Supporting Information

Carboxylic Acid-Catalyzed Three-Component Aza-Friedel-Crafts Reactions in Water for the Synthesis of 3-Substituted Indoles

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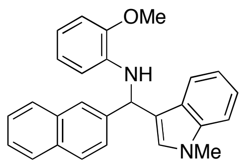
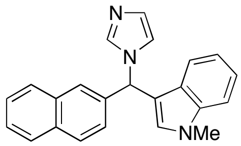
General: ^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECX-600 spectrometer in $CDCl_3$. Tetramethylsilane (TMS) served as internal standard (0 ppm) for ^{1}H NMR, and $CDCl_3$ was used as internal standard (77.0 ppm) for ^{13}C NMR. IR spectra were measured with JASCO FT/IR-610 spectrometers. High resolution mass spectrometry was carried out using BRUCKER DALTONICS BioTOFII. Preparative thin-layer chromatography was carried out using Wakogel B-5F.

General experimental procedure for aza-Friedel Crafts reactions in water (Table 1): Catalyst (0.010 mmol), 2-naphthaldehyde (0.20 mmol), *o*-anisidine (0.20 mmol), and 1-methylindole (0.20 mmol) were added to H_2O (2.0 mL), and the mixture was stirred for 24 h at rt. After CH_2Cl_2 (5.0 mL) and $NaHCO_3$ aq. (5.0 mL) were added to the mixture, the aqueous layer was extracted with CH_2Cl_2 (2 x 5.0 mL) and the combined organic layer was dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was dissolved in $CDCl_3$ (2.0 mL). Mesitylene (0.20 mmol) as an internal standard was dissolved in the solution, and the yields of the products were determined by ^{1}H NMR analyses.

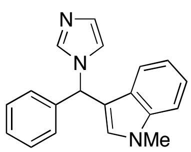


4a : ^{1}H NMR (600 MHz, $CDCl_3$) □ 8.00 (s, 1H), 7.79–7.83 (m, 3H), 7.59–7.64 (m, 2H), 7.42–7.46 (m, 2H), 7.28–7.30 (m, 1H), 7.22–7.24 (m, 1H), 7.08 (t, J = 7.6 Hz, 1H), 6.79 (dd, J = 1.4, 7.6 Hz, 1H), 6.71 (dt, J = 1.4, 7.6 Hz, 1H), 6.64 (dt, J = 1.4, 7.6 Hz, 1H), 6.62 (s, 1H), 6.53 (dd, J = 1.4, 7.6 Hz, 1H), 5.95 (s, 1H), 5.03 (brs, 1H), 3.81 (s, 3H), 3.66 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) □ 146.8, 140.5, 137.6, 137.3, 133.5, 132.8, 128.20, 128.18, 128.05, 127.6, 126.6, 125.9, 125.5, 125.4, 121.9, 121.1, 119.5, 119.3, 117.5, 116.4, 111.1, 109.3, 109.2, 55.7, 55.4, 32.7 ppm; IR (neat) 3427, 3052, 2935, 2823, 1598, 1507, 1457, 1226, 1123, 737 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{25}N_2O$: 393.1961 ([M+H]$^+$), found 393.1957 ([M+H]$^+$).

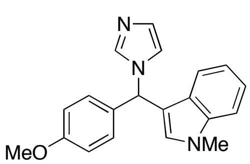
Typical experimental procedure for the synthesis of aromatase inhibitor type compounds 7 (Entry 1 of Table 2): $C_9H_{19}COOH$ (0.020 mmol), 2-naphthaldehyde (0.20 mmol), *o*-anisidine (0.20 mmol), and 1-methylindole (0.20 mmol) were added to H_2O (2.0 mL), and the mixture was stirred for 24 h at rt. After CH_2Cl_2 (5.0 mL) and $NaHCO_3$ aq. (5.0 mL) were added to the mixture, the aqueous layer was extracted with CH_2Cl_2 (2 X 5.0 mL) and the combined organic layer was dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was dissolved in toluene (2.0 mL). $Sc(OTf)_3$ (0.02 mmol) and CDI (0.40 mmo) were added to the solution, and the reaction mixture was stirred 70 ˚C for 3 h. After cooled to rt, CH_2Cl_2 (5.0 mL) and $NaHCO_3$ aq. (5.0 mL) were added to the reaction mixture. The aqueous layer was extracted with CH_2Cl_2 (2 X 5.0 mL), and the combined organic layer was dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was purified by preparative thin layer chromatography (AcOEt/CH_2Cl_2 = 1:1) to afford the desired product.



7a : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74–7.77 (m, 2H), 7.66–7.68 (m, 1H), 7.50 (s, 1H), 7.46 (s, 1H), 7.39–7.43 (m, 2H), 7.24–7.28 (m, 2H), 7.16–7.19 (m, 2H), 7.03 (s, 1H), 6.97 (t, J = 7.9 Hz, 1H), 6.91 (s, 1H), 6.83 (s, 1H), 6.48 (s, 1H), 3.62 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.5, 137.4, 137.2, 133.2, 132.9, 129.3, 129.1, 128.5, 128.1, 127.6, 126.5, 126.4, 125.6, 125.1, 122.4, 120.0, 119.2, 119.0, 113.3, 109.6, 58.4, 32.8 ppm; IR (neat) 3112, 3054, 2934, 1478, 1223, 1069, 909, 738 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{20}N_3$: 338.1652 ([M+H]$^+$), found 338.1638 ([M+H]$^+$).

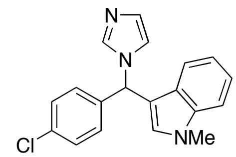


7b : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.22–7.28 (m, 4H), 7.16 (dt, J = 1.4, 7.6 Hz, 1H), 7.09–7.12 (m, 3H), 6.99 (s, 1H), 6.96 (dt, J = 1.4, 7.6 Hz, 1H), 6.85 (d, J = 1.4 Hz, 1H), 6.66 (s, 1H), 6.47 (s, 1H), 3.62 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.9, 137.3, 137.1, 129.1, 129.0, 128.6, 128.0, 127.0, 126.3, 122.3, 119.9, 119.1, 119.0, 113.4, 109.5, 58.3, 32.8 ppm; IR (neat) 3112, 3057, 2934, 1484, 1224, 1070, 913, 739 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{18}N_3$: 288.1495 ([M+H]$^+$), found 288.1493 ([M+H]$^+$).

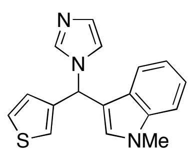


7c : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.31 (d, J = 8.2 Hz, 1H), 7.24 (dt, J = 1.4, 8.2 Hz, 1H), 7.17 (d, J = 8.2 Hz, 1H), 7.12 (d, J = 8.6 Hz, 2H), 7.06 (d, J = 1.4 Hz, 1H), 7.04 (dt, J = 1.4, 8.2 Hz, 1H), 6.93 (s, 1H), 6.87 (d, J = 8.6 Hz, 2H), 6.69 (s, 1H), 6.55 (s, 1H), 3.80 (s, 3H), 3.71 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.2, 137.3, 137.0, 131.9, 128.9, 128.8, 128.3, 126.2, 122.3, 119.8, 119.1, 119.0, 113.95, 113.89, 109.5, 57.9, 55.2, 32.8 ppm; IR (neat) 3109, 3050, 2933, 2836, 1507, 1469, 1247, 1068, 1028, 741 cm^{-1};

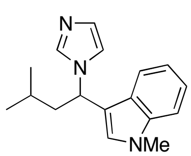
HRMS (ESI) calcd for $C_{20}H_{20}N_3O$: 318.1601 ([M+H]$^+$), found 318.1596 ([M+H]$^+$).



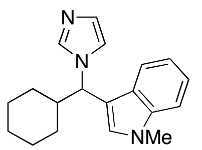
7d : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.31–7.34 (m, 3H), 7.26 (dt, *J* = 1.4, 7.6 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.05–7.11 (m, 4H), 6.91 (t, *J* = 1.4 Hz, 1H), 6.73 (s, 1H), 6.56 (s, 1H), 3.73 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.5, 137.4, 137.0, 133.9, 129.2, 129.0, 128.9, 128.4, 126.1, 122.5, 120.0, 118.9, 112.9, 109.6, 57.7, 32.9 ppm; IR (neat) 3111, 3055, 2930, 1485, 1224, 1081, 740 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{17}ClN_3$: 322.1106 ([M+H]$^+$), found 322.1098 ([M+H]$^+$).



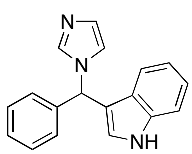
7e : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.31–7.33 (m, 2H), 7.23–7.26 (m, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.04–7.06 (m, 2H), 6.95–6.97 (m, 3H), 6.80 (s, 1H), 6.70 (s, 1H), 3.73 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 141.2, 137.3, 136.8, 129.0, 128.5, 126.8, 126.5, 126.1, 122.8, 122.3, 119.9, 119.0, 118.8, 113.3, 109.6, 54.5, 32.8 ppm; IR (neat) 3106, 3055, 2932, 2887, 1477, 1221, 1070, 909, 775, 741 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{15}N_3NaS$: 316.0879 ([M+Na]$^+$), found 316.0878 ([M+Na]$^+$).



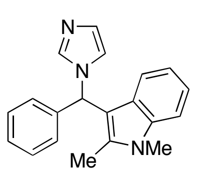
7f : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.34 (d, *J* = 8.2 Hz, 1H), 7.30 (d, *J* = 8.2 Hz, 1H), 7.22 (dt, *J* = 1.4, 7.6 Hz, 1H), 7.07 (dt, *J* = 1.4, 7.6 Hz, 1H), 7.02 (s, 1H), 6.99 (s, 1H), 6.97 (s, 1H), 5.54 (dd, *J* = 6.5, 8.6 Hz, 1H), 3.77 (s, 3H), 2.10–2.18 (m, 2H), 1.46–1.53 (m, 1H), 1.02 (d, *J* = 6.5 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.2, 136.3, 129.0, 126.4, 126.2, 122.2, 119.6, 118.7, 117.7, 114.2, 109.5, 52.7, 44.6, 32.8, 24.7, 22.8, 22.0 ppm; IR (neat) 3109, 3054, 2954, 2870, 1471, 1221, 1072, 742 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{22}N_3$: 268.1808 ([M+H]$^+$), found 268.1812 ([M+H]$^+$).



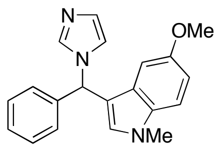
7g : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.08 (s, 1H), 6.98 (s, 2H), 5.09 (d, *J* = 9.6 Hz, 1H), 3.77 (s, 3H), 2.23–2.29 (m, 1H), 1.66–1.83 (m, 4H), 1.43–1.46 (m, 1H), 1.13–1.30 (m, 3H), 0.93–1.04 (m, 2H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 136.9, 136.7, 128.8, 126.9, 126.8, 122.1, 119.6, 118.7, 118.0, 112.7, 109.5, 60.2, 42.4, 32.8, 31.3, 30.1, 26.2, 26.0, 25.8 ppm; IR (neat) 3108, 3055, 2927, 2852, 1483, 1226, 1073, 740 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{24}N_3$: 294.1965 ([M+H]$^+$), found 294.1957 ([M+H]$^+$).



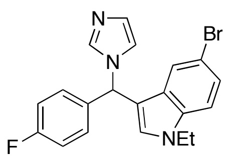
7h : ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.81 (brs,1H), 7.54 (s, 1H), 7.32–7.39 (m, 4H), 7.17–7.22 (m, 4H), 7.09 (s, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.96 (s, 1H), 6.76 (s, 1H), 6.72–6.73 (m, 1H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.8, 137.1, 136.7, 129.0, 128.7, 128.1, 127.1, 125.9, 124.8, 122.8, 120.3, 119.2, 118.9, 115.0, 111.5, 58.5 ppm; IR (neat) 3144, 3114, 2916, 2855, 1493, 1452, 1217, 1071, 912, 732 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{16}N_3$: 274.1339 ([M+H]$^+$), found 274.1328 ([M+H]$^+$).



7i : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.25–7.30 (m, 4H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.07 (s, 1H), 7.05 (d, *J* = 6.2 Hz, 2H), 6.95 (s, 1H), 6.91 (t, *J* = 7.6 Hz, 1H), 6.83 (d, *J* = 7.6 Hz, 1H), 6.77 (s, 1H), 3.66 (s, 3H), 2.30 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 140.3, 137.2, 136.6, 135.4, 129.0, 128.6, 127.6, 126.8, 126.2, 121.1, 119.8, 119.3, 118.7, 108.9, 57.9, 29.5, 10.5 ppm; IR (neat) 3102, 3056, 3035, 2933, 2909, 1473, 1221, 1072, 908, 740 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{19}N_3Na$: 324.1471 ($[M+Na]^+$), found 324.1460 ($[M+Na]^+$).



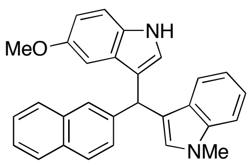
7j : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (s, 1H), 7.32–7.37 (m, 3H), 7.17–7.22 (m, 3H), 7.08 (s, 1H), 6.95 (s, 1H), 6.90 (dd, *J* = 2.7, 8.9 Hz, 1H), 6.71 (s, 1H), 6.57 (d, *J* = 2.0 Hz, 1H), 6.53 (s, 1H), 3.69 (s, 6H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.3, 140.0, 137.1, 132.6, 129.6, 129.0, 128.7, 128.0, 127.0, 126.7, 119.1, 112.8, 112.6, 110.4, 100.7, 58.4, 55.7, 33.0 ppm; IR (neat) 3111, 3060, 3032, 2997, 2939, 2832, 1492, 1219 1067, 725 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{19}N_3NaO$: 340.1420 ($[M+Na]^+$), found 340.1430 ($[M+Na]^+$).



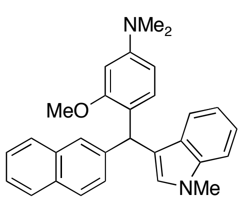
2 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.30–7.32 (m, 2H), 7.23 (d, *J* = 8.9 Hz, 1H), 7.12–7.14 (m, 2H), 7.09 (s, 1H), 7.03–7.07 (m, 2H), 6.90 (s, 1H), 6.68 (s, 1H), 6.61 (s, 1H), 4.09 (q, *J* = 7.3 Hz, 2H), 1.41 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.2, 161.6, 136.9, 135.27, 135.25, 135.1, 129.3, 128.8, 128.7, 128.2, 127.9, 125.3, 121.5, 118.9, 115.8, 115.7, 113.3, 113.1, 111.2, 57.5, 41.3, 15.3 ppm; IR (neat) 3115, 3062, 2979, 2935, 2888, 1509, 1223, 1072, 822, 785, 732 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{18}BrFN_3$: 398.0663 ($[M+H]^+$), found 398.0667 ($[M+H]^+$).

Experimental procedure for larger scale synthesis (Scheme 3): $C_9H_{19}COOH$ (1.0 mmol), 2-naphthaldehyde (10 mmol), *o*-anisidine (10 mmol), and 1-methylindole (10 mmo) were added to H_2O (100 mL), and the mixture was stirred for 24 h at rt. Then $NaHCO_3$ aq. (30 mL) was added, and the mixture was stirred for 15 min at rt. The resulting slightly brown white solid was collected by filtration and dried under reduced pressure. Purification of the resulting solid by recrystallization from CH_2Cl_2/hexane provided the pure product **4a** as a white solid.

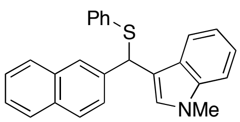
General experimental procedure for transformations of 4a (Scheme 4): AFC product **4a** (0.20 mmol) was dissolved in toluene (2.0 mL), and then $Sc(OTf)_3$ (0.02 mmol) and a nucleophile were added to the solution. The reaction mixture was stirred 70 ˚C for 24 h, and then cooled to rt. CH_2Cl_2 (5.0 mL) and $NaHCO_3$ aq. (5.0 mL) were added to the reaction mixture. The aqueous layer was extracted with CH_2Cl_2 (2 X 5.0 mL) and the combined organic layer was dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was purified by preparative thin layer chromatography to afford the desired product.



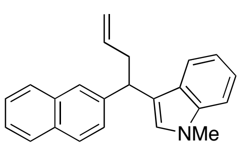
8 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69–7.81 (m, 5H), 7.51 (dd, *J* = 1.4, 8.2 Hz, 1H), 7.37–7.42 (m, 3H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.23 (d, *J* = 8.9 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.82–6.85 (m, 2H), 6.65 (d, *J* = 2.0 Hz, 1H), 6.52 (s, 1H), 5.99 (s, 1H), 3.66 (s, 6H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.7, 141.7, 137.4, 133.6, 132.3, 131.8, 128.5, 127.9, 127.8, 127.7, 127.53, 127.48, 127.43, 126.6, 125.6, 125.2, 124.6, 121.4, 120.1, 119.3, 118.6, 117.8, 111.9, 111.7, 109.1, 101.8, 55.8, 40.2, 32.7 ppm; IR (neat) 3414, 3051, 2938, 2830, 1587, 1478, 1211, 907, 738 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{25}N_2O$: 417.1961 ($[M+H]^+$), found 417.1903 ($[M+H]^+$).



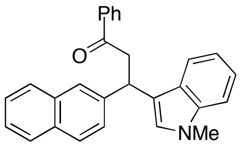
9 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.77–7.80 (m, 1H), 7.67–7.73 (m, 2H), 7.58 (s, 1H), 7.37–7.43 (m, 3H), 7.27 (t, *J* = 7.2 Hz, 2H), 7.17 (t, *J* = 7.2 Hz, 1H), 6.94 (t, *J* = 7.9 Hz, 1H), 6.89 (d, J = 8.9 Hz, 1H), 6.40 (s, 1H), 6.33 (brs, 1H), 6.22 (brd, J = 7.6 Hz, 1H), 6.12 (s, 1H), 3.74 (s, 3H), 3.66 (s, 3H), 2.93 (s, 6H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.7, 150.3, 142.7, 137.4, 133.5, 132.1, 130.5, 128.8, 128.3, 127.8, 127.6, 127.5, 127.3, 126.6, 125.4, 125.0, 121.3, 120.2, 118.54, 118.52, 108.9, 104.6, 96.5, 55.6, 40.8, 40.6, 32.6 ppm; IR (neat) 3050, 2935, 2880, 2831, 2802, 1615, 1516, 1469, 1353, 1239, 737 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{29}N_2O$: 421.2274 ([M+H]$^+$), found 421.2306 ([M+H]$^+$).



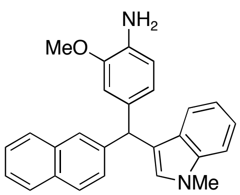
10 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.74–7.81 (m, 3H), 7.67 (dd, J = 1.4, 8.9 Hz, 2H), 7.43–7.45 (m, 2H), 7.28–7.30 (m, 3H), 7.22 (t, J = 7.6 Hz, 1H), 7.15 (t, J = 7.6 Hz, 2H), 7.07–7.12 (m, 2H), 6.90 (s, 1H), 5.96 (s, 1H), 3.70 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.9, 137.4, 136.7, 133.3, 132.7, 130.2, 128.7, 128.6, 128.2, 128.0, 127.6, 126.8, 126.7, 126.6, 126.3, 126.0, 125.8, 122.0, 119.7, 119.2, 114.6, 109.4, 50.1, 32.8 ppm; IR (neat) 3053, 2925, 1587, 1472, 1367, 1335, 1121, 1020, 903, 736, 691 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{21}NNaS$: 402.1287 ([M+Na]$^+$), found 402.1310 ([M+Na]$^+$).



11 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76–7.78 (m, 3H), 7.73 (d, J = 8.9 Hz, 1H), 7.38–7.45 (m, 4H), 7.25 (d, J = 8.3 Hz, 1H), 7.16 (t, J = 7.2 Hz, 1H), 6.97 (t, J = 7.6 Hz, 1H), 6.90 (s, 1H), 5.80–5.86 (m, 1H), 5.06–5.10 (m, 1H), 4.94–4.96 (m, 1H), 4.44 (t, J = 7.6 Hz, 1H), 3.74 (s, 3H), 2.99–3.04 (m, 1H), 2.85–2.90 (m, 1H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 142.3, 137.3, 137.1, 133.5, 132.2, 127.9, 127.7, 127.5, 127.3, 126.7, 126.2, 126.1, 125.7, 125.2, 121.5, 119.5, 118.7, 118.1, 116.0, 109.1, 43.1, 40.4, 32.7 ppm; IR (neat) 3053, 2921, 1633, 1474, 1370, 1329, 1124, 1005, 910, 813, 740 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{22}N$: 312.1747 ([M+H]$^+$), found 312.1723 ([M+H]$^+$).

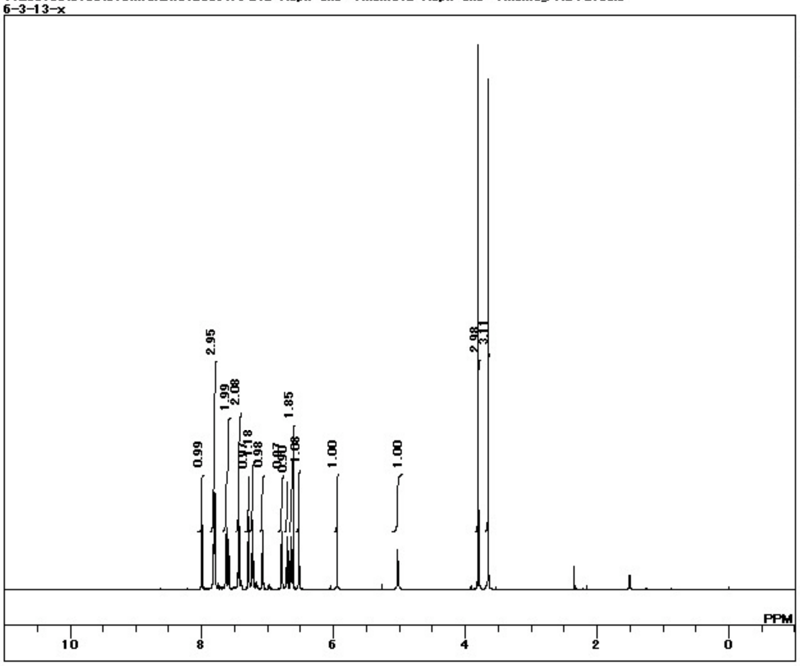


12 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.80 (s, 1H), 7.72–7.76 (m, 3H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 9.6 Hz, 2H), 7.38–7.43 (m, 4H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.85 (s, 1H), 5.23 (t, *J* = 7.2 Hz, 1H), 3.82–3.90 (m, 2H), 3.70 (s, 3H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 198.4, 141.8, 137.3, 137.0, 133.5, 133.0, 132.2, 128.5, 128.1, 127.7, 127.5, 127.0, 126.7, 126.4, 125.8, 125.3, 121.7, 119.5, 118.9, 117.6, 109.2, 45.1, 38.1, 32.7 ppm; IR (neat) 3054, 2931, 1685, 1597, 1474, 1328, 1272, 906, 737, 691 cm^{-1}; HRMS (ESI) calcd for $C_{28}H_{24}NO$: 390.1852 ($[M+H]^+$), found 390.1853 ($[M+H]^+$).



13 : ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69–7.80 (m, 3H), 7.61 (s, 1H), 7.39–7.43 (m, 3H), 7.25–7.29 (m, 2H), 7.18 (t, *J* = 7.6 Hz, 1H), 6.95 (t, *J* = 7.6 Hz, 1H), 6.75 (s, 1H), 6.64 (s, 2H), 6.43 (s, 1H), 5.73 (s, 1H), 3.71 (s, 3H), 3.68 (s, 3H), 3.50 (br, 2H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$) δ 147.4, 142.2, 137.4, 134.6, 133.8, 133.5, 132.1, 128.8, 127.92, 127.85, 127.6, 127.5, 127.4, 126.9, 125.7, 125.2, 121.5, 121.4, 120.0, 118.7, 118.5, 115.0, 111.6, 109.0, 55.4, 48.4, 32.6 ppm; IR (neat) 3449, 3369, 3048, 2930, 1617, 1516, 1465, 1228, 1145, 1033, 906, 737 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{25}N_2O$: 393.1961 ($[M+H]^+$), found 393.1998 ($[M+H]^+$).

¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2-Naph-oMP-1MeInd¥2-Naph-oMP-1MeInd_PROTON.als
6-3-13-x



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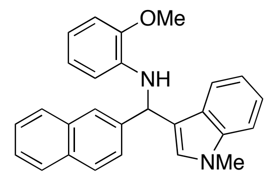
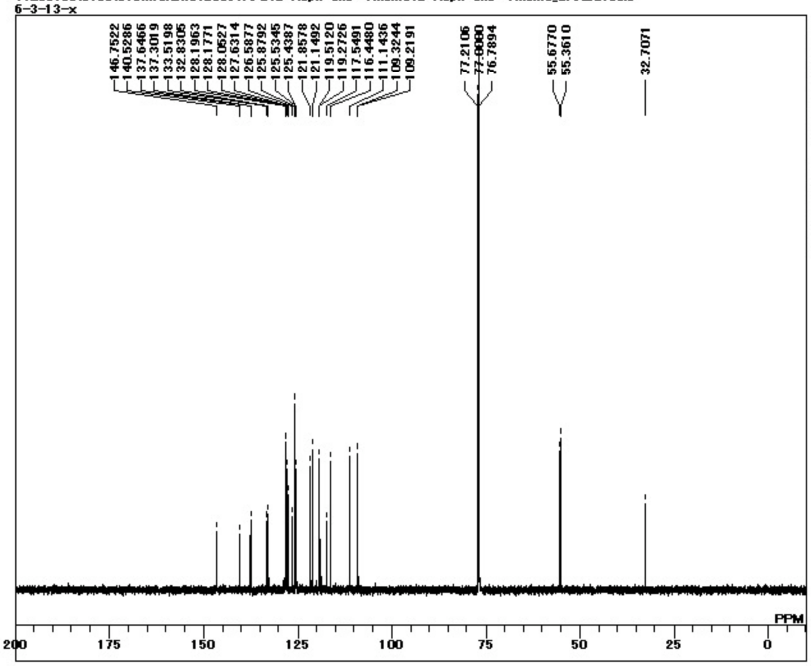
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42



4a

¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2-Naph-oMP-1MeInd¥2-Naph-oMP-1MeInd_CARBON.als
6-3-13-x



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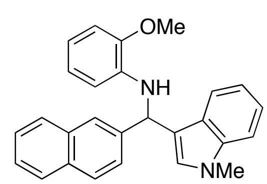
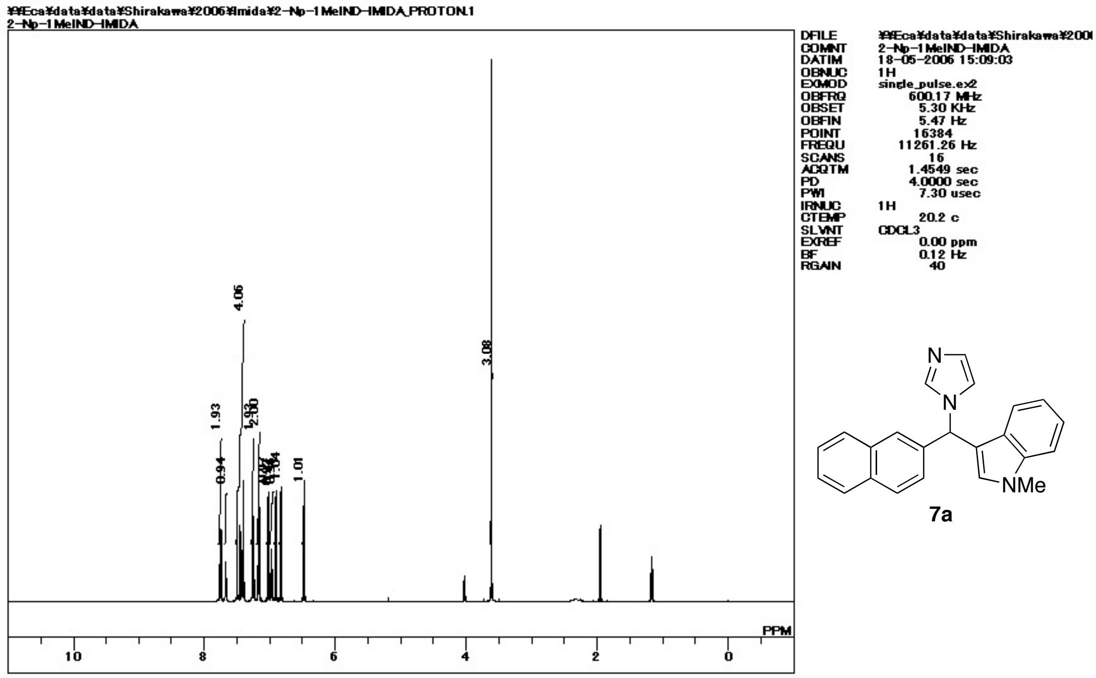
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 406
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



4a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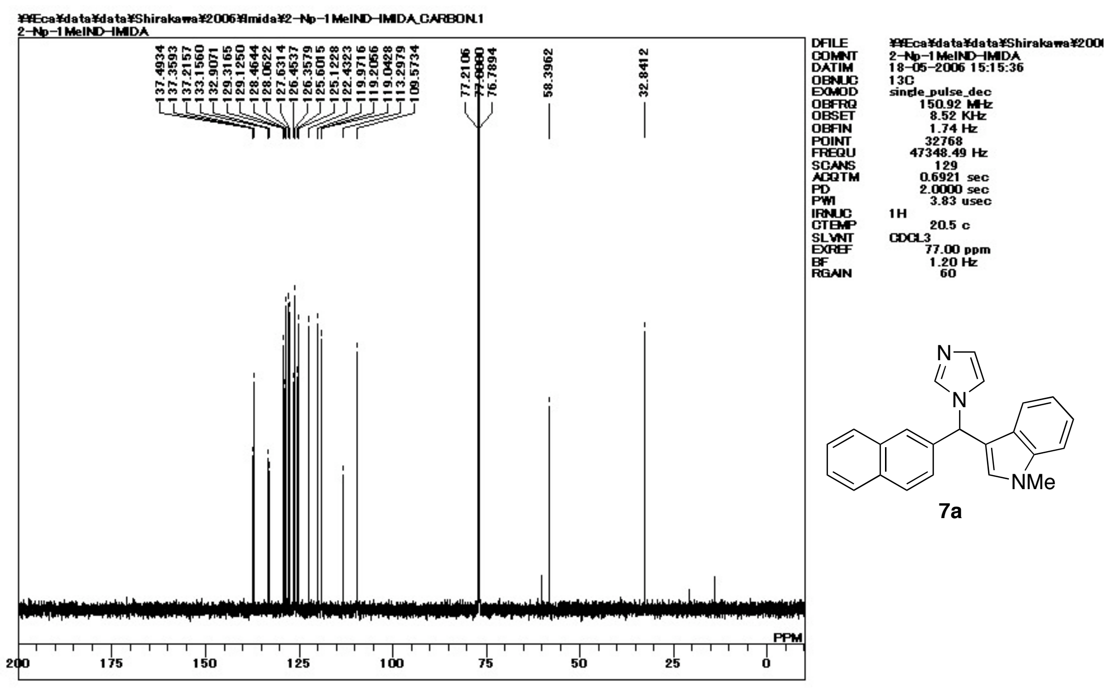
2-Np-1MeIND-IMIDA
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 2-Np-1MeIND-IMIDA
DATIM 18-05-2006 15:09:03
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
4.06
3.08
1.93
2.00
0.94
1.01
PPM
10
8
6
4
2
0
N
N
NMe
7a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2-Np-1MeIND-IMIDA
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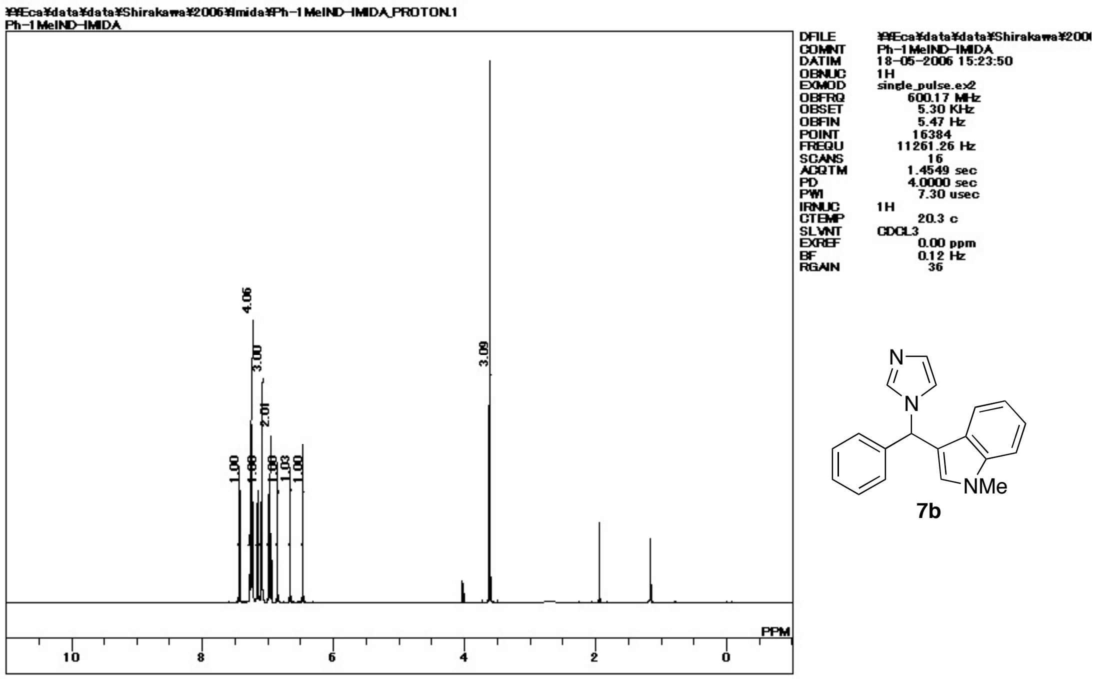
137.2157
133.1560
132.9071
129.3165
129.1250
128.4644
128.0622
127.6314
126.4537
126.3579
125.6015
125.1228
122.4323
119.9716
119.2056
119.0428
113.2979
109.5734
77.2106
77.0000
76.7894
58.3962
32.8412
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 2-Np-1MeIND-IMIDA
DATIM 18-05-2006 15:15:36
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 129
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0
N
N
NMe
7a



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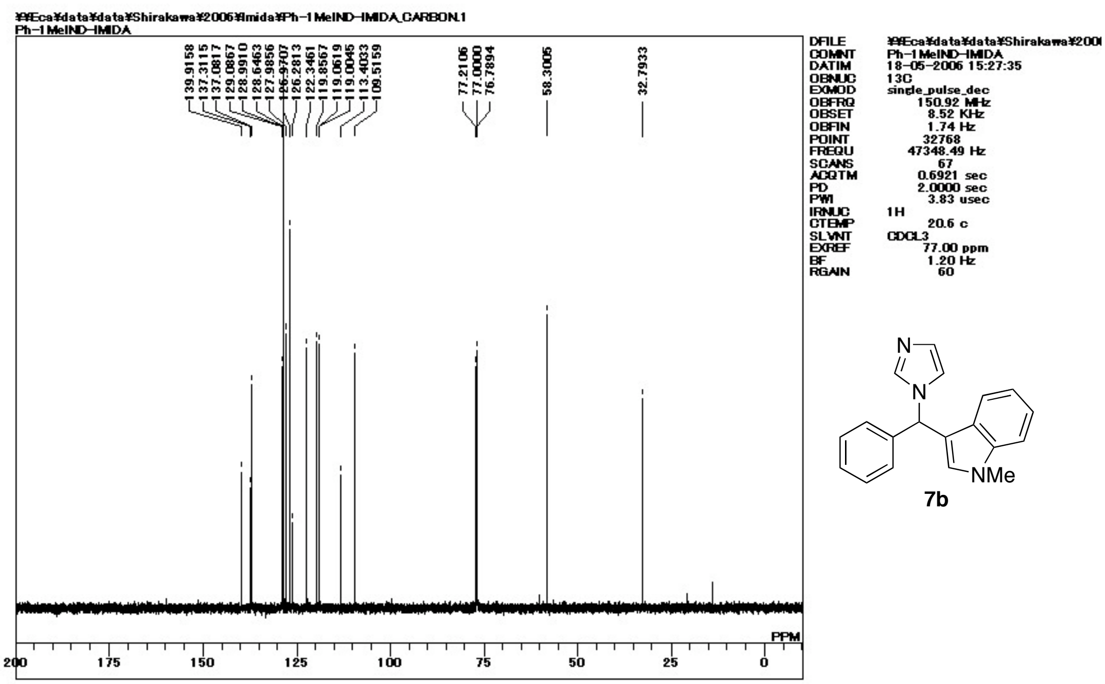

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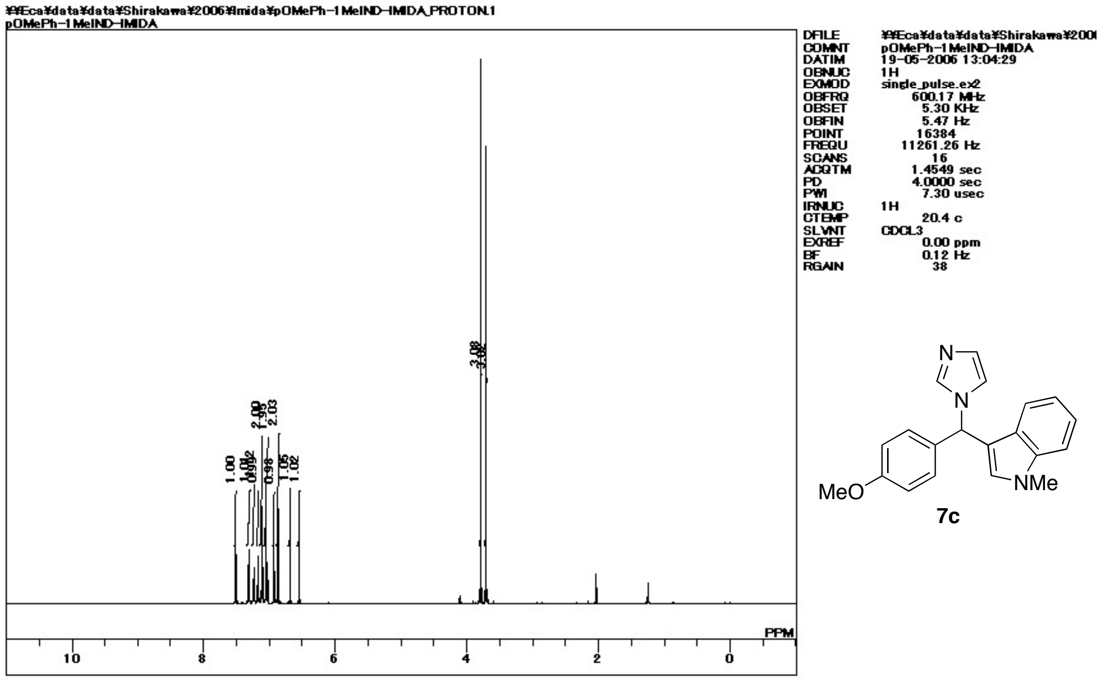
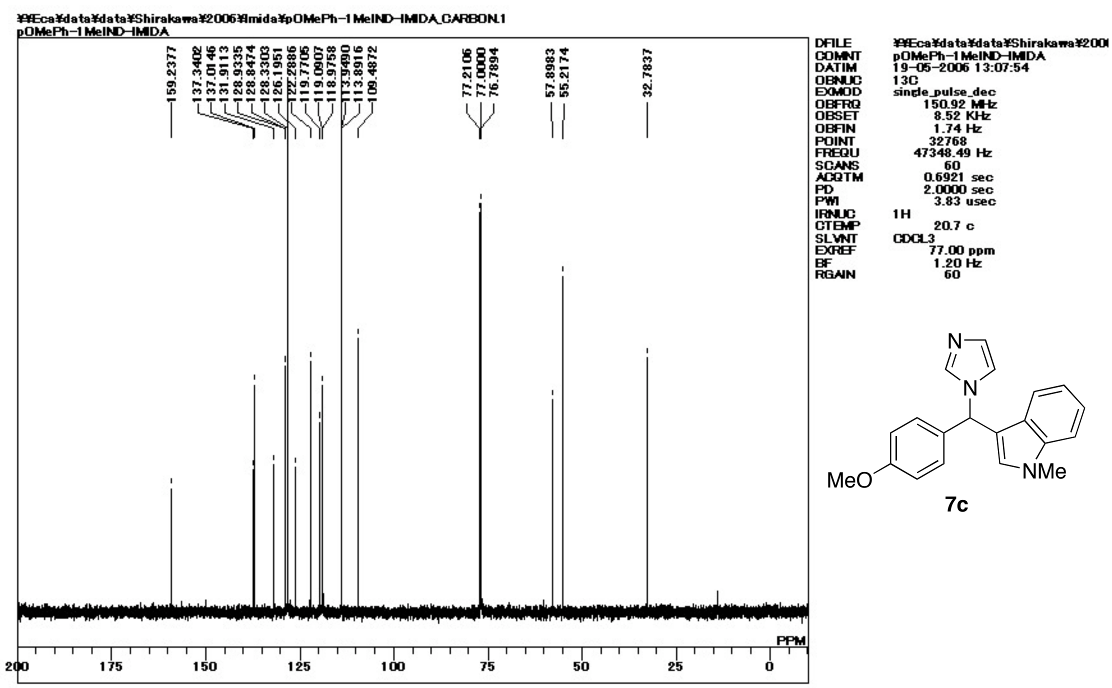


¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥pOMePh-1MeIND-IMIDA_PROTON.1
pOMePh-1MeIND-IMIDA
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT pOMePh-1MeIND-IMIDA
DATIM 19-05-2006 13:04:29
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
1.00
2.00
1.95
2.03
0.98
1.05
1.02
3.08
PPM
10
8
6
4
2
0
MeO
N
NMe
7c



¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥pOMePh-1MeIND-IMIDA_CARBON.1
pOMePh-1MeIND-IMIDA
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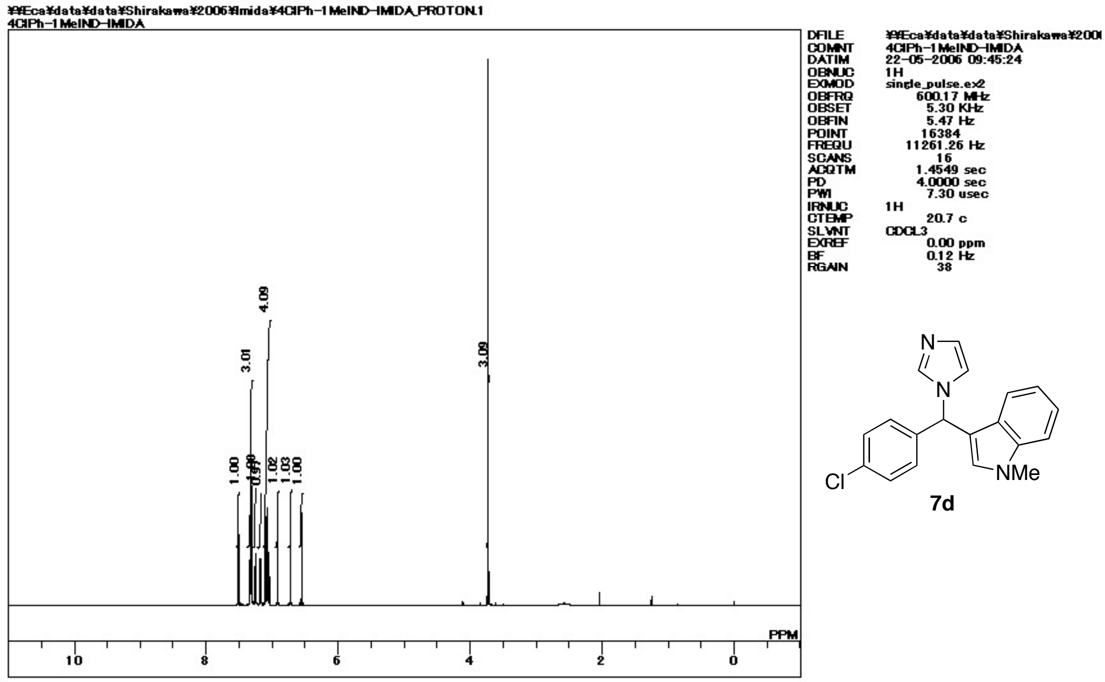
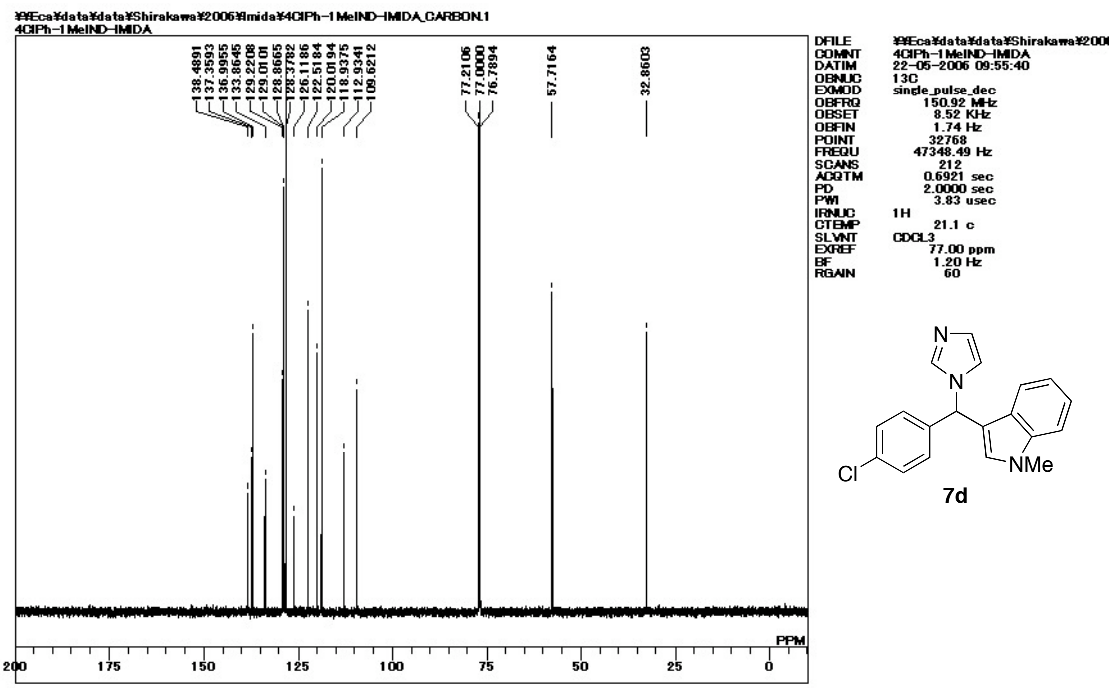
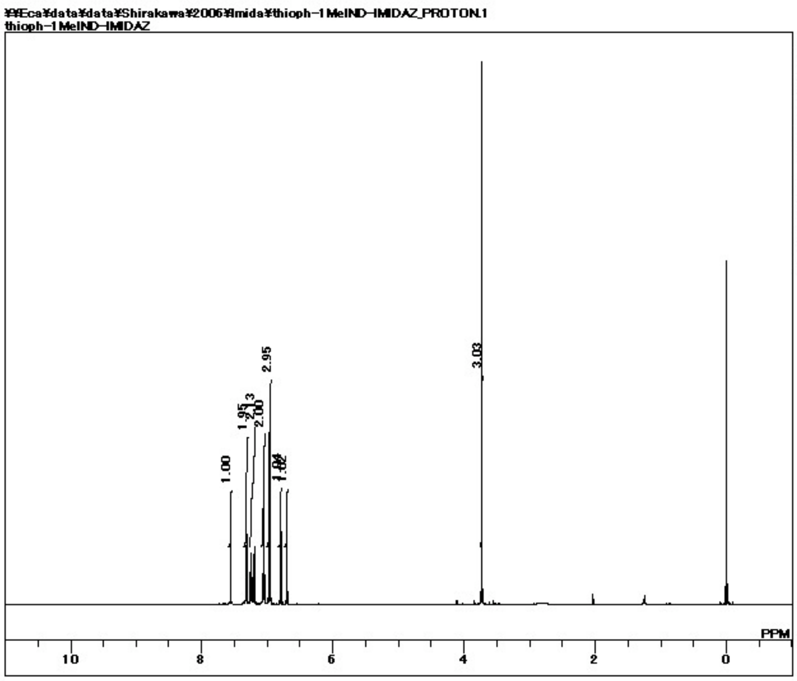
137.3402
137.0146
131.9113
128.9335
128.8474
128.3303
126.1951
122.2886
119.7705
119.0907
118.9758
113.9490
113.8916
109.4872
77.2106
77.0000
76.7894
57.8983
55.2174
32.7837
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT pOMePh-1MeIND-IMIDA
DATIM 19-05-2006 13:07:54
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 60
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0
MeO
N
NMe
7c






¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥thioph-1MeIND-IMIDAZ_PROTON.1
thioph-1MeIND-IMIDAZ
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT thioph-1MeIND-IMIDAZ
DATIM 25-05-2006 09:43:10
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
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1.95
2.13
2.00
2.95
1.04
1.02
3.03
PPM
10
8
6
4
2
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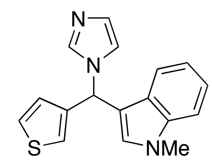
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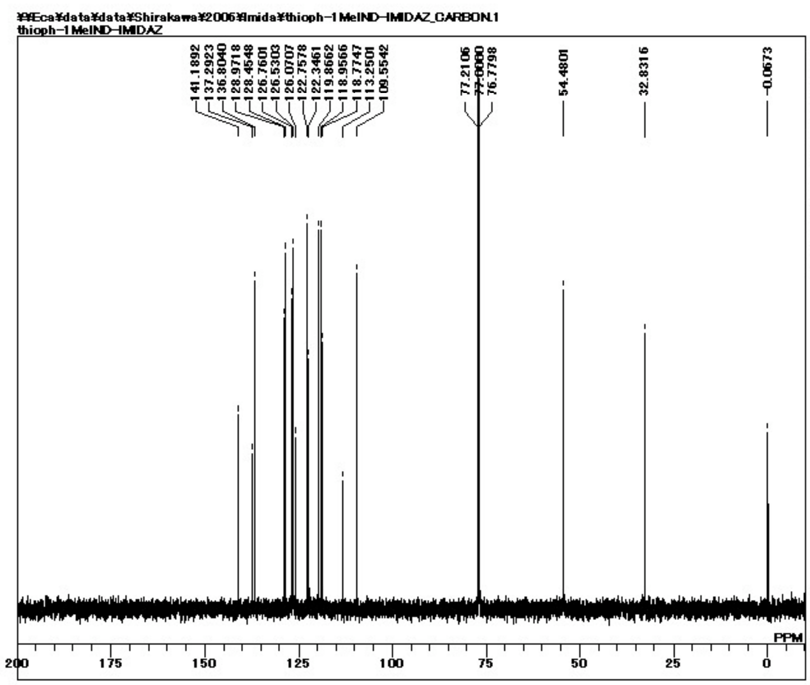
S
NMe

7e


¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥thioph-1MeIND-IMIDAZ_CARBON.1
thioph-1MeIND-IMIDAZ
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136.8040
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128.4548
126.7601
126.5303
126.0707
122.7578
122.3461
119.8662
118.9566
118.7747
113.2501
109.5542
77.2106
77.0000
76.7798
54.4801
32.8316
-0.0673
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT thioph-1MeIND-IMIDAZ
DATIM 25-05-2006 09:46:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 61
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
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BF 1.20 Hz
RGAIN 60
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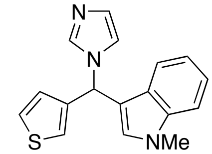
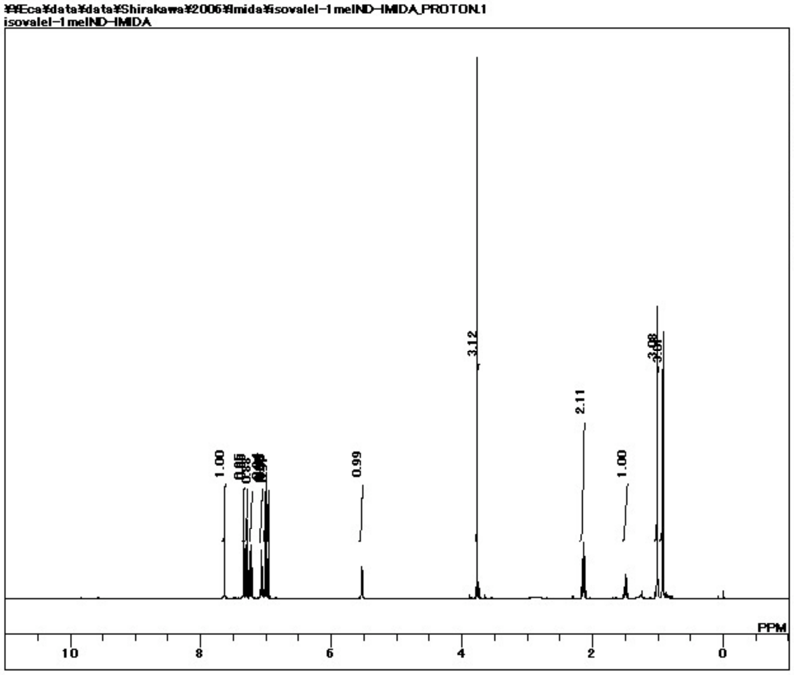
100
75
50
25
0


N
N
S
NMe

7e


¥¥Eca¥data¥data¥Shirakawa¥2006¥imida¥isovalel-1meIND-IMIDA_PROTON.1
isovalel-1meIND-IMIDA
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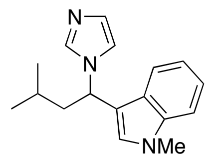
COMNT isovalel-1meIND-IMIDA
DATIM 06-06-2006 16:40:47
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
1.00
0.99
3.12
2.11
1.00
PPM
10
8
6
4
2
0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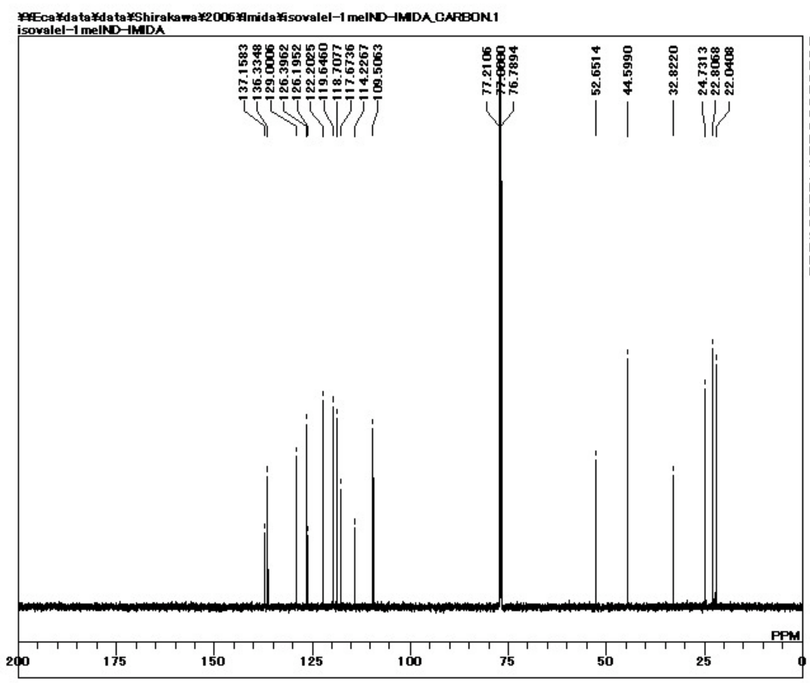
N
NMe

7f


¥¥Eca¥data¥data¥Shirakawa¥2006¥imida¥isovalel-1meIND-IMIDA_CARBON.1
isovalel-1meIND-IMIDA
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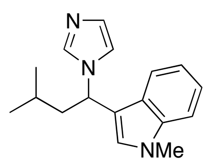
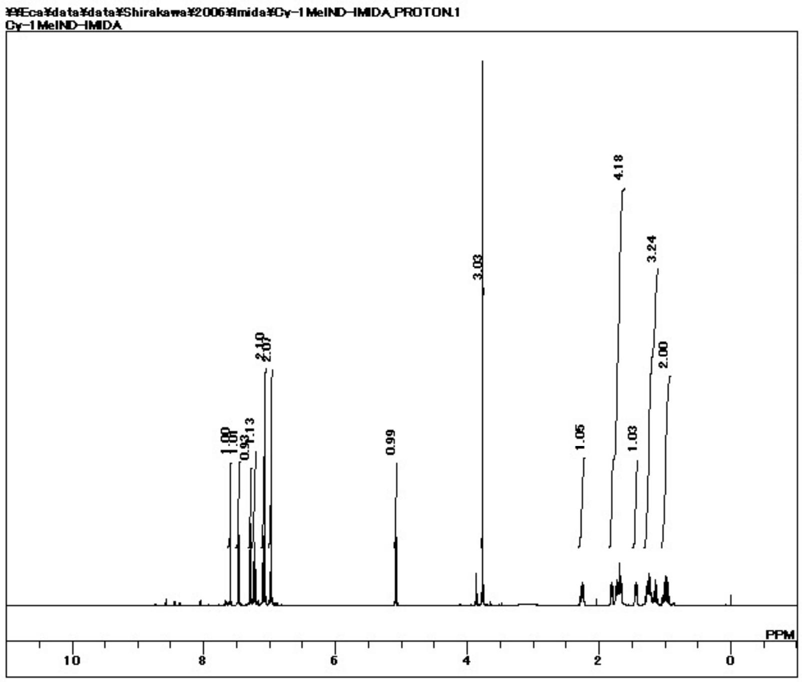
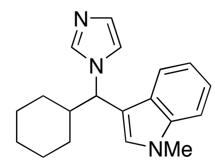
136.3348
129.0006
126.3962
126.1952
122.2025
119.6460
118.7077
117.6736
114.2267
109.5063
77.2106
76.7894
52.6514
44.5990
32.8220
24.7313
22.8068
22.0408
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT isovalel-1meIND-IMIDA
DATIM 06-06-2006 16:57:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 357
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0


N
N
NMe

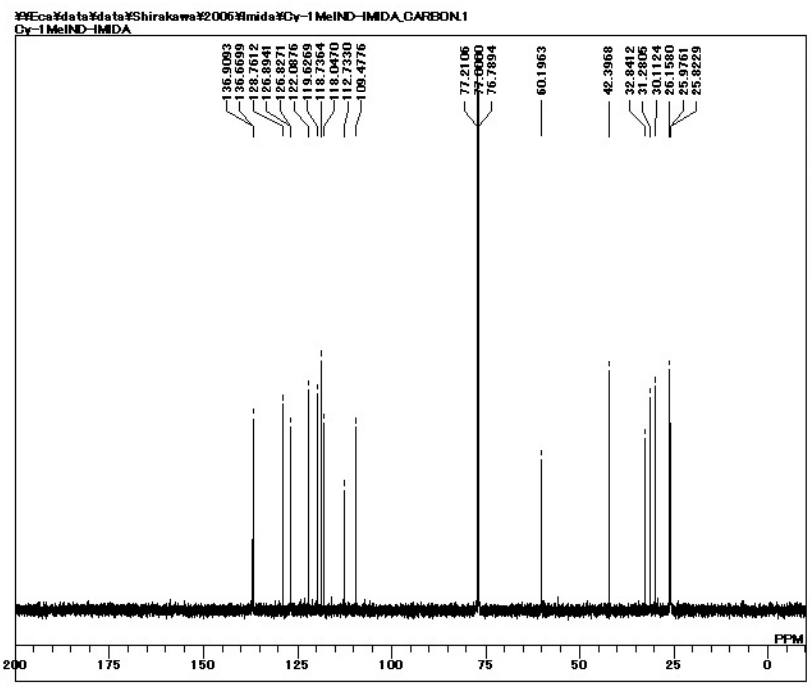
7f



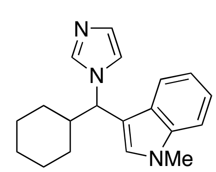
¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥Cy-1MeIND-IMIDA_PROTON.1
Cy-1MeIND-IMIDA
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Cy-1MeIND-IMIDA
DATIM 22-05-2006 10:03:32
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
PPM



7g

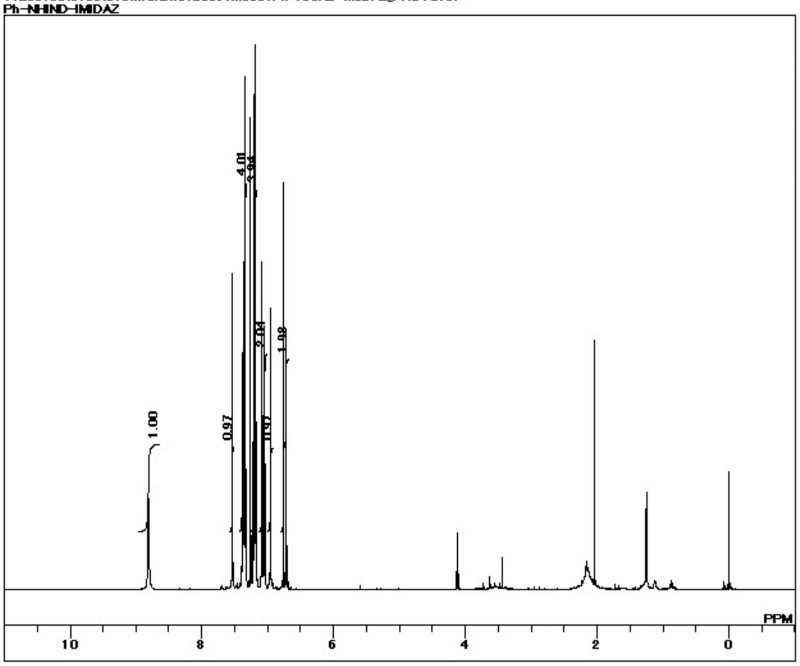


¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥Cy-1MeIND-IMIDA_CARBON.1
Cy-1MeIND-IMIDA
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Cy-1MeIND-IMIDA
DATIM 22-05-2006 10:10:14
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 129
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM

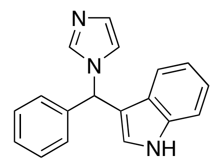


7g

¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥Ph-NHIND-IMIDAZ_PROTON.1
Ph-NHIND-IMIDAZ

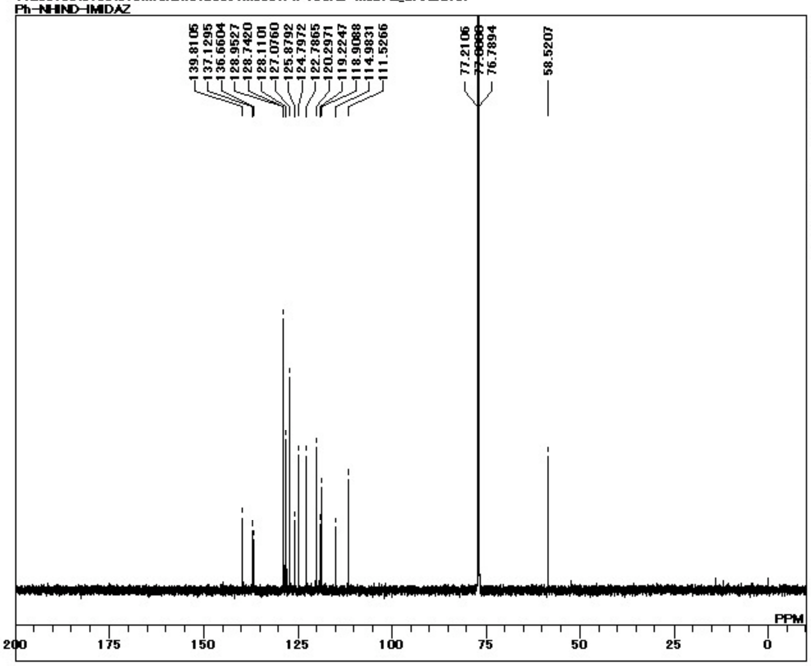


DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-NHIND-IMIDAZ
DATIM 24-05-2006 09:33:13
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

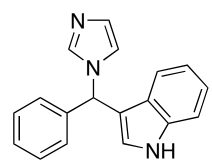


7h

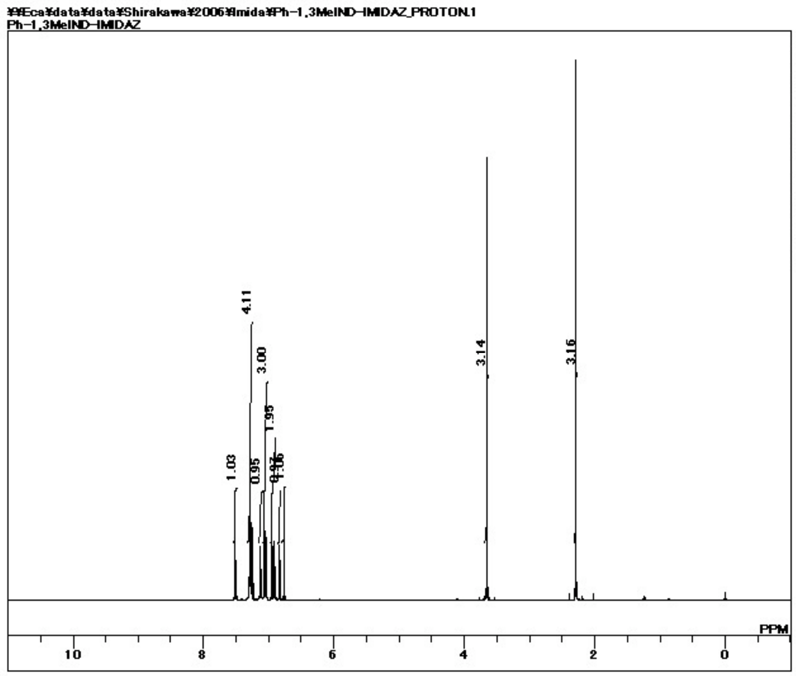
¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥Ph-NHIND-IMIDAZ_CARBON.1
Ph-NHIND-IMIDAZ



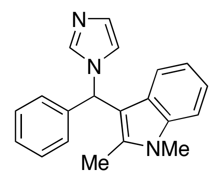
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-NHIND-IMIDAZ
DATIM 24-05-2006 10:06:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 716
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



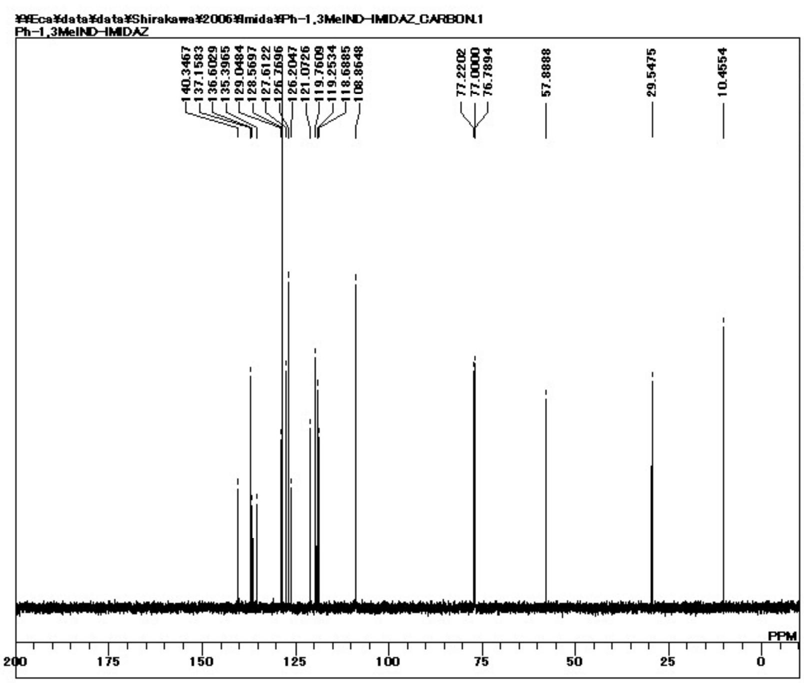
7h



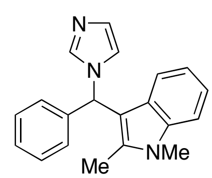
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-1,3MeIND-IMIDAZ
DATIM 24-05-2006 10:13:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36



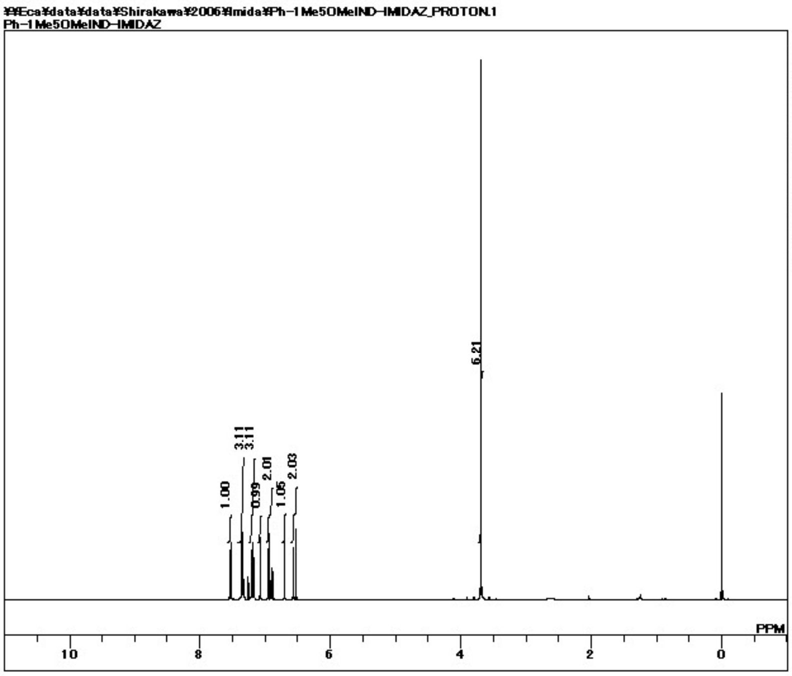
7i

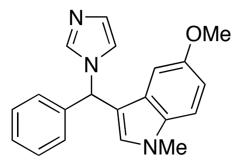


DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-1,3MeIND-IMIDAZ
DATIM 24-05-2006 10:16:52
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 55
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

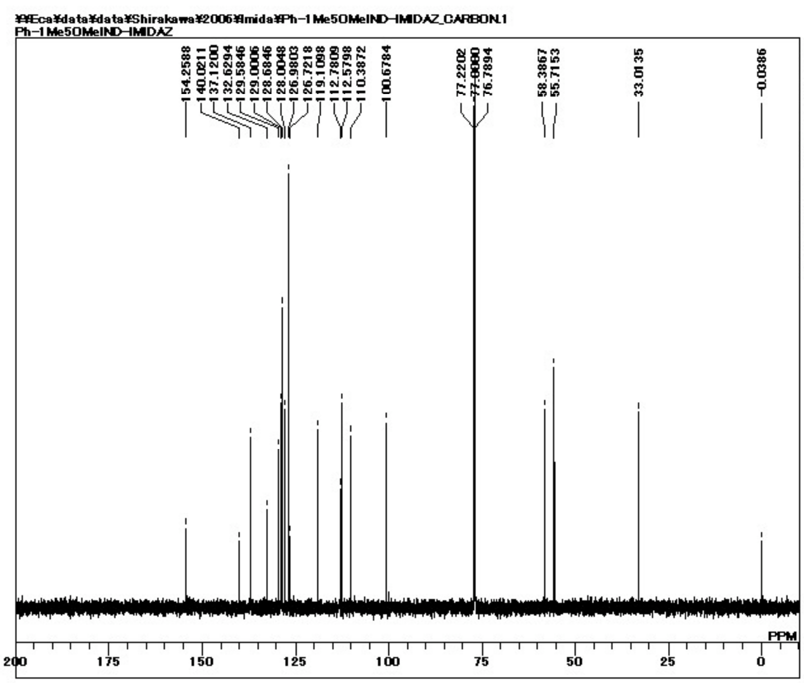


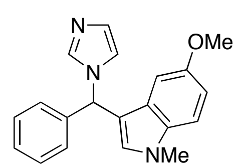
7i


¥¥Eca¥data¥data¥Shirakawa¥2006¥imida¥Ph-1Me5OMeIND-IMIDAZ_PROTON.1
Ph-1Me5OMeIND-IMIDAZ
1.00
3.11
3.11
0.99
2.01
1.05
2.03
6.21
PPM
10
8
6
4
2
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-1Me5OMeIND-IMIDAZ
DATIM 25-05-2006 09:55:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

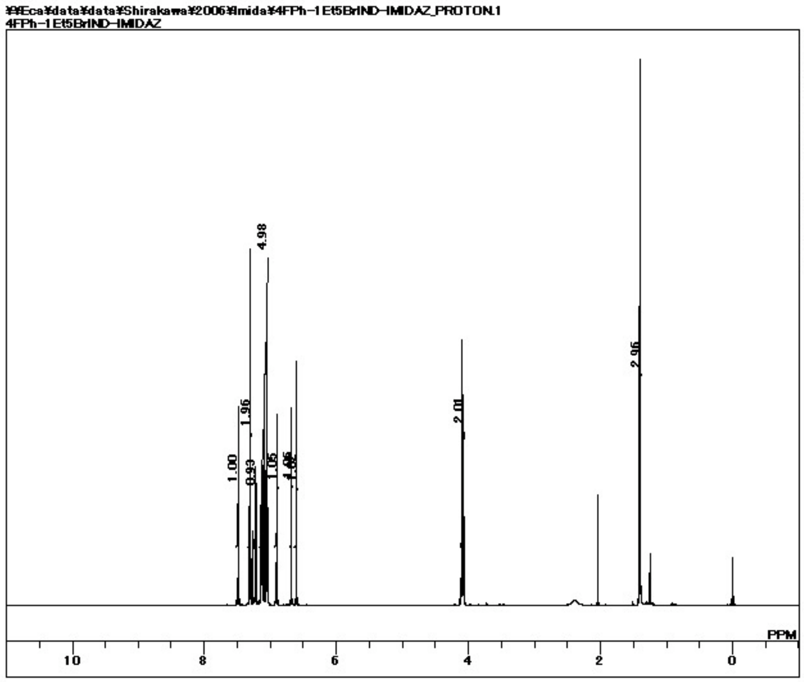

N
N
OMe
NMe

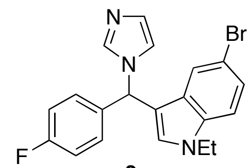
7j


¥¥Eca¥data¥data¥Shirakawa¥2006¥imida¥Ph-1Me5OMeIND-IMIDAZ_CARBON.1
Ph-1Me5OMeIND-IMIDAZ
154.2588
140.0211
137.1200
132.6294
129.5846
129.0006
128.6846
128.0048
126.9803
126.7218
119.1098
112.7809
112.5798
110.3872
100.6784
77.2202
77.0080
76.7894
58.3867
55.7153
33.0135
-0.0386
PPM
200
175
150
125
100
75
50
25
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT Ph-1Me5OMeIND-IMIDAZ
DATIM 25-05-2006 10:00:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 91
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

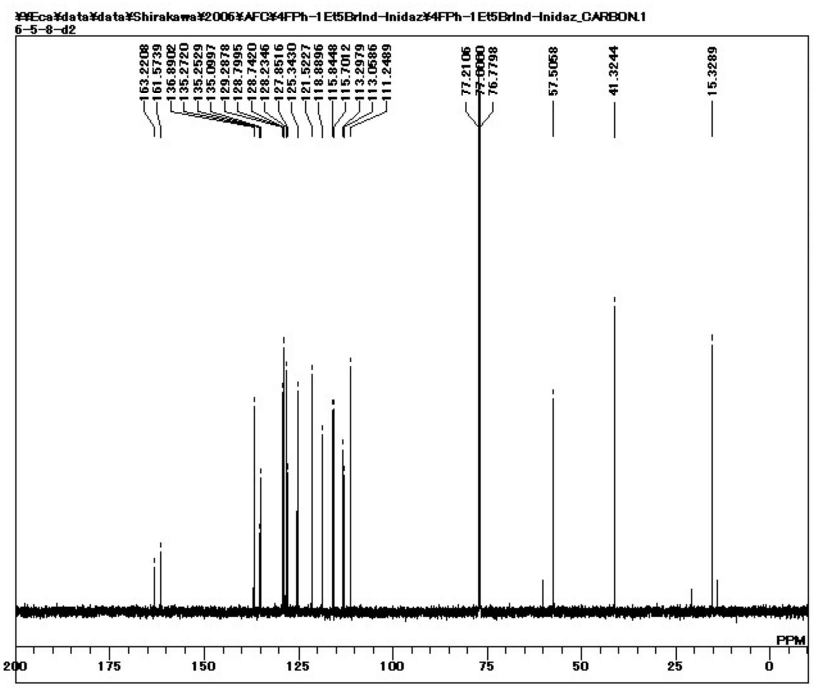

N
N
OMe
NMe

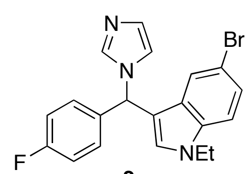
7j


¥¥Eca¥data¥data¥Shirakawa¥2006¥Imida¥4FPh-1Et5BrIND-IMIDAZ_PROTON.1
4FPh-1Et5BrIND-IMIDAZ
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 4FPh-1Et5BrIND-IMIDAZ
DATIM 26-05-2006 09:49:19
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
1.00
0.93
1.96
1.05
4.98
1.06
2.01
2.96
PPM
10
8
6
4
2
0


N
N
Br
F
NEt

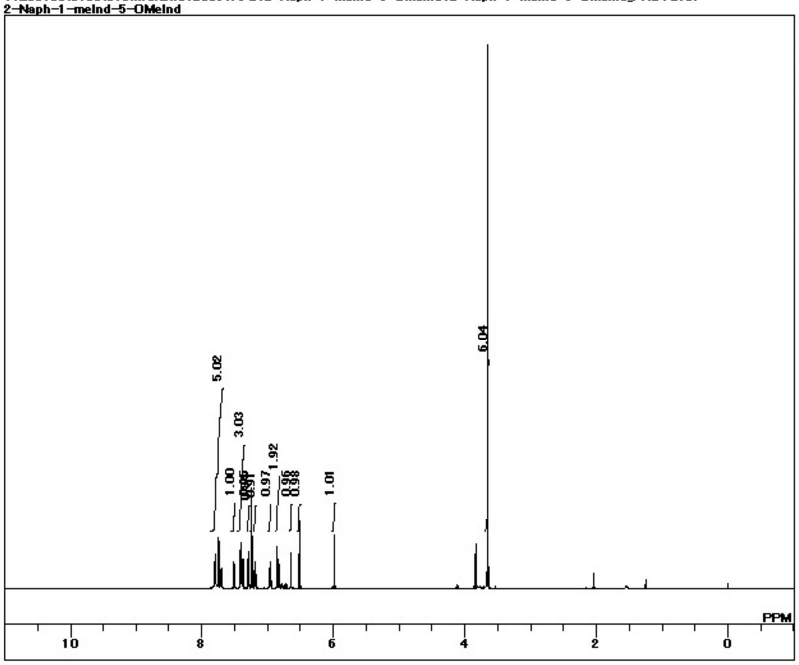
2


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥4FPh-1Et5BrInd-Inidaz¥4FPh-1Et5BrInd-Inidaz_CARBON.1
6-5-8-d2
163.2208
161.5739
136.8902
135.2720
135.2529
135.0997
129.2878
128.7995
128.7420
128.2346
127.8516
125.3430
121.5227
118.8896
115.8448
115.7012
113.2979
113.0586
111.2489
77.2106
77.0000
76.7798
57.5058
41.3244
15.3289
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-5-8-d2
DATIM 10-04-2006 15:31:18
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 202
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0

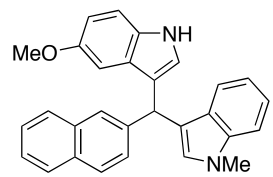

N
N
Br
F
NEt

2

¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2-Naph-1-meInd-5-OMeInd¥2-Naph-1-meInd-5-OMeInd_PROTON.1
2-Naph-1-meInd-5-OMeInd

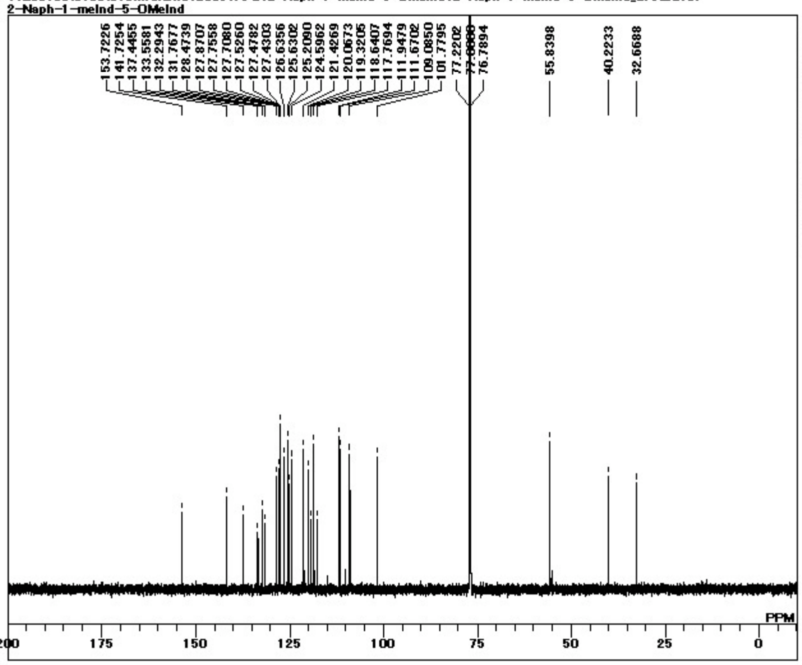


DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 2-Naph-1-meInd-5-OMeInd
DATIM 21-03-2006 16:57:24
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

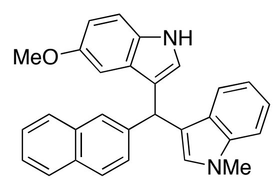


8

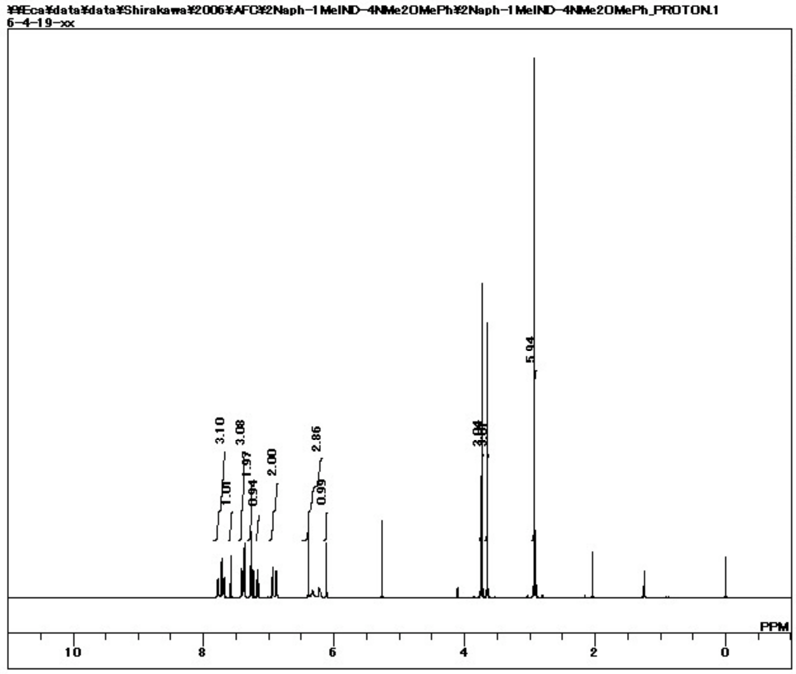
¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2-Naph-1-meInd-5-OMeInd¥2-Naph-1-meInd-5-OMeInd_CARBON.1
2-Naph-1-meInd-5-OMeInd

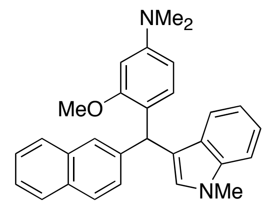


DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 2-Naph-1-meInd-5-OMeInd
DATIM 21-03-2006 17:34:03
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 721
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

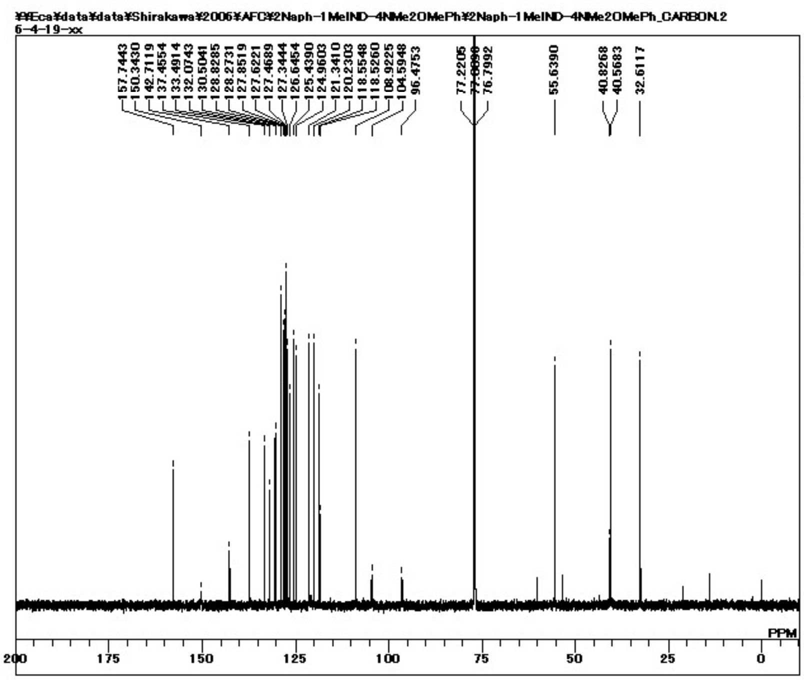


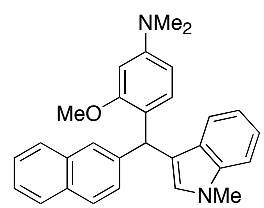
8


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-4NMe2OMePh¥2Naph-1MeIND-4NMe2OMePh_PROTON.1
6-4-19-xx
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-19-xx
DATIM 19-04-2006 12:47:19
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
3.10
1.01
3.08
1.97
0.94
2.00
2.86
0.99
3.04
3.01
5.94
PPM
10
8
6
4
2
0

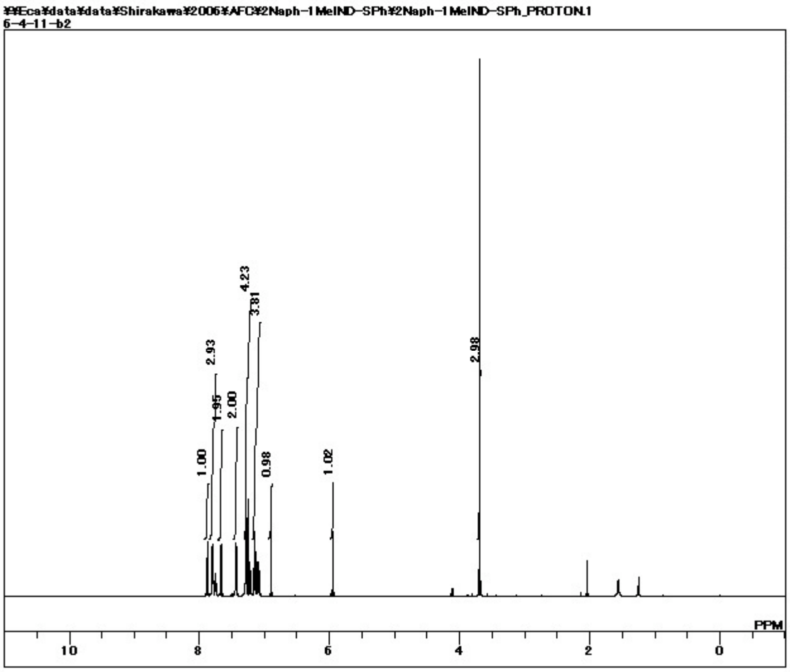

NMe2
MeO
NMe

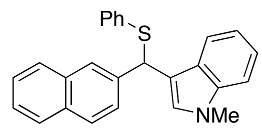
9


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-4NMe2OMePh¥2Naph-1MeIND-4NMe2OMePh_CARBON.2
6-4-19-xx
157.7443
150.3430
142.7119
137.4554
133.4914
132.0743
130.5041
128.8285
128.2731
127.8519
127.6221
127.4689
127.3444
126.6454
125.4390
124.9603
121.3410
120.2303
118.5548
118.5260
108.9225
104.5948
96.4753
77.2205
77.0098
76.7992
55.6390
40.8268
40.5683
32.6117
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-19-xx
DATIM 19-04-2006 13:45:18
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 1024
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 60
PPM
200
175
150
125
100
75
50
25
0

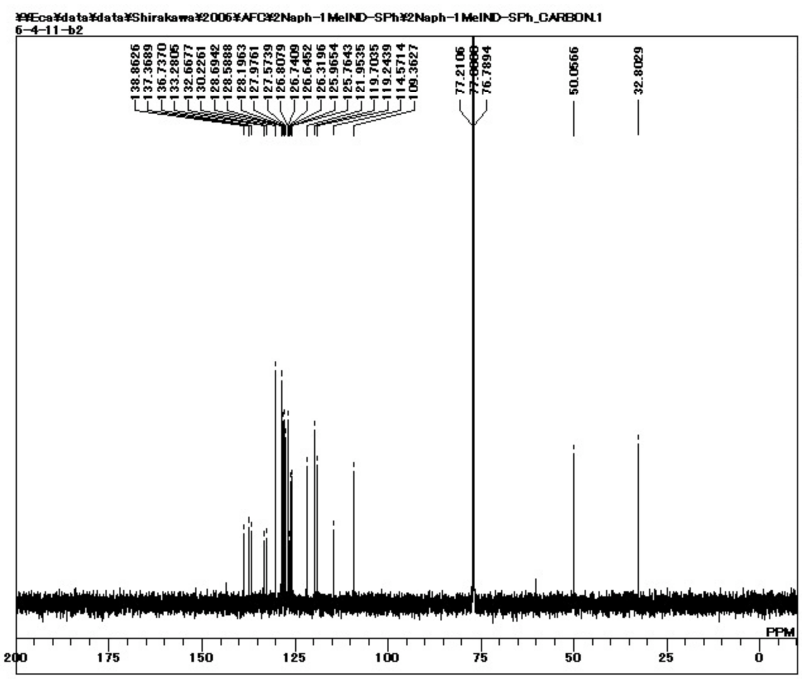

NMe2
MeO
NMe

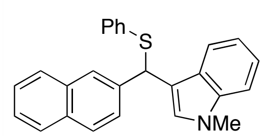
9


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-SPh¥2Naph-1MeIND-SPh_PROTON.1
6-4-11-b2
1.00
1.95
2.93
2.00
4.23
3.81
0.98
1.02
2.98
PPM
10
8
6
4
2
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-11-b2
DATIM 13-04-2006 14:50:23
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

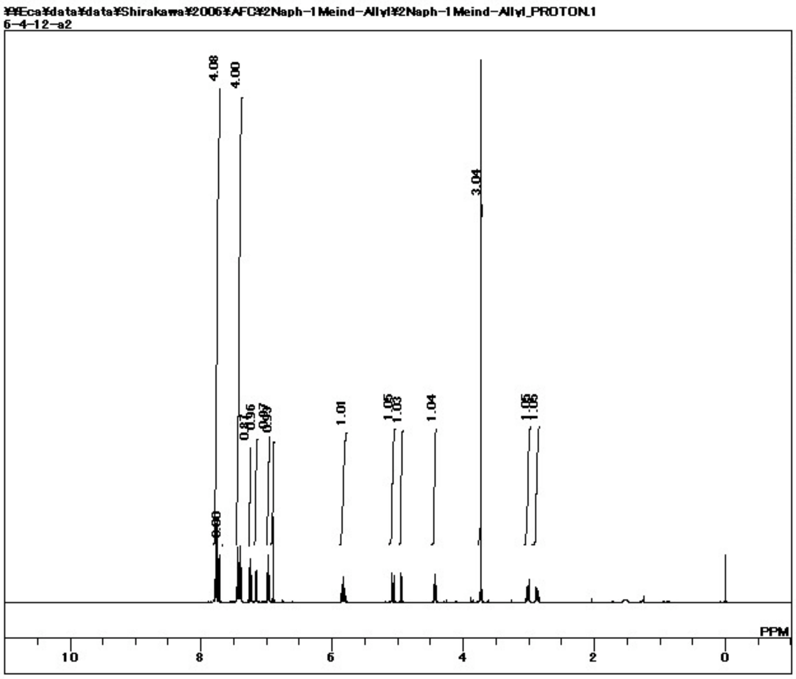

Ph
S
NMe

10

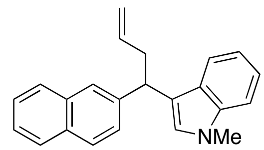

¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-SPh¥2Naph-1MeIND-SPh_CARBON.1
6-4-11-b2
138.8626
137.3689
136.7370
133.2805
132.6677
130.2261
128.6942
128.5888
128.1963
127.9761
127.5739
126.8079
126.7409
126.6452
126.3196
125.9654
125.7643
121.9535
119.7035
119.2439
114.5714
109.3627
77.2106
77.0000
76.7894
50.0566
32.8029
PPM
200
175
150
125
100
75
50
25
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-11-b2
DATIM 13-04-2006 15:17:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 198
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60


Ph
S
NMe

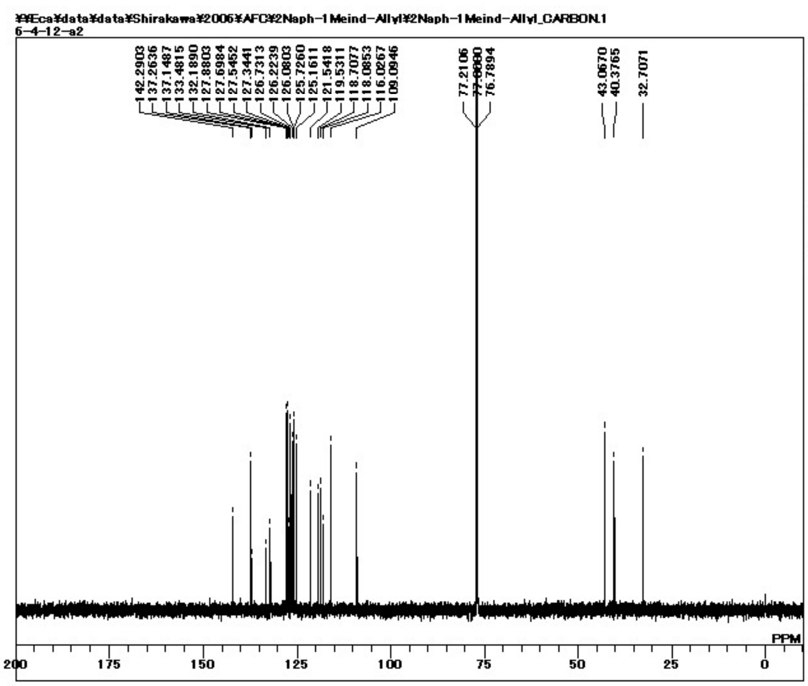
10



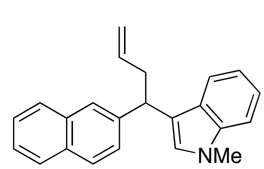
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-12-a2
DATIM 13-04-2006 21:53:51
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



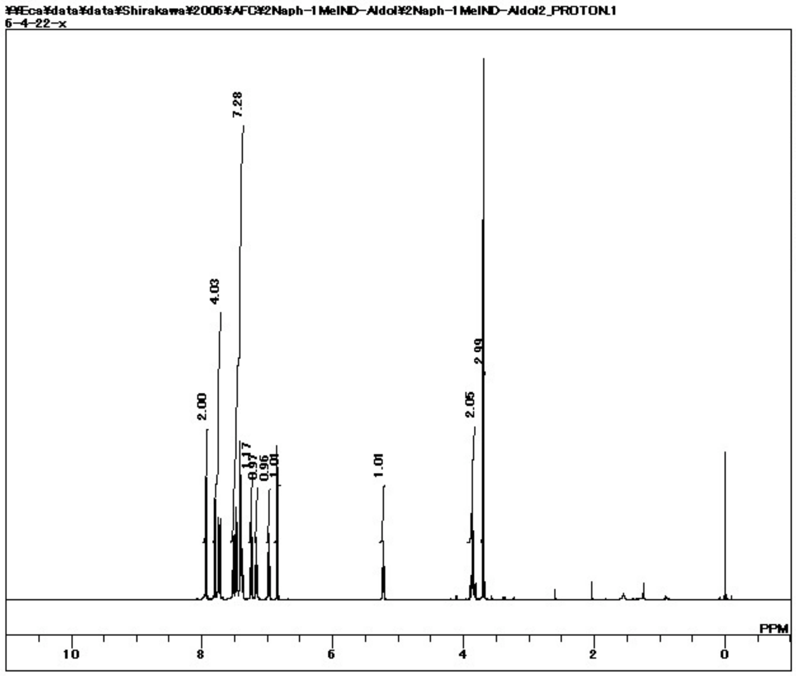
11

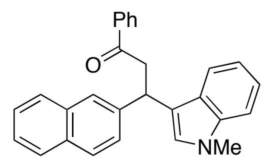


DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-12-a2
DATIM 13-04-2006 22:33:36
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 91
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

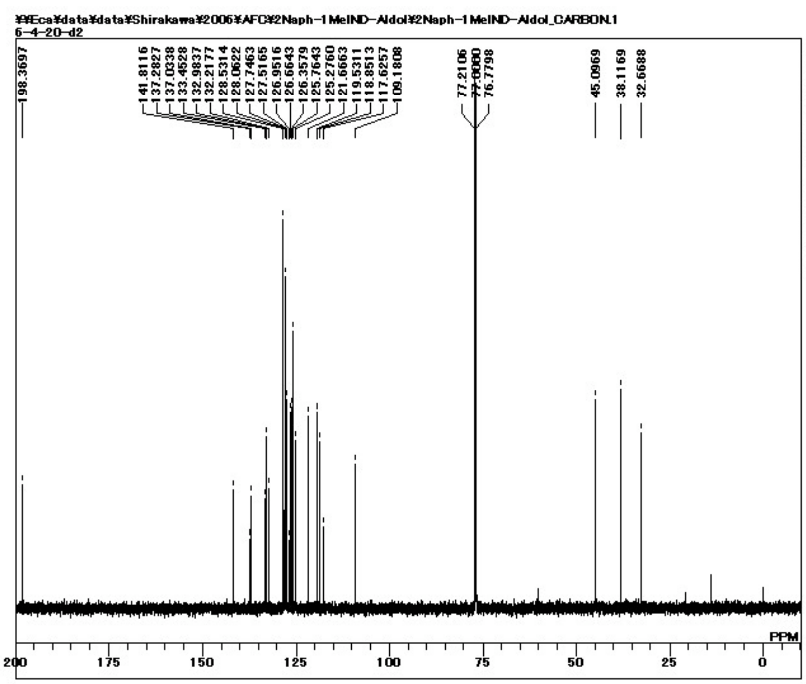


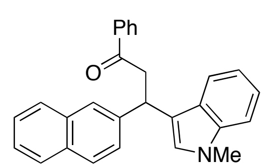
11


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-Aldol¥2Naph-1MeIND-Aldol2_PROTON.1
6-4-22-x
7.28
4.03
2.00
1.17
0.97
0.96
1.01
1.01
2.05
2.99
PPM
10
8
6
4
2
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-22-x
DATIM 22-04-2006 15:34:19
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

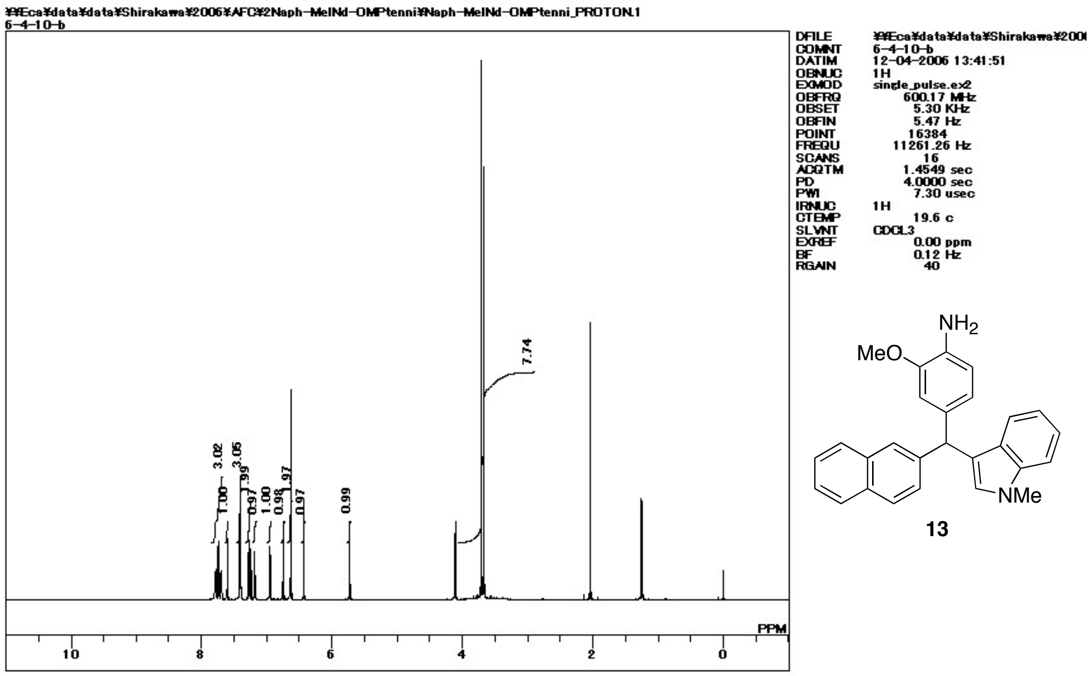

Ph
O
NMe

12


¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-1MeIND-Aldol¥2Naph-1MeIND-Aldol_CARBON.1
6-4-20-d2
198.3697
141.8116
137.2827
137.0338
133.4528
132.9837
132.2177
128.5314
128.0622
127.7463
127.5165
126.9516
126.6643
126.3579
125.7643
125.2760
121.6663
119.5311
118.8513
117.6257
109.1808
77.2106
77.0000
76.7798
45.0969
38.1169
32.6688
PPM
200
175
150
125
100
75
50
25
0
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-20-d2
DATIM 21-04-2006 22:48:46
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 148
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

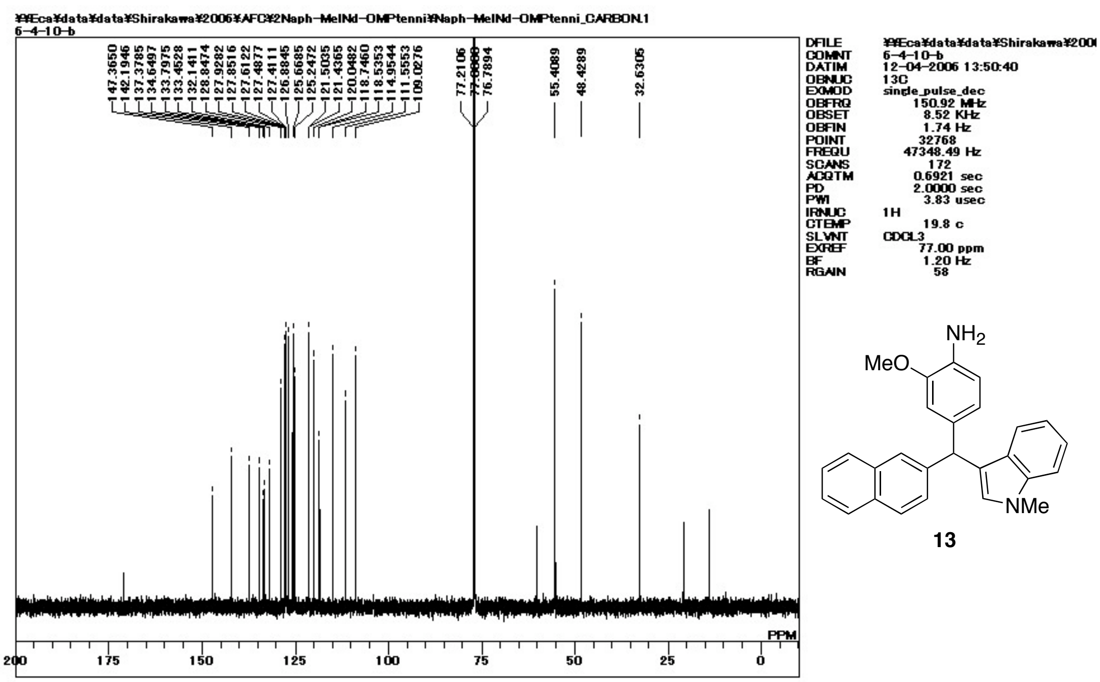

Ph
O
NMe

12



¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-MeInd-OMPtenni¥Naph-MeInd-OMPtenni_PROTON.1
6-4-10-b
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-10-b
DATIM 12-04-2006 13:41:51
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
3.02
1.00
3.05
.99
0.97
1.00
0.98
.97
0.97
0.99
7.74
PPM
10
8
6
4
2
0
NH2
MeO
NMe

13



¥¥Eca¥data¥data¥Shirakawa¥2006¥AFC¥2Naph-MeInd-OMPtenni¥Naph-MeInd-OMPtenni_CARBON.1
6-4-10-b
147.3650
142.1946
137.3785
134.6497
133.7975
133.4528
132.1411
128.8474
127.9282
127.8516
127.6122
127.4877
127.4111
126.8845
125.6685
125.2472
121.5035
121.4365
120.0482
118.7460
118.5353
114.9544
111.5553
109.0276
77.2106
76.7894
55.4089
48.4289
32.6305
DFILE ¥¥Eca¥data¥data¥Shirakawa¥200
COMNT 6-4-10-b
DATIM 12-04-2006 13:50:40
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.49 Hz
SCANS 172
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58
PPM
200
175
150
125
100
75
50
25
0
NH2
MeO
NMe

13