Supporting Information

Palladium-Catalyzed Direct 2-Alkylation of Indoles by Norbornene-Mediated Regioselective Cascade C-H Activation

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1. General Information

Air and moisture sensitive reactions were carried out in oven-dried glassware sealed with rubber septa under a positive pressure of dry argon. Similarly, sensitive liquids and solutions were transferred via syringe. Reactions were stirred using Teflon-coated magnetic stir bars. Elevated temperatures were maintained using Thermostat-controlled silicone oil baths. Organic solutions were concentrated using a rotary evaporator with a diaphragm vacuum pump. Analytical TLC was performed on Merck silica gel 60 F_{254} plates. The TLC plates were visualized by either ultraviolet light or treatment with a ceric ammonium molybdate (CAM) or potassium permanganate ($KMnO_4$) stain followed by gentle heating. Purification of products was accomplished by flash column chromatography on silica gel (Merck silica gel 60, 230-400 mesh).

NMR spectra were measured on Bruker AV250 (^{1}H at 250 MHz, ^{13}C at 62.9 MHz), Bruker AV360 (^{1}H at 360 MHz, ^{13}C at 90.6 MHz), and Bruker AV500 (^{1}H at 500 MHz, ^{13}C at 125 MHz) nuclear magnetic resonance spectrometers. The ^{1}H-NMR spectra were calibrated against the peak of tetramethylsilane (TMS, 0 ppm) and the ^{13}C-NMR spectra were calibrated against the peak of $CDCl_3$ (77.16 ppm[1]). Data for ^{1}H-NMR spectra were reported as follows: chemical shift (ppm), peak shape (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets), coupling constant (Hz), and integration. Data for ^{13}C-NMR were reported in terms of chemical shift (ppm). Infrared spectra were recorded on a JASCO FT/IR-4100 spectrometer. Mass spectra and high-resolution mass spectra were performed on a Finnigan MAT 8200 or a Thermo Scientific DFS mass spectrometer.

1. Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. *J. Org. Chem.* **1997**, *62*, 7512.

2. Experimental Procedures and Characterization Data

2.1 Source of Chemical Reagents

Solvents. Acetonitrile and DMF were purchased from Acros as anhydrous solvents sealed in AcroSeal bottles. Reagent grade DMA was purchased from Aldrich. "Newly distilled" DMA was distilled from the reagent grade material under reduced pressure (b.p. 50 °C/8 mbar). DMA with indicated concentration of water was made by adding a certain amount of deionized water to the newly distilled DMA and was stored in tightly sealed bottles.

Organic reagents. Indole and substituted indoles **7a-j**, norbornene, butyl bromide, and alkyl bromides **5a-c**, **5g-k**, **5m** were purchased from commercial sources and were used without further purification. Alkyl bromides **5d**,[2] **5e**,[3] and **5f**[4] were synthesized following the published procedures. The synthesis of alkyl bromide **5l** was described in section 2.3.

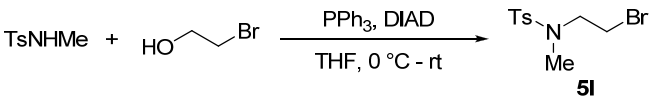
Palladium(II) complexes. $Pd(OAc)_2$ and $PdCl_2(MeCN)_2$ were purchased from Aldrich and $Pd(OCOCF_3)_2$ was purchased from Acros. The palladium(II) complexes were used as received.

Bases. K_2CO_3, $KHCO_3$, and KOAc was purchased from Acros. Cs_2CO_3 was purchased from Alfa-Aesar. K_2HPO_4 (anhydrous) was purchased from Aldrich.

2.2 Screen of Reaction Conditions (Table 1)

Procedure for the screening reactions. An oven dried Schlenk vial equipped with a magnetic stirring bar and a rubber stopper was charged with indole (0.2 mmol), norbornene (0.4 mmol), the base (as indicated, 0.4 mmol), the Pd(II) complex (as indicated, 0.02 mmol), and the solvent (as indicated, 1 mL). The vial was briefly evacuated and backfilled with argon, which was repeated three times. Butyl bromide (0.8 or 0.4 mmol, as indicated) was added via syringe. The reaction vial was placed in a preheated parallel reactor (50 or 70 °C, as indicated) and the reaction was stirred under a balloon pressure of argon for the indicated period of time. After the reaction, the reaction vial was cooled to rt and 1,3,5-trimethoxybenzene (ca. 6 mg, weighted on a precise electronic balance for microanalysis) was transferred into the reaction vial by CH_2Cl_2 as the internal standard. The reaction mixture was diluted with CH_2Cl_2, washed with water (twice) and brine (once), dried over Na_2SO_4, and concentrated. The residue was directly submitted to ^1H NMR, and the conversion and yields of the products were calculated according to the integration of corresponding peaks relative to the characteristic peak of the internal standard (singlet at 6.09 ppm, ArH).

2.3 Synthesis of Alkyl Bromide 5l



5l

To a stirred solution of *N*-methyl tosylamide (926 mg, 5.00 mmol) and triphenylphosphine (2.62 g, 10.0 mmol) in anhydrous THF (40 mL) was added 2-bromoethanol (1.25 g, 10.0 mmol) and then diisopropyl azodicarboxy-

2. Cai, Z. R.; Jabri, S. Y.; Jin, H.; Lansdown, R. A.; Metobo, S. E.; Mish, M. L.; Pastor, R. PCT Int. Appl. WO 2007/136714 A2, 2007.
3. Gant, T. G.; Sarshar, S. Patent US Pat. Appl. US 2010/0069356 A1, 2010.
4. Brawn, R. A.; Welzel, M.; Lowe, J. T.; Panek, J. S. *Org. Lett.* **2010**, *12*, 336.

late (340 mg, 1.99 mmol) dropwise at 0 ºC. The mixture was then allowed to warm to rt and stirred for 6 h. The reaction mixture was concentrated and filtered through a pad of silica gel (eluted with pentane/ether 2:1). The filtrate was concentrated and the crude product was purified by flash column chromatography (eluted with pentane/ether 4:1 to 2:1). The eluent was concentrated and then colorless crystals formed. The crystalline product was washed with pentane/ether 5:1 and collected by filtration to afford alkyl bromide **5l** as a colorless crystalline solid (634 mg, 43% yield).

M.p. 71-72 °C. TLC: R_f = 0.55 (pentane/ether 1:1) [$KMnO_4$].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.44 (s, 3H), 2.83 (s, 3H), 3.38-3.40 (m, 2H), 3.46-3.49 (m, 2H), 7.32-7.34 (m, 2H), 7.68-7.69 (m, 2H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 21.7, 29.1, 36.2, 52.0, 127.5, 130.0, 134.7, 143.9.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2923, 1597, 1492, 1454, 1338, 1156, 1090, 946.

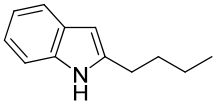
MS (EI, 70 eV): *m*/*z* (%) = 293 (2) [M^+ with ^{81}Br], 291 (2) [M^+ with ^{79}Br], 198 (100) [$(M - CH_2Br)^+$], 155 (56), 91 (64).

HRMS (EI, 70 eV) calcd for $C_9H_{12}NO_2S^+$ [$(M - CH_2Br)^+$]: 198.0583; found: 198.0581.

2.4 General Procedure for the Pd(II)-Catalyzed Norbornene-Mediated 2-Alkylation Reaction of Free *N*-H Indoles and Product Characterization Data (Table 2 and Schemes 3–5)

General procedure for 2-alkylation of free *N*-H indoles. A Schlenk flask equipped with a magnetic stirring bar and a rubber stopper was charged with indole substrate (1 equiv. or 2 equiv., as indicated), norbornene (2 equiv.), the base (2 equiv. K_2CO_3, 3 equiv. $KHCO_3$, or 3 equiv. K_2HPO_4, as indicated), and $PdCl_2(MeCN)_2$ (10 mol %). A solution of water in DMA (0.5 M) was added via syringe as the solvent to prepare a 0.2 M solution of the substrate. Then either of the following two procedures could be used: 1. the resulting solution was briefly evacuated and then backfilled with argon (3 times), and then the alkyl bromide (2 equiv. or 1 equiv., as indicated) was added via syringe; 2. the alkyl bromide (2 equiv. or 1 equiv., as indicated) was added via syringe and the resulting solution was degassed by three freeze-pump-thaw cycles using liquid nitrogen under high vacuum. The reaction mixture was then placed in a preheated oil bath at 70 °C (or 90 °C, as indicated). Vigorous stirring was applied and the mixture was reacted under a balloon pressure of argon. The reaction was monitored by TLC. Upon completion, the reaction mixture was cooled to room temperature, diluted with ether, and filtered. The filtrate was concentrated in a water bath (60 °C, 8-10 mbar) to remove ether and most of DMA. The residue was directly submitted to flash column chromatography (by dry loading) to afford the 2-alkylindole product.

2-Butyl-1*H*-indole (1)



Synthesized according to the general procedure using indole (119 mg, 1.02 mmol, 1 equiv.) and butyl bromide (310 mg, 2.27 mmol, 2.23 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column

chromatography (eluted with pentane/ether 40:1 to 30:1) to afford 2-alkylindole **1** as a colorless oil (119 mg, 67% yield) and 2,3-disubstituted indole **2** as a colorless oil (25.4 mg, 11% yield).

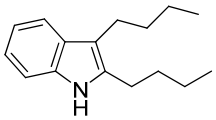
R_f = 0.52 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.94 (t, J = 7.4 Hz, 3H), 1.39 (app. sextet, J = 7.4 Hz, 2H), 1.66 (app. quintet, J = 7.6 Hz, 2H), 2.68 (t, J = 7.7 Hz, 2H), 6.21-6.22 (m, 1H), 7.05 (app. dt, J = 1.4, 7.4 Hz, 1H), 7.10 (app. dt, J = 1.3, 7.4 Hz, 1H), 7.22 (d, J = 7.7 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.68 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.0, 22.5, 28.0, 31.4, 99.5, 110.4, 119.7, 119.8, 121.0, 129.0, 135.9, 140.1.

The ^{1}H and ^{13}C NMR spectra are consistent with those reported in the literature.[5]

2,3-Dibutyl-1*H*-indole (2)



Pale-yellow oil. TLC: R_f = 0.63 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.93 (t, J = 7.3 Hz, 3H), 0.94 (t, J = 7.3 Hz, 3H), 1.381 (app. sextet, J = 7.4 Hz, 2H), 1.384 (app. sextet, J = 7.4 Hz, 2H), 1.61 (app. sextet, J = 7.7 Hz, 4H), 2.68 (t, J = 7.7 Hz, 2H), 2.70 (t, J = 7.7 Hz, 2H), 7.05 (app. dt, J = 1.3, 7.4 Hz, 1H), 7.09 (app. dt, J = 1.4, 7.5 Hz, 1H), 7.23 (d, J = 7.8 Hz, 1H), 7.51 (d, J = 7.7 Hz, 1H), 7.62 (br s, 1H).

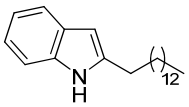
^{13}C NMR (63 MHz, $CDCl_3$): δ (ppm) = 14.1, 14.2, 22.7, 22.9, 24.1, 26.0, 32.2, 33.4, 110.3, 112.3, 118.5, 119.0, 120.9, 129.0, 135.3, 135.4.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3406, 2955, 2929, 2870, 1634, 1526, 1460, 1378, 1338, 1199.

MS (EI, 70 eV): m/z (%) = 229 (32) [M^+], 186 (100) [$(M - C_3H_7)^+$], 144 (52).

HRMS (EI, 70 eV) calcd for $C_{16}H_{23}N^+$ [M^+]: 229.1825; found: 229.1824.

2-Tetradecyl-1*H*-indole (6a)



Synthesized according to the general procedure using indole (118 mg, 1.01 mmol, 1 equiv.) and 1-bromotetradecane (551 mg, 1.99 mmol, 1.97 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 40:1 to 30:1) to afford 2-alkylindole **6a** as a white solid (213 mg, 67% yield) and 2,3-disubstituted indole **6a'** as a pale-yellow oil (47.1 mg, 9% yield).

5. Ambrogio, I.; Cacchi, S.; Fabrizi, G.; Prastaro, A. *Tetrahedron* **2009**, *65*, 8916.

M.p. 58-60 °C. TLC: R_f = 0.64 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.88 (t, J = 7.0 Hz, 3H), 1.23-1.34 (m, 20H), 1.35-1.42 (m, 2H), 1.71 (app. quintet, J = 7.5 Hz, 2H), 2.74 (t, J = 7.6 Hz, 2H), 6.21 (br s, 1H), 7.06 (app. dt, J = 0.9, 7.5 Hz, 1H), 7.10 (app. dt, J = 0.9, 7.5 Hz, 1H), 7.29 (d, J = 7.9 Hz, 1H), 7.52 (d, J = 7.2 Hz, 1H), 7.83 (br s, 1H).

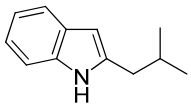
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.3, 22.8, 28.5, 29.3, 29.49, 29.52, 29.6, 29.7, 29.81, 29.85, 32.1, 99.6, 110.4, 119.7, 119.9, 121.1, 129.0, 136.0, 140.2.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3413, 2916, 2847, 1616, 1584, 1551, 1457, 1408, 1290, 1231, 1011.

MS (EI, 70 eV): m/z (%) = 313 (45) [M^+], 144 (45) [$(M - C_{12}H_{25})^+$], 130 (100) [$(M - C_{13}H_{27})^+$].

HRMS (EI, 70 eV) calcd for $C_{22}H_{35}N^+$ [M^+]: 313.2764; found: 313.2759.

2-Isobutyl-1*H*-indole (6b)



Synthesized according to the general procedure using indole (118 mg, 1.00 mmol, 1 equiv.) and isobutyl bromide (277 mg, 2.02 mmol, 2.02 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 90 °C; reaction time: 61 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 40:1 to 30:1) to afford 2-alkylindole **6b** as a pale-yellow oil (103 mg, 59% yield) and 2,3-disubstituted indole **6b'** as a colorless oil (9.9 mg, 4% yield).

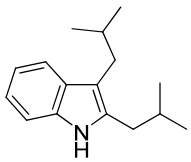
TLC: R_f = 0.89 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.97 (d, J = 6.6 Hz, 6H), 1.96 (app. nonet, J = 6.8 Hz, 1H), 2.60 (d, J = 7.2 Hz, 2H), 6.22 (s, 1H), 7.06 (app. dt, J = 1.2, 7.4 Hz, 1H), 7.10 (app. dt, J = 1.2, 7.5 Hz, 1H), 7.27 (d, J = 7.8 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 7.77 (br s, 1H).

^{13}C NMR (62.9 MHz, $CDCl_3$): δ (ppm) = 22.7, 29.1, 37.9, 100.6, 110.4, 119.7, 119.9, 121.0, 129.1, 136.0, 139.0.

The ^{1}H NMR spectrum is consistent with that reported in the literature.[6]

2,3-Diisobutyl-1*H*-indole (6b')



TLC: R_f = 0.63 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.93 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H), 1.96 (app. nonet, J = 6.8 Hz, 1H), 1.99 (app. nonet, J = 6.8 Hz, 1H), 2.55 (d, J = 7.3 Hz, 2H), 2.59 (d, J =7.4 Hz, 2H), 7.05 (app. dt, J

6. Zhao, D.; Hughes, D. L.; Bender, D. R.; DeMarco, A. M.; Reider, P. J. *J. Org. Chem.* **1991**, *56*, 3001

= 1.2, 7.4 Hz, 1H), 7.10 (app. dt, *J* = 1.3, 7.4 Hz, 1H), 7.26 (d, *J* = 7.9 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.68 (br s, 1H).

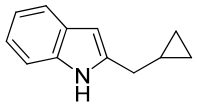
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 22.8, 23.0, 29.4, 29.9, 33.8, 35.7, 110.2, 112.3, 118.8, 118.9, 120.9, 129.3, 135.1, 135.4.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3412, 2952, 2866, 1586, 1461, 1304, 1167, 1011.

MS (EI, 70 eV): *m*/*z* (%) = 229 (22) [M^+], 186 (100) [$(M - C_3H_7)^+$], 144 (21).

HRMS (EI, 70 eV) calcd for $C_{16}H_{23}N^+$ [M^+]: 229.1825; found: 229.1823.

2-Cyclopropylmethyl-1*H*-indole (6c)



Synthesized according to the general procedure using indole (118 mg, 1.00 mmol, 1 equiv.) and cyclopropylmethyl bromide (280 mg, 2.07 mmol, 2.07 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 40:1 to 30:1) to afford 2-alkylindole **6c** as a pale-yellow oil (102 mg, 59% yield) and 2,3-disubstituted indole **6c'** as a colorless oil (24 mg, 11% yield).

TLC: R_f = 0.42 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.22-0.25 (m, 2H), 0.56-0.60 (m, 2H), 0.99-1.07 (m, 1H), 2.62 (d, *J* = 6.9 Hz, 2H), 6.28 (s, 1H), 7.06 (app. t, *J* = 7.3 Hz, 1H), 7.10 (app. t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 7.9 Hz, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.85 (br s, 1H).

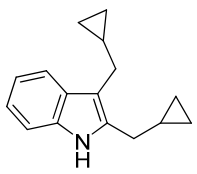
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 4.7, 10.2, 33.1, 99.7, 110.5, 119.7, 119.9, 121.1, 128.9, 136.0, 139.5.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3392, 3073, 3001, 2890, 1683, 1616, 1548, 1486, 1457, 1413, 1293, 1010.

MS (EI, 70 eV): *m*/*z* (%) = 171 (66) [M^+], 143 (25) [$(M - C_2H_4)^+$], 130 (100) [$(M - C_3H_5)^+$], 117 (12), 103 (7).

HRMS (EI, 70 eV) calcd for $C_{12}H_{13}N^+$ [M^+]: 171.1043; found: 171.1043.

2,3-Bis(cyclopropylmethyl)-1*H*-indole (6c')



TLC: R_f = 0.47 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.17-0.20 (m, 2H), 0.26-0.29 (m, 2H), 0.42-0.45 (m, 2H), 0.59-0.62 (m, 2H), 0.96-1.05 (m, 2H), 2.66 (d, *J* = 6.4 Hz, 2H), 2.68 (d, *J* = 6.9 Hz, 2H), 7.07 (app. dt, *J* = 1.1, 7.4 Hz, 1H), 7.11 (app. dt, *J* = 1.1, 7.5 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 7.57 (d, *J* = 7.7 Hz, 1H), 7.96 (br s, 1H).

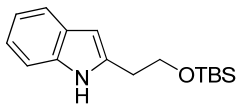
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 4.8, 5.0, 10.5, 12.2, 28.7, 30.9, 110.4, 111.3, 118.8, 119.2, 121.1, 129.1, 135.0, 135.4.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3407, 3074, 3001, 2911, 1619, 1460, 1338, 1291, 1013.

MS (EI, 70 eV): *m*/*z* (%) = 225 (86) [M^+], 184 (100) [$(M - C_3H_5)^+$], 170 (91) [$(M - C_4H_7)^+$], 143 (42), 130 (44).

HRMS (EI, 70 eV) calcd for $C_{16}H_{19}N^+$ [M^+]: 225.1512; found: 225.1513.

2-(2-*tert*-Butyldimethylsilyloxyethyl)-1*H*-indole (6d)



Synthesized according to the general procedure using indole (116 mg, 0.99 mmol, 1 equiv.) and bromide **5d** (484 mg, 2.03 mmol, 2.05 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 40:1) to afford 2-alkylindole **6d** as a pale-yellow solid (225 mg, 82% yield). Another run using bromide **5d** (231 mg, 0.97 mmol, 1 equiv.) and indole (234 mg, 2.00 mmol, 2.07 equiv.) under identical conditions produced 2-alkylindole **6d** (189 mg) in 71% yield.

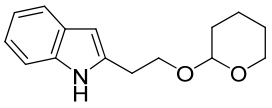
M.p. 64-65 °C. TLC: R_f = 0.57 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.12 (s, 6H), 1.00 (s, 9H), 3.00 (t, *J* = 5.8 Hz, 2H), 3.96 (t, *J* = 5.8 Hz, 2H), 6.26 (s, 1H), 7.10 (app. dt, *J* = 0.9, 7.5 Hz, 1H), 7.15 (app. dt, *J* = 0.9, 7.5 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 8.66 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = –5.3, 18.4, 26.1, 31.3, 63.2, 99.8, 110.5, 119.6, 119.9, 121.1, 128.5, 136.1, 138.4.

The ^{1}H NMR spectrum is consistent with that reported in the literature.[7]

2-(2-(Tetrahydro-2*H*-pyran-2-yloxy)ethyl)-1*H*-indole (6e)



Synthesized according to the general procedure using indole (117 mg, 1.00 mmol, 1 equiv.) and bromide **5e** (404 mg, 1.93 mmol, 1.93 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 5:1 to 3:1) to afford 2-alkylindole **6e** as a colorless oil (179 mg, 73% yield). Another run using bromide **5e** (40.5 mg, 0.194 mmol, 1 equiv.) and indole (48.1 mg, 0.410 mmol, 2.16 equiv.) under identical conditions, except that 10 mol % of $Pd(OCOCF_3)_2$ was used as the catalyst, produced 2-alkylindole **6e** (36.2 mg) in 76% yield.

7. Bennani, Y. L.; Campbell, M. G.; Dastrup, D.; Porter Huck, E. Patent PCT Int. Appl. WO 2007/047775 A1, 2007.

TLC: R_f = 0.49 (pentane/ether 3:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.52-1.69 (m, 4H), 1.75-1.80 (m, 1H), 1.84-1.90 (m, 1H), 3.04, (t, J = 6.0 Hz, 2H), 3.48-3.53 (m, 1H), 3.71 (dt, J = 9.6, 6.0 Hz, 1H), 3.83 (ddd, J = 3.0, 7.7, 11.0 Hz, 1H), 4.07 (dt, J = 9.6, 6.0 Hz, 1H), 4.64 (dd, J = 3.0, 4.6 Hz, 1H), 6.25 (s, 1H), 7.04-7.07 (m, 1H), 7.09-7.13 (m, 1H), 7.29 (d, J = 8.0 Hz, 1H), 7.53 (d, J = 7.7 Hz, 1H), 8.54 (br s, 1H).

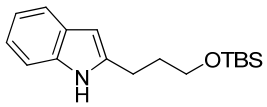
^{13}C NMR (63 MHz, $CDCl_3$): δ (ppm) = 20.0, 25.5, 28.8, 31.0, 62.8, 67.4, 99.5, 100.1, 110.6, 119.6, 120.0, 121.1, 128.6, 136.2, 137.9.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3333, 2938, 2867, 1685, 1617, 1457, 1323, 1200, 1119, 1072, 1022.

MS (EI, 70 eV): m/z (%) = 245 (31) [M^+], 161 (82) [$(M - C_5H_8O)^+$], 144 (31) [$(M - C_5H_9O_2)^+$], 130 (88), 86 (93), 85 (100).

HRMS (EI, 70 eV) calcd for $C_{15}H_{19}NO_2^+$ [M^+]: 245.1410; found: 245.1407.

2-(3-*tert*-Butyldimethylsilyloxypropyl)-1*H*-indole (6f)



Synthesized according to the general procedure using indole (48.6 mg, 0.415 mmol, 1 equiv.) and bromide **5f** (208 mg, 0.821 mmol, 2.00 equiv.), except that $Pd(OCOCF_3)_2$ (10 mol %) was used as the catalyst. Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 30:1 to 20:1) to afford 2-alkylindole **6f** as a pale-yellow solid (43.0 mg, 36% yield). Another run using bromide **5f** (111 mg, 0.438 mmol, 1 equiv.) and indole (96.3 mg, 0.822 mmol, 1.86 equiv.) under identical conditions produced 2-alkylindole **6f** (50.6 mg) in 40% yield.

M.p. 63-64°C. TLC: R_f = 0.36 (pentane/ether 9:1) [CAM].

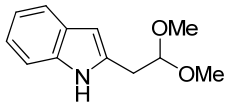
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.10 (s, 6H), 0.94 (s, 9H), 1.92 (app. quintet, J = 6.6 Hz, 2H), 2.87 (t, J = 7.1 Hz, 2H), 3.72 (t, J = 5.9 Hz, 2H), 6.22 (s, 1H), 7.06 (app. dt, J = 1.0, 7.5 Hz, 1H), 7.10 (app. dt, J = 1.1, 7.7 Hz, 1H), 7.28 (d, J = 7.9 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 8.37 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = –5.1, 18.5, 25.1, 26.2, 32.0, 62.7, 99.6, 110.4, 119.6, 119.9, 121.0, 129.0, 136.1, 139.7.

The ^{1}H NMR spectrum is consistent with that reported in the literature.[8]

8. Wender, P. A.; Cooper, C. B. *Tetrahedron* **1986**, *42*, 2985.

2-(2,2-Dimethoxyethyl)-1*H*-indole (6g)



Synthesized according to the general procedure using indole (117 mg, 1.00 mmol, 1 equiv.) and bromide **5g** (351 mg, 2.07 mmol, 2.07 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 90 °C; reaction time: 20 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 5:1 to 3:1) to afford 2-alkylindole **6g** as a pale-brown oil (133 mg, 65% yield).

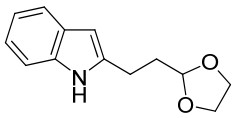
TLC: R_f = 0.36 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 3.06 (d, J = 5.3 Hz, 2H), 3.41 (s, 6H), 4.56 (t, J = 5.3 Hz, 1H), 6.28 (s, 1H), 7.05 (app. dt, J = 1.0, 7.5 Hz, 1H), 7.12 (app. dt, J = 1.1, 7.5 Hz, 1H), 7.29 (d, J = 8.1 Hz, 1H), 7.53 (d, J = 7.9 Hz, 1H), 8.44 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 32.4, 53.9, 101.3, 104.5, 110.7, 119.6, 120.0, 121.3, 128.5, 134.5, 136.3.

The ^{1}H NMR spectrum is consistent with that reported in the literature.[9]

2-[2-(1,3-Dioxylan-2-yl)ethyl]-1*H*-indole (6h)



Synthesized according to the general procedure using indole (116 mg, 0.99 mmol, 1 equiv.) and bromide **5h** (359 mg, 1.99 mmol, 2.01 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 3:1 to 2:1) to afford 2-alkylindole **6h** as a pale-yellow solid (155 mg, 72% yield). Another run using bromide **5h** (178 mg, 0.98 mmol, 1 equiv.) and indole (233 mg, 1.98 mmol, 2.02 equiv.) under identical conditions produced 2-alkylindole **6h** (115 mg) in 54% yield.

M.p. 107-108 °C. TLC: R_f = 0.15 (pentane/ether 3:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.10 (dt, J = 4.5, 7.3 Hz, 2H), 2.91 (t, J = 7.3 Hz, 2H), 3.88-3.94 (m, 2H), 4.00-4.06 (m, 2H), 4.98 (t, J = 4.5 Hz, 1H), 6.24 (s, 1H), 7.06 (app. t, J = 7.3 Hz, 1H), 7.11 (app. t, J = 7.2 Hz, 1H), 7.29 (d, J = 7.9 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 8.29 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 22.3, 33.0, 65.2, 99.8, 103.8, 110.5, 119.7, 119.9, 121.1, 128.9, 136.1, 139.1.

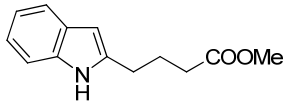
IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3315, 2923, 2853, 1584, 1551, 1457, 1413, 1287, 1124, 1072, 1024.

MS (EI, 70 eV): m/z (%) = 217 (36) [M^+], 144 (20) [$(M - C_3H_5O_2)^+$], 130 (100) [$(M - C_4H_7O_2)^+$], 117 (11), 73 (15) [$C_3H_5O_2^+$].

9. Kraus, G. A.; Frazier, K. *Tetrahedron Lett.* **1978**, *35*, 3195.

HRMS (EI, 70 eV) calcd for $C_{13}H_{15}NO_2^+$ [M^+]: 217.1097; found: 217.1094.

Methyl 4-(1*H*-indol-2-yl)butanoate (6i)



Synthesized according to the general procedure using indole (118 mg, 1.01 mmol, 1 equiv.) and bromide **5i** (355 mg, 1.96 mmol, 1.94 equiv.). Base: 2 equiv. K_2CO_3; degassing method: evacuation/backfill with argon; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 4:1 to 2:1) to afford 2-alkylindole **6i** as a white solid (134 mg, 61% yield) and 2,3-dialkyl indole **6i'** as a yellow oil (37.7 mg, 12% yield). Another run using bromide **5i** (191 mg, 1.06 mmol, 1 equiv.) and indole (233 mg, 1.99 mmol, 1.88 equiv.) under identical conditions produced 2-alkylindole **6i** (120 mg) in 52% yield, together with intramolecular cyclization byproduct **6i"** as a yellow oil (20.5 mg, 10% yield).

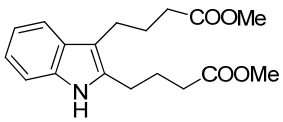
M.p. 60-62 °C (lit. m.p. 69-71 °C[10]). TLC: R_f = 0.33 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.09 (app. quintet, J = 7.4 Hz, 2H), 2.45 (t, J = 7.3 Hz, 2H), 2.83 (t, J = 7.4 Hz, 2H), 3.74 (s, 3H), 6.30-6.31 (m, 1H), 7.14 (app. dt, J = 1.1, 7.4 Hz, 1H), 7.18 (app. dt, J = 1.2, 7.5 Hz, 1H), 7.33 (d, J = 7.8 Hz, 1H), 7.60 (d, J = 7.7 Hz, 1H), 8.11 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 24.6, 27.5, 33.3, 51.7, 100.0, 110.5, 119.7, 119.9, 121.2, 128.8, 136.1, 138.5, 174.1.

The ^{1}H and ^{13}C NMR spectra are consistent with those reported in the literature.[10]

Methyl 4-(1*H*-indol-2-yl)butanoate (6i')



TLC: R_f = 0.16 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.93-2.01 (m, 4H), 2.35 (t, J = 7.5 Hz, 2H), 2.36 (t, J = 7.3 Hz, 2H), 2.73 (t, J = 7.5 Hz, 2H), 2.77 (t, J = 7.5 Hz, 2H), 3.65 (s, 3H), 3.68 (s, 3H), 7.07 (app. dt, J = 1.1, 7.4 Hz, 1H), 7.12 (app. dt, J = 1.2, 7.5 Hz, 1H), 7.28 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 8.00 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 23.5, 25.1, 25.2, 26.0, 33.2, 33.7, 51.5, 51.8, 110.5, 111.4, 118.4, 119.2, 121.3, 128.5, 134.4, 135.5, 174.1, 174.3.

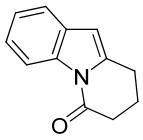
IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3382, 2951, 2850, 1716, 1620, 1460, 1436, 1199, 1156, 1011.

MS (EI, 70 eV): m/z (%) = 317 (20) [M^+], 285 (17), 230 (37), 198 (18), 170 (39), 101 (46), 84 (100).

HRMS (EI, 70 eV) calcd for $C_{18}H_{23}NO_4^+$ [M^+]: 317.1622; found: 317.1619.

10. Bunce, R. A.; Nammalwar, B. *J. Heterocyclic Chem.* **2009,** *46*, 172.

8,9-Dihydropyrido[1,2-*a*]indol-6(7*H*)-one (6i")



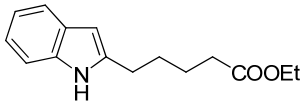
TLC: R_f = 0.50 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.09 (app. quintet, J = 6.4 Hz, 2H), 2.79 (t, J = 6.4 Hz, 2H), 2.99 (dt, J = 1.2, 6.3 Hz, 2H), 6.32-6.33 (m, 1H), 7.24 (app. dt, J = 1.2, 7.4 Hz, 1H), 7.28 (app. dt, J = 1.4, 7.7 Hz, 1H), 7.46 (d, J = 7.5 Hz, 1H), 8.44 (d, J = 8.0 Hz, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 21.6, 23.9, 34.6, 105.0, 116.5, 119.8, 124.0, 124.2, 129.9, 135.0, 138.2, 169.5.

The ^{1}H and ^{13}C NMR spectra are consistent with those reported in the literature.[10]

Ethyl 5-(1*H*-indol-2-yl)pentanoate (6j)



Synthesized according to the general procedure using indole (118 mg, 1.01 mmol, 1 equiv.) and bromide **5j** (404 mg, 1.93 mmol, 1.91 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 3:1 to 2:1) to afford 2-alkylindole **6j** as a pale-yellow solid (160 mg, 66% yield) and 2,3-dialkyl indole **6j'** as a yellow oil (39.9 mg, 11% yield). Another run using bromide **5j** (202 mg, 0.97 mmol, 1 equiv.) and indole (236 mg, 2.02 mmol, 2.08 equiv.) under identical conditions produced 2-alkylindole **6j** (163 mg) in 69% yield and 2,3-dialkyl indole **6j'** (22.4 mg) in 12% yield.

M.p. 87-88 °C (lit. m.p. 93-95 °C[11]). TLC: R_f = 0.45 (pentane/ether 2:1) [CAM].

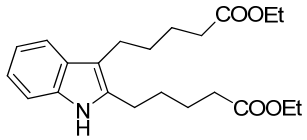
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.25 (t, J = 7.1 Hz, 3H), 1.69-1.80 (m, 4H), 2.35 (t, J = 7.0 Hz, 2H), 2.77 (t, J = 7.2 Hz, 2H), 4.13 (q, J = 7.1 Hz, 2H), 6.22 (s, 1H), 7.06 (app. dt, J = 1.1, 7.4 Hz, 1H), 7.11 (app. dt, J = 1.2, 7.5 Hz, 1H), 7.29 (d, J = 7.9 Hz, 1H), 7.52 (d, J = 7.8 Hz, 1H), 8.02 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.4, 24.6, 28.0, 28.7, 34.1, 60.5, 99.8, 110.5, 119.7, 119.9, 121.1, 129.0, 136.0, 139.4, 173.8.

The ^{1}H NMR spectrum is consistent with that reported in the literature.[11]

11. Bailey, A. S.; Seager, J. F. *J. Chem. Soc., Perkin Trans. 1* **1974**, *7*, 763.

Ethyl 5-(1*H*-indol-2-yl)pentanoate (6j')



TLC: R_f = 0.33 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.23 (t, J = 7.2 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H), 1.62-1.72 (m, 8H), 2.30-2.35 (m, 4H), 2.68-2.75 (m, 4H), 4.11 (q, J = 7.2 Hz, 2H), 4.13 (q, J = 7.2 Hz, 2H), 7.05 (app. dt, J = 1.0, 7.4 Hz, 1H), 7.10 (app. dt, J = 1.1, 7.4 Hz, 1H), 7.26 (d, J = 7.8 Hz, 1H), 7.49 (d, J = 7.7 Hz, 1H), 7.93 (br s, 1H).

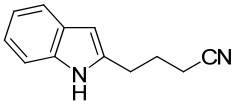
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.4, 24.0, 24.6, 25.2, 25.8, 29.3, 30.6, 34.1, 34.5, 60.3, 60.5, 110.5, 111.8, 118.4, 119.1, 121.0, 128.7, 134.8, 135.5, 173.8, 173.9.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3380, 2935, 2863, 1730, 1714, 1461, 1372, 1179, 1026.

MS (EI, 70 eV): m/z (%) = 373 (52) [M^+], 328 (17) [$(M - C_2H_5O)^+$], 258 (100) [$(M - CH_2CH_2CH_2COOEt)^+$], 184 (35), 144 (54), 130 (82).

HRMS (EI, 70 eV) calcd for $C_{22}H_{31}NO_4^+$ [M^+]: 373.2248; found: 373.2245.

4-(1*H*-Indol-2-yl)butyronitrile (6k)



Synthesized according to the general procedure using indole (115 mg, 0.98 mmol, 1 equiv.) and bromide **5k** (291 mg, 1.97 mmol, 2.01 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 2:1 to 1:2) to afford 2-alkylindole **6k** as a pale-red oil (101 mg, 56% yield) and 2,3-dialkyl indole **6k'** as a pale-red oil (46.8 mg, 19% yield). Another run using bromide **5k** (144 mg, 0.98 mmol, 1 equiv.) and indole (233 mg, 1.99 mmol, 2.03 equiv.) under identical conditions produced 2-alkylindole **6k** (114 mg) in 64% yield.

TLC: R_f = 0.26 (pentane/ether 1:1) [CAM].

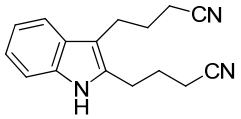
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.97 (app. quintet, J = 7.2 Hz, 2H), 2.30 (t, J = 7.3 Hz, 2H), 2.81 (t, J = 7.3 Hz, 2H), 6.23 (s, 1H), 7.08 (app. dt, J = 1.0, 7.2 Hz, 1H), 7.13 (app. dt, J = 1.1, 7.8 Hz, 1H), 7.27 (d, J = 8.0 Hz, 1H), 7.52 (d, J = 7.8 Hz, 1H), 7.93 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 16.4, 25.0, 26.9, 100.3, 110.7, 119.5, 119.9, 120.0, 121.5, 128.7, 136.1, 136.8.

The ^{1}H and ^{13}C NMR spectra are consistent with those reported in the literature.[12]

12. Miyazaki, Y.; Kobayashi, S. *J. Comb. Chem.* **2008**, *10*, 355.

2,3-Bis(3-cyanopropyl)-1*H*-indole (6k')



TLC: R_f = 0.22 (pentane/ether 1:2) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.97-2.03 (m, 4H), 2.30 (t, J = 7.0 Hz, 2H), 2.35 (t, J = 7.0 Hz, 2H), 2.88 (t, J =7.3 Hz, 2H), 2.91 (t, J = 7.5 Hz, 2H), 7.10 (app. dt, J = 1.0, 7.5 Hz, 1H), 7.16 (app. dt, J = 1.3, 7.5 Hz, 1H), 7.30 (d, J = 8.0 Hz, 1H), 7.50 (d, J = 7.8 Hz, 1H), 8.06 (br s, 1H).

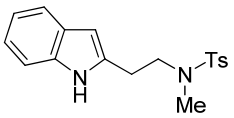
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 16.6, 22.8, 24.8, 25.7, 26.2, 110.4, 110.8, 118.3, 119.5, 119.7, 120.0, 121.9, 128.1, 133.2, 135.7.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3362, 2935, 2866, 2361, 2246, 1732, 1620, 1565, 1490, 1460, 1335, 1309, 1240.

MS (EI, 70 eV): m/z (%) = 251 (26) [M^+], 197 (92) [$(M - C_3H_4N)^+$], 149 (26), 84 (100).

HRMS (EI, 70 eV) calcd for $C_{16}H_{17}N_3^+$ [M^+]: 251.1417; found: 251.1411.

***N*-(2-(1*H*-Indol-2-yl)ethyl)-*N*-methyl-4-toluenesulfonamide (6l)**



Synthesized according to the general procedure using indole (55.0 mg, 0.469 mmol, 1 equiv.) and bromide **5l** (257 mg, 0.880 mmol, 1.87 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 90 °C; reaction time: 68 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 5:1 to 1:1) to afford 2-alkylindole **6l** as an off-white solid (65.7 mg, 43% yield) and recovered indole (27.4 mg, 50%). The yield of **6l** based on recovered starting material was 85%.

M.p. 98-99 °C. TLC: R_f = 0.26 (pentane/ether 1:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.41 (s, 3H), 2.72 (s, 3H), 3.04 (t, J = 7.1 Hz, 2H), 3.33 (t, J = 7.1 Hz, 2H), 6.24 (s, 1H), 7.06 (app. t, J = 7.5 Hz, 1H), 7.13 (app. t, J = 7.6 Hz, 1H), 7.27-7.29 (m, 2H), 7.32 (d, J = 8.1 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.64-7.66 (m, 2H), 8.30 (br s, 1H).

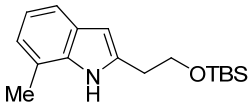
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 21.6, 27.5, 35.5, 50.2, 100.5, 110.9, 119.7, 119.9, 121.4, 127.5, 128.6, 129.9, 134.2, 135.8, 136.2, 143.7.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3392, 2977, 2921, 1597, 1547, 1457, 1418, 1339, 1293, 1158, 1119, 1084.

MS (EI, 70 eV): m/z (%) = 328 (39) [M^+], 198 (100), 155 (84), 149 (23), 130 (56), 91 (73).

HRMS (EI, 70 eV) calcd for $C_{18}H_{20}N_2O_2S^+$ [M^+]: 328.1240; found: 328.1233.

7-Methyl-2-(2-*tert*-butyldimethylsilyloxyethyl)-1*H*-indole (8a)



Synthesized according to the general procedure using indole **7a** (131 mg, 1.00 mmol, 1 equiv.) and bromide **5d** (474 mg, 1.98 mmol, 1.98 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 18 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 40:1 to 30:1) to afford 2-alkylindole **8a** as a pale-yellow oil (196 mg, 68% yield).

TLC: R_f = 0.65 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.10 (s, 6H), 0.97 (s, 9H), 2.45 (s, 3H), 2.98 (t, J = 5.6 Hz, 2H), 3.94 (t, J = 5.6 Hz, 2H), 6.22 (s, 1H), 6.91 (d, J = 7.2 Hz, 1H), 6.98 (app. t, J = 7.5 Hz, 1H), 7.38 (d, J = 7.8 Hz, 1H), 8.70 (br s, 1H).

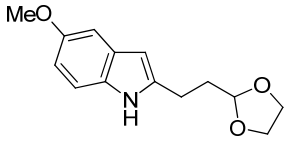
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = –5.3, 16.9, 18.3, 26.1, 31.2, 63.3, 100.3, 117.7, 119.68, 119.70, 121.7, 127.9, 135.7, 138.3.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3437, 2954, 2927, 2856, 1614, 1559, 1496, 1461, 1329, 1254, 1086, 1054.

MS (EI, 70 eV): *m*/*z* (%) = 289 (21) [M^+], 232 (100) [$(M - C_4H_9)^+$], 158 (26) [$(M - TBSO)^+$], 109 (36).

HRMS (EI, 70 eV) calcd for $C_{17}H_{27}NOSi^+$ [M^+]: 289.1856; found: 289.1856.

2-(2-(1,3-Dioxolan-2-yl)ethyl)-5-methoxy-1*H*-indole (8b)



Synthesized according to the general procedure using indole **7b** (143 mg, 0.97 mmol, 1 equiv.) and bromide **5h** (353 mg, 1.95 mmol, 2.01 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 3:1 to 1:1) to afford 2-alkylindole **8b** as a colorless oil (150 mg, 62% yield) and recovered indole **7b** as a pale-yellow oil (32.5 mg, 23% recovered). The yield of product **8b** based on recovered starting material was 78%.

TLC: R_f = 0.15 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.08 (dt, J = 4.4, 7.4 Hz, 2H), 2.87 (t, J = 7.4 Hz, 2H), 3.83 (s, 3H), 3.88-3.91 (m, 2H), 4.00-4.04 (m, 2H), 4.96 (t, J = 4.4 Hz, 1H), 6.16 (br s, 1H), 6.77 (dd, J = 2.4, 8.7 Hz, 1H), 7.00 (d, J = 2.4 Hz, 1H), 7.16 (d, J = 8.7 Hz, 1H), 8.19 (br s, 1H).

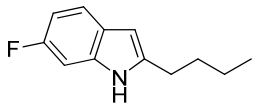
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 22.4, 33.0, 56.0, 65.1, 99.5, 102.1, 103.7, 110.9, 111.2, 129.2, 131.2, 139.9, 154.1.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3389, 2919, 2885, 2847, 1621, 1586, 1482, 1451, 1407, 1222, 1131, 1024.

MS (EI, 70 eV): *m*/*z* (%) = 247 (3) [M^+], 220 (2), 205 (9), 161 (9), 121 (10), 84 (100), 73 (27).

HRMS (EI, 70 eV) calcd for $C_{14}H_{17}NO_3^+$ [M^+]: 247.1203; found: 247.1196.

2-Butyl-6-fluoro-1*H*-indole (8c)



Synthesized according to the general procedure using indole **7c** (134 mg, 0.99 mmol, 1 equiv.) and butyl bromide (280 mg, 2.05 mmol, 2.07 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 20 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 30:1 to 20:1) to afford 2-alkylindole **8c** as a pale-yellow oil (111 mg, 59% yield) and 2,3-dialkylindole **8c'** as a pale-yellow oil (34.0 mg, 14% yield).

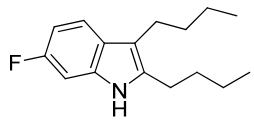
TLC: R_f = 0.50 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.94 (t, *J* = 7.3 Hz, 3H), 1.40 (app. sextet, *J* = 7.4 Hz, 2H), 1.68 (app. quintet, *J* = 7.7 Hz, 2H), 2.71 (t, *J* = 7.6 Hz, 2H), 6.18-6.19 (m, 1H), 6.82 (ddd, *J* = 2.3, 8.6, 9.8 Hz, 1H), 6.95 (dd, *J* = 2.0, 9.7 Hz, 1H), 7.40 (dd, *J* = 5.4, 8.6 Hz, 1H), 7.78 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): 14.0, 22.5, 28.0, 31.3, 96.9 (d, *J* = 26.1 Hz), 99.4, 108.1 (d, *J* = 24.2 Hz), 120.3 (d, *J* = 10.0 Hz), 125.4, 135.8 (d, *J* = 12.2 Hz), 140.5 (d, *J* = 3.7 Hz), 159.4 (d, *J* = 236.0 Hz).

This is a known compound, but no spectroscopic data were given.[13]

2,3-Dibutyl-6-fluoro-1*H*-indole (8c')



TLC: R_f = 0.59 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.93 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 3H), 1.33-1.43 (m, 4H), 1.54-1.65 (m, 4H), 2.65 (t, *J* = 7.6 Hz, 2H), 2.69 (t, *J* = 7.6 Hz, 2H), 6.82 (ddd, *J* = 2.3, 8.7, 9.7 Hz, 1H), 6.94 (dd, *J* = 2.3, 9.7 Hz, 1H), 7.39 (dd, *J* = 5.4, 8.7 Hz, 1H), 7.65 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): 14.0, 14.2, 22.7, 22.9, 24.0, 26.0, 32.2, 33.4, 96.8 (d, *J* = 26.0 Hz), 107.4 (d, *J* = 24.1 Hz), 112.3, 119.0 (d, *J* = 10.0 Hz), 125.5, 135.2 (d, *J* = 12.3 Hz), 135.5 (d, *J* = 3.7 Hz), 159.5 (d, *J* = 235.8 Hz).

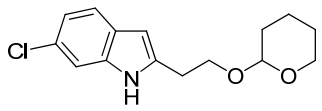
IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3415, 2955, 2858, 1626, 1592, 1526, 1495, 1465, 1329, 1229, 1134.

MS (EI, 70 eV): *m*/*z* (%) = 247 (45) [M^+], 204 (100) [$(M - C_3H_7)^+$], 162 (64), 148 (10), 133 (5).

HRMS (EI, 70 eV) calcd for $C_{16}H_{22}FN^+$ [M^+]: 247.1731; found: 247.1723.

13. Sanz, R.; Guilarte, V.; Castroviejo, M. P. *Synlett* **2008**, 3006.

6-Chloro-2-(2-(tetrahydro-2*H*-pyran-2-yloxy)ethyl)-1*H*-indole (8d)



Synthesized according to the general procedure using indole **7d** (151 mg, 0.99 mmol, 1 equiv.) and bromide **5e** (404 mg, 1.93 mmol, 1.95 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 4:1 to 2:1) to afford 2-alkylindole **8d** as a pale-yellow oil (212 mg, 76% yield).

TLC: R_f = 0.29 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.53-1.68 (m, 4H), 1.75-1.81 (m, 1H), 1.83-1.89 (m, 1H), 3.02 (t, J = 5.9 Hz, 2H), 3.48-3.53 (m, 1H), 3.71 (dt, J = 9.6, 5.9 Hz, 1H), 3.81-3.85 (m, 1H), 4.05 (dt, J = 9.6, 5.9 Hz, 1H), 4.63 (dd, J = 2.8, 4.8 Hz, 1H), 6.21-6.22 (m, 1H), 7.02 (dd, J = 1.9, 8.4 Hz, 1H), 7.26-7.29 (m, 1H), 7.41 (d, J = 8.4 Hz, 1H), 8.64 (br s, 1H).

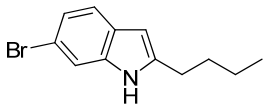
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 20.1, 25.4, 28.7, 31.0, 63.0, 67.3, 99.7, 100.1, 110.6, 120.2, 120.7, 126.9, 127.1, 136.5, 138.8.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3256, 2950, 2878, 1616, 1580, 1541, 1457, 1293, 1201, 1143, 1118, 1059, 1023.

MS (EI, 70 eV): *m*/*z* (%) = 281 (11) [M^+ with ^{37}Cl], 279 (25) [M^+ with ^{35}Cl], 232 (6), 195 (73), 177 (31), 164 (100), 85 (61).

HRMS (EI, 70 eV) calcd for $C_{15}H_{18}{}^{35}ClNO_2{}^+$ [M^+]: 279.1021; found: 279.1017.

6-Bromo-2-butyl-1*H*-indole (8e)



Synthesized according to the general procedure using indole **7e** (194 mg, 0.99 mmol, 1 equiv.) and butyl bromide (278 mg, 2.03 mmol, 2.05 equiv.). Base: 3 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 15 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 40:1 to 30:1) to afford 2-alkylindole **8e** as a white solid (185 mg, 74% yield).

TLC: R_f = 0.45 (pentane/ether 9:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.94 (t, J = 7.4 Hz, 3H), 1.40 (app. sextet, J = 7.4 Hz, 2H), 1.68 (app. quintet, J = 7.6 Hz, 2H), 2.71 (t, J = 7.6 Hz, 2H), 6.19-6.20 (m, 1H), 7.15 (dd, J = 1.7, 8.4 Hz, 1H), 7.36 (d, J = 8.4 Hz, 1H), 7.39 (m, 1H), 7.78 (br s, 1H).

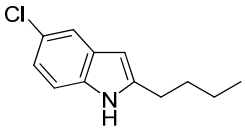
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.0, 22.5, 28.0, 31.2, 99.7, 113.3, 114.3, 121.0, 122.9, 127.9, 136.7, 141.0.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3402, 2954, 2856, 1605, 1577, 1542, 1455, 1398, 1340, 1287, 1050.

MS (EI, 70 eV): *m*/*z* (%) = 253 (41) [M^+ with ^{81}Br], 251 (41) [M^+ with ^{79}Br], 210 (100), 208 (96), 129 (30).

HRMS (EI, 70 eV) calcd for $C_{12}H_{14}BrN^+$ [M^+]: 251.0304; found: 251.0300.

2-Butyl-5-chloro-1*H*-indole (8f)



Synthesized according to the general procedure using indole **7f** (152 mg, 1.00 mmol, 1 equiv.) and butyl bromide (268 mg, 1.96 mmol, 1.96 equiv.). Base: 3 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ethyl acetate 40:1 to 20:1) to afford 2-alkylindole **8f** as a pale-yellow oil (176 mg, 85% yield).

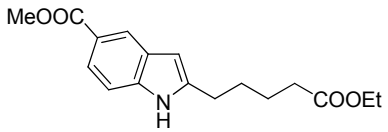
TLC: R_f = 0.30 (pentane/ether 9:1) [CAM].

^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.94 (t, *J* = 7.4 Hz, 3H), 1.40 (app. sextet, *J* = 7.5 Hz, 2H), 1.68 (app. quintet, *J* = 7.7 Hz, 2H), 2.71 (t, *J* = 7.7 Hz, 2H), 6.16 (s, 1H), 7.04 (dd, *J* = 2.0, 8.5 Hz, 1H), 7.16 (d, *J* = 8.5 Hz, 1H), 7.47 (d, *J* = 2.0 Hz, 1H), 7.82 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.0, 22.5, 28.1, 31.3, 99.4, 111.3, 119.3, 121.2, 125.3, 130.1, 134.3, 141.7.

The ^1H and ^{13}C NMR spectra are consistent with those reported in the literature.[14]

Methyl 2-(5-ethoxy-5-oxopentyl)-1*H*-indole-5-carboxylate (8g)



Synthesized according to the general procedure using indole **7g** (176 mg, 1.00 mmol, 1 equiv.) and bromide **5j** (426 mg, 2.04 mmol, 2.04 equiv.). Base: 3 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 3:1 to 1:1) to afford 2-alkylindole **8g** as a white solid (266 mg, 87% yield).

M.p. 91-92 °C. TLC: R_f = 0.30 (pentane/ether 9:1) [CAM].

^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.25 (t, *J* = 7.1 Hz, 3H), 1.69-1.80 (m, 4H), 2.35 (t, *J* = 7.0 Hz, 2H), 2.77 (t, *J* = 7.0 Hz, 2H), 3.92 (s, 3H), 4.13 (q, *J* = 7.1 Hz, 2H), 6.31 (m, 1H), 7.28 (d, *J* = 8.5 Hz, 1H), 7.83 (dd, *J*

14. Lai, R.-Y.; Surekha, K.; Hayashi, A.; Ozawa, F.; Liu, Y.-H.; Peng, S.-M.; Liu, S.-T. *Organometallics* **2007**, *26,* 1062.

= 1.7, 8.5 Hz, 1H), 8.28 (m, 1H), 8.52 (br s, 1H).

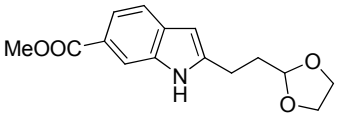
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.3, 24.5, 27.9, 28.5, 34.0, 51.9, 60.5, 100.9, 110.2, 121.7, 122.7, 122.8, 128.5, 138.8, 141.0, 168.6, 173.8.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3336, 2932, 2861, 1712, 1695, 1614, 1556, 1439, 1325, 1292, 1238, 1179, 1086.

MS (EI, 70 eV): *m*/*z* (%) = 303 (56) [M^+], 257 (34), 201 (100), 188 (68), 170 (26), 129 (21).

HRMS (EI, 70 eV) calcd for $C_{17}H_{21}NO_4^+$ [M^+]: 303.1465; found: 303.1456.

Methyl 2-(2-(1,3-dioxolan-2-yl)ethyl)-6-methoxy-1*H*-indole-6-carboxylate (8h)



Synthesized according to the general procedure using indole **7h** (175 mg, 1.00 mmol, 1 equiv.) and bromide **5h** (350 mg, 1.94 mmol, 1.94 equiv.). Base: 3 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 1:1 to 1:1.5) to afford 2-alkylindole **8h** as a pale-yellow solid (236 mg, 86% yield).

M.p. 89-91 °C. TLC: R_f = 0.18 (pentane/ether 1:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 2.13 (dt, *J* = 4.3, 7.2 Hz, 2H), 2.95 (t, *J* = 7.2 Hz, 2H), 3.92 (s, 3H), 3.91-3.96 (m, 2H), 4.01-4.06 (m, 2H), 4.99 (t, *J* = 4.3 Hz, 1H), 6.29 (s, 1H), 7.52 (d, *J* = 8.3 Hz, 1H), 7.76 (dd, *J* = 1.5, 8.3 Hz, 1H), 8.07 (m, 1H), 8.82 (br s, 1H).

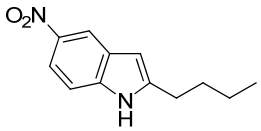
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 22.3, 32.6, 52.0, 65.2, 100.3, 103.6, 112.9, 119.3, 120.9, 122.6, 132.7, 135.4, 143.2, 168.6.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3336, 2948, 2873, 1693, 1621, 1574, 1539, 1435, 1304, 1215, 1126.

MS (EI, 70 eV): *m*/*z* (%) = 275 (6) [M^+], 217 (16), 189 (16), 101 (27), 99 (56), 73 (100).

HRMS (EI, 70 eV) calcd for $C_{15}H_{17}NO_4^+$ [M^+]: 275.1152; found: 275.1148.

2-Butyl-5-nitro-1*H*-indole (8i)



Synthesized according to the general procedure using indole **7i** (160 mg, 0.99 mmol, 1 equiv.) and butyl bromide (298 mg, 2.18 mmol, 2.20 equiv.). Base: 3 equiv. K_2HPO_4; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 17 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 3:1) to afford 2-alkylindole **8i** as a light-yellow solid (194 mg, 90% yield).

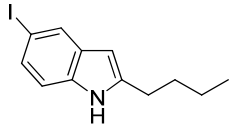
M.p. 99-100 °C. TLC: R_f = 0.33 (pentane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.97 (t, J = 7.4 Hz, 3H), 1.43 (app. sextet, J = 7.5 Hz, 2H), 1.73 (app. quintet, J = 7.6 Hz, 2H), 2.79 (t, J = 7.7 Hz, 2H), 6.39-6.40 (m, 1H), 7.32 (d, J = 9.0 Hz, 1H), 8.03 (dd, J = 2.2, 9.0 Hz, 1H), 8.41 (br s, 1H), 8.47 (d, J = 2.2 Hz, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 13.9, 22.5, 28.0, 31.1, 101.8, 110.3, 116.9, 117.0, 128.4, 139.2, 141.9, 143.7.

This is a known compound, but no spectroscopic data were given.[13]

2-Butyl-5-iodo-1*H*-indole (8j)



Synthesized according to the general procedure using indole **7j** (242 mg, 0.996 mmol, 1 equiv.) and butyl bromide (554 mg, 4.04 mmol, 4.06 equiv.). Base: 4 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 14 h. The crude product was purified by flash column chromatography (eluted with hexane/ether 40:1 to 2:1) to afford 2-butyl-5-iodoindole **8j** as a pale-yellow solid (194 mg, 65% yield) and 2,5'-bisindole byproduct **8j'** as a dark-yellow solid (16.1 mg, 8% yield).

M.p. 50-52 °C. TLC: R_f = 0.50 (hexane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0.94 (t, J = 7.4 Hz, 3H), 1.40 (app. sextet, J = 7.4 Hz, 2H), 1.68 (app. quintet, J = 7.6 Hz, 2H), 2.72 (t, J = 7.7 Hz, 2H), 6.14-6.15 (m, 1H), 7.04 (d, J = 8.4 Hz, 1H), 7.34 (dd, J = 1.6, 8.4 Hz, 1H), 7.83-7.86 (m, 2H).

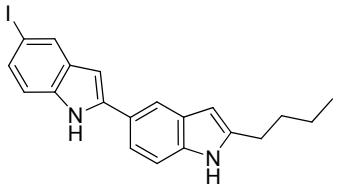
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.0, 22.5, 28.0, 31.3, 83.1, 99.0, 112.3, 128.6, 129.3, 131.6, 135.0, 141.1.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3410, 2953, 2926, 2857, 1572, 1457, 1407, 1302.

MS (EI, 70 eV): m/z (%) =299 (76) [M^+], 257 (100), 256 (90), 129 (35), 102 (10).

HRMS (EI, 70 eV) calcd for $C_{12}H_{14}{}^{127}IN^+$ [M^+]: 299.0166; found: 299.0162.

2'-Butyl-5-iodo-1*H*,1'*H*-2,5'-bisindole (8j')



M.p. 150-152 °C. TLC: R_f = 0.17 (hexane/ether 2:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 0. 96 (t, J = 7.4 Hz, 3H), 1.43 (app. sextet, J = 7.4 Hz, 2H), 1.72 (app.

quintet, J = 7.6 Hz, 2H), 2.76 (t, J = 7.6 Hz, 2H), 6.27-6.28 (m, 1H), 6.66 (m, 1H), 7.14 (d, J = 8.4 Hz, 1H), 7.32 (d, J = 8.4 Hz, 1H), 7.38 (dd, J = 1.7, 8.4 Hz, 1H), 7.41 (dd, J = 1.7, 8.4 Hz, 1H), 7.76-7.77 (m, 1H), 7.92-7.93 (m, 2H), 8.34 (br s, 1H).

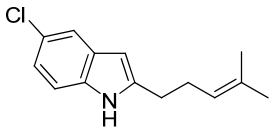
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 14.0, 22.5, 28.1, 31.3, 83.5, 97.8, 100.0, 111.1, 112.7, 116.7, 119.3, 123.8, 129.0, 129.5, 129.8, 132.4, 135.8, 135.9, 140.9, 141.5.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3400, 2958, 2923, 2857, 1760, 1601, 1571, 1444, 1396, 1306.

MS (EI, 70 eV): m/z (%) = 414 (100) [M^+], 370 (31), 288 (16), 244 (16), 185 (12), 167 (10), 111 (23).

HRMS (EI, 70 eV) calcd for $C_{20}H_{19}{}^{127}IN_2{}^+$ [M^+]: 414.0588; found: 414.0582.

5-Chloro-2-(4-methylpent-3-enyl)-1*H*-indole (8k)



Synthesized according to the general procedure using indole **7f** (151 mg, 0.99 mmol, 1 equiv.) and bromide **5m** (321 mg, 1.97 mmol, 1.99 equiv.). Base: 3 equiv. $KHCO_3$; degassing method: freeze-pump-thaw using liquid N_2; reaction temperature: 70 °C; reaction time: 38 h. The crude product was purified by flash column chromatography (eluted with pentane/ether 20:1 to 10:1) to afford 2-alkylindole **8j** as a pale-yellow solid (130 mg, 56% yield), and the recovered indole **7f** as a pale-yellow solid (36.5 mg, 24%). The yield based on recovered starting material was 74%.

M.p. 50-52 °C. TLC: R_f = 0.54 (pentane/ether 4:1) [CAM].

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 1.58-1.59 (m, 3H), 1.70-1.71 (m, 3H), 2.37 (app. q, J = 7.3 Hz, 2H), 2.73 (t, J = 7.5 Hz, 2H), 5.19 (app. tseptet, J = 7.1, 1.4 Hz, 1H), 6.16-6.17 (m, 1H), 7.04 (dd, J = 2.0, 8.5 Hz, 1H), 7.14 (d, J = 8.5 Hz, 1H), 7.47 (d, J = 2.0 Hz, 1H), 7.85 (br s, 1H).

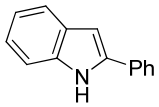
^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 17.9, 25.8, 27.7, 28.5, 99.5, 111.3, 119.3, 121.2, 123.3, 125.3, 130.0, 133.3, 134.3, 141.5.

IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3397, 2972,2910, 1575, 1543, 1466, 1442, 1409.

MS (EI, 70 eV): m/z (%) = 235 (4) [M^+ with ^{37}Cl], 233 (12) [M^+ with ^{35}Cl], 164 (100), 149 (4), 128 (5).

HRMS (EI, 70 eV) calcd for $C_{14}H_{16}{}^{35}ClN^+$ [M^+]: 233.0966; found: 233.0968.

2-Phenyl-1*H*-indole (9)



Synthesized according to the general procedure using indole (25.0 mg, 0.213 mmol, 1 equiv.) and iodobenzene (83.2 mg, 0.408 mmol, 1.91 equiv.). Base: 2 equiv. K_2CO_3; degassing method: freeze-pump-thaw

using liquid N_2; reaction temperature: 70 °C; reaction time: 16 h. The crude product was purified by flash column chromatography (eluted with hexane/ethyl acetate 15:1) to afford 2-phenylindole (**9**) as an off-white solid (35.7 mg, 86% yield).

M.p. 166-167 °C. TLC: R_f = 0.35 (hexane/ethyl acetate 9:1) [CAM].

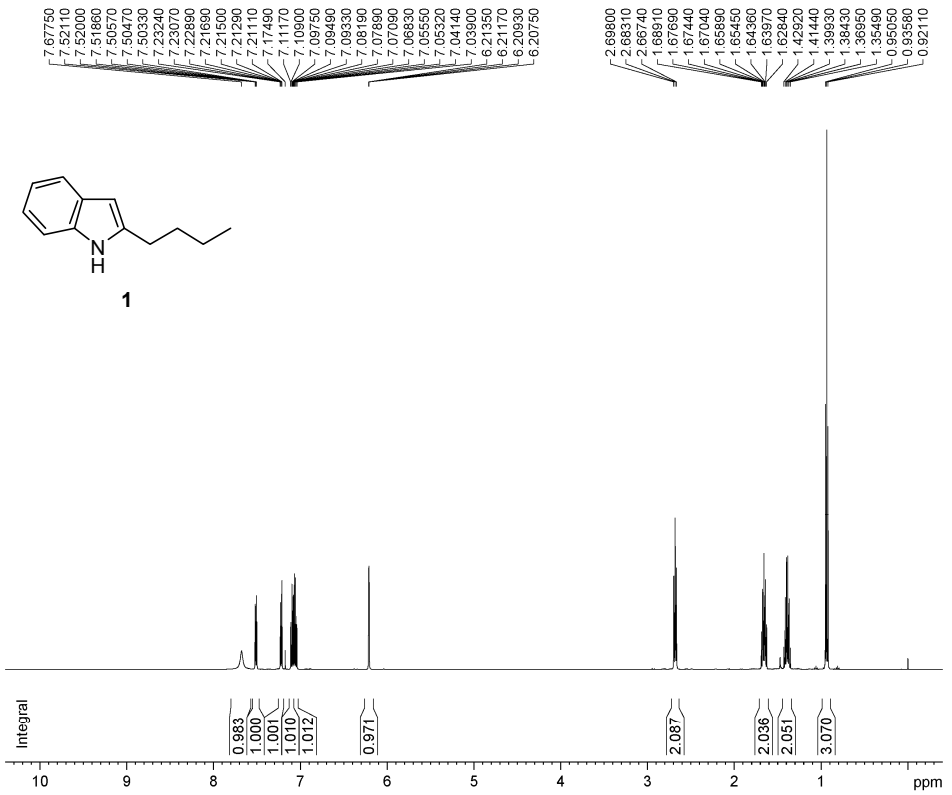
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 6.81 (m, 1H), 7.11 (app. dt, J = 1.0, 7.5 Hz, 1H), 7.18 (app. dt, J = 1.2, 7.6 Hz, 1H), 7.30 (app. tt, J = 1.2, 7.4 Hz, 1H), 7.36 (d, J = 8.1 Hz, 1H), 7.40-7.43 (m, 2H), 7.61-7.63 (m, 3H), 8.26 (br s, 1H).

^{13}C NMR (90.6 MHz, $CDCl_3$): δ (ppm) = 100.1, 111.0, 120.5, 120.8, 122.5, 125.3, 127.8, 129.1, 129.4, 132.5, 137.0, 138.0.

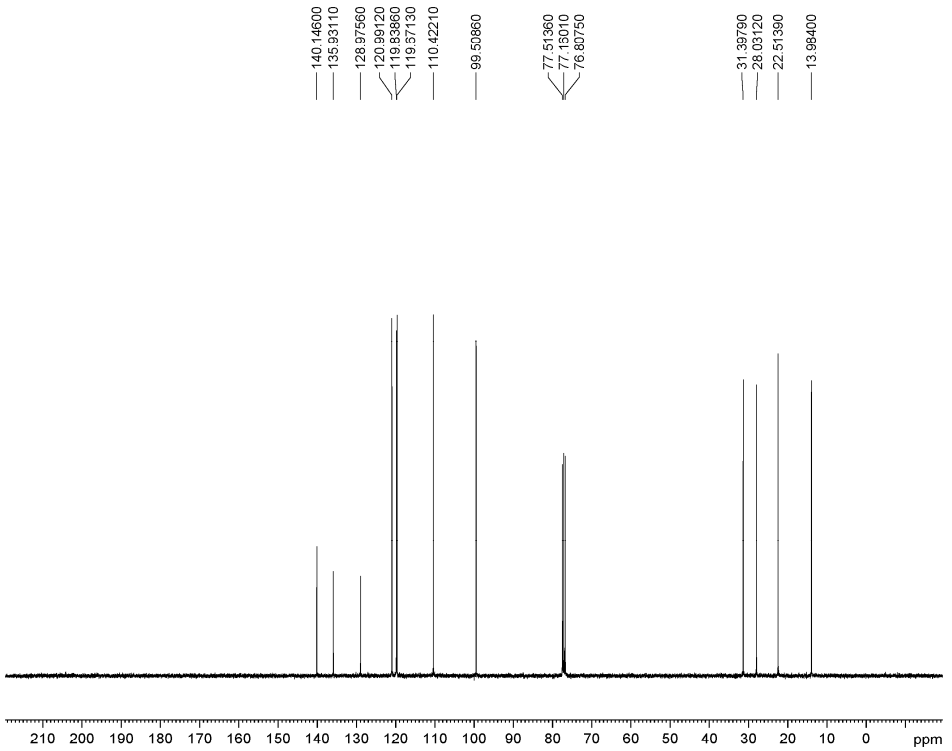
The ^{1}H and ^{13}C NMR spectra are consistent with those reported in the literature.[15]

15. Maizuru, N.; Inami, T.; Kurahashi, T.; Matsubara, S. *Org. Lett.* **2011**, *13*, 1206.

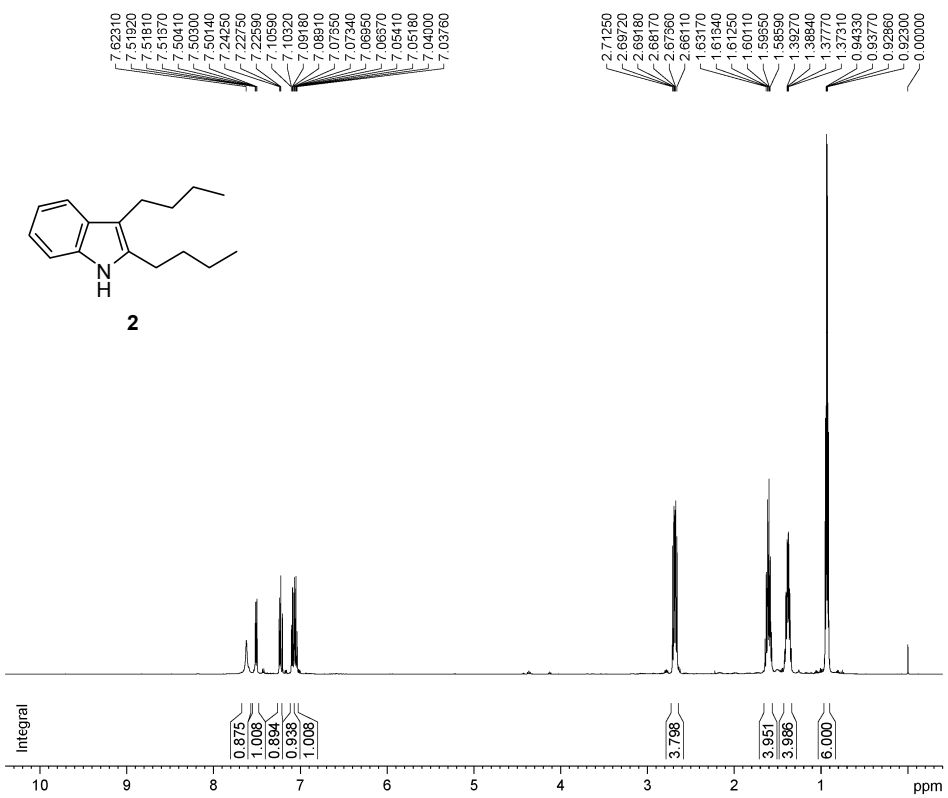
3. ^{1}H and ^{13}C-NMR Spectra for Synthesized Compounds



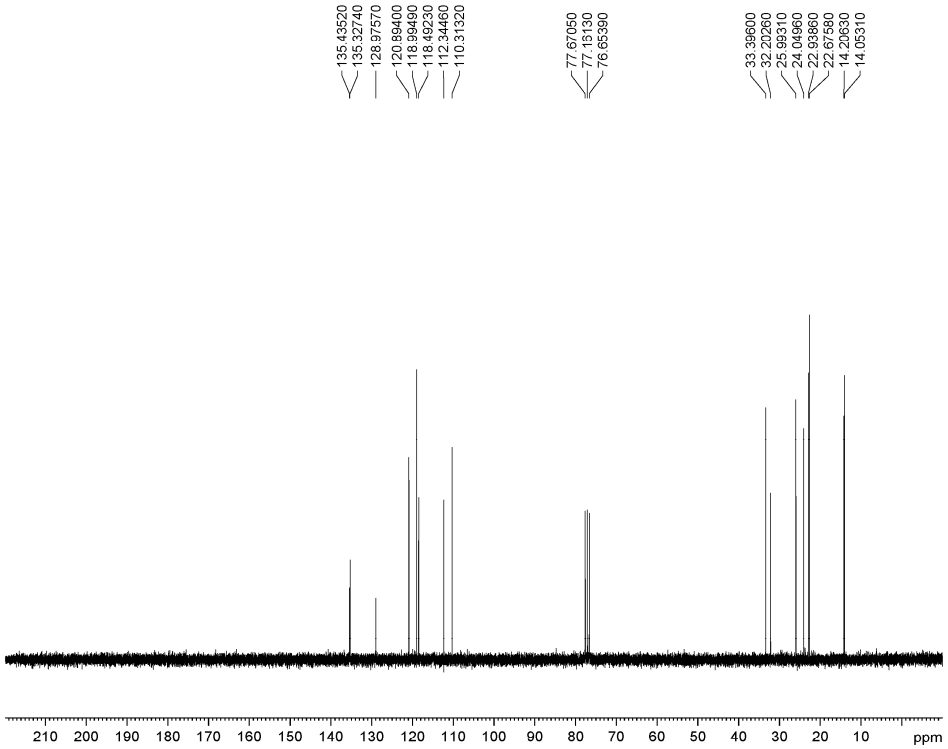

```
NAME              JLB256-2
EXPNO                   10
PROCNO                   1
Date_             20110531
Time                 20.59
INSTRUM              spect
PROBHD    5 mm SEI 1H-13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       4
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1720407 sec
RG                    35.9
DW                  48.400 usec
DE                   6.00 usec
TE                  299.8 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    8.20 usec
PL1                  -1.00 dB
PL1W           12.18109322 W
SFO1           500.1330885 MHz
SI                   65536
SF             500.1300558 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



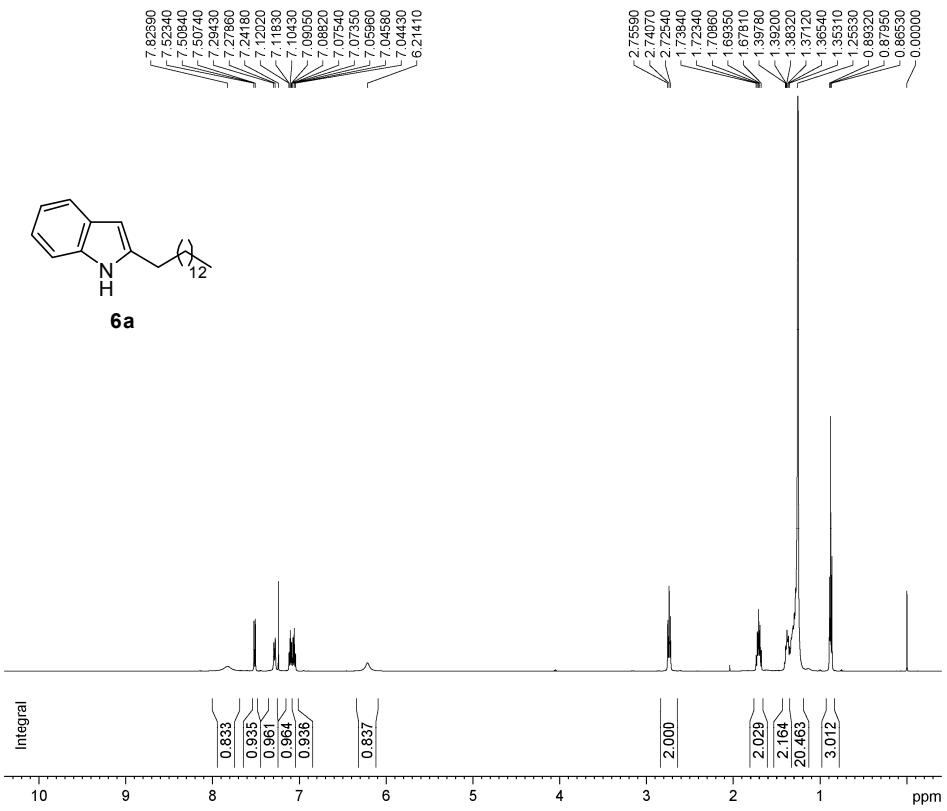

```
NAME              JLB256-2
EXPNO                   11
PROCNO                   1
Date_             20110531
Time                 16.16
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH              21645.021 Hz
FIDRES           0.330277 Hz
AQ               1.5139316 sec
RG                    3251
DW                  23.100 usec
DE                   6.50 usec
TE                  299.0 K
D1              2.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    7.00 usec
PL1                  -2.00 dB
SFO1           90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                115.00 usec
PL2                   0.00 dB
PL12                 23.37 dB
PL13                 26.00 dB
SFO2          360.1314405 MHz
SI                   32768
SF             90.5547564 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```




```
NAME            JLB156-2
EXPNO                 10
PROCNO                 1
Date_           20110310
Time                21.08
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            10330.578 Hz
FIDRES             0.157632 Hz
AQ              3.1720407 sec
RG                  35.9
DW                 48.400 usec
DE                  6.00 usec
TE                299.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                 65536
SF            500.1300393 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```




```
NAME            JLB156-2
EXPNO                 11
PROCNO                 1
Date_           20110309
Time                17.04
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     2
SWH            15060.241 Hz
FIDRES             0.229801 Hz
AQ              2.1758451 sec
RG                   362
DW                 33.200 usec
DE                  6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.50 usec
PL1                 0.00 dB
SFO1          62.9015280 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              95.00 usec
PL2                 0.00 dB
PL12               18.00 dB
PL13               24.00 dB
SFO2          250.1310005 MHz
SI                 32768
SF             62.8952303 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```

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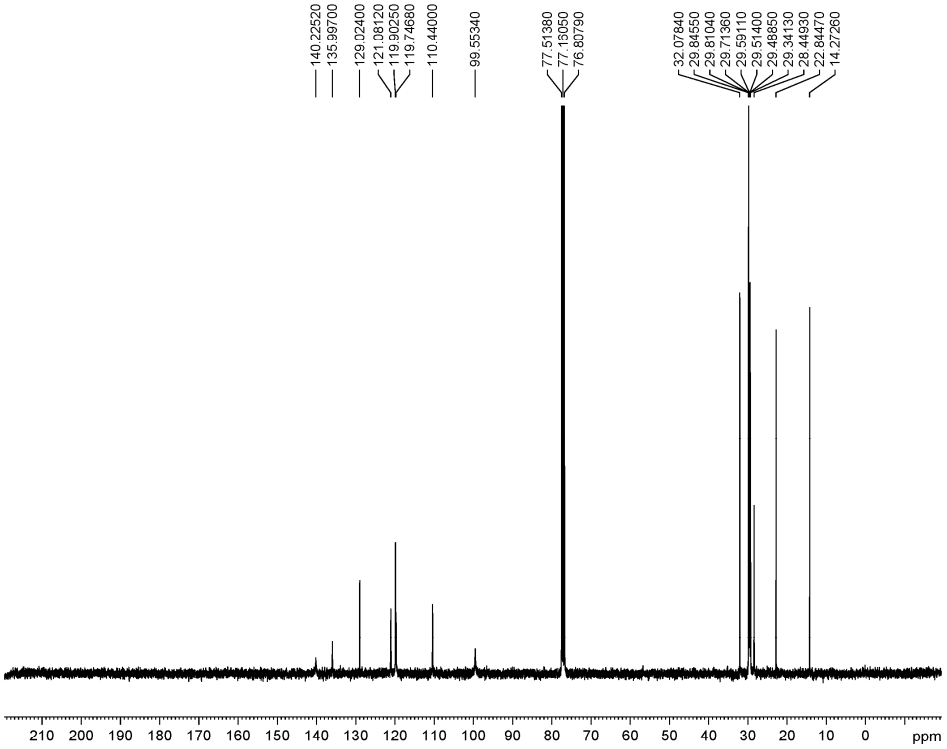
NAME            JLB227-2
EXPNO                 10
PROCNO                 1
Date_           20110428
Time                13.33
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                    57
DW               48.400 usec
DE                6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300225 MHz
WDW                   EM
SSB                    0
LB                0.20 Hz
GB                     0
PC                1.00
```




```
NAME            JLB226-2
EXPNO                 11
PROCNO                 1
Date_           20110430
Time                0.21
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                4597.6
DW               23.100 usec
DE                6.50 usec
TE                297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF         90.5547499 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
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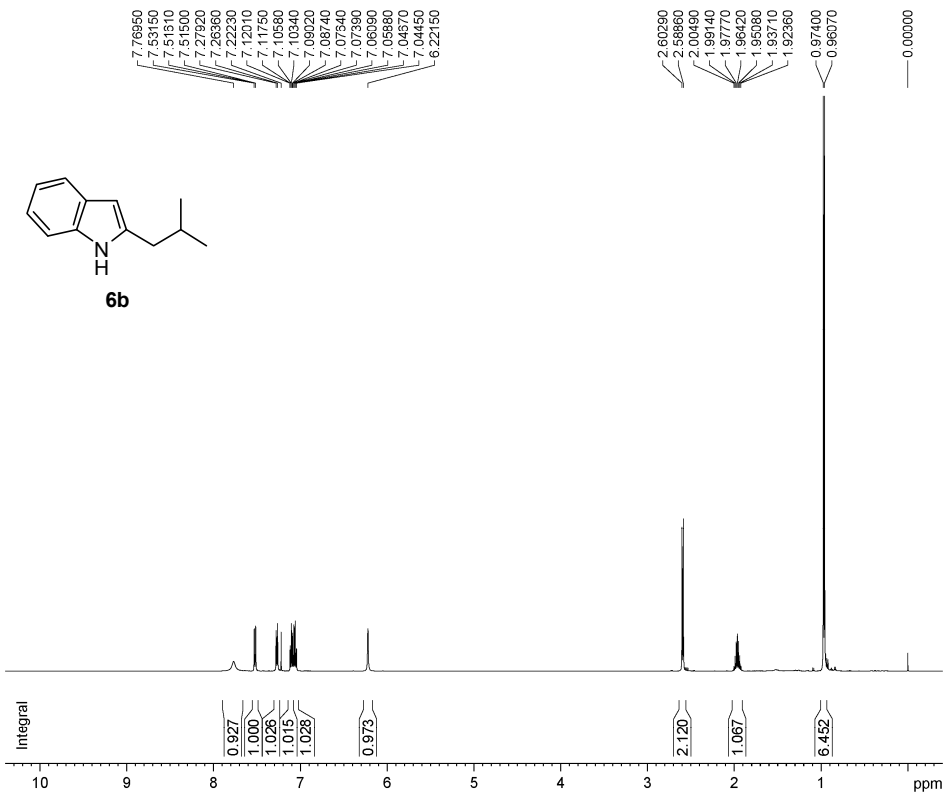
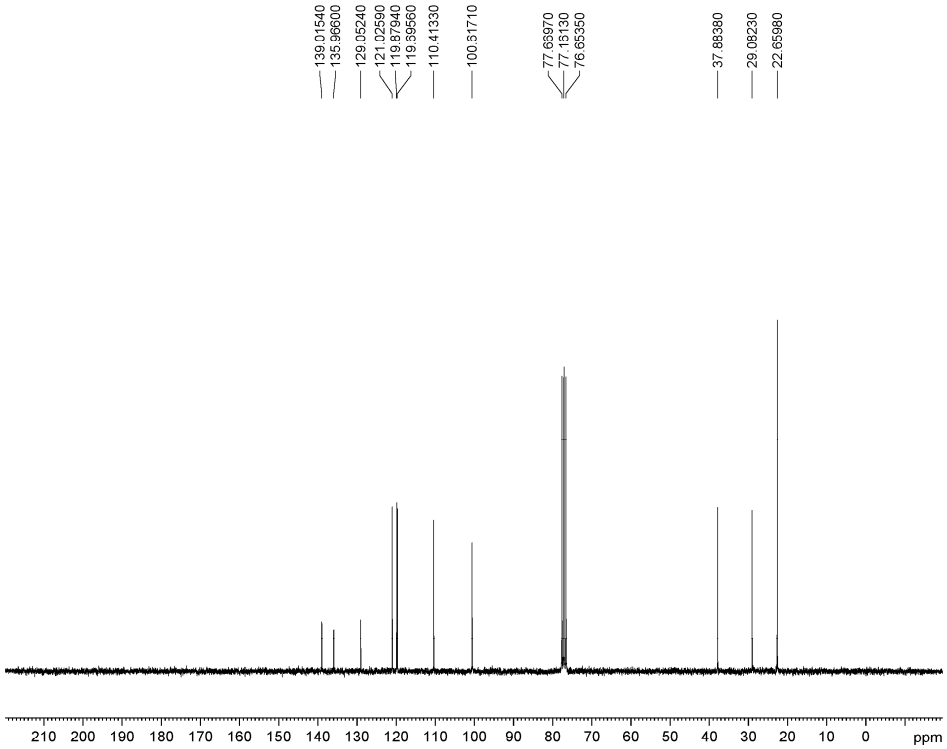
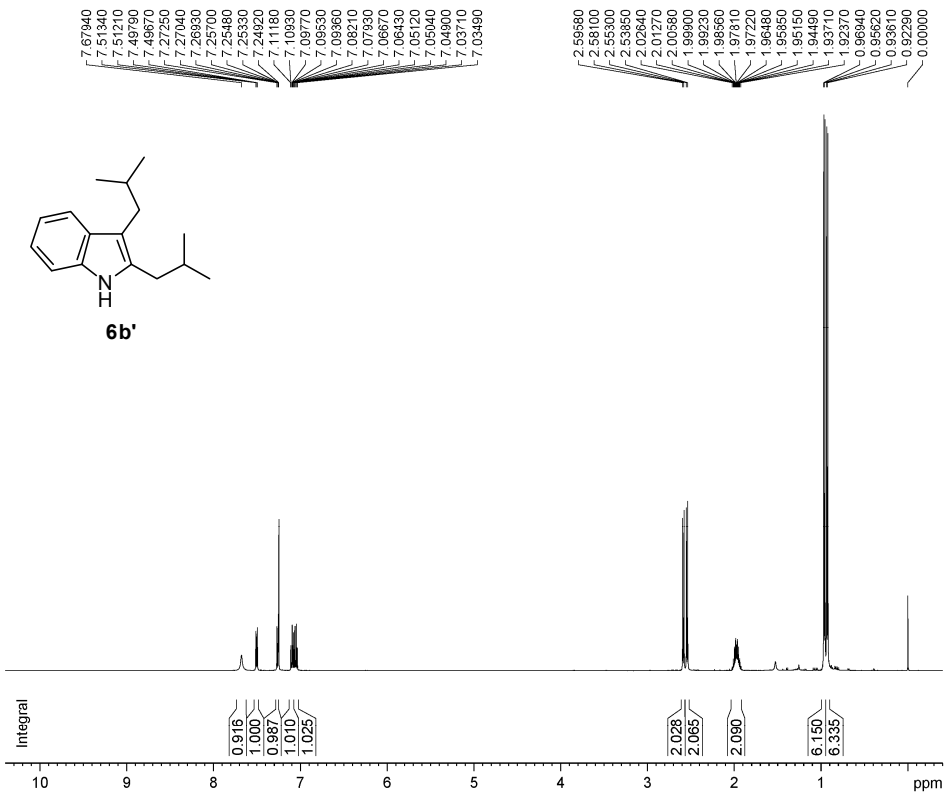


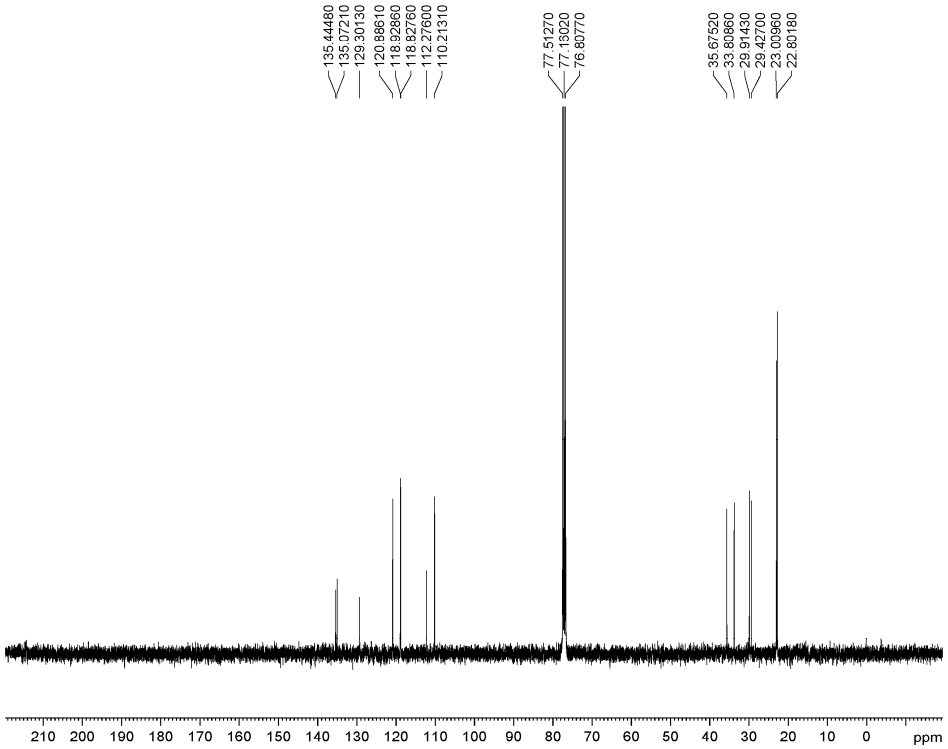

```
NAME          JLB154-2-Re
EXPNO                  10
PROCNO                  1
Date_           20110313
Time                18.33
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                     57
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                  65536
SF           500.1300323 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



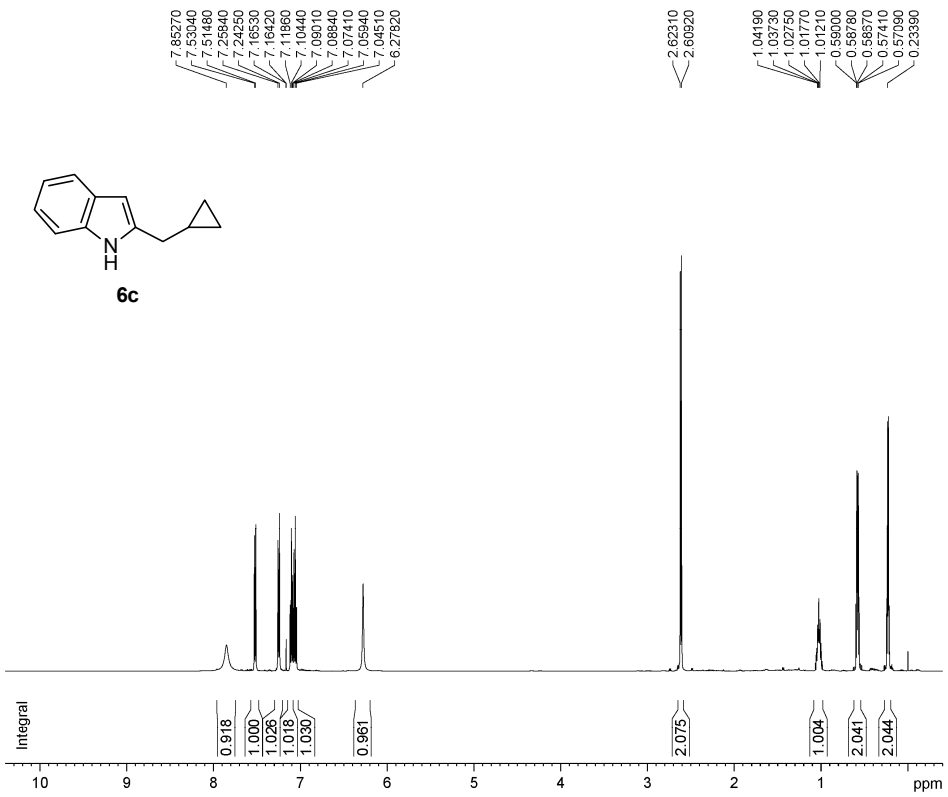

```
NAME          JLB154-2-Re
EXPNO                  11
PROCNO                  1
Date_           20110313
Time                15.31
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    768
DS                      2
SWH           15060.241 Hz
FIDRES         0.229801 Hz
AQ             2.1758451 sec
RG                    362
DW                33.200 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.50 usec
PL1                  0.00 dB
SFO1         62.9015280 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               95.00 usec
PL2                  0.00 dB
PL12                18.00 dB
PL13                24.00 dB
SFO2        250.1310005 MHz
SI                  32768
SF           62.8952303 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



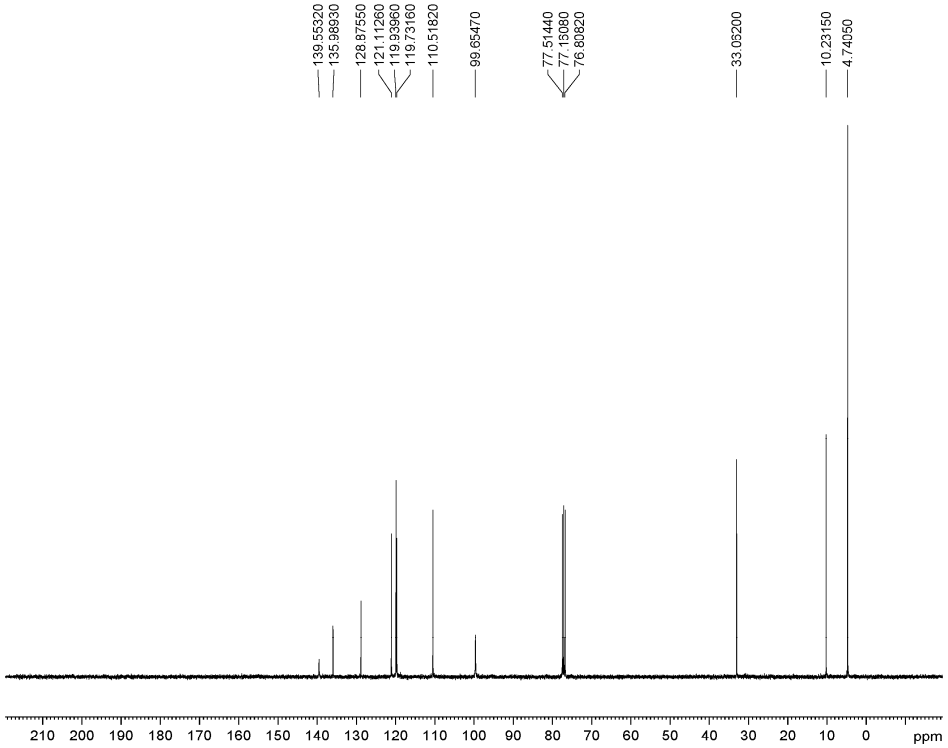

```
NAME            JLB154-1
EXPNO                 10
PROCNO                 1
Date_           20110310
Time                8.03
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                   128
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W         12.18109322 W
SFO1        500.1330885 MHz
SI                 65536
SF          500.1300189 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



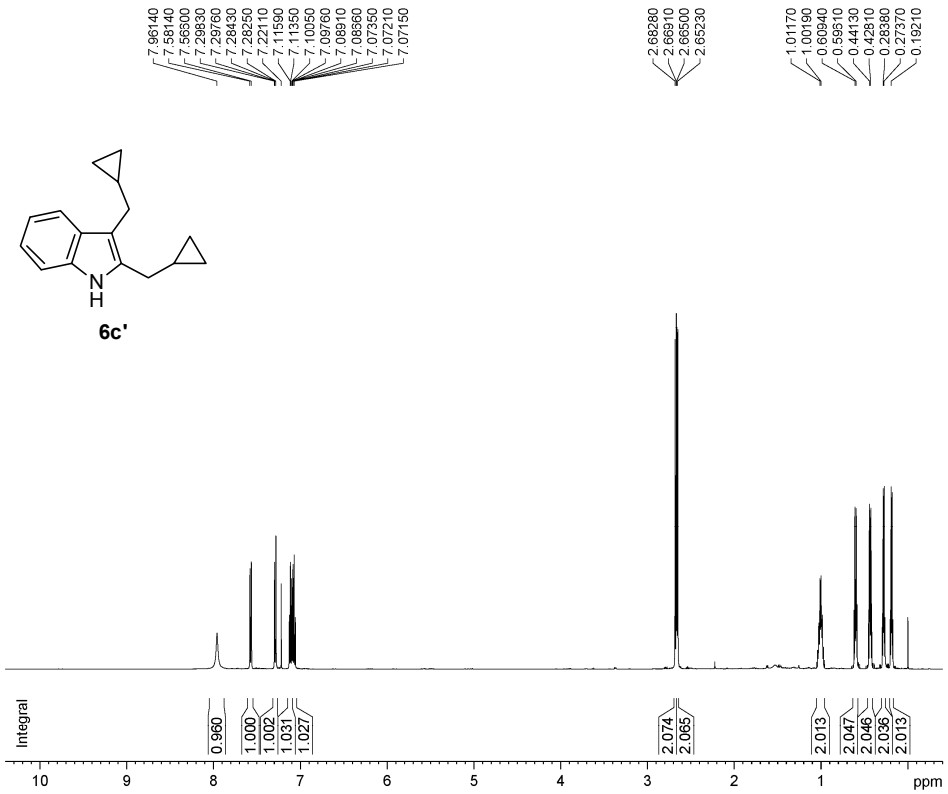

```
NAME            JLB154-1
EXPNO                 11
PROCNO                 1
Date_           20110309
Time               13.33
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   996
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                  3251
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF          90.5547470 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```



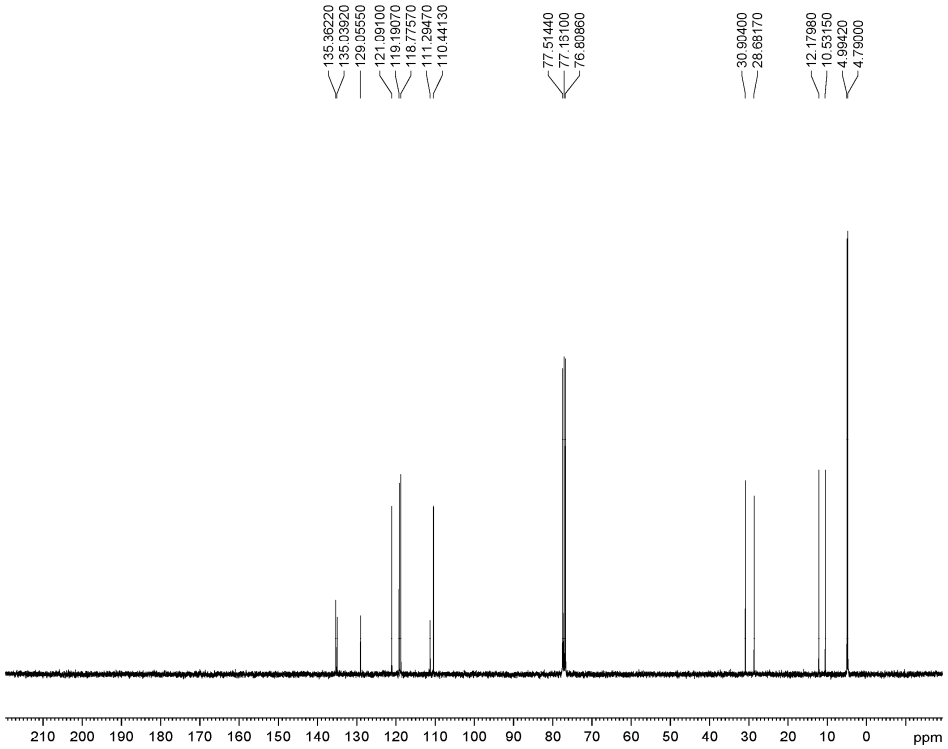

```
NAME            JLB210-2
EXPNO                 10
PROCNO                 1
Date_           20110408
Time                11.49
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300608 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



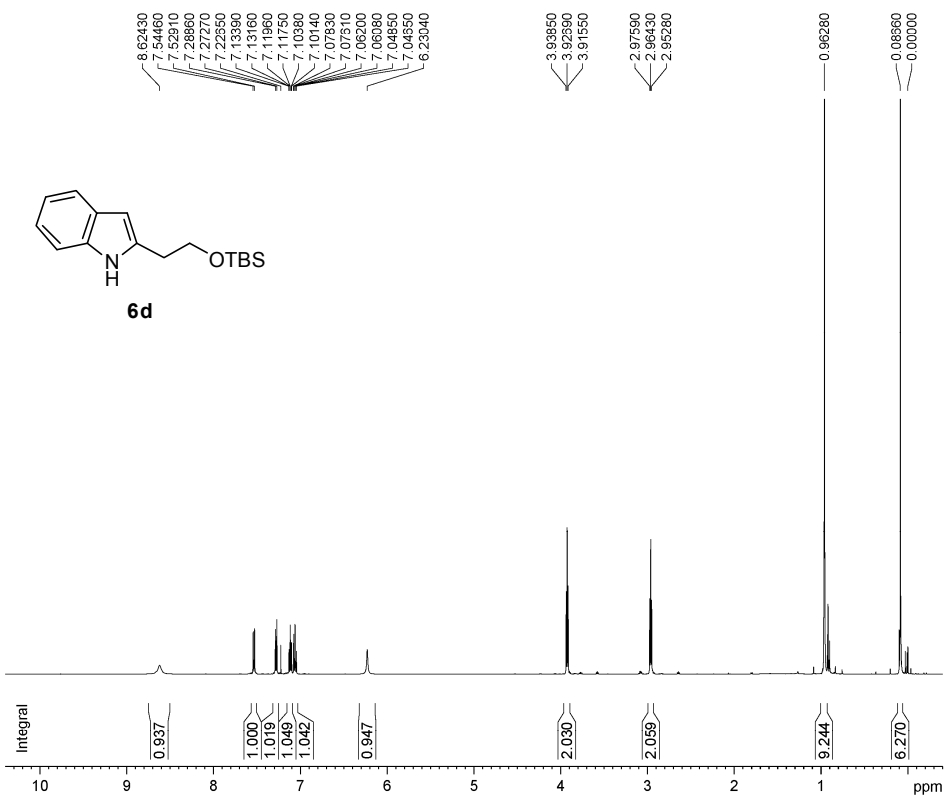

```
NAME            JLB210-2
EXPNO                 11
PROCNO                 1
Date_           20110408
Time                8.44
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                2298.8
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547577 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



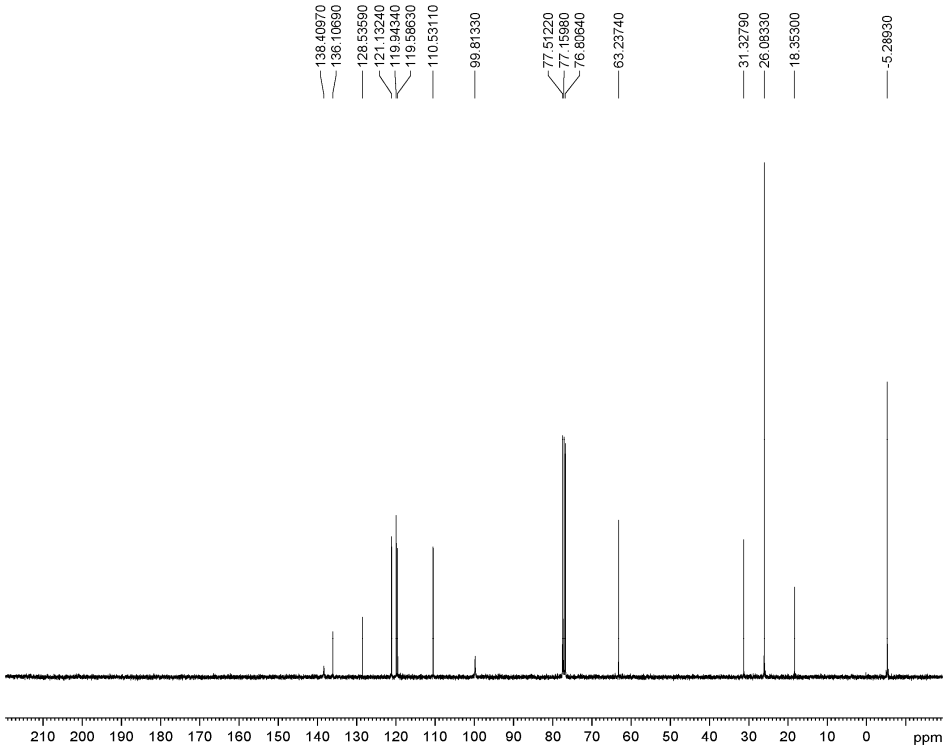

```
NAME            JLB210-1
EXPNO                 10
PROCNO                 1
Date_           20110407
Time                18.04
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300327 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



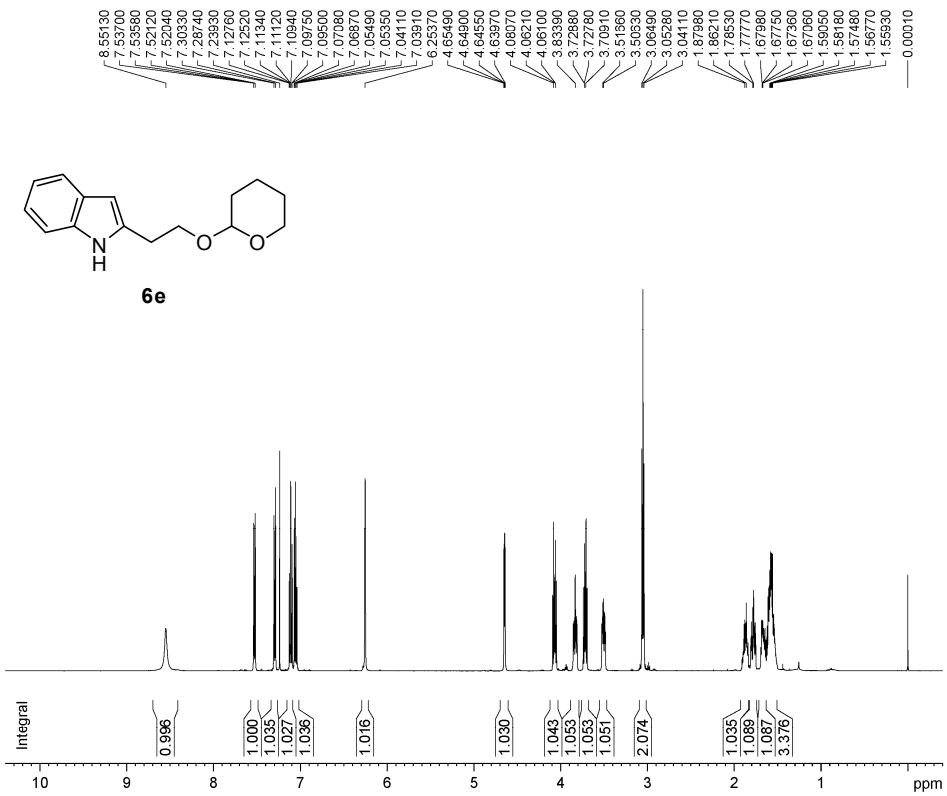

```
NAME            JLB210-1
EXPNO                 11
PROCNO                 1
Date_           20110408
Time                13.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                2298.8
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF         90.5547504 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```



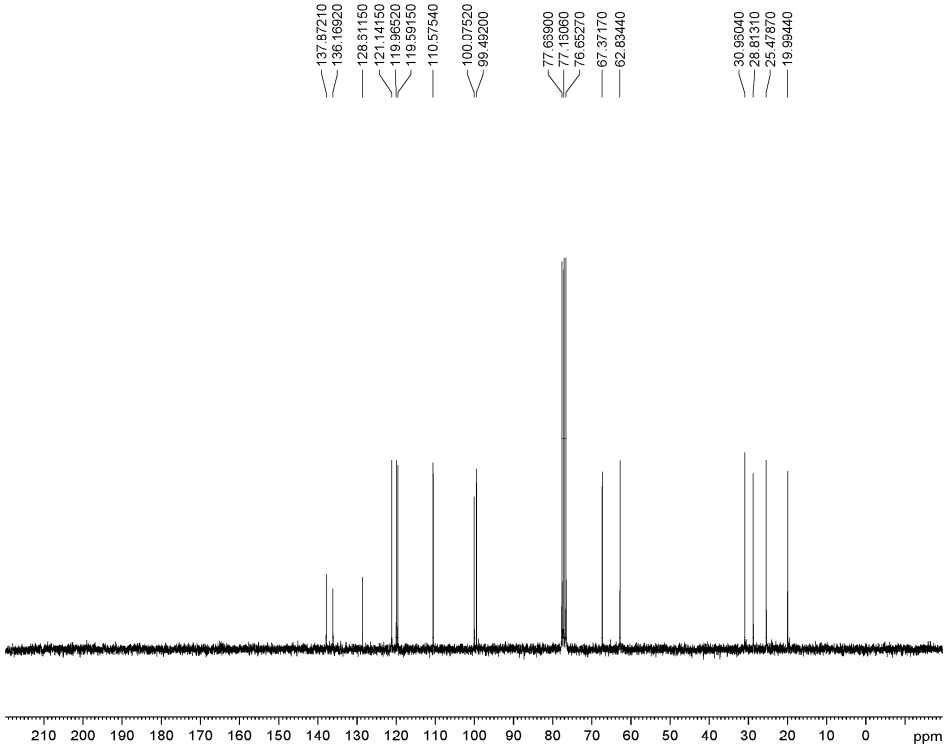

```
NAME              JLB212
EXPNO                 10
PROCNO                 1
Date_           20110411
Time                20.02
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300302 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



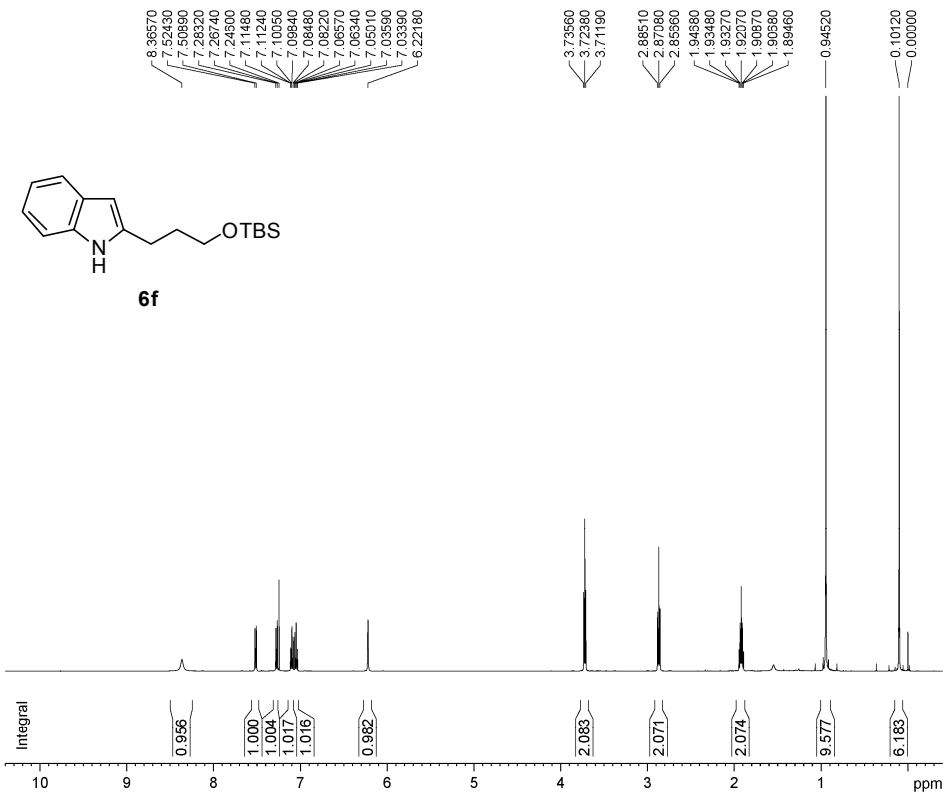

```
NAME              JLB212
EXPNO                 11
PROCNO                 1
Date_           20110411
Time                17.36
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES        0.330277 Hz
AQ            1.5139316 sec
RG                2580.3
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF         90.5547509 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```




```
NAME           JLB180-Re
EXPNO                 10
PROCNO                 1
Date_           20110320
Time                12.17
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1720407 sec
RG                  71.8
DW                 48.400 usec
DE                  6.00 usec
TE                 299.8 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                  65536
SF            500.1300237 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

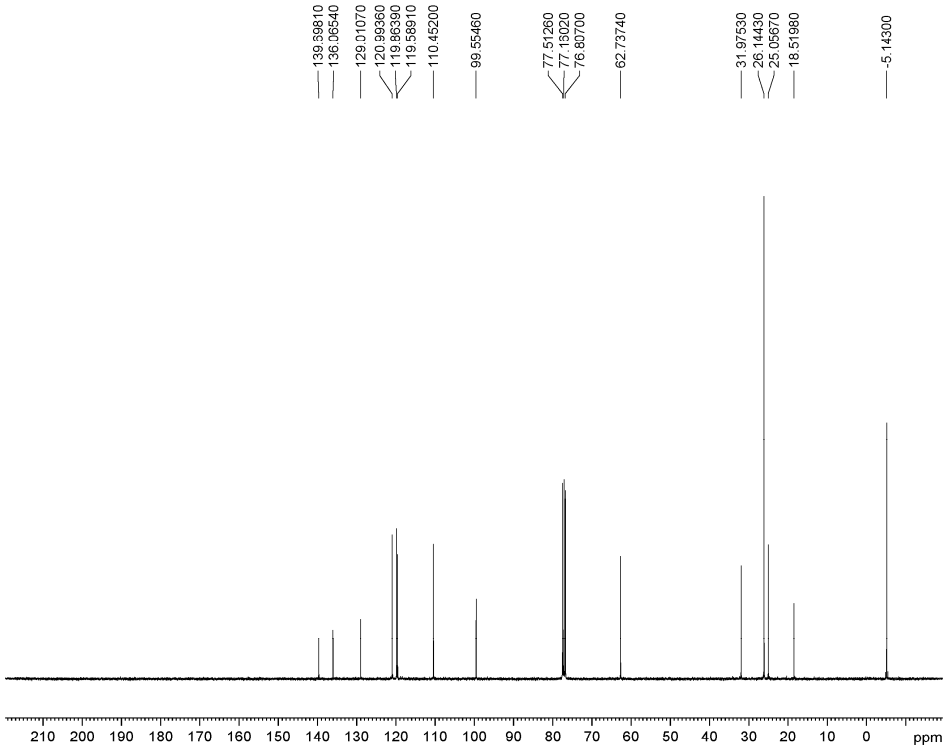



```
NAME             JLB178
EXPNO                11
PROCNO                1
Date_          20110317
Time               12.47
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           15060.241 Hz
FIDRES         0.229801 Hz
AQ             2.1758451 sec
RG                574.7
DW                33.200 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PL1                0.00 dB
SFO1          62.9015280 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              95.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              24.00 dB
SFO2         250.1310005 MHz
SI                 32768
SF            62.8952304 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

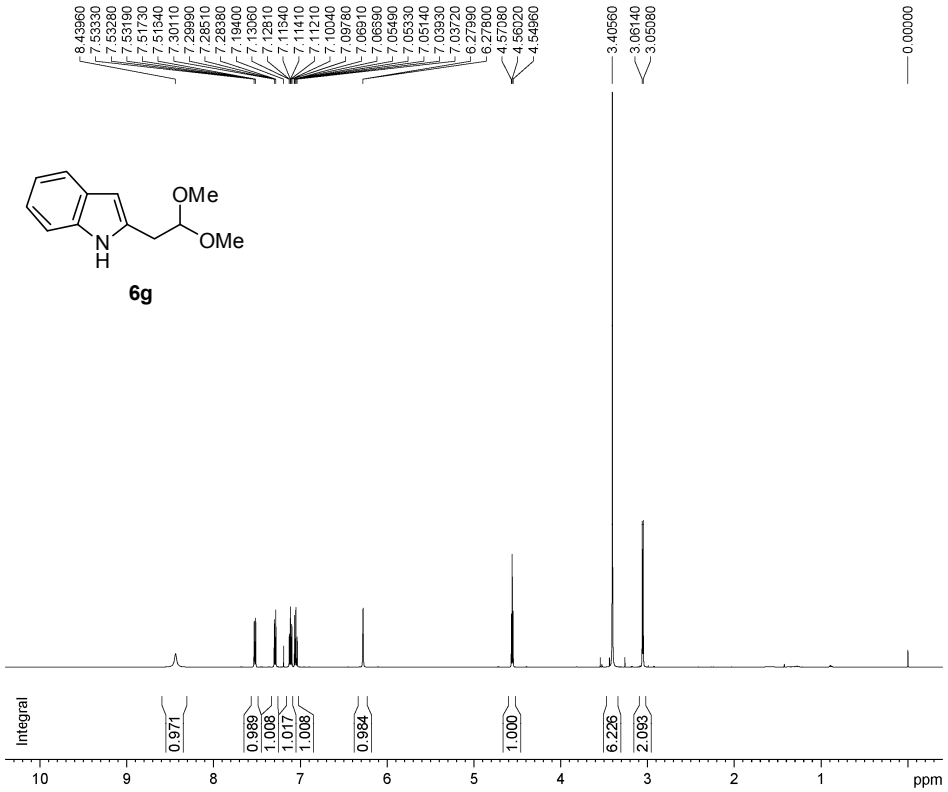


6f

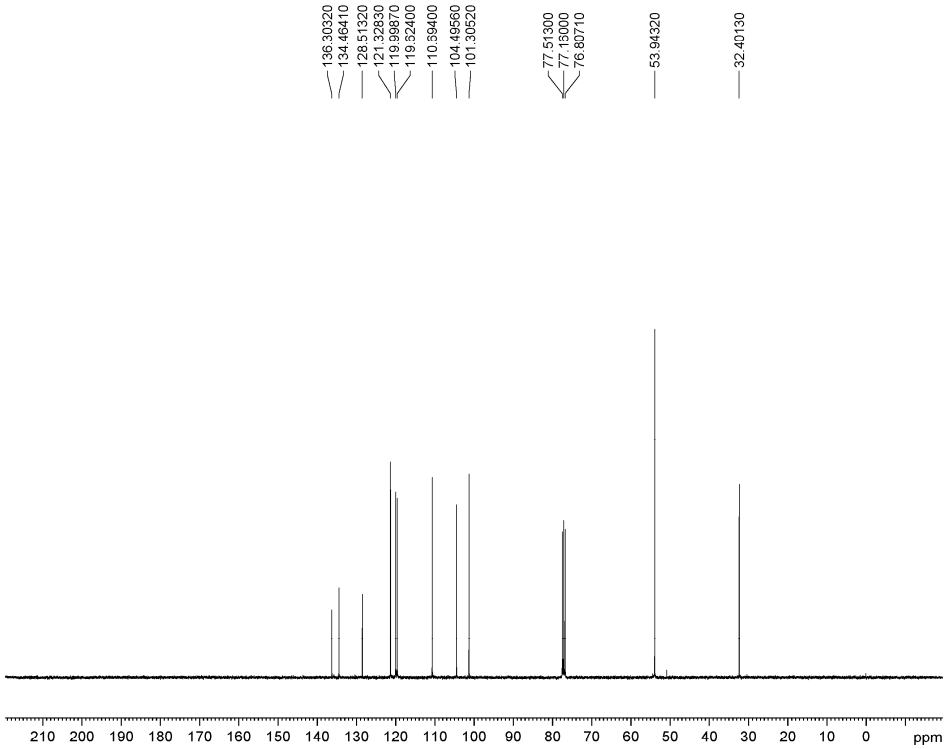
```
NAME          JLB202-2-Re
EXPNO                  10
PROCNO                  1
Date_           20110405
Time                12.03
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  90.5
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                  65536
SF           500.1300203 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```



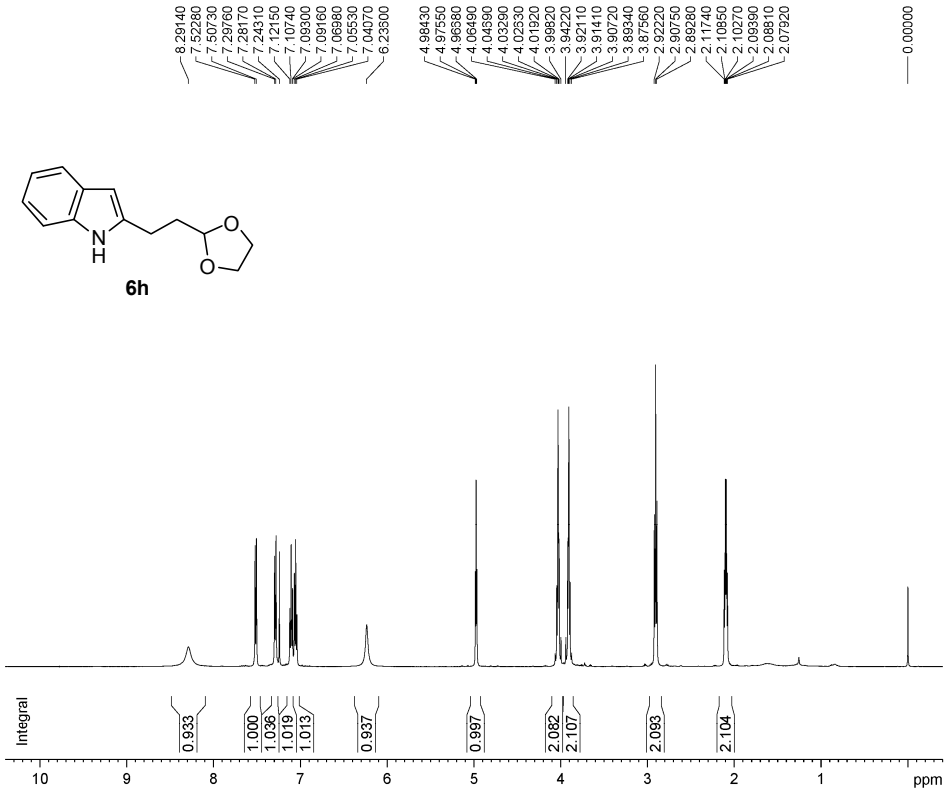

```
NAME          JLB202-2-Re
EXPNO                  11
PROCNO                  1
Date_           20110406
Time                1.05
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                1625.5
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2         360.1314405 MHz
SI                  32768
SF            90.5547509 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```



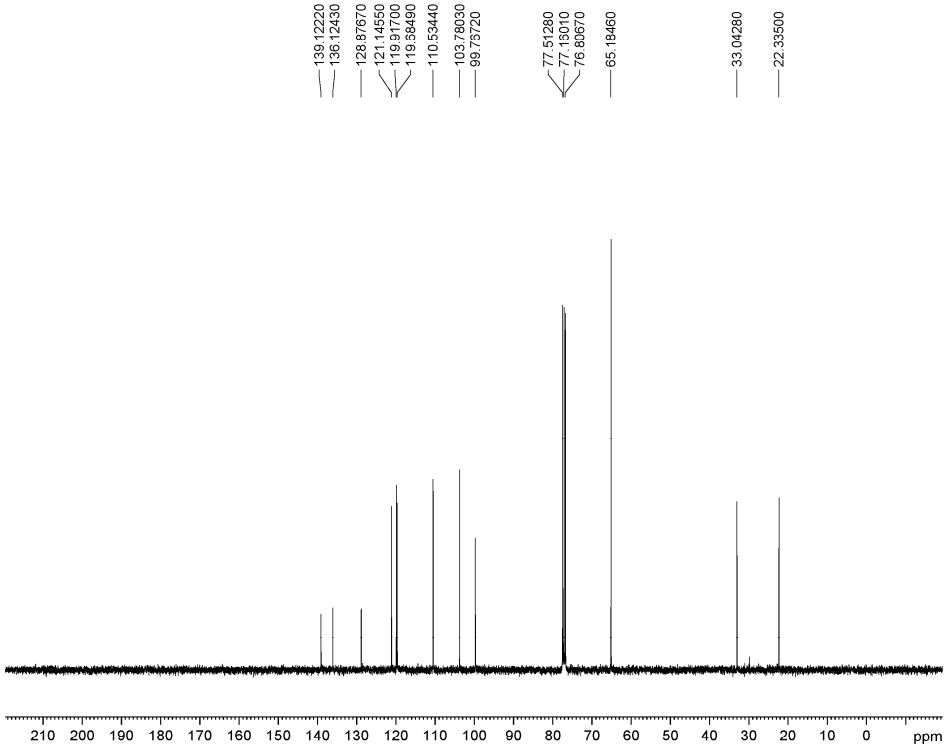

```
NAME              JLB223
EXPNO                 10
PROCNO                 1
Date_           20110420
Time               15.23
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W        12.18109322 W
SFO1        500.1330885 MHz
SI                 65536
SF          500.1300462 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



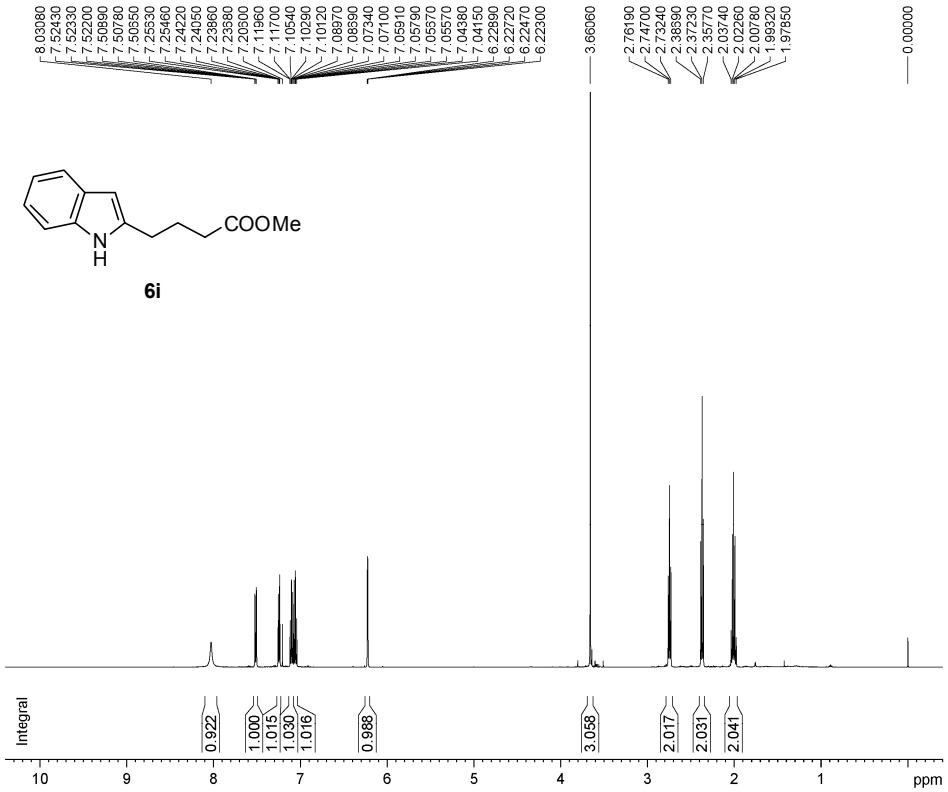

```
NAME              JLB223
EXPNO                 11
PROCNO                 1
Date_           20110421
Time               15.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                3649.1
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547563 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



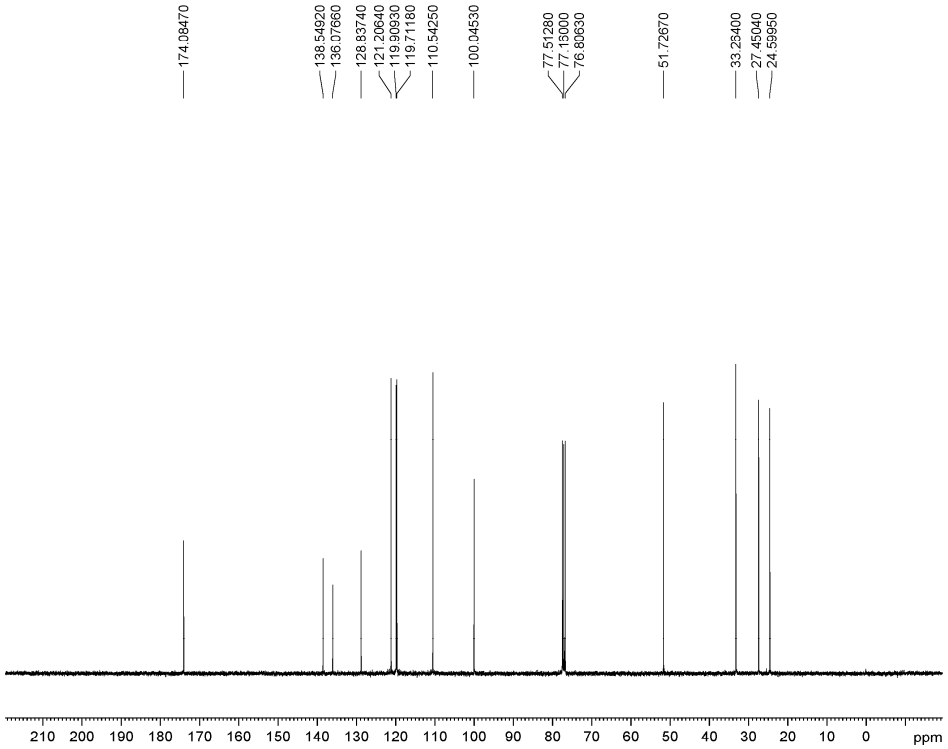

```
NAME           JLB211-Re
EXPNO                 10
PROCNO                 1
Date_           20110411
Time               18.32
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1720407 sec
RG                  90.5
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300217 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



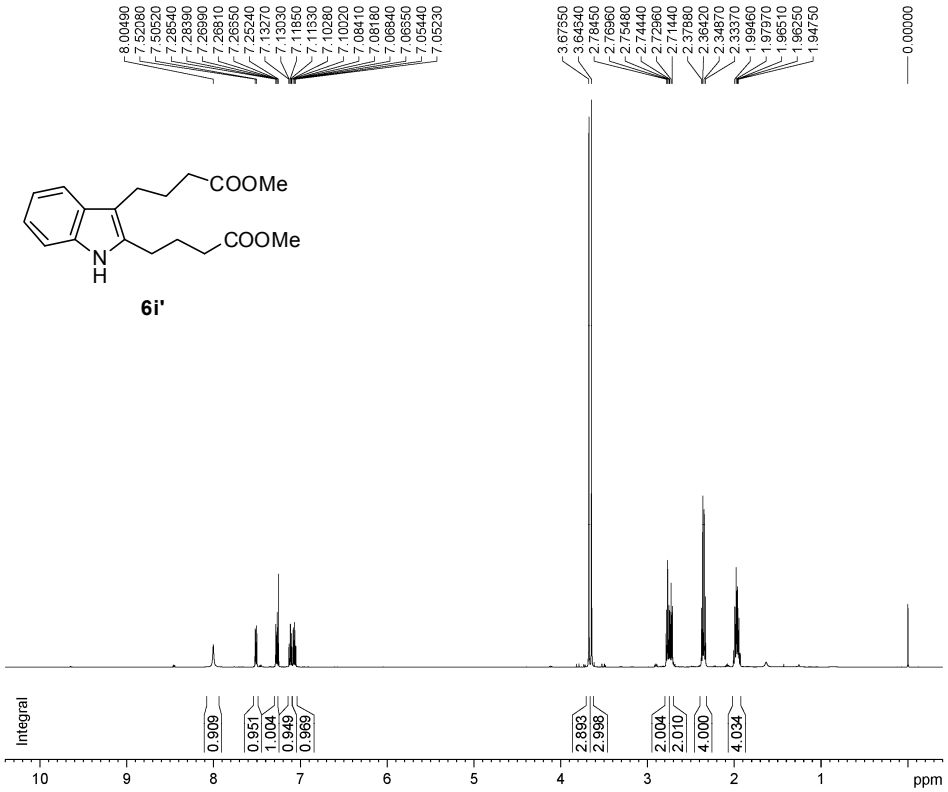

```
NAME             JLB209
EXPNO                11
PROCNO                1
Date_         20110408
Time              9.13
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                  300
DS                    4
SWH          21645.021 Hz
FIDRES        0.330277 Hz
AQ            1.5139316 sec
RG               2298.8
DW               23.100 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                7.00 usec
PL1              -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2               0.00 dB
PL12             23.37 dB
PL13             26.00 dB
SFO2       360.1314405 MHz
SI                32768
SF          90.5547508 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                 1.40
```




```
NAME              JLB217
EXPNO                 10
PROCNO                 1
Date_           20110415
Time                10.33
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                322.5
DW               48.400 usec
DE                6.00 usec
TE               299.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1        500.1330885 MHz
SI                 65536
SF          500.1300400 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.00
```

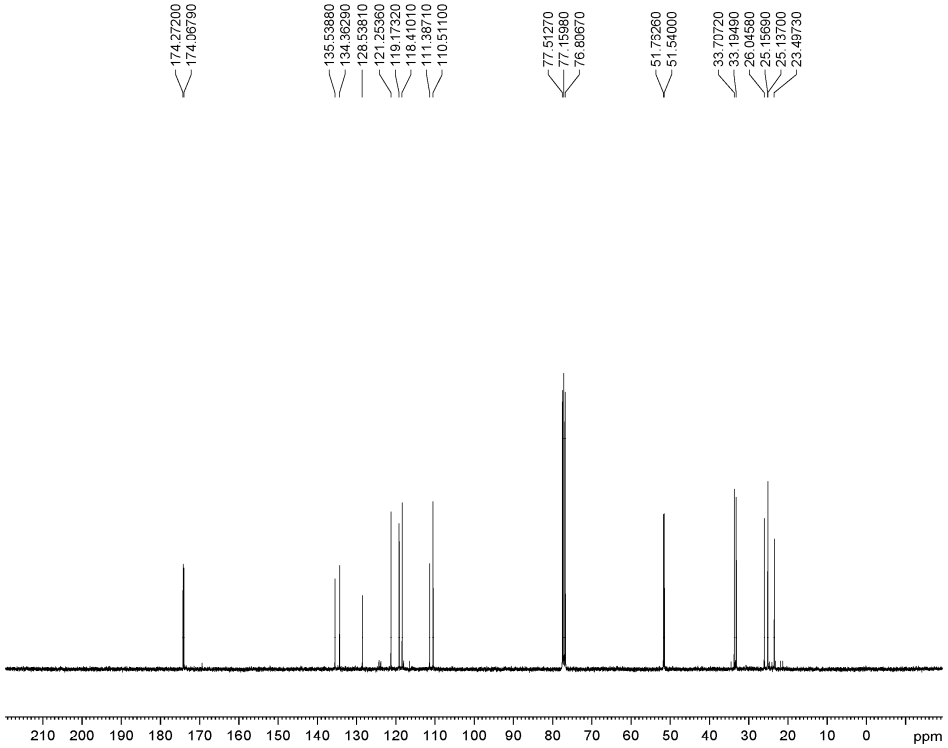



```
NAME              JLB217
EXPNO                 11
PROCNO                 1
Date_           20110415
Time                0.09
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES        0.330277 Hz
AQ            1.5139316 sec
RG                2298.8
DW               23.100 usec
DE                6.50 usec
TE               300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF          90.5547561 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

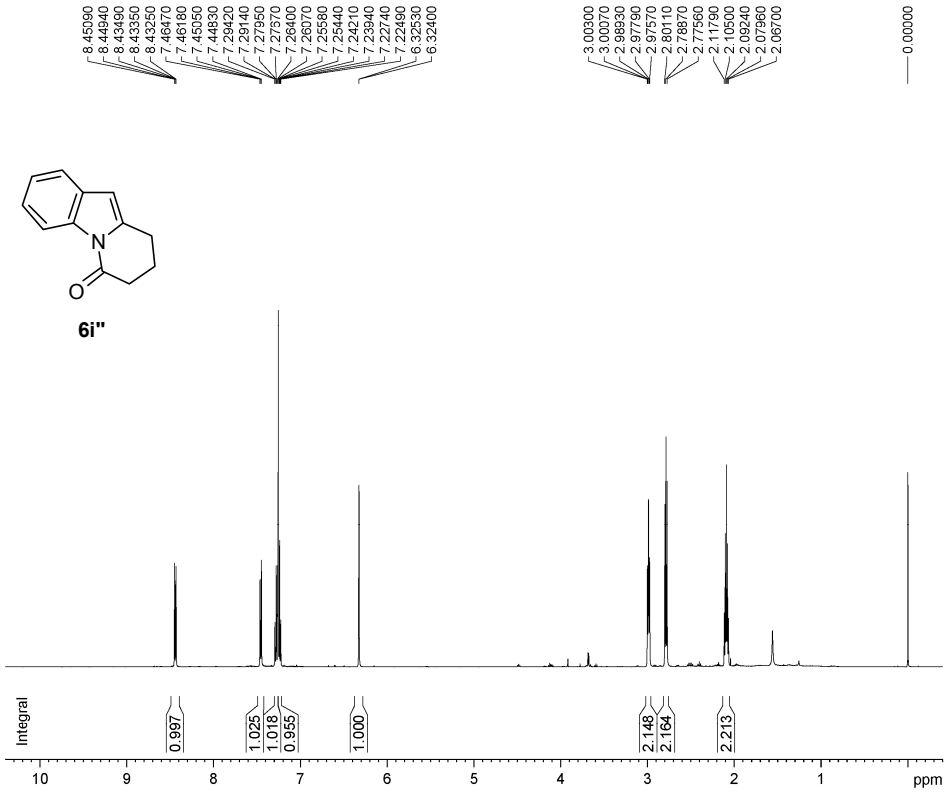


6i'

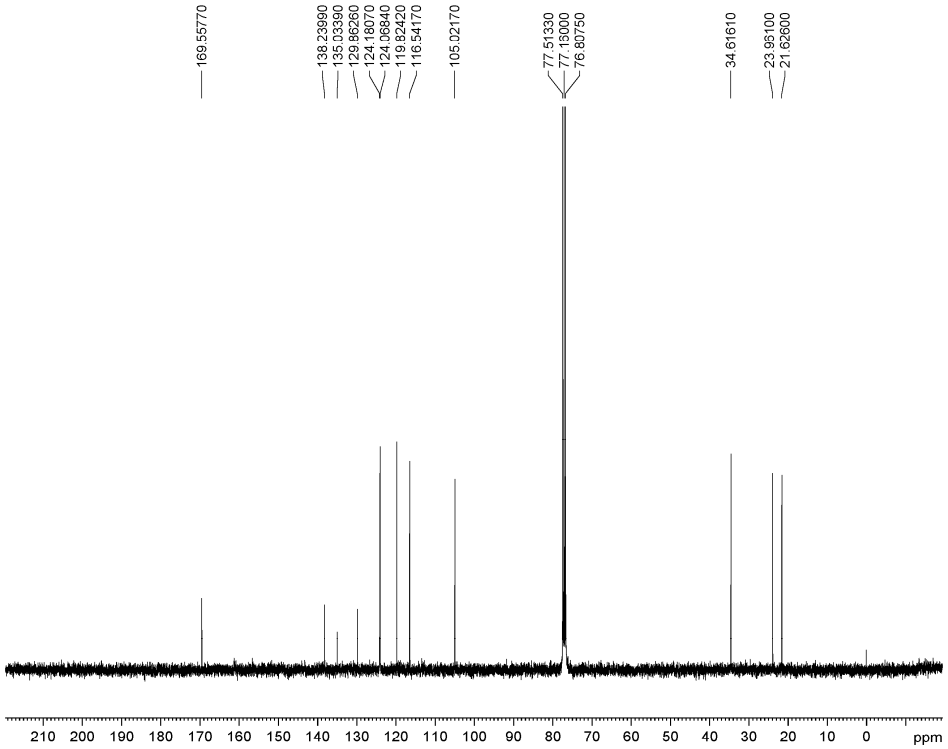
```
NAME          JLB234-2-Re
EXPNO                  10
PROCNO                  1
Date_           20110509
Time                16.58
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES            0.157632 Hz
AQ              3.1720407 sec
RG                   90.5
DW                 48.400 usec
DE                  6.00 usec
TE                 299.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W          12.18109322 W
SFO1         500.1330885 MHz
SI                  65536
SF           500.1300171 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



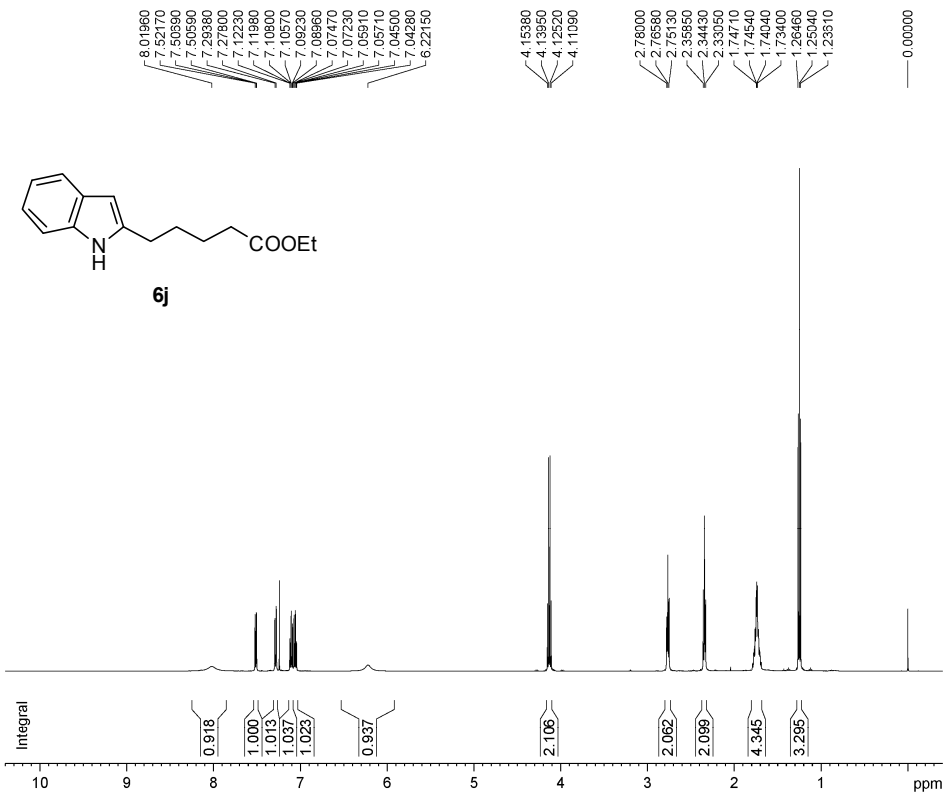

```
NAME           JLB234-2
EXPNO                11
PROCNO                1
Date_          20110506
Time                19.28
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH           21645.021 Hz
FIDRES            0.330277 Hz
AQ              1.5139316 sec
RG                 2896.3
DW                 23.100 usec
DE                  6.50 usec
TE                 297.5 K
D1              2.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              115.00 usec
PL2                  0.00 dB
PL12                23.37 dB
PL13                26.00 dB
SFO2         360.1314405 MHz
SI                  32768
SF            90.5547537 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```




```
NAME      JLB217-unknown-Re
EXPNO                   10
PROCNO                   1
Date_             20110516
Time                 0.35
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1720407 sec
RG                    181
DW                48.400 usec
DE                 6.00 usec
TE                 299.8 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                  65536
SF            500.1300156 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```




```
NAME      JLB217-unknown-Re
EXPNO                   11
PROCNO                   1
Date_             20110516
Time                19.23
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             21645.021 Hz
FIDRES          0.330277 Hz
AQ              1.5139316 sec
RG                 1625.5
DW                23.100 usec
DE                 6.50 usec
TE                 297.0 K
D1              2.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               115.00 usec
PL2                  0.00 dB
PL12                23.37 dB
PL13                26.00 dB
SFO2          360.1314405 MHz
SI                  32768
SF            90.5547485 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



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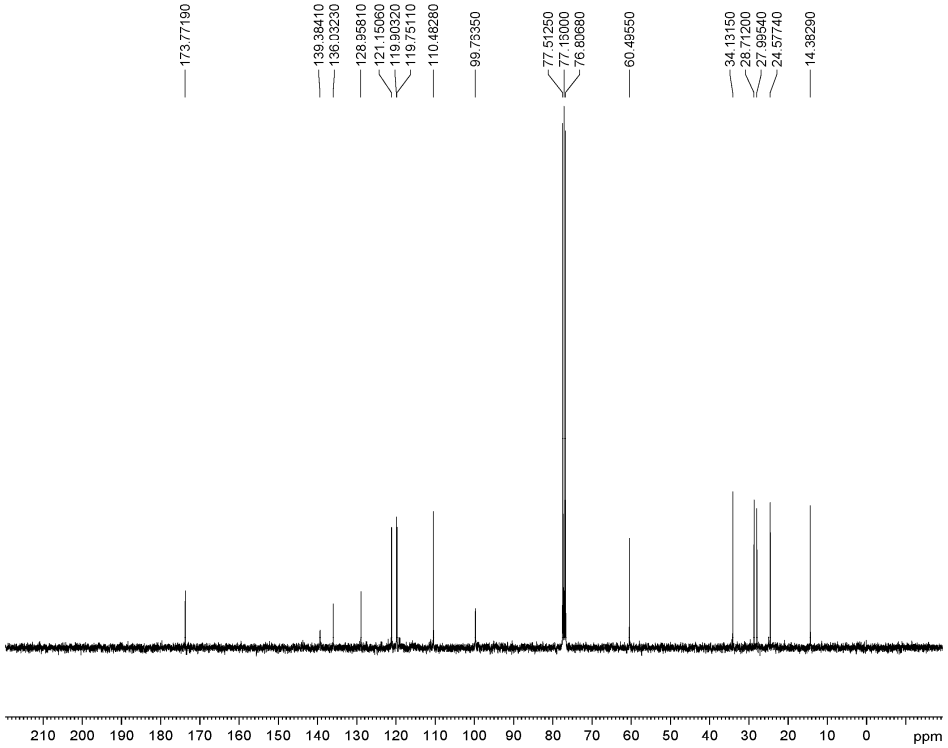
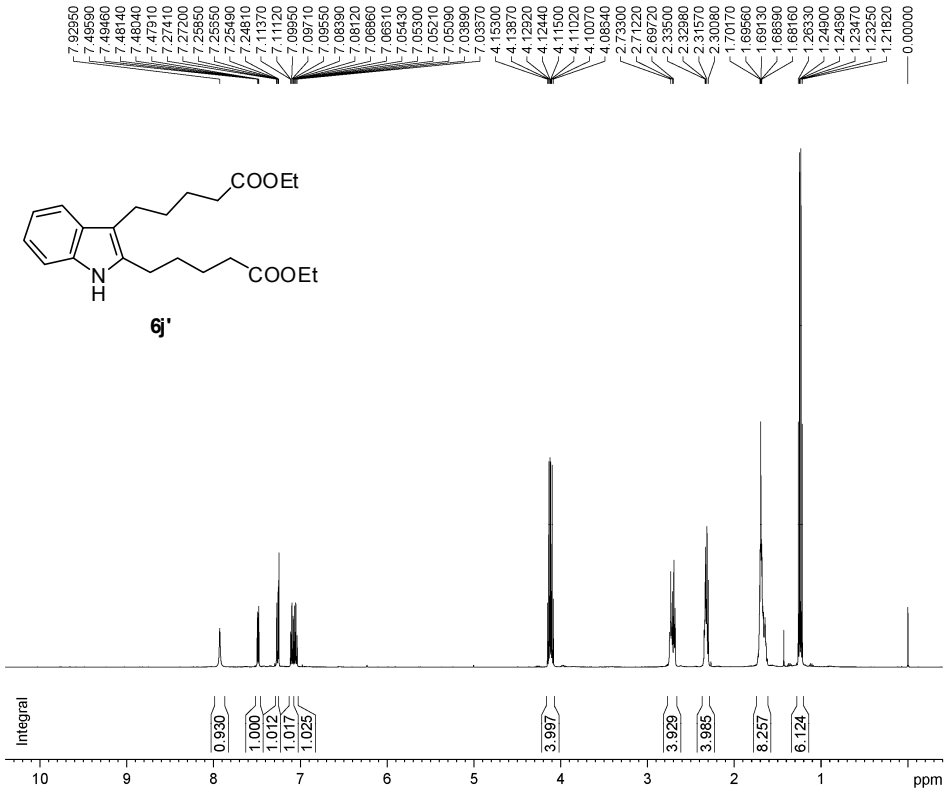
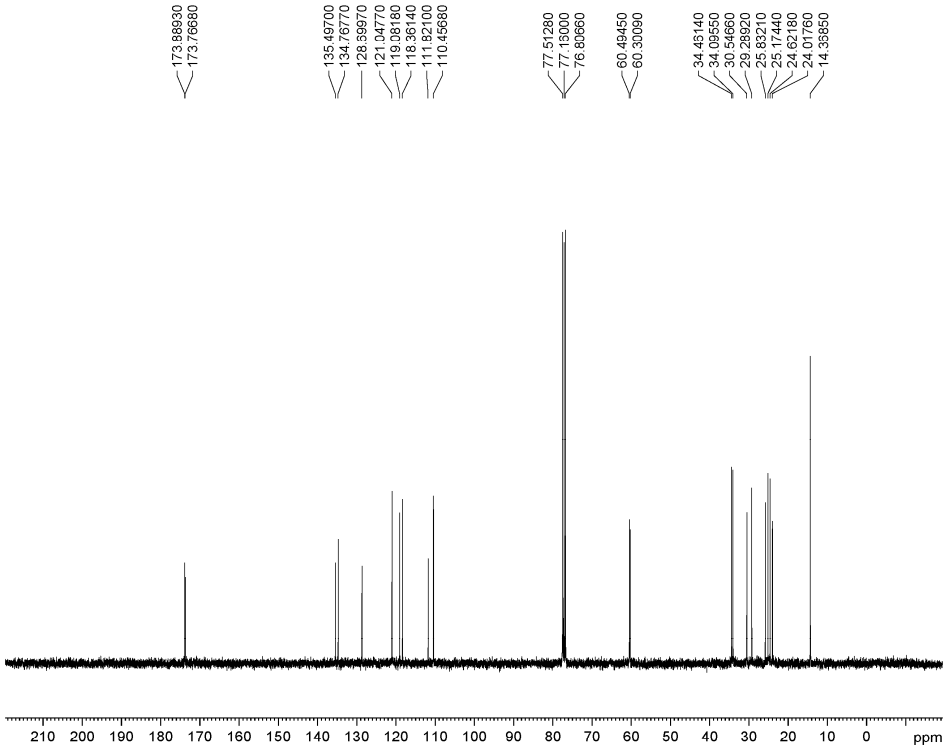
```
NAME          JLB236-1-Re
EXPNO                  10
PROCNO                  1
Date_           20110504
Time                12.47
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  90.5
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                  65536
SF           500.1300219 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



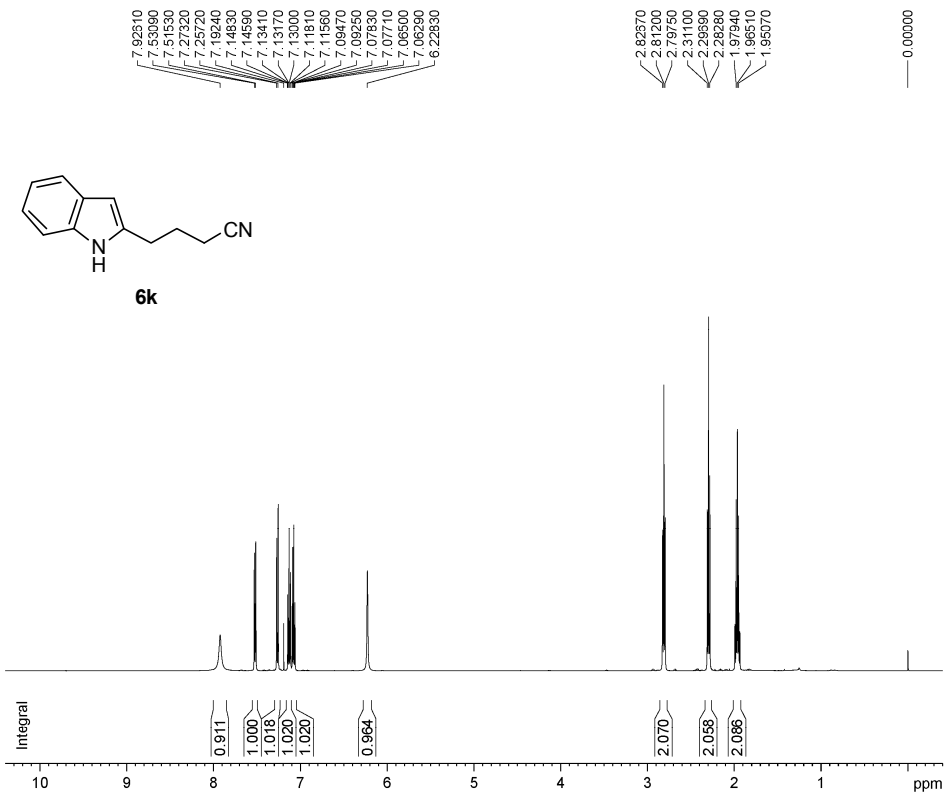

```
NAME          JLB219-1
EXPNO                 11
PROCNO                 1
Date_           20110416
Time                19.06
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                   2048
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              115.00 usec
PL2                  0.00 dB
PL12                23.37 dB
PL13                26.00 dB
SFO2         360.1314405 MHz
SI                  32768
SF            90.5547490 MHz
WDW                    EM
SSB                     0
LB                   1.50 Hz
GB                      0
PC                   1.40
```



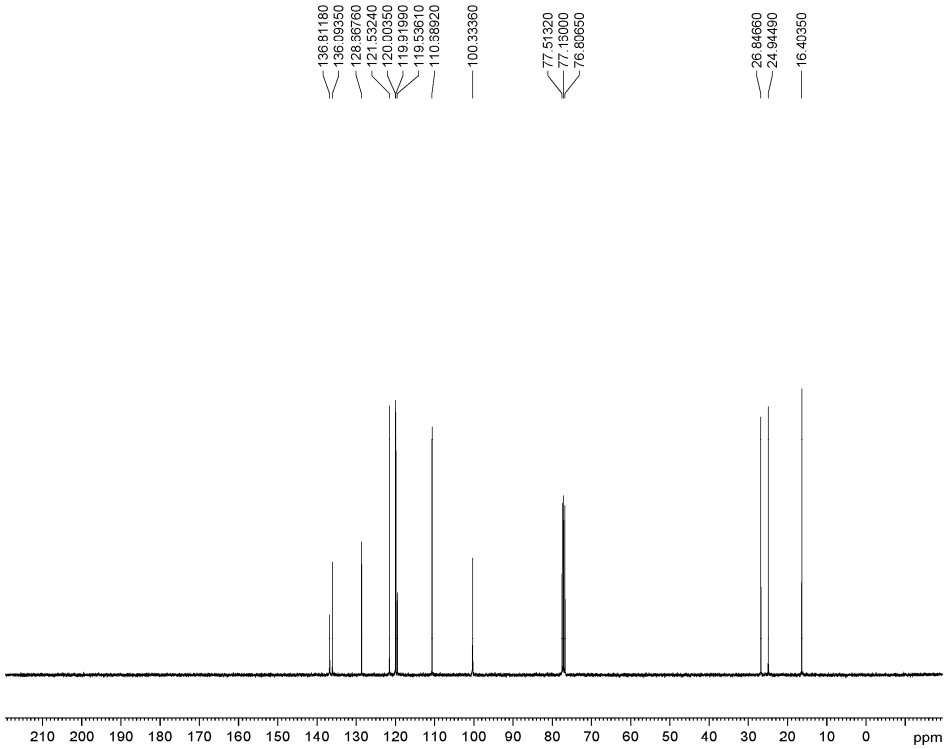

```
NAME            JLB219-2
EXPNO                 10
PROCNO                 1
Date_           20110415
Time                18.29
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                   181
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300193 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



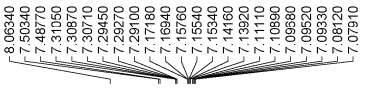

```
NAME            JLB219-2
EXPNO                 11
PROCNO                 1
Date_           20110418
Time                12.34
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                1625.5
DW                23.100 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF         90.5547494 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```

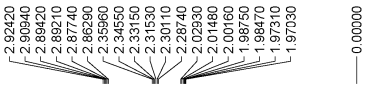


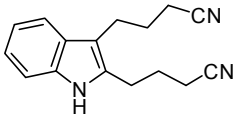

```
NAME              JLB214
EXPNO                 10
PROCNO                 1
Date_           20110412
Time                18.26
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            10330.578 Hz
FIDRES             0.157632 Hz
AQ              3.1720407 sec
RG                  35.9
DW                 48.400 usec
DE                  6.00 usec
TE                 299.9 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                 65536
SF            500.1300469 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```




```
NAME              JLB214
EXPNO                 11
PROCNO                 1
Date_           20110412
Time                17.22
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            21645.021 Hz
FIDRES             0.330277 Hz
AQ              1.5139316 sec
RG                2580.3
DW                 23.100 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2          360.1314405 MHz
SI                 32768
SF            90.5547615 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```







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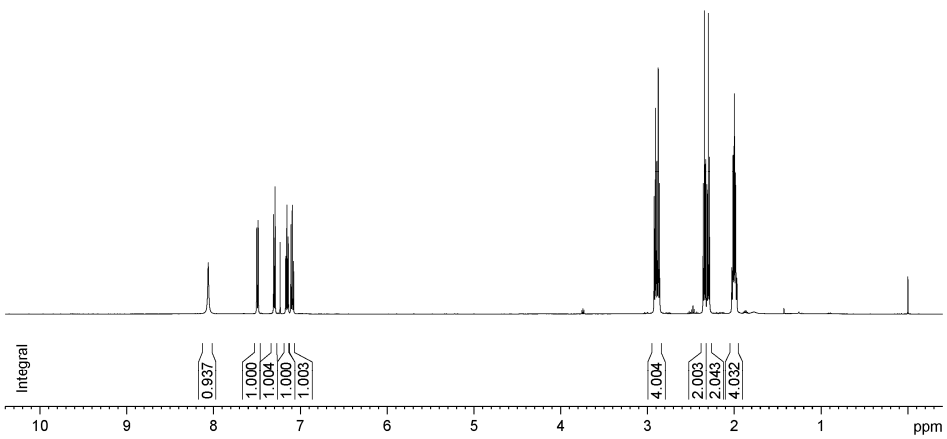
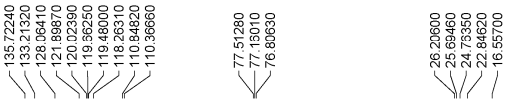
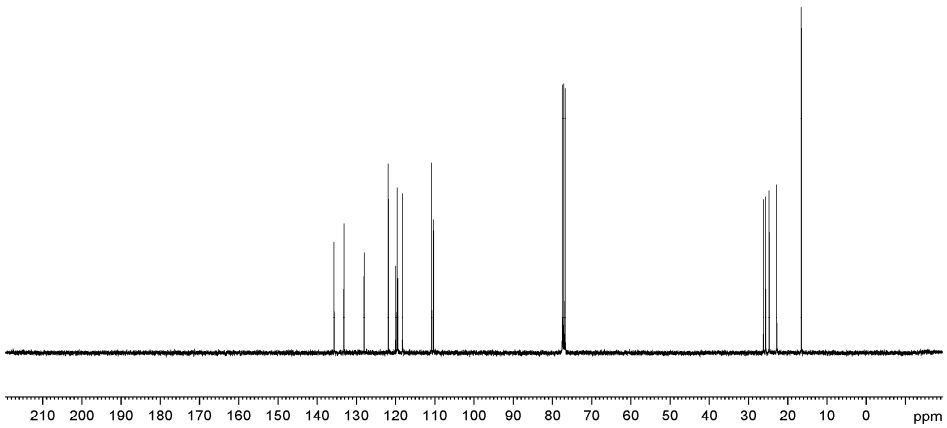
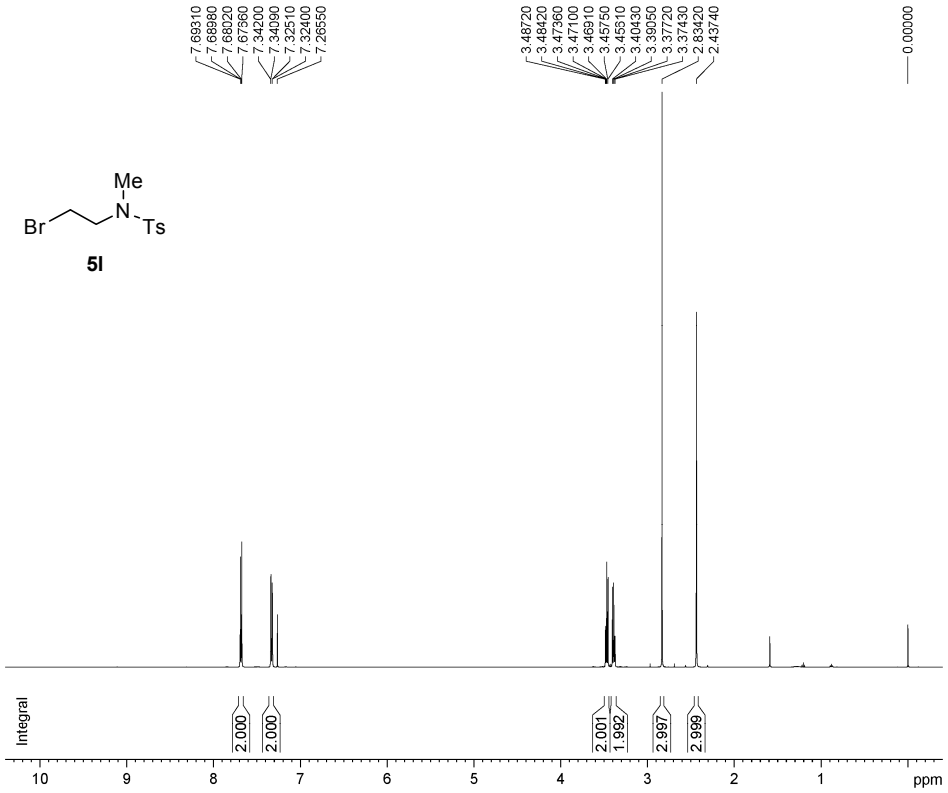
```
NAME            JLB237-2
EXPNO                 10
PROCNO                 1
Date_           20110506
Time                19.26
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300259 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



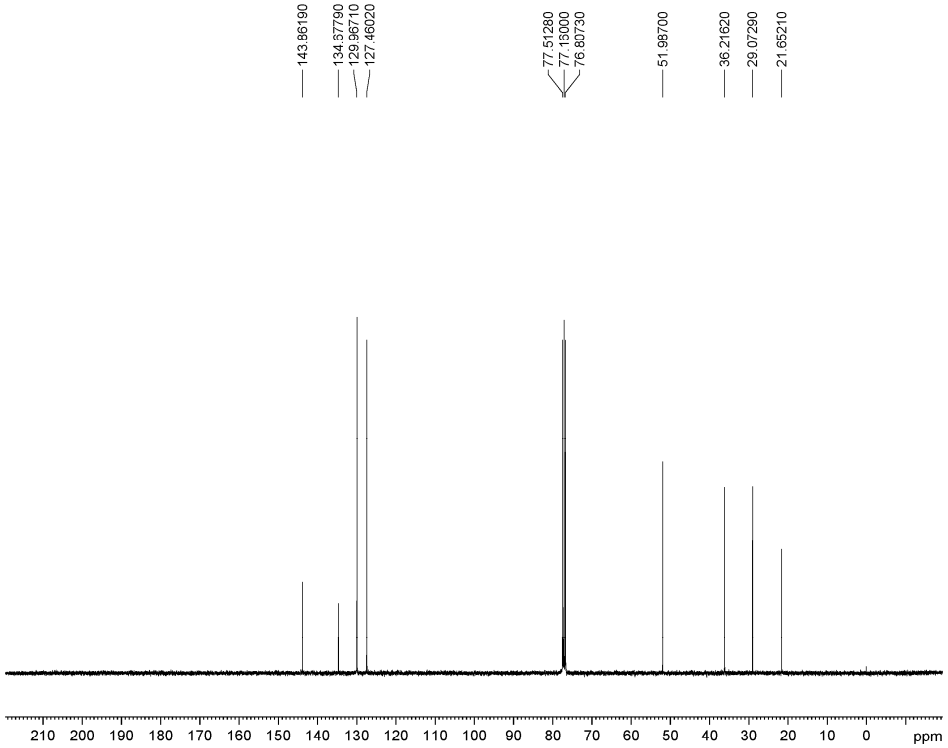



```
NAME            JLB237-2
EXPNO                 11
PROCNO                 1
Date_           20110505
Time                 9.12
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                3649.1
DW                23.100 usec
DE                 6.50 usec
TE                297.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2         360.1314405 MHz
SI                 32768
SF           90.5547575 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

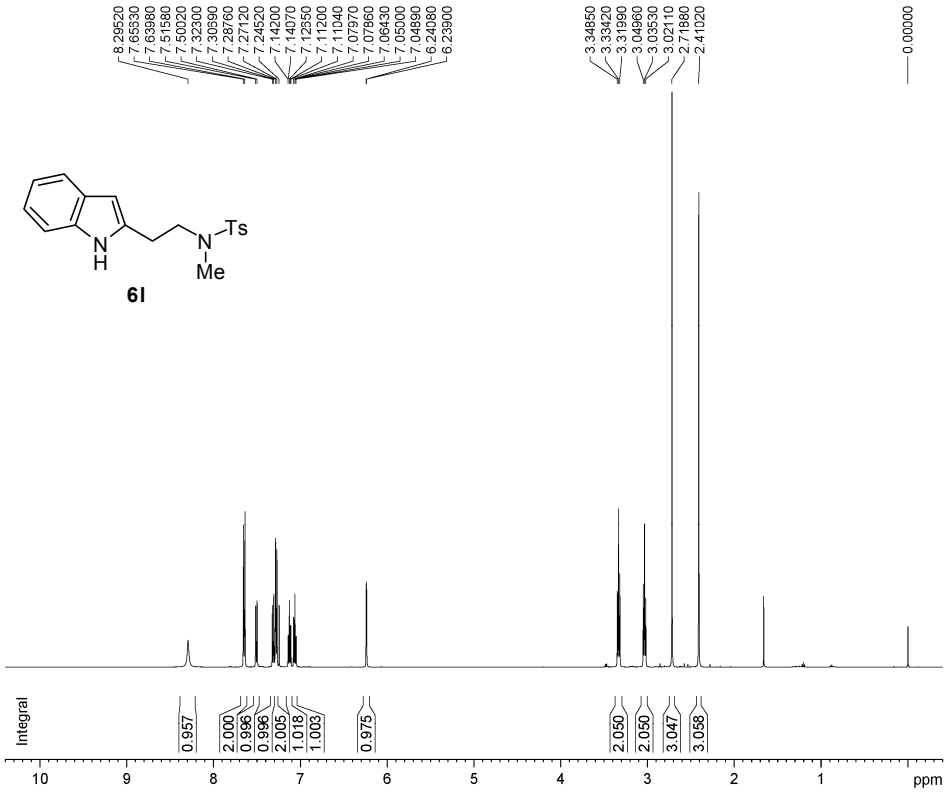





```
NAME              JLB245
EXPNO                 10
PROCNO                 1
Date_           20110513
Time               13.32
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                 574.7
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300109 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```




```
NAME              JLB245
EXPNO                 11
PROCNO                 1
Date_           20110514
Time               11.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                  2048
DW                23.100 usec
DE                 6.50 usec
TE                297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF         90.5547504 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



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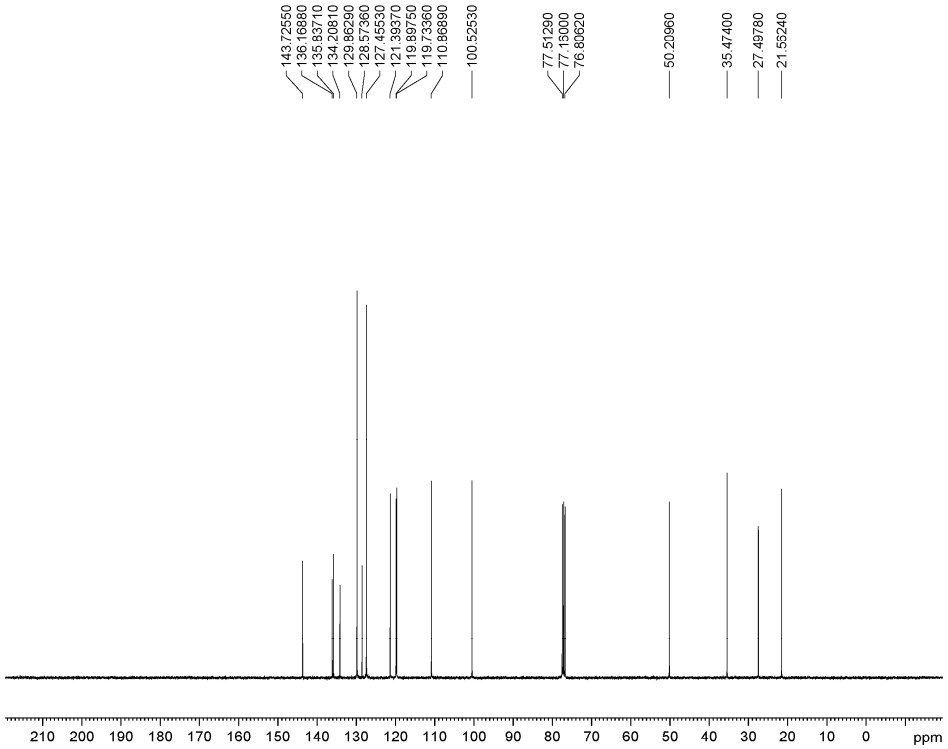
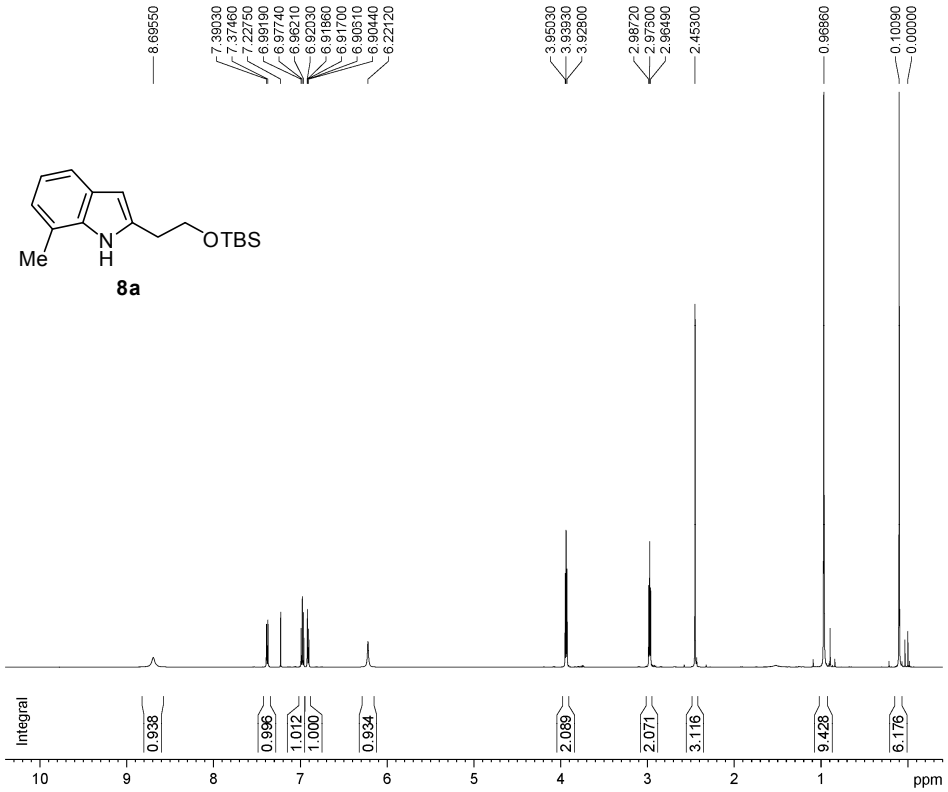
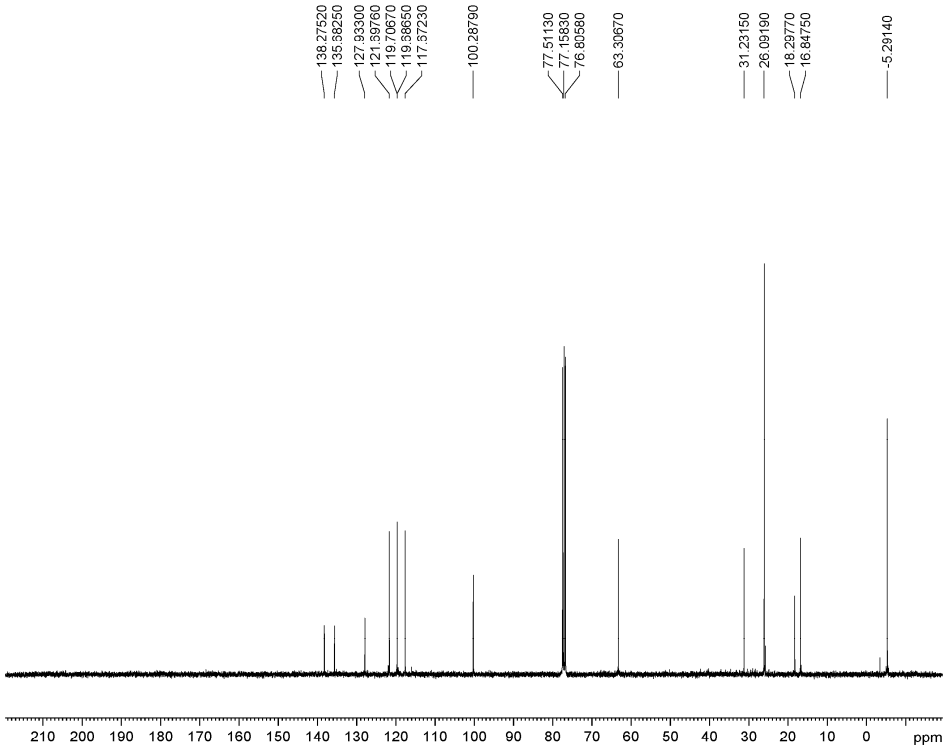
```
NAME                JLB249
EXPNO                   10
PROCNO                   1
Date_             20110520
Time                 18.36
INSTRUM              spect
PROBHD   5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1720407 sec
RG                   90.5
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                  65536
SF            500.1300210 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



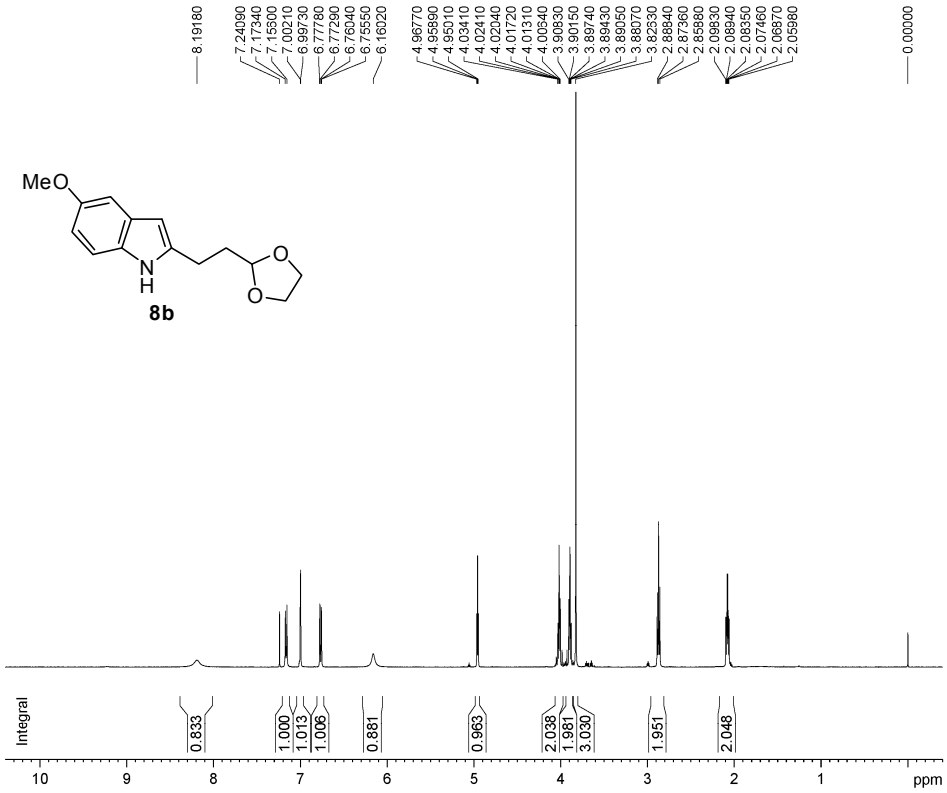

```
NAME                JLB249
EXPNO                   11
PROCNO                   1
Date_             20110522
Time                  0.55
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    768
DS                      4
SWH             21645.021 Hz
FIDRES          0.330277 Hz
AQ              1.5139316 sec
RG                   4096
DW                23.100 usec
DE                 6.50 usec
TE                297.9 K
D1             2.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              115.00 usec
PL2                  0.00 dB
PL12                23.37 dB
PL13                26.00 dB
SFO2          360.1314405 MHz
SI                  32768
SF            90.5547594 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



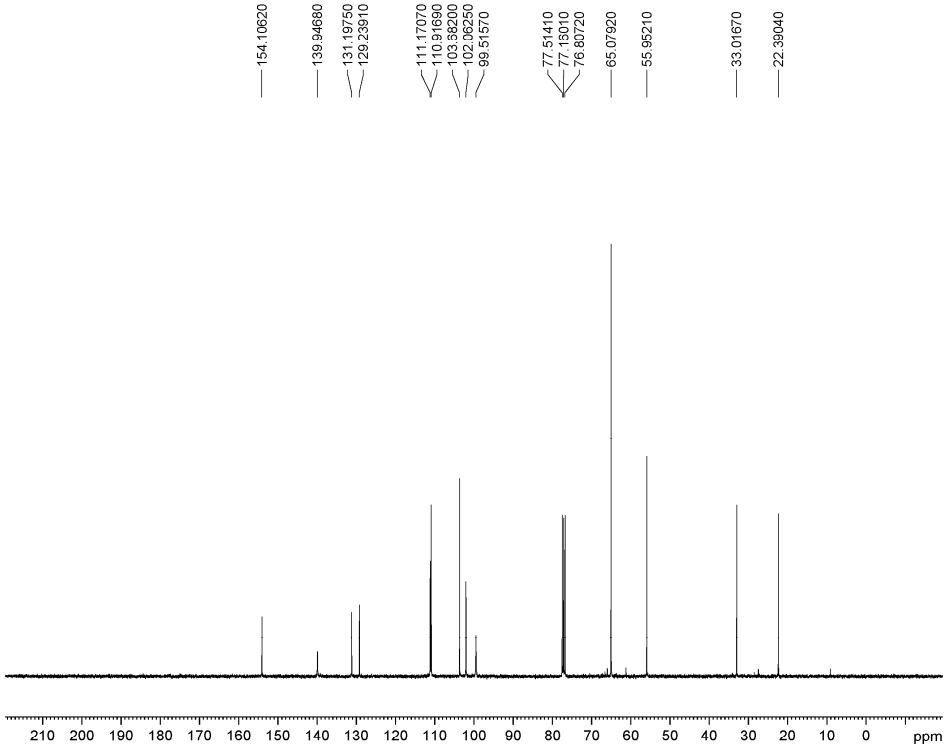

```
NAME            JLB235-Re
EXPNO                  10
PROCNO                  1
Date_           20110508
Time                15.10
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300296 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



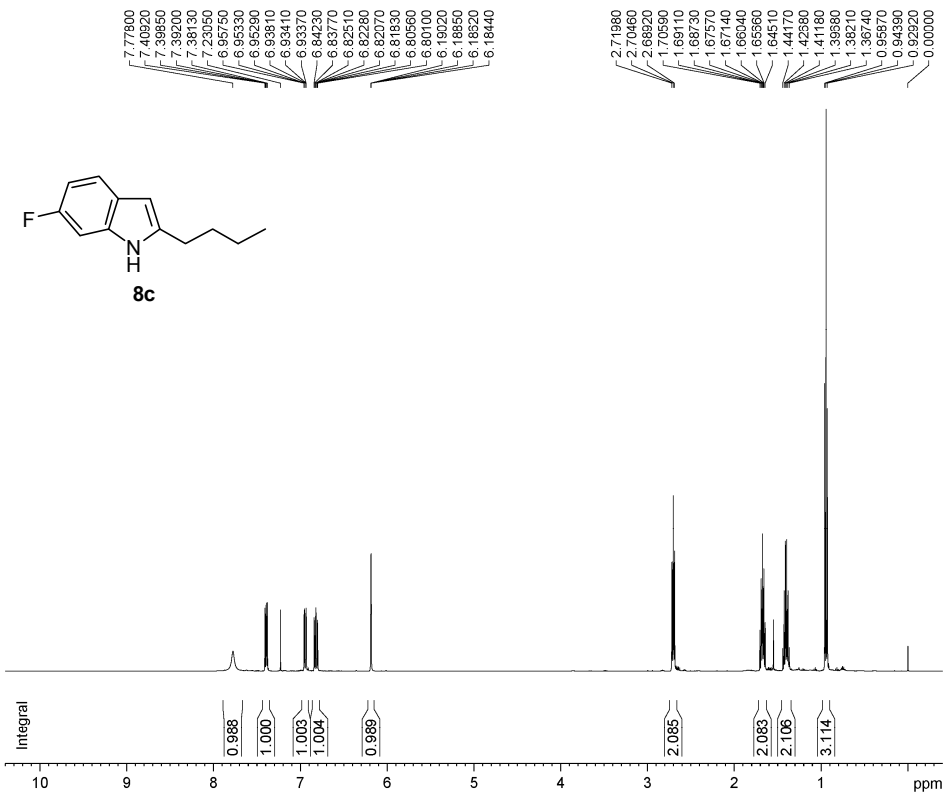

```
NAME              JLB235
EXPNO                 21
PROCNO                 1
Date_          20110504
Time               18.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  768
DS                    4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                 2048
DW                23.100 usec
DE                 6.50 usec
TE                296.9 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547513 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



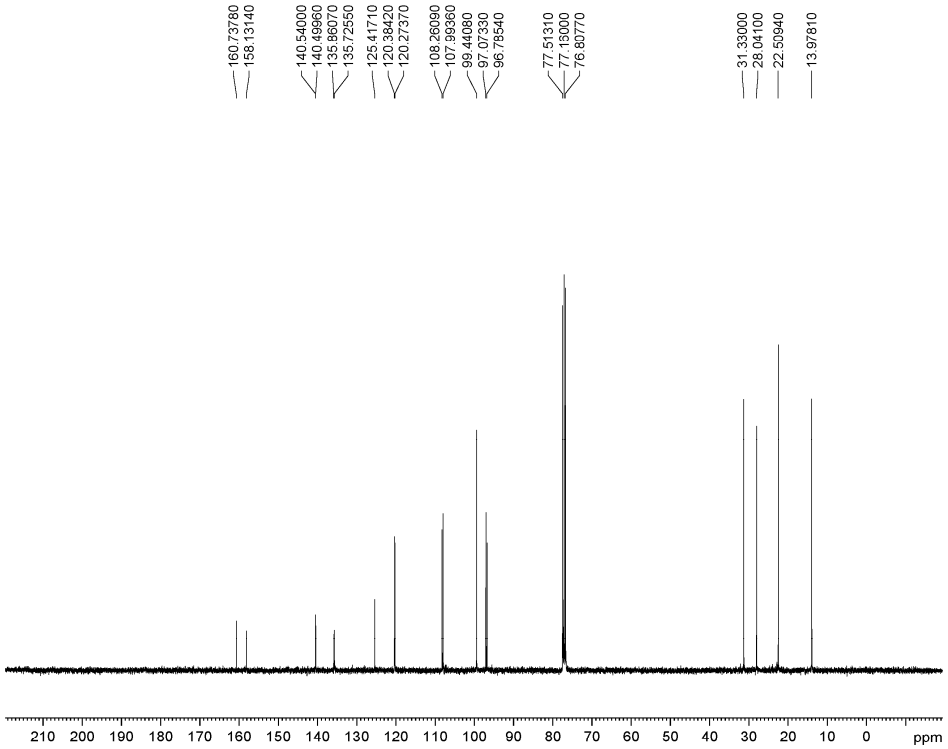

```
NAME            JLB238-Re
EXPNO                  10
PROCNO                  1
Date_            20110508
Time                17.21
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                1290.2
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                  65536
SF            500.1300228 MHz
WDW                    EM
SSB                     0
LB                   0.20 Hz
GB                      0
PC                   1.00
```



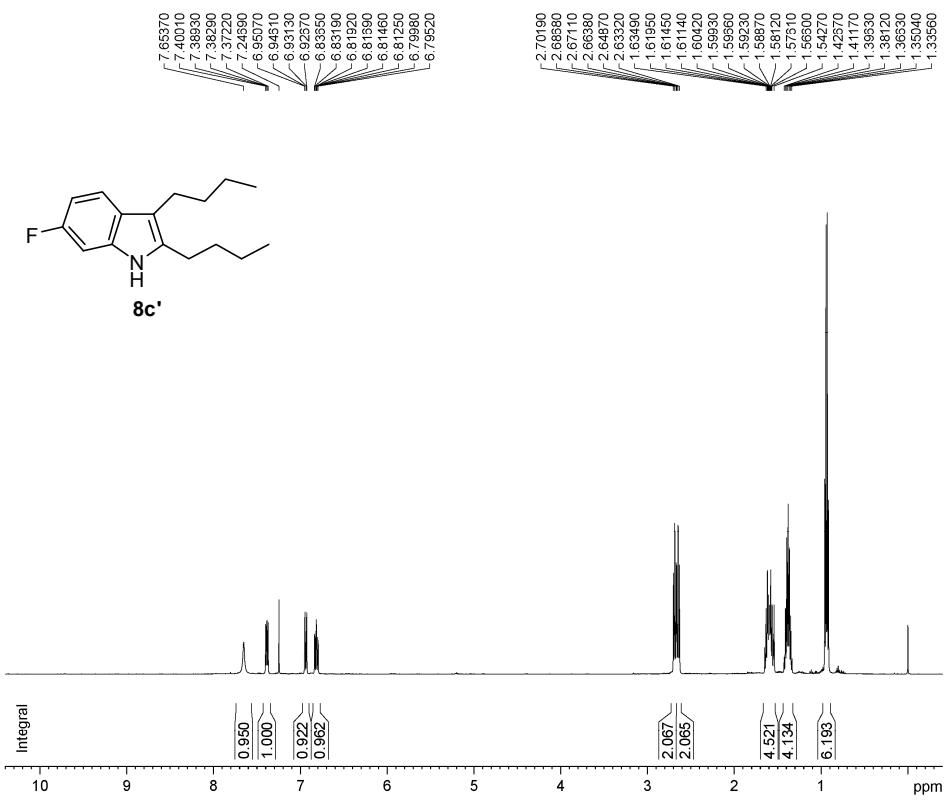

```
NAME              JLB238
EXPNO                 11
PROCNO                 1
Date_          20110506
Time               7.26
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH          21645.021 Hz
FIDRES        0.330277 Hz
AQ            1.5139316 sec
RG               4597.6
DW               23.100 usec
DE                6.50 usec
TE               297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547592 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



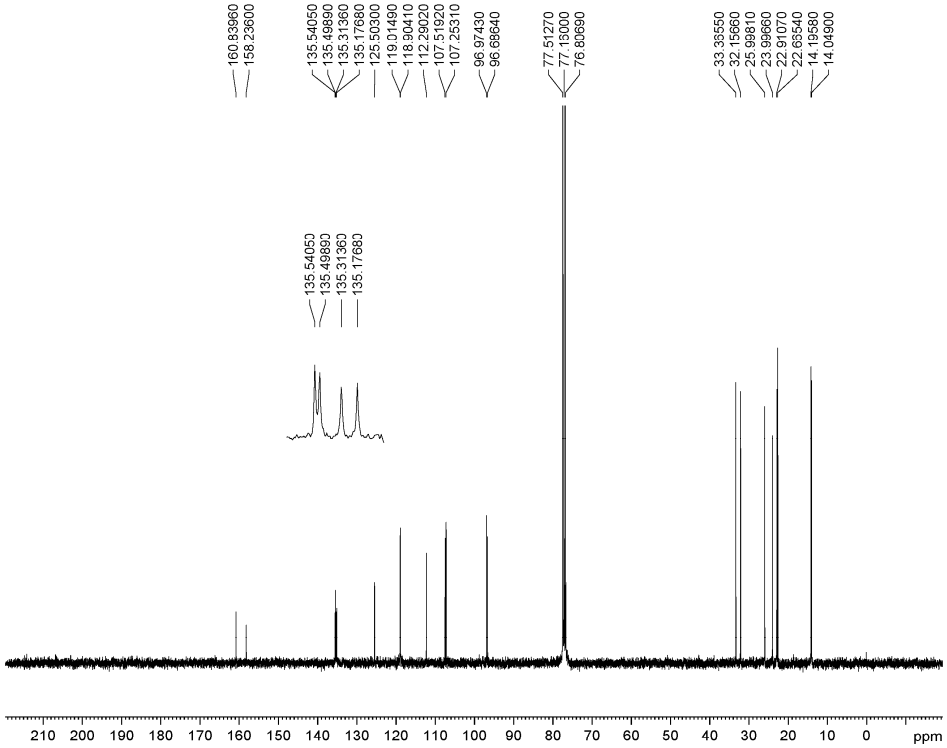

```
NAME            JLB250-2
EXPNO                 10
PROCNO                 1
Date_           20110520
Time                14.26
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            10330.578 Hz
FIDRES             0.157632 Hz
AQ              3.1720407 sec
RG                    57
DW                 48.400 usec
DE                  6.00 usec
TE                 299.9 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                 65536
SF            500.1300281 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



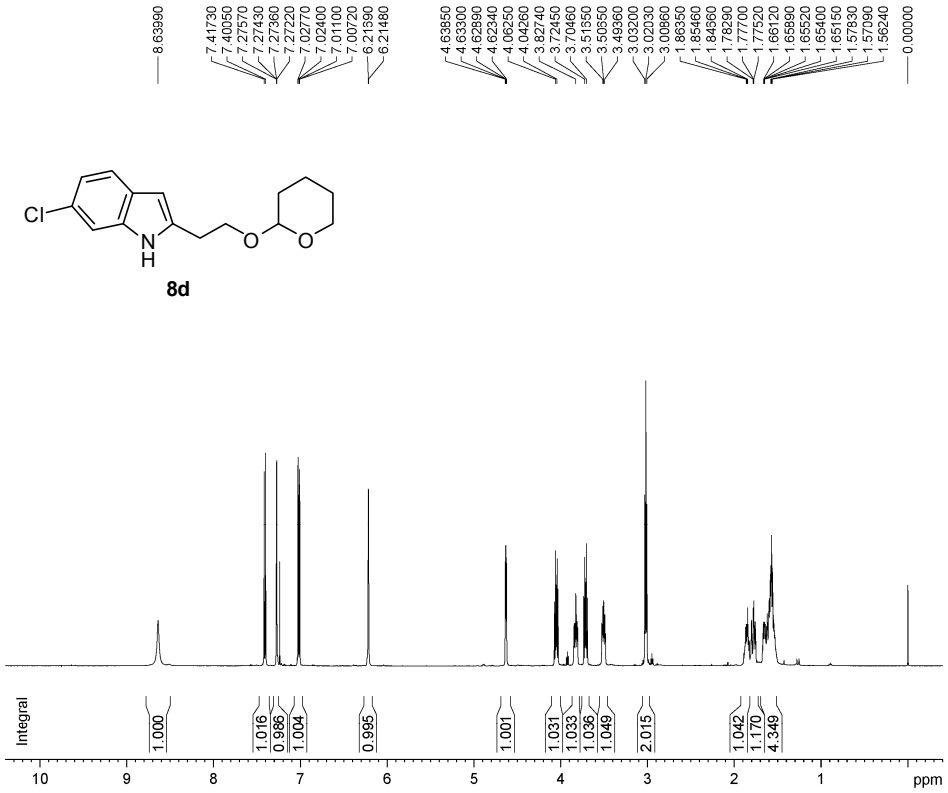

```
NAME            JLB250-2
EXPNO                 11
PROCNO                 1
Date_           20110520
Time                10.40
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH            21645.021 Hz
FIDRES             0.330277 Hz
AQ              1.5139316 sec
RG                 3649.1
DW                 23.100 usec
DE                  6.50 usec
TE                 297.7 K
D1              2.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2         360.1314405 MHz
SI                 32768
SF            90.5547504 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



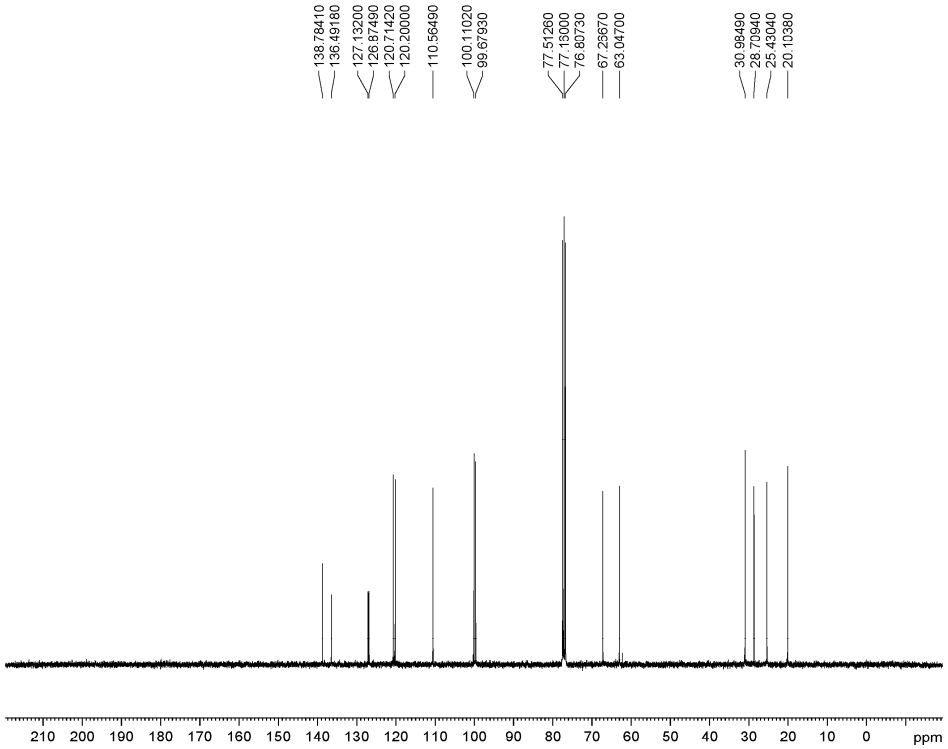

```
NAME          JLB250-1-Re
EXPNO                  10
PROCNO                  1
Date_           20110522
Time                22.53
INSTRUM             spect
PROBHD    5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  71.8
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                  65536
SF            500.1300197 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



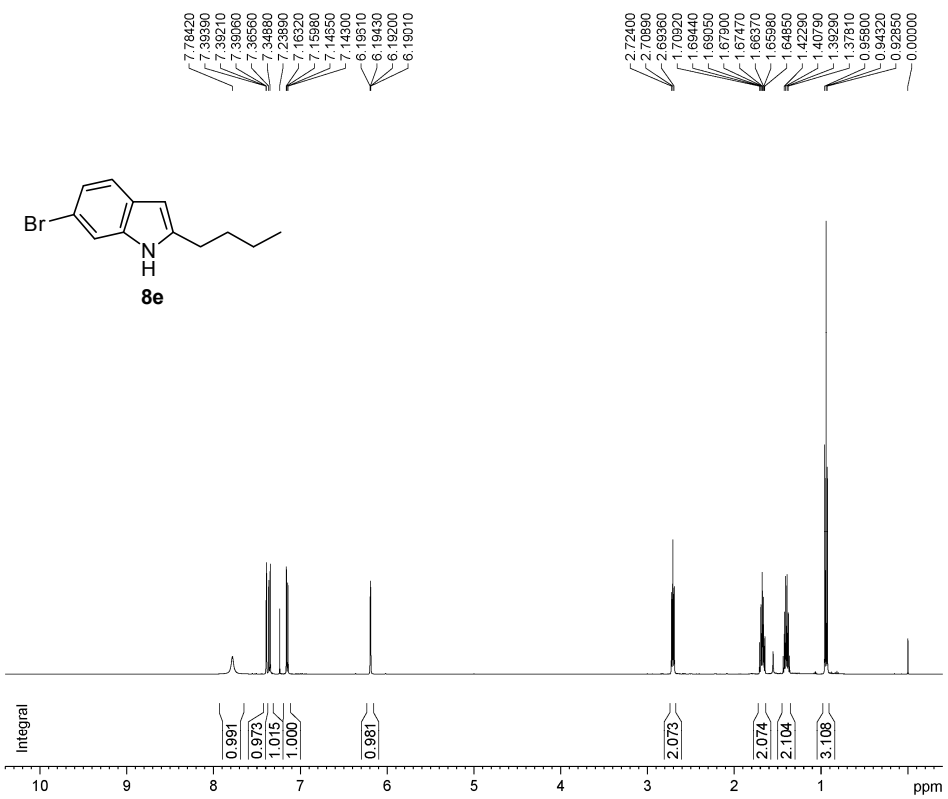

```
NAME          JLB250-1-Re
EXPNO                  11
PROCNO                  1
Date_           20110522
Time                16.54
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1536
DS                      4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                  4096
DW                23.100 usec
DE                 6.50 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              115.00 usec
PL2                  0.00 dB
PL12                23.37 dB
PL13                26.00 dB
SFO2         360.1314405 MHz
SI                  32768
SF            90.5547474 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



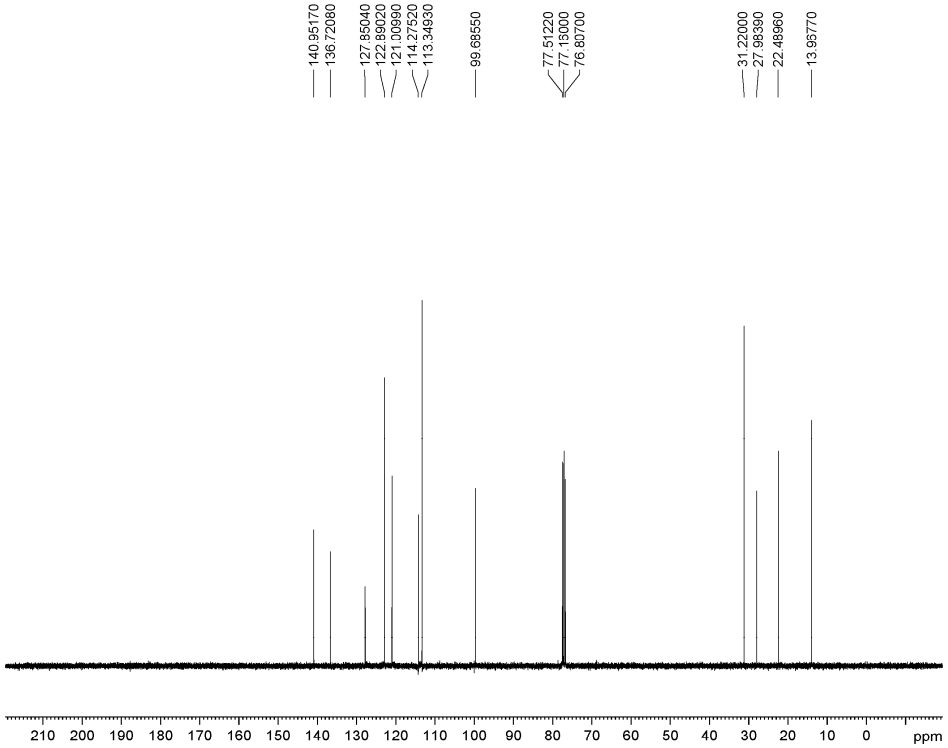

```
NAME              JLB239
EXPNO                 10
PROCNO                 1
Date_           20110506
Time                14.35
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300244 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```



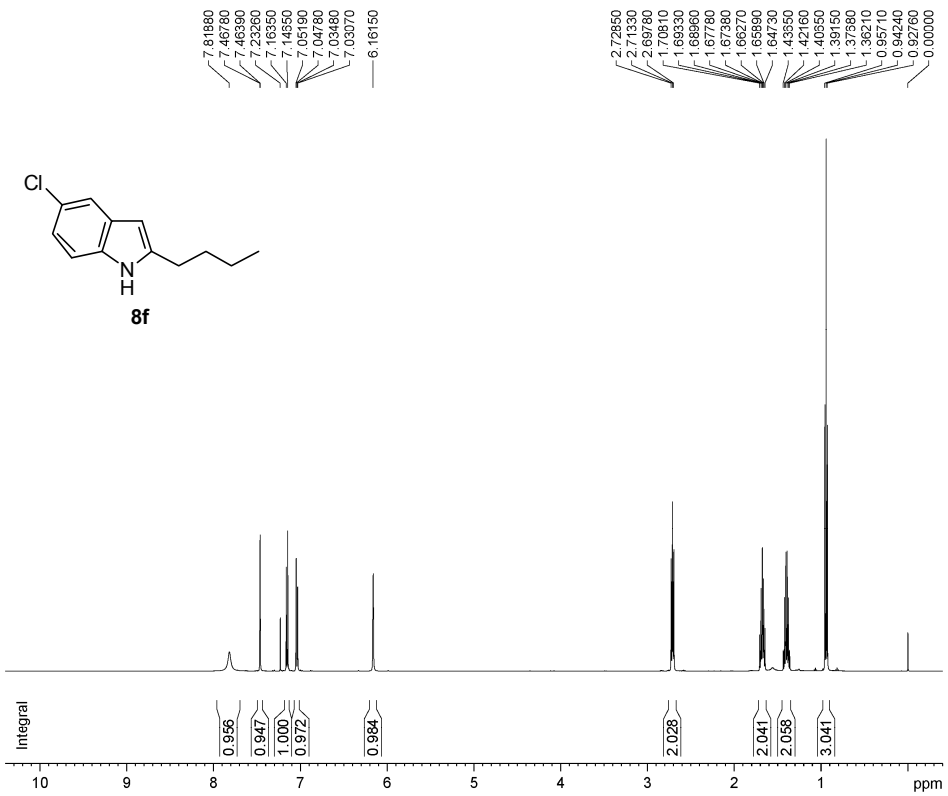

```
NAME          JLB239-Re
EXPNO                 10
PROCNO                 1
Date_           20110508
Time                19.23
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                2298.8
DW                23.100 usec
DE                 6.50 usec
TE                298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2      360.1314405 MHz
SI                 32768
SF         90.5547504 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```



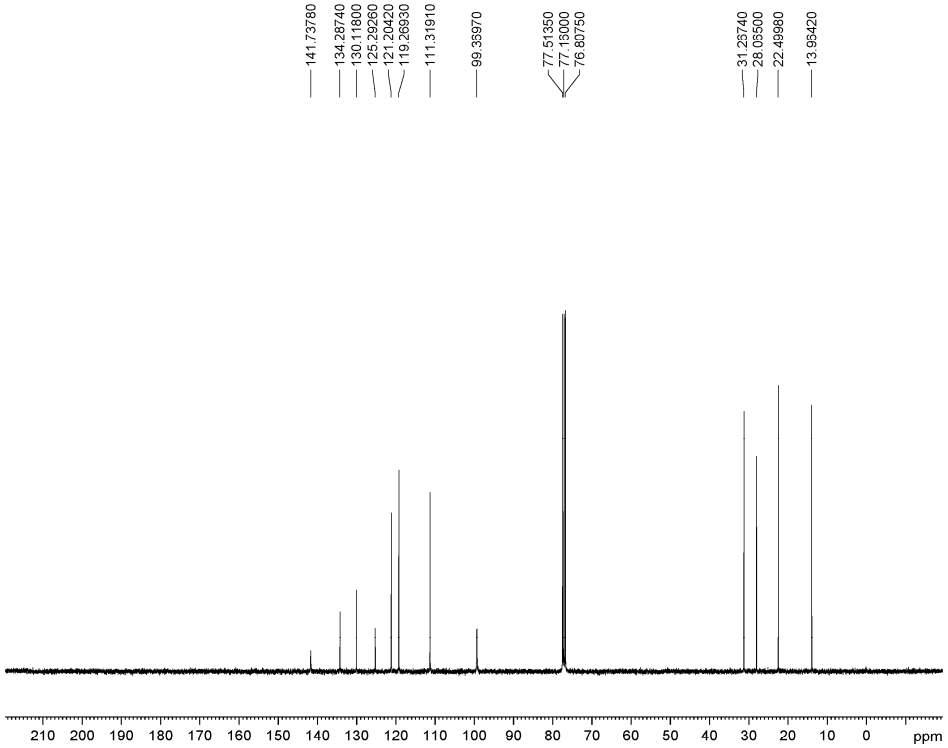

```
NAME              JLB253
EXPNO                 10
PROCNO                 1
Date_           20110526
Time               14.40
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  71.8
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300238 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



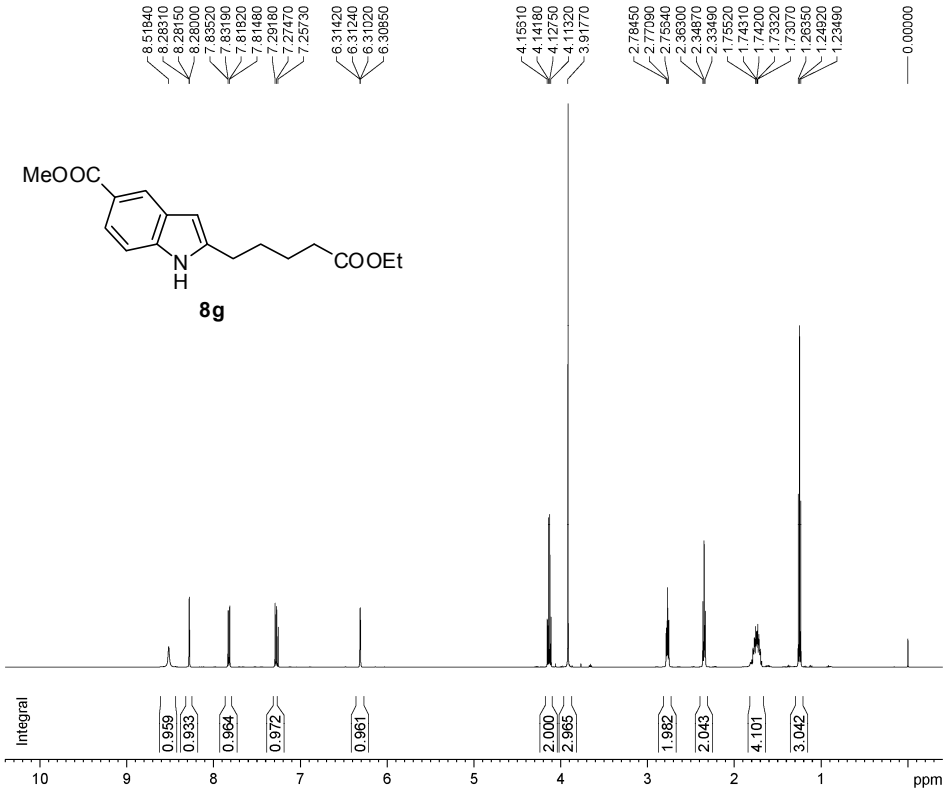

```
NAME              JLB253
EXPNO                 11
PROCNO                 1
Date_           20110525
Time               15.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                2896.3
DW                23.100 usec
DE                 6.50 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547529 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```



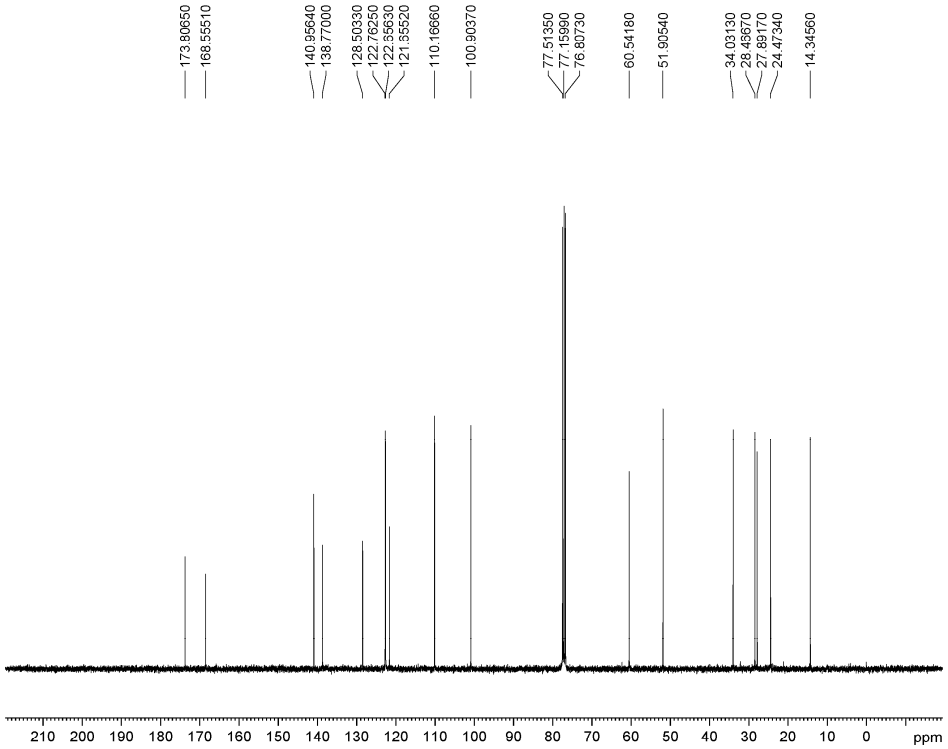

```
NAME              JLB247
EXPNO                 20
PROCNO                 1
Date_           20110517
Time               18.44
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  90.5
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300270 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```



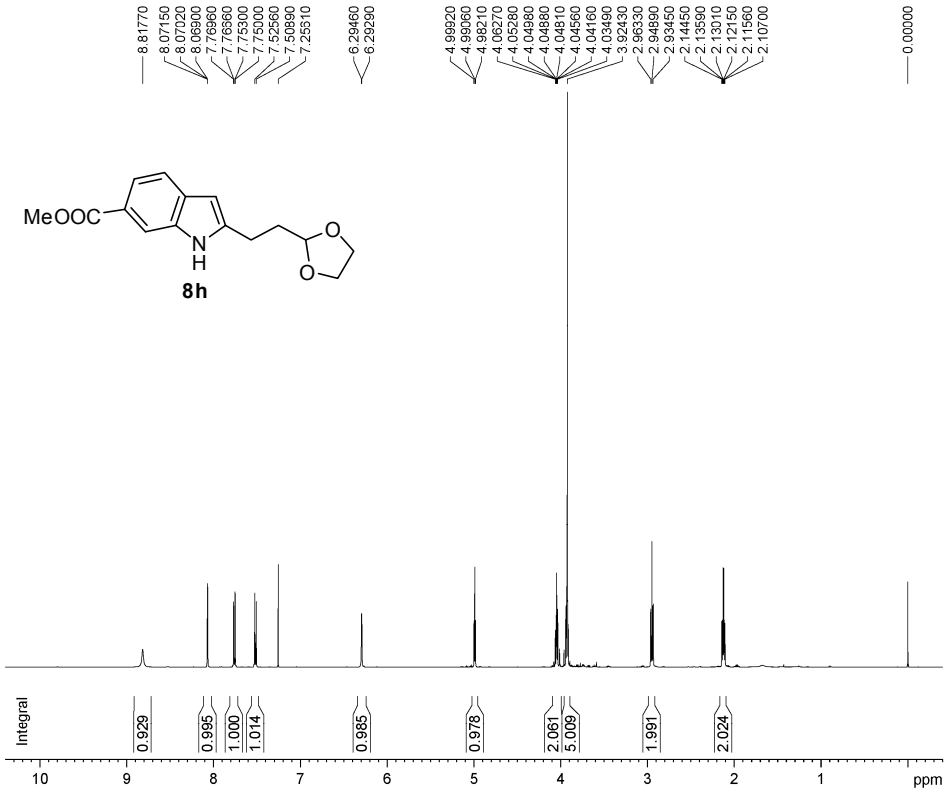

```
NAME              JLB247
EXPNO                 11
PROCNO                 1
Date_           20110518
Time               19.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                3649.1
DW                23.100 usec
DE                 6.50 usec
TE                297.8 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF          90.5547513 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



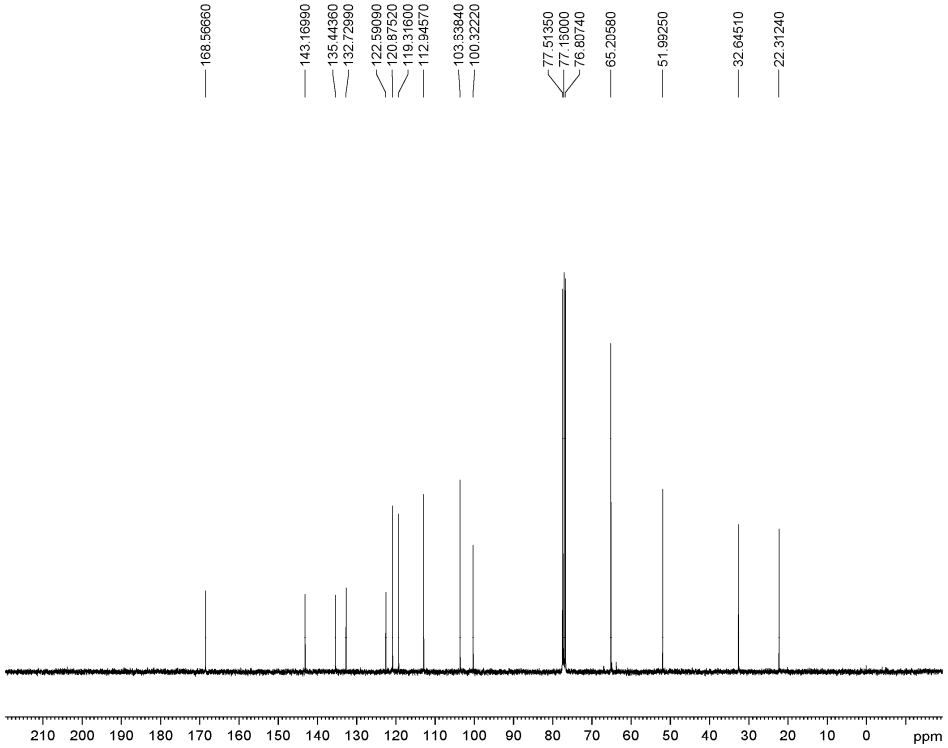

```
NAME            JLB254-1
EXPNO                10
PROCNO                1
Date_          20110527
Time              10.12
INSTRUM           spect
PROBHD   5 mm SEI 1H-13
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                 45.3
DW               48.400 usec
DE                6.00 usec
TE               299.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                65536
SF         500.1300145 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



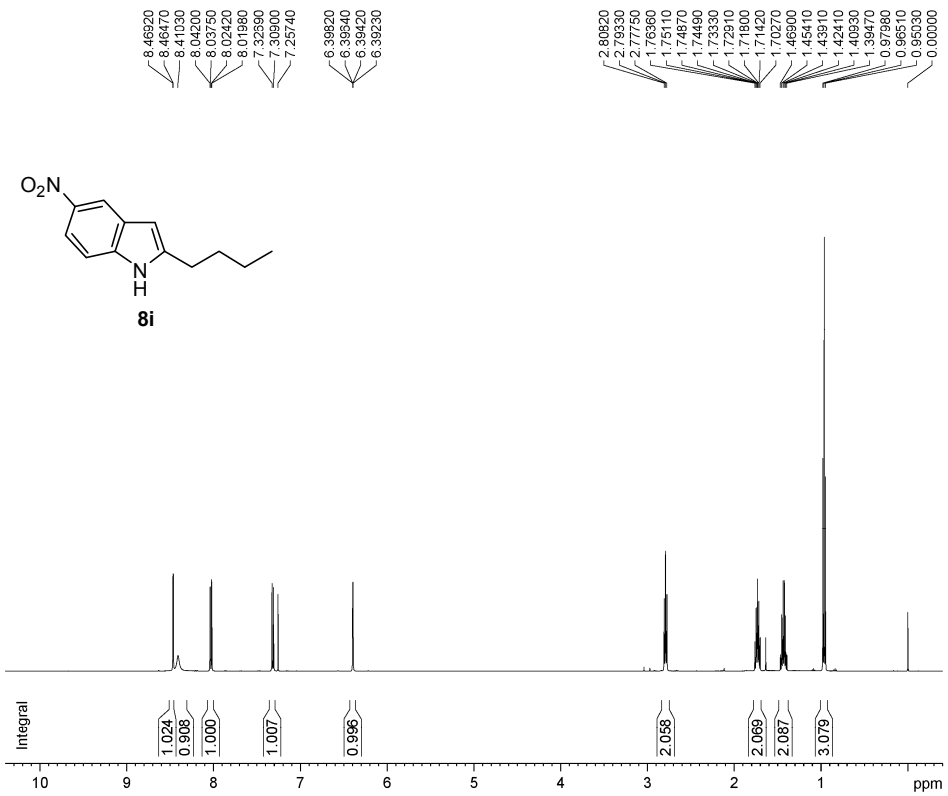

```
NAME            JLB254-1
EXPNO                11
PROCNO                1
Date_          20110526
Time              15.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  768
DS                    4
SWH           21645.021 Hz
FIDRES        0.330277 Hz
AQ            1.5139316 sec
RG               5160.6
DW               23.100 usec
DE                6.50 usec
TE               299.0 K
D1           2.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                32768
SF          90.5547511 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



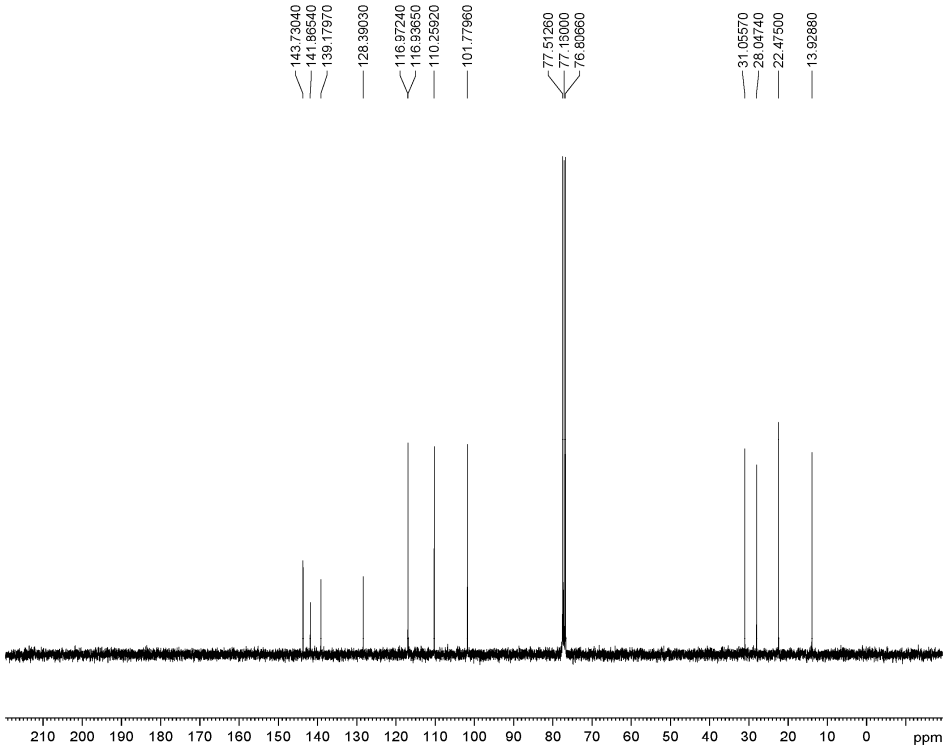

```
NAME              JLB248
EXPNO                 10
PROCNO                 1
Date_           20110518
Time                16.57
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1720407 sec
RG                161.3
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W          12.18109322 W
SFO1          500.1330885 MHz
SI                 65536
SF            500.1300152 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```



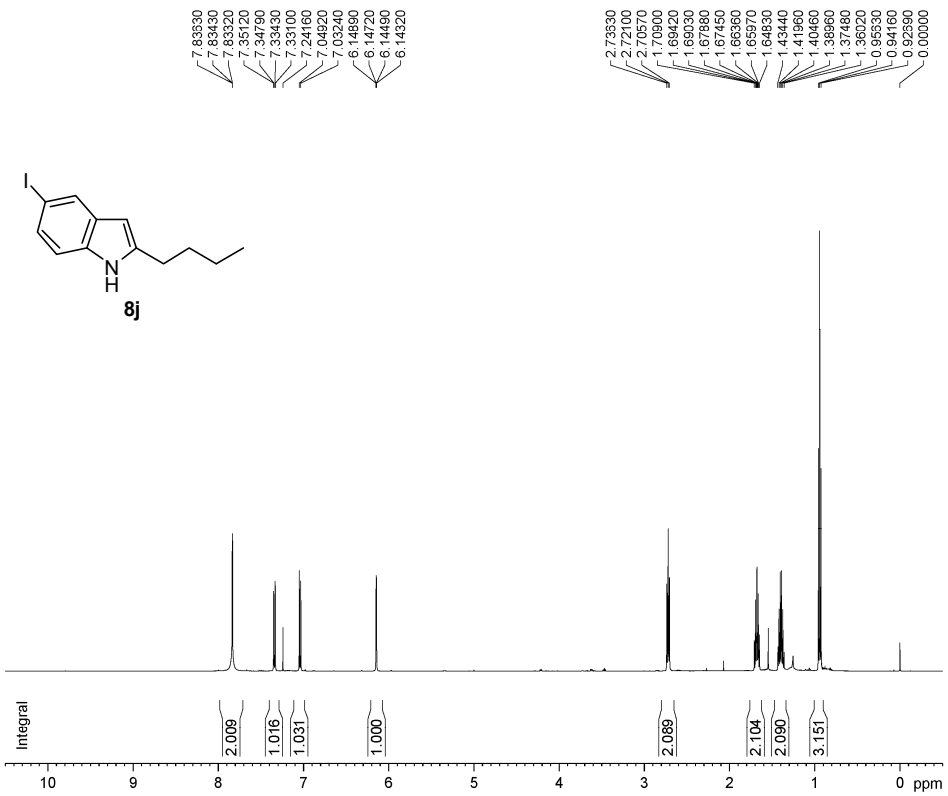

```
NAME              JLB248
EXPNO                 11
PROCNO                 1
Date_           20110519
Time                12.33
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH            21645.021 Hz
FIDRES          0.330277 Hz
AQ              1.5139316 sec
RG                2580.3
DW                23.100 usec
DE                 6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1          90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2         360.1314405 MHz
SI                 32768
SF            90.5547512 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



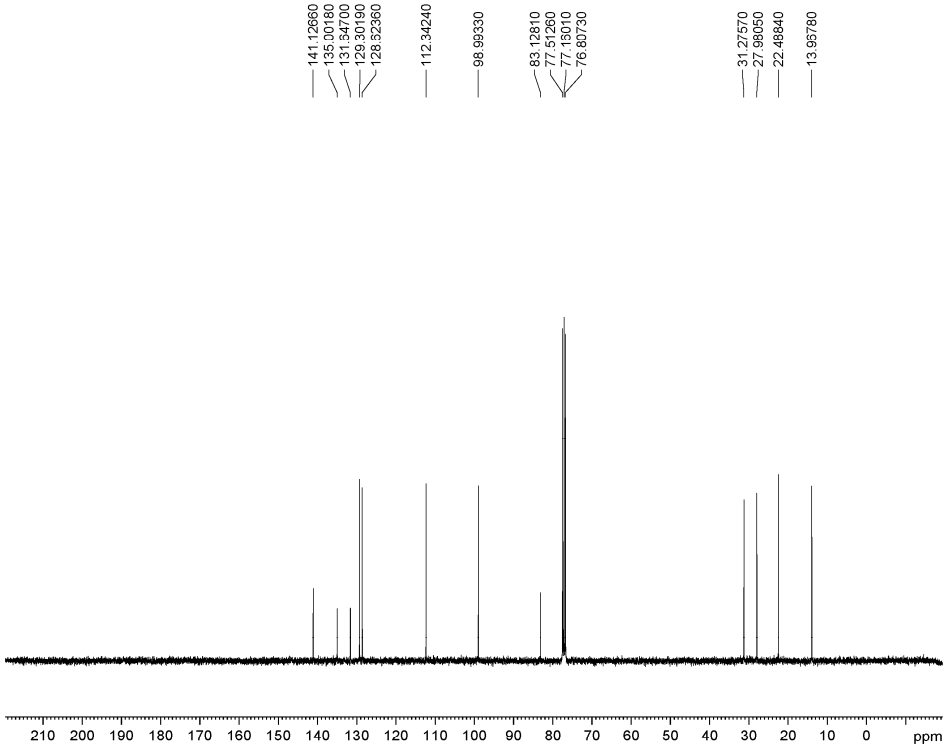

```
NAME              JLB252
EXPNO                 10
PROCNO                 1
Date_           20110525
Time                1.47
INSTRUM            spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                   114
DW                48.400 usec
DE                 6.00 usec
TE                299.8 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300146 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



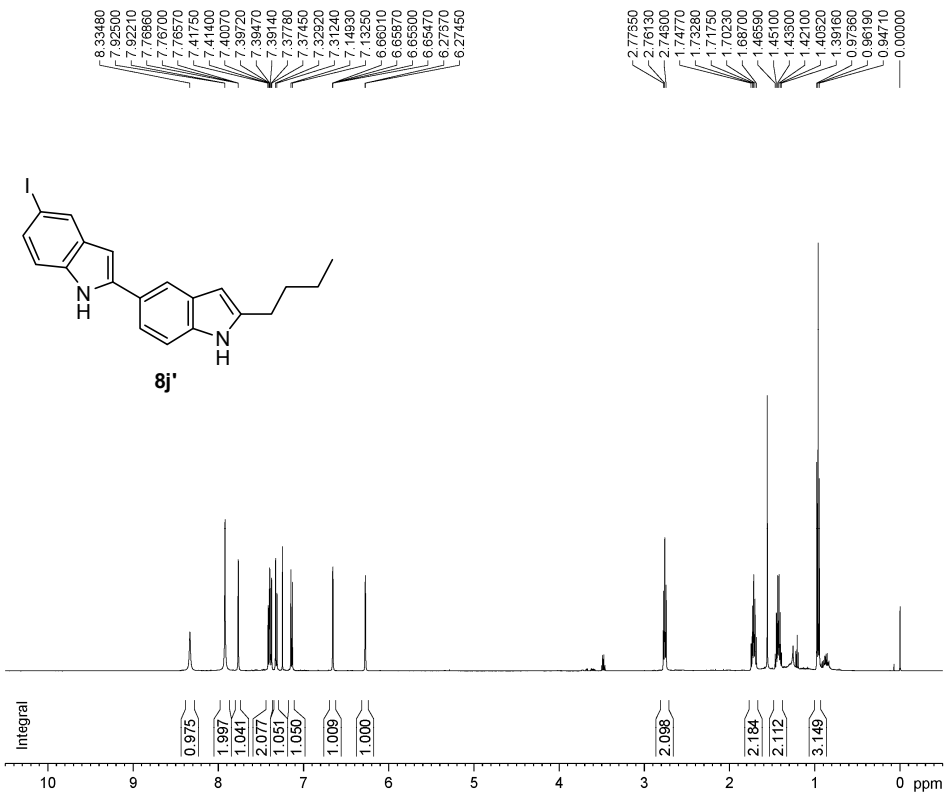

```
NAME              JLB252
EXPNO                 11
PROCNO                 1
Date_           20110524
Time               17.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                5160.6
DW                23.100 usec
DE                 6.50 usec
TE                298.4 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                0.00 dB
PL12              23.37 dB
PL13              26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547495 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



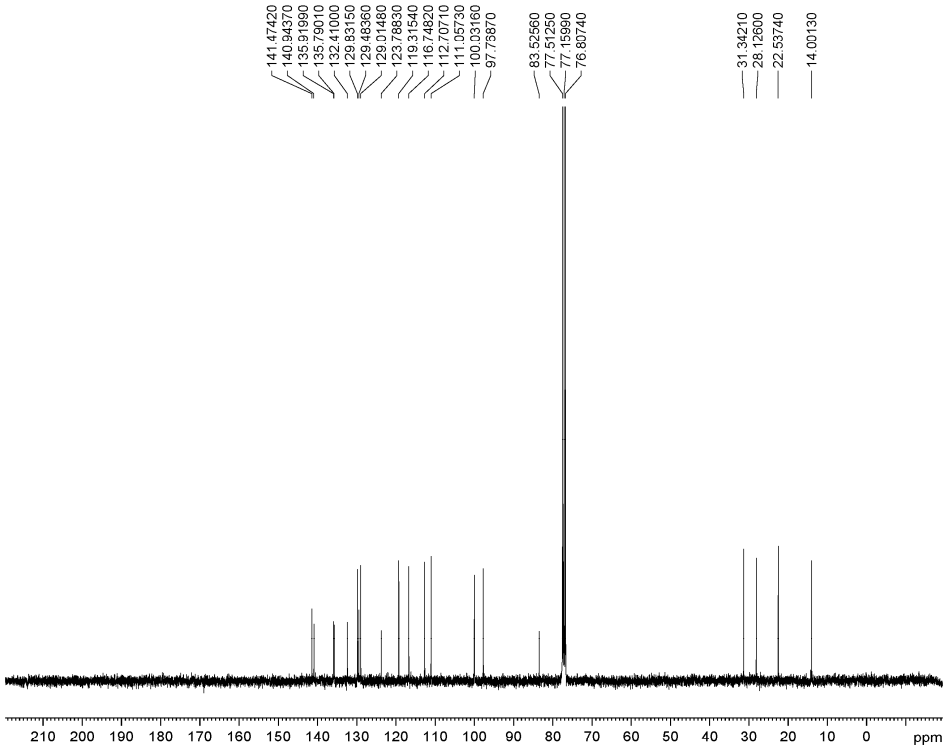

```
NAME              JLB290
EXPNO                 10
PROCNO                 1
Date_           20110715
Time                12.41
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  71.8
DW                48.400 usec
DE                 6.00 usec
TE                299.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W        12.18109322 W
SFO1       500.1330885 MHz
SI                 65536
SF         500.1300227 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```




```
NAME              JLB290
EXPNO                 11
PROCNO                 1
Date_           20110715
Time                15.16
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                4597.6
DW                23.100 usec
DE                 6.50 usec
TE                299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1       90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              20.39 dB
PL13              26.00 dB
SFO2      360.1314405 MHz
SI                 32768
SF         90.5547503 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

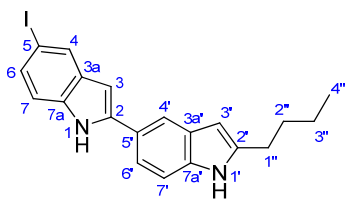



```
NAME            JLB292-2
EXPNO                 10
PROCNO                 1
Date_           20110719
Time                16.12
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                   128
DW                48.400 usec
DE                 6.00 usec
TE                299.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
PL1               -1.00 dB
PL1W         12.18109322 W
SFO1        500.1330885 MHz
SI                 65536
SF          500.1300194 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```




```
NAME            JLB292-2
EXPNO                 11
PROCNO                 1
Date_           20110720
Time                20.18
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                2298.8
DW                23.100 usec
DE                 6.50 usec
TE                299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 7.00 usec
PL1               -2.00 dB
SFO1        90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            115.00 usec
PL2                0.00 dB
PL12              20.39 dB
PL13              26.00 dB
SFO2       360.1314405 MHz
SI                 32768
SF          90.5547491 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```

COSY spectrum of **8j'**(partial)



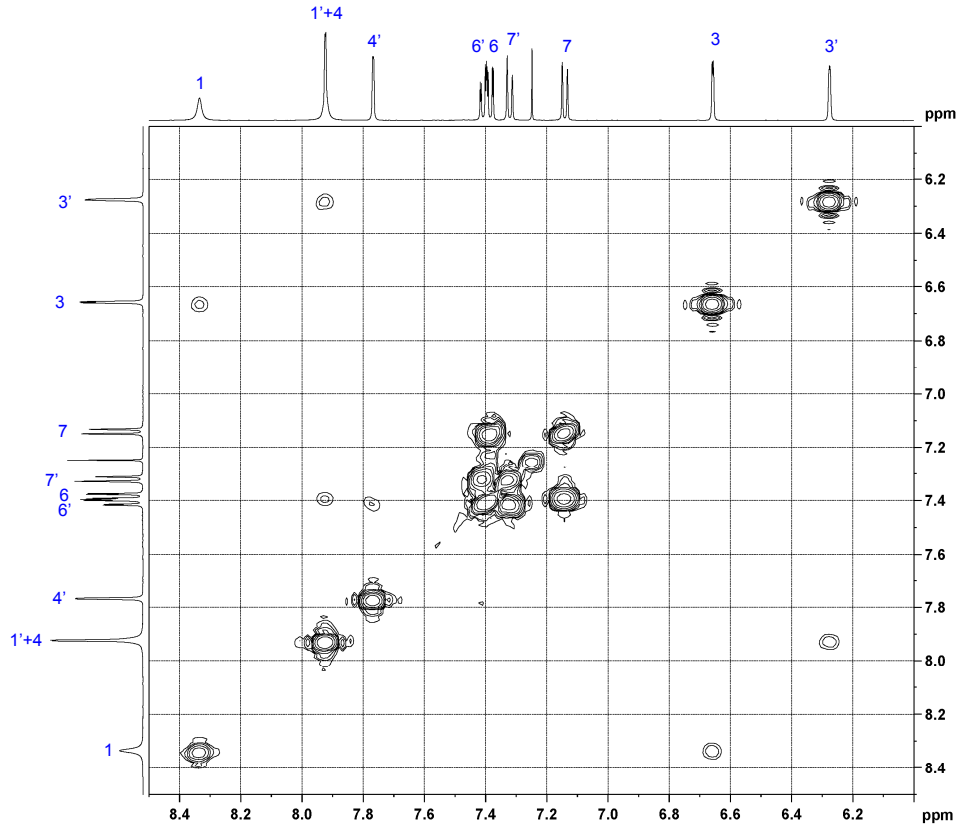
Key Correlations

Three-bond correlations:

H(6)-H(7), H(6')-H(7'), H(1")-H(2")
H(2")-H(3"), H(3")-H(4")

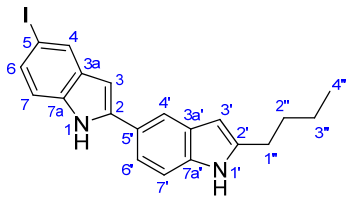
Four-bond correlations:

H(4)-H(6), H(3)-H(1), H(4')-H(6'),
H(3')-H(1')



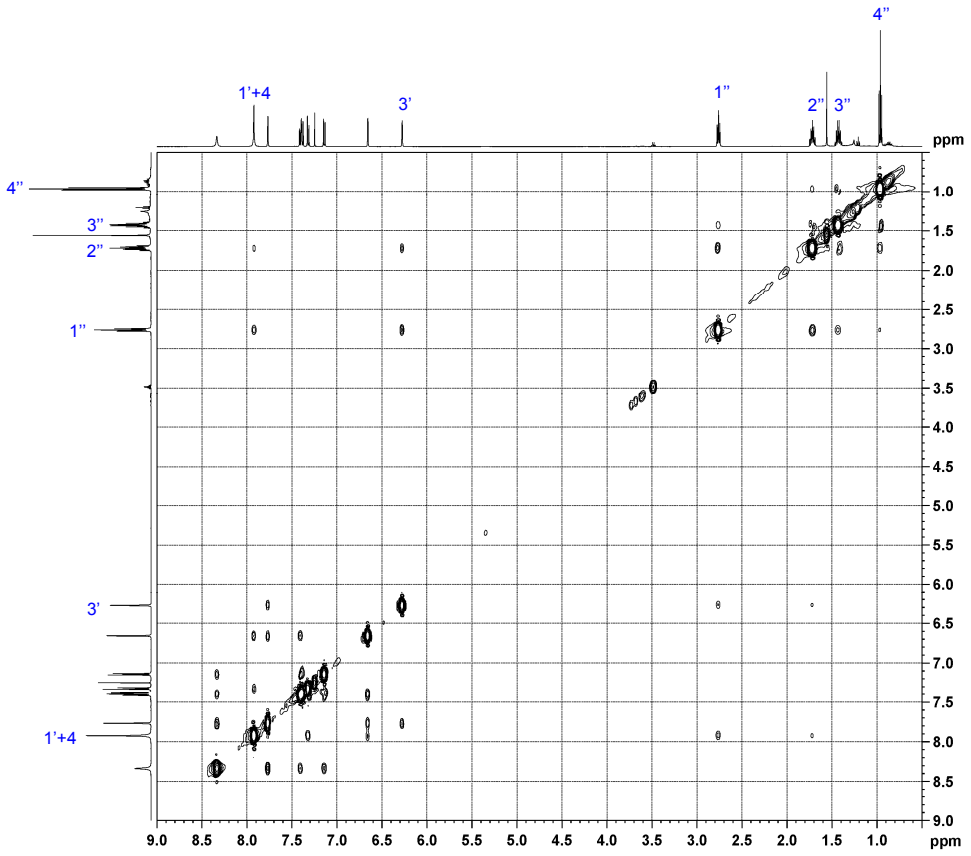

```
NAME                JLB292-2
EXPNO                     20
PROCNO                     1
Date_               20110719
Time                   16.13
INSTRUM                spect
PROBHD   5 mm SEI 1H-13
PULPROG            cosygpqf
TD                      1024
SOLVENT                CDCl3
NS                         1
DS                         8
SWH                 6510.417 Hz
FIDRES              6.357829 Hz
AQ                 0.0787700 sec
RG                    9195.2
DW                    76.800 usec
DE                     8.00 usec
TE                    299.0 K
D0                 0.00000300 sec
D1                 2.00000000 sec
D13                0.00000400 sec
D16                0.00010000 sec
IN0                0.00015360 sec

======== CHANNEL f1 ========
NUC1                       1H
P0                      8.20 usec
P1                      8.20 usec
PL1                    -1.00 dB
PL1W            12.18109322 W
SFO1            500.1331008 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPZ1                  20.00 %
P16                 1000.00 usec
ND0                        1
TD                       256
SFO1                500.1331 MHz
FIDRES             25.431377 Hz
SW                    13.017 ppm
FnMODE                    QF
SI                      1024
SF              500.1300154 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40
SI                      1024
MC2                       QF
SF              500.1300124 MHz
WDW                     SINE
SSB                        0
LB                      0.00 Hz
GB                         0
```

NOESY spectrum of **8j'**



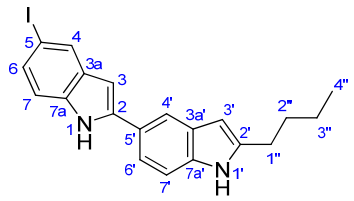
Key Correlations

H(1")-H(3'), H(1")-H(1'), H(3')-H(4'), H(4')-H(1), H(4')-H(3), H(6')-H(1), H(6')-H(3), H(7')-H(1'), H(1)-H(7), H(3)-H(4), H(7)-H(1)



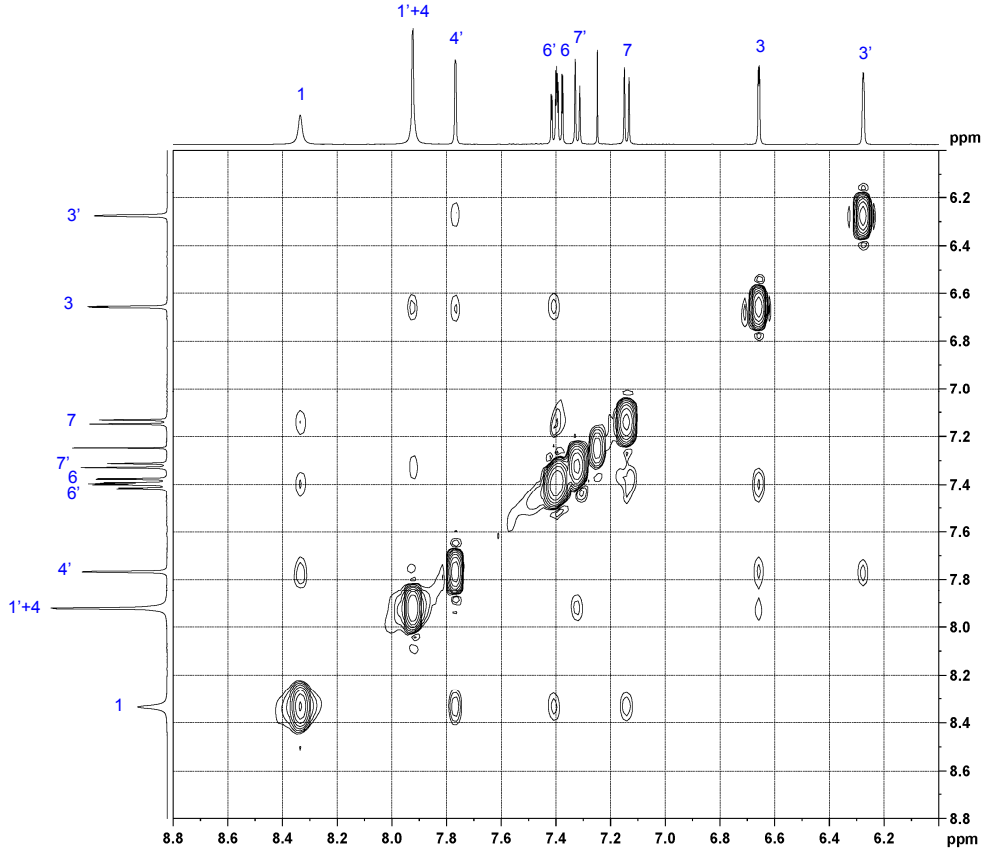

```
NAME            JLB292-2
EXPNO                 23
PROCNO                 1
Date_           20110719
Time_              23.25
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG      noesygpphpp
TD                  1024
SOLVENT             CDCl3
NS                    16
DS                    16
SWH             6510.417 Hz
FIDRES          6.357829 Hz
AQ              0.0787700 sec
RG                574.7
DW                76.800 usec
DE                 6.00 usec
TE                299.0 K
D0              0.00006636 sec
D1              2.00000000 sec
D8              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
D16             0.00010000 sec
IN0             0.00015360 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
P2                 16.40 usec
P17              2500.00 usec
PL1                -1.00 dB
PL10               10.83 dB
PL1W         12.18109322 W
PL10W         0.79925668 W
SFO1         500.1331008 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPZ1               40.00 %
GPZ2              -40.00 %
P16              1000.00 usec
ND0                   1
TD                  128
SFO1          500.1331 MHz
FIDRES         50.862755 Hz
SW                13.017 ppm
FnMODE              TPPI
SI                  1024
SF           500.1300161 MHz
WDW                QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                  1024
MC2                 TPPI
SF           500.1300161 MHz
WDW                QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
```

NOESY spectrum of **8j'** (partial)



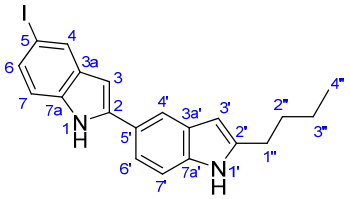
Key Correlations

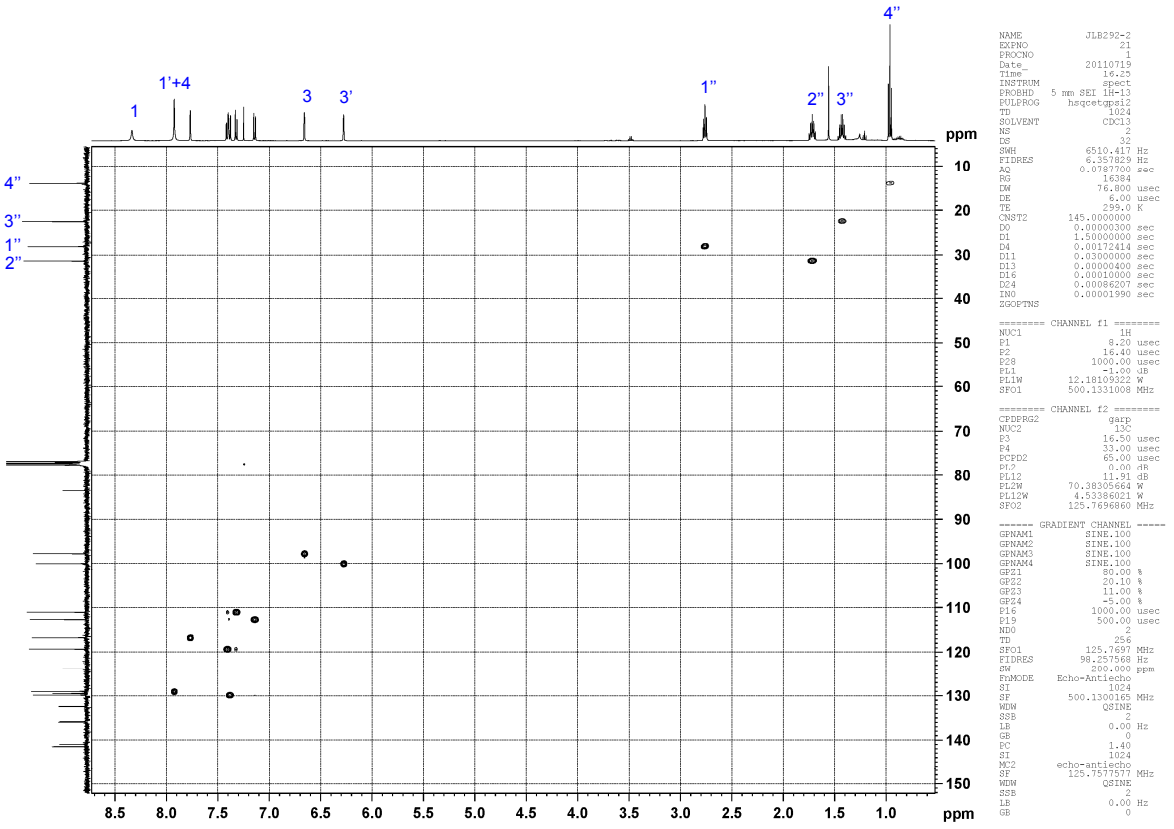
H(1")-H(3'), H(1")-H(1'), H(3')-H(4'), H(4')-H(1), H(4')-H(3), H(6')-H(1), H(6')-H(3), H(7')-H(1'), H(1)-H(7), H(3)-H(4), H(7)-H(1)




```
NAME                JLB292-2
EXPNO                     23
PROCNO                     1
Date_               20110719
Time                   23.25
INSTRUM                spect
PROBHD    5 mm SEI 1H-13
PULPROG          noesygpphpp
TD                       1024
SOLVENT                CDCl3
NS                         16
DS                         16
SWH                6510.417 Hz
FIDRES             6.357829 Hz
AQ                 0.0787700 sec
RG                     574.7
DW                    76.800 usec
DE                     8.00 usec
TE                    299.0 K
D0                 0.00006636 sec
D1                 2.00000000 sec
D8                 1.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec
D16                0.00010000 sec
IN0                0.00015360 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       8.20 usec
P2                      16.40 usec
P17                   2500.00 usec
PL1                     -1.00 dB
PL10                    10.83 dB
PL1W               12.18109322 W
PL10W               0.79925668 W
SFO1               500.1331008 MHz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                     40.00 %
GPZ2                    -40.00 %
P16                    1000.00 usec
NDO                        1
TD                       128
SFO1                500.1331 MHz
FIDRES             50.862755 Hz
SW                    13.017 ppm
FnMODE                   TPPI
SI                       1024
SF                 500.1300161 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40
SI                       1024
MC2                      TPPI
SF                 500.1300161 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

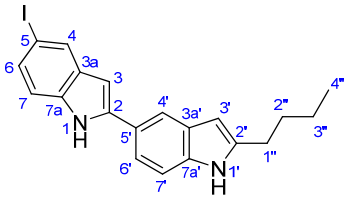
HSQC spectrum of **8j'**

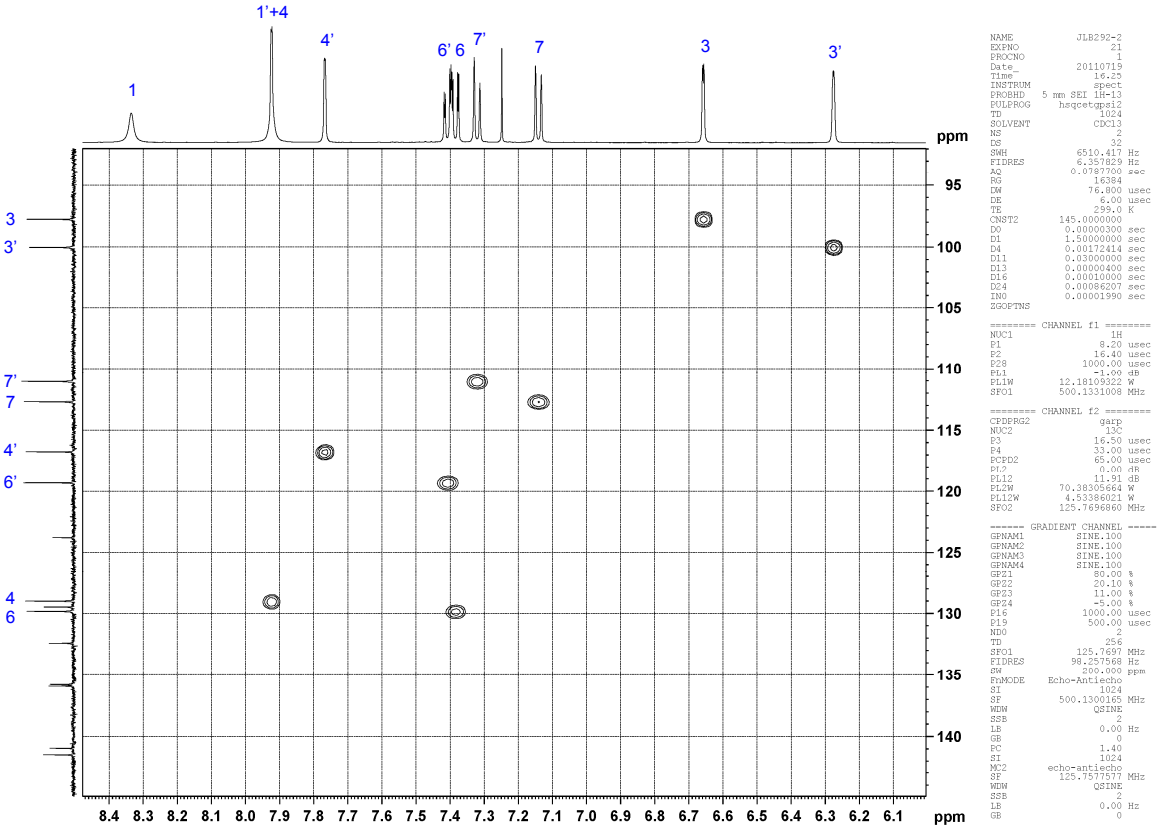





```
NAME                JLB292-2
EXPNO                     21
PROCNO                     1
Date_               20110719
Time                   16.25
INSTRUM                spect
PROBHD   5 mm BEI 1H-13 
PULPROG         hsqcetgpsi2
TD                       1024
SOLVENT                CDCl3
NS                         2
DS                        32
SWH                6510.417 Hz
FIDRES             6.357829 Hz
AQ                 0.0787700 sec
RG                     16384
DW                  76.800 usec
DE                    6.00 usec
TE                   299.0 K
CNST2              145.0000000
D0                 0.00000300 sec
D1                 1.50000000 sec
D4                 0.00172414 sec
D11                0.03000000 sec
D13                0.00004400 sec
D16                0.00010000 sec
D24                0.00086207 sec
IN0                0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                       1H
P1                       8.20 usec
P2                      16.40 usec
P28                   1000.00 usec
PL1                     -1.00 dB
PL1W               12.18109322 W
SFO1            500.1331008 MHz

======== CHANNEL f2 ========
CPDPRG2                 garp
NUC2                     13C
P3                      16.50 usec
P4                      33.00 usec
PCPD2                   65.00 usec
PL2                      0.00 dB
PL12                    11.91 dB
PL2W               70.38305664 W
PL12W               4.53386021 W
SFO2            125.7696860 MHz

====== GRADIENT CHANNEL =====
GPNAM1               SINE.100
GPNAM2               SINE.100
GPNAM3               SINE.100
GPNAM4               SINE.100
GPZ1                    80.00 %
GPZ2                    20.10 %
GPZ3                    11.00 %
GPZ4                    -5.00 %
P16                   1000.00 usec
P19                    500.00 usec
ND0                         2
TD                        256
SFO1            125.7697 MHz
FIDRES            98.257568 Hz
SW                   200.000 ppm
FnMODE         Echo-Antiecho
SI                       1024
SF              500.1300165 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40
SI                       1024
MC2            echo-antiecho
SF              125.7577577 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

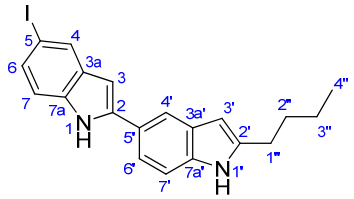
HSQC spectrum of **8j'** (partial)





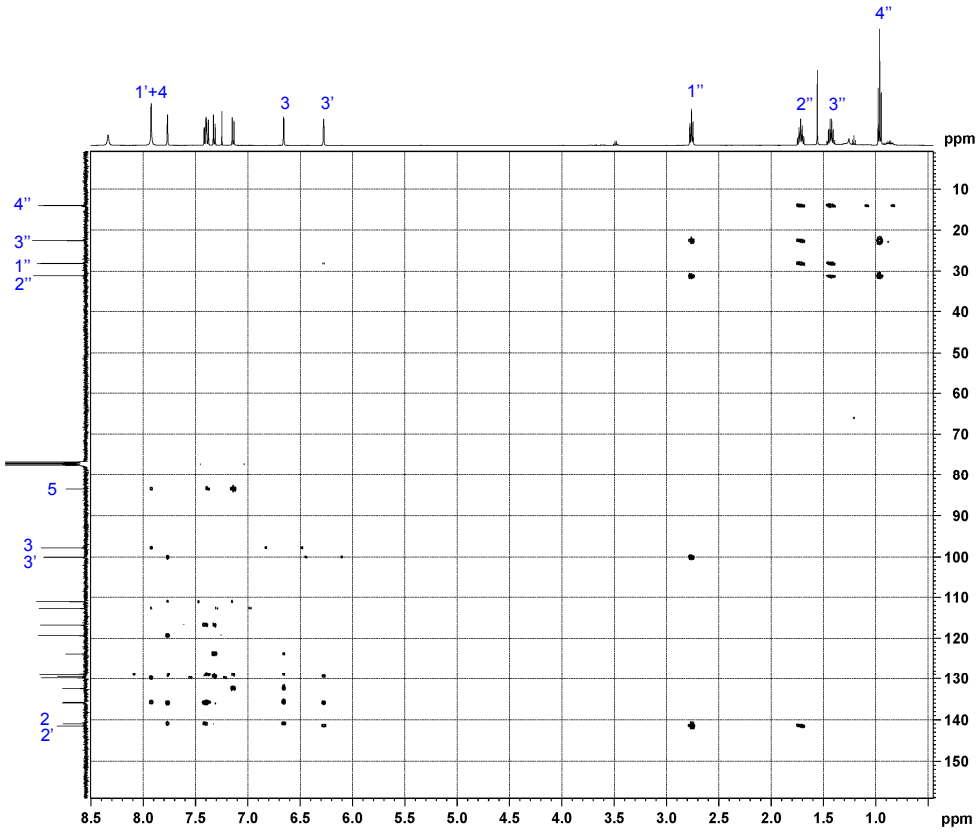

```
NAME                JLB292-2
EXPNO                     21
PROCNO                     1
Date_               20110719
Time_                  16.25
INSTRUM                spect
PROBHD   5 mm BEI 1H-13 
PULPROG         hsqcetgpsi2
TD                       1024
SOLVENT                CDCl3
NS                          2
DS                         32
SWH                 6510.417 Hz
FIDRES              6.357829 Hz
AQ                  0.0787700 sec
RG                      16384
DW                     76.800 usec
DE                      6.00 usec
TE                     299.0 K
CNST2              145.0000000
D0                  0.00000300 sec
D1                  1.50000000 sec
D4                  0.00172414 sec
D11                 0.03000000 sec
D13                 0.00004400 sec
D16                 0.00010000 sec
D24                 0.00086207 sec
IN0                 0.00001990 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                       1H
P1                       8.20 usec
P2                      16.40 usec
P28                   1000.00 usec
PL1                     -1.00 dB
PL1W               12.18109222 W
SFO1              500.1331008 MHz

======== CHANNEL f2 ========
CPDPRG2                 garp
NUC2                     13C
P3                      16.50 usec
P4                      33.00 usec
PCPD2                   65.00 usec
PL2                      0.00 dB
PL12                    11.91 dB
PL2W               70.38305664 W
PL12W               4.53386021 W
SFO2              125.7696860 MHz

====== GRADIENT CHANNEL =====
GPNAM1               SINE.100
GPNAM2               SINE.100
GPNAM3               SINE.100
GPNAM4               SINE.100
GPZ1                    80.00 %
GPZ2                    20.10 %
GPZ3                    11.00 %
GPZ4                    -5.00 %
P16                   1000.00 usec
P19                    500.00 usec
ND0                         2
TD                        256
SFO1              125.7697 MHz
FIDRES             98.257568 Hz
SW                   200.000 ppm
FnMODE          Echo-Antiecho
SI                       1024
SF                500.1300165 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40
SI                       1024
MC2             echo-antiecho
SF                125.7577577 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

HMBC spectrum of **8j'**



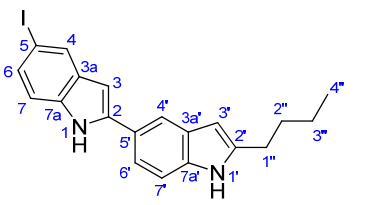
Key Correlations

C(1")-H(3'),
C(2')-H(1")/H(2"),
C(3')-H(1")



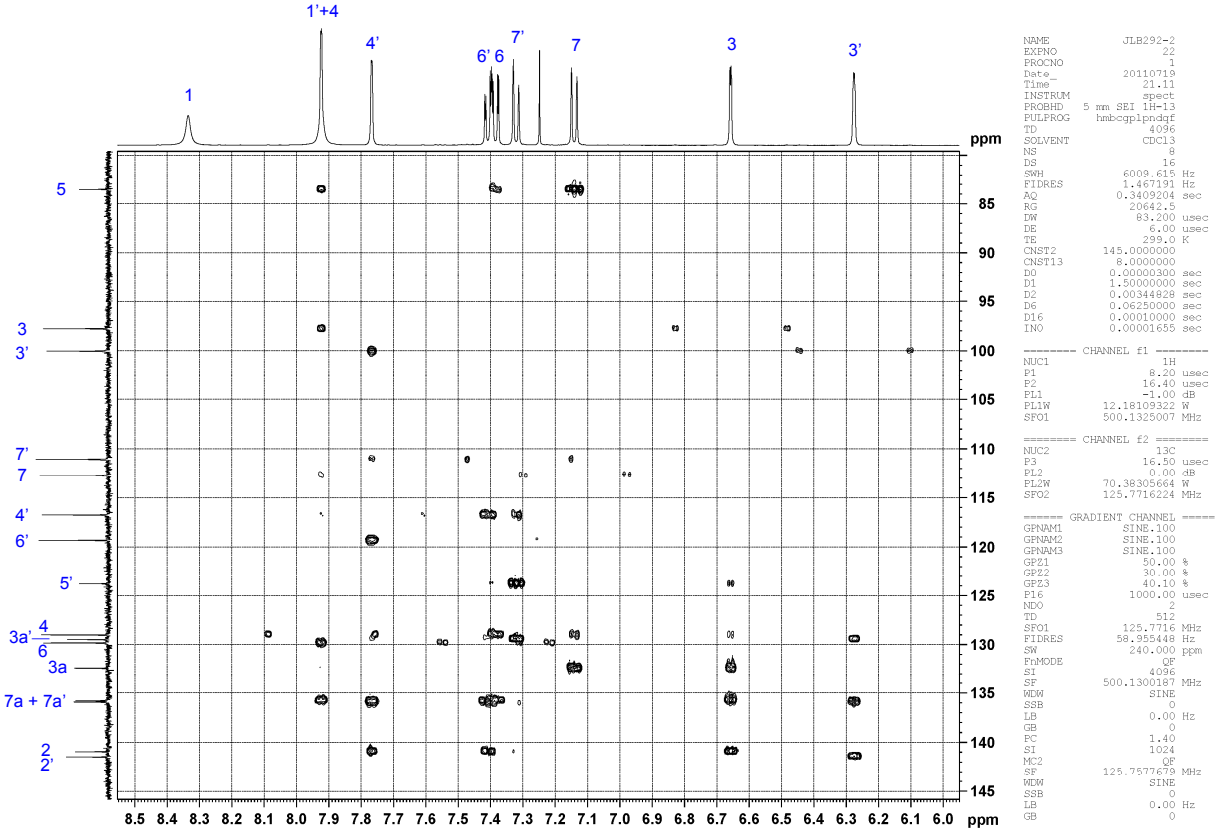

```
NAME                JLB292-2
EXPNO                     22
PROCNO                     1
Date_               20110719
Time                   21.11
INSTRUM                spect
PROBHD   5 mm SEI 1H-13
PULPROG     hmbcgplpndqf
TD                      4096
SOLVENT              CDCl3
NS                         8
DS                        16
SWH                6009.615 Hz
FIDRES             1.467191 Hz
AQ                 0.3409204 sec
RG                   20642.5
DW                    83.200 usec
DE                     6.00 usec
TE                   299.0 K
CNST2              145.0000000
CNST13               8.0000000
D0                 0.00000300 sec
D1                 1.50000000 sec
D2                 0.00344828 sec
D6                 0.06250000 sec
D16                0.00010000 sec
IN0                0.00001655 sec

--------- CHANNEL f1 ---------
NUC1                       1H
P1                       8.20 usec
P2                      16.40 usec
PL1                     -1.00 dB
PL1W            12.18109322 W
SFO1            500.1325007 MHz

--------- CHANNEL f2 ---------
NUC2                      13C
P3                      16.50 usec
PL2                      0.00 dB
PL2W            70.38305664 W
SFO2            125.7716224 MHz

------ GRADIENT CHANNEL -----
GPNAM1                SINE.100
GPNAM2                SINE.100
GPNAM3                SINE.100
GPZ1                     50.00 %
GPZ2                     30.00 %
GPZ3                     40.10 %
P16                    1000.00 usec
NDO                          2
TD                         512
SFO1                125.7716 MHz
FIDRES             58.955448 Hz
SW                    240.000 ppm
FnMODE                      QF
SI                        4096
SF                500.1300187 MHz
WDW                      SINE
SSB                         0
LB                       0.00 Hz
GB                          0
PC                       1.40
SI                       1024
MC2                         QF
SF                125.7577679 MHz
WDW                      SINE
SSB                         0
LB                       0.00 Hz
GB                          0
```

HMBC spectrum of **8j'** (partial)

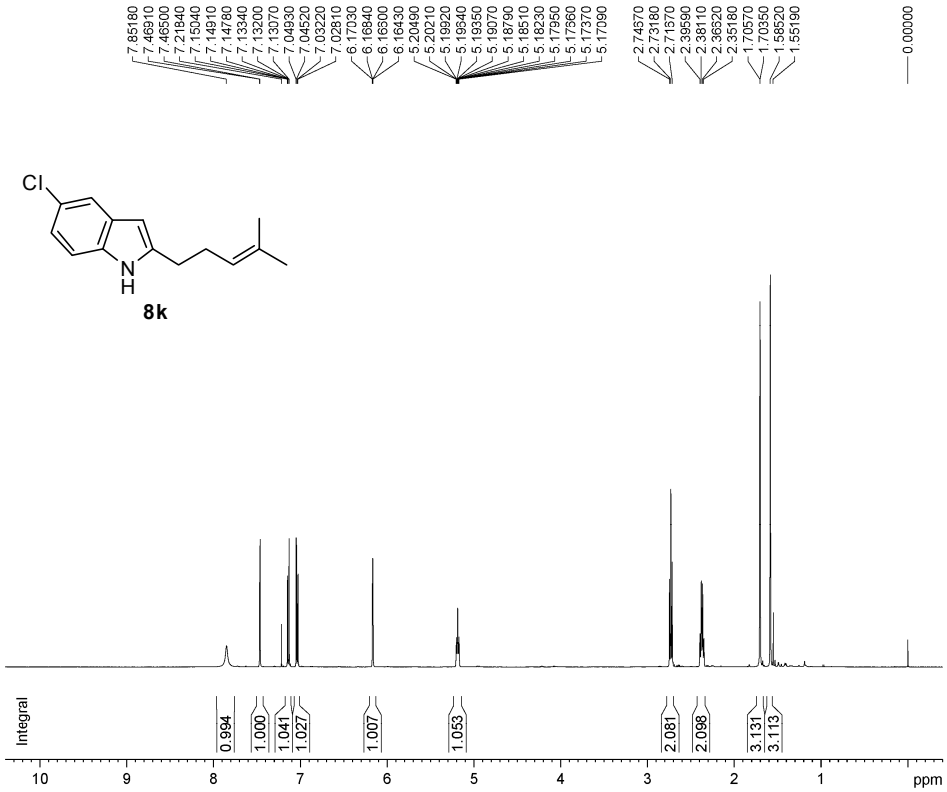


Key Correlations

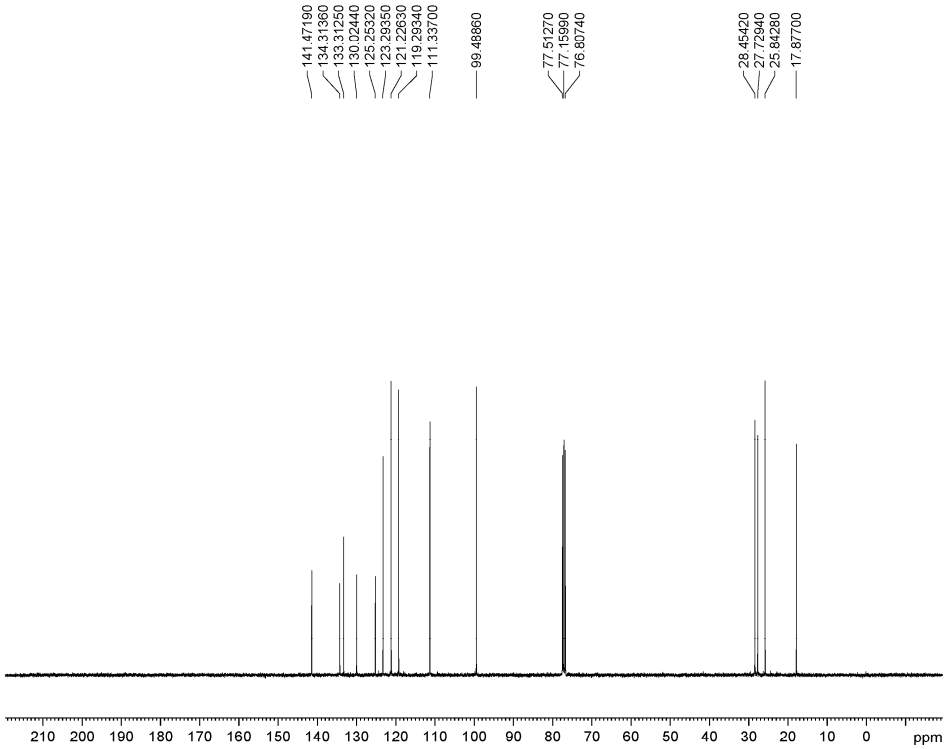
C(2')-H(3'), C(3')-H(4'), C(3a')-H(7')/H(3'), C(4')-H(6')/H(7'), C(5')-H(3)/H(6')/H(7'), C(6')-H(4'), C(7')-H(4'), C(7a')-H(3')/H(4')/H(6'), C(2)-H(3)/H(4')/H(6'), C(3)-H(4), C(4)-H(6)/H(7), C(5)-H(4)/H(6)/H(7), C(6)-H(4), C(7)-H(4), C(7a)-H(3)/H(4)/H(6)



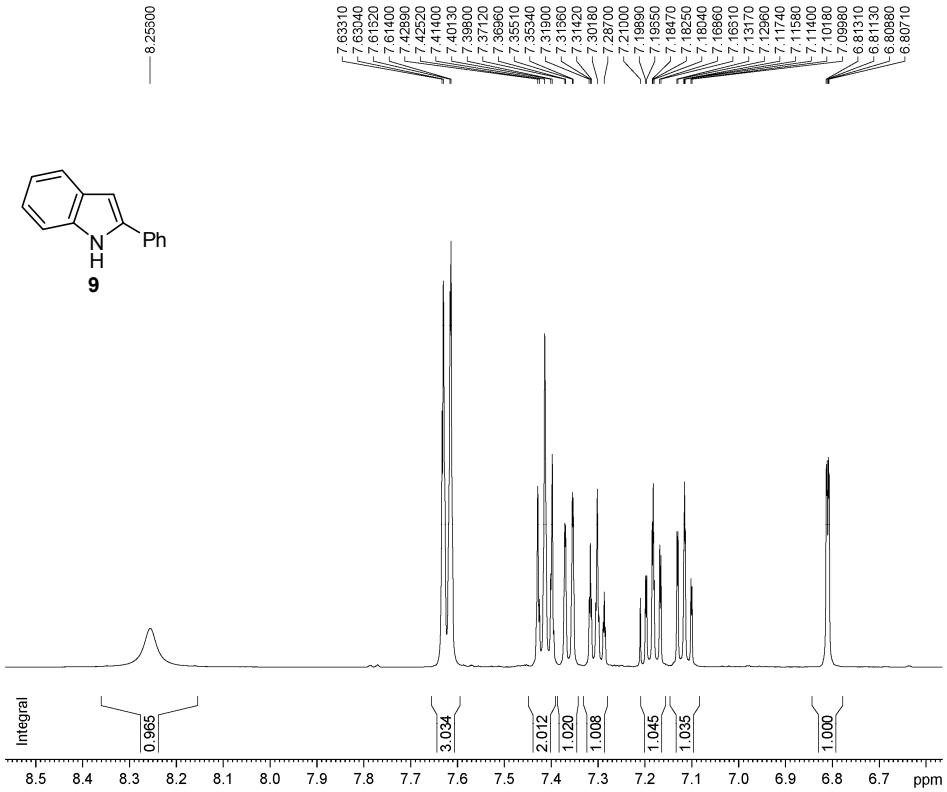

```
NAME              JLB292-2
EXPNO                   22
PROCNO                   1
Date_              20110719
Time_                 21.11
INSTRUM                spect
PROBHD  5 mm SEI 1H-13
PULPROG    hmbcgplpndqf
TD                     4096
SOLVENT             CDCl3
NS                        8
DS                       16
SWH               6009.615 Hz
FIDRES            1.467191 Hz
AQ                0.3409204 sec
RG                  20642.5
DW                   83.200 usec
DE                    6.00 usec
TE                  299.0 K
CNST2           145.0000000
CNST13            8.0000000
D0                0.00000300 sec
D1                1.50000000 sec
D2                0.00344828 sec
D6                0.06250000 sec
D16               0.00010000 sec
IN0               0.00001655 sec

-------- CHANNEL f1 --------
NUC1                     1H
P1                     8.20 usec
P2                    16.40 usec
PL1                   -1.00 dB
PL1W            12.18109322 W
SFO1           500.1325007 MHz

======== CHANNEL f2 ========
NUC2                    13C
P3                    16.50 usec
PL2                    0.00 dB
PL2W            70.38305664 W
SFO2           125.7716224 MHz

====== GRADIENT CHANNEL =====
GPNAM1             SINE.100
GPNAM2             SINE.100
GPNAM3             SINE.100
GPZ1                  50.00 %
GPZ2                  30.00 %
GPZ3                  40.10 %
P16                 1000.00 usec
NDO                      2
TD                     512
SFO1           125.7716 MHz
FIDRES          58.955448 Hz
SW                240.000 ppm
FnMODE                  QF
SI                     4096
SF             500.1300187 MHz
WDW                   SINE
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40
SI                     1024
MC2                     QF
SF             125.7577679 MHz
WDW                   SINE
SSB                      0
LB                    0.00 Hz
GB                       0
```



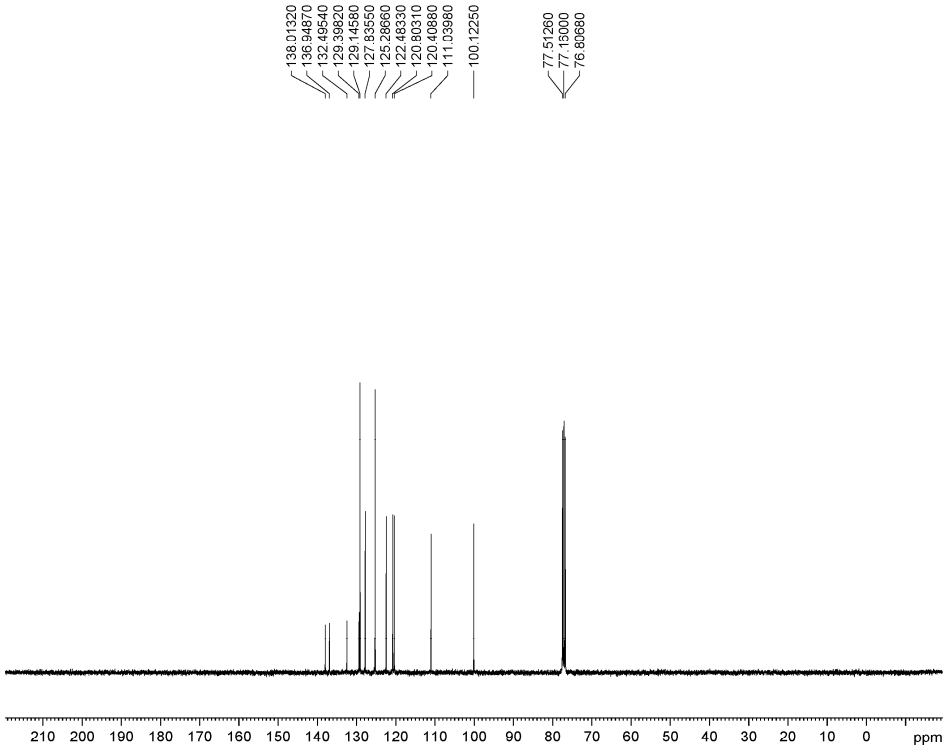

```
NAME              JLB255
EXPNO                 10
PROCNO                 1
Date_           20110530
Time                14.07
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  35.9
DW                48.400 usec
DE                 6.00 usec
TE                299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W         12.18109322 W
SFO1         500.1330885 MHz
SI                 65536
SF           500.1300342 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```




```
NAME              JLB255
EXPNO                 11
PROCNO                 1
Date_           20110530
Time                15.00
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    768
DS                      4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                  4096
DW                23.100 usec
DE                 6.50 usec
TE                299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               23.37 dB
PL13               26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547530 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME              JLB285
EXPNO                 10
PROCNO                 1
Date_           20110712
Time                11.52
INSTRUM             spect
PROBHD   5 mm SEI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  71.8
DW                48.400 usec
DE                 6.00 usec
TE                299.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                -1.00 dB
PL1W        12.18109322 W
SFO1        500.1330885 MHz
SI                 65536
SF          500.1300384 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```




```
NAME              JLB285
EXPNO                 11
PROCNO                 1
Date_           20110712
Time                14.09
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH           21645.021 Hz
FIDRES         0.330277 Hz
AQ             1.5139316 sec
RG                  3251
DW                23.100 usec
DE                 6.50 usec
TE                299.0 K
D1            2.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.00 usec
PL1                -2.00 dB
SFO1         90.5638160 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             115.00 usec
PL2                 0.00 dB
PL12               20.39 dB
PL13               26.00 dB
SFO2        360.1314405 MHz
SI                 32768
SF           90.5547530 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```