Table

Line#:1 R.Time:12.7(Scan#:1160)
MassPeaks:10 BasePeak:234(10176)
RawMode:Averaged 12.6-12.7(1154-1168)
BG Mode:Averaged 13.6-15.3(1272-1477)

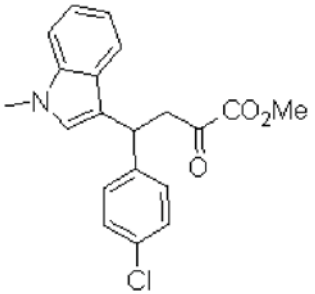
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	115.10	4.08	4	218.15	9.26	7	235.20	18.98	10	336.30	6.20
2	178.10	4.60	5	232.15	4.39	8	248.10	3.09			
3	217.20	4.67	6	234.15	100.00	9	335.20	30.20			

BN538 - File:uo27bnru.3172

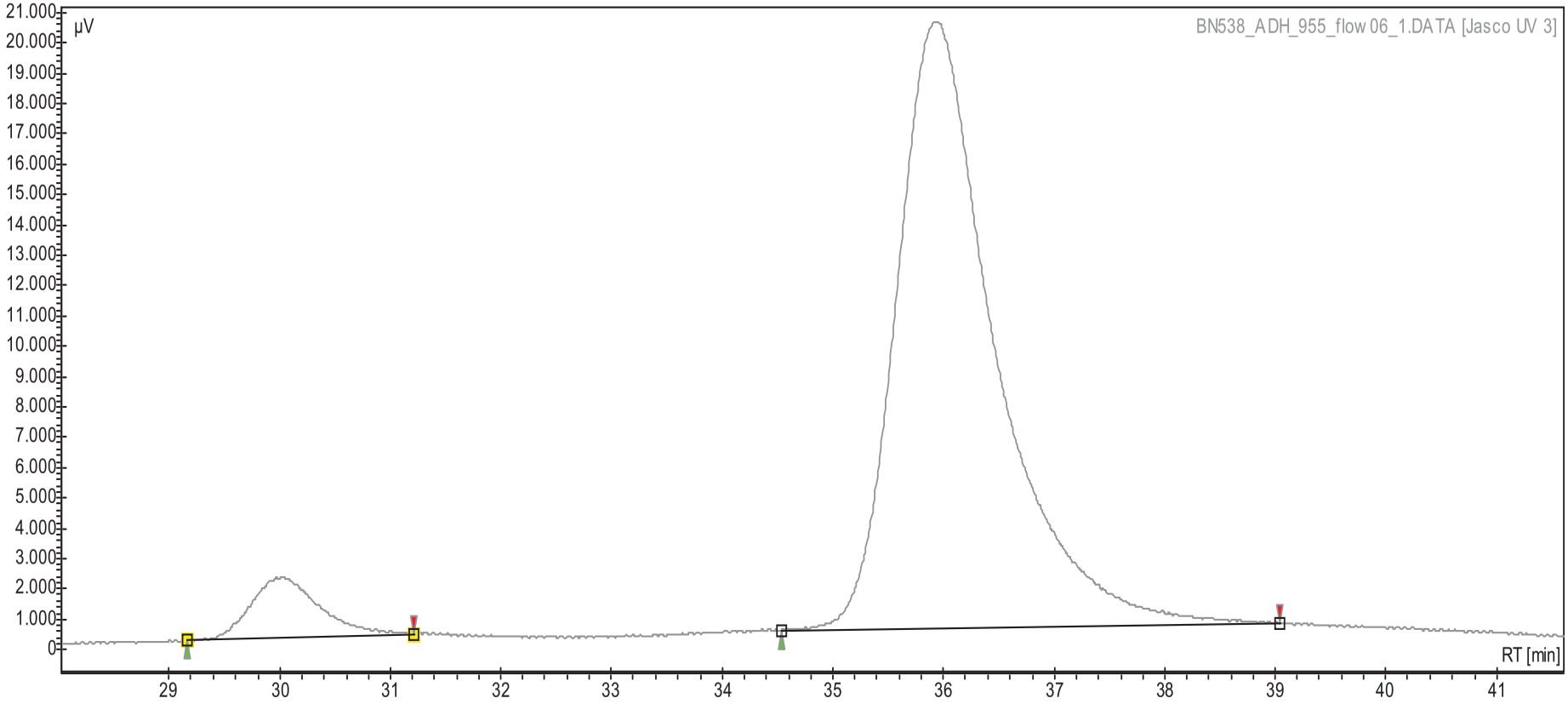



192.265
161.229
141.940
137.353
132.268
129.156
128.658
126.625
126.249
122.005
119.332
119.142
116.326
109.328
52.980
45.596
37.058
32.766
BN538 - File:uo27bnru.3172
N
O
O
O
Cl
150
100
50
ppm (f1)

Chromatogram : BN538_ADH_955_flow06_1



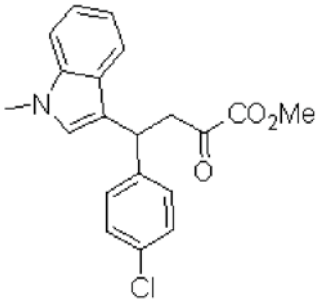
Data file: BN538_ADH_955_flow06_1.DATA
Method: HPLC1_ADH_955_flow06_acq_90
Date: 27.03.2007 22:24:35



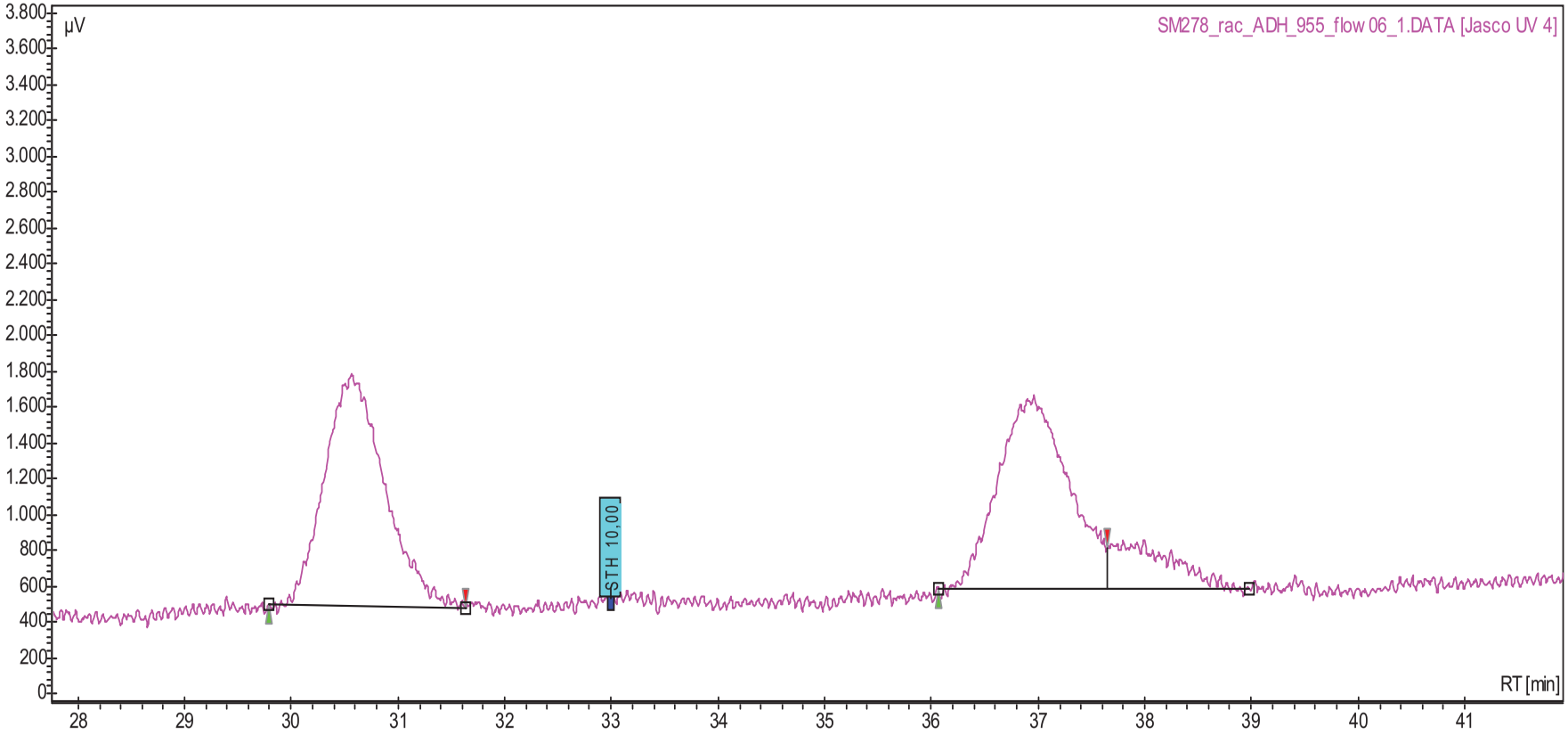
BN538_ADH_955_flow06_1.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	29,165	30,033	31,218	0,000	6,50	2002,1	1435,1	6,500
2	UNKNOWN	34,534	35,925	39,034	0,000	93,50	20000,5	20644,5	93,500
Total						100,00	22002,6	22079,6	100,000

Chromatogram : SM278_rac_ADH_955_flow06_1



Data file: SM278_rac_ADH_955_flow06_1.DATA
Method: HPLC1_ADH_955_flow06_acq_90
Date: 26.03.2007 11:16:24



SM278_rac_ADH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[%]	[µV]	[µV.Min]	[%]
1	UNKNOWN	29,788	30,575	31,633	0,000	0,00	1282,4	840,6	50,816
2	UNKNOWN	36,068	36,900	37,649	0,000	0,00	1055,5	813,6	49,184
Total						0,00	2337,8	1654,2	100,000

Sample Information

Analyzed : 28.03.2007 14:45:35
Sample Name : BN538

Method

====== Analytical Line 1 ======

[GC-2010]

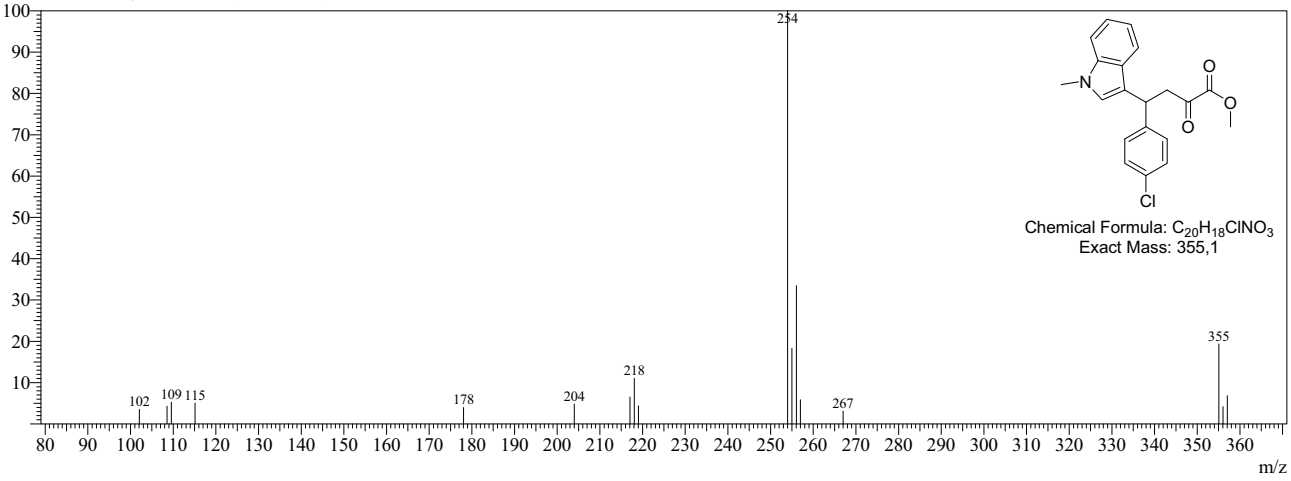
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:13.0(Scan#:1198)
MassPeaks:17 BasePeak:254(183667)
RawMode:Averaged 12.9-13.1(1191-1214)
BG Mode:Averaged 14.1-16.3(1332-1600)

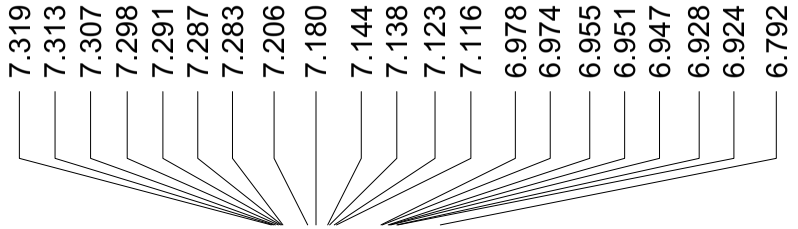


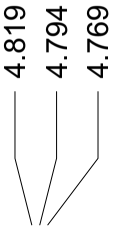
Mass Table

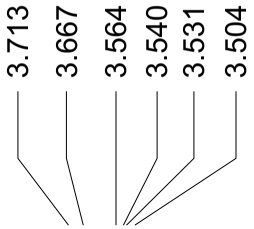
Line#:1 R.Time:13.0(Scan#:1198)
MassPeaks:17 BasePeak:254(183667)
RawMode:Averaged 12.9-13.1(1191-1214)
BG Mode:Averaged 14.1-16.3(1332-1600)

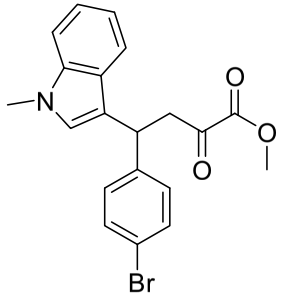
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	102.05	3.53	6	204.00	4.81	11	255.00	18.28	16	356.05	4.20
2	108.55	4.31	7	217.05	6.50	12	256.05	33.46	17	357.05	6.80
3	109.55	5.25	8	218.05	11.05	13	257.00	5.84			
4	115.10	5.04	9	219.05	4.37	14	267.00	3.08			
5	178.05	3.97	10	254.00	100.00	15	355.05	19.35			

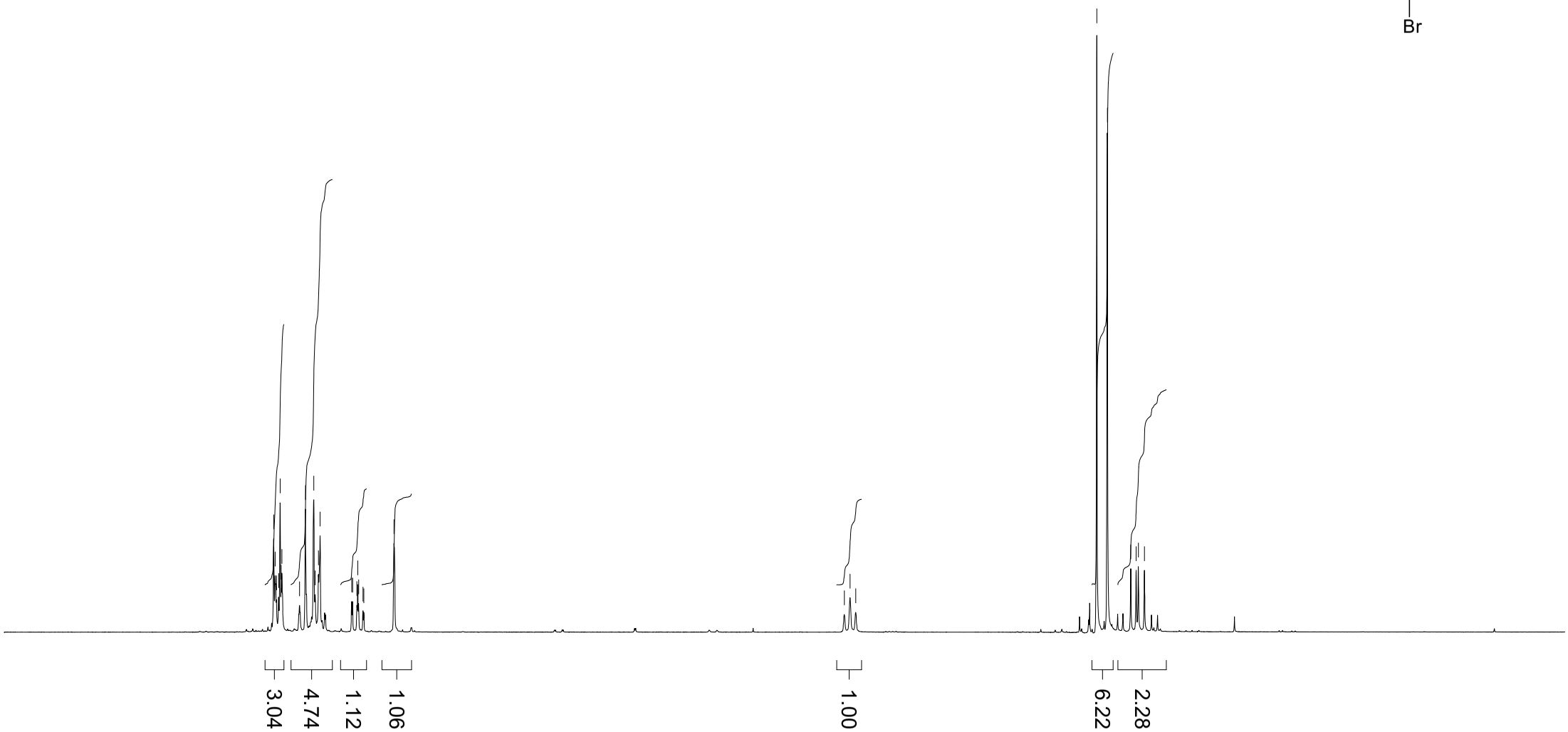
BN 552 - File: up03bnru.3239



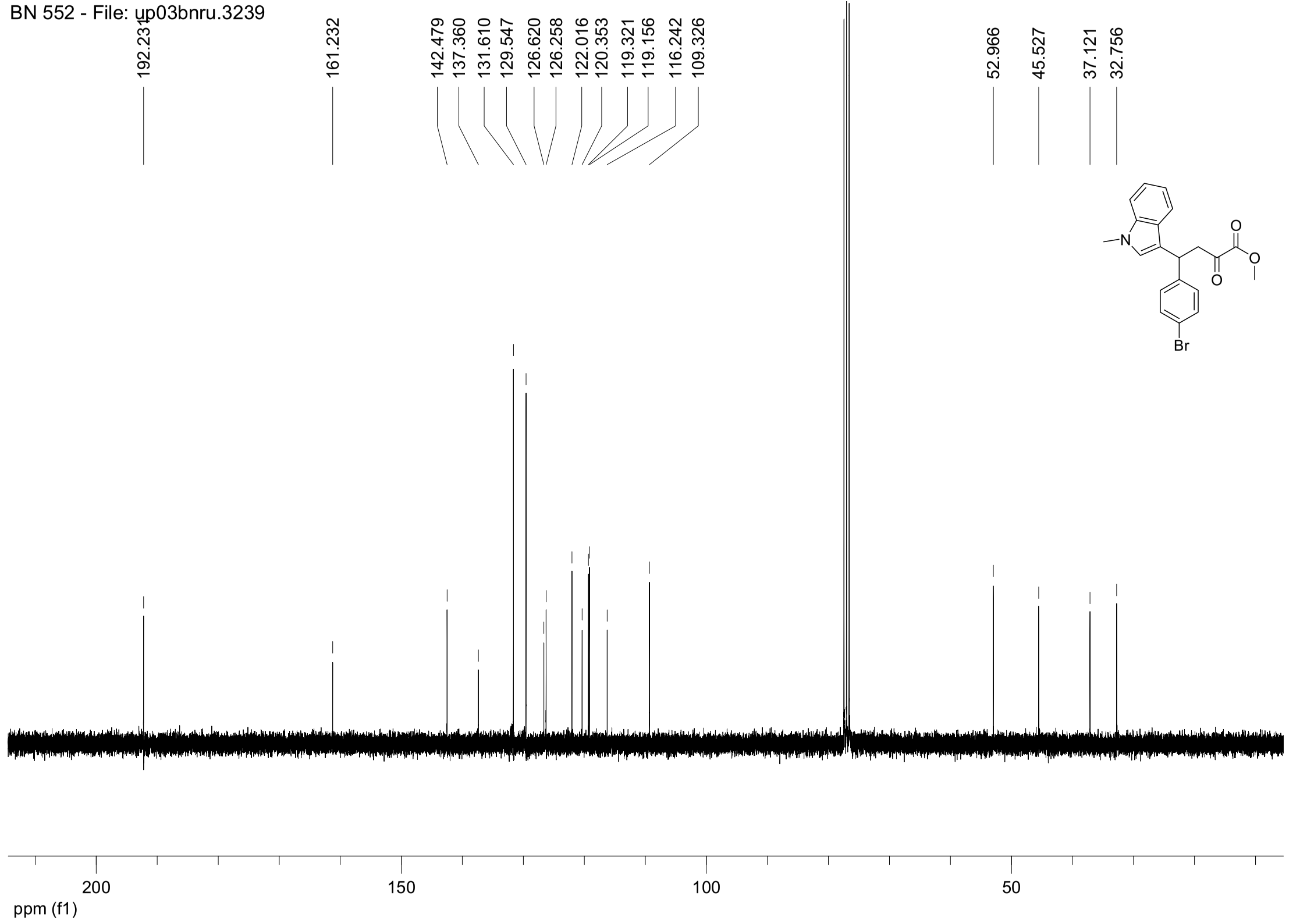






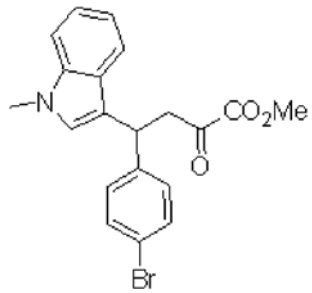


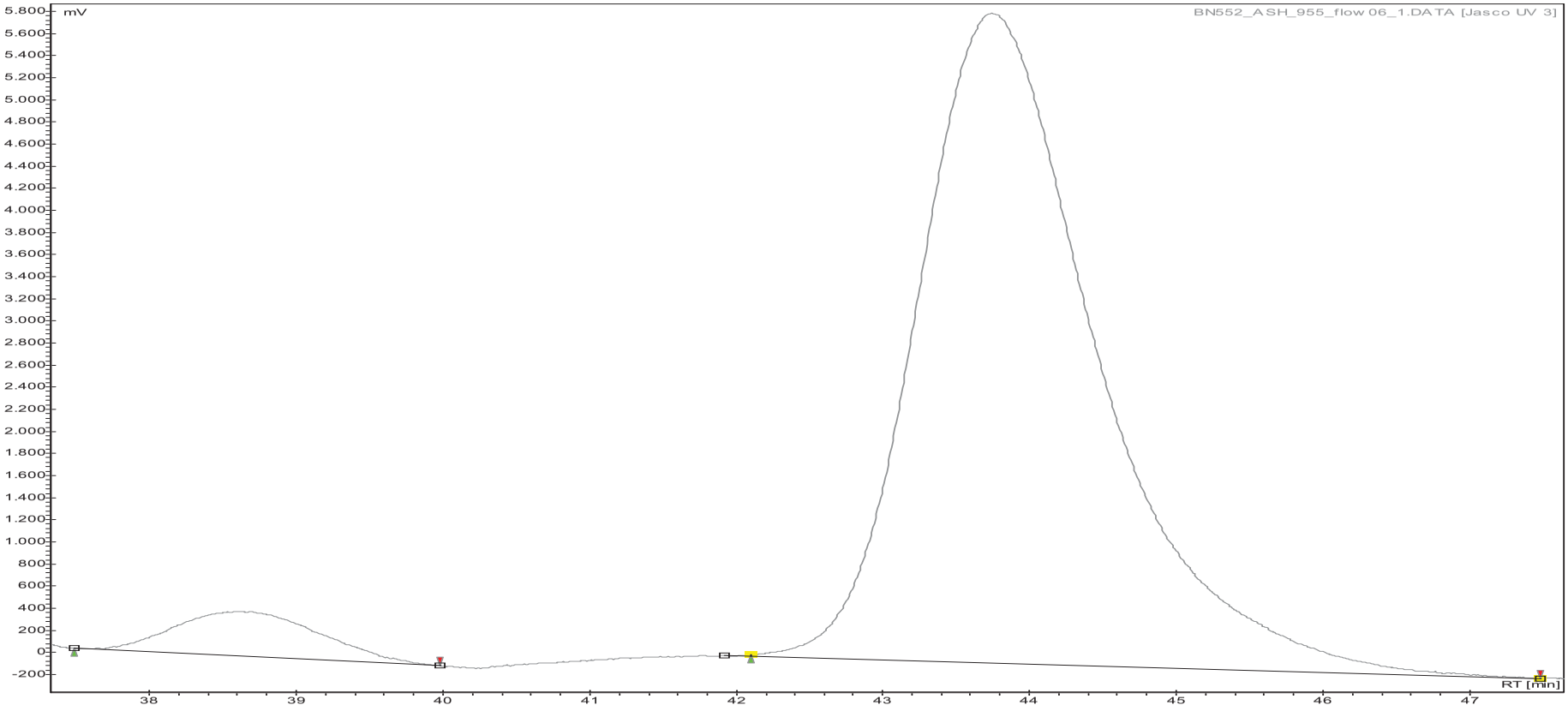
BN 552 - File: up03bnru.3239



Chromatogram : BN552_ASH_955_flow06_1

Data file: BN552_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 22:47:27

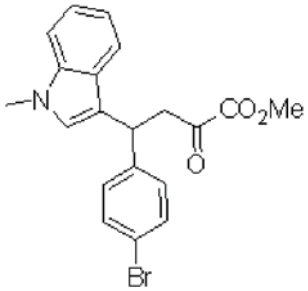




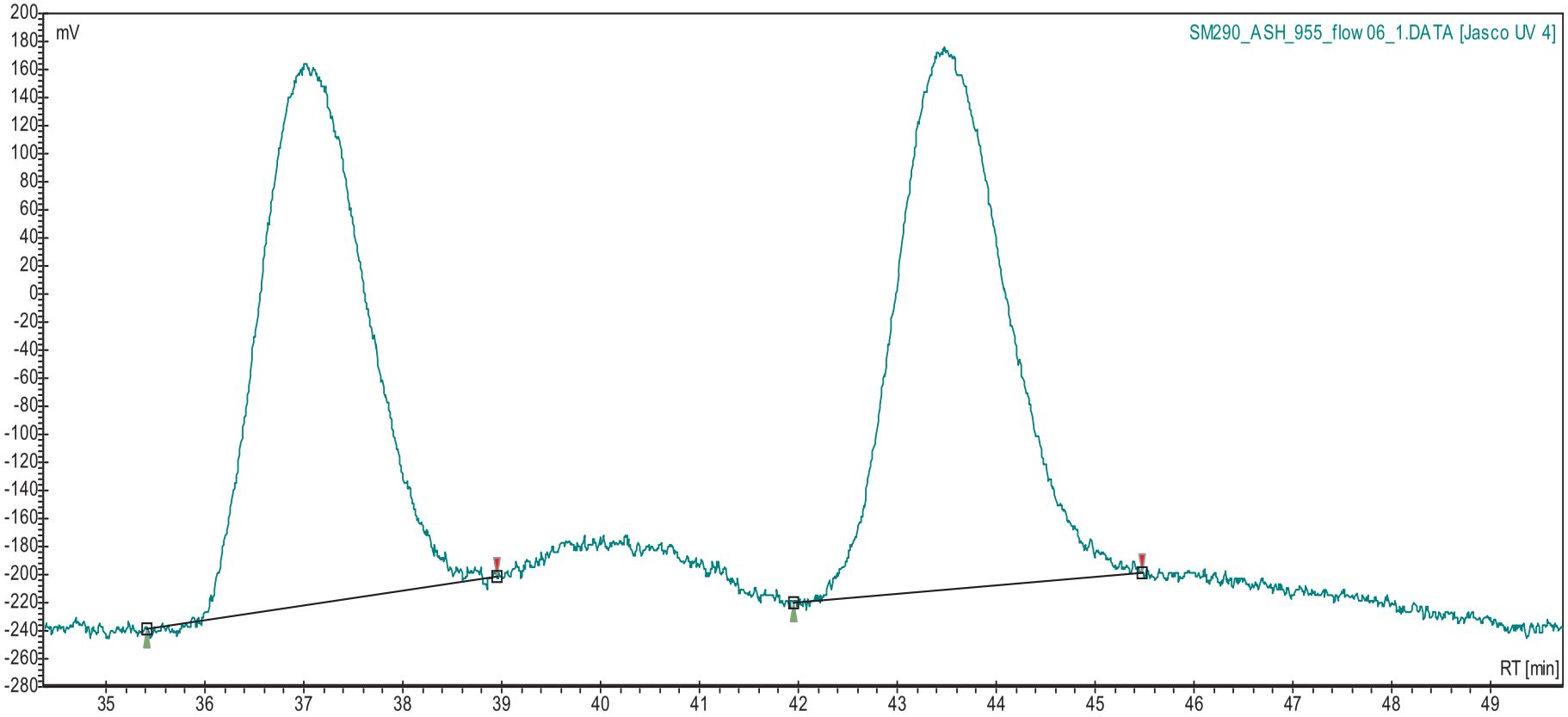
BN552_ASH_955_flow06_1.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
2	UNKNOWN	37,480	38,658	39,981	0,000	5,29	401,6	463,8	5,293
1	UNKNOWN	42,099	43,742	47,484	0,000	94,71	5879,3	8299,4	94,707
Total						100,00	6280,8	8763,2	100,000

Chromatogram : SM290_ASH_955_flow06_1



Data file: SM290_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 11:21:16



SM290_ASH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	35,415	37,017	38,952	0,000	49,12	385,6	474,3	49,117
2	UNKNOWN	41,951	43,475	45,472	0,000	50,88	386,5	491,3	50,883
Total						100,00	772,1	965,6	100,000

Sample Information

Analyzed : 10.04.2007 20:01:31
Sample Name : BN552

Method

====== Analytical Line 1 ======

[GC-2010]

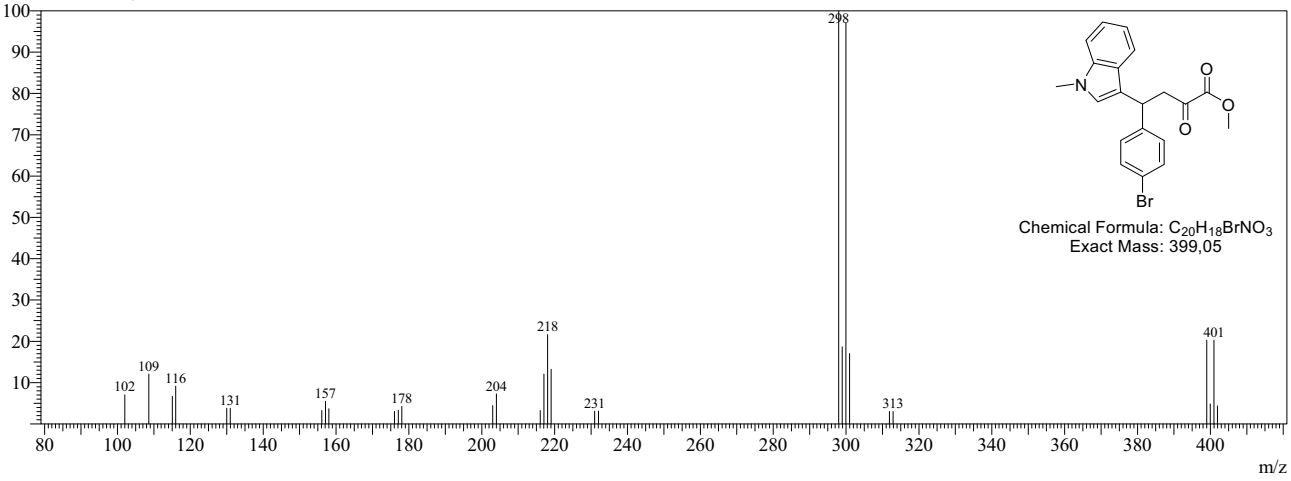
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:13.6(Scan#:1275)
MassPeaks:30 BasePeak:298(126306)
RawMode:Averaged 13.5-13.8(1261-1293)
BG Mode:Averaged 14.7-16.4(1405-1609)

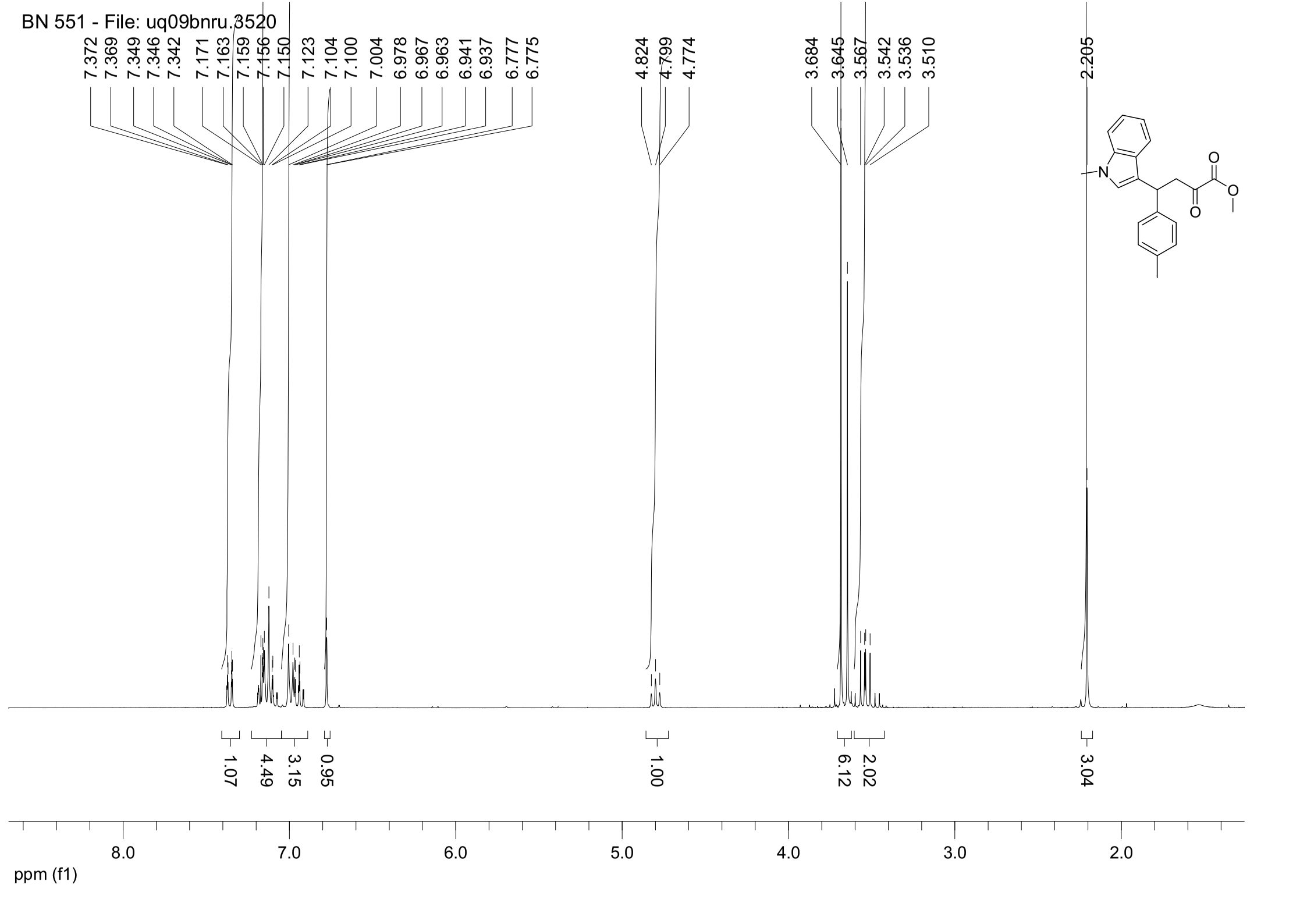


Mass Table

Line#:1 R.Time:13.6(Scan#:1275)
MassPeaks:30 BasePeak:298(126306)
RawMode:Averaged 13.5-13.8(1261-1293)
BG Mode:Averaged 14.7-16.4(1405-1609)

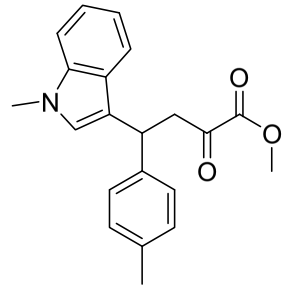
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	102.00	7.07	9	158.00	3.67	17	218.05	21.62	25	311.90	3.01
2	108.60	12.10	10	176.05	3.03	18	219.05	13.23	26	312.90	3.01
3	115.05	6.70	11	177.10	3.34	19	231.00	3.10	27	399.00	20.28
4	116.00	9.14	12	178.05	4.25	20	232.05	3.08	28	399.95	4.84
5	130.00	3.78	13	203.00	4.45	21	297.95	100.00	29	400.95	20.31
6	130.95	3.78	14	204.00	7.29	22	298.95	18.68	30	401.95	4.38
7	156.10	3.24	15	216.05	3.26	23	299.95	97.17			
8	157.10	5.46	16	217.05	12.08	24	300.95	17.05			

BN 551 - File: uq09bnru.3520



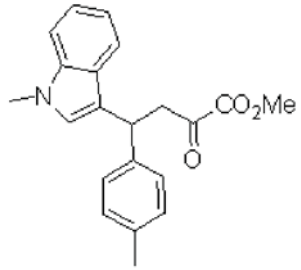
BN 551 - File: uq09bnru.3520

192.707
161.375
140.330
137.333
136.030
129.208
127.595
126.846
126.276
121.803
119.498
118.972
117.057
109.197
52.841
45.903
37.339
32.705
20.993

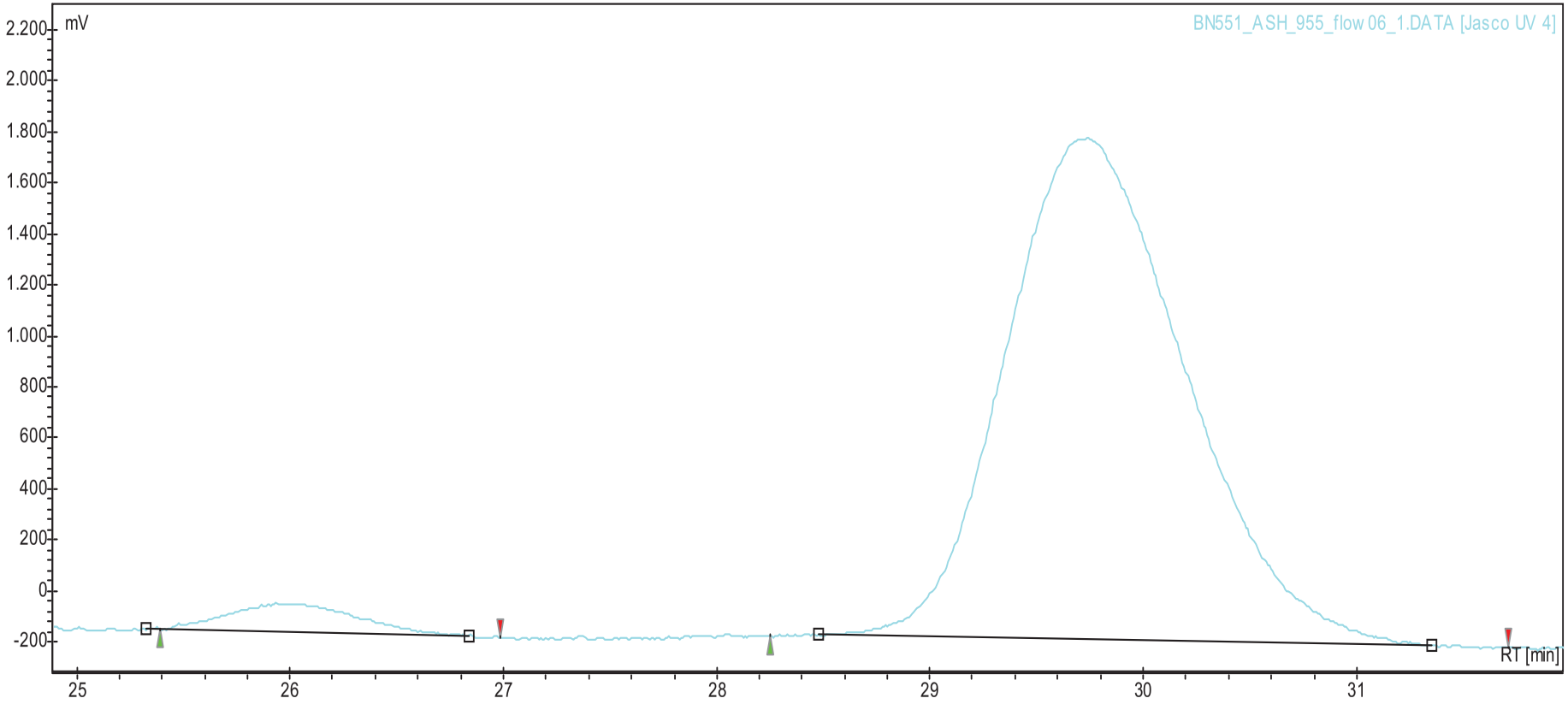


200
150
100
50
ppm (f1)

Chromatogram : BN551_ASH_955_flow06_1

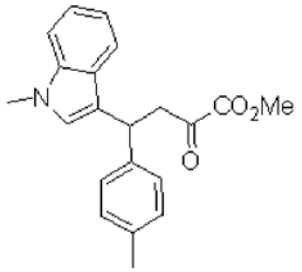


Data file: BN551_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 21:14:51



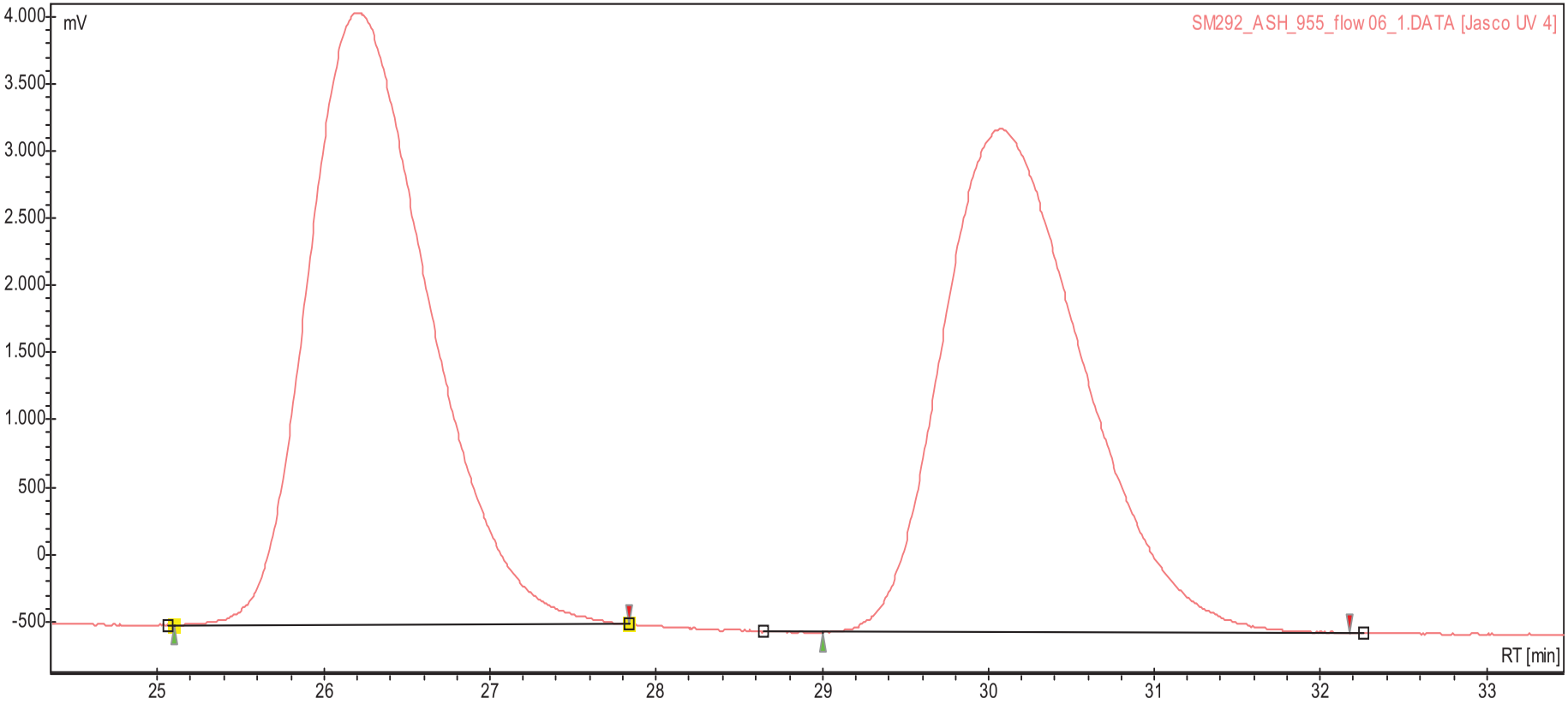
BN551_ASH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	25,389	26,008	26,984	0,000	3,88	110,3	77,0	3,875
2	UNKNOWN	28,250	29,733	31,712	0,000	96,12	1965,1	1910,0	96,125
Total						100,00	2075,4	1987,0	100,000



Chromatogram : SM292_ASH_955_flow06_1

Data file: SM292_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 14:26:30



SM292_ASH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	25,103	26,208	27,842	0,000	51,00	4561,0	3822,7	50,996
2	UNKNOWN	29,004	30,075	32,178	0,000	49,00	3750,0	3673,4	49,004
Total						100,00	8311,0	7496,2	100,000

Sample Information

Analyzed : 10.04.2007 19:34:29
Sample Name : BN551

Method

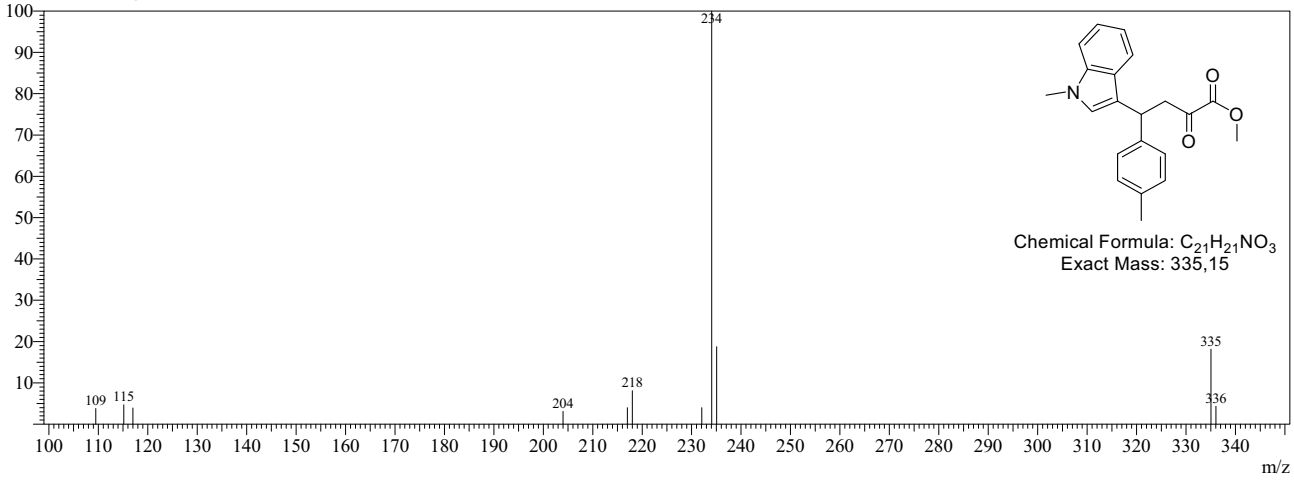
====== Analytical Line 1 ======

[GC-2010]

Column Oven Temp.	:80.0 °C	
Injection Temp.	:280.00 °C	
Injection Mode	:Split	
Flow Control Mode	:Linear Velocity	
Pressure	:34.3 kPa	
Total Flow	:16.9 mL/min	
Column Flow	:0.66 mL/min	
Linear Velocity	:30.0 cm/sec	
Purge Flow	:3.0 mL/min	
Split Ratio	:20.0	
Oven Temp. Program		
Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:12.4(Scan#:1129)
MassPeaks:11 BasePeak:234(625278)
RawMode:Averaged 12.3-12.5(1121-1143)
BG Mode:Averaged 13.6-17.4(1277-1734)

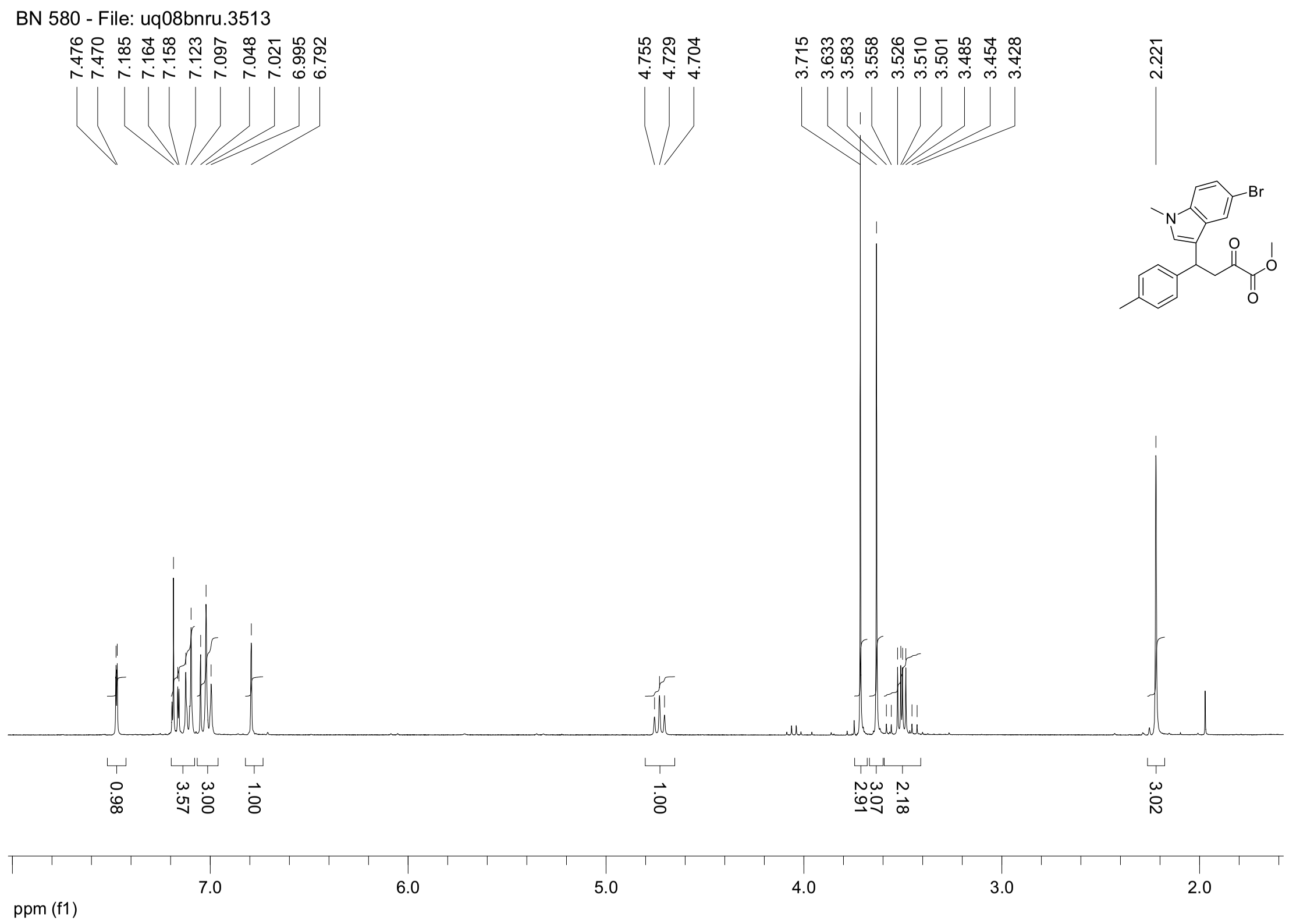


Mass Table

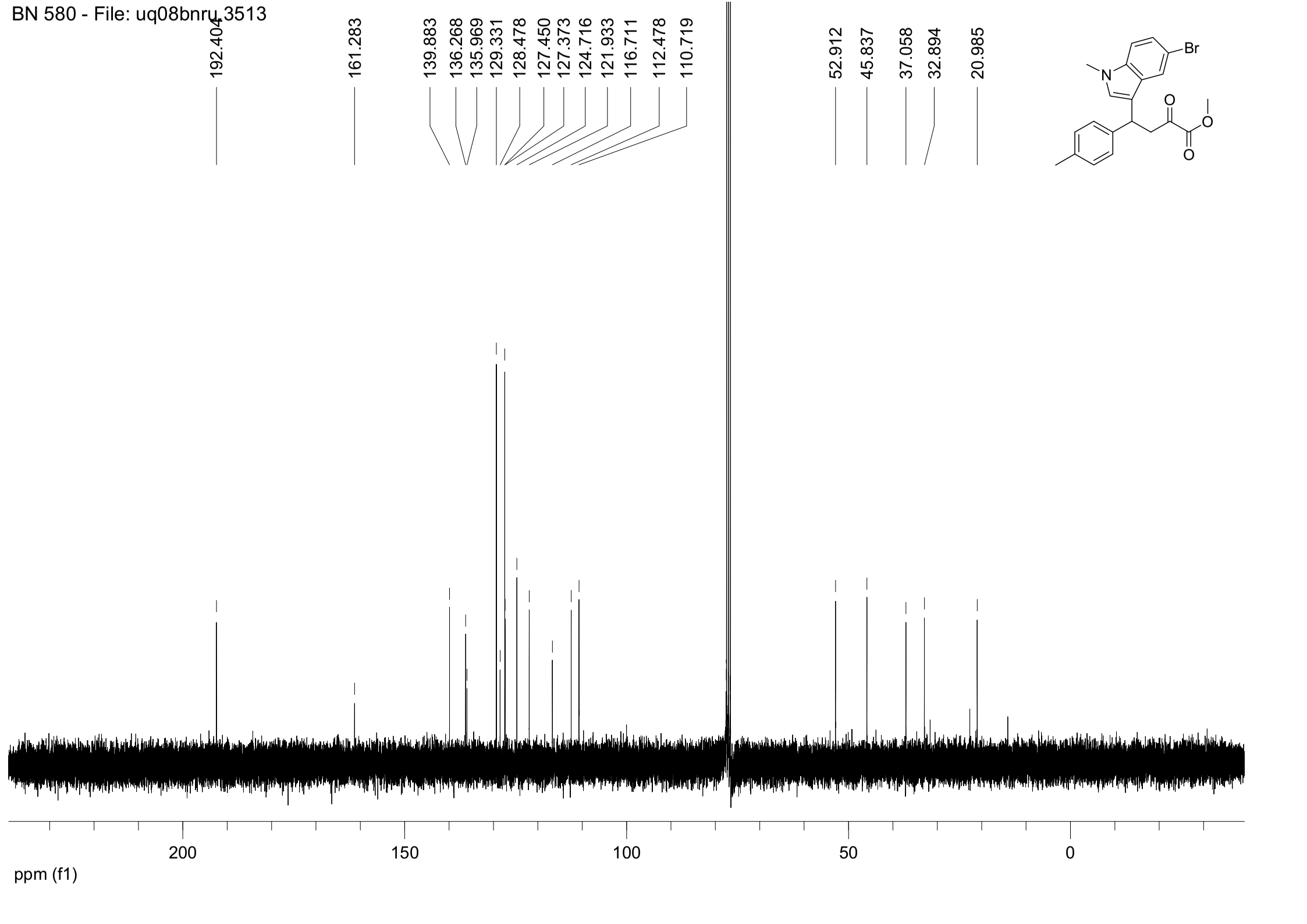
Line#:1 R.Time:12.4(Scan#:1129)
MassPeaks:11 BasePeak:234(625278)
RawMode:Averaged 12.3-12.5(1121-1143)
BG Mode:Averaged 13.6-17.4(1277-1734)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	109.50	3.80	4	204.00	3.10	7	232.05	3.98	10	335.05	18.18
2	115.15	4.68	5	217.00	3.98	8	234.05	100.00	11	336.05	4.34
3	117.00	3.92	6	218.00	8.11	9	235.05	18.75			

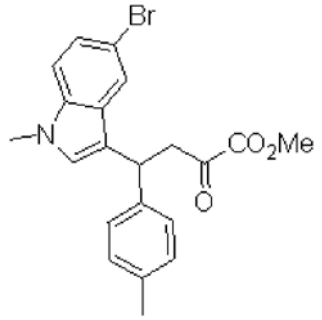
BN 580 - File: uq08bnru.3513



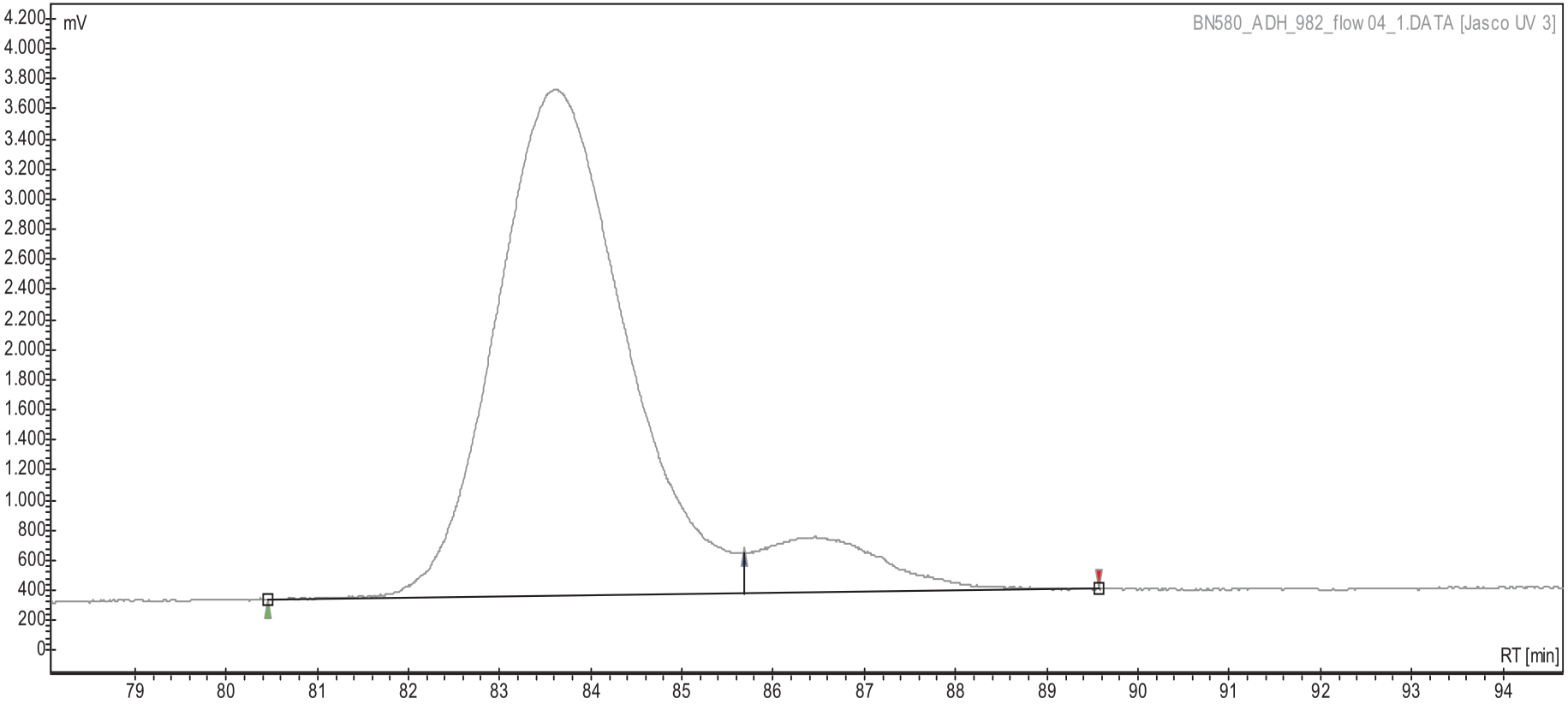
BN 580 - File: uq08bnru.3513



Chromatogram : BN580_ADH_982_flow04_1



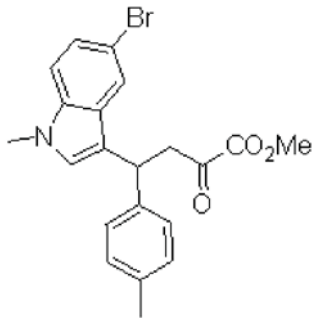
Data file: BN580_ADH_982_flow04_1.DATA
Method: HPLC2_ADH_982_flow04_acq150
Date: 21.05.2007 12:24:03



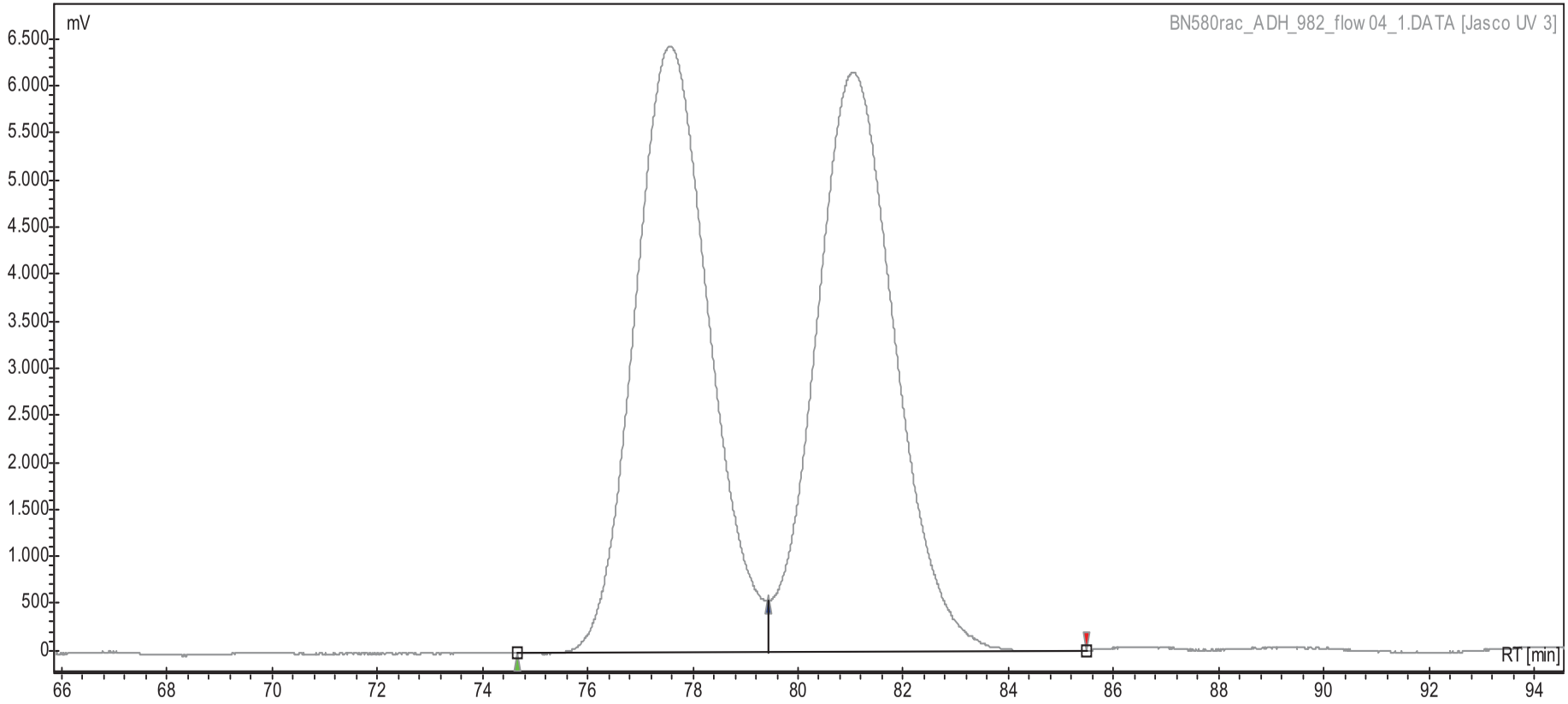
BN580_ADH_982_flow04_1.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	80,460	83,617	85,683	0,000	90,28	3368,4	5504,3	90,282
2	UNKNOWN	85,683	86,458	89,571	0,000	9,72	369,5	592,5	9,718
Total						100,00	3737,9	6096,8	100,000

Chromatogram : BN580rac_ADH_982_flow04_1

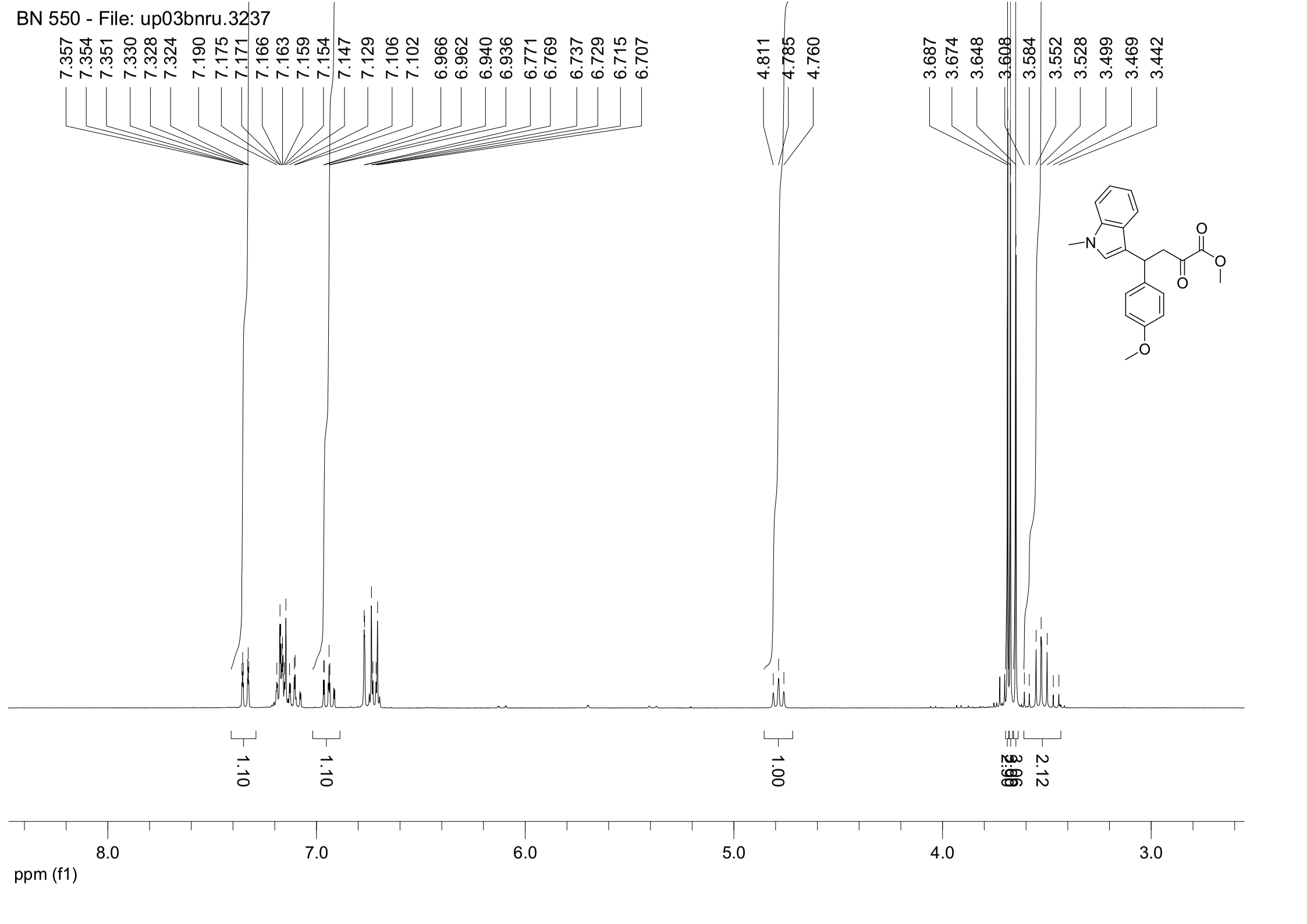


Data file: BN580rac_ADH_982_flow04_1.DATA
Method: HPLC2_ADH_982_flow04_acq150
Date: 04.06.2007 23:11:39

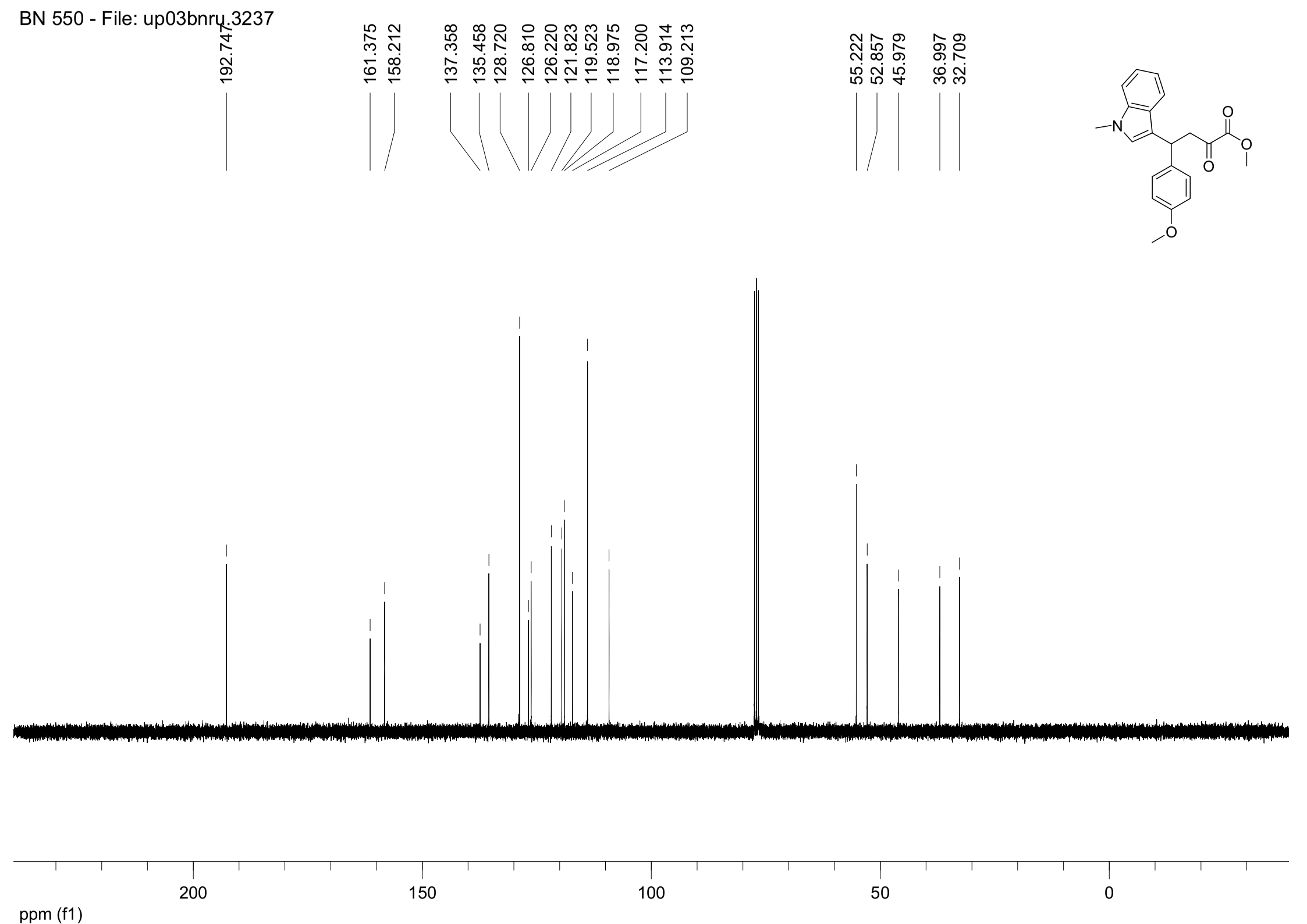


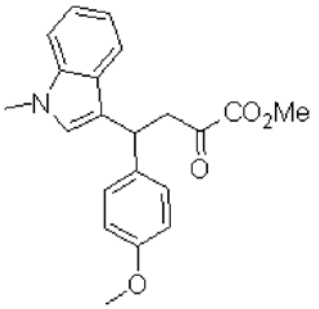
BN580rac_ADH_982_flow04_1.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	74,662	77,558	79,436	0,000	49,67	6447,2	10588,7	49,665
2	UNKNOWN	79,436	81,050	85,486	0,000	50,33	6158,3	10731,5	50,335
Total						100,00	12605,5	21320,3	100,000


BN 550 - File: up03bnru.3237
7.357
7.354
7.351
7.330
7.328
7.324
7.190
7.175
7.171
7.166
7.163
7.159
7.154
7.147
7.129
7.106
7.102
6.966
6.962
6.940
6.936
6.771
6.769
6.737
6.729
6.715
6.707
4.811
4.785
4.760
3.687
3.674
3.648
3.608
3.584
3.552
3.528
3.499
3.469
3.442
1.10
1.10
1.00
2.12
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

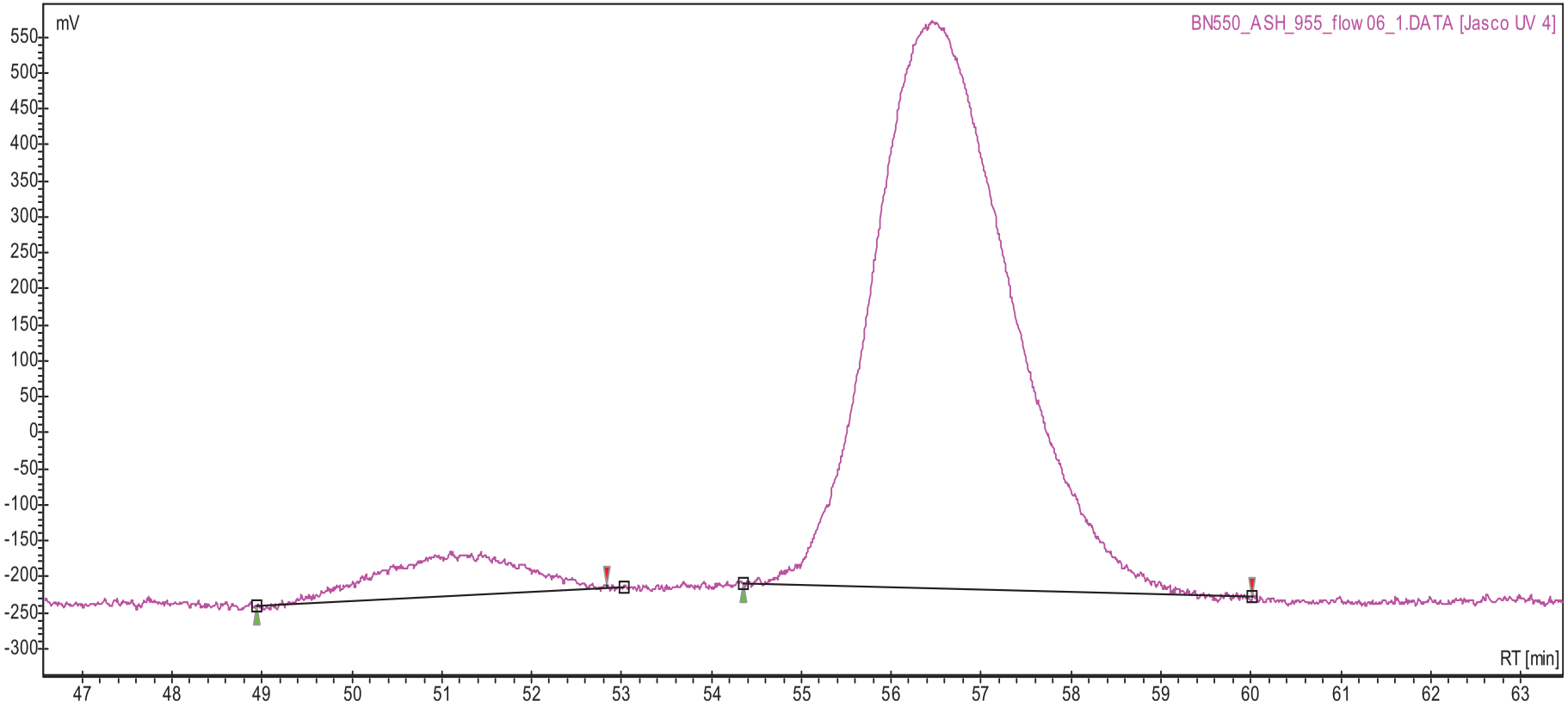
BN 550 - File: up03bnru.3237





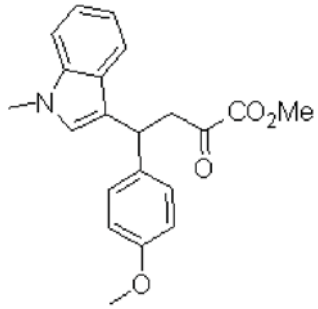
Chromatogram : BN550_ASH_955_flow06_1

Data file: BN550_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 19:42:16



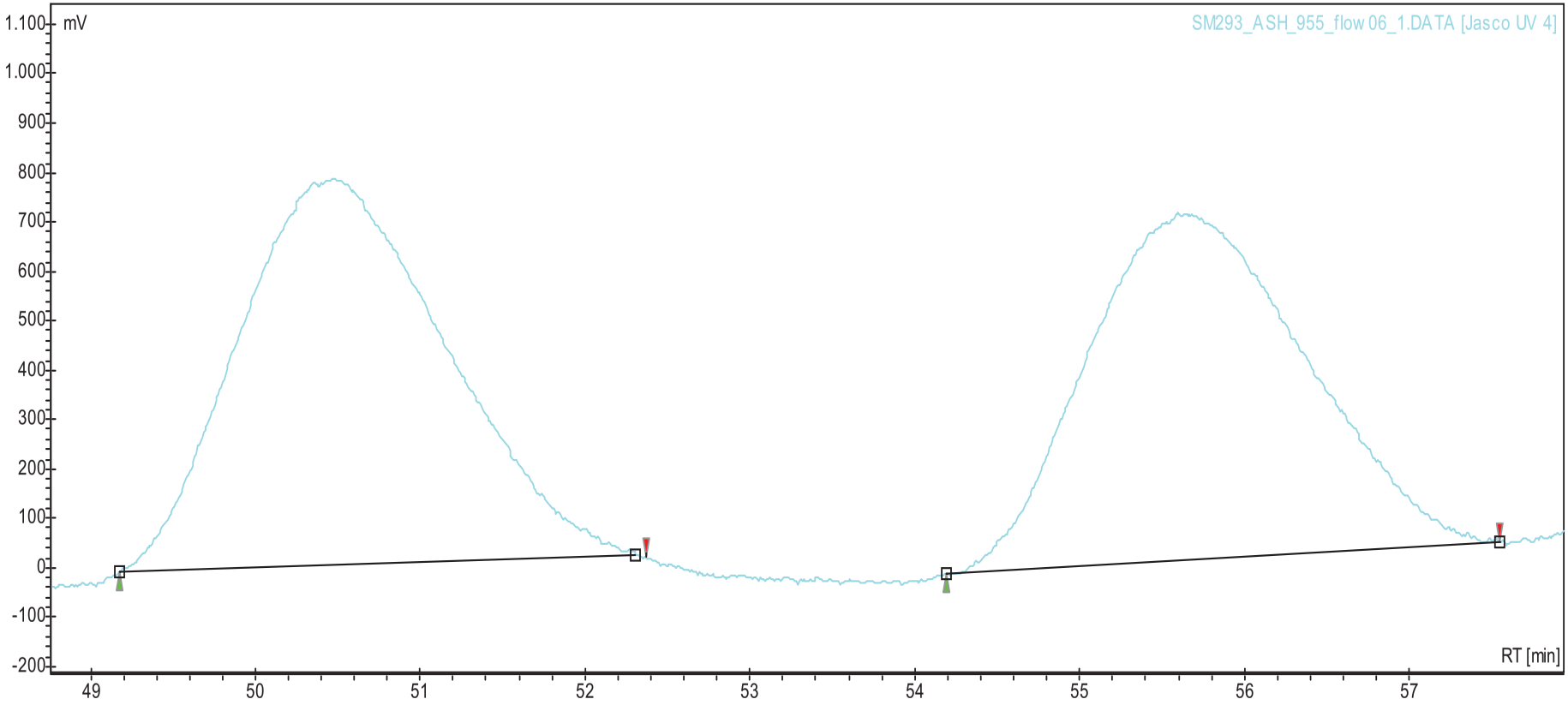
BN550_ASH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	48,937	51,100	52,831	0,000	7,29	61,6	108,7	7,294
2	UNKNOWN	54,357	56,475	60,018	0,000	92,71	785,9	1381,6	92,706
Total						100,00	847,5	1490,4	100,000



Chromatogram : SM293_ASH_955_flow06_1

Data file: SM293_ASH_955_flow06_1.DATA
Method: HPLC2_ASH_955_flow06_acq90
Date: 05.04.2007 15:59:08



SM293_ASH_955_flow06_1.DATA [Jasco UV 4]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	49,175	50,483	52,371	0,000	51,53	780,6	1139,9	51,535
2	UNKNOWN	54,192	55,600	57,550	0,000	48,47	703,4	1072,0	48,465
Total						100,00	1483,9	2211,9	100,000

Sample Information

Analyzed : 10.04.2007 19:07:26
Sample Name : BN550

Method

====== Analytical Line 1 ======

[GC-2010]

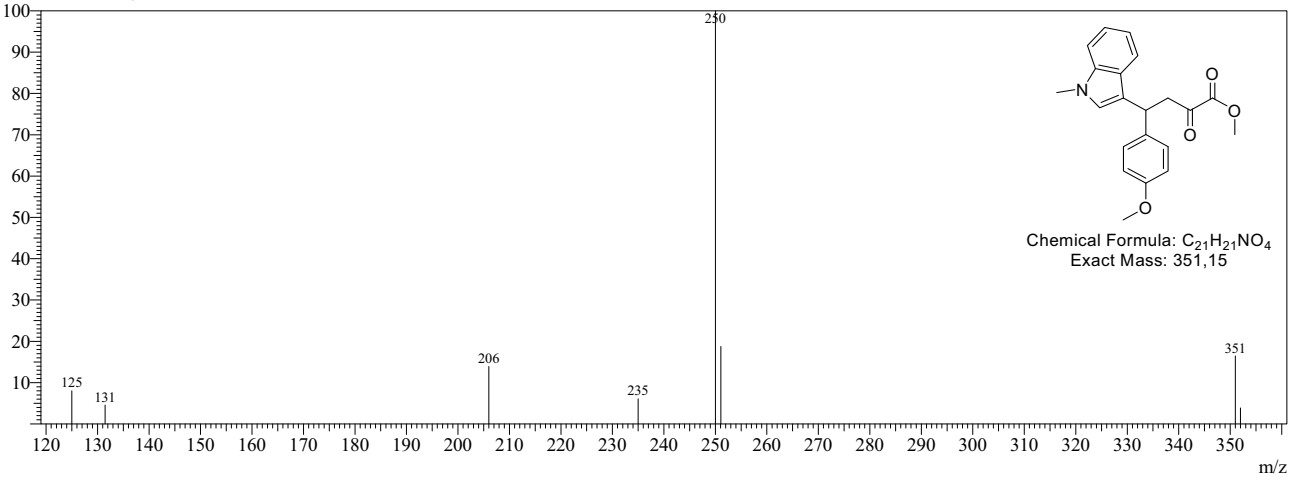
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:13.2(Scan#:1228)
MassPeaks:8 BasePeak:250(505470)
RawMode:Averaged 13.2-13.3(1223-1242)
BG Mode:Averaged 14.5-16.5(1386-1619)

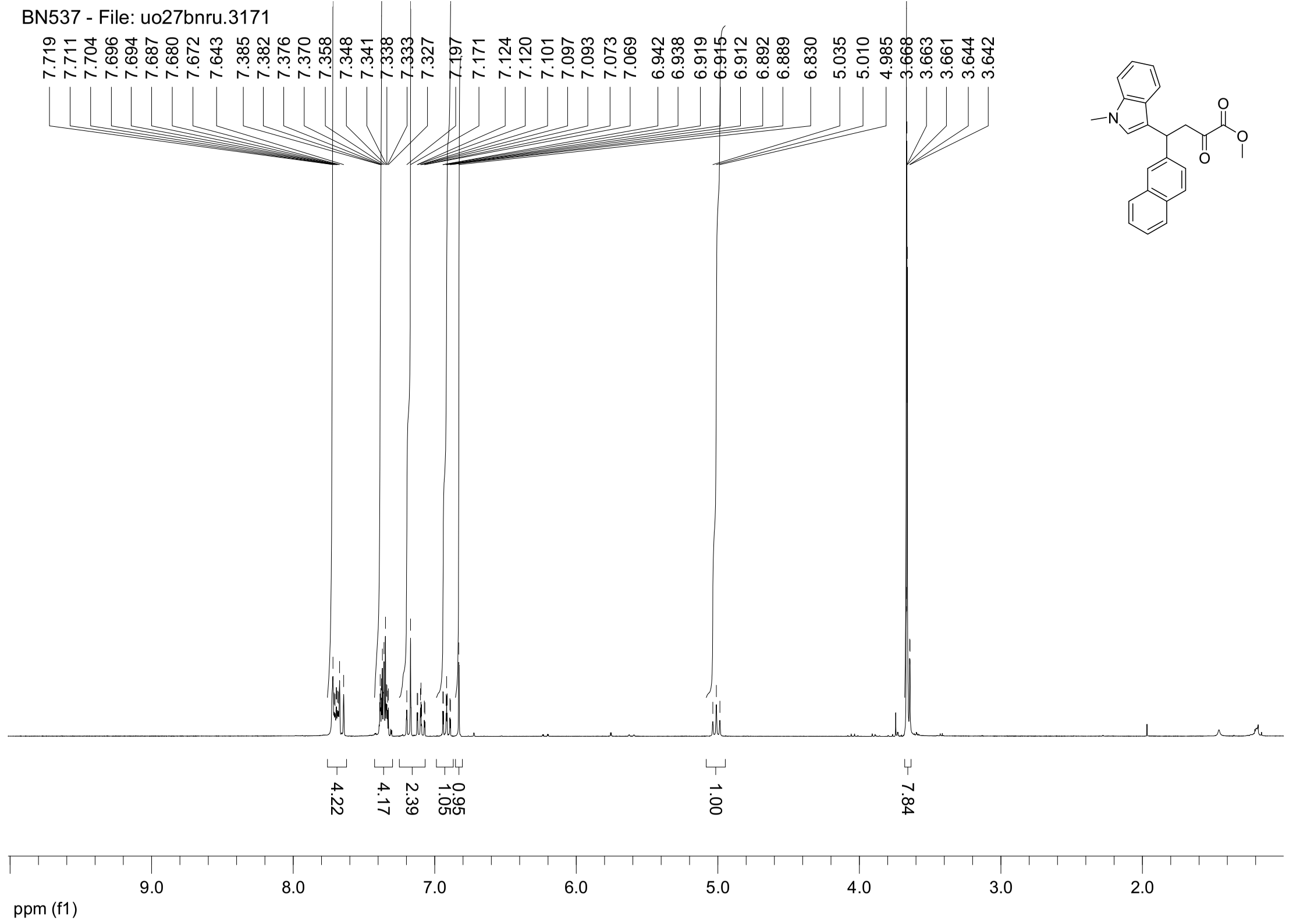


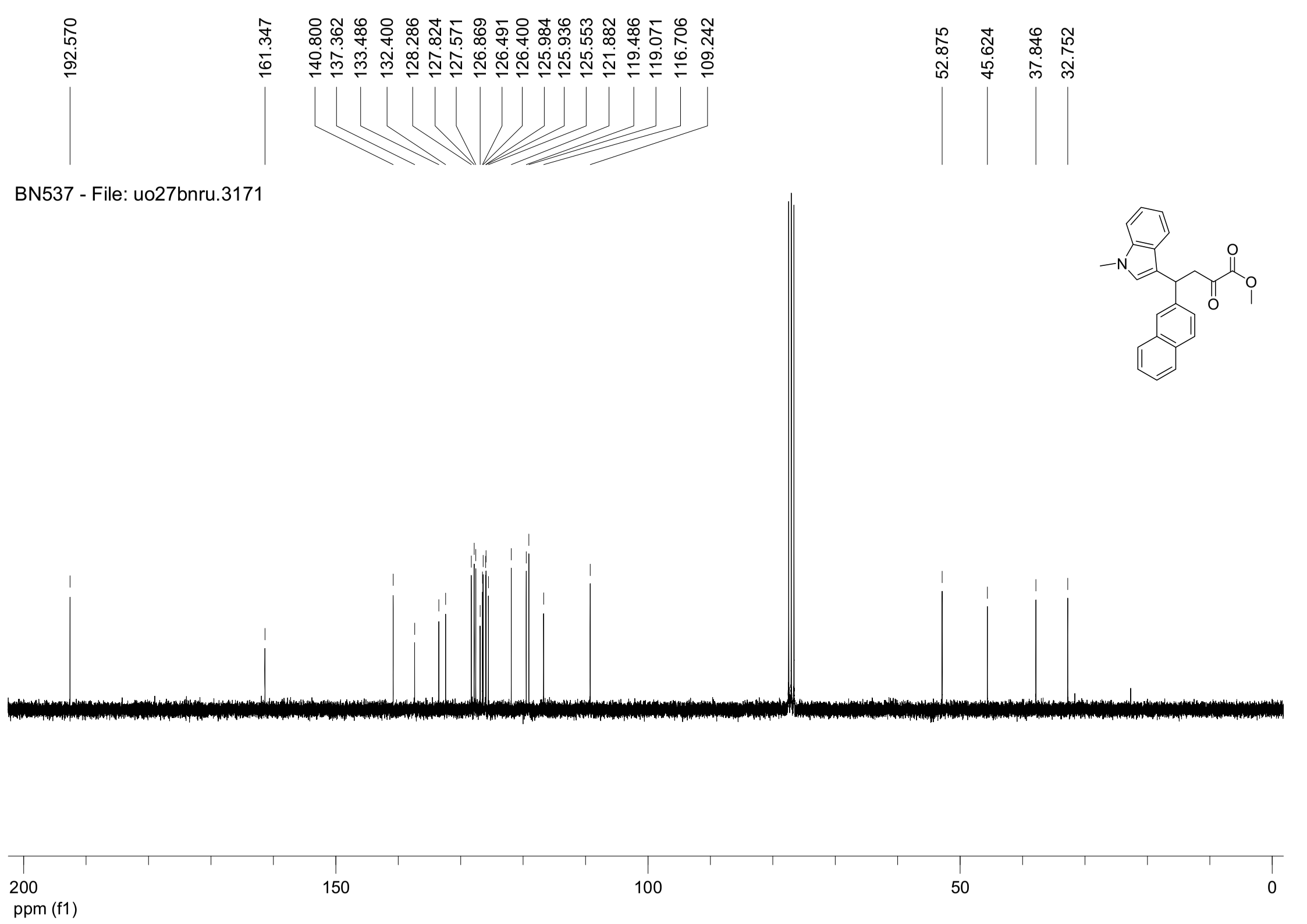
Mass Table

Line#:1 R.Time:13.2(Scan#:1228)
MassPeaks:8 BasePeak:250(505470)
RawMode:Averaged 13.2-13.3(1223-1242)
BG Mode:Averaged 14.5-16.5(1386-1619)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	125.00	8.02	3	206.00	13.89	5	250.00	100.00	7	351.00	16.48
2	131.45	4.58	4	235.00	6.08	6	251.05	18.75	8	352.00	3.87

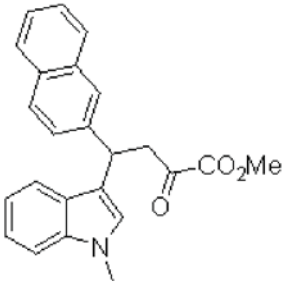
BN537 - File: uo27bnru.3171

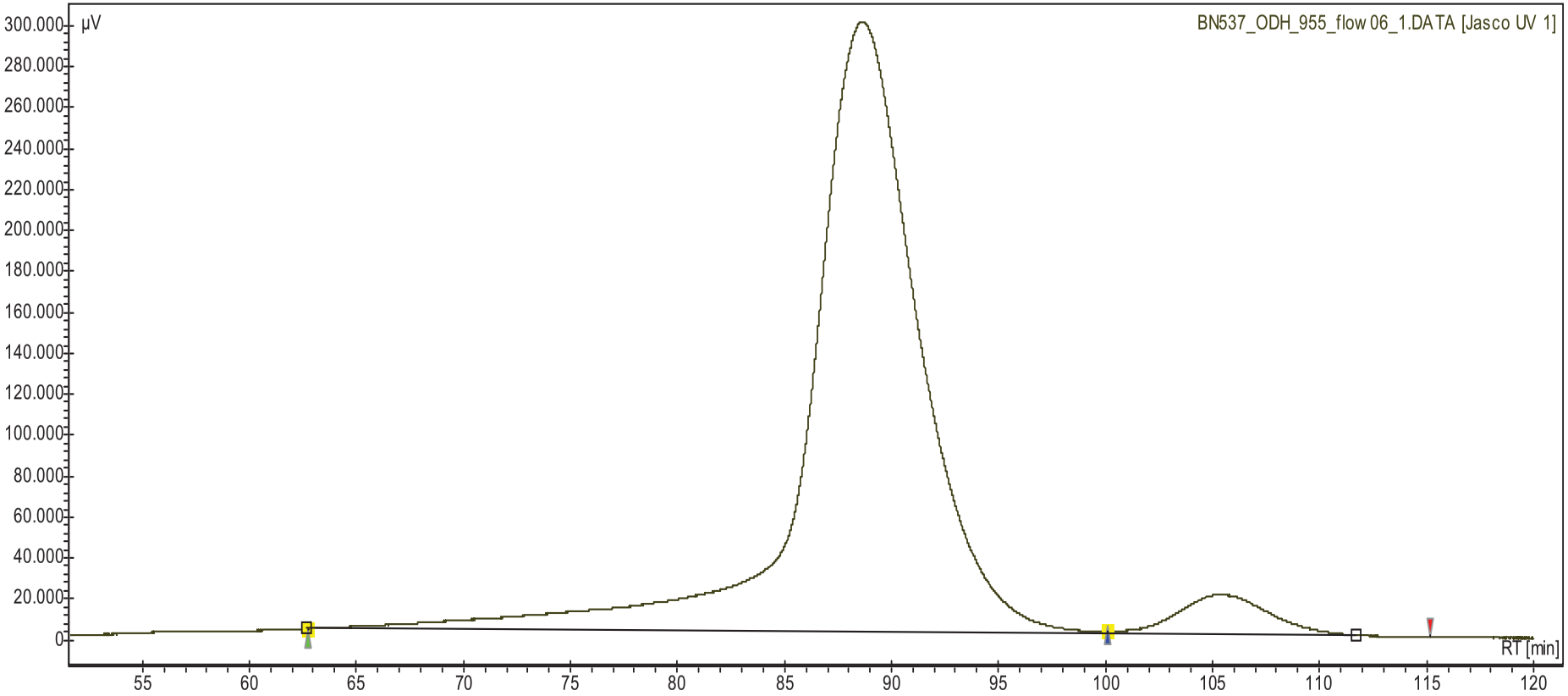



192.570
161.347
140.800
137.362
133.486
132.400
128.286
127.824
127.571
126.869
126.491
126.400
125.984
125.936
125.553
121.882
119.486
119.071
116.706
109.242
52.875
45.624
37.846
32.752
BN537 - File: uo27bnru.3171
N
O
O
O
200
150
100
50
0
ppm (f1)

Chromatogram : BN537_ODH_955_flow06_1

Data file: BN537_ODH_955_flow06_1.DATA
Method: HPLC1_ODH_955_flow06_acq_120
Date: 27.03.2007 15:24:07



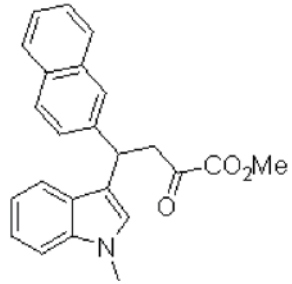


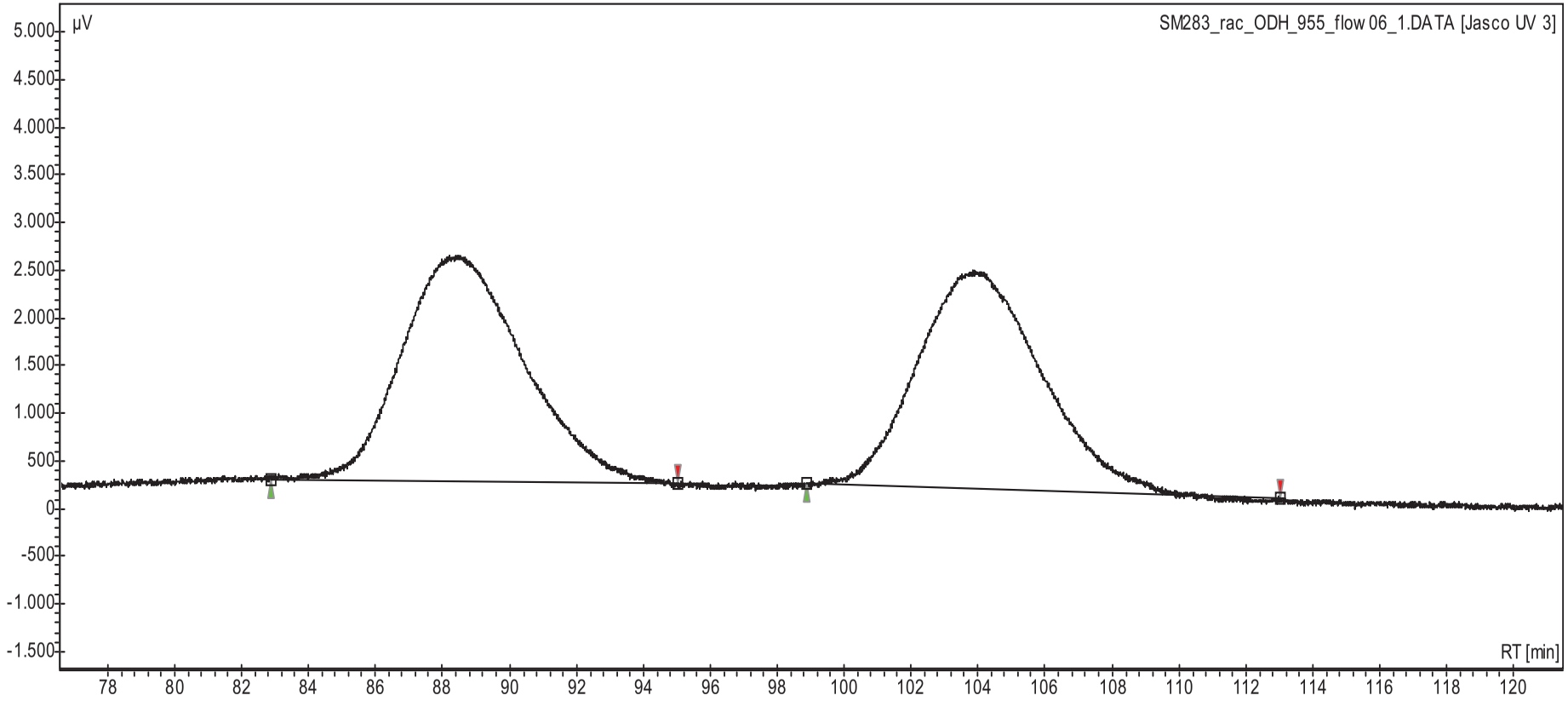
BN537_ODH_955_flow06_1.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	62,746	88,592	100,094	94,934
2	100,094	105,308	115,163	5,066
Total				100,000

Chromatogram : SM283_rac_ODH_955_flow06_1

Data file: SM283_rac_ODH_955_flow06_1.DATA
Method: HPLC1_ODH_955_flow06_acq_135
Date: 27.03.2007 00:01:24





SM283_rac_ODH_955_flow06_1.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	82,874	88,467	95,047	0,000	50,78	2372,1	10021,8	50,780
2	UNKNOWN	98,883	103,908	113,050	0,000	49,22	2282,3	9714,0	49,220
Total						100,00	4654,5	19735,7	100,000

Sample Information

Analyzed : 28.03.2007 14:18:18
Sample Name : BN537

Method

====== Analytical Line 1 ======

[GC-2010]

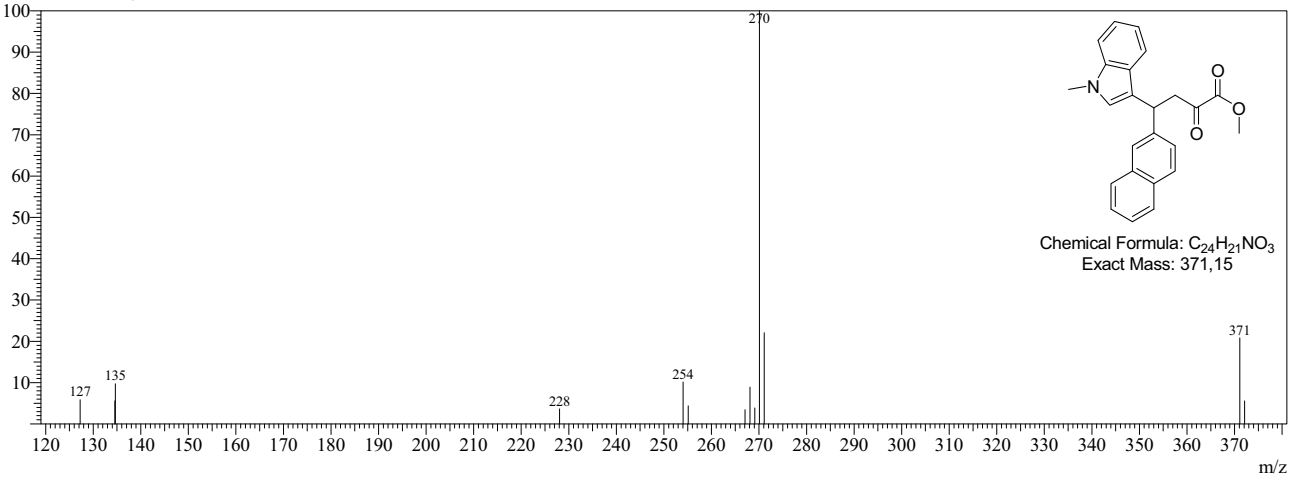
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:16.9 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	310.0	10.00

Spectrum

Line#:1 R.Time:15.7(Scan#:1526)
MassPeaks:13 BasePeak:270(264576)
RawMode:Averaged 15.6-15.8(1517-1539)
BG Mode:Averaged 17.2-18.7(1699-1887)

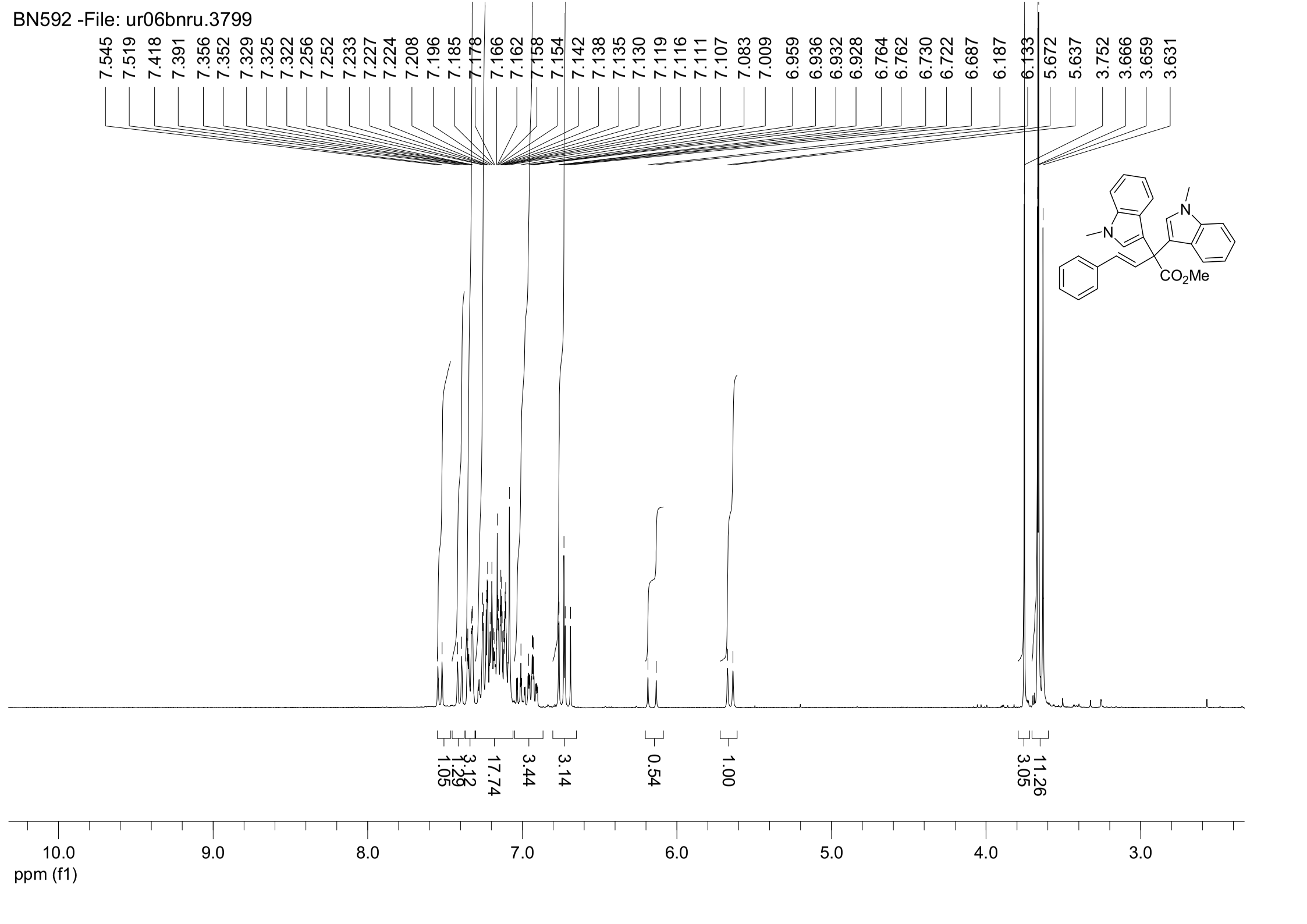


Mass Table

Line#:1 R.Time:15.7(Scan#:1526)
MassPeaks:13 BasePeak:270(264576)
RawMode:Averaged 15.6-15.8(1517-1539)
BG Mode:Averaged 17.2-18.7(1699-1887)

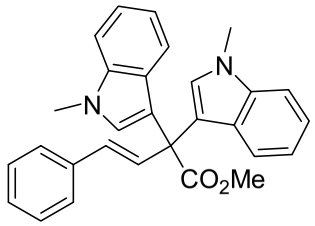
#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	127.25	5.86	5	254.05	10.16	9	269.10	3.89	13	372.10	5.58
2	134.55	5.58	6	255.10	4.37	10	270.10	100.00			
3	134.65	9.72	7	267.05	3.45	11	271.10	22.07			
4	228.05	3.64	8	268.10	8.91	12	371.10	20.84			

BN592 -File: ur06bnru.3799



BN 592 - File: ur06bnru.3799

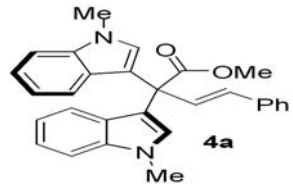
174.321
168.902
143.609
138.338
137.640
137.486
131.186
129.131
129.051
128.491
128.421
128.242
128.227
128.166
127.142
127.078
126.809
126.691
126.493
126.362
126.337
125.317
121.999
121.637
121.602
121.441
120.105
119.950
119.939
119.086
118.912
116.987
115.332
112.409
109.499
109.275
109.174
53.259
52.490
51.818
42.833
32.807
32.790
32.717
21.462



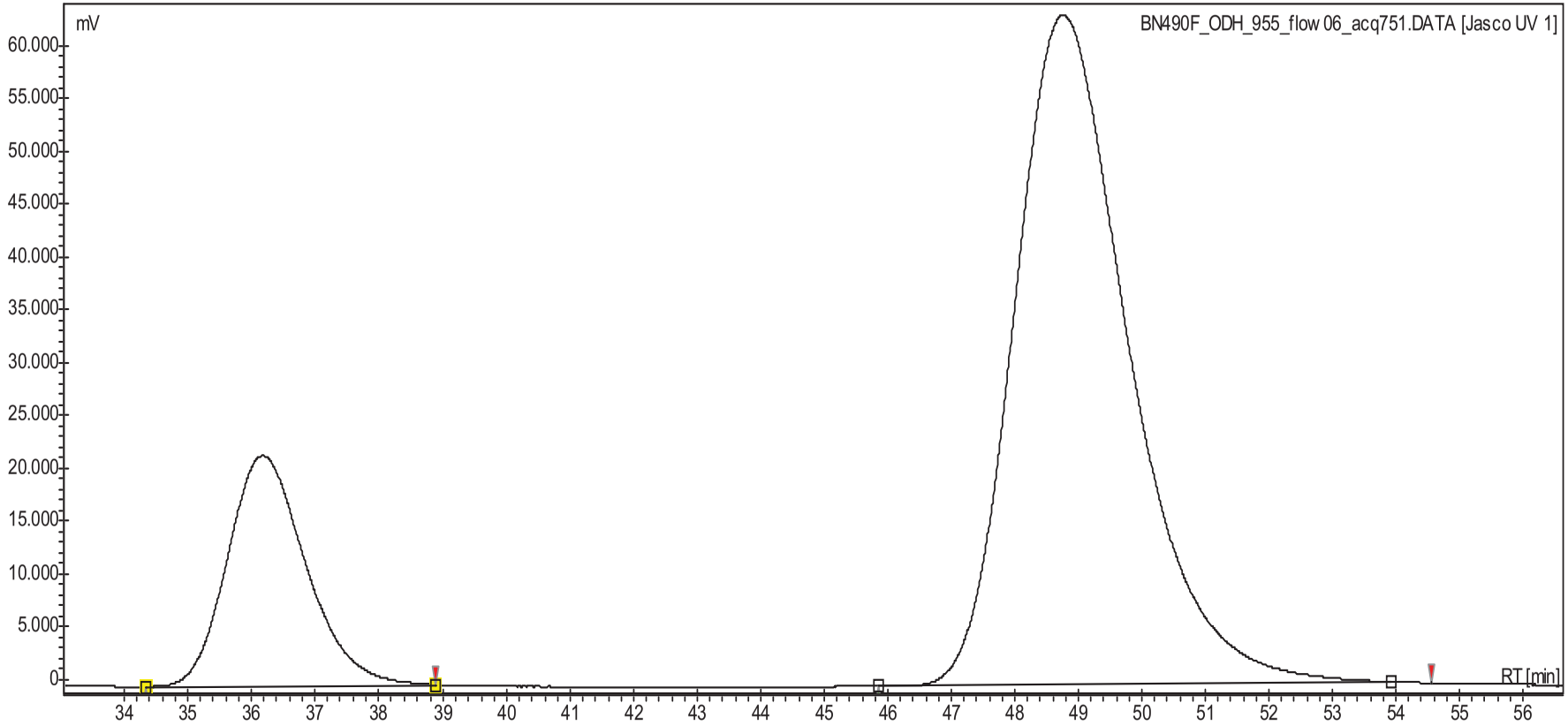
200
150
100
50
0

ppm (f1)

Chromatogram : BN490F_ODH_955_flow06_acq751



Data file: BN490F_ODH_955_flow06_acq751.DATA
Method: HPLC2_ODH_955_flow06_acq75
Date: 06.02.2007 20:07:42

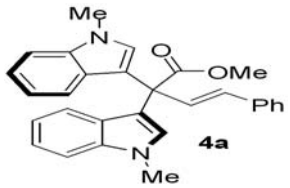


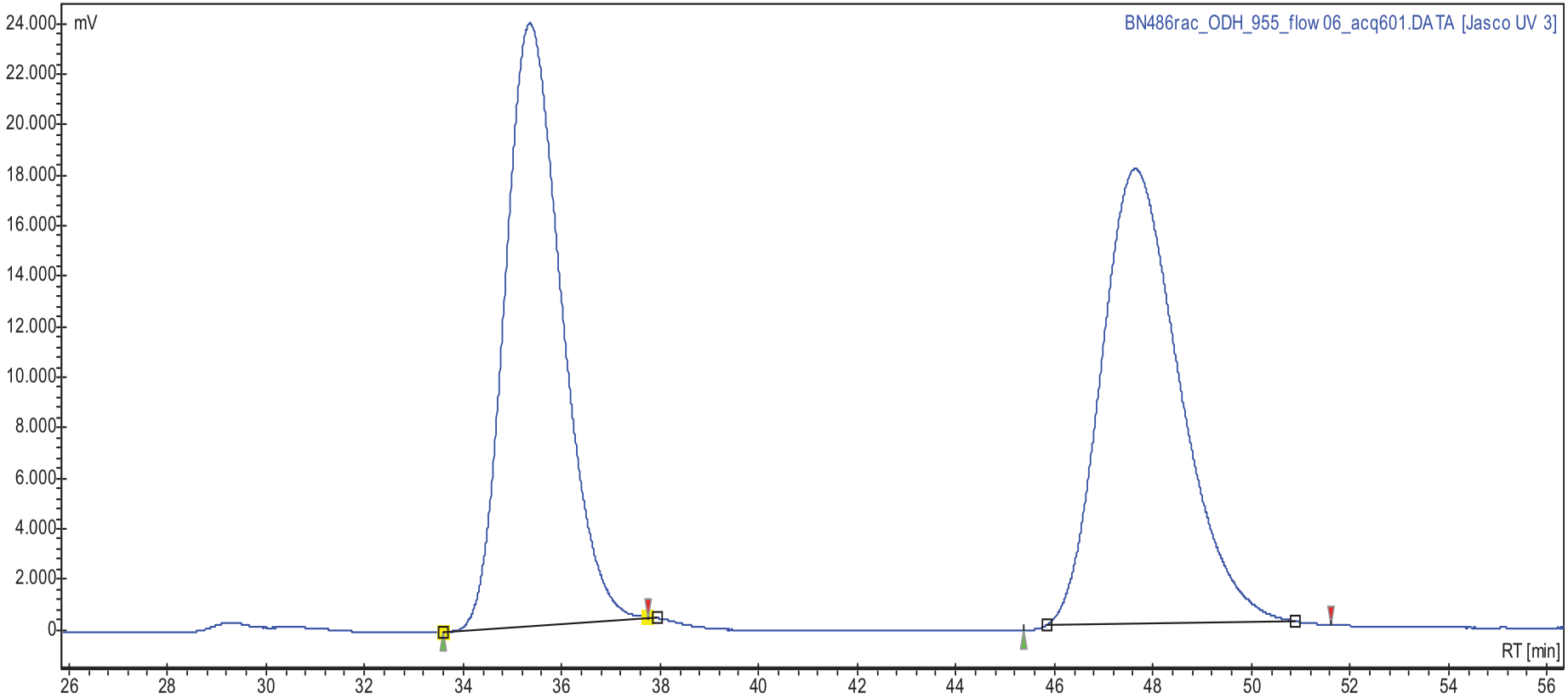
BN490F_ODH_955_flow06_acq751.DATA [Jasco UV 1]

Index	Start	Time	End	Area %
	[Min]	[Min]	[Min]	[%]
1	34,323	36,175	38,895	19,098
2	45,868	48,767	54,545	80,902
Total				100,000

Chromatogram : BN486rac_ODH_955_flow06_acq601

Data file: BN486rac_ODH_955_flow06_acq601.DATA
Method: HPLC2_ODH_955_flow06_acq60
Date: 05.02.2007 14:15:10





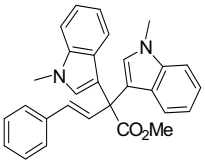
BN486rac_ODH_955_flow06_acq601.DATA [Jasco UV 3]

Index	Name	Start	Time	End	Ret. time Offset	Quantity	Height	Area	Area %
		[Min]	[Min]	[Min]	[Min]	[% Area]	[mV]	[mV.Min]	[%]
1	UNKNOWN	33,595	35,358	37,748	0,000	49,07	23885,9	31778,5	49,066
2	UNKNOWN	45,372	47,642	51,632	0,000	50,93	17990,8	32987,9	50,934
Total						100,00	41876,7	64766,4	100,000

Sample Information

Analyzed : 06.06.2007 12:15:10
Sample Name : BN592 I blau

Method



Chemical Formula: $C_{29}H_{26}N_2O_2$
Exact Mass: 434,2
m/z: 434,20 (100,0%), 435,20 (32,2%), 436,21 (4,9%)

====== Analytical Line 1 ======

[GC-2010]

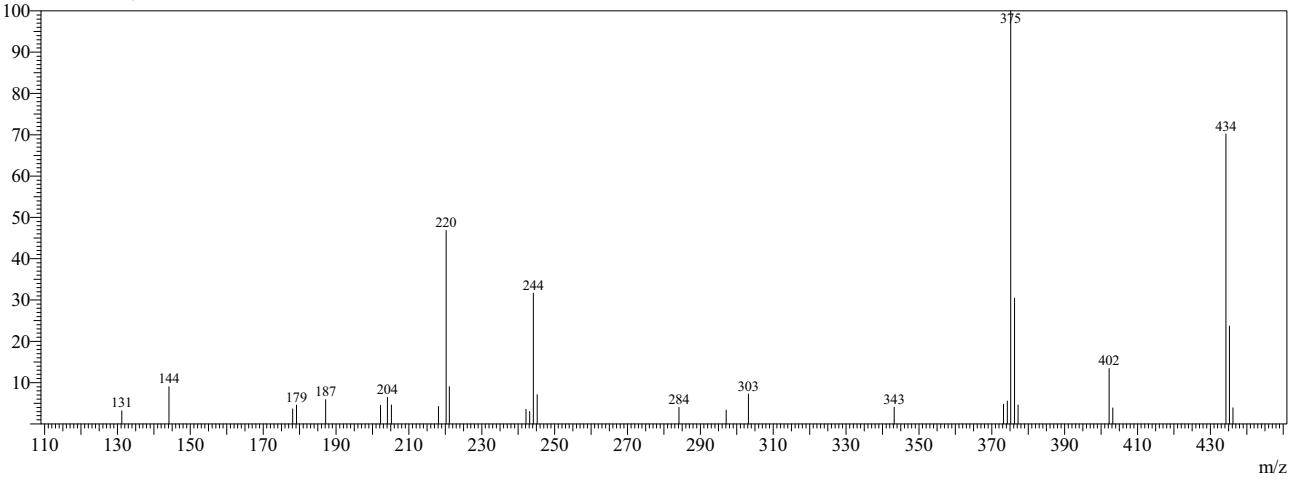
Column Oven Temp.	:80.0 °C
Injection Temp.	:280.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:34.3 kPa
Total Flow	:10.3 mL/min
Column Flow	:0.66 mL/min
Linear Velocity	:30.0 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:10.0

Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	80.0	0.00
20.00	320.0	10.00

Spectrum

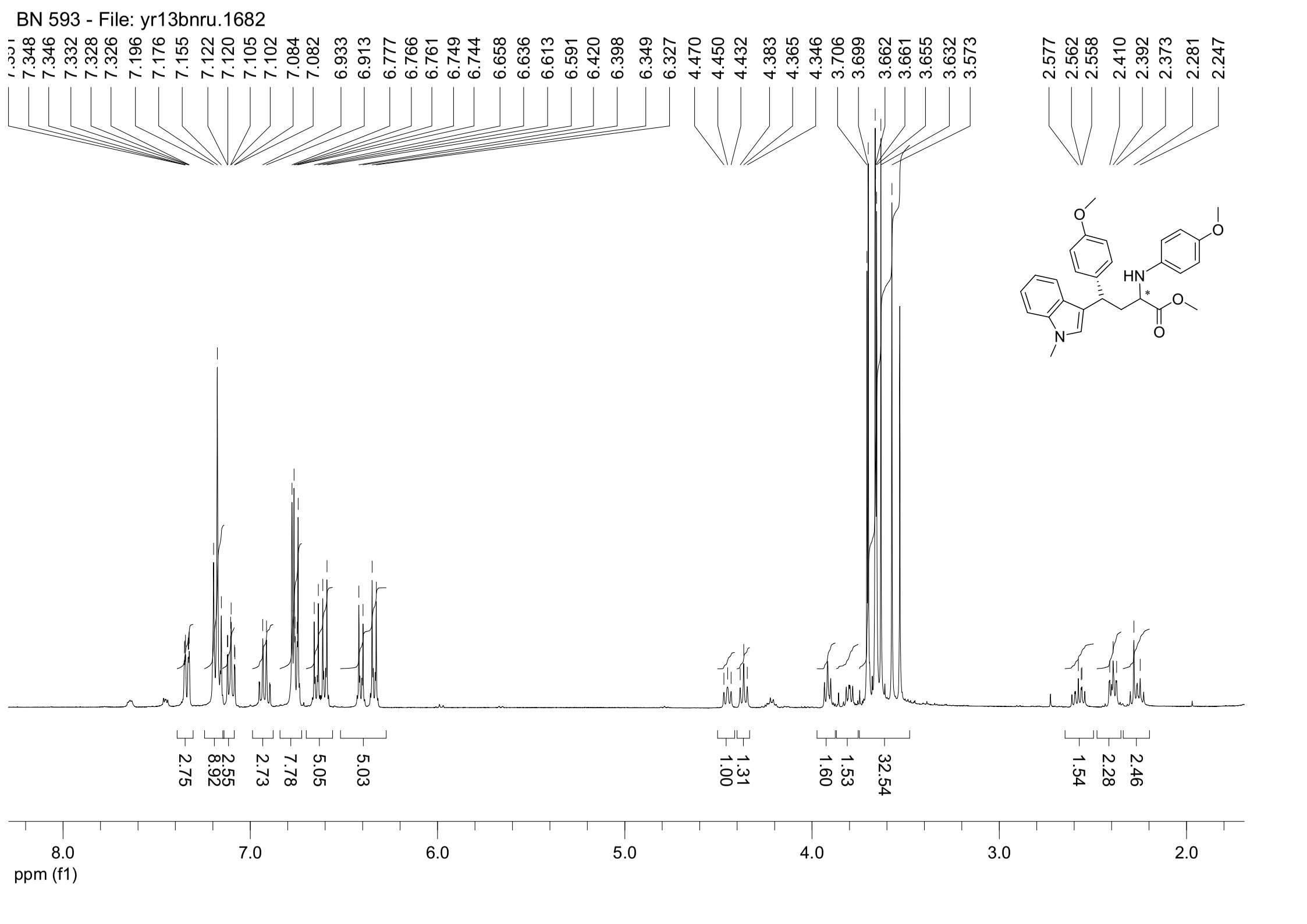
Line#:1 R.Time:18.5(Scan#:1864)
MassPeaks:29 BasePeak:375(12478)
RawMode:Averaged 18.4-18.8(1852-1896)
BG Mode:Averaged 13.7-16.6(1284-1628)



Mass Table
Line#:1 R.Time:18.5(Scan#:1864)
MassPeaks:29 BasePeak:375(12478)
RawMode:Averaged 18.4-18.8(1852-1896)
BG Mode:Averaged 13.7-16.6(1284-1628)

#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.	#	m/z	Rel. Int.
1	131.20	3.19	9	218.10	4.23	17	297.10	3.37	25	402.20	13.42
2	144.15	9.02	10	220.20	46.82	18	303.20	7.26	26	403.20	3.89
3	178.10	3.65	11	221.10	9.02	19	343.20	4.02	27	434.25	70.18
4	179.15	4.57	12	242.15	3.58	20	373.20	4.78	28	435.25	23.69
5	187.15	5.88	13	243.15	3.02	21	374.25	5.54	29	436.20	3.93
6	202.20	4.50	14	244.15	31.66	22	375.20	100.00			
7	204.10	6.48	15	245.20	7.08	23	376.25	30.45			
8	205.20	4.62	16	284.15	4.05	24	377.20	4.60			

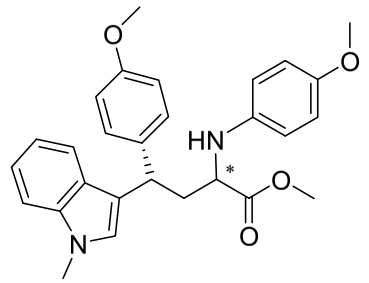
BN 593 - File: yr13bnru.1682



BN 593 - DEPT135 - File: yr13bnru.1682

129.156
128.867
126.163
125.760
121.743
121.710
119.649
119.583
118.896
118.853
115.618
115.406
114.826
114.804
113.969
113.876
109.207
109.181

56.641
56.445
55.734
55.297
55.282
52.079
52.020
40.155
39.679
38.460
38.267
32.779
32.751



150
100
50
ppm (f1)