Carbazoles via $AuCl_3$-Catalyzed Cyclization of 1-(Indol-2-yl)-3-alkyn-1-ols

Youai Qiu, Wangqing Kong, Chunling Fu, and Shengming Ma*

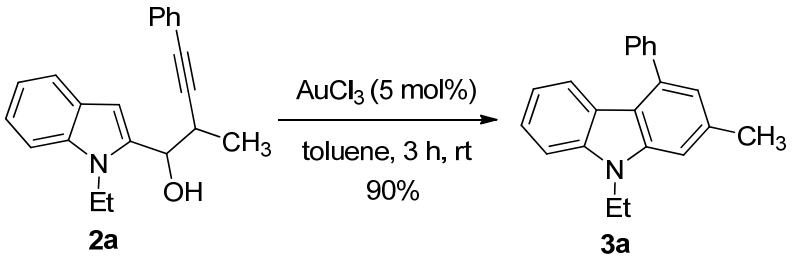
Laboratory of Molecular Recognition and Synthesis, Department of Chemistry, Zhejiang University, Hangzhou 310027, Zhejiang, P. R. China

E-mail: masm@sioc.ac.cn Fax: (+86) 21-64167510

Supporting Information

General: ^{1}H and ^{13}C NMR spectra were recorded with a Bruker AM 300 MHz. IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were recorded with a Carlo-Erba EA1110 elementary analysis instrument. Mass spectrometry was performed with an HP 5989A system. High-resolution mass data were determined with a Finnigan MAT 8430 or Bruker APEXIII instrument. Toluene was distilled from Na/benzophenone before use. Unless otherwise indicated, chemicals and solvents were purchased from commercial suppliers.

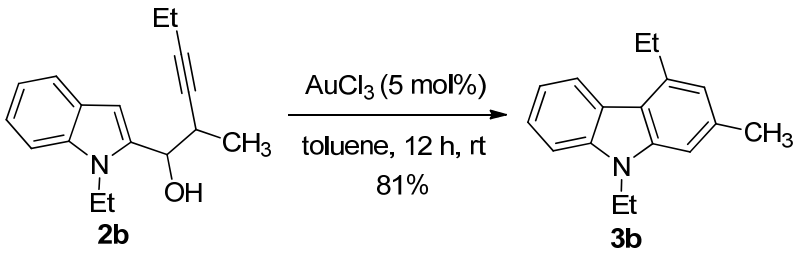
(1) **9-Ethyl-2-methyl-4-phenyl-9*H*-carbazole** (**3a**)



Typical Procedure: To a dry Schlenk tube were added sequentially $AuCl_3$ (3.1 mg, 0.01 mmol), **2a** (60.6 mg, 0.2 mmol), and toluene (1.0 mL) under N_2. After continuous stirring for 3 h at rt, the reaction was complete as monitored by TLC. Filtration, evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate = 40/l) afforded **3a** (51.3 mg, 90%): solid; m. p. 92-93 °C (ethyl acetate/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68-7.57 (m, 2H, ArH), 7.56-7.43 (m, 4H, ArH), 7.41-7.31 (m, 2H, ArH), 7.21 (s, 1H, ArH), 7.02-6.90 (m, 2H, ArH), 4.38 (q, *J* = 7.2 Hz, 2H, NCH_2), 2.59 (s, 3H, $ArCH_3$), 1.46 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.4, 140.6, 140.1, 137.5, 135.5, 129.1, 128.3, 127.3, 124.9, 122.5, 122.1, 122.0, 118.3, 118.0, 108.1, 107.5, 37.3, 22.1, 13.7; IR (KBr) ν (cm^{-1}) 3052, 3025, 2974, 2914, 2865, 1621, 1599, 1571, 1470, 1460, 1418, 1348, 1324, 1282, 1190, 1121, 1084, 1028; MS (70 ev, EI) *m/z* (%) 286 (M^++1, 18.69), 285 (M^+, 84.44), 270 (100); Elemental analysis calcd (%) for $C_{21}H_{19}N$: C, 88.38; H, 6.71; N, 4.91; Found: C, 88.34, H, 6.87; N, 4.86.

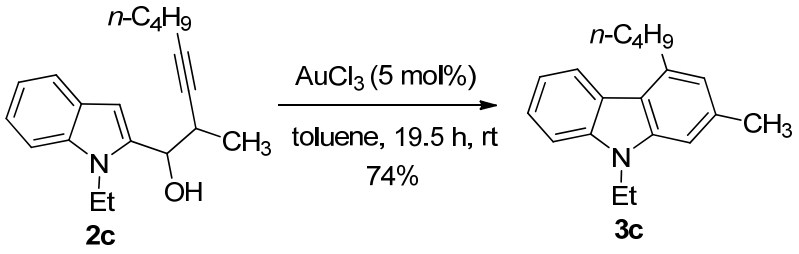
The following compounds **3b-3l** were prepared according to this procedure.

(2) **4,9-Diethyl-2-methyl-9*H*-carbazole (3b)**



The reaction of $AuCl_3$ (3.5 mg, 0.012 mmol) and **2b** (51.1 mg, 0.20 mmol) in toluene (1.0 mL) at rt for 12 h afforded **3b** (38.3 mg, 81%) (petroleum ether/ethyl acetate = 80/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.1 Hz, 1H, ArH), 7.54-7.41 (m, 2H, ArH), 7.37-7.23 (m, 1H, ArH), 7.13 (s, 1H, ArH), 6.94 (s, 1H, ArH), 4.38 (q, *J* = 7.2 Hz, 2H, NCH_2), 3.29 (q, *J* = 7.5 Hz, 2H, $ArCH_2$), 2.61 (s, 3H, $ArCH_3$), 1.60-1.40 (m, 6H, 2×CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.6, 139.8, 139.4, 135.7, 124.4, 122.9, 122.3, 120.1, 118.6, 118.3, 108.1, 106.3, 37.3, 27.3, 22.2, 14.2, 13.7; IR (neat) ν (cm^{-1}) 2970, 2943, 2877, 1618, 1599, 1570, 1468, 1458, 1432, 1374, 1350, 1327, 1258, 1189, 1156, 1080; MS (70 ev, EI) *m/z* (%) 238 (M^++1, 17.18), 237 (M^+, 78.06), 222 (100); HRMS Calcd for $C_{17}H_{19}N$ (M^+): 237.1517, Found: 237.1519.

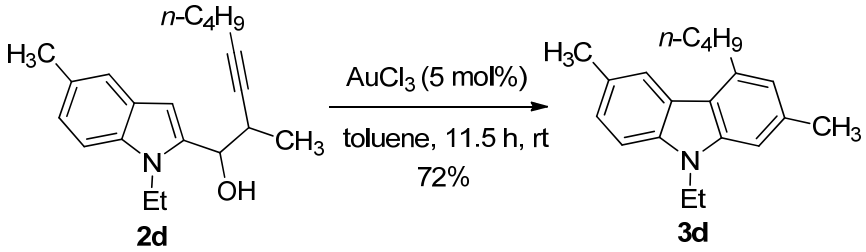
(3) **4-Butyl-9-ethyl-2-methyl-9*H*-carbazole (3c)**



The reaction of $AuCl_3$ (4.6 mg, 0.015 mmol) and **2c** (83.5 mg, 0.30 mmol) in toluene (2.0 mL) at rt for 19.5 h afforded **3c** (57.8 mg, 74%) (petroleum ether/ethyl acetate = 60/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.8 Hz, 1H, ArH), 7.47-7.31 (m, 2H, ArH), 7.27-7.17 (m, 1H, ArH), 7.05 (s, 1H, ArH), 6.85 (s, 1H, ArH),

4.31 (q, J = 7.4 Hz, 2H, NCH_2), 3.26-3.07 (m, 2H, $ArCH_2$), 2.53 (s, 3H, $ArCH_3$), 1.88-1.75 (m, 2H, CH_2), 1.61-1.45 (m, 2H, CH_2), 1.40 (t, J = 7.4 Hz, 3H, $ArCH_3$), 0.99 (t, J = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.7, 139.9, 138.2, 135.5, 124.4, 123.0, 122.2, 121.2, 118.6, 118.5, 108.1, 106.3, 37.3, 34.1, 32.0, 23.0, 22.1, 14.1, 13.7; IR (neat) ν (cm^{-1}) 3050, 3013, 2955, 2928, 2860, 1623, 1601, 1577, 1491, 1468, 1459, 1450, 1432, 1377, 1350, 1328, 1299, 1268, 1257, 1210, 1188, 1156, 1112, 1084, 1029; MS (70 ev, EI) *m/z* (%) 265 (M^+, 100); HRMS Calcd for $C_{19}H_{23}N$ (M^+): 265.1830, Found: 265.1827.

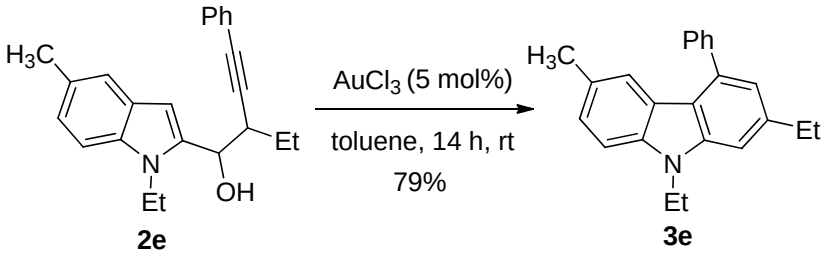
(4) **4-Butyl-9-ethyl-2,6-dimethyl-9*H*-carbazole (3d)**



The reaction of $AuCl_3$ (4.7 mg, 0.015 mmol) and **2d** (89.0 mg, 0.30 mmol) in toluene (2.0 mL) at rt for 11.5 h afforded **3d** (60.5 mg, 72%) (petroleum ether/ethyl acetate = 80/l): liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H, ArH), 7.36-7.19 (m, 2H, ArH), 7.02 (s, 1H, ArH), 6.81 (s, 1H, ArH), 4.26 (q, J = 7.2 Hz, 2H, NCH_2), 3.24-3.11 (m, 2H, $ArCH_2$), 2.54 (s, 3H, $ArCH_3$), 2.52 (s, 3H, $ArCH_3$), 1.91-1.76 (m, 2H, CH_2), 1.62-1.48 (m, 2H, CH_2), 1.37 (t, J = 7.2 Hz, 3H, $ArCH_3$), 1.01 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.0, 138.19, 138.17, 135.3, 127.7, 125.6, 123.1, 122.4, 120.9, 118.3, 107.8, 106.2, 37.3, 34.1, 32.0, 22.9, 22.1, 21.7, 14.1, 13.7; IR (neat) ν (cm^{-1}) 2956, 2929, 2861, 1626, 1607, 1576, 1481, 1462, 1376, 1350, 1325, 1307, 1271, 1254, 1190, 1146, 1117; MS (70 ev, EI) *m/z* (%) 280 (M^++1, 21.93), 279

(M^+, 100); HRMS Calcd for $C_{20}H_{25}N$ (M^+): 279.1987, Found: 279.1982.

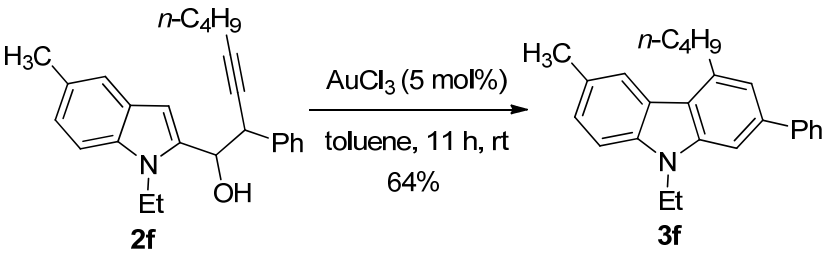
(5) **2,9-Diethyl-6-methyl-4-phenyl-9*H*-carbazole (3e)**



2e **3e**

The reaction of $AuCl_3$ (4.6 mg, 0.015 mmol) and **2e** (97.5 mg, 0.30 mmol) in toluene (2.0 mL) at rt for 14 h afforded **3e** (72.9 mg, 79%) (petroleum ether/ethyl acetate = 80/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68-7.60 (m, 2H, ArH), 7.54-7.38 (m, 3H, ArH), 7.28 (s, 1H, ArH), 7.24-7.11 (m, 3H, ArH), 6.94 (d, *J* = 1.2 Hz, 1H, ArH), 4.28 (q, *J* = 7.1 Hz, 2H, NCH_2), 2.85 (q, *J* = 7.8 Hz, 2H, $ArCH_2$), 2.29 (s, 3H, $ArCH_3$), 1.42-1.31 (m, 6H, 2×CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.8, 141.6, 140.9, 138.5, 137.5, 129.2, 128.2, 127.3, 126.2, 122.7, 122.1, 120.8, 118.1, 107.8, 106.2, 37.3, 29.5, 21.4, 16.1, 13.7; IR (neat) ν (cm^{-1}) 3055, 3027, 2966, 2931, 2870, 1622, 1603, 1568, 1499, 1486, 1470, 1462, 1409, 1378, 1350, 1334, 1307, 1292, 1282, 1227, 1192, 1176, 1146, 1126, 1085, 1073, 1062, 1028; MS (70 ev, EI) *m/z* (%) 314 (M^++1, 24.85); 313 (M^+, 100); HRMS Calcd for $C_{23}H_{23}N$ (M^+): 313.1830, Found: 313.1824.

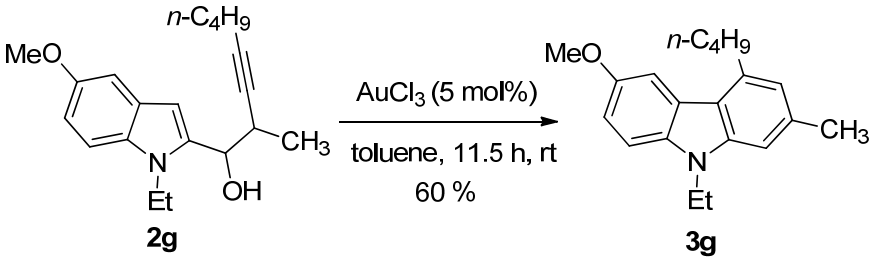
(6) **4-Butyl-9-ethyl-6-methyl-2-phenyl-9*H*-carbazole (3f)**



2f **3f**

The reaction of $AuCl_3$ (4.6 mg, 0.015 mmol) and **2f** (102.5 mg, 0.29 mmol) in toluene (2.0 mL) at rt for 11 h afforded **3f** (62.5 mg, 64%) (petroleum ether/CH_2Cl_2 = 20/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.99 (s, 1H, ArH), 7.85-7.75 (m, 2H, ArH), 7.60-7.46 (m, 3H, ArH), 7.45-7.27 (m, 4H, ArH), 4.42 (q, *J* = 7.2 Hz, 2H, NCH_2), 3.46-3.20 (m, 2H, $ArCH_2$), 2.63 (s, 3H, $ArCH_3$), 2.02-1.87 (m, 2H, CH_2), 1.72-1.56 (m, 2H, CH_2), 1.48 (t, *J* = 7.2 Hz, 3H, CH_3), 1.09 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.4, 141.1, 138.7, 138.64, 138.63, 128.7, 128.0, 127.6, 126.9, 126.2, 122.8, 122.7, 119.9, 119.1, 107.9, 104.6, 37.4, 34.3, 31.9, 22.9, 21.7, 14.1, 13.7; IR (neat) ν (cm^{-1}) 3026, 2955, 2929, 2868, 1600, 1567, 1486, 1470, 1375, 1351, 1309, 1281, 1228, 1153, 1117, 1075; MS (70 ev, EI) *m/z* (%) 342 (M^++1, 27.48), 341 (M^+, 100); HRMS Calcd for $C_{25}H_{27}N$ (M^+): 341.2144, Found: 341.2142.

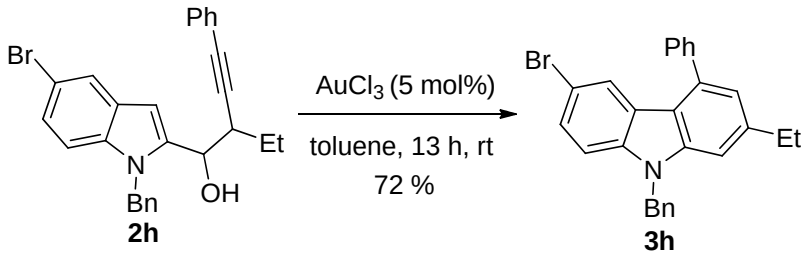
(7) **4-Butyl-9-ethyl-6-methoxy-2-methyl-9*H*-carbazole (3g)**



The reaction of $AuCl_3$ (3.2 mg, 0.01 mmol) and **2g** (63.5 mg, 0.20 mmol) in toluene (1.0 mL) at rt for 11.5 h afforded **3g** (36.0 mg, 60%) (petroleum ether/ethyl acetate = 40/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (d, *J* = 2.4 Hz, 1H, ArH), 7.30 (d, *J* = 9.0 Hz, 1H, ArH), 7.11 (dd, *J* = 8.7 and 2.4 Hz, 1H, ArH), 7.06 (s, 1H, ArH), 6.84 (s, 1H, ArH), 4.31 (q, *J* = 7.2 Hz, 2H, NCH_2), 3.95 (s, 3H, OCH_3), 3.30-2.97 (m, 2H, $ArCH_2$), 2.56 (s, 3H, $ArCH_3$), 1.94-1.78 (m, 2H, CH_2), 1.66-1.50 (m, 2H, CH_2), 1.42 (t, *J* = 7.2 Hz, 3H, CH_3), 1.04 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ

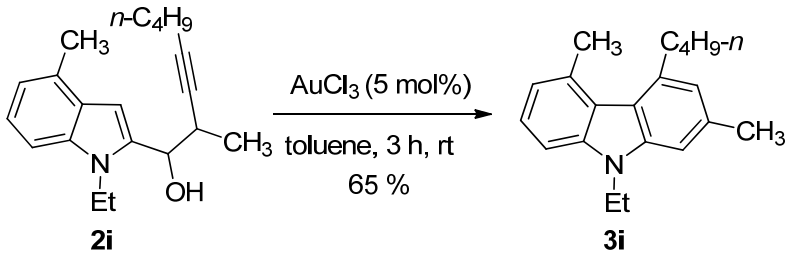
153.2, 141.3, 138.2, 135.6, 135.0, 123.3, 120.8, 118.3, 112.7, 108.4, 106.35, 106.32, 56.2, 37.4, 34.1, 32.1, 23.1, 22.1, 14.1, 13.7; IR (neat) ν (cm^{-1}) 2954, 2931, 2862, 2830, 1625, 1609, 1579, 1482, 1378, 1351, 1310, 1227, 1170, 1138, 1040; MS (70 ev, EI) *m/z* (%) 296 (M^++1, 21.91), 295 (M^+, 100); HRMS Calcd for $C_{20}H_{25}NO$ (M^+): 295.1936, Found: 295.1936.

(8) **9-Benzyl-6-bromo-2-ethyl-4-phenyl-9*H*-carbazole (3h)**



The reaction of $AuCl_3$ (3.4 mg, 0.01 mmol) and **2h** (92.0 mg, 0.20 mmol) in toluene (2.0 mL) at rt for 13 h afforded **3h** (63.6 mg, 72%) (petroleum ether/ethyl acetate = 100/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66-7.56 (m, 3H, ArH), 7.55-7.41 (m, 3H, ArH), 7.31 (dd, *J* = 8.7 and 1.8 Hz, 1H, ArH), 7.26-7.16 (m, 3H, ArH), 7.13 (s, 1H, ArH), 7.10-7.02 (m, 3H, ArH), 6.99 (d, *J* = 0.9 Hz, 1H, ArH), 5.36 (s, 2H, $ArCH_2$), 2.78 (q, *J* = 7.5 Hz, 2H, CH_2), 1.28 (t, *J* = 7.5 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.1, 141.7, 140.7, 139.4, 137.7, 136.6, 129.0, 128.8, 128.5, 127.8, 127.7, 127.5, 126.2, 124.5, 124.4, 121.8, 117.4, 111.6, 110.0, 106.8, 46.4, 29.4, 15.9; IR (neat) ν (cm^{-1}) 3087, 3049, 3012, 2955, 2928, 2862, 1645, 1495, 1574, 1502, 1463, 1392 ,1327 1302, 1225, 1162, 1118, 1088, 1029; MS (70 ev, EI) *m/z* (%) 442 (M^+ (^{81}Br)+1, 27.88), 441 (M^+ (^{81}Br), 100), 440 (M^+ (^{79}Br)+1, 29.53), 439 (M^+ (^{79}Br), 96.50); HRMS Calcd for $C_{27}H_{22}N^{79}Br$ (M^+): 439.0936, Found: 439.0932.

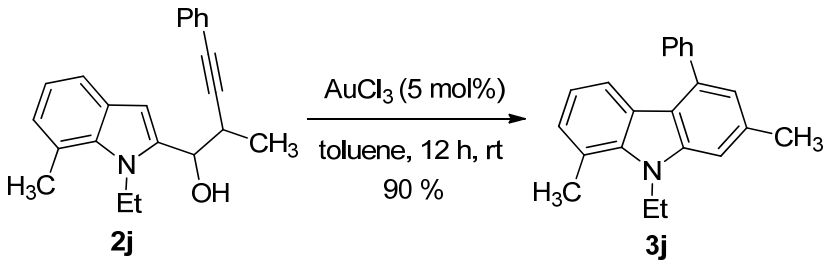
(9) **4-Butyl-9-ethyl-2,5-dimethyl-9*H*-carbazole (3i)**



2i **3i**

The reaction of $AuCl_3$ (4.6 mg, 0.015 mmol) and **2i** (89.2 mg, 0.30 mmol) in toluene (2.0 mL) at rt for 3 h afforded **3i** (54.5 mg, 65%) (petroleum ether/ethyl acetate = 40/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.36 (m, 1H, ArH), 7.32 (d, *J* = 7.8 Hz, 1H, ArH), 7.14 (s, 1H, ArH), 7.09 (d, *J* = 7.2 Hz, 1H, ArH), 6.97 (s, 1H, ArH), 4.38 (q, *J* = 7.1 Hz, 2H, NCH_2), 3.48-3.30 (m, 2H, $ArCH_2$), 3.04 (s, 3H, CH_3), 2.60 (s, 3H, CH_3), 1.86-1.74 (m, 2H, CH_2), 1.60-1.40 (m, 5H, CH_3+CH_2), 1.02 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.2, 140.6, 137.7, 135.1, 132.1, 124.6, 122.8, 122.4, 122.3, 119.4, 106.2, 106.0, 37.2, 36.8, 34.8, 25.3, 22.6, 21.7, 14.1, 13.3; IR (neat) ν (cm^{-1}) 3048, 2956, 2926, 2870, 1619, 1597, 1570, 1451, 1377, 1349, 1317, 1274, 1216, 1148, 1078, 1044; MS (70 ev, EI) *m/z* (%) 280 (M^++1, 22.02), 279 (M^+, 100); HRMS Calcd for $C_{20}H_{25}N$ (M^+): 279.1987, Found: 279.1994.

(10) **9-Ethyl-1,7-dimethyl-5-phenyl-9*H*-carbazole (3j)**

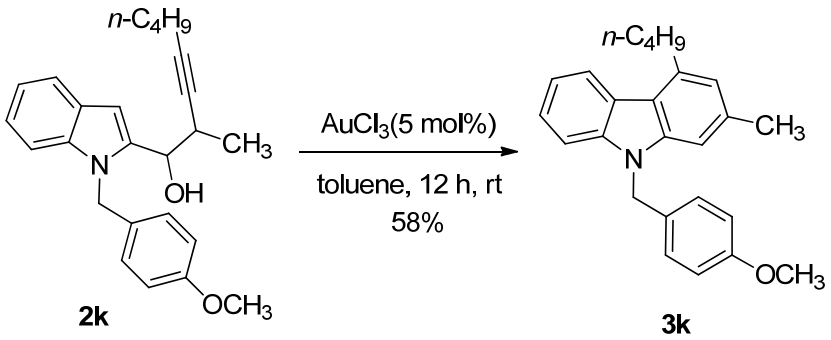


2j **3j**

The reaction of $AuCl_3$ (4.9 mg, 0.016 mmol) and **2j** (96.2 mg, 0.30 mmol) in toluene (1.5 mL) at rt for 12 h afforded **3j** (82.1 mg, 90%) (petroleum ether/ethyl

acetate = 80/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77-7.69 (m, 2H, ArH), 7.67-7.52 (m, 3H, ArH), 7.42 (d, *J* = 7.8 Hz, 1H, ArH), 7.32 (s, 1H, ArH), 7.20 (d, *J* = 7.2 Hz, 1H, ArH), 7.06 (d, *J* = 0.6 Hz, 1H, ArH), 6.96 (t, *J* = 7.5 Hz, 1H, ArH), 4.69 (q, *J* = 7.1 Hz, 2H, NCH_2), 2.91 (s, 3H, $ArCH_3$), 2.71 (s, 3H, $ArCH_3$), 1.56 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.5, 141.4, 138.8, 137.2, 135.2, 129.2, 128.3, 127.3, 123.5, 122.4, 120.0, 119.5, 118.4, 118.2, 107.8, 39.3, 22.1, 20.3, 15.6; IR (neat) ν (cm^{-1}) 3027, 2959, 2927, 2855, 1622, 1603, 1569, 1486, 1463, 1409, 1332, 1306, 1227, 1192, 1146, 1084, 1028; MS (70 ev, EI) *m/z* (%) 300 (M^++1, 19.63), 299 (M^+, 78.14), 284 (100); HRMS Calcd for $C_{22}H_{21}N$ (M^+): 299.1674, Found: 299.1678.

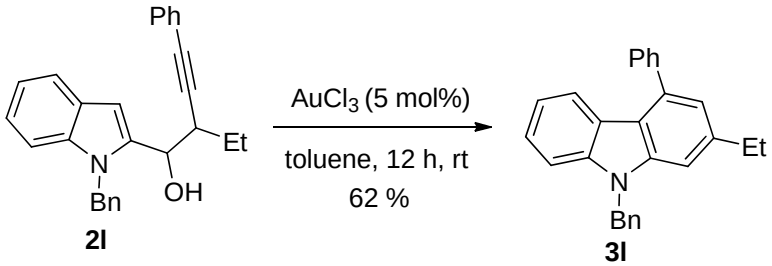
(11) **4-Butyl-9-(4-methoxybenzyl)-2-methyl-9*H*-carbazole (3k)**



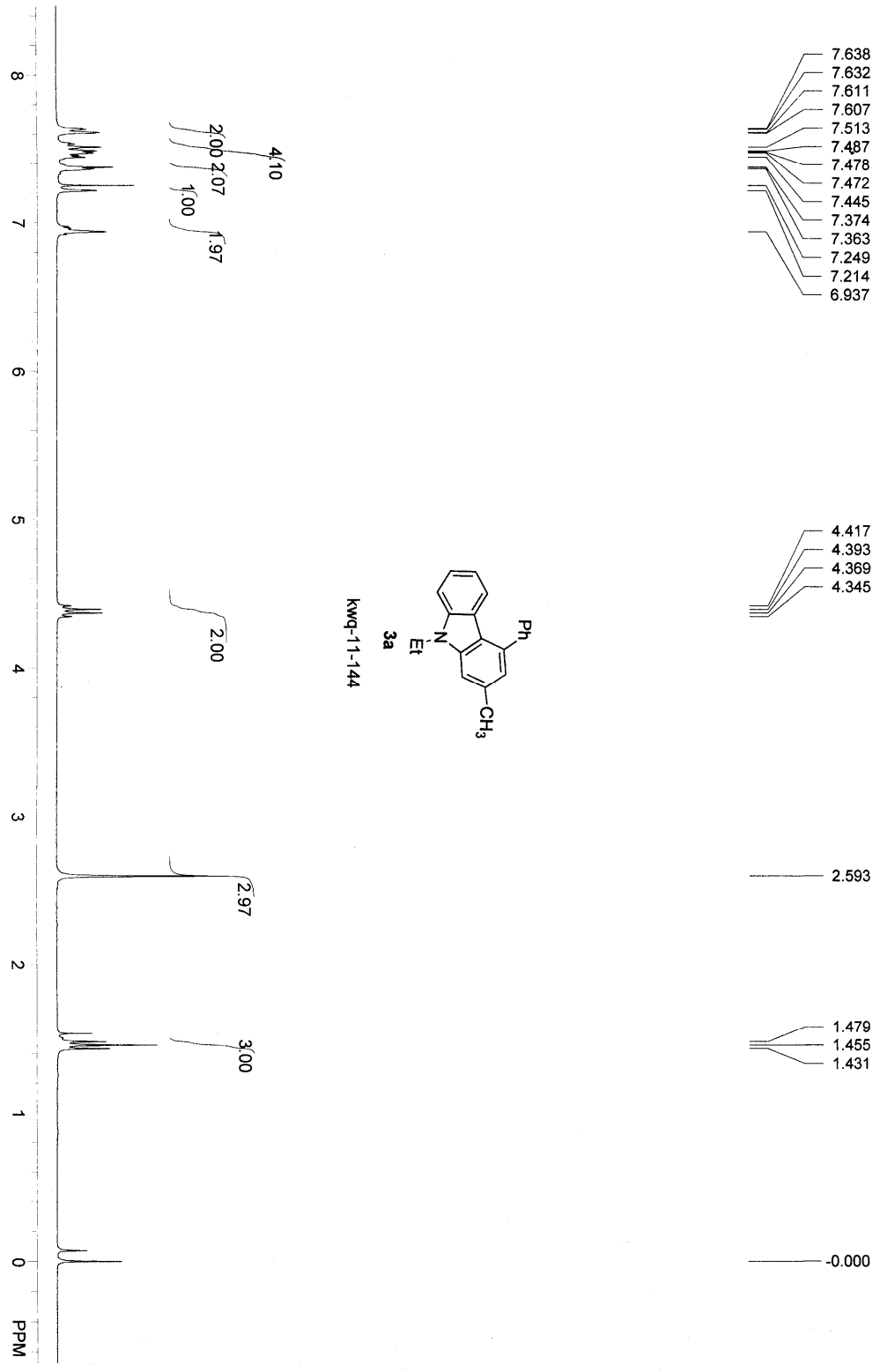
The reaction of $AuCl_3$ (3.1 mg, 0.01 mmol) and **2k** (75.0 mg, 0.20 mmol) in toluene (1.5 mL) at rt for 12 h afforded **3k** (41.7 mg, 58%) (petroleum ether/ethyl acetate = 60/l): solid, m.p. 92~93 ^{o}C (*n*-hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (q, *J* = 7.8 Hz, 1H, ArH), 7.42-7.28 (m, 2H, ArH), 7.27-7.18 (m, 1H, ArH), 7.10-6.98 (m, 3H, ArH), 6.86 (s, 1H, ArH), 6.80-6.71 (m, 2H, ArH), 5.38 (s, 2H, $ArCH_2$), 3.70 (s, 3H, OCH_3), 3.24-3.15 (m, 2H, $ArCH_2$), 2.47 (s, 3H, $ArCH_3$), 1.90-1.68 (m, 2H, CH_2), 1.62-1.43 (m, 2H, CH_2), 1.00 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 141.5, 140.6, 138.1, 135.7, 129.3, 127.5, 124.6,

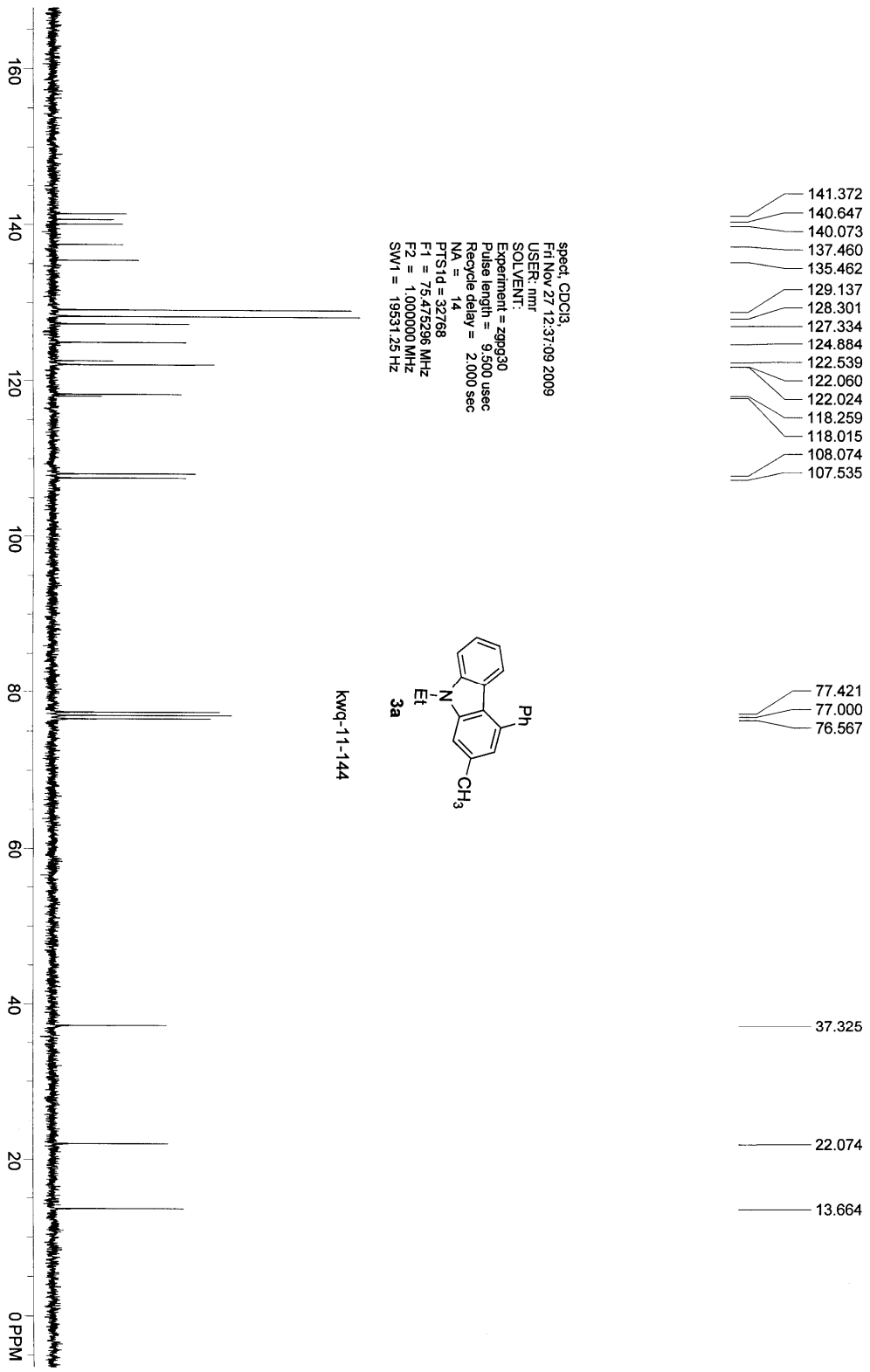
123.1, 122.2, 121.5, 119.0, 118.5, 114.1, 108.6, 106.7, 55.2, 45.8, 34.1, 32.0, 23.0, 22.1, 14.1; IR (KBr) ν (cm^{-1}) 3052, 2954, 2928, 2860, 2835, 1613, 1601, 1586, 1575, 1513, 1481, 1463, 1446, 1353, 1327, 1295, 1248, 1175, 1034; MS (70 ev, EI) *m/z* (%) 358 (M^++1, 7.06), 357 (M^+, 24.84), 121 (100); Elemental analysis calcd (%) for $C_{25}H_{27}NO$: C, 83.99; H, 7.61; N, 3.92; Found: C, 83.57, H, 7.66; N, 4.02.

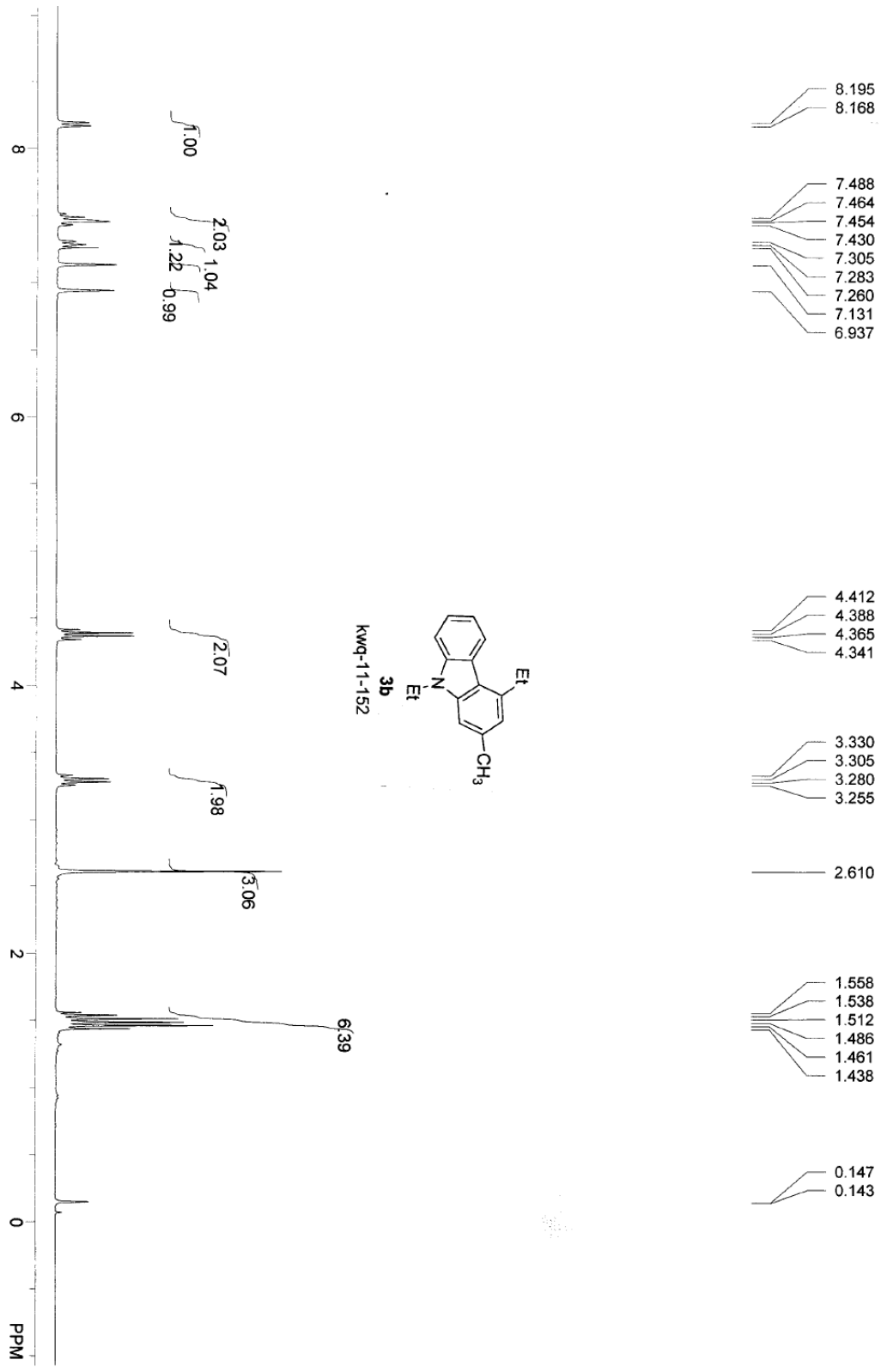
(12) **9-Benzyl-2-ethyl-4-phenyl-9*H*-carbazole (3l)**

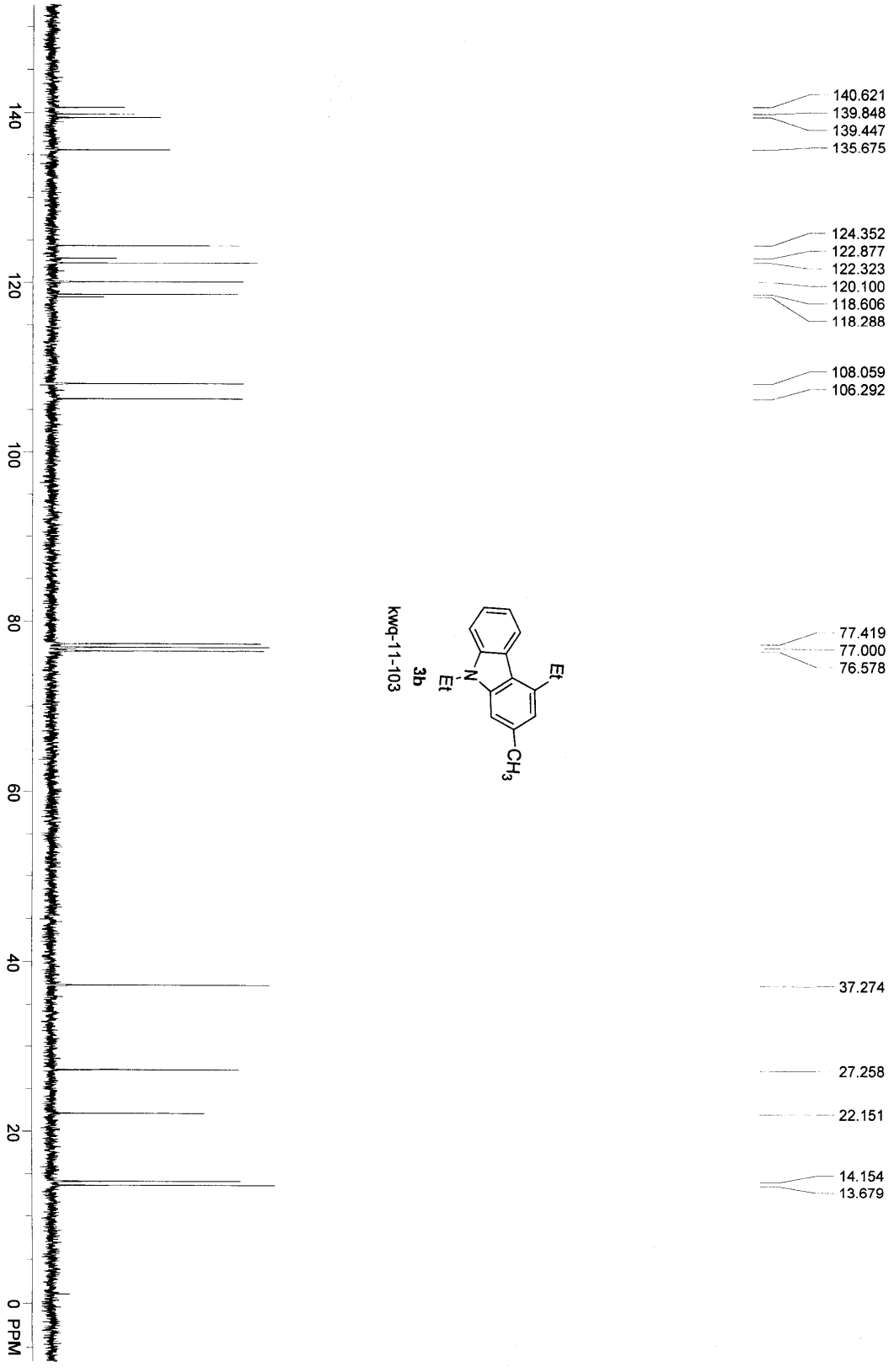


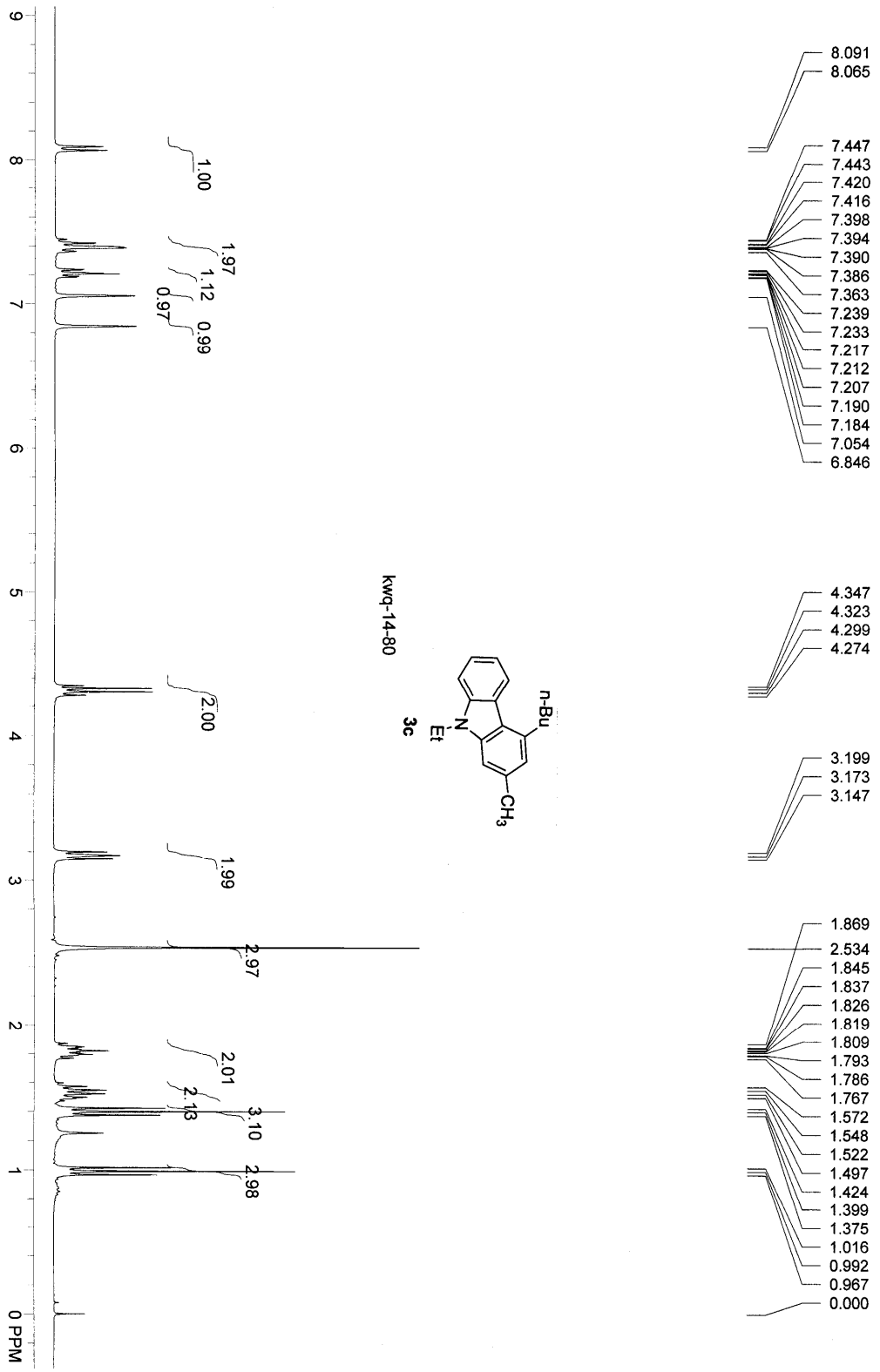
The reaction of $AuCl_3$ (3.2 mg, 0.01 mmol) and **2l** (76.2 mg, 0.20 mmol) in toluene (1.0 mL) at rt for 12 h afforded **3l** (45.0 mg, 62%) (petroleum ether/ethyl acetate = 50/l): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71-7.63 (m, 2H, ArH), 7.58-7.44 (m, 4H, ArH), 7.35-7.22 (m, 5H, ArH), 7.22-7.12 (m, 3H, ArH), 7.03-6.92 (m, 2H, ArH), 5.55 (s, 2H, $ArCH_2$), 2.83 (q, *J* = 7.7 Hz, 2H, CH_2), 1.32 (t, *J* = 7.7 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.3, 141.5, 141.4, 140.9, 137.5, 137.2, 129.2, 128.7, 128.3, 127.40, 127.37, 126.4, 125.1, 122.7, 122.0, 121.4, 118.7, 118.4, 108.6, 106.7, 46.4, 29.4, 16.0; IR (neat) ν (cm^{-1}) 3055, 3030, 2962, 2926, 2853, 1620, 1600, 1571, 1496, 1480, 1463, 1453, 1418, 1354, 1324, 1292, 1174; MS (70 ev, EI) *m/z* (%) 362 (M^++1, 29.50), 361 (M^+, 100); HRMS Calcd for $C_{27}H_{23}N$ (M^+): 361.1830, Found: 361.1828.

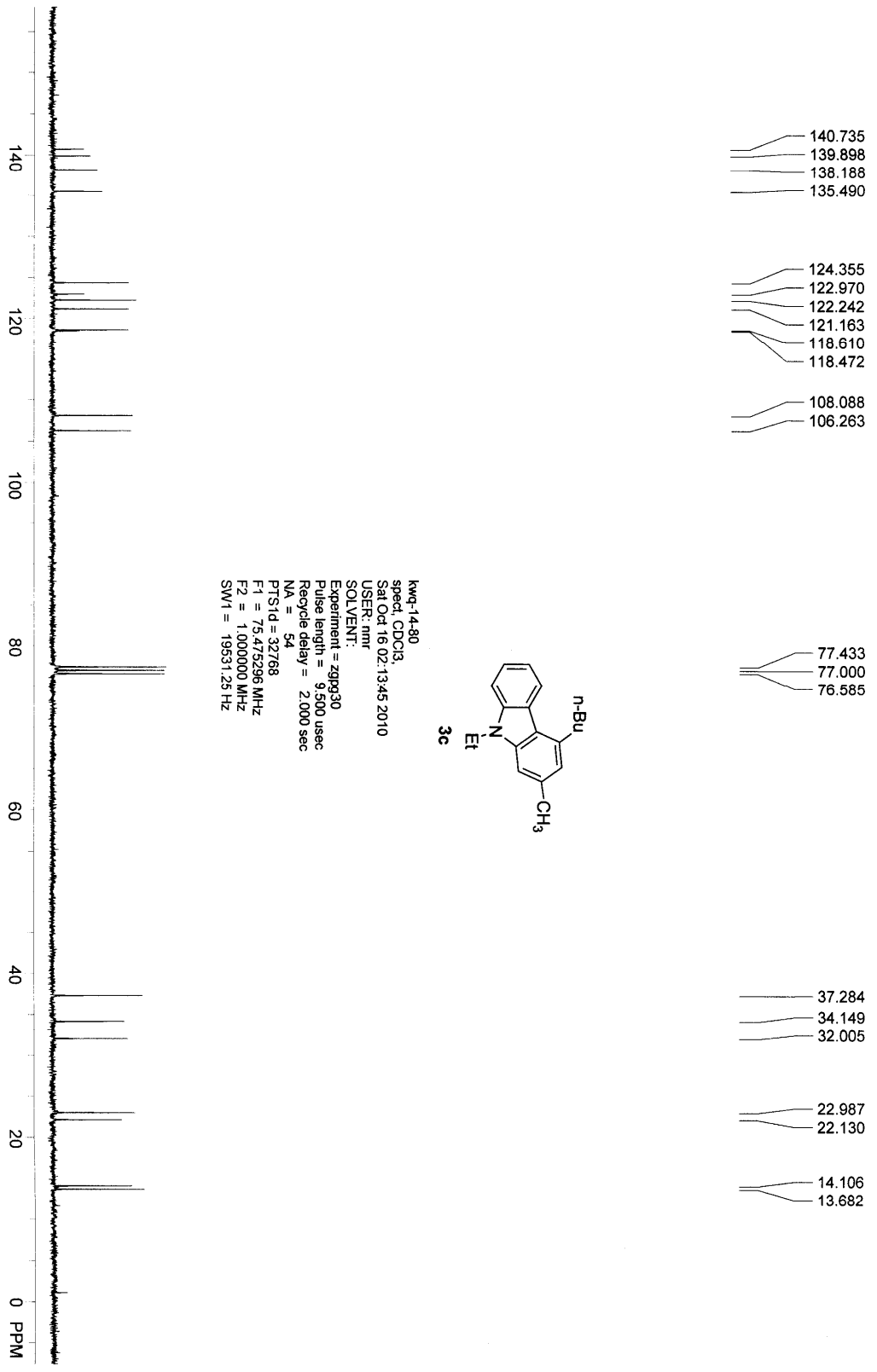

7.638
7.632
7.611
7.607
7.513
7.487
7.478
7.472
7.445
7.374
7.363
7.249
7.214
6.937
4.417
4.393
4.369
4.345
2.593
1.479
1.455
1.431
-0.000
4.10
2.00
2.07
1.00
1.97
2.00
2.97
3.00
Ph
N
Et
CH3
3a
kwq-11-144
PPM
8
7
6
5
4
3
2
1
0

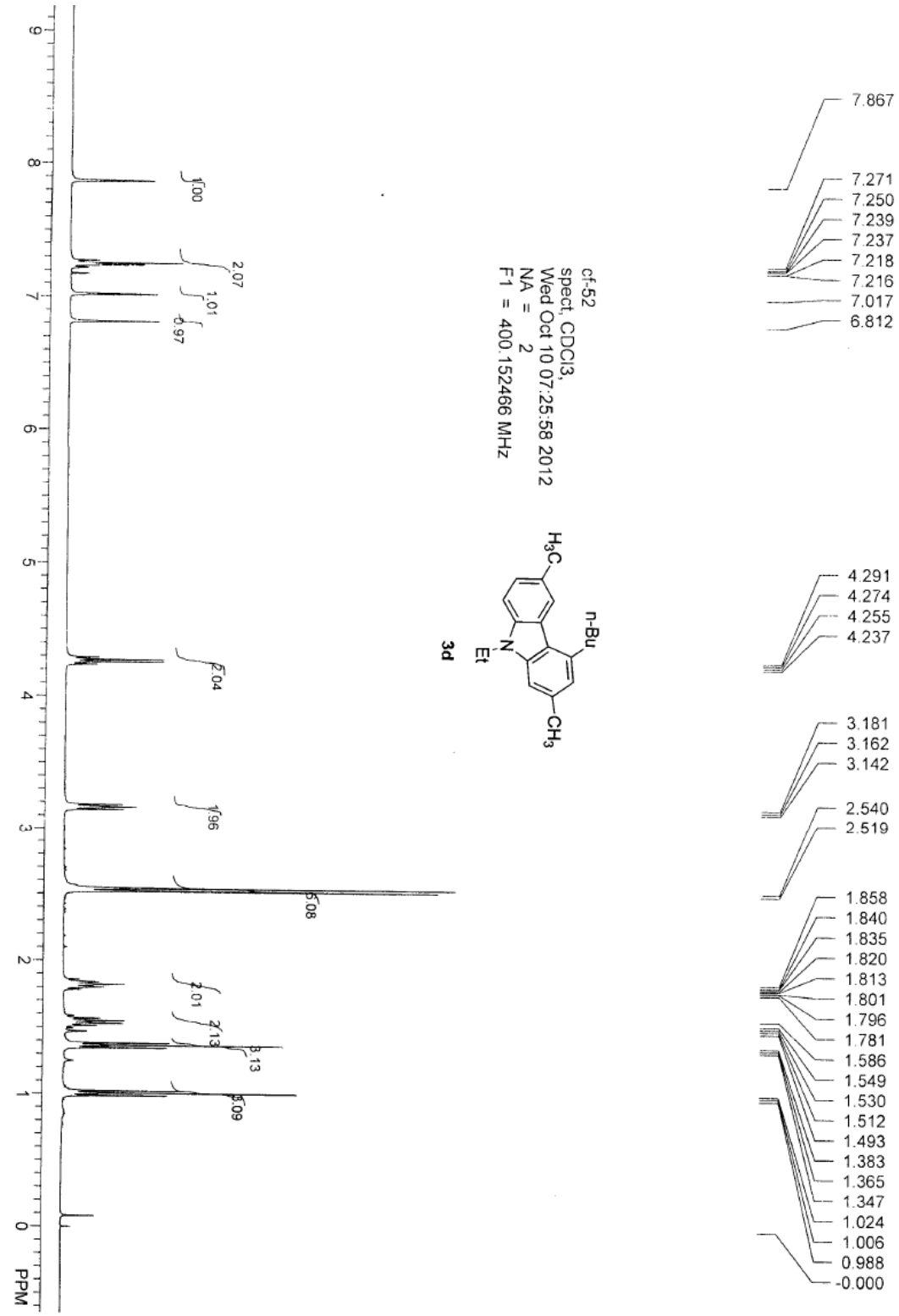

160
140
120
100
80
60
40
20
0 PPM
spect. CDCl3
Fri Nov 27 12:37:09 2009
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 14
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
kwq-11-144
N
Et
Ph
CH3
3a
141.372
140.647
140.073
137.460
135.462
129.137
128.301
127.334
124.884
122.539
122.060
122.024
118.259
118.015
108.074
107.535
77.421
77.000
76.567
37.325
22.074
13.664

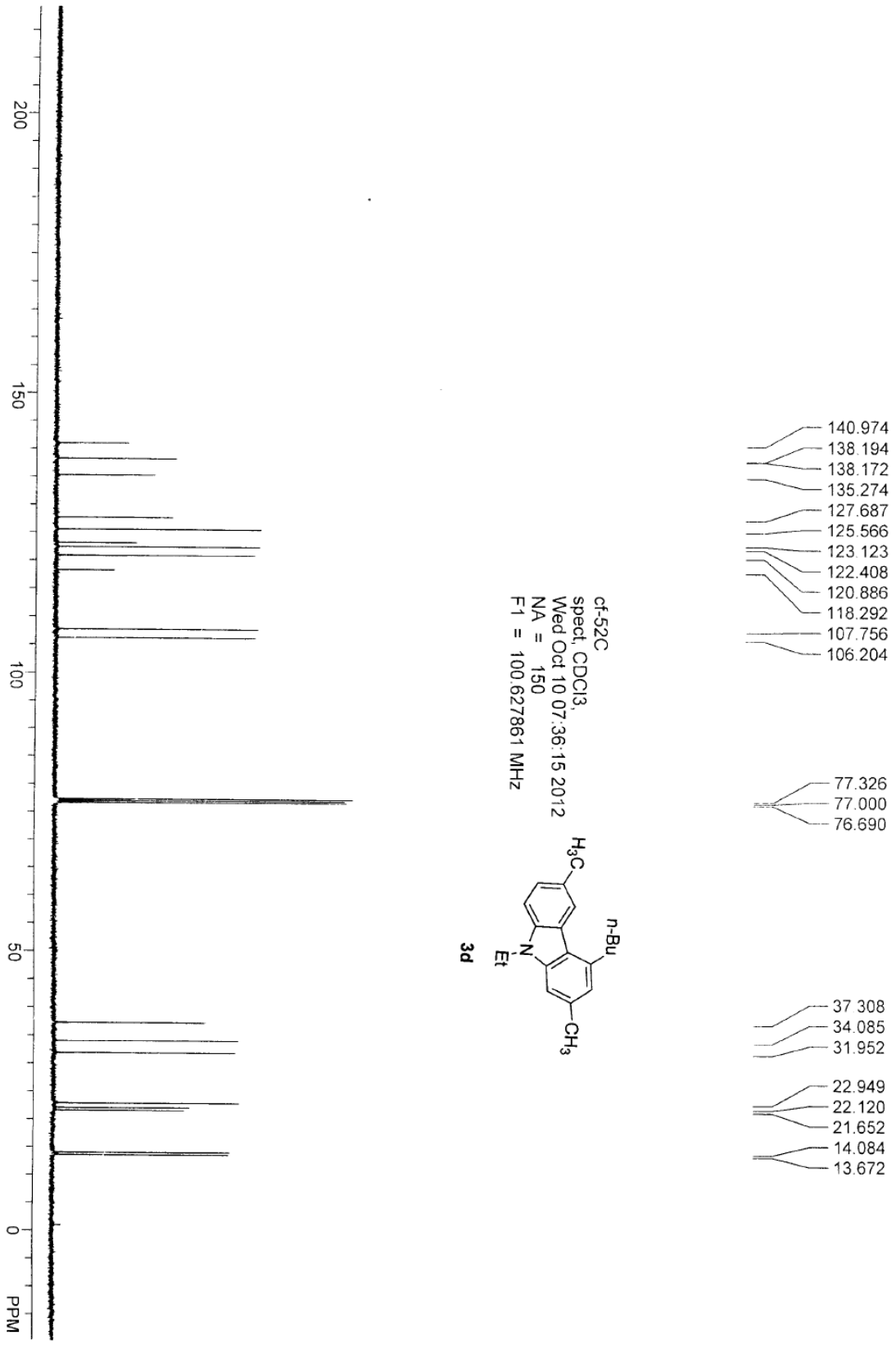

8.195
8.168
7.488
7.464
7.454
7.430
7.305
7.283
7.260
7.131
6.937
4.412
4.388
4.365
4.341
3.330
3.305
3.280
3.255
2.610
1.558
1.538
1.512
1.486
1.461
1.438
0.147
0.143
1.00
2.03
1.04
1.22
0.99
2.07
1.98
3.06
6.39
8
6
4
2
0
PPM
Et
N
Et
CH3
3b
kwq-11-152

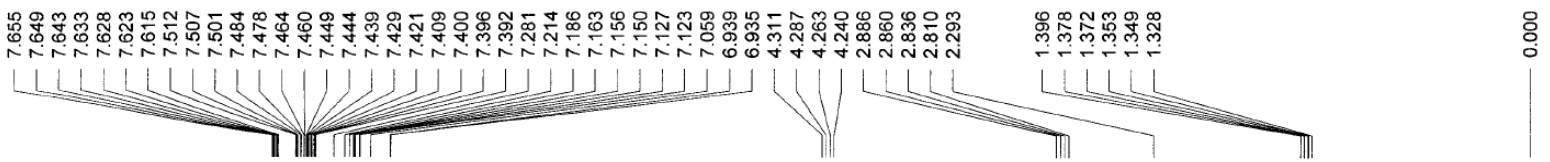

140.621
139.848
139.447
135.675
124.352
122.877
122.323
120.100
118.606
118.288
108.059
106.292
77.419
77.000
76.578
37.274
27.258
22.151
14.154
13.679
140
120
100
80
60
40
20
0 PPM
kwq-11-103
3b
N
Et
Et
CH3

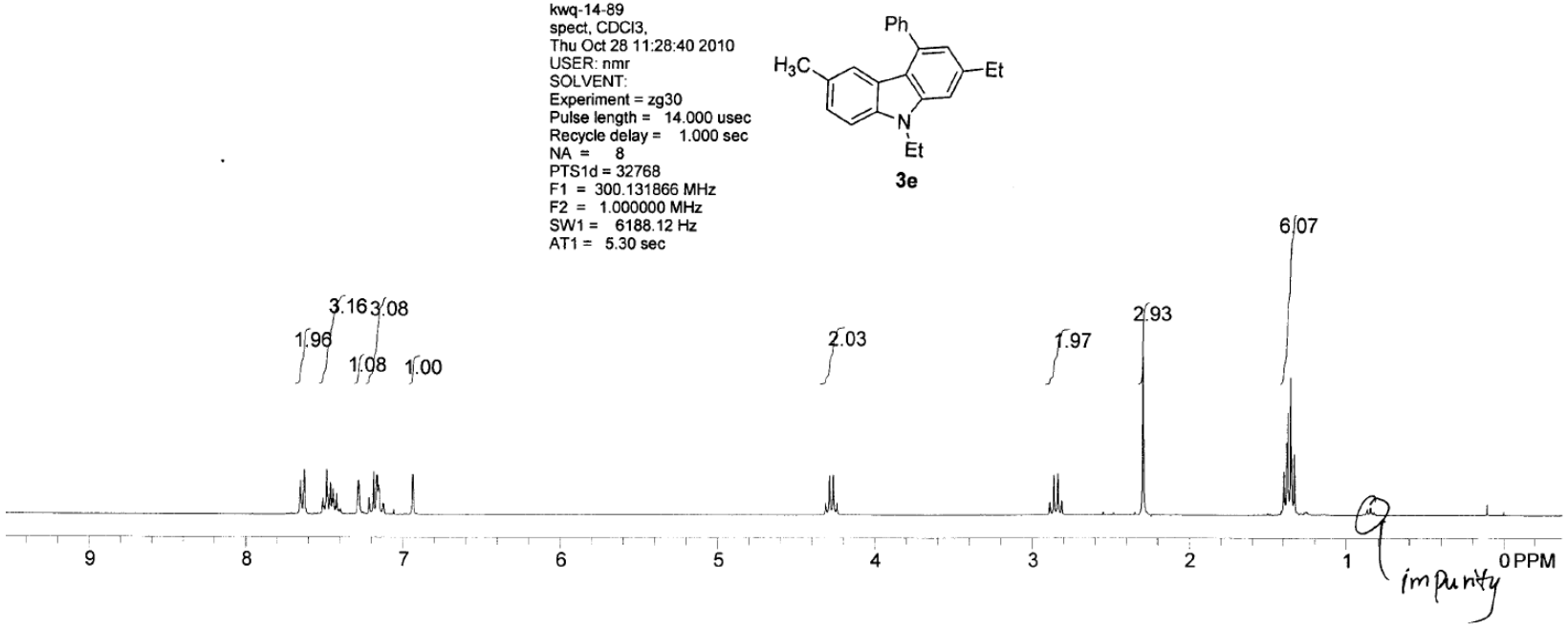

8.091
8.065
7.447
7.443
7.420
7.416
7.398
7.394
7.390
7.386
7.363
7.239
7.233
7.217
7.212
7.207
7.190
7.184
7.054
6.846
4.347
4.323
4.299
4.274
3.199
3.173
3.147
1.869
2.534
1.845
1.837
1.826
1.819
1.809
1.793
1.786
1.767
1.572
1.548
1.522
1.497
1.424
1.399
1.375
1.016
0.992
0.967
0.000
kwq-14-80
3c
n-Bu
N
Et
CH3
1.00
1.97
1.12
0.97
0.99
2.00
1.99
2.97
2.01
2.13
3.10
2.98
9
8
7
6
5
4
3
2
1
0 PPM

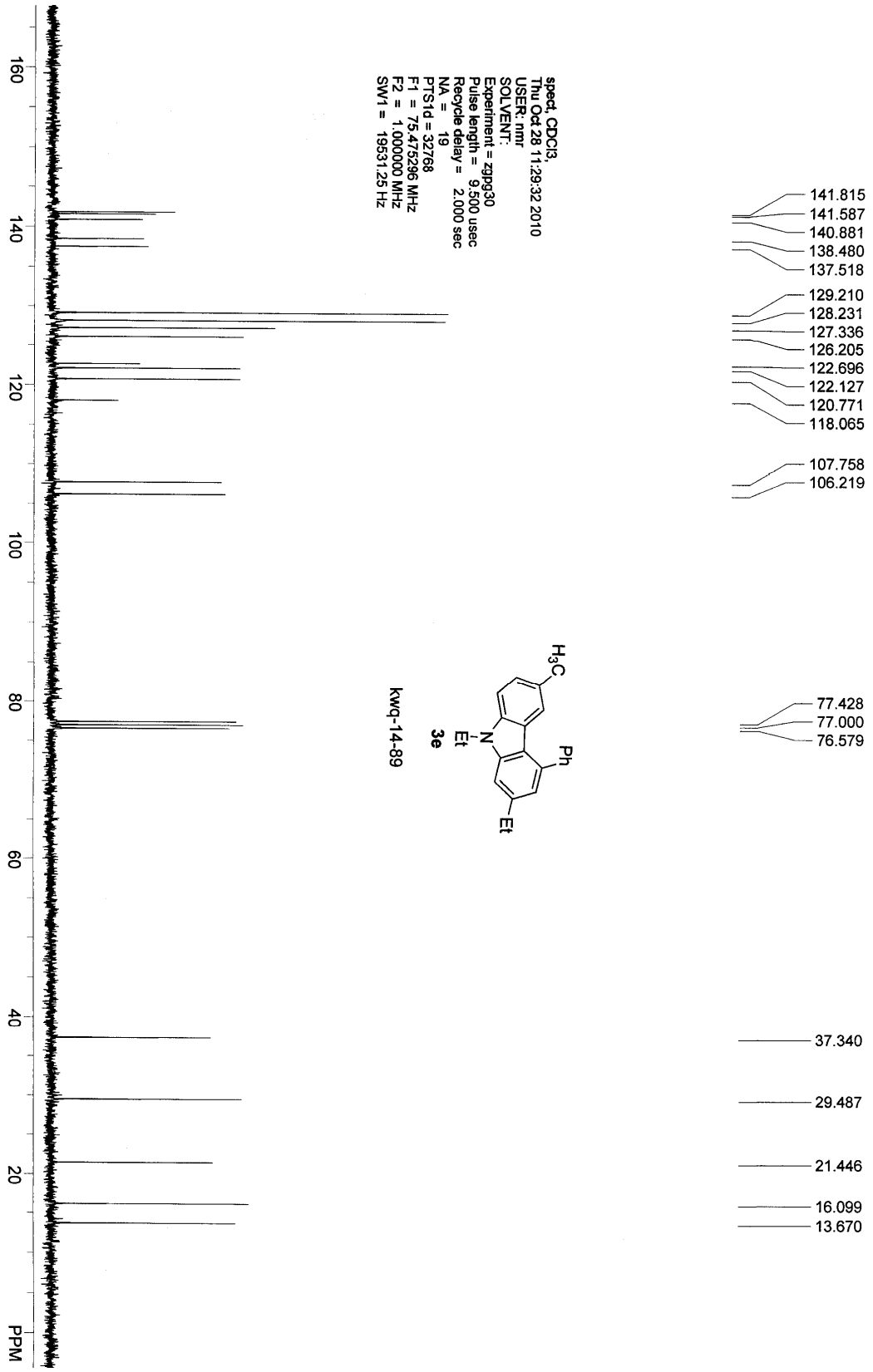

140.735
139.898
138.188
135.490
124.355
122.970
122.242
121.163
118.610
118.472
108.088
106.263
77.433
77.000
76.585
37.284
34.149
32.005
22.987
22.130
14.106
13.682
kwq-14-80
spect, CDCl3,
Sat Oct 16 02:13:45 2010
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 54
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
n-Bu
N
Et
CH3
3c
140
120
100
80
60
40
20
0 PPM

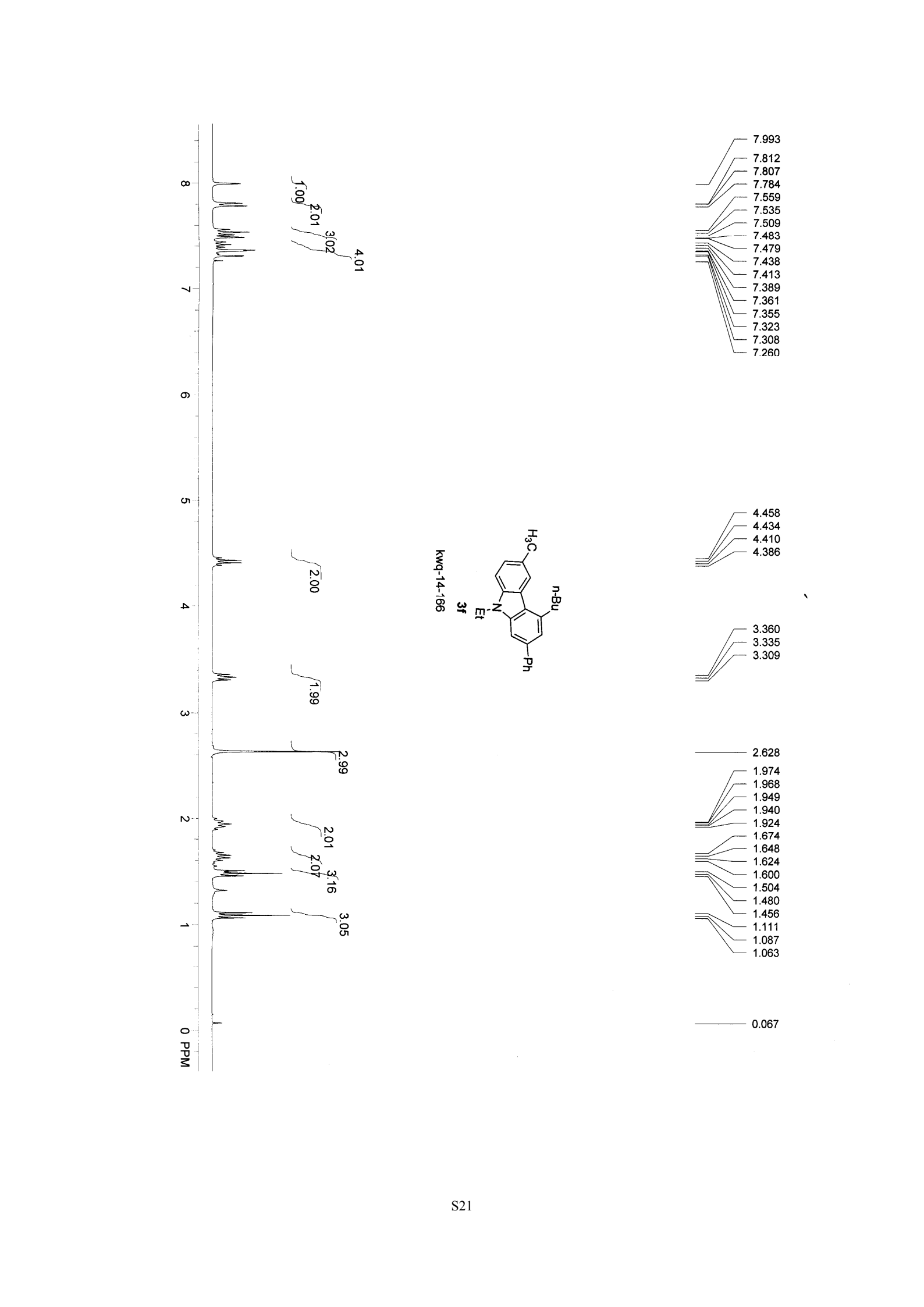

cf-52
spect, CDCl3,
Wed Oct 10 07:25:58 2012
NA = 2
F1 = 400.152466 MHz
3d
n-Bu
H3C
CH3
N
Et
7.867
7.271
7.250
7.239
7.237
7.218
7.216
7.017
6.812
4.291
4.274
4.255
4.237
3.181
3.162
3.142
2.540
2.519
1.858
1.840
1.835
1.820
1.813
1.801
1.796
1.781
1.586
1.549
1.530
1.512
1.493
1.383
1.365
1.347
1.024
1.006
0.988
-0.000
1.00
2.07
1.01
0.97
2.04
1.96
6.08
2.01
2.13
3.13
5.09
PPM

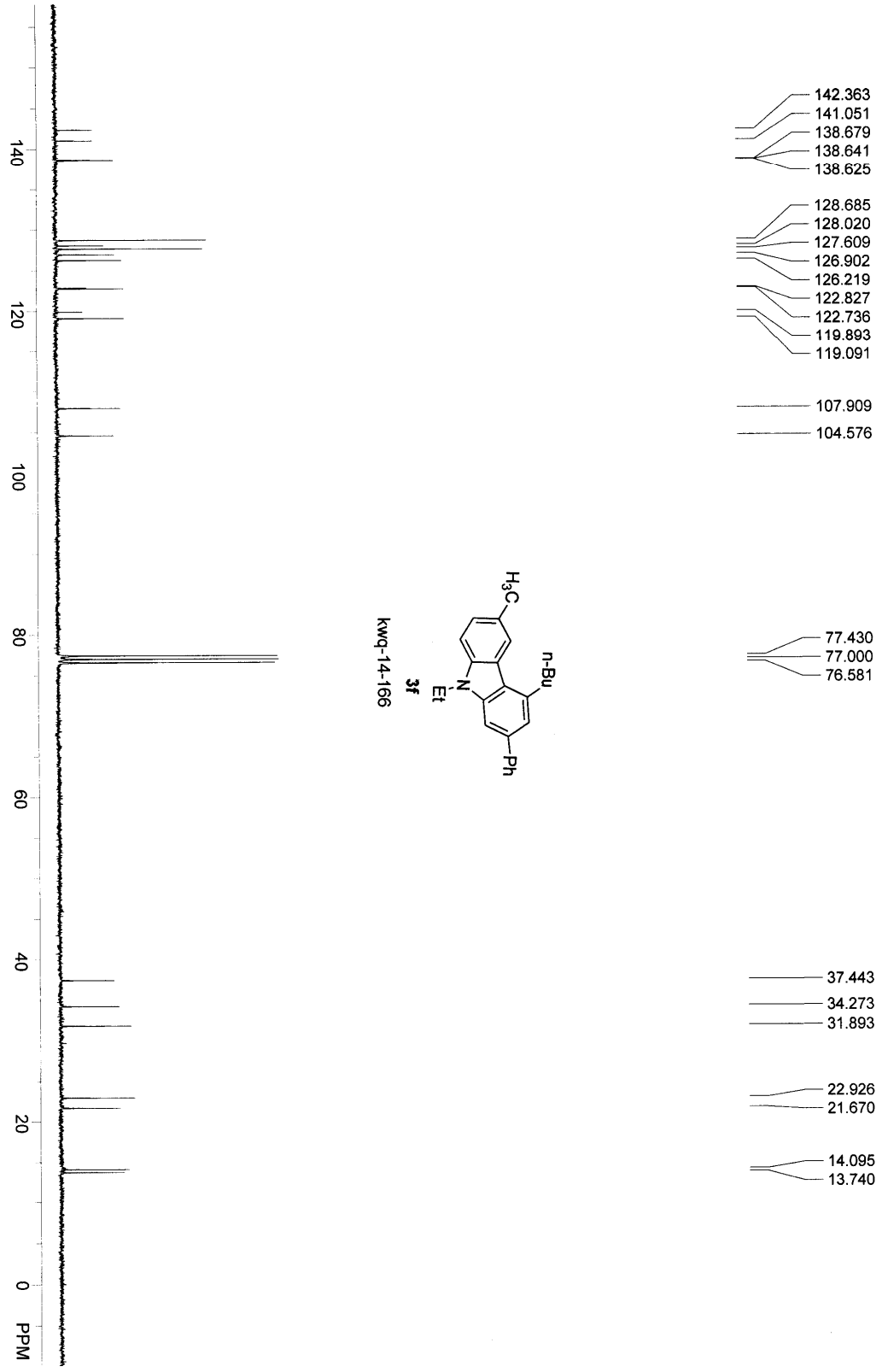

cf-52C
spect, CDCl3,
Wed Oct 10 07:36:15 2012
NA = 150
F1 = 100.627861 MHz
3d
H3C
n-Bu
N
Et
CH3
140.974
138.194
138.172
135.274
127.687
125.566
123.123
122.408
120.886
118.292
107.756
106.204
77.326
77.000
76.690
37.308
34.085
31.952
22.949
22.120
21.652
14.084
13.672
200
150
100
50
0
PPM

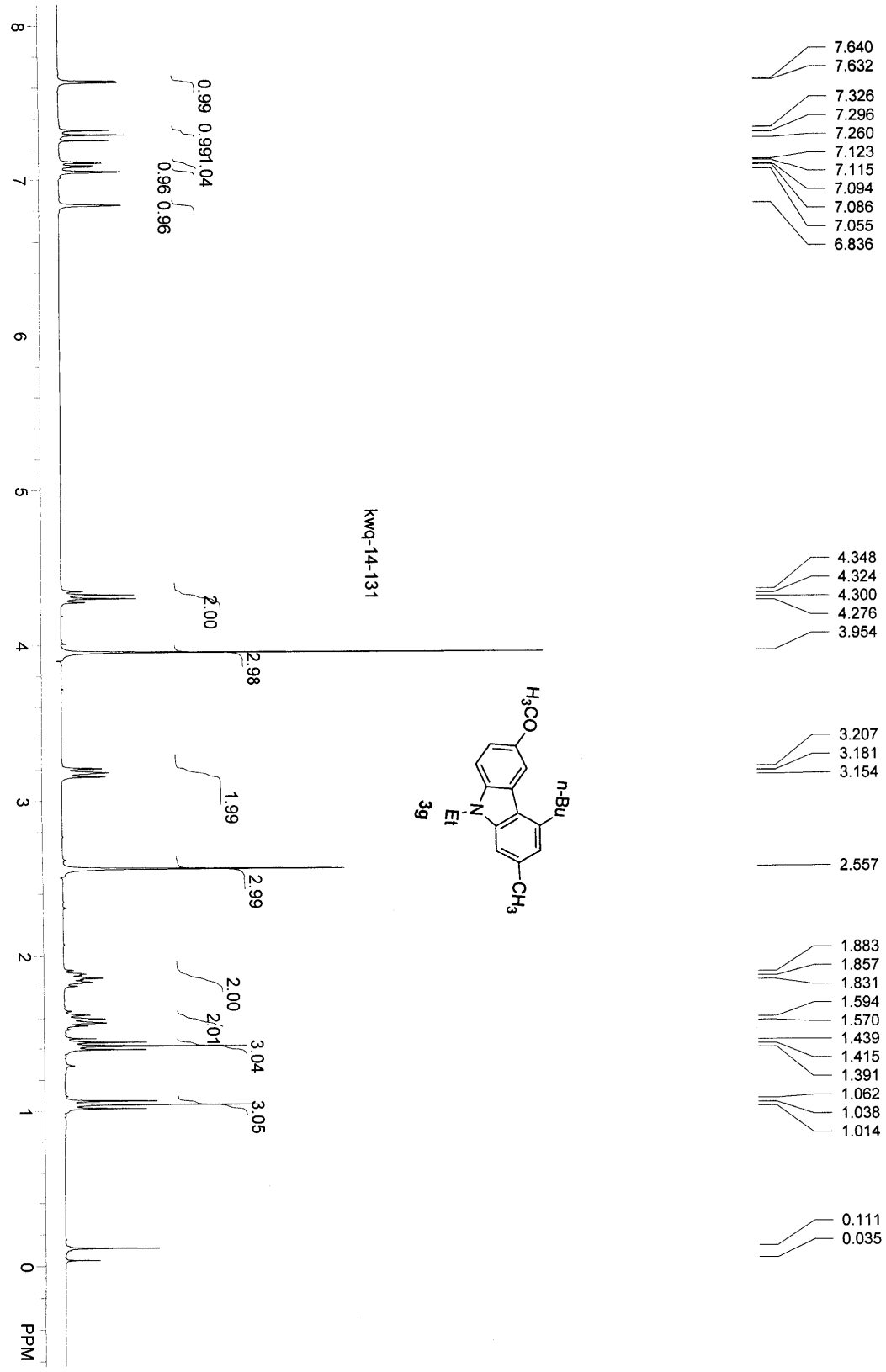

7.655
7.649
7.643
7.633
7.628
7.623
7.615
7.512
7.507
7.501
7.484
7.478
7.464
7.460
7.449
7.444
7.439
7.429
7.421
7.409
7.400
7.396
7.392
7.281
7.214
7.186
7.163
7.156
7.150
7.127
7.123
7.059
6.939
6.935
4.311
4.287
4.263
4.240
2.886
2.860
2.836
2.810
2.293
1.396
1.378
1.372
1.353
1.349
1.328
0.000

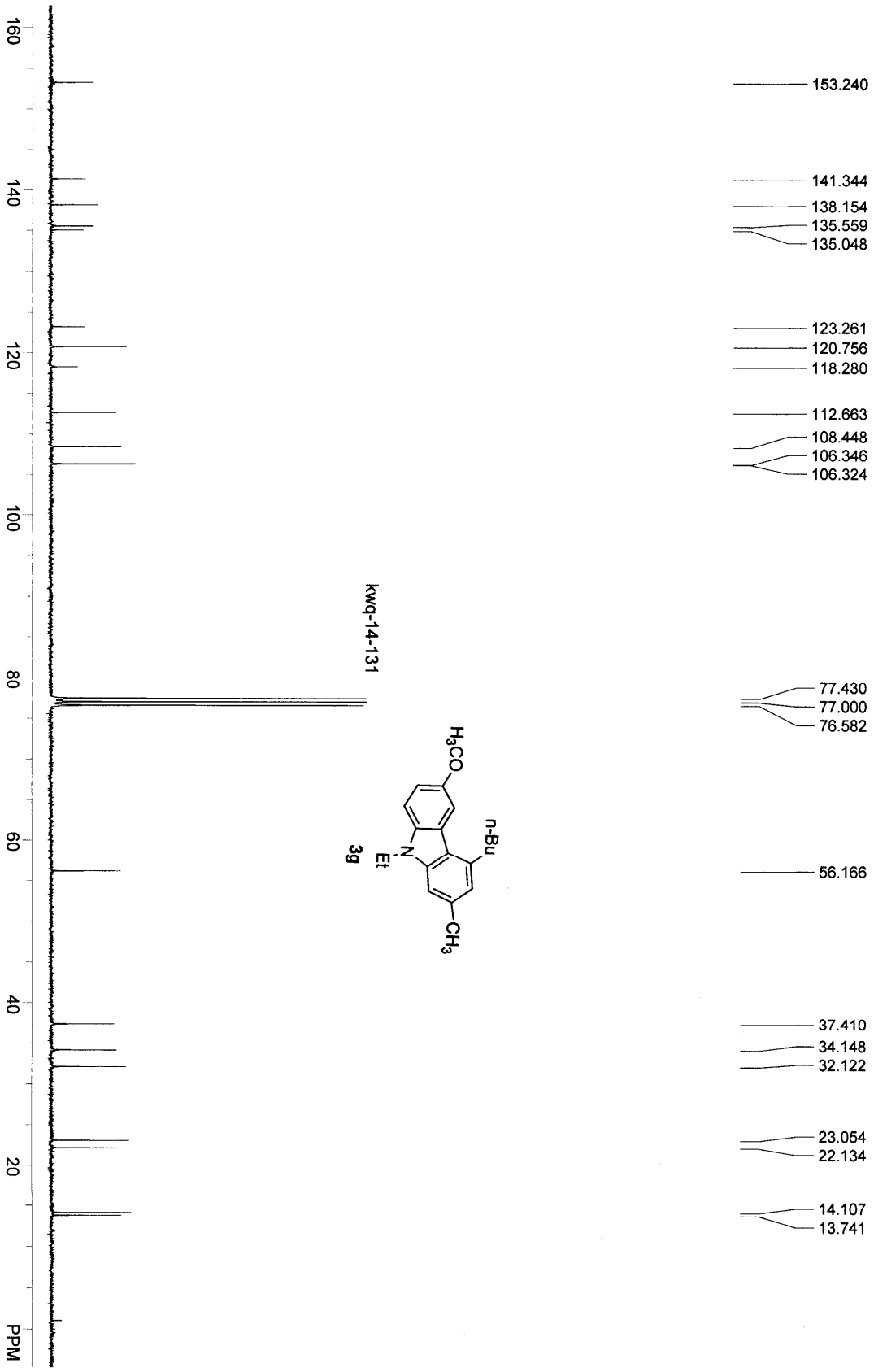

kwq-14-89
spect, CDCl3,
Thu Oct 28 11:28:40 2010
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
AT1 = 5.30 sec
Ph
H3C
Et
N
Et
3e
6.07
3.16
3.08
2.93
1.96
2.03
1.97
1.08
1.00
9
8
7
6
5
4
3
2
1
0 PPM
impurity

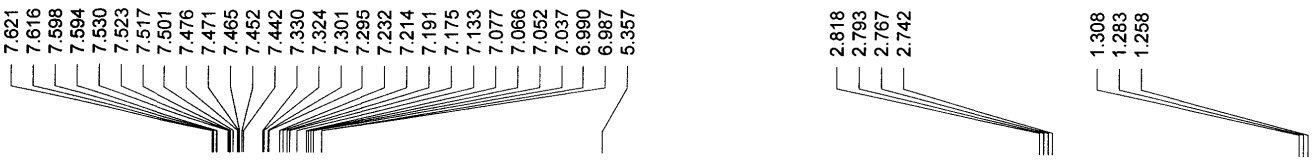

spect, CDCl3,
Thu Oct 28 11:29:32 2010
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 19
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
kwq-14-89
3e
141.815
141.587
140.881
138.480
137.518
129.210
128.231
127.336
126.205
122.696
122.127
120.771
118.065
107.758
106.219
77.428
77.000
76.579
37.340
29.487
21.446
16.099
13.670
160
140
120
100
80
60
40
20
PPM

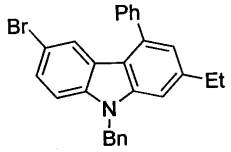

7.993
7.812
7.807
7.784
7.559
7.535
7.509
7.483
7.479
7.438
7.413
7.389
7.361
7.355
7.323
7.308
7.260
4.458
4.434
4.410
4.386
3.360
3.335
3.309
2.628
1.974
1.968
1.949
1.940
1.924
1.674
1.648
1.624
1.600
1.504
1.480
1.456
1.111
1.087
1.063
0.067
H3C
n-Bu
N
Et
Ph
3f
kwq-14-166
4.01
1.00
2.01
3.02
2.00
1.99
2.99
2.01
2.07
3.16
3.05
8
7
6
5
4
3
2
1
0 PPM


142.363
141.051
138.679
138.641
138.625
128.685
128.020
127.609
126.902
126.219
122.827
122.736
119.893
119.091
107.909
104.576
77.430
77.000
76.581
37.443
34.273
31.893
22.926
21.670
14.095
13.740
140
120
100
80
60
40
20
0
PPM
kwq-14-166
3f
Et
N
n-Bu
Ph
H3C


kwq-14-131
3g
H3CO
n-Bu
N
Et
CH3
PPM
0 1 2 3 4 5 6 7 8
0.99 0.99 1.04
0.96 0.96
2.00
2.98
1.99
2.99
2.00
2.01
3.04
3.05
7.640
7.632
7.326
7.296
7.260
7.123
7.115
7.094
7.086
7.055
6.836
4.348
4.324
4.300
4.276
3.954
3.207
3.181
3.154
2.557
1.883
1.857
1.831
1.594
1.570
1.439
1.415
1.391
1.062
1.038
1.014
0.111
0.035

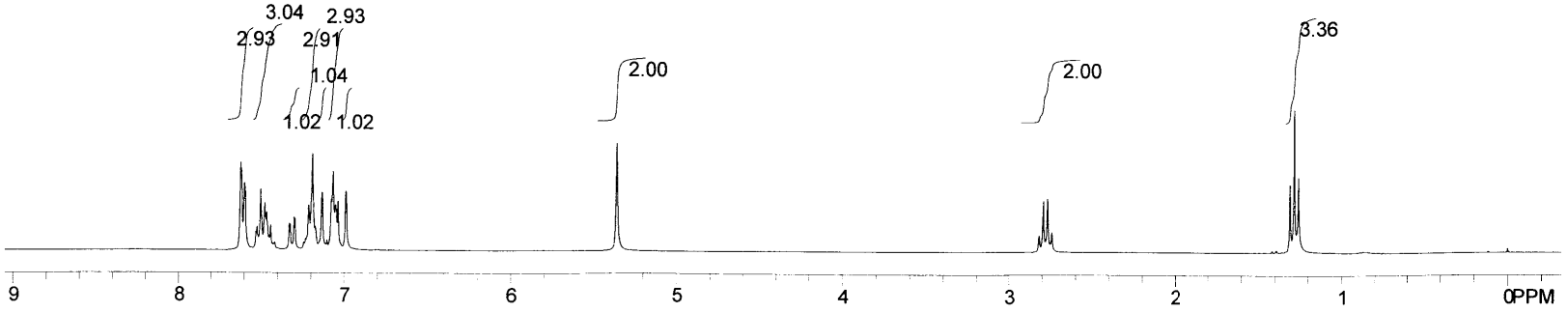

160
140
120
100
80
60
40
20
PPM
kwq-14-131
H3CO
n-Bu
N
Et
CH3
3g
153.240
141.344
138.154
135.559
135.048
123.261
120.756
118.280
112.663
108.448
106.346
106.324
77.430
77.000
76.582
56.166
37.410
34.148
32.122
23.054
22.134
14.107
13.741

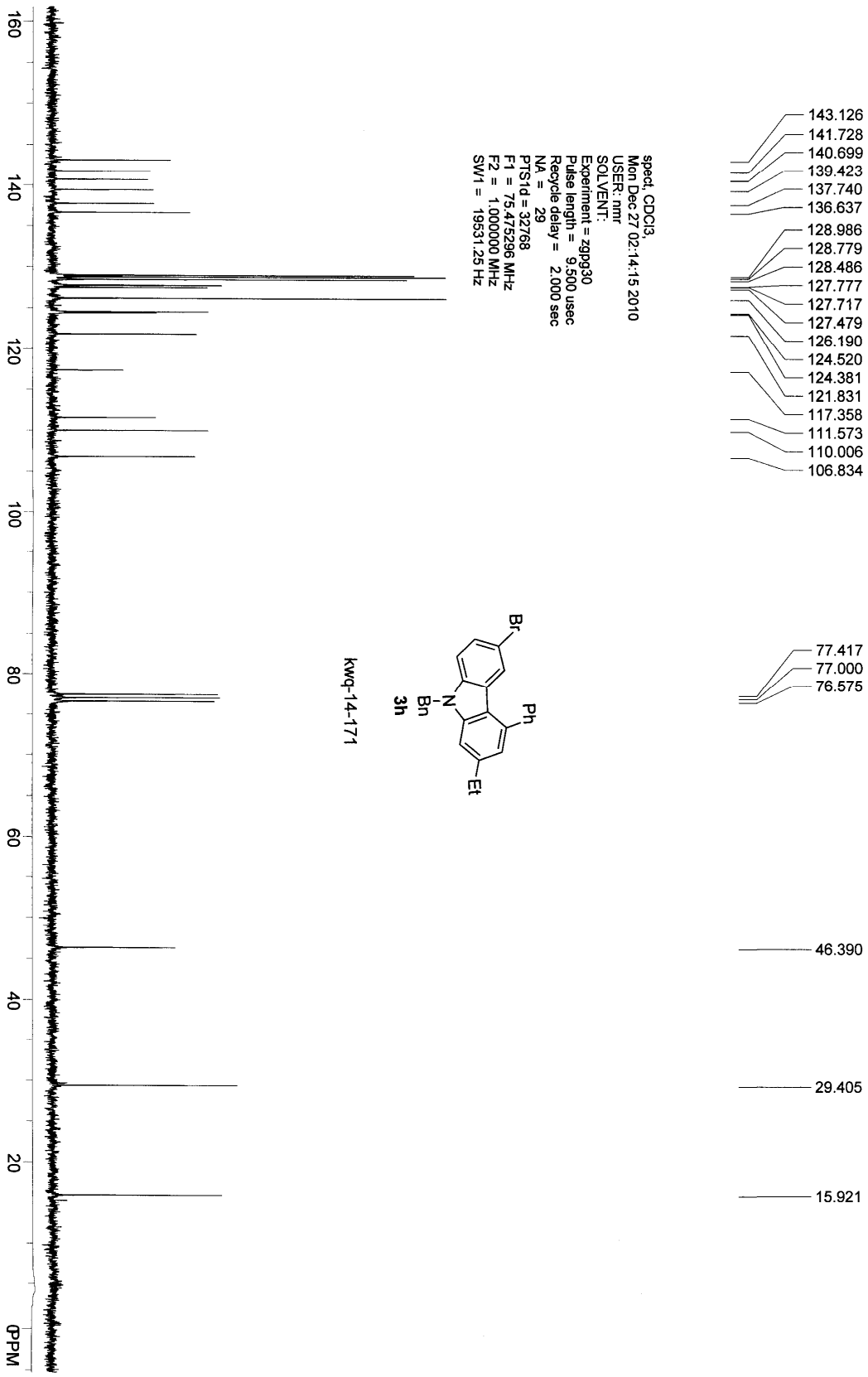

7.621
7.616
7.598
7.594
7.530
7.523
7.517
7.501
7.476
7.471
7.465
7.452
7.442
7.330
7.324
7.301
7.295
7.232
7.214
7.191
7.175
7.133
7.077
7.066
7.052
7.037
6.990
6.987
5.357
2.818
2.793
2.767
2.742
1.308
1.283
1.258

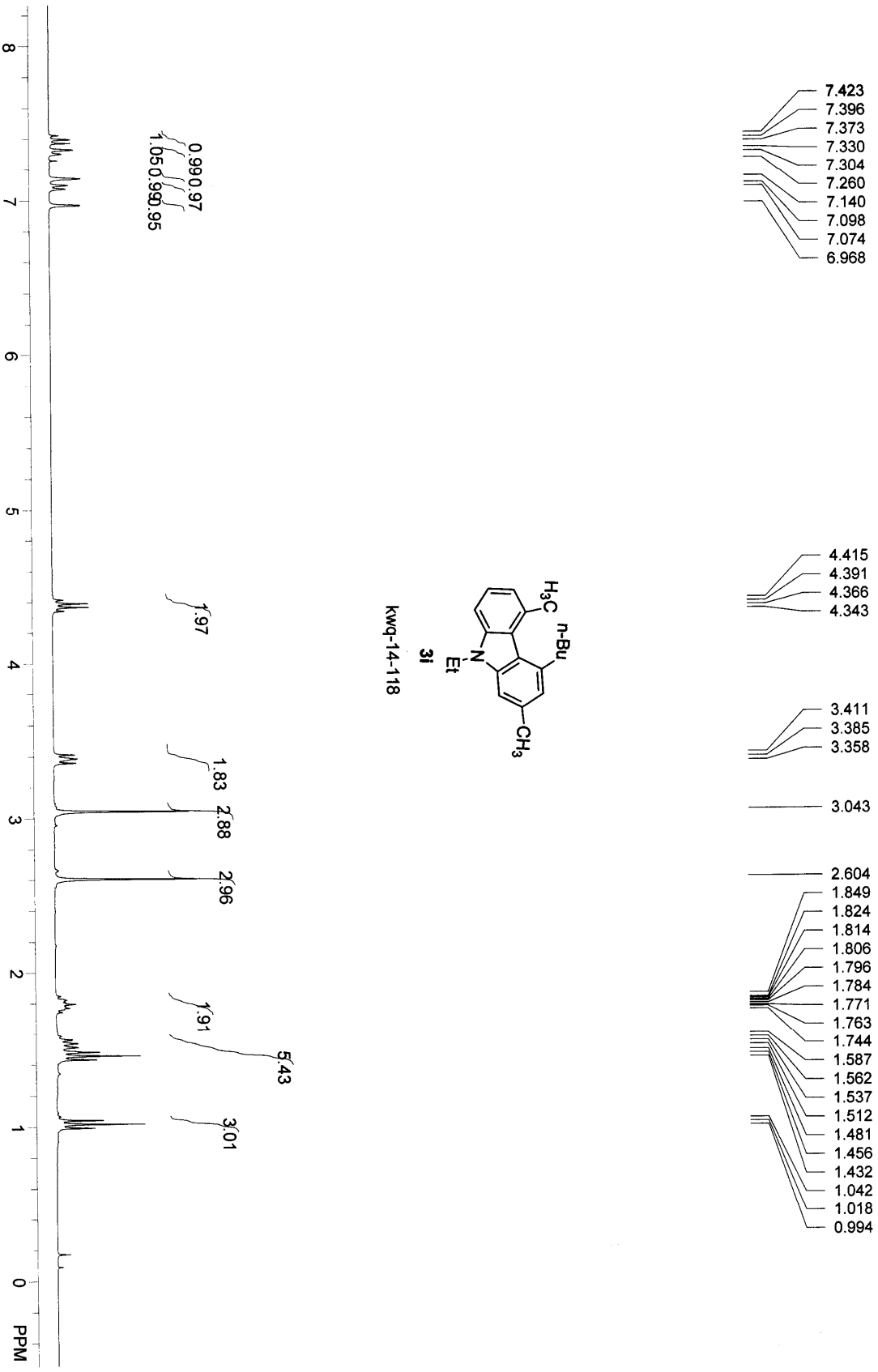

Br
Ph
Et
N
Bn

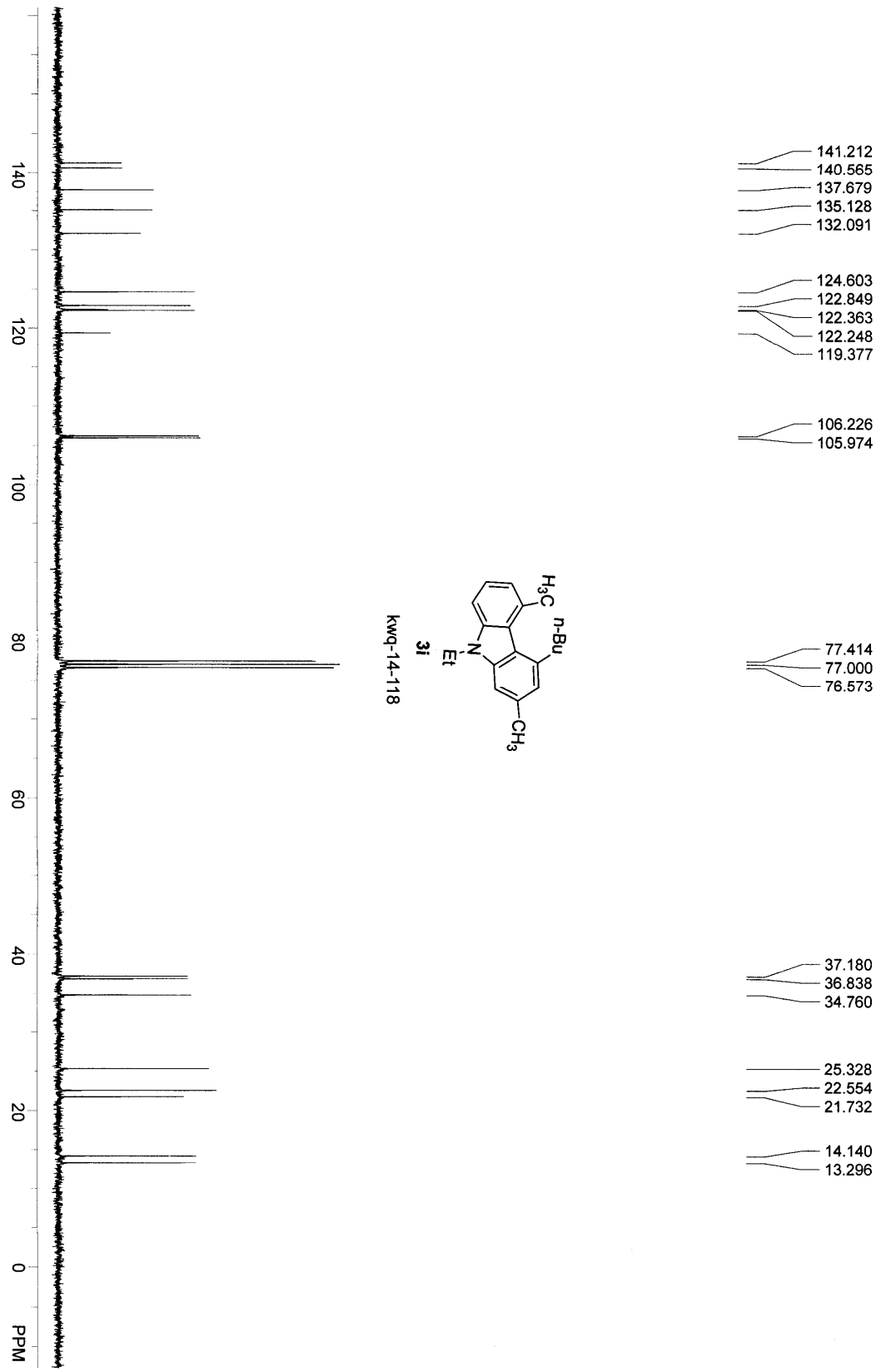
3h

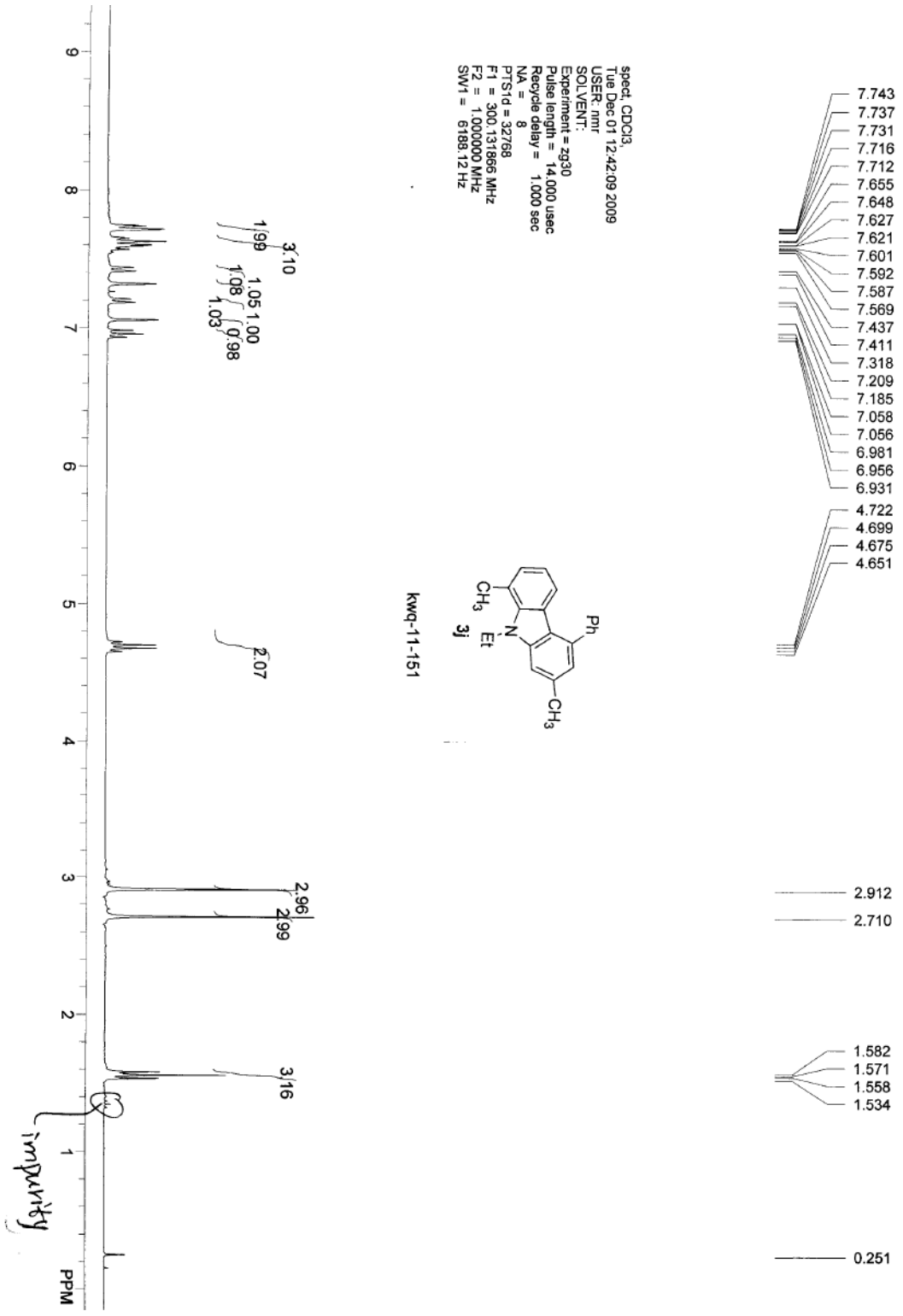
kwq-14-171

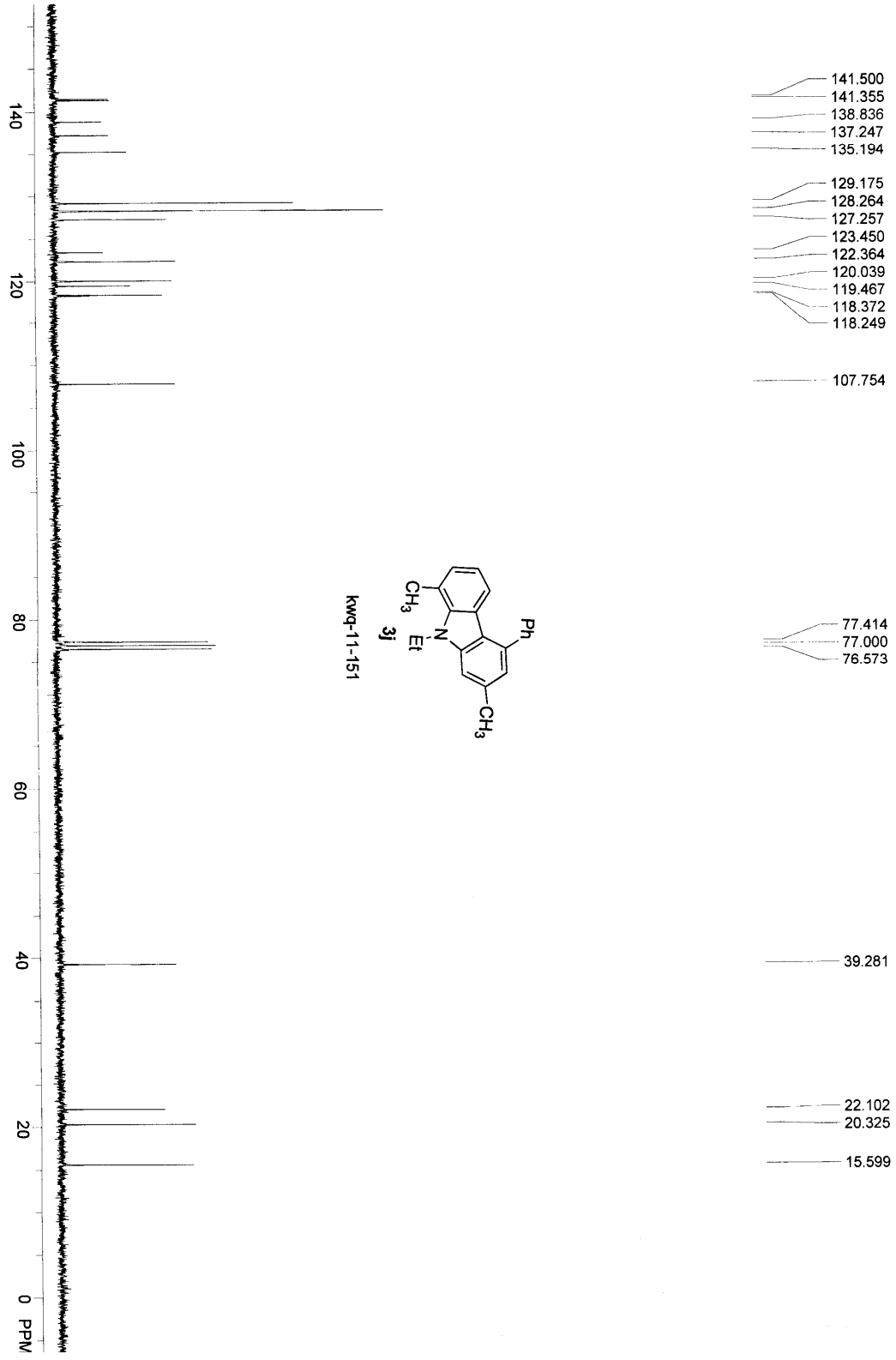

3.04
2.93
2.93
2.91
1.04
1.02
1.02
2.00
2.00
3.36
9
8
7
6
5
4
3
2
1
0PPM

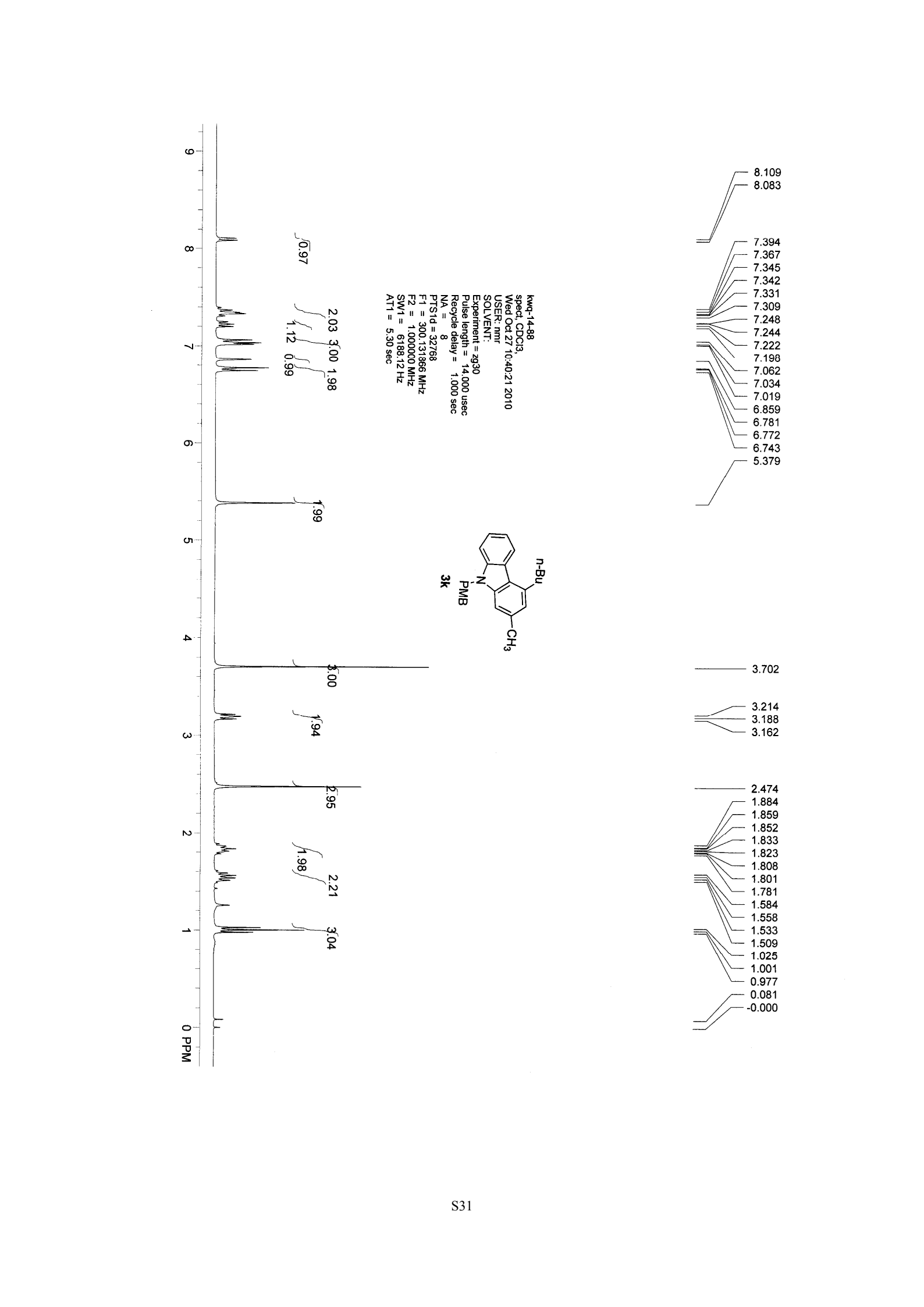

spect, CDCl3,
Mon Dec 27 02:14:15 2010
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 29
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
kwq-14-171
3h
Br
Ph
Et
N
Bn
143.126
141.728
140.699
139.423
137.740
136.637
128.986
128.779
128.486
127.777
127.717
127.479
126.190
124.520
124.381
121.831
117.358
111.573
110.006
106.834
77.417
77.000
76.575
46.390
29.405
15.921
160
140
120
100
80
60
40
20
0
PPM

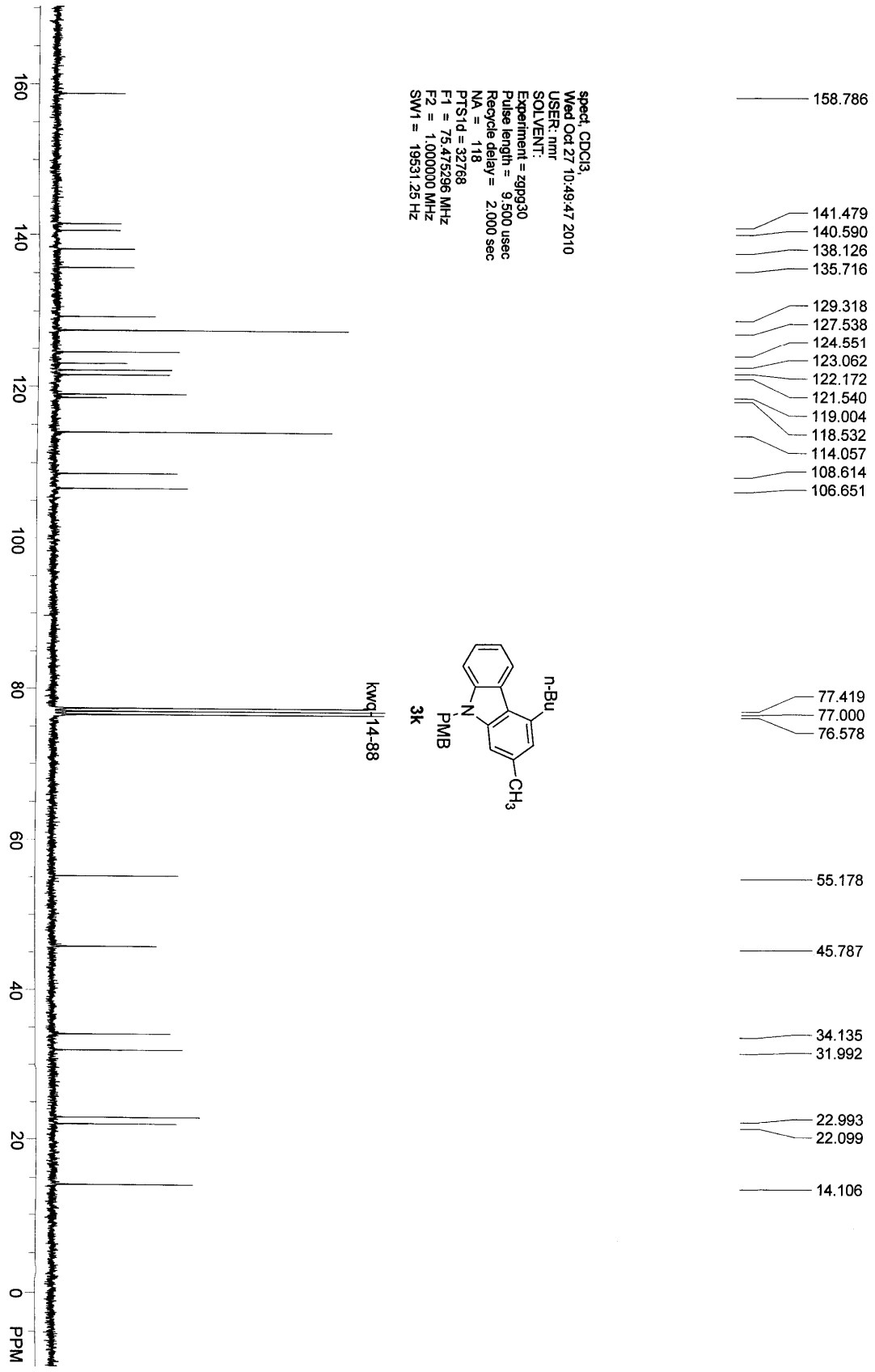

7.423
7.396
7.373
7.330
7.304
7.260
7.140
7.098
7.074
6.968
4.415
4.391
4.366
4.343
3.411
3.385
3.358
3.043
2.604
1.849
1.824
1.814
1.806
1.796
1.784
1.771
1.763
1.744
1.587
1.562
1.537
1.512
1.481
1.456
1.432
1.042
1.018
0.994
H3C
n-Bu
N
Et
CH3
3i
kwq-14-118
0.99
0.97
1.05
0.99
0.95
1.97
1.83
2.88
2.96
1.91
5.43
3.01
8
7
6
5
4
3
2
1
0
PPM

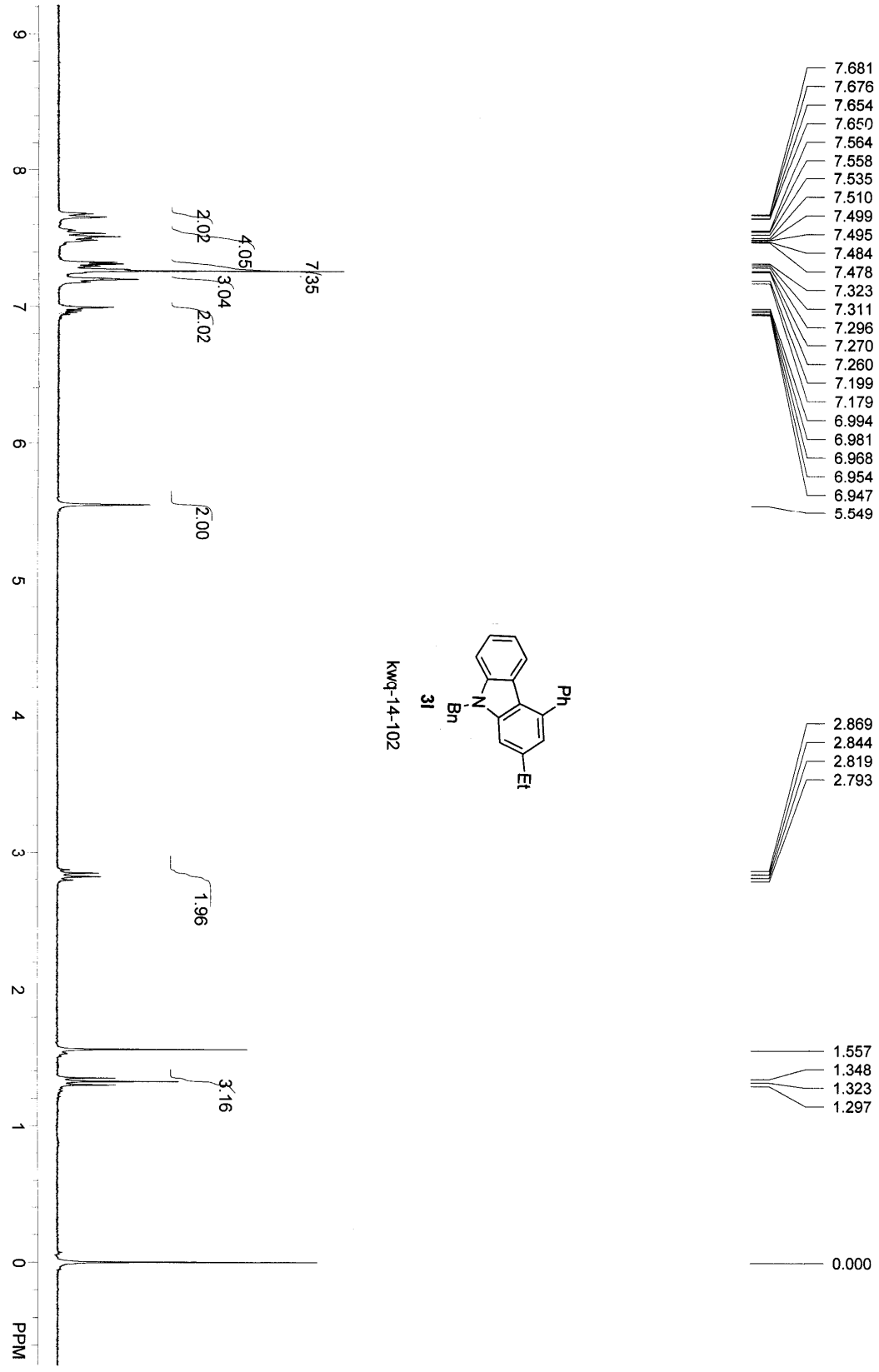

141.212
140.565
137.679
135.128
132.091
124.603
122.849
122.363
122.248
119.377
106.226
105.974
77.414
77.000
76.573
37.180
36.838
34.760
25.328
22.554
21.732
14.140
13.296
140
120
100
80
60
40
20
0
PPM
kwq-14-118
3i
Et
N
n-Bu
H3C
CH3

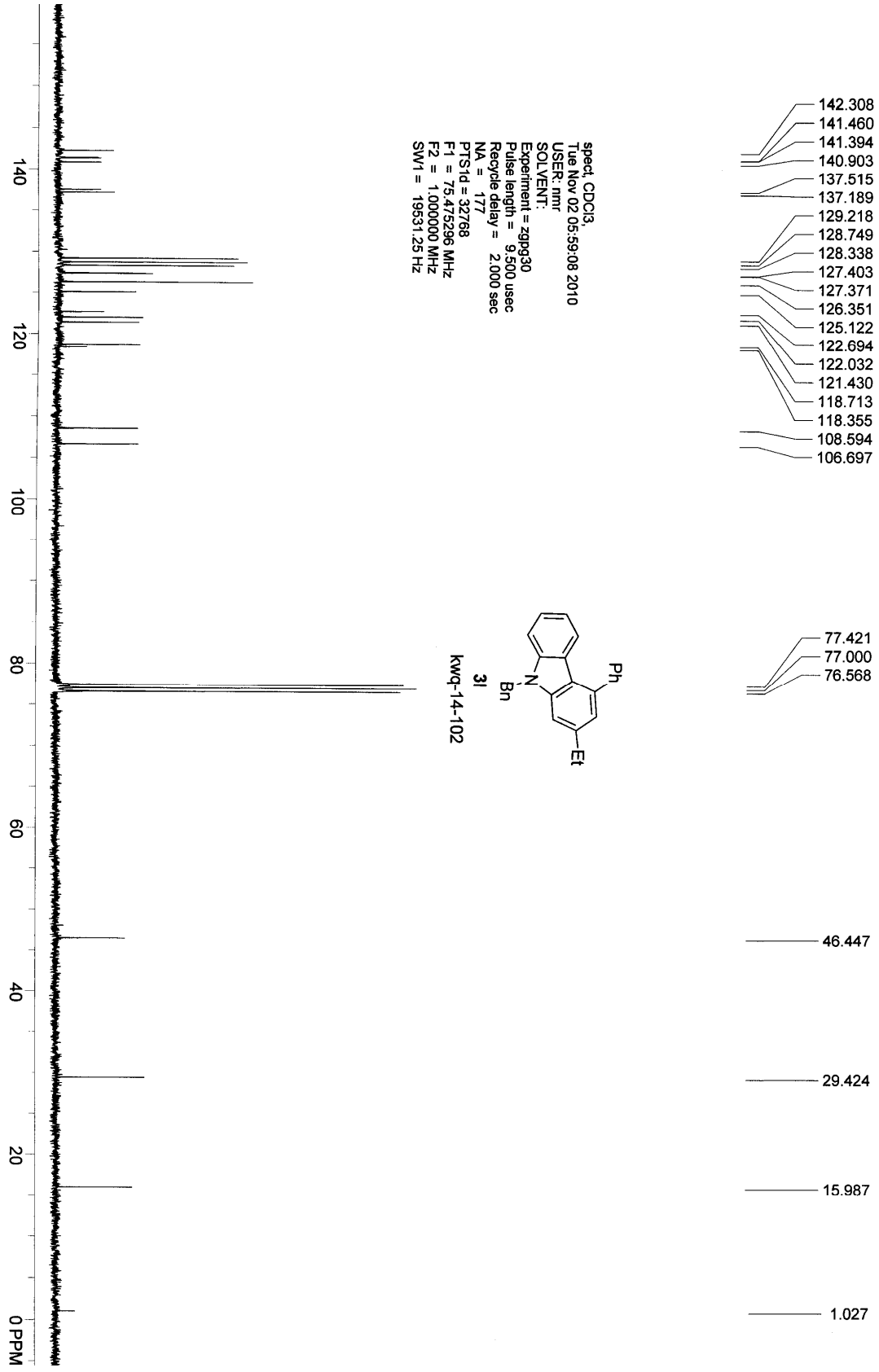

spect. CDCl3
Tue Dec 01 12:42:09 2009
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
kwq-11-151
3j
Ph
CH3
N
Et
CH3
7.743
7.737
7.731
7.716
7.712
7.655
7.648
7.627
7.621
7.601
7.592
7.587
7.569
7.437
7.411
7.318
7.209
7.185
7.058
7.056
6.981
6.956
6.931
4.722
4.699
4.675
4.651
2.912
2.710
1.582
1.571
1.558
1.534
0.251
3.10
1.99
1.05
1.00
1.08
0.98
1.03
2.07
2.96
2.99
3.16
impurity
PPM


141.500
141.355
138.836
137.247
135.194
129.175
128.264
127.257
123.450
122.364
120.039
119.467
118.372
118.249
107.754
77.414
77.000
76.573
39.281
22.102
20.325
15.599
kwq-11-151
3j
CH3
N
Et
Ph
CH3
140
120
100
80
60
40
20
0 PPM


kwq-14-88
spect, CDCl3,
Wed Oct 27 10:40:21 2010
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
AT1 = 5.30 sec
n-Bu
N
PMB
CH3
3k
8.109
8.083
7.394
7.367
7.345
7.342
7.331
7.309
7.248
7.244
7.222
7.198
7.062
7.034
7.019
6.859
6.781
6.772
6.743
5.379
3.702
3.214
3.188
3.162
2.474
1.884
1.859
1.852
1.833
1.823
1.808
1.801
1.781
1.584
1.558
1.533
1.509
1.025
1.001
0.977
0.081
-0.000
0.97
2.03
3.00
1.98
1.12
0.99
1.99
3.00
1.94
2.95
1.98
2.21
3.04
9
8
7
6
5
4
3
2
1
0 PPM


spect, CDCl3,
Wed Oct 27 10:49:47 2010
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 118
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
kwq14-88
3k
n-Bu
N
PMB
CH3
158.786
141.479
140.590
138.126
135.716
129.318
127.538
124.551
123.062
122.172
121.540
119.004
118.532
114.057
108.614
106.651
77.419
77.000
76.578
55.178
45.787
34.135
31.992
22.993
22.099
14.106
160
140
120
100
80
60
40
20
0
PPM


7.681
7.676
7.654
7.650
7.564
7.558
7.535
7.510
7.499
7.495
7.484
7.478
7.323
7.311
7.296
7.270
7.260
7.199
7.179
6.994
6.981
6.968
6.954
6.947
5.549
2.869
2.844
2.819
2.793
1.557
1.348
1.323
1.297
0.000
7.35
4.05
2.02
3.04
2.02
2.00
1.96
3.16
PPM
kwq-14-102
3l
Ph
N
Bn
Et


spect, CDCl3,
Tue Nov 02 05:59:08 2010
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 177
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz
3l
kwq-14-102
Ph
N
Bn
Et
142.308
141.460
141.394
140.903
137.515
137.189
129.218
128.749
128.338
127.403
127.371
126.351
125.122
122.694
122.032
121.430
118.713
118.355
108.594
106.697
77.421
77.000
76.568
46.447
29.424
15.987
1.027
140
120
100
80
60
40
20
0 PPM