Supplementary Material (ESI) for Chemical Communications

Chemical Communications

Electronic Supporting Information

Ruthenium/TFA-catalyzed regioselective C-3-alkylation of indoles with terminal alkynes in water: Efficient and unprecedented access to 3-(1-methyl-alkyl)-1*H*-indoles

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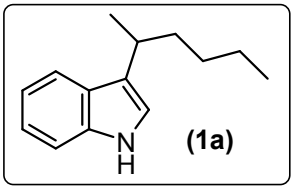
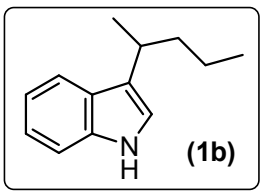
Experimental procedures, characterization data and copies of the ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of all compounds synthesized.

General methods: Infrared spectra were recorded on a Perkin-Elmer 1720-XFT spectrometer. NMR spectra were recorded on a Bruker DPX-300 instrument at 300 MHz (^{1}H), 282.4 MHz (^{19}F) or 75.4 MHz (^{13}C) using $SiMe_4$ or $CFCl_3$ as standards. DEPT experiments have been carried out for all the compounds reported. GC/MS measurements were performed on a Agilent 6890N equipment coupled to a 5973 mass detector (70eV electron impact ionization) using a HP-1MS column. All reagents were obtained from commercial suppliers and used without any further purification, with the exception of complex $[\{RuCl(\mu\text{-}Cl)(\eta^3\text{:}\eta^3\text{-}C_{10}H_{16})\}_2]$,[1] which was prepared by following the methods reported in the literature.

General procedure for the catalytic alkylation of indoles with terminal alkynes: The corresponding indole derivative (1 mmol), the appropriate terminal alkyne (2.5 mmol) and water (1 cm^3) were introduced into a sealed tube under nitrogen atmosphere. $[\{RuCl(\mu\text{-}Cl)(\eta^3\text{:}\eta^3\text{-}C_{10}H_{16})\}_2]$ (0.006 g, 0.01 mmol; 2 mol% of Ru) and TFA (0.037 cm^3, 0.5 mmol) were then added at room temperature, and the resulting suspension heated at 100 ºC for 24 h. After removal of volatiles under vacuum, the residue was purified by column chromatography (silica gel) using a mixture of EtOAc/hexanes (1:50) as eluent. Characterization data of the resulting compounds **1a-r** are as follows:

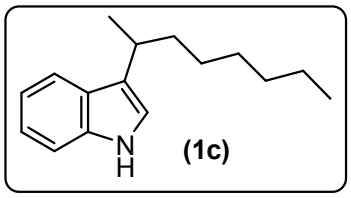


3-(1-Methylpentyl)-1*H*-indole (1a):[2] Yield: 84% (0.169 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1093, 1226, 1337, 1375, 1418, 1456, 1618, 2870, 2926, 2956, 3056, 3417 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.92 (t, 3H, J = 6.6 Hz), 1.35 (m, 4H), 1.39 (d, 3H, J = 7.0 Hz), 1.64 (m, 1H), 1.84 (m, 1H), 3.07 (m, 1H), 6.98 (d, 1H, J = 2.2 Hz), 7.12-7.25 (m, 2H), 7.38 (d, 1H, J = 7.9 Hz), 7.70 (d, 1H, J = 7.9 Hz), 7.90 (br, 1H) ppm; ^{13}C{^{1}H} NMR ($CDCl_3$): δ 14.1, 21.5, 22.9, 30.0, 30.8, 37.4, 111.1, 118.9, 119.4, 119.8, 121.7, 122.9, 127.0, 136.5 ppm; MS (EI, 70eV): *m/z* 201 (M$^+$, 15), 144 (100), 115 (10).

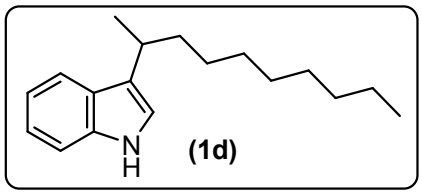


3-(1-Methylbutyl)-1*H*-indole (1b):[3] Yield: 79% (0.148 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1095, 1222, 1245, 1338, 1373, 1418, 1455, 1618, 2869, 2927, 2956, 3056, 3417 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.95 (t, 3H, J = 7.4 Hz), 1.31-1.56 (m, 2H), 1.39 (d, 3H, J = 6.8 Hz), 1.62 (m, 1H), 1.84 (m, 1H), 3.10 (m, 1H), 6.97 (d, 1H, J = 2.1 Hz),

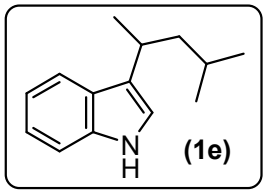
7.12-7.25 (m, 2H), 7.37 (d, 1H, *J* = 8.1 Hz), 7.71 (d, 1H, *J* = 8.3 Hz), 7.88 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.3, 20.8, 21.4, 30.6, 40.0, 111.1, 118.9, 119.5, 119.8, 121.7, 122.9, 127.0, 136.5 ppm; MS (EI, 70eV): *m/z* 187 (M^+, 20), 144 (100), 115 (10).



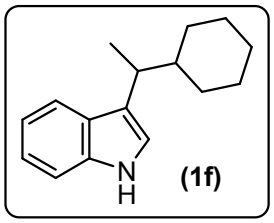
3-(1-Methylheptyl)-1*H*-indole (1c):[2] Yield: 76% (0.174 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1094, 1227, 1244, 1338, 1376, 1418, 1456, 1486, 1618, 2854, 2924, 2955, 3056, 3418 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.92 (t, 3H, *J* = 6.6 Hz), 1.33 (br, 8H), 1.40 (d, 3H, *J* = 7.2 Hz), 1.67 (m, 1H), 1.85 (m, 1H), 3.08 (m, 1H), 6.95 (s, 1H), 7.13-7.28 (m, 2H), 7.38 (d, 1H, *J* = 7.8 Hz), 7.70 (d, 1H, *J* = 7.9 Hz), 7.88 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.4, 22.7, 27.7, 29.5, 30.9, 31.9, 37.7, 111.1, 118.9, 119.5, 119.8, 121.7, 122.9, 127.0, 136.5 ppm; MS (EI, 70eV): *m/z* 229 (M^+, 15), 144 (100), 115 (10).



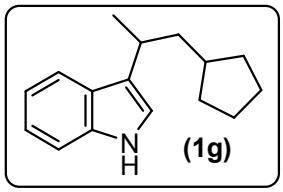
3-(1-Methylnonyl)-1*H*-indole (1d): Yield: 57% (0.147 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1093, 1224, 1337, 1375, 1418, 1456, 1617, 2853, 2924, 2955, 3056, 3417 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.93 (t, 3H, *J* = 6.6 Hz), 1.30 (br, 12H), 1.40 (d, 3H, *J* = 7.0 Hz), 1.64 (m, 1H), 1.82 (m, 1H), 3.07 (m, 1H), 6.97 (s, 1H), 7.13-7.29 (m, 2H), 7.38 (d, 1H, *J* = 7.8 Hz), 7.71 (d, 1H, *J* = 7.8 Hz), 7.87 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.5, 22.7, 27.8, 29.4, 29.7, 29.9, 30.9, 31.9, 37.7, 111.1, 118.9, 119.5, 119.8, 121.7, 122.9, 127.0, 136.5 ppm; MS (EI, 70eV): *m/z* 257 (M^+, 20), 144 (100), 115 (10).



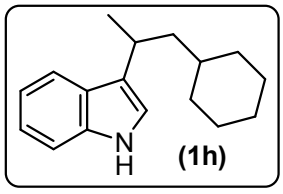
3-(1,3-Dimethylbutyl)-1*H*-indole (1e): Yield: 64% (0.128 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1092, 1222, 1247, 1337, 1365, 1383, 1418, 1455, 1618, 2867, 2925, 2953, 3056, 3418 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.96 (m, 6H), 1.38 (d, 3H, *J* = 6.8 Hz), 1.50 (m, 1H), 1.63-1.80 (m, 2H), 3.16 (m, 1H), 6.97 (s, 1H), 7.13-7.27 (m, 2H), 7.38 (d, 1H, *J* = 8.0 Hz), 7.71 (d, 1H, *J* = 8.0 Hz), 7.87 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 21.9, 22.7, 23.0, 25.8, 28.5, 47.2, 111.2, 119.0, 119.4, 119.8, 121.8, 123.1, 127.1, 136.6 ppm; MS (EI, 70eV): *m/z* 201 (M^+, 20), 144 (100), 115 (20).



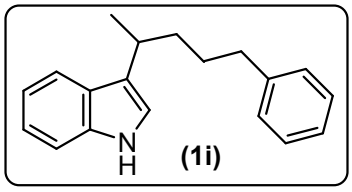
3-(1-Cyclohexylethyl)-1*H*-indole (1f): Yield: 69% (0.157 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1011, 1095, 1224, 1261, 1337, 1418, 1455, 1618, 2850, 2922, 3054, 3417 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.19 (m, 2H), 1.41 (d, 3H, *J* = 7.0 Hz), 1.53-1.66 (m, 5H), 1.82-1.91 (m, 4H), 3.13 (m, 1H), 6.98 (s, 1H), 7.14-7.28 (m, 2H), 7.38 (d, 1H, *J* = 7.9 Hz), 7.73 (d, 1H, *J* = 7.9 Hz), 7.88 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 21.9, 25.2, 30.0, 32.8, 33.0, 38.1, 44.5, 111.2, 118.9, 119.5, 119.8, 121.7, 123.1, 126.9, 136.5 ppm; MS (EI, 70eV): *m/z* 227 (M^+, 10), 144 (100), 115 (20).



3-(2-Cyclopentyl-1-methylethyl)-1*H*-indole (1g): Yield: 83% (0.188 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1092, 1223, 1337, 1374, 1419, 1457, 1617, 2865, 2951, 3055, 3415 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.97-1.31 (m, 4H), 1.36 (d, 3H, *J* = 7.0 Hz), 1.67-1.89 (m, 7H), 2.92 (m, 1H), 6.96 (s, 1H), 7.11-7.28 (m, 2H), 7.38 (d, 1H, *J* = 7.8 Hz), 7.68 (d, 1H, *J* = 7.8 Hz), 7.92 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 18.0, 26.7, 26.8, 30.4, 31.5, 36.5, 43.7, 111.0, 118.9, 119.7, 120.7, 121.6, 121.7, 127.4, 136.3 ppm; MS (EI, 70eV): *m/z* 227 (M^+, 10), 144 (100), 115 (20).

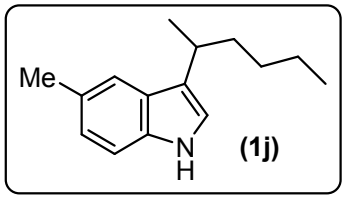


3-(2-Cyclohexyl-1-methylethyl)-1*H*-indole (1h): Yield: 68% (0.164 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1010, 1093, 1225, 1337, 1372, 1417, 1455, 1619, 2848, 2920, 3054, 3417 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.00 (m, 3H), 1.25 (m, 3H), 1.36 (d, 3H, *J* = 7.0 Hz), 1.48 (m, 1H), 1.69-1.80 (m, 6H), 3.20 (m, 1H), 6.95 (s, 1H), 7.11-7.26 (m, 2H), 7.36 (d, 1H, *J* = 7.8 Hz), 7.69 (d, 1H, *J* = 7.8 Hz), 7.85 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 21.8, 26.3, 26.7, 27.6, 33.4, 33.8, 35.2, 45.6, 111.1, 118.9, 119.3, 119.7, 121.7, 123.1, 126.8, 136.5 ppm; MS (EI, 70eV): *m/z* 241 (M^+, 10), 144 (100), 115 (15).

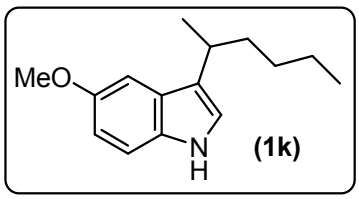


3-(1-Methyl-4-phenylbutyl)-1*H*-indole (1i): Yield: 60% (0.158 g); Brown oil; IR (neat, cm^{-1}): *v* 1010, 1094, 1226, 1259, 1337, 1373, 1418, 1455, 1495, 1602, 2855, 2927, 3025, 3058, 3083, 3420 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.39 (d, 3H, *J* = 7.3 Hz), 1.61-1.93 (m, 4H), 2.65 (t, 2H, *J* = 7.2 Hz), 3.11 (m, 1H), 6.94 (d, 1H, *J* = 1.8 Hz), 7.11-7.31 (m, 7H), 7.38 (d, 1H, *J* = 8.1 Hz), 7.66 (d, 1H, *J* = 7.9 Hz), 7.89 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 21.5, 29.5, 30.8, 36.1, 37.2, 111.1, 119.0,

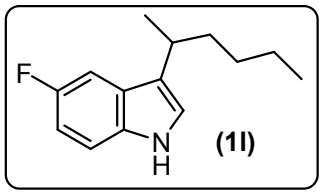
119.4, 119.9, 121.8, 122.5, 125.6, 126.9, 128.2, 128.4, 136.5, 142.8 ppm; MS (EI, 70eV): *m/z* 263 (M^+, 20), 144 (100), 115 (10), 91 (10).



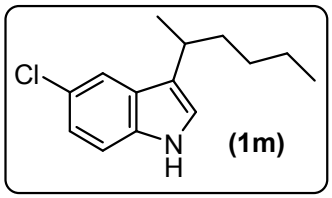
5-Methyl-3-(1-methylpentyl)-1*H*-indole (1j): Yield: 82% (0.176 g); Brown oil; IR (neat, cm^{-1}): *v* 1095, 1184, 1226, 1329, 1376, 1420, 1459, 1481, 1581, 2856, 2924, 2956, 3015, 3412 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.94 (t, 3H, J = 7.2 Hz), 1.37-1.42 (m, 7H), 1.67 (m, 1H), 1.85 (m, 1H), 2.54 (s, 3H), 3.06 (m, 1H), 6.94 (d, 1H, J = 2.1 Hz), 7.07 (dd, 1H, J = 8.3 and 1.3 Hz), 7.28 (d, 1H, J = 8.3 Hz), 7.50 (s, 1H), 7.76 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.2, 21.5, 21.6, 22.9, 30.0, 30.8, 37.4, 110.8, 119.1, 120.0, 122.4, 123.3, 127.2, 128.1, 134.9 ppm; MS (EI, 70eV): *m/z* 215 (M^+, 20), 158 (100), 142 (15), 115 (5).



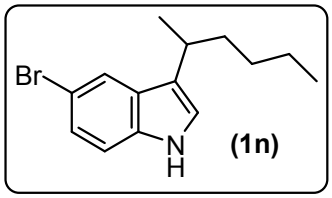
5-Methoxy-3-(1-methylpentyl)-1*H*-indole (1k): Yield: 79% (0.183 g); Brown oil; IR (neat, cm^{-1}): *v* 1032, 1097, 1146, 1174, 1215, 1284, 1375, 1439, 1455, 1483, 1581, 1623, 2857, 2927, 2955, 3418 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.90 (t, 3H, J = 5.8 Hz), 1.33 (m, 4H), 1.35 (d, 3H, J = 6.9 Hz), 1.63 (m, 1H), 1.79 (m, 1H), 2.99 (m, 1H), 3.88 (s, 3H), 6.86 (dd, 1H, J = 8.8 and 2.5 Hz), 6.93 (d, 1H, J = 2.1 Hz), 7.11 (d, 1H, J = 2.5 Hz), 7.24 (d, 1H, J = 8.8 Hz), 7.81 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.3, 22.8, 29.9, 30.6, 37.2, 55.9, 101.5, 111.6, 120.7, 122.5, 127.3, 131.6, 153.5 ppm; MS (EI, 70eV): *m/z* 231 (M^+, 25), 174 (100), 158 (10), 130 (10).



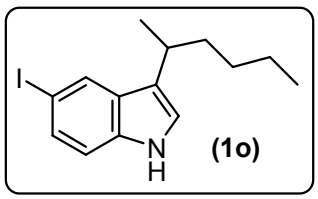
5-Fluoro-3-(1-methylpentyl)-1*H*-indole (1l): Yield: 69% (0.151 g); Brown oil; IR (neat, cm^{-1}): *v* 935, 1094, 1141, 1174, 1221, 1274, 1376, 1454, 1483, 1579, 1627, 2870, 2927, 2957, 3429, 3474 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.92 (t, 3H, J = 7.0 Hz), 1.32 (m, 4H), 1.36 (d, 3H, J = 6.8 Hz), 1.58 (m, 1H), 1.75 (m, 1H), 2.99 (m, 1H), 6.93-7.02 (m, 2H), 7.25-7.34 (m, 2H), 7.89 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.3, 22.9, 29.9, 30.8, 37.3, 104.3 (d, J = 23.3 Hz), 110.0 (d, J = 26.5 Hz), 111.6 (d, J = 10.1 Hz), 121.7, 123.1 (d, J = 4.7 Hz), 127.3 (d, J = 9.5 Hz), 133.0, 157.5 (d, J = 233.6 Hz) ppm; $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ -125.2 (s) ppm; MS (EI, 70eV): *m/z* 219 (M^+, 20), 162 (100), 133 (10), 115 (5).



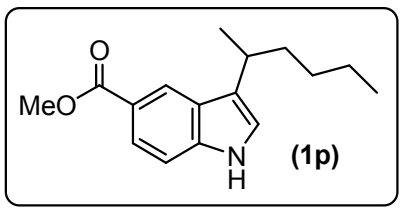
5-Chloro-3-(1-methylpentyl)-1*H*-indole (1m): Yield: 67% (0.158 g); Yellow oil; IR (neat, cm^{-1}): *v* 1090, 1147, 1170, 1222, 1264, 1371, 1450, 1482, 1573, 1625, 2876, 2917, 2952, 3414 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.90 (t, 3H, *J* = 7.1 Hz), 1.30 (m, 4H), 1.34 (d, 3H, *J* = 6.9 Hz), 1.58 (m, 1H), 1.77 (m, 1H), 2.97 (m, 1H), 6.98 (d, 1H, *J* = 1.6 Hz), 7.14 (dd, 1H, *J* = 7.4 and 1.6 Hz), 7.27 (d, 1H, *J* = 2.5 Hz), 7.62 (d, 1H, *J* = 1.4 Hz), 7.93 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.4, 22.8, 29.9, 30.7, 37.3, 112.0, 118.9, 121.3, 122.0, 122.7, 124.7, 128.0, 134.8 ppm; MS (EI, 70eV): *m/z* 235 (M^+, 20), 178 (100), 162 (15), 143 (50), 115 (25).



5-Bromo-3-(1-methylpentyl)-1*H*-indole (1n): Yield: 57% (0.160 g); Brown oil; IR (neat, cm^{-1}): *v* 1088, 1222, 1259, 1370, 1419, 1456, 1563, 1617, 2845, 2940, 2959, 3399 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.90 (t, 3H, *J* = 6.9 Hz), 1.30 (m, 4H), 1.32 (d, 3H, *J* = 6.7 Hz), 1.61 (m, 1H), 1.76 (m, 1H), 2.96 (m, 1H), 6.95 (d, 1H, *J* = 1.7 Hz), 7.20-7.26 (m, 2H), 7.76 (s, 1H), 7.98 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 13.6, 20.9, 22.3, 29.4, 30.2, 36.8, 111.8, 112.0, 120.6, 121.5, 122.2, 124.0, 128.2, 134.6 ppm; MS (EI, 70eV): *m/z* 281 (M^+, 10), 222 (100), 195 (5), 143 (90), 115 (60).

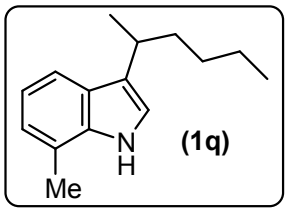


5-Iodo-3-(1-methylpentyl)-1*H*-indole (1o): Yield: 82% (0.268 g); Brown oil; IR (neat, cm^{-1}): *v* 1091, 1223, 1261, 1375, 1414, 1456, 1558, 1615, 2855, 2924, 2956, 3424 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.89 (t, 3H, *J* = 6.8 Hz), 1.28 (m, 4H), 1.33 (d, 3H, *J* = 7.1 Hz), 1.63 (m, 1H), 1.76 (m, 1H), 2.97 (m, 1H), 6.93 (s, 1H), 7.14 (d, 1H, *J* = 8.2 Hz), 7.43 (d, 1H, *J* = 8.2 Hz), 7.93 (br, 1H), 7.99 (s, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.5, 22.8, 29.9, 30.6, 37.3, 82.5, 113.1, 120.7, 122.4, 128.3, 129.6, 130.0, 135.5 ppm; MS (EI, 70eV): *m/z* 327 (M^+, 30), 270 (100), 143 (30), 115 (10).

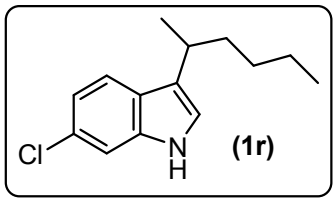


3-(1-Methylpentyl)-1*H*-indole-5-carboxylic acid methyl ester (1p): Yield: 87% (0.225 g); Yellow oil; IR (neat, cm^{-1}): *v* 974, 1046, 1106, 1246, 1291, 1310,

1373, 1435, 1580, 1616, 1693, 2857, 2927, 2956, 3348 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.88 (br, 3H), 1.28-1.37 (m, 7H), 1.66 (m, 1H), 1.77 (m, 1H), 3.07 (m, 1H), 3.96 (s, 3H), 7.02 (s, 1H), 7.36 (d, 1H, J = 8.4 Hz), 7.90 (d, 1H, J = 8.4 Hz), 8.26 (br, 1H), 8.44 (s, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.6, 22.8, 29.9, 30.7, 37.4, 51.8, 110.7, 121.1, 122.4, 123.2, 124.4, 126.6, 139.1, 168.4 ppm; MS (EI, 70eV): *m/z* 259 (M^+, 30), 228 (10), 202 (100), 170 (15), 143 (25), 115 (10).



7-Methyl-3-(1-methylpentyl)-1*H*-indole (1q): Yield: 60% (0.129 g); Pale yellow oil; IR (neat, cm^{-1}): *v* 1016, 1063, 1113, 1260, 1343, 1377, 1435, 1455, 1494, 1611, 1687, 2857, 2927, 2956, 3050, 3419 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.92 (t, 3H, J = 6.8 Hz), 1.36 (m, 4H), 1.40 (d, 3H, J = 7.0 Hz), 1.65 (m, 1H), 1.84 (m, 1H), 2.52 (s, 3H), 3.07 (m, 1H), 6.98 (s, 1H), 7.03-7.10 (m, 2H), 7.56 (d, 1H, J = 7.9 Hz), 7.82 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 16.6, 21.5, 22.9, 30.0, 31.0, 37.4, 117.2, 119.2, 119.6, 120.2, 122.3, 123.5, 126.5, 136.1 ppm; MS (EI, 70eV): *m/z* 215 (M^+, 20), 158 (100), 140 (20), 115 (5).



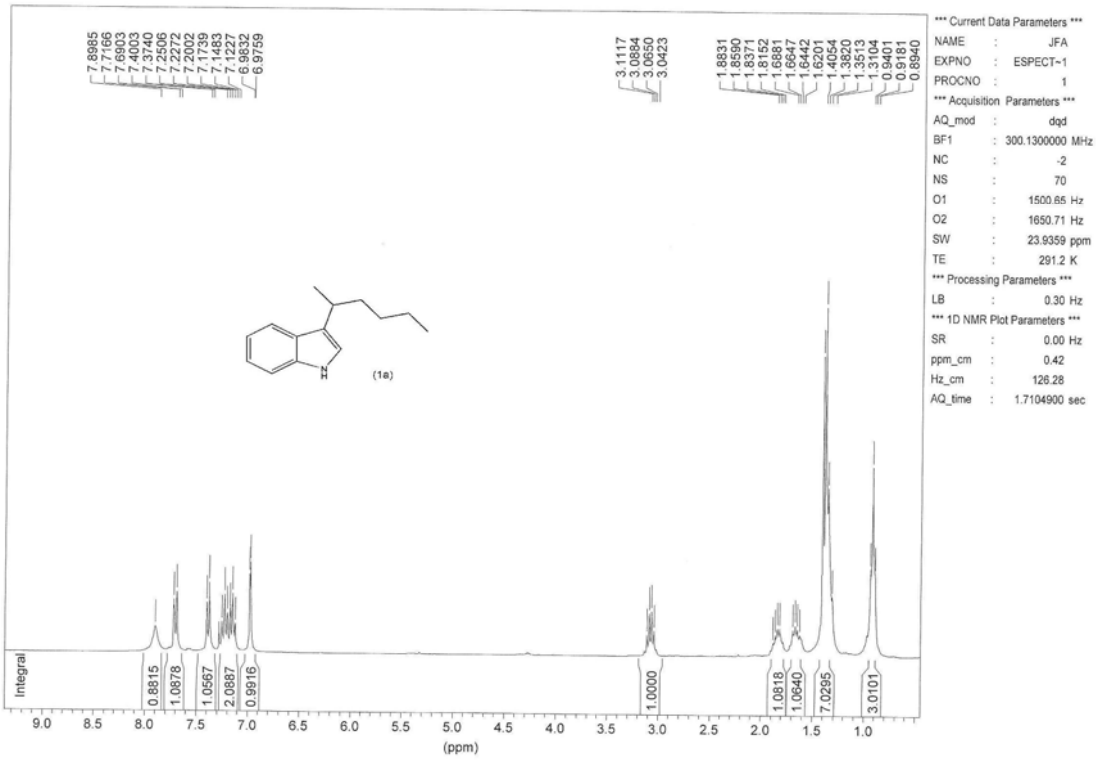
6-Chloro-3-(1-methylpentyl)-1*H*-indole (1r): Yield: 65% (0.153 g); Yellow oil; IR (neat, cm^{-1}): *v* 1062, 1091, 1224, 1333, 1375, 1398, 1456, 1544, 1617, 2857, 2927, 2956, 3427 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.89 (t, 3H, J = 6.6 Hz), 1.32 (m, 4H), 1.35 (d, 3H, J = 6.8 Hz), 1.59 (m, 1H), 1.79 (m, 1H), 3.01 (m, 1H), 6.95 (d, 1H, J = 2.2 Hz), 7.08 (dd, 1H, J = 8.5 and 1.3 Hz), 7.35 (d, 1H, J = 1.3 Hz), 7.57 (d, 1H, J = 8.5 Hz), 7.91 (br, 1H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 14.1, 21.4, 22.8, 29.9, 30.7, 37.4, 111.0, 119.7, 120.2, 120.5, 123.1, 125.6, 127.7, 136.8 ppm; MS (EI, 70eV): *m/z* 235 (M^+, 10), 178 (100), 163 (10), 143 (40), 115 (10).

References

1 (*a*) L. Porri, M. C. Gallazzi, A. Colombo and G. Allegra, *Tetrahedron Lett.*, 1965, **47**, 4187; (*b*) A. Salzer, A. Bauer, S. Geyser and F. Podewils, *Inorg. Synth.*, 2004, **34**, 59.

2 A.-M. L. Hogan and D. F. O´Shea, *J. Org. Chem.*, 2008, **73**, 2503.

3 B. Cardillo, G. Casnati, A. Pochini and A. Ricca, *Tetrahedron*, 1967, **23**, 3771.


7.8985
7.7166
7.6903
7.4403
7.3740
7.3596
7.2272
7.2002
7.1739
7.1483
7.1227
6.9632
6.9759
3.1117
3.0884
3.0650
3.0423
1.8831
1.8590
1.8371
1.8152
1.6881
1.6647
1.6442
1.6201
1.4054
1.3820
1.3513
1.3104
0.9401
0.9181
0.8940
(1a)
Integral
0.8815
1.0878
1.0567
2.0887
0.9916
1.0000
1.0818
1.0640
7.0295
3.0101
(ppm)
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PROCNO : 1
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NC : -2
NS : 70
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O2 : 1650.71 Hz
SW : 23.9359 ppm
TE : 291.2 K
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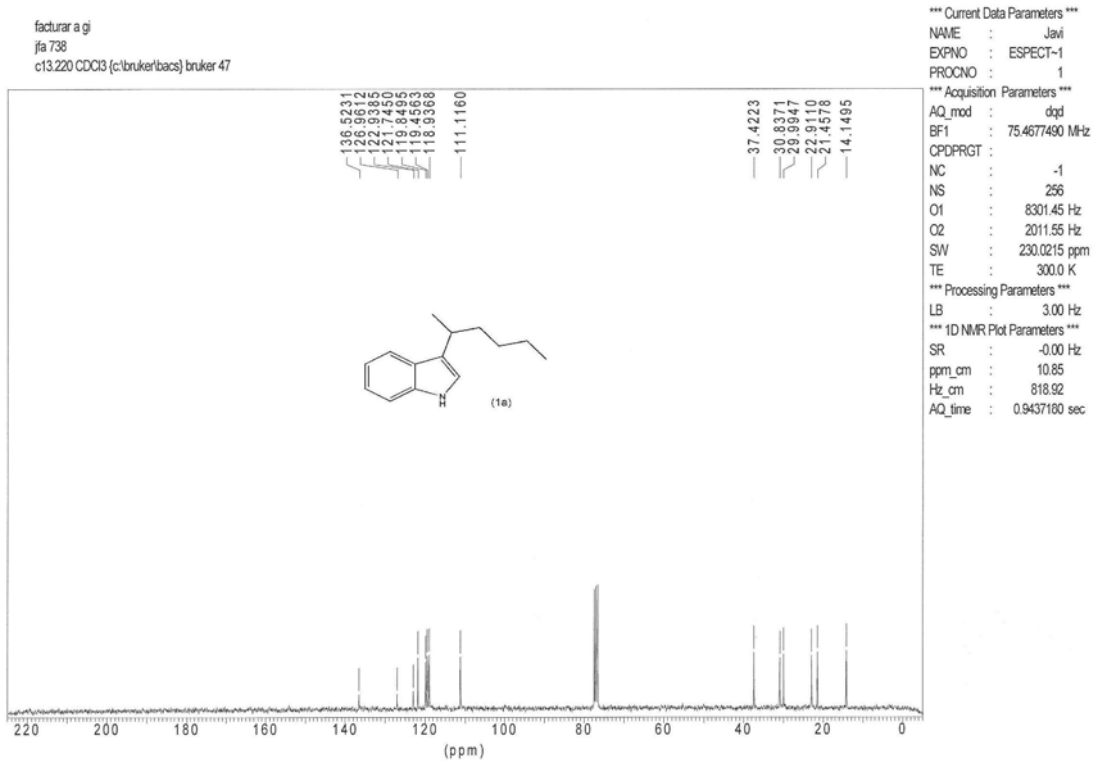
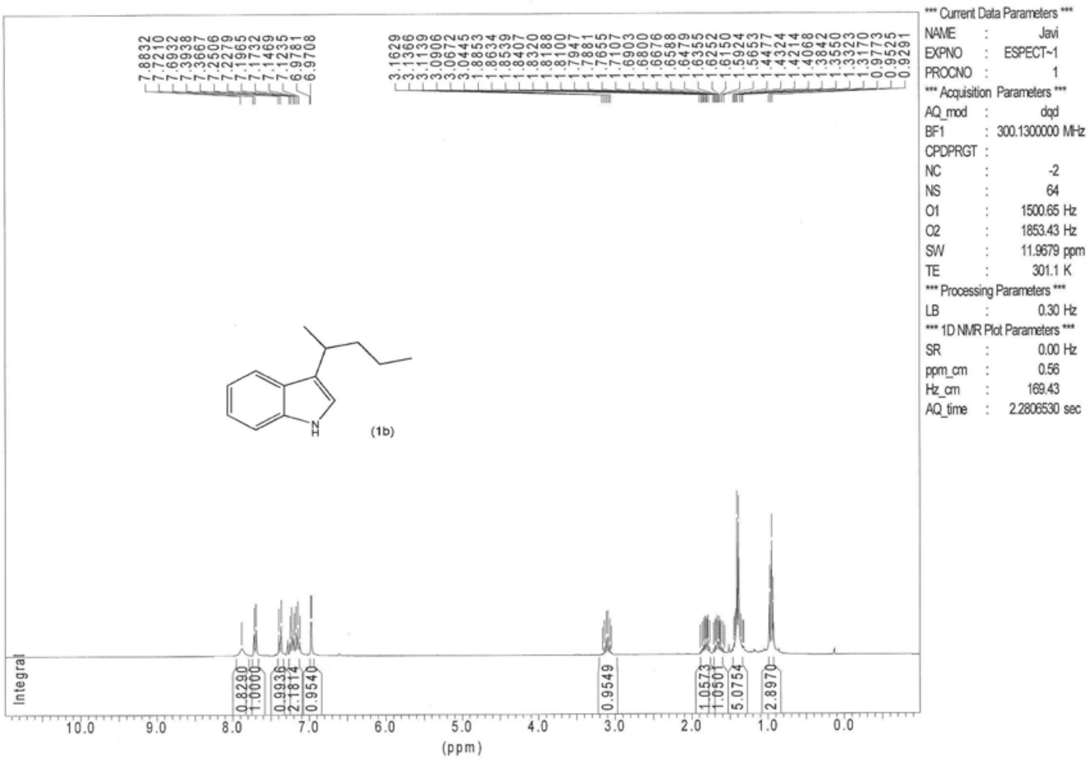
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.42
Hz_cm : 126.28
AQ_time : 1.7104900 sec


facturar a gi
jfa 738
c13.220 CDCl3 {c:\bruker\bacs} bruker 47
136.5231
136.0612
122.9355
121.7450
119.8455
119.4563
118.9368
111.1160
37.4223
30.8371
29.9947
22.9110
21.4578
14.1495
(1a)
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 256
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec


(1b)
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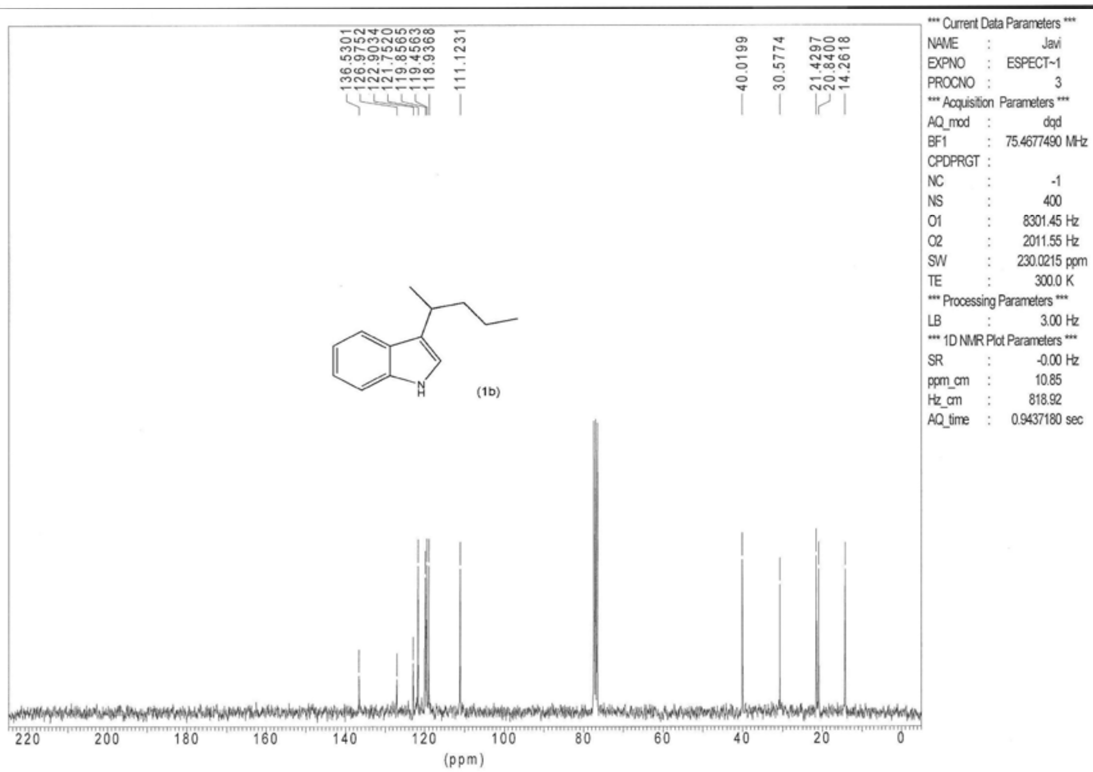
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 301.1 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
Integral
(ppm)


(1b)
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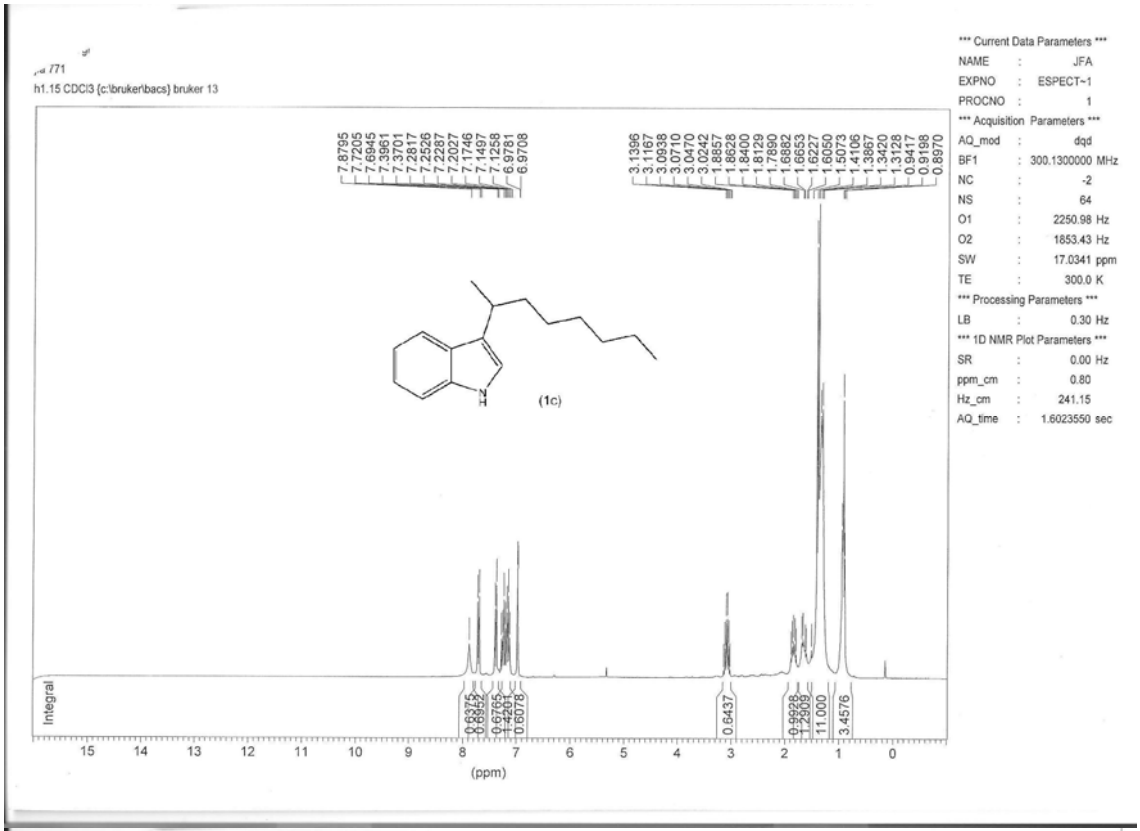
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
136.5301
126.9752
122.9034
121.7520
119.8565
119.4563
118.9368
111.1231
40.0199
30.5774
21.4297
20.8400
14.2618
(ppm)


jfa 771
h1.15 CDCl3 {c:\bruker\bacs} bruker 13
7.8795
7.7205
7.6945
7.3961
7.3701
7.2817
7.2526
7.2287
7.2027
7.1746
7.1497
7.1258
6.9781
6.9708
3.1396
3.1167
3.0938
3.0710
3.0470
3.0242
1.8857
1.8628
1.8400
1.8129
1.7890
1.7652
1.6653
1.6527
1.6050
1.5073
1.4106
1.3867
1.3420
1.3128
0.9417
0.9198
0.8970
(1c)
Integral
0.6375
0.6952
0.6765
1.4261
0.6070
0.6437
0.9928
1.2909
11.000
3.4576
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
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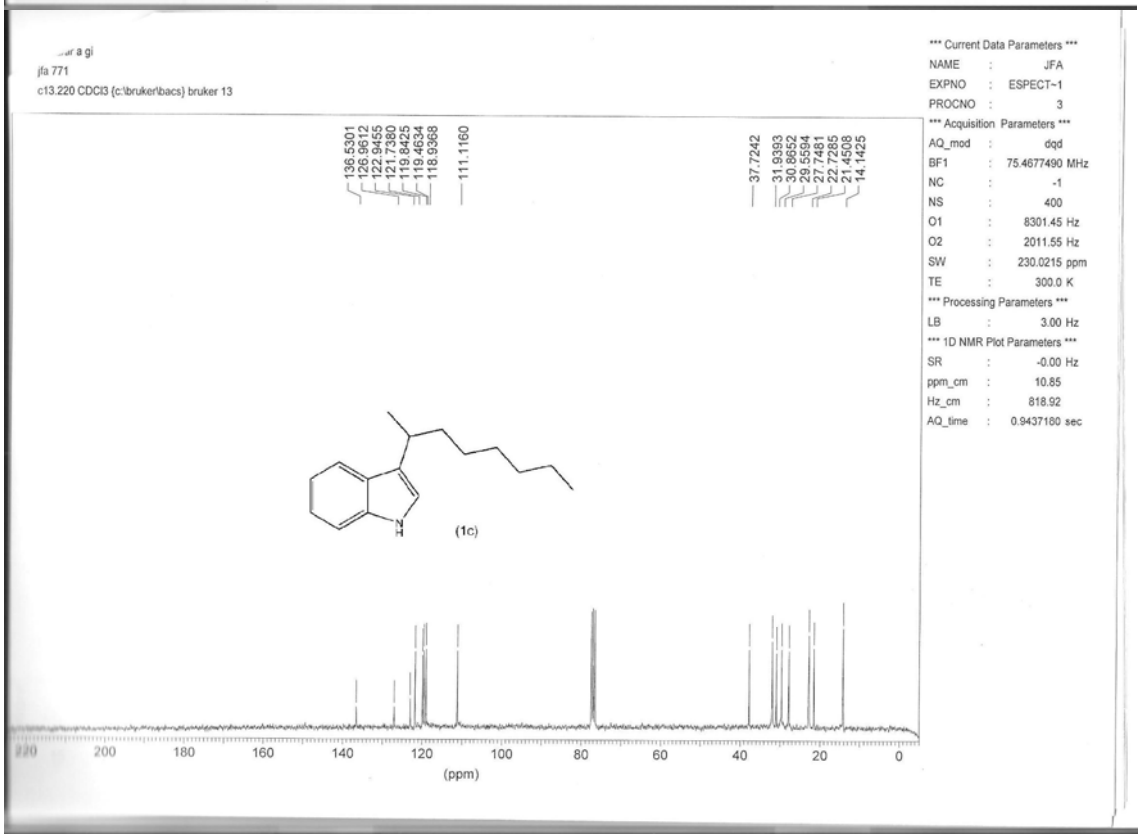
NC : -2
NS : 64
O1 : 2250.98 Hz
O2 : 1853.43 Hz
SW : 17.0341 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.80
Hz_cm : 241.15
AQ_time : 1.6023550 sec


jfa 771
c13.220 CDCl3 {c:\bruker\bacs} bruker 13
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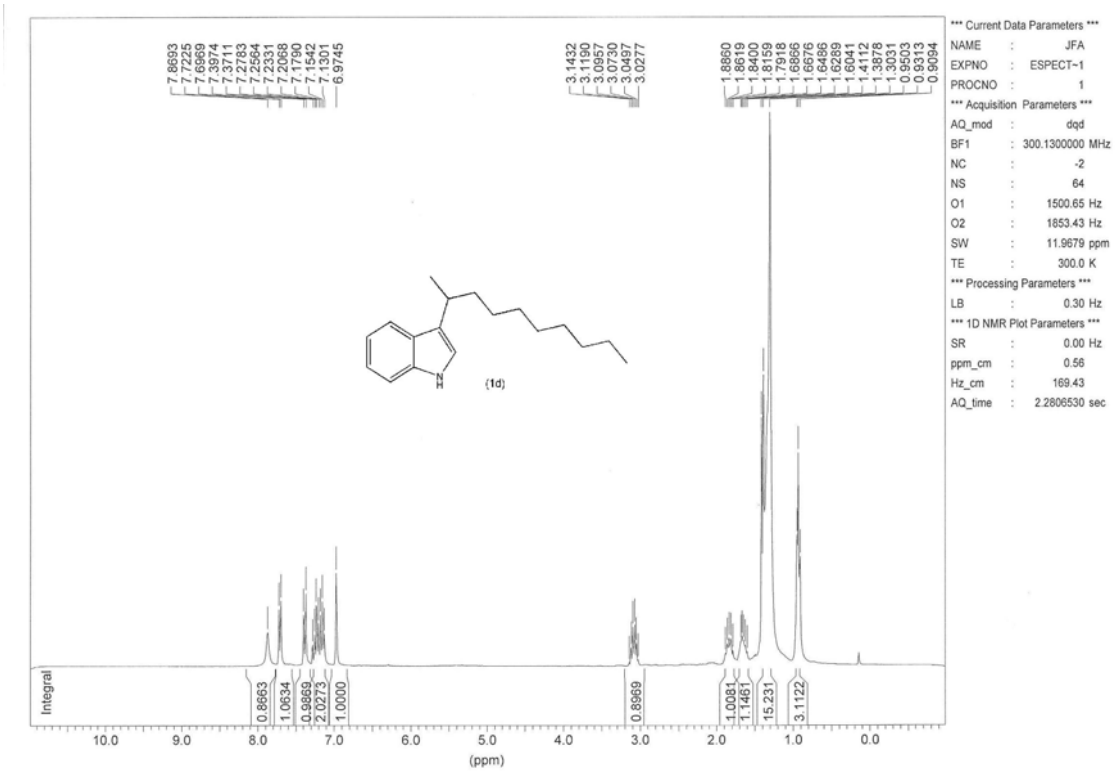
126.9612
122.9455
121.7380
119.8425
119.4634
118.9368
111.1160
37.7242
31.9393
30.8652
29.5594
27.7481
22.7285
21.4508
14.1425
(1c)
220 200 180 160 140 120 100 80 60 40 20 0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec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7.7225
7.6969
7.3974
7.3711
7.2783
7.2584
7.2331
7.2068
7.1790
7.1542
7.1301
6.9745
3.1432
3.1190
3.0957
3.0730
3.0497
3.0277
1.8860
1.8619
1.8400
1.8159
1.7918
1.6886
1.6676
1.6486
1.6289
1.6041
1.4112
1.3878
1.3031
0.9503
0.9313
0.9094
(1d)
Integral
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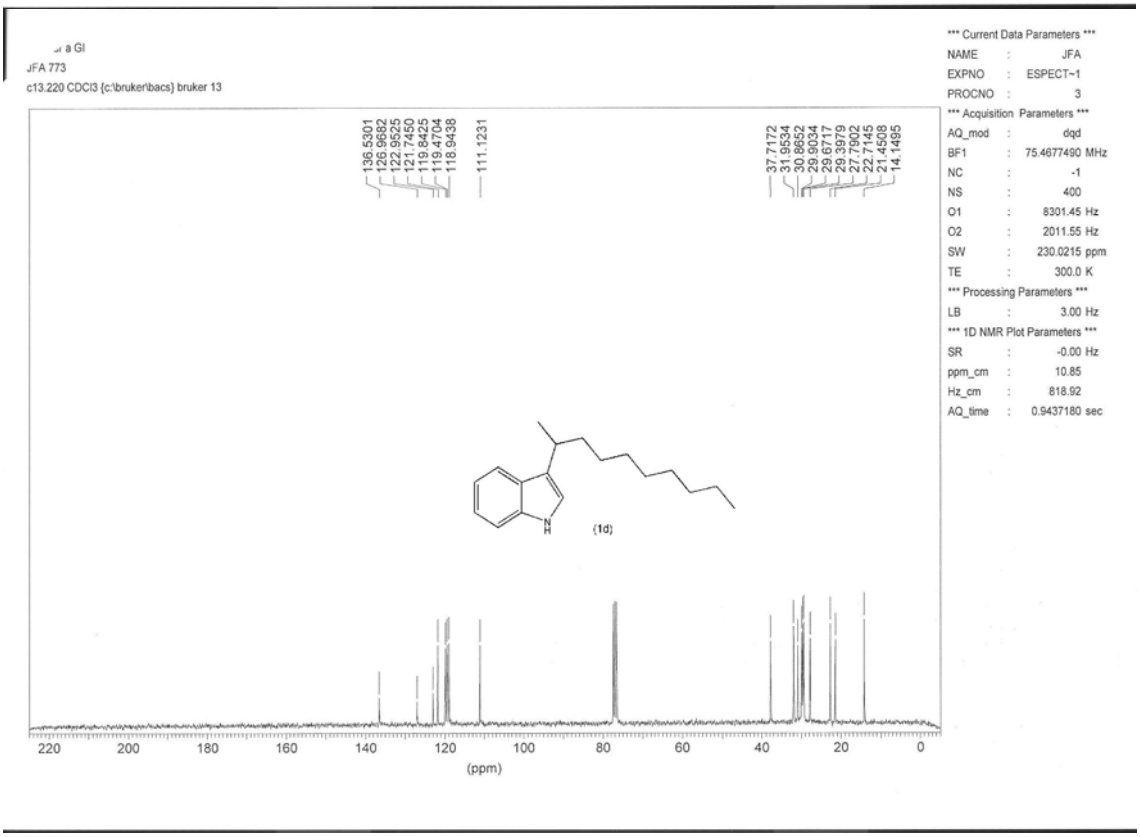
1.0634
0.9869
2.0273
1.0000
0.8969
1.0081
1.1461
15.231
3.1122
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec


JFA 773
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136.5301
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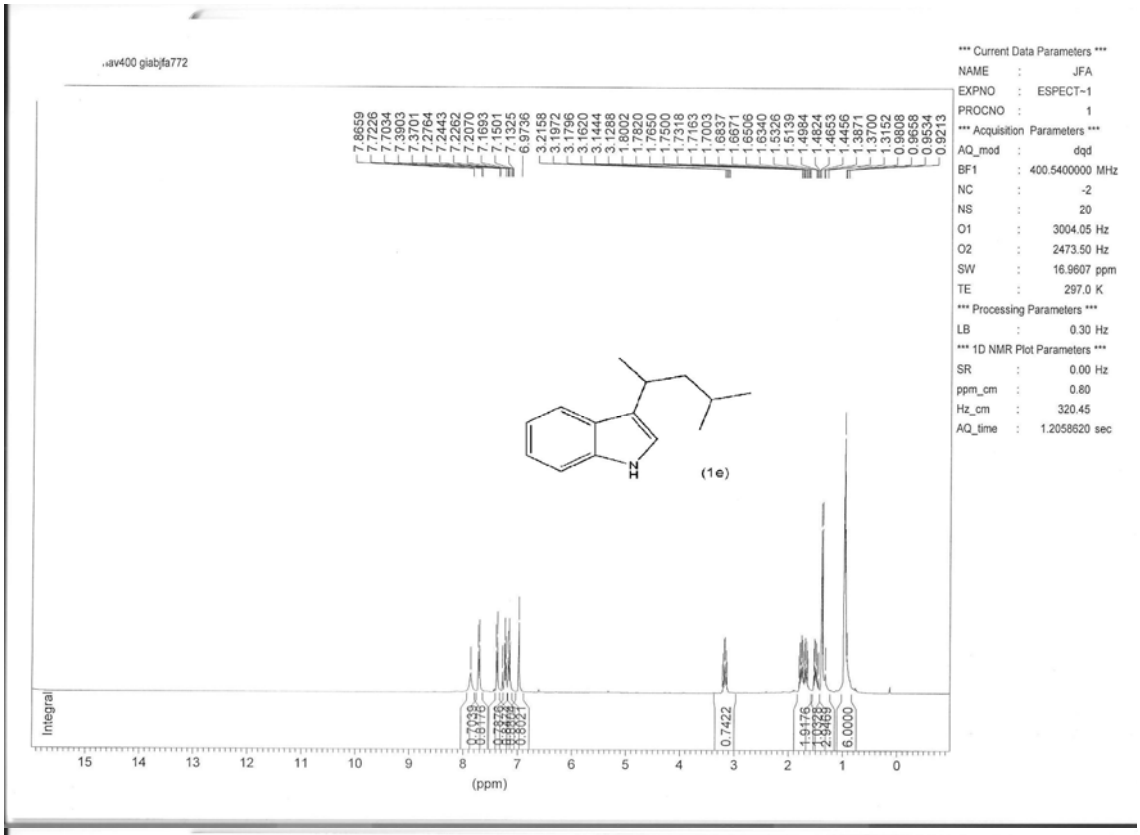
121.7450
119.8425
119.4704
118.9438
111.1231
37.7172
31.9634
30.8652
29.8934
29.5977
29.3979
29.1902
27.7145
21.4508
14.1495
(1d)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec


nav400 giabjfa772
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NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
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NC : -2
NS : 20
O1 : 3004.05 Hz
O2 : 2473.50 Hz
SW : 16.9607 ppm
TE : 297.0 K
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LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
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ppm_cm : 0.80
Hz_cm : 320.45
AQ_time : 1.2058620 sec
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Integral
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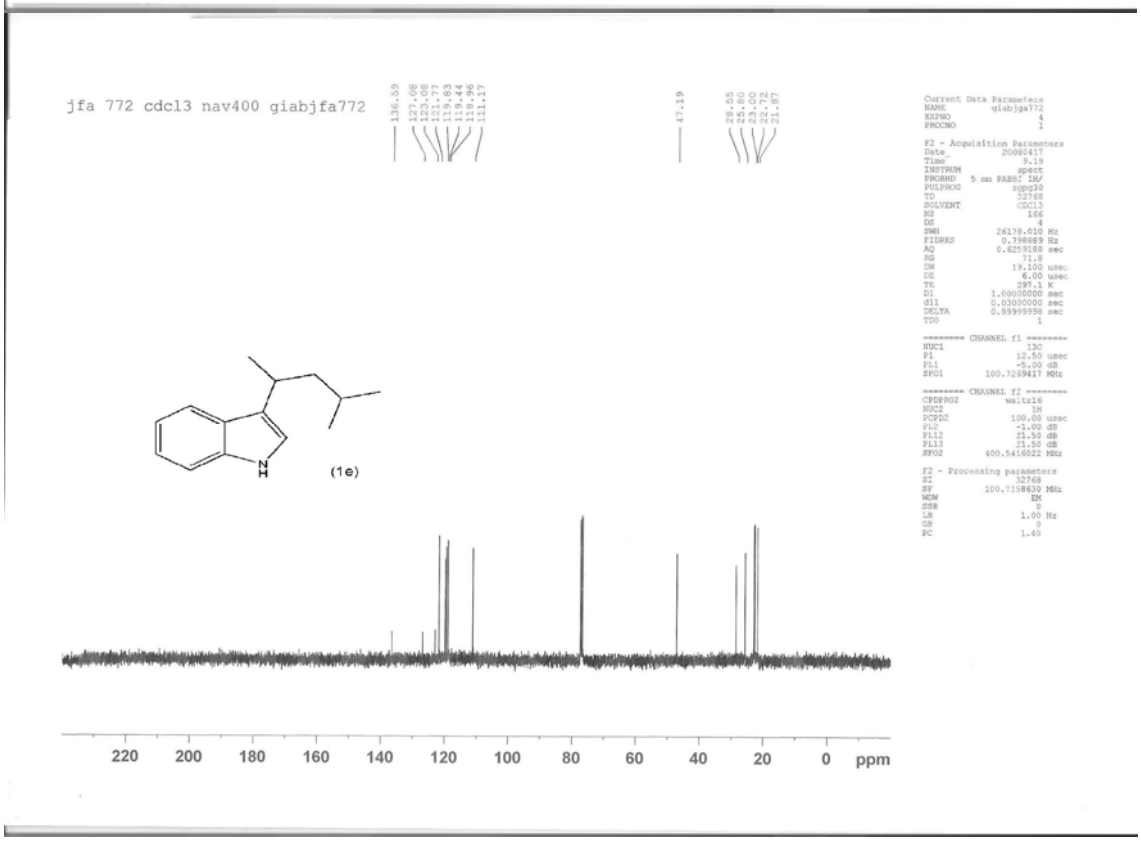
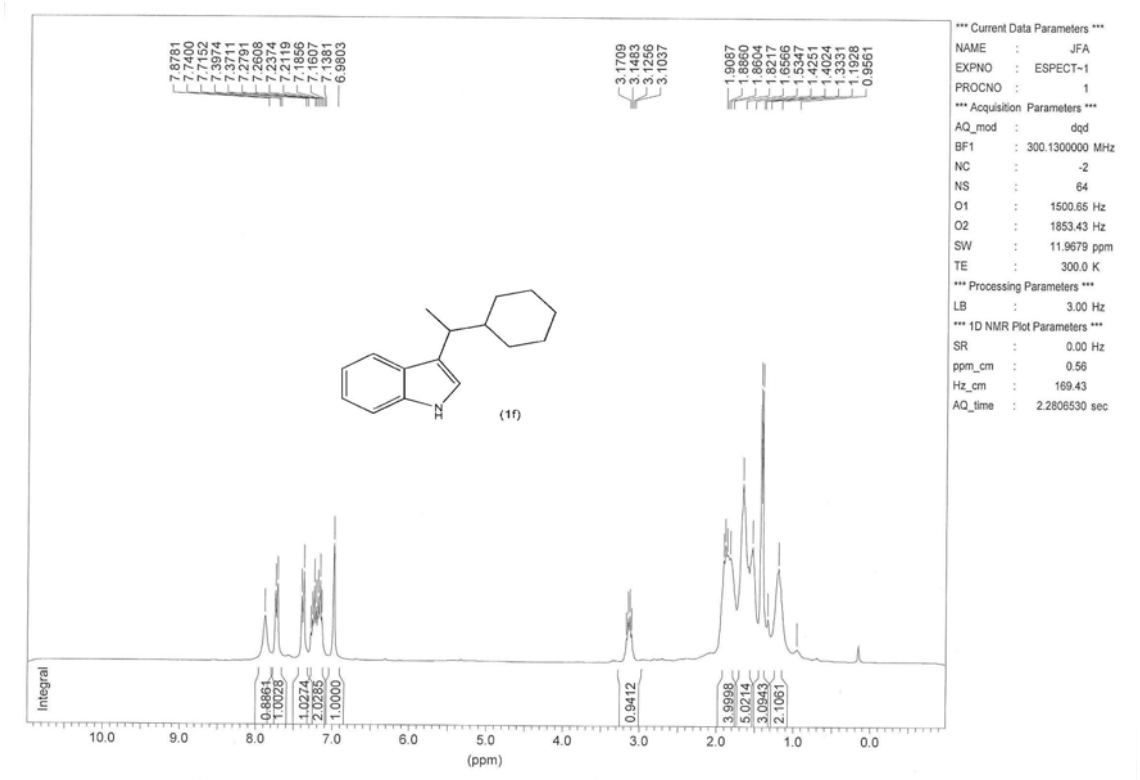
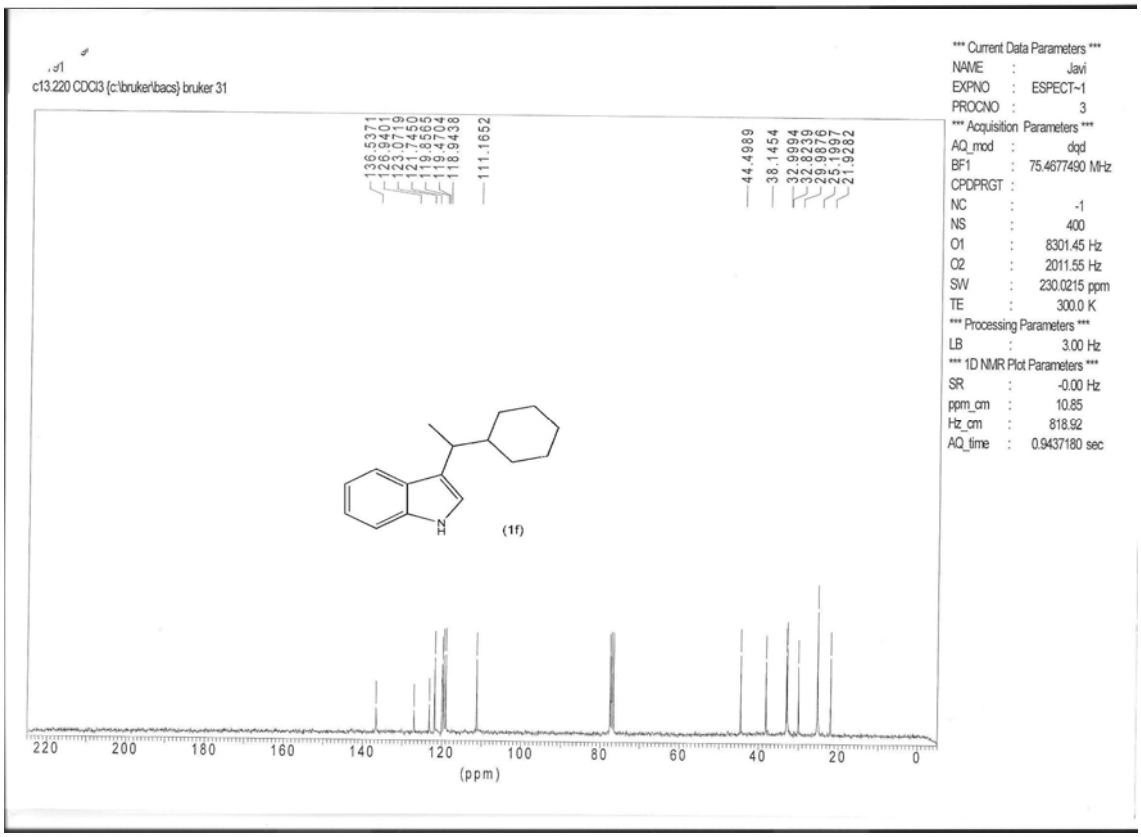
6.0000
(ppm)


jfa 772 cdcl3 nav400 giabjfa772
(1e)
ppm


7.8781
7.7400
7.7152
7.3974
7.3711
7.2791
7.2608
7.2374
7.2119
7.1856
7.1607
7.1381
6.9803
3.1709
3.1483
3.1256
3.1037
1.9087
1.8860
1.8604
1.8217
1.6566
1.5347
1.4251
1.4024
1.3331
1.1928
0.9561
(1f)
Integral
0.8861
1.0028
1.0274
2.0285
1.0000
0.9412
3.9998
5.0214
3.0943
2.1061
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec


c13.220 CDCl3 {c:\bruker\bacs} bruker 31
136.5371
126.9401
126.0719
121.7450
119.8555
119.4704
118.9438
111.1652
44.4989
38.1454
32.9994
32.8239
29.9876
25.1997
21.9282
(1f)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
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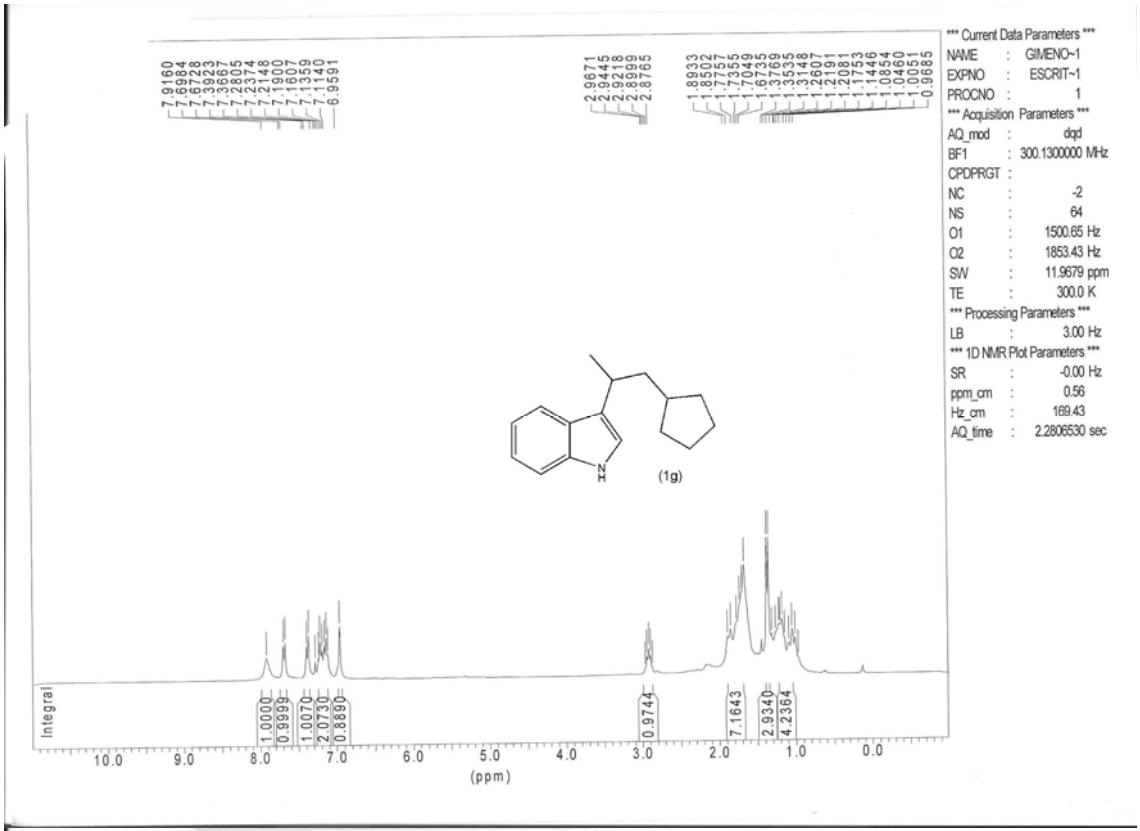
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec


*** Current Data Parameters ***
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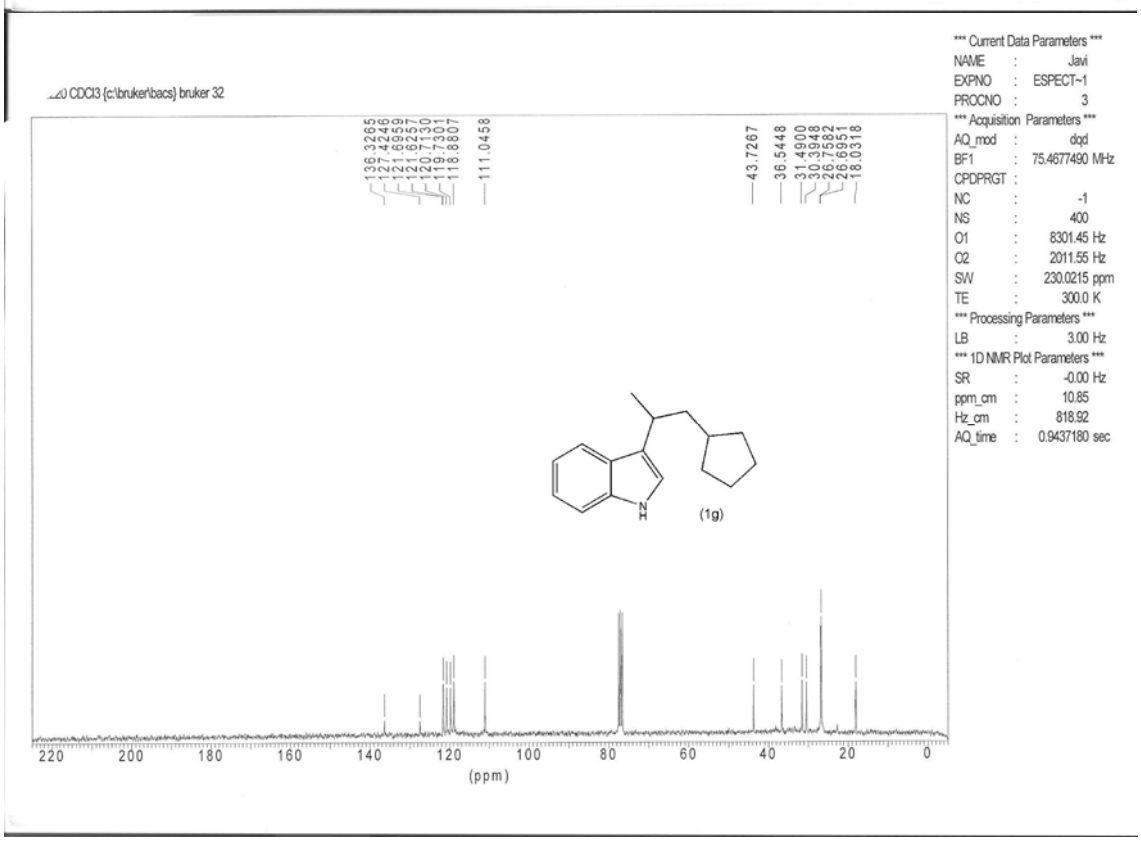
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
(1g)
Integral
1.0000
0.999
1.0070
2.0730
0.8890
0.9744
7.1643
2.9340
4.2364
(ppm)


*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
CDCl3 {c:\bruker\bacs} bruker 32
136.3265
127.4246
121.6597
121.2257
120.7130
119.7301
118.8807
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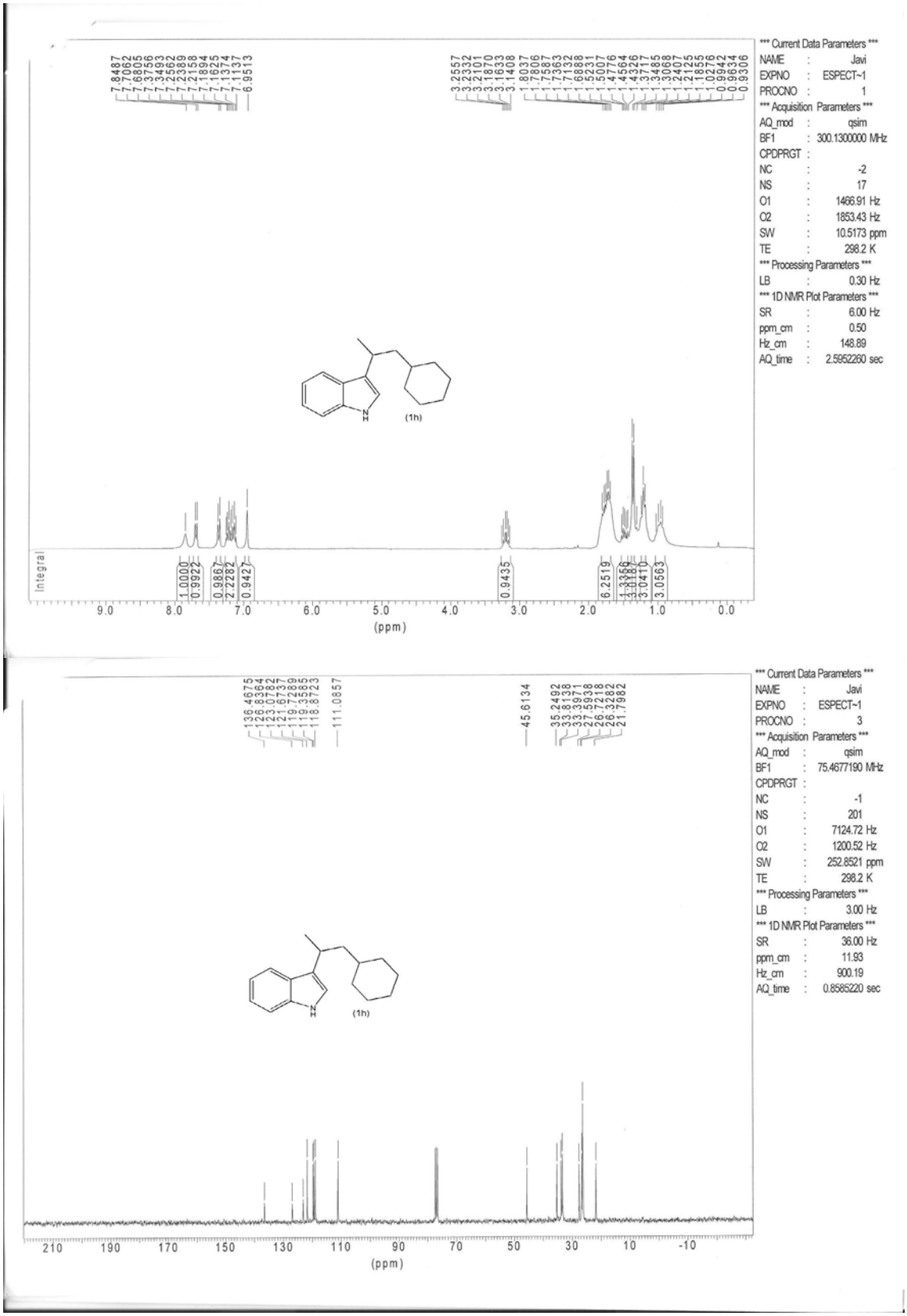
26.7882
26.6931
18.0318
(1g)
(ppm)


*** Current Data Parameters ***
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EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
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CPDPRGT :
NC : -2
NS : 17
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O2 : 1853.43 Hz
SW : 10.5173 ppm
TE : 298.2 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 6.00 Hz
ppm_cm : 0.50
Hz_cm : 148.89
AQ_time : 2.5952260 sec
(1h)
Integral
(ppm)
*** Current Data Parameters ***
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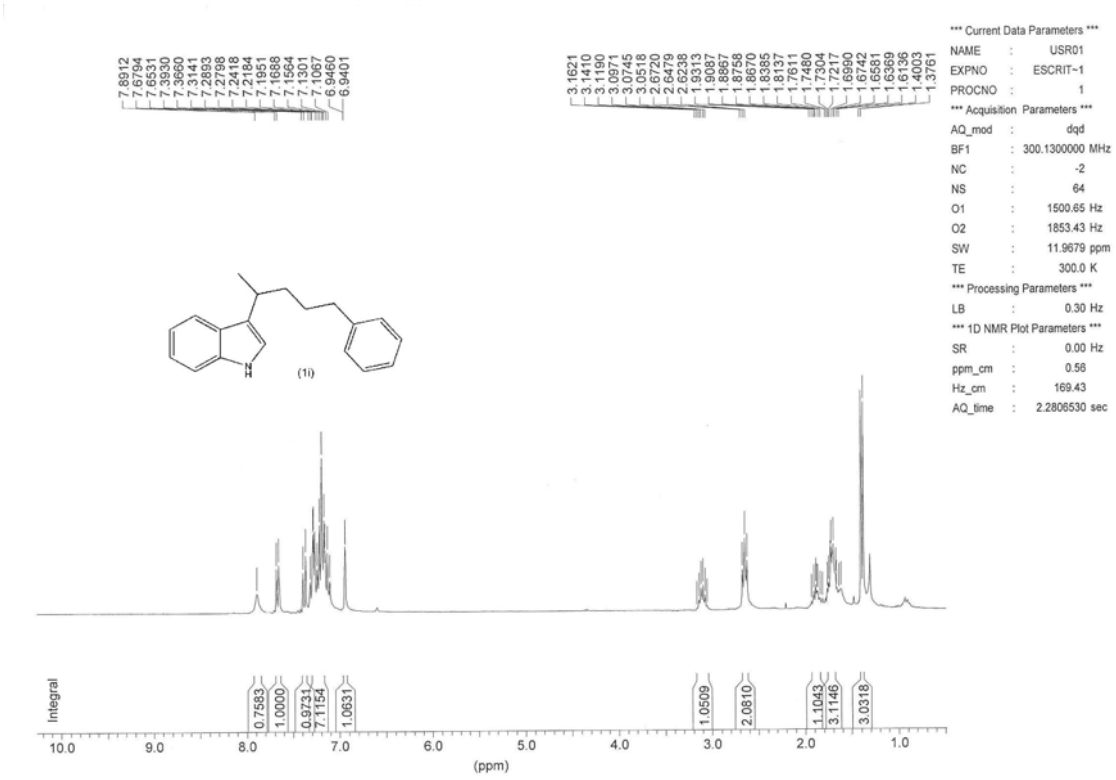
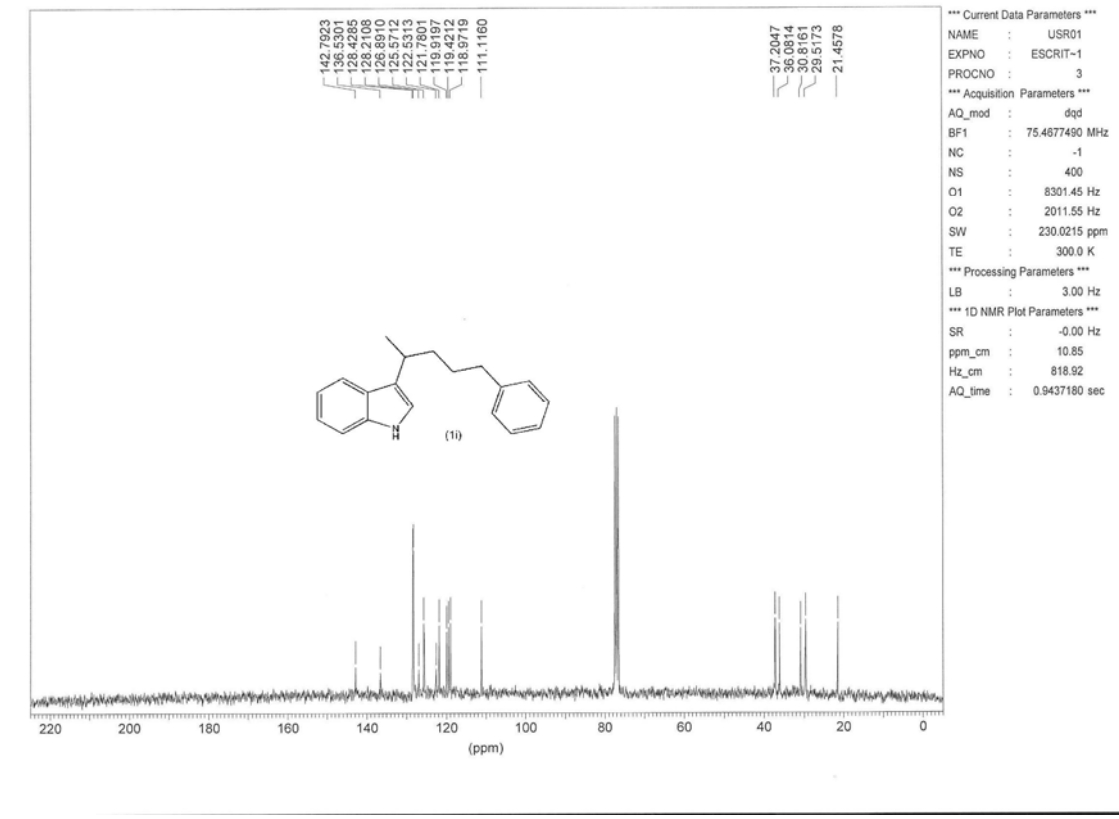
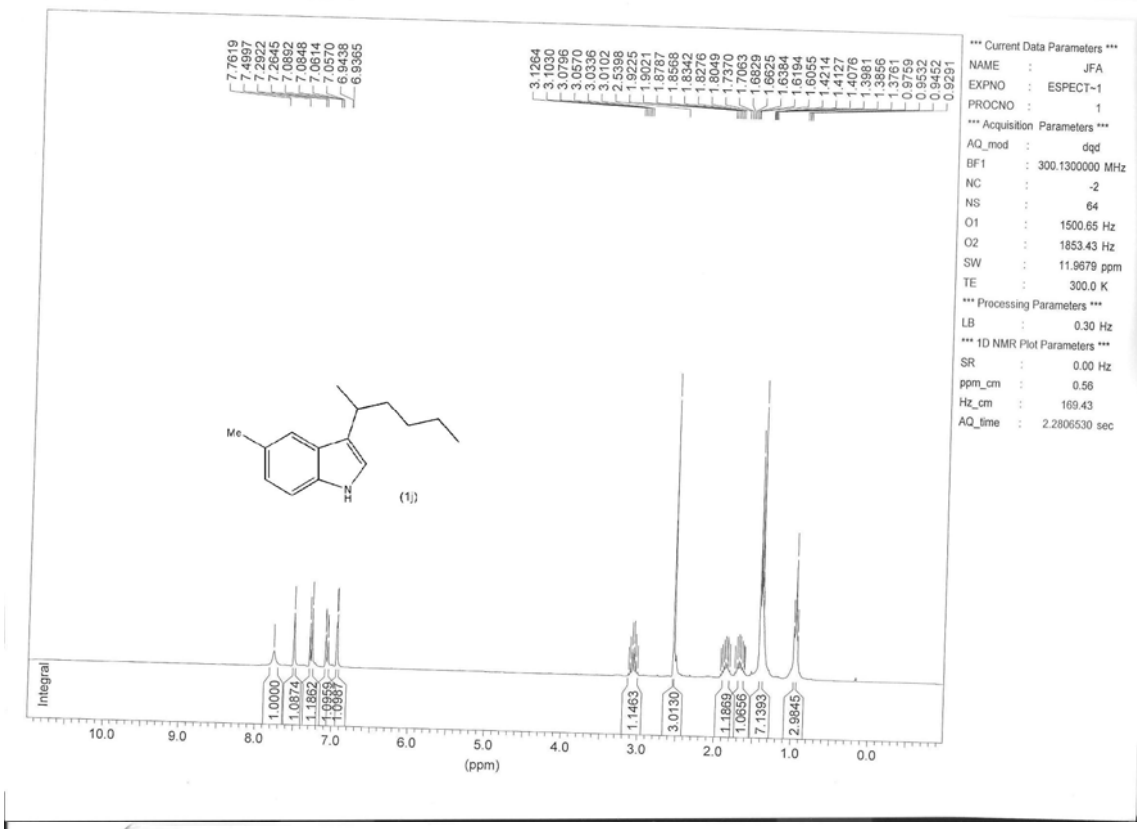
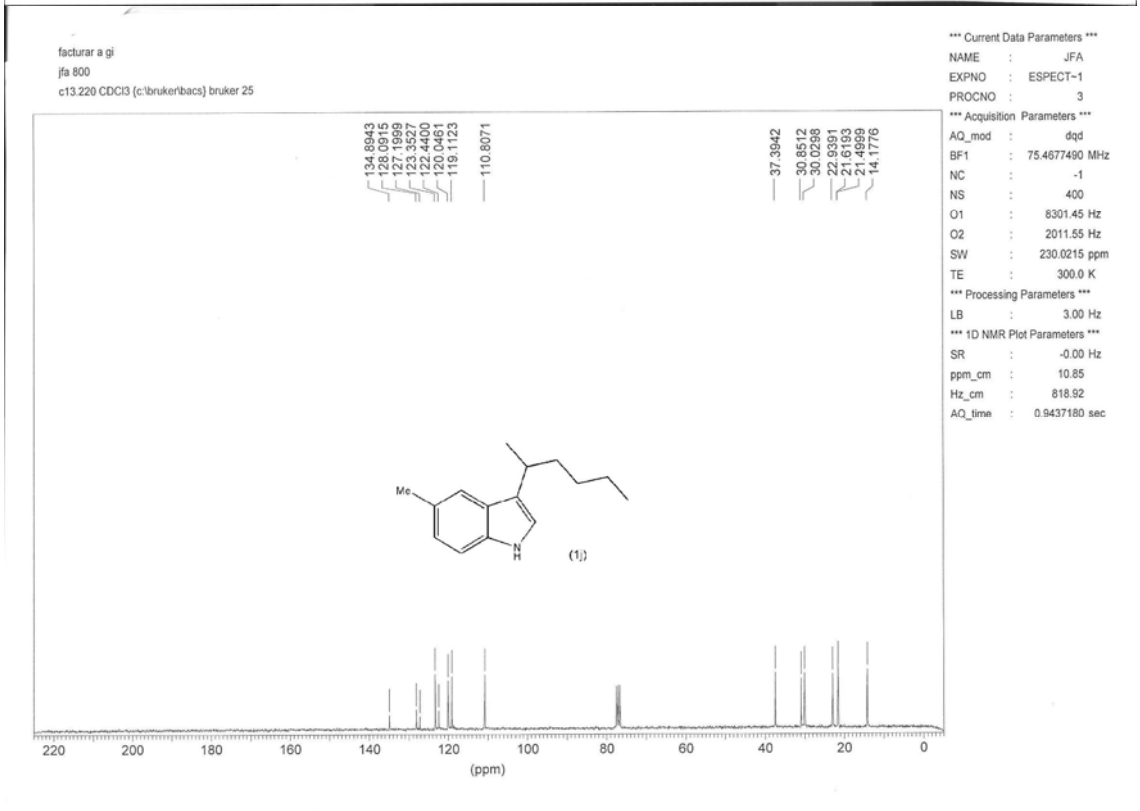
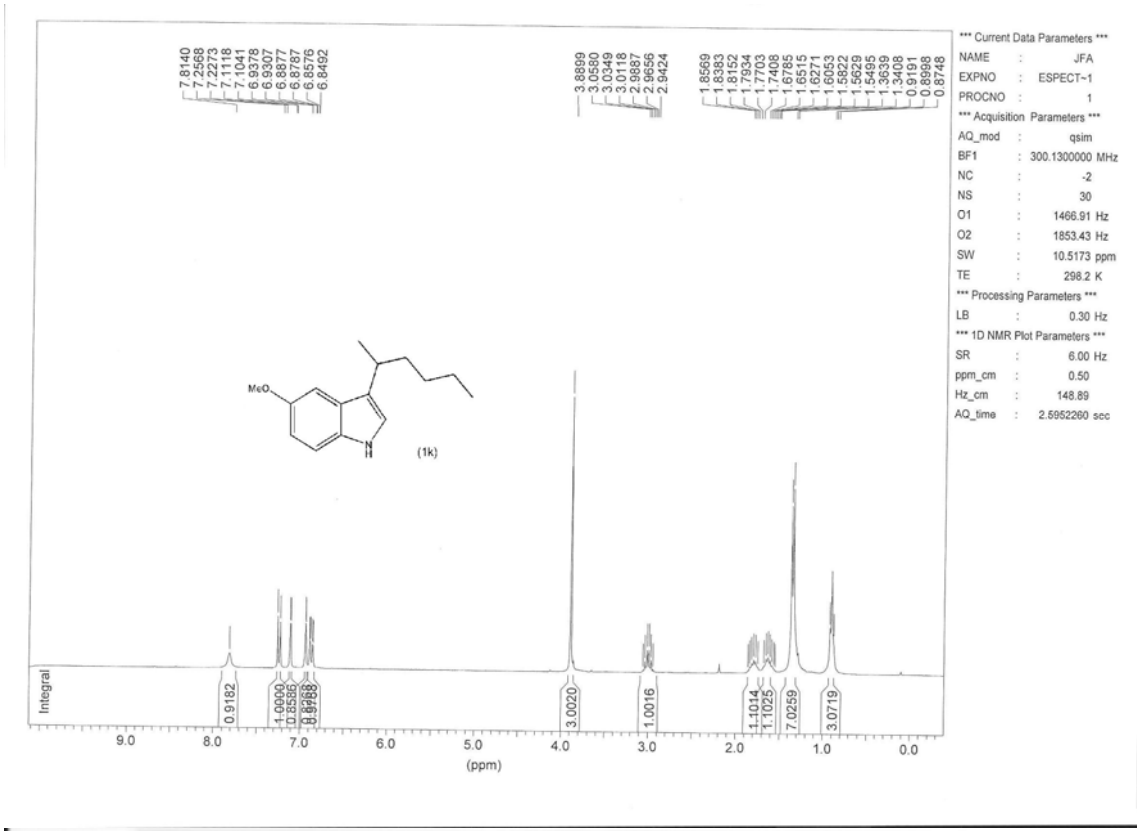
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 75.4677190 MHz
CPDPRGT :
NC : -1
NS : 201
O1 : 7124.72 Hz
O2 : 1200.52 Hz
SW : 252.8521 ppm
TE : 298.2 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 36.00 Hz
ppm_cm : 11.93
Hz_cm : 900.19
AQ_time : 0.8585220 sec
(1h)
(ppm)

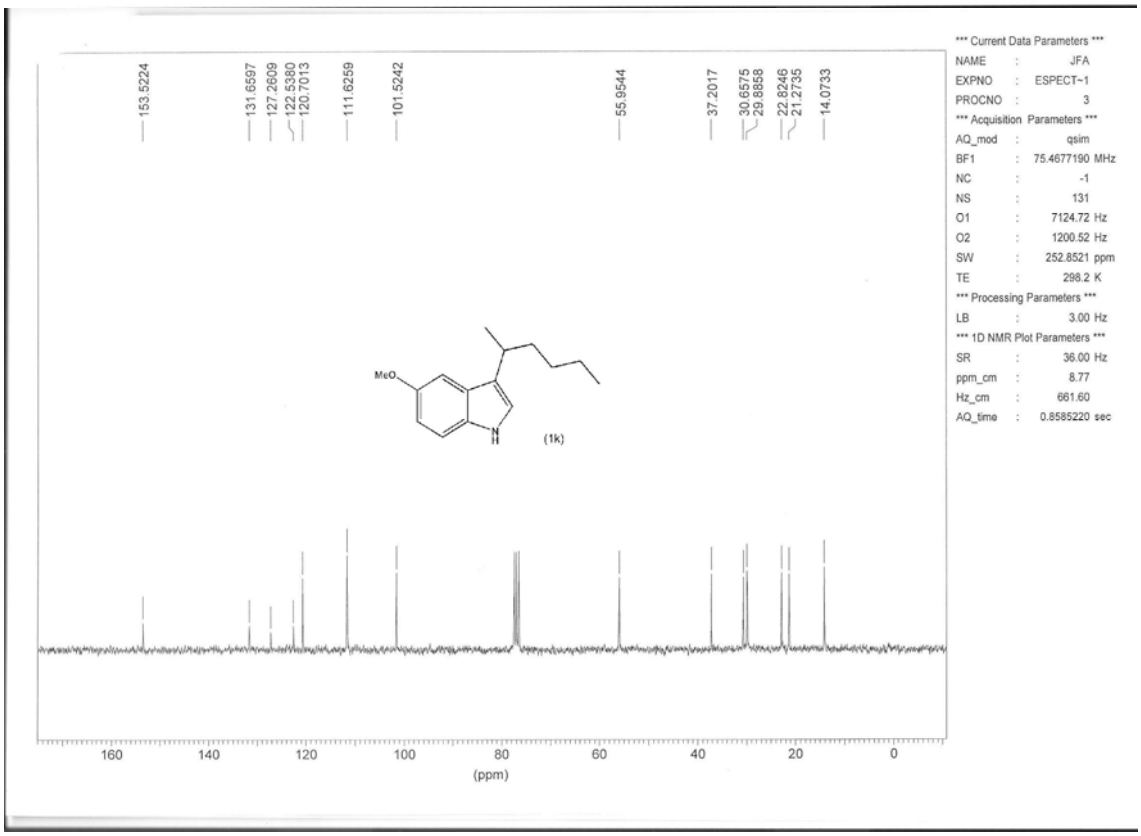

7.8912
7.6794
7.6531
7.3930
7.3660
7.3141
7.2893
7.2798
7.2418
7.2184
7.1951
7.1688
7.1564
7.1301
7.1067
6.9460
6.9401
3.1621
3.1410
3.1190
3.0971
3.0745
3.0518
2.6720
2.6479
2.6238
1.9313
1.9087
1.8867
1.8758
1.8670
1.8385
1.8137
1.7611
1.7480
1.7304
1.7217
1.6990
1.6742
1.6581
1.6369
1.6136
1.4003
1.3761
(1i)
*** Current Data Parameters ***
NAME : USR01
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
Integral
0.7583
1.0000
0.9731
7.1154
1.0631
1.0509
2.0810
1.1043
3.1146
3.0318
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)

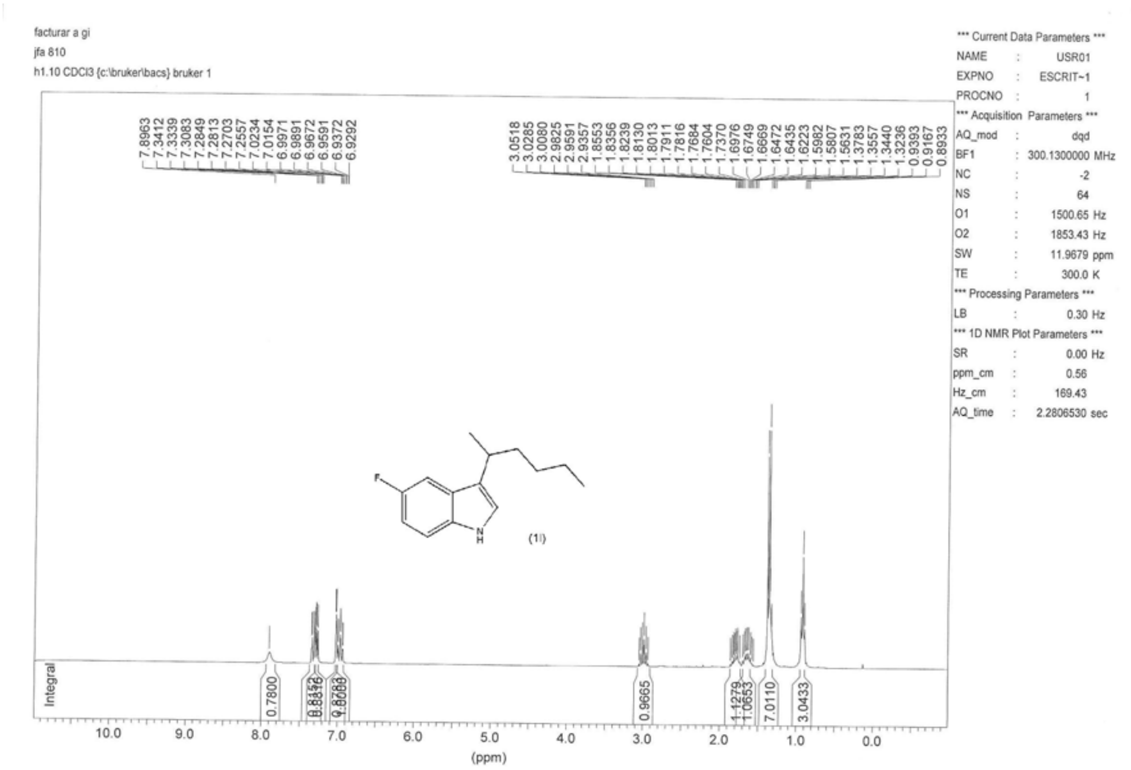

142.7923
136.5301
128.4285
128.2108
126.8910
125.5712
122.5313
121.7801
119.9197
119.4212
118.9719
111.1160
37.2047
36.0814
30.8161
29.5173
21.4578
(1i)
*** Current Data Parameters ***
NAME : USR01
EXPNO : ESCRIT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

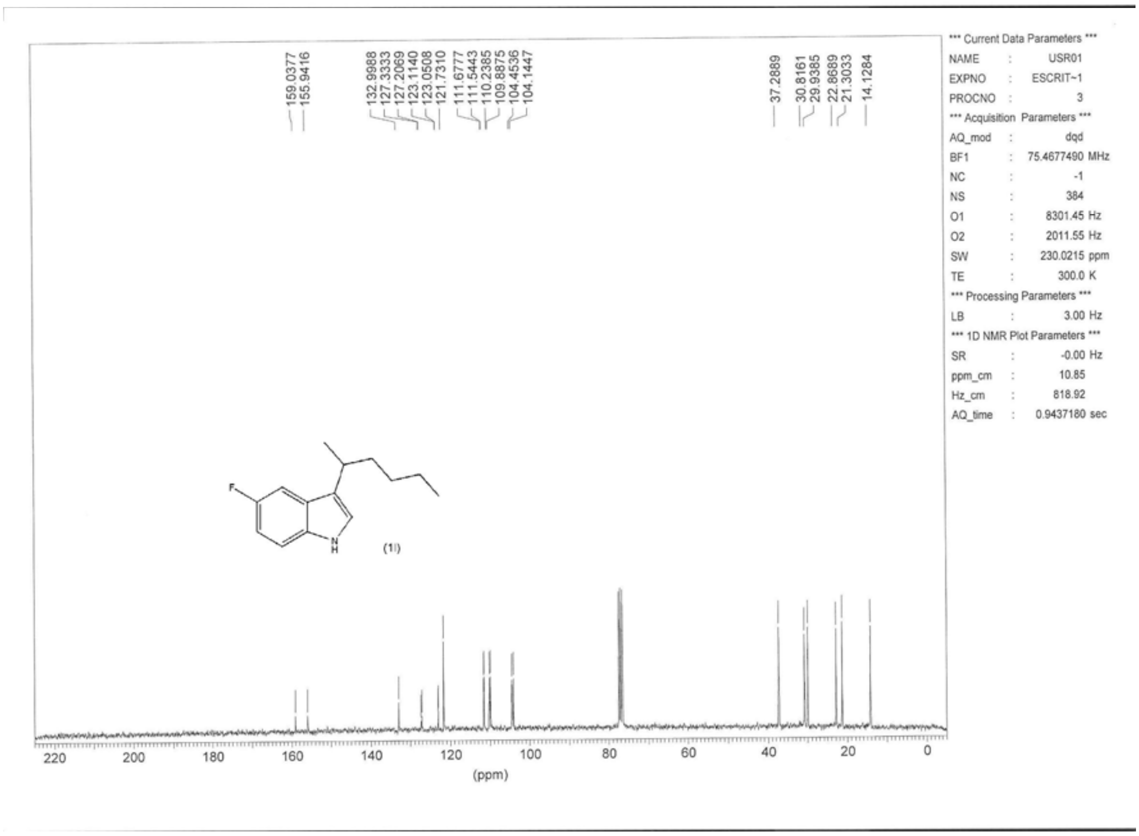

7.7619
7.4997
7.2922
7.2645
7.0892
7.0848
7.0614
7.0570
6.9438
6.9365
3.1264
3.1030
3.0796
3.0570
3.0336
3.0102
2.9398
1.9225
1.8971
1.8767
1.8568
1.8342
1.8276
1.8049
1.7370
1.7063
1.6829
1.6625
1.6384
1.6194
1.6055
1.4214
1.4127
1.4076
1.3981
1.3856
1.3761
0.9759
0.9532
0.9452
0.9291
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
Me
N
H
(1j)
Integral
1.0000
1.0874
1.1862
1.0569
1.0987
1.1463
3.0130
1.1869
1.0656
7.1393
2.9845
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

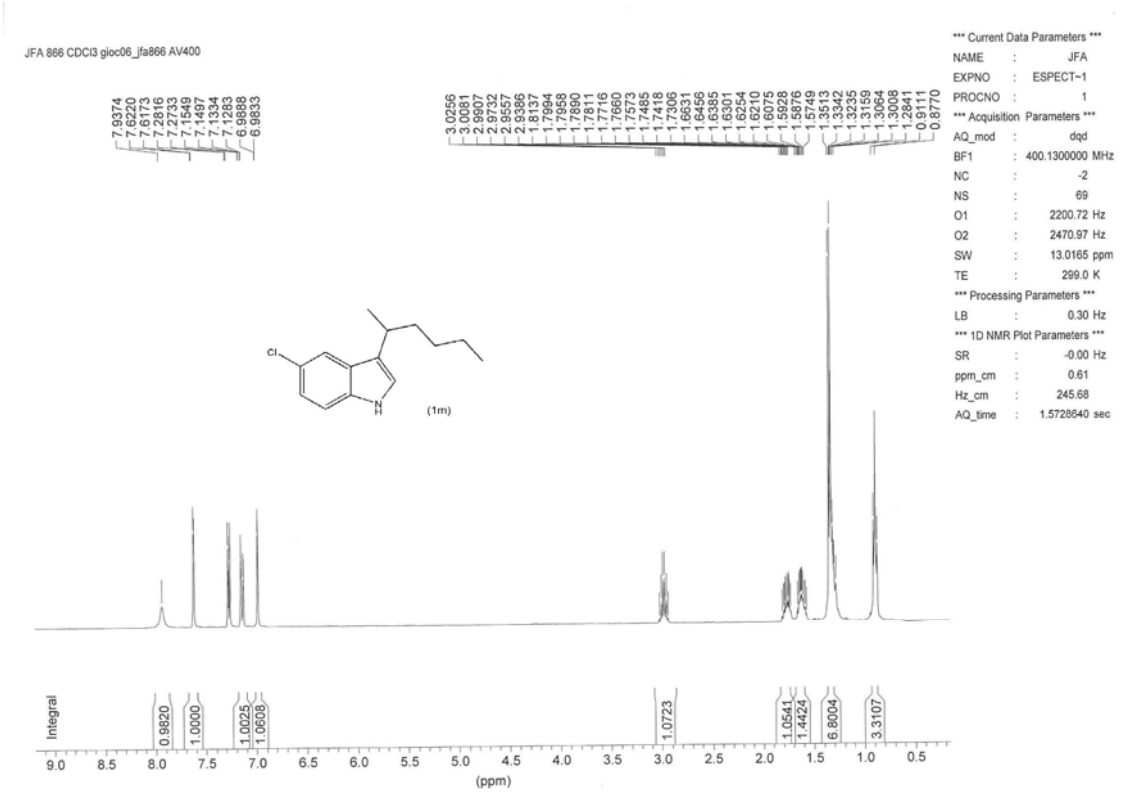

facturar a gi
jfa 800
c13.220 CDCl3 {c:\bruker\bacs} bruker 25
134.8943
128.0915
127.1999
123.3527
122.4400
120.0461
119.1123
110.8071
37.3942
30.8512
30.0298
22.9391
21.6193
21.4999
14.1776
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
Me
N
H
(1j)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

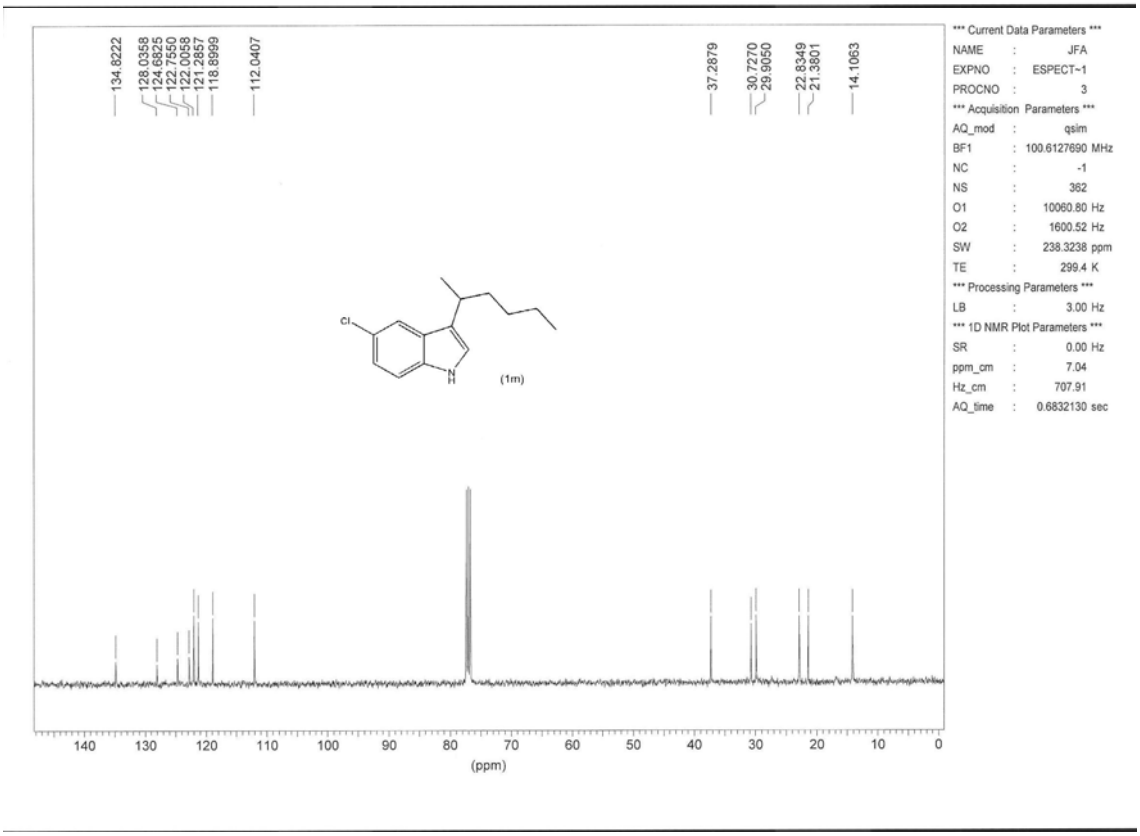

7.8140
7.2568
7.2273
7.1118
7.0941
6.9378
6.8977
6.8787
6.8576
6.8492
3.8899
3.0580
3.0349
3.0118
2.9887
2.9656
2.9424
1.8569
1.8383
1.8152
1.7934
1.7703
1.7408
1.6785
1.6515
1.6271
1.6053
1.5822
1.5629
1.5495
1.3639
1.3469
0.9191
0.8988
0.8748
MeO
(1k)
Integral
0.9182
1.0000
0.8586
0.8068
0.9768
3.0020
1.0016
1.1014
1.1025
7.0259
3.0719
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 300.1300000 MHz
NC : -2
NS : 30
O1 : 1466.91 Hz
O2 : 1853.43 Hz
SW : 10.5173 ppm
TE : 298.2 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 6.00 Hz
ppm_cm : 0.50
Hz_cm : 148.89
AQ_time : 2.5952260 sec

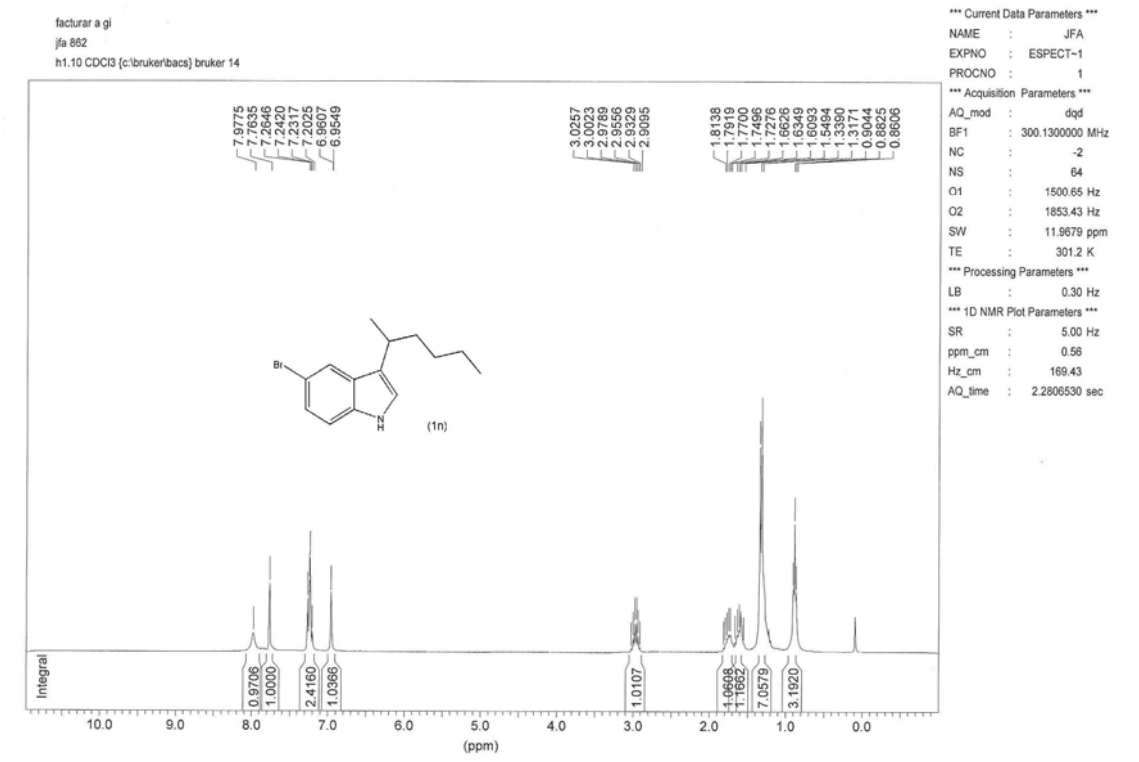

153.5224
131.6597
127.2609
122.5380
120.7013
111.6259
101.5242
55.9544
37.2017
30.6575
29.8858
22.8246
21.2735
14.0733
MeO
(1k)
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 75.4677190 MHz
NC : -1
NS : 131
O1 : 7124.72 Hz
O2 : 1200.52 Hz
SW : 252.8521 ppm
TE : 298.2 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 36.00 Hz
ppm_cm : 8.77
Hz_cm : 661.60
AQ_time : 0.8585220 sec

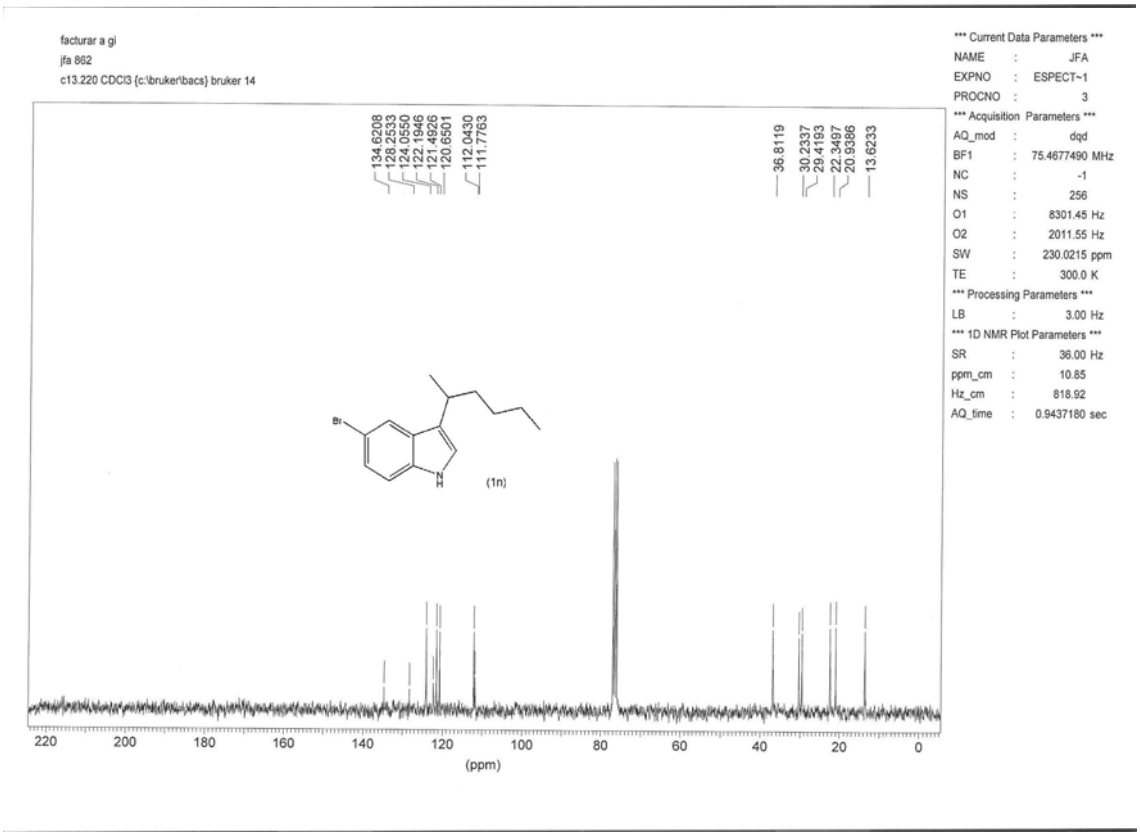

facturar a gi
jfa 810
h1.10 CDCl3 {c:\bruker\bacs} bruker 1
7.8963 7.3412 7.3339 7.3083 7.2849 7.2813 7.2703 7.2557 7.0234 7.0154 6.9971 6.9891 6.9672 6.9591 6.9372 6.9292
3.0518 3.0285 3.0080 2.9825 2.9591 2.9357 1.8553 1.8356 1.8239 1.8130 1.8013 1.7911 1.7816 1.7684 1.7604 1.7370 1.6976 1.6749 1.6669 1.6472 1.6435 1.6223 1.5982 1.5807 1.5631 1.3783 1.3557 1.3440 1.3236 0.9393 0.9167 0.8933
*** Current Data Parameters ***
NAME : USR01
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
F
N
H
(1l)
Integral
0.7800
0.9665
7.0110
3.0433
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
(ppm)

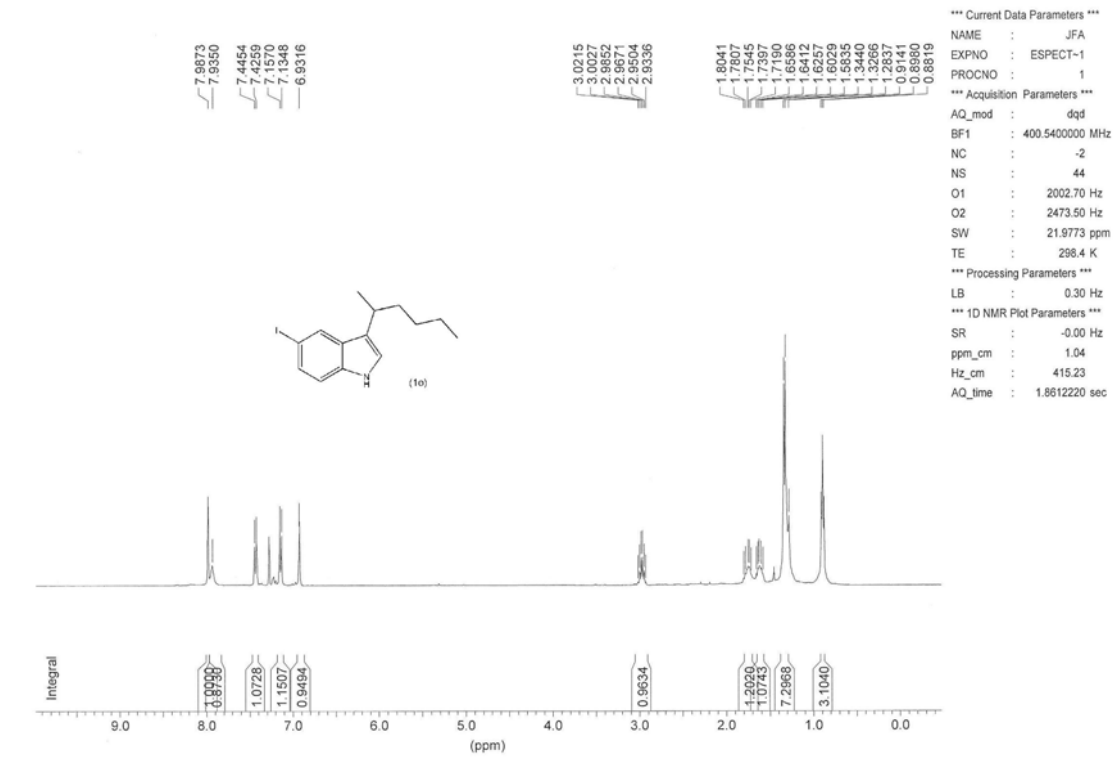

159.0377 155.9416
132.9988 127.3333 127.2069 123.1140 123.0508 121.7310 111.6777 111.5443 110.2385 109.8875 104.4536 104.1447
37.2889 30.8161 29.9385 22.8689 21.3033 14.1284
*** Current Data Parameters ***
NAME : USR01
EXPNO : ESCRIT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 384
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
F
N
H
(1l)
220 200 180 160 140 120 100 80 60 40 20 0
(ppm)

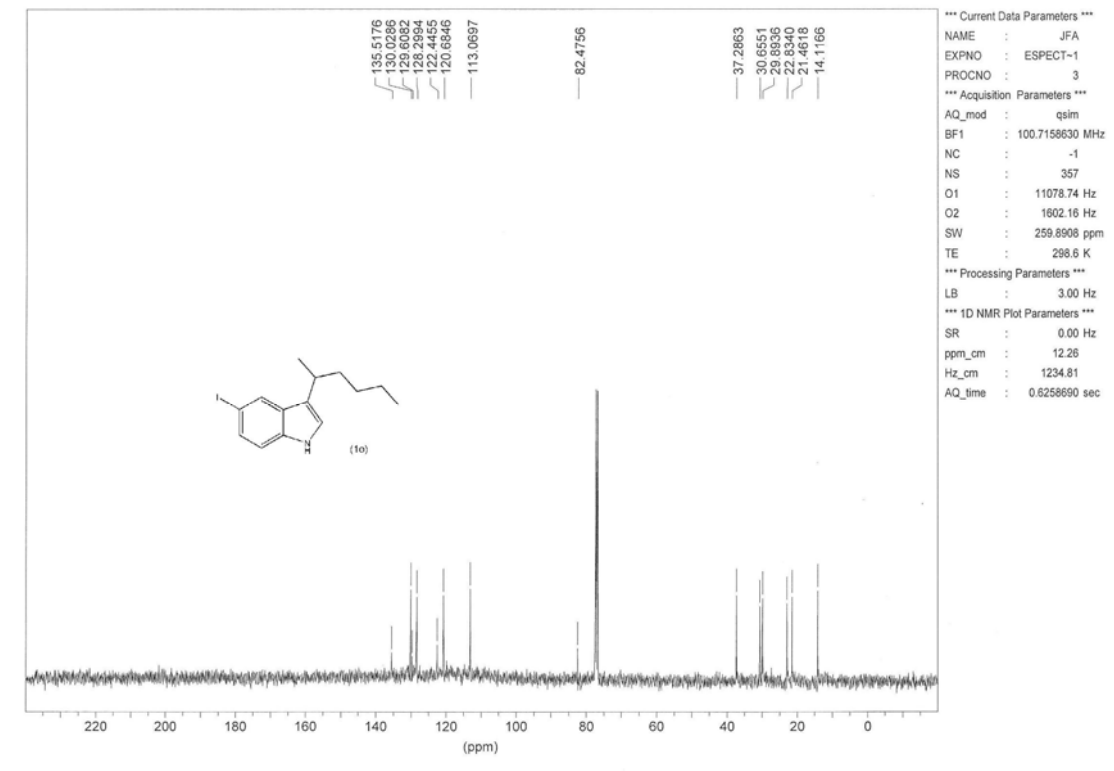

JFA 866 CDCl3 gioc06_jfa866 AV400
7.9374
7.6220
7.6173
7.2816
7.2733
7.1549
7.1497
7.1334
7.1283
6.9888
6.9833
3.0256
3.0081
2.9907
2.9732
2.9557
2.9386
1.8137
1.7984
1.7958
1.7880
1.7811
1.7716
1.7660
1.7573
1.7485
1.7418
1.7306
1.6631
1.6456
1.6385
1.6301
1.6254
1.6210
1.6075
1.5928
1.5876
1.5749
1.3513
1.3342
1.3235
1.3159
1.3064
1.3008
1.2841
0.9111
0.8770
Cl
N
(1m)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 400.1300000 MHz
NC : -2
NS : 69
O1 : 2200.72 Hz
O2 : 2470.97 Hz
SW : 13.0165 ppm
TE : 299.0 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.61
Hz_cm : 245.68
AQ_time : 1.5728640 sec
Integral
0.9820
1.0000
1.0025
1.0608
1.0723
1.0541
1.4424
6.8004
3.3107
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
(ppm)

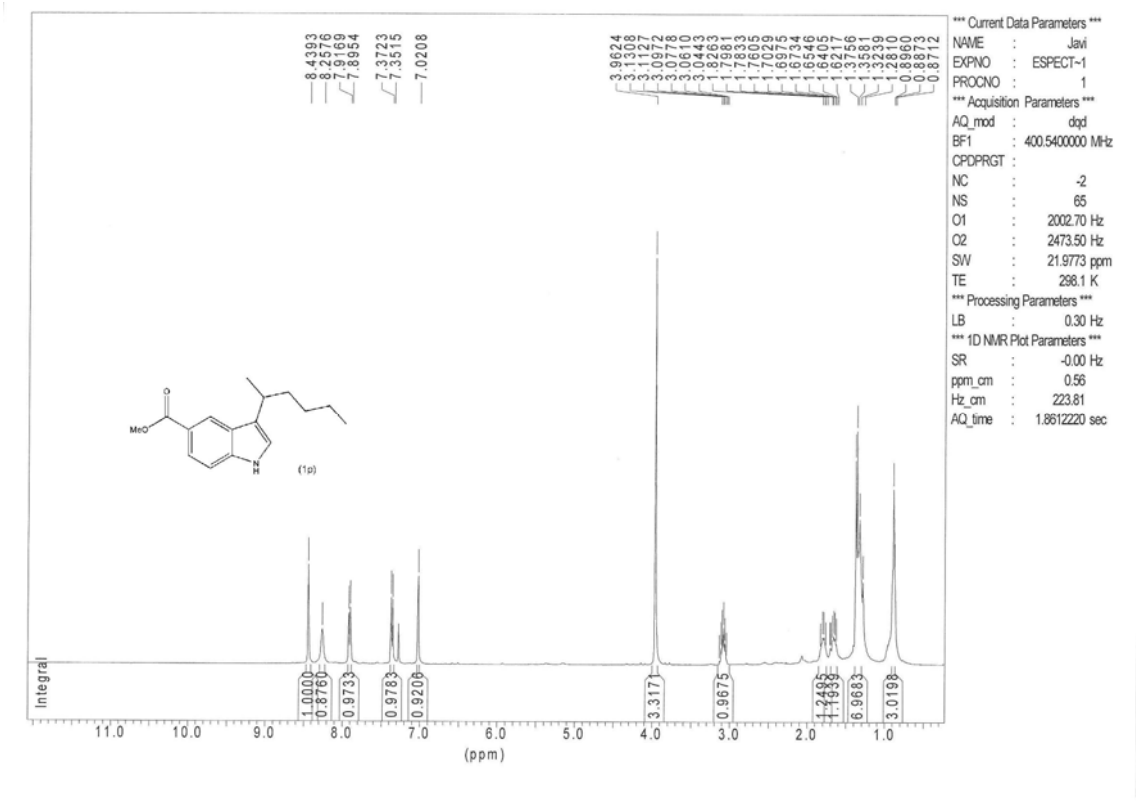

134.8222
128.0358
124.6825
122.7550
122.0058
121.2857
118.8999
112.0407
37.2879
30.7270
29.9050
22.8349
21.3801
14.1063
Cl
N
(1m)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 100.6127690 MHz
NC : -1
NS : 382
O1 : 10060.80 Hz
O2 : 1600.52 Hz
SW : 238.3238 ppm
TE : 299.4 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 7.04
Hz_cm : 707.91
AQ_time : 0.6832130 sec
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

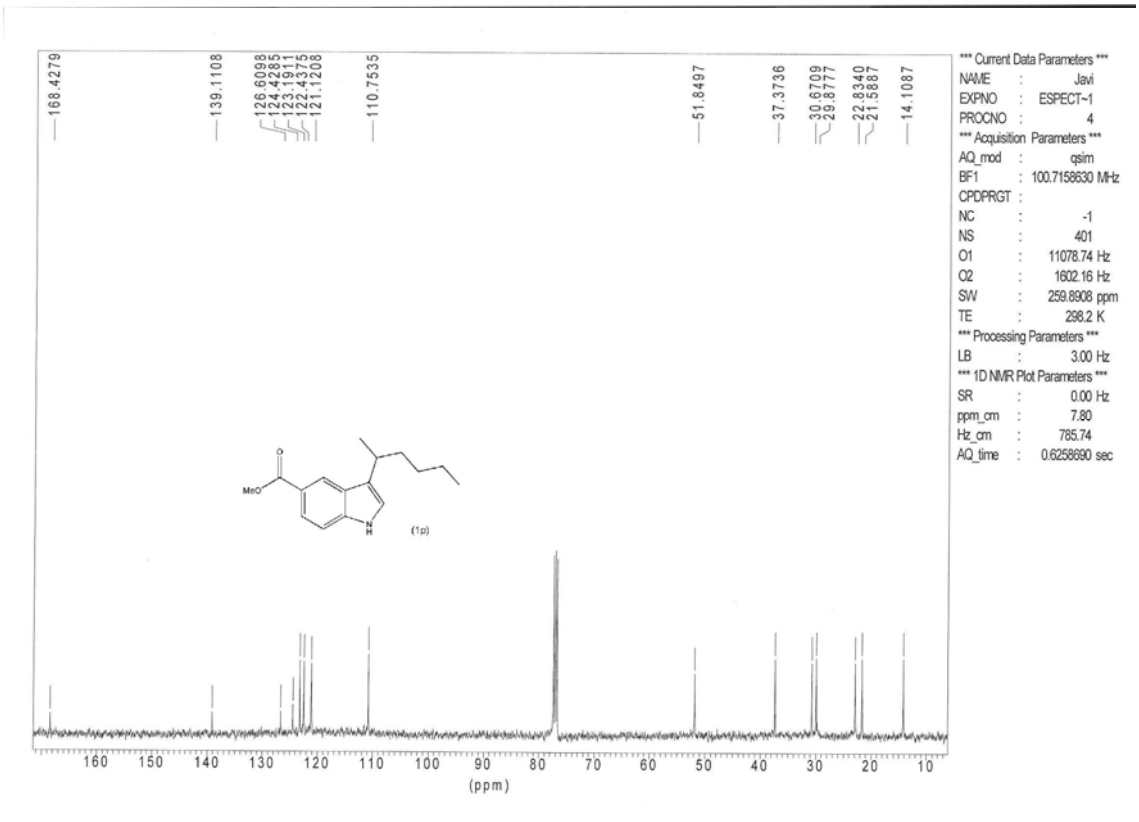

facturar a gi
jfa 862
h1.10 CDCl3 {c:\bruker\bacs} bruker 14
7.9775
7.7635
7.2846
7.2420
7.2317
7.2025
6.9607
6.9549
3.0257
3.0023
2.9769
2.9755
2.9329
2.9095
1.8138
1.7919
1.7700
1.7496
1.7276
1.6626
1.6549
1.6093
1.5494
1.3390
1.3171
0.9044
0.8825
0.8606
Br
N
H
(1n)
Integral
0.9706
1.0000
2.4160
1.0366
1.0107
1.0608
1.1662
7.0579
3.1920
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 301.2 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 5.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec

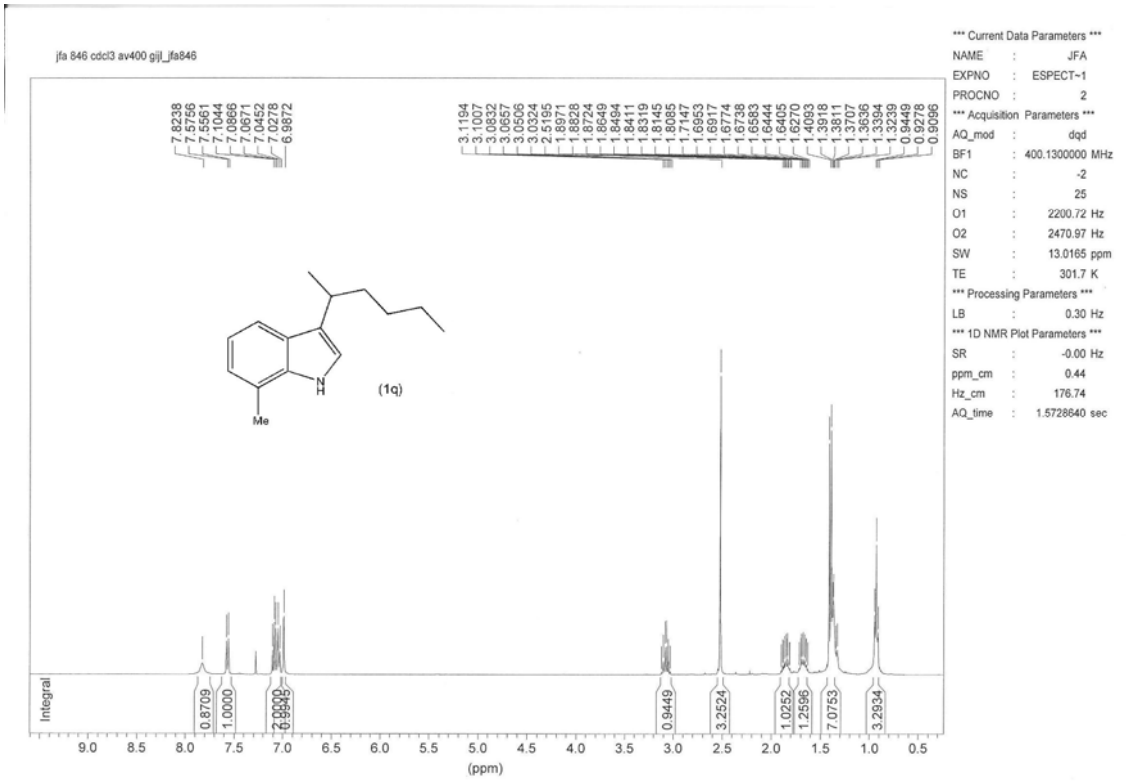

facturar a gi
jfa 862
c13.220 CDCl3 {c:\bruker\bacs} bruker 14
134.6208
128.2533
124.0550
122.1946
121.4926
120.6501
112.0430
111.7763
36.8119
30.2337
29.4193
22.3497
20.9386
13.6233
Br
N
H
(1n)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
NC : -1
NS : 256
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 36.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec

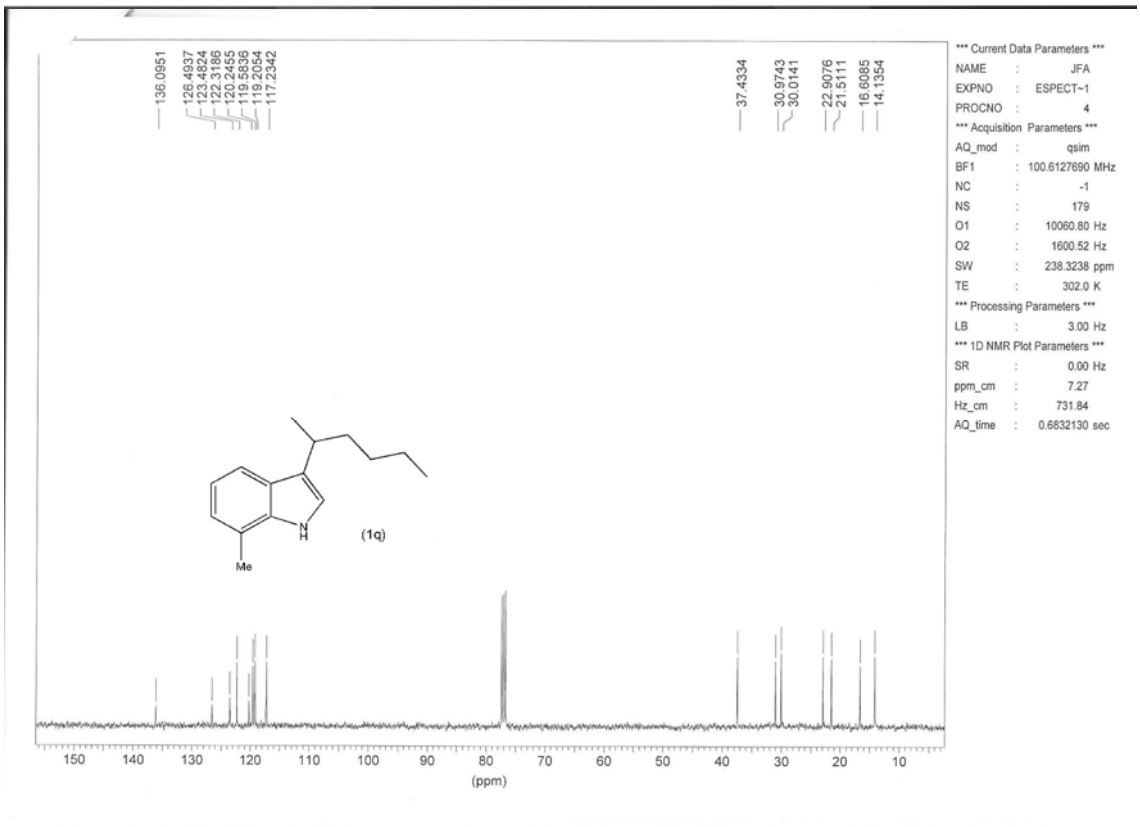

7.9873
7.9350
7.4454
7.4259
7.1570
7.1348
6.9316
3.0215
3.0027
2.9852
2.9671
2.9504
2.9336
1.8041
1.7807
1.7545
1.7397
1.7190
1.6586
1.6412
1.6257
1.6029
1.5835
1.3440
1.3266
1.2837
0.9141
0.8980
0.8819
(1o)
Integral
1.0000
0.8798
1.0728
1.1507
0.9494
0.9634
1.2020
1.0743
7.2968
3.1040
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 400.5400000 MHz
NC : -2
NS : 44
O1 : 2002.70 Hz
O2 : 2473.50 Hz
SW : 21.9773 ppm
TE : 298.4 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 1.04
Hz_cm : 415.23
AQ_time : 1.8612220 sec

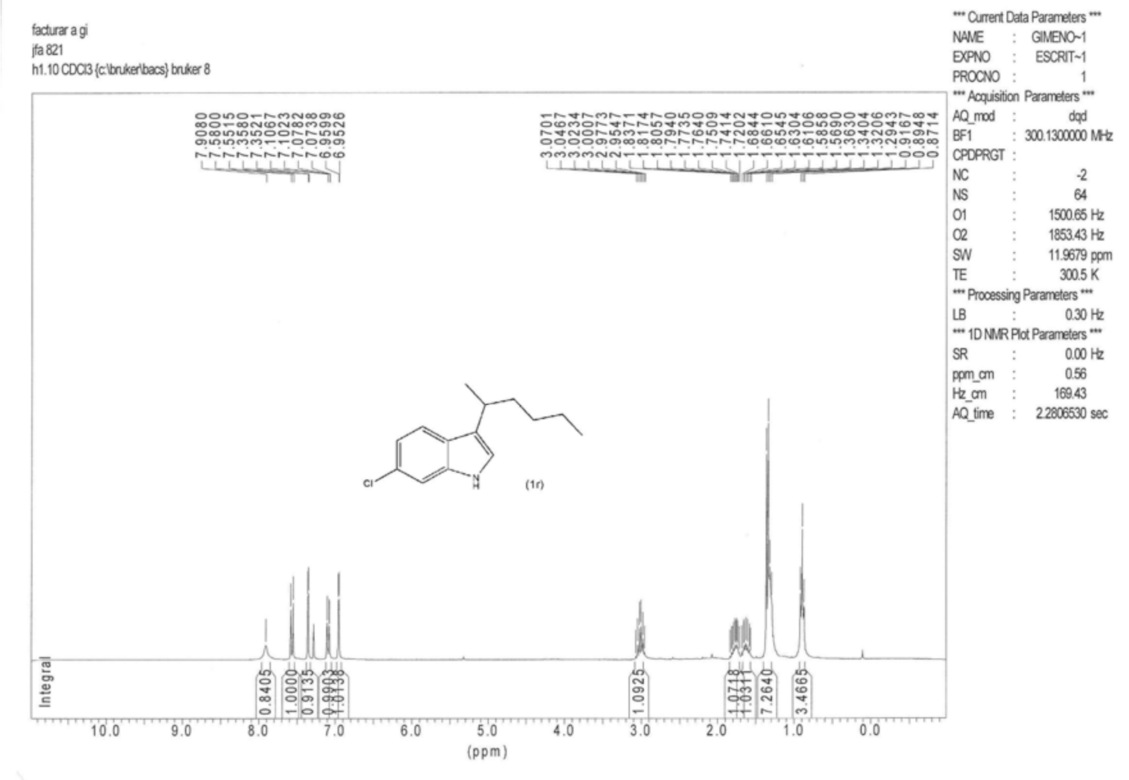

135.5176
130.0286
129.6082
128.2994
122.4455
120.6846
113.0697
82.4756
37.2863
30.6551
29.8836
22.8340
21.4618
14.1166
(1o)
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 100.7158630 MHz
NC : -1
NS : 357
O1 : 11078.74 Hz
O2 : 1602.16 Hz
SW : 259.8908 ppm
TE : 298.6 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 12.26
Hz_cm : 1234.81
AQ_time : 0.6258690 sec

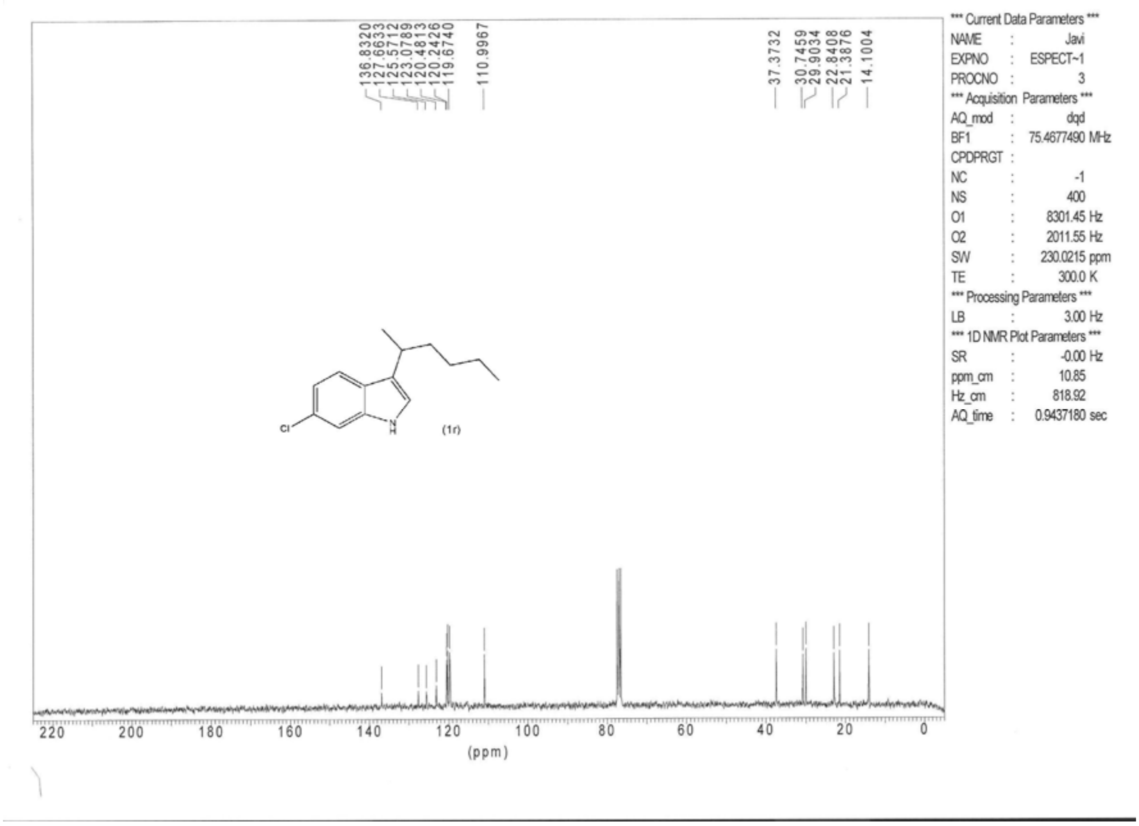

8.4393
8.2576
7.9169
7.8954
7.3723
7.3515
7.0208
3.9624
3.1309
3.1172
3.0778
3.0610
3.0443
1.8263
1.7981
1.7833
1.7605
1.7029
1.6975
1.6734
1.6546
1.6405
1.6217
1.3756
1.3581
1.3239
1.2810
0.8960
0.8873
0.8712
MeO
(1p)
Integral
1.0000
0.8760
0.9733
0.9783
0.9206
3.3171
0.9675
1.2495
1.1939
6.9683
3.0198
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 400.5400000 MHz
CPDPRGT :
NC : -2
NS : 65
O1 : 2002.70 Hz
O2 : 2473.50 Hz
SW : 21.9773 ppm
TE : 298.1 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.56
Hz_cm : 223.81
AQ_time : 1.8612220 sec


168.4279
139.1108
126.6098
124.4285
123.1911
122.4375
121.1208
110.7535
51.8497
37.3736
30.6709
29.8777
22.8340
21.5887
14.1087
MeO
(1p)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)
*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 4
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 100.7158630 MHz
CPDPRGT :
NC : -1
NS : 401
O1 : 11078.74 Hz
O2 : 1602.16 Hz
SW : 259.8908 ppm
TE : 298.2 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 7.80
Hz_cm : 785.74
AQ_time : 0.6258690 sec


jfa 846 cdcl3 av400 gjjl_jfa846
7.8238
7.5756
7.5561
7.1044
7.0866
7.0671
7.0452
7.0278
6.9872
3.1194
3.1007
3.0832
3.0657
3.0506
3.0324
2.5195
1.8971
1.8828
1.8724
1.8649
1.8494
1.8411
1.8319
1.8145
1.8085
1.7147
1.6953
1.6917
1.6774
1.6738
1.6583
1.6444
1.6405
1.6270
1.4093
1.3918
1.3811
1.3707
1.3636
1.3394
1.3239
0.9449
0.9278
0.9096
N
H
Me
(1q)
Integral
0.8709
1.0000
2.0000
0.9948
0.9449
3.2524
1.0252
1.2596
7.0753
3.2934
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 2
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 400.1300000 MHz
NC : -2
NS : 25
O1 : 2200.72 Hz
O2 : 2470.97 Hz
SW : 13.0165 ppm
TE : 301.7 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 0.44
Hz_cm : 176.74
AQ_time : 1.5728640 sec


136.0951
126.4937
123.4824
122.3186
120.2455
119.5836
119.2054
117.2342
37.4334
30.9743
30.0141
22.9076
21.5111
16.6085
14.1354
N
H
Me
(1q)
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)
*** Current Data Parameters ***
NAME : JFA
EXPNO : ESPECT~1
PROCNO : 4
*** Acquisition Parameters ***
AQ_mod : qsim
BF1 : 100.6127690 MHz
NC : -1
NS : 179
O1 : 10060.80 Hz
O2 : 1600.52 Hz
SW : 238.3238 ppm
TE : 302.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 7.27
Hz_cm : 731.84
AQ_time : 0.6832130 sec


facturar a gi
jfa 821
h1.10 CDCl3 {c:\bruker\bacs} bruker 8
*** Current Data Parameters ***
NAME : GIMENO~1
EXPNO : ESCRIT~1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 300.1300000 MHz
CPDPRGT :
NC : -2
NS : 64
O1 : 1500.65 Hz
O2 : 1853.43 Hz
SW : 11.9679 ppm
TE : 300.5 K
*** Processing Parameters ***
LB : 0.30 Hz
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
ppm_cm : 0.56
Hz_cm : 169.43
AQ_time : 2.2806530 sec
7.9080 7.5800 7.5515 7.3580 7.3521 7.1067 7.1023 7.0782 7.0738 6.9599 6.9526
3.0701 3.0467 3.0234 3.0007 2.9773 2.9547 1.8371 1.8174 1.8057 1.7940 1.7735 1.7640 1.7509 1.7414 1.7202 1.6844 1.6610 1.6545 1.6304 1.6106 1.5858 1.5690 1.3630 1.3404 1.3206 1.2943 0.9167 0.8948 0.8714
Cl
N
H
(1r)
Integral
0.8405 1.0000 0.9135 0.9903 1.0138 1.0925 1.0718 1.0311 7.2640 3.4665
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
(ppm)


*** Current Data Parameters ***
NAME : Javi
EXPNO : ESPECT~1
PROCNO : 3
*** Acquisition Parameters ***
AQ_mod : dqd
BF1 : 75.4677490 MHz
CPDPRGT :
NC : -1
NS : 400
O1 : 8301.45 Hz
O2 : 2011.55 Hz
SW : 230.0215 ppm
TE : 300.0 K
*** Processing Parameters ***
LB : 3.00 Hz
*** 1D NMR Plot Parameters ***
SR : -0.00 Hz
ppm_cm : 10.85
Hz_cm : 818.92
AQ_time : 0.9437180 sec
136.8320 127.6633 125.5712 123.0789 120.4813 120.2426 119.6740
110.9967
37.3732 30.7459 29.9034 22.8408 21.3876 14.1004
Cl
N
H
(1r)
220 200 180 160 140 120 100 80 60 40 20 0
(ppm)