Synthesis of functionalized *H*-pyrazolo[5,1-*a*]isoquinolines via sequential reactions of *N'*-(2-alkynylbenzylidene)hydrazides

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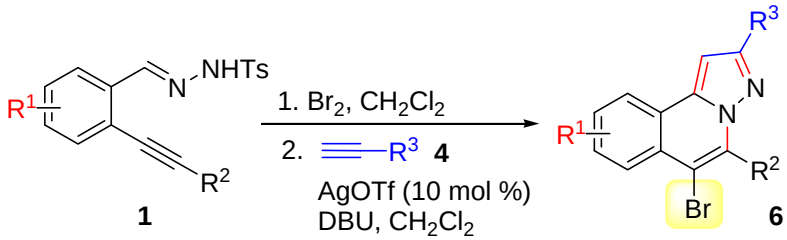
Supporting Information

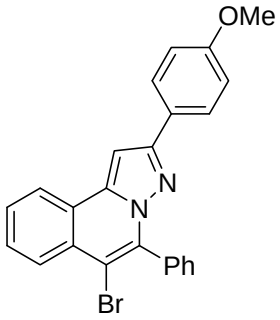
General experimental methods:

All reactions were performed in reaction tubes. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35°C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

General procedure for reactions of N'-(2-alkynylbenzylidene)hydrazides with bromine and alkynes

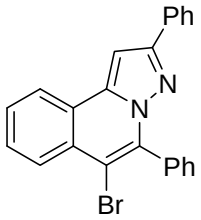


Bromine (0.3 mmol, 1.0 equiv) in 2.0 mL of CH_2Cl_2 was added dropwisely to a mixture of *N*'-(2-alkynylbenzylidene)hydrazide **1** (0.30 mmol) in CH_2Cl_2 (4.0 mL). The reaction was stirred at room temperature. After completion of reaction as indicated by TLC, the reaction mixture was then diluted with CH_2Cl_2 (25 mL), washed with saturated aqueous $Na_2S_2O_3$ (25 mL), dried (Na_2SO_4) and filtered. The solvent was then evaporated and the residue was dissolved in 2.0 mL of CH_2Cl_2. Then DBU (2.5 equiv) and AgOTf (10 mol %) were added. Subsequently, alkyne **4** (1.5 equiv) in 1.0 mL of CH_2Cl_2 was added dropwisely at room temperature under air atmosphere. The reaction mixture was stirred at room temperature for about 5 hours. After completion of reaction as indicated by TLC, the mixture was diluted with CH_2Cl_2, washed by water. The organic layer was combined, dried over Na_2SO_4, and purified by column chromatography on silica gel to afford the desired product **6**.



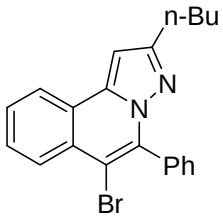
6a. 6-bromo-2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

Yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.81 (s, 3H), 6.89-6.91 (m, 2H), 7.28 (s, 1H), 7.54-7.65 (m, 7H), 7.76-7.79 (m, 2H), 8.12-8.15 (m, 1H), 8.21-8.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.4, 94.6, 108.5, 114.1, 123.8, 123.9, 125.8, 127.8, 127.9, 128.2, 128.3, 128.6, 128.7, 129.3, 130.7, 134.2, 137.9, 139.7, 152.6, 159.9; IR (cm^{-1}): 2965, 2904, 2832, 1618, 1521, 1459, 1434, 1378, 1251, 1173, 1024; *m/z* (ESI): 429 (M^++H); HRMS calcd for $C_{24}H_{18}BrN_2O$ (M+H) 429.0603, found 429.0621.



6b. 6-bromo-2,5-diphenylpyrazolo[5,1-a]isoquinoline

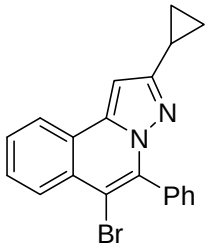
Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): 7.27-7.31 (m, 1H), 7.34-7.38 (m, 3H), 7.52-7.64 (m, 7H), 7.84-7.86 (m, 2H), 8.12-8.14 (m, 1H), 8.20-8.22 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 95.1, 108.9, 123.7, 123.9, 126.5, 127.9, 128.3, 128.4, 128.5, 128.6, 128.7, 129.3, 130.7, 133.0, 134.1, 137.9, 139.7, 152.7. IR (cm^{-1}): 3052, 2945, 2919, 2842, 1618, 1598, 1540, 1492, 1470, 1456, 1381, 1319, 1173, 1079; *m/z* (ESI): 399 (M^++H); HRMS calcd for $C_{23}H_{16}BrN_2$ (M+H) 399.0497, found 399.0513.



6c. 6-bromo-2-butyl-5-phenylpyrazolo[5,1-a]isoquinoline

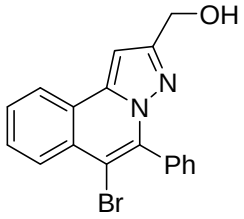
Yield: 71%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.92 (t, *J* = 7.3 Hz, 3H), 1.36-1.41 (m, 2H),

1.64-1.71 (m, 2H), 2.75 (t, *J* = 7.80 Hz, 2H), 6.86 (s, 1H), 7.51-7.62 (m, 7H), 8.05-8.07 (m, 1H), 8.16-8.19 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.6, 28.4, 31.9, 96.7, 107.9, 123.6, 123.8, 127.7, 128.1, 128.4, 128.5, 129.3, 130.5, 134.4, 137.7, 139.0, 155.9. IR (cm^{-1}): 3057, 2955, 2928, 2852, 1588, 1541, 1493, 1481, 1465, 1383, 1322; *m/z* (ESI): 379 (M^++H); HRMS calcd for $C_{21}H_{20}BrN_2$ (M+H) 379.0810, found 379.0828.



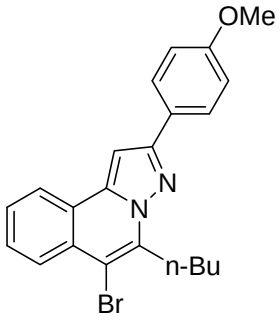
6d. 6-bromo-2-cyclopropyl-5-phenylpyrazolo[5,1-a]isoquinoline

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.74-0.78 (m, 2H), 0.93-0.99 (m, 2H), 2.04-2.12 (m, 1H), 6.61 (s, 1H), 7.51-7.61 (m, 7H), 8.00-8.02 (m, 1H), 8.16-8.18 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.31, 9.84, 93.2, 107.8, 123.5, 123.6, 127.7, 128.1, 128.4, 128.5, 129.3, 130.5, 134.3, 137.6, 139.1, 157.9. IR (cm^{-1}): 2955, 2924, 2854, 1618, 1588, 1543, 1492, 1444, 1383, 1337, 1045; *m/z* (ESI): 363 (M^++H); HRMS calcd for $C_{20}H_{16}BrN_2$ (M+H) 363.0497, found 363.0510.



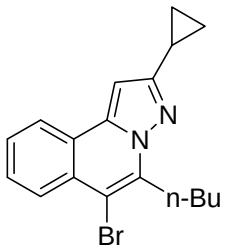
6e. (6-bromo-5-phenylpyrazolo[5,1-a]isoquinolin-2-yl)methanol

Yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$)): 4.70 (s, 2H), 7.00 (s, 1H), 7.45-7.62 (m, 7H), 8.01-8.04 (m, 1H), 8.15-8.18 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 59.0, 96.6, 109.1, 123.6, 123.7, 127.8, 128.3, 128.4, 128.6, 128.8, 129.5, 130.3, 134.0, 137.5, 139.2, 154.3. IR (cm^{-1}): 3401, 3052, 2925, 2847, 1613, 1588, 1540, 1492, 1444, 1411, 1381, 1319, 1032; *m/z* (ESI): 353 (M^++H); HRMS calcd for $C_{18}H_{14}BrN_2O$ (M+H) 353.0290, found 353.0304.



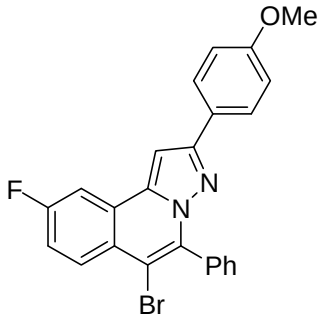
6f. 6-bromo-5-butyl-2-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline

Yield: 72%; ^{1}H NMR (400 MHz, $CDCl_3$): 1.03 (t, *J* = 7.32 Hz, 3H), 1.54-1.58 (m, 2H), 1.83-1.87 (m, 2H), 3.56 (t, *J* = 7.80 Hz, 2H), 3.86 (s, 3H), 6.99-7.01 (m, 2H), 7.21 (s, 1H), 7.50-7.59 (m, 2H), 7.92-7.98 (m, 2H), 8.04-8.06 (m, 1H), 8.08-8.12(m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.9, 28.8, 31.3, 55.4, 94.4, 107.4, 114.2, 123.2, 123.6, 126.1, 127.1, 127.4, 127.7, 128.3, 128.4, 139.1, 139.5, 152.1, 159.9. IR (cm^{-1}): 2960, 2919, 2852, 1613, 1525, 1460, 1437, 1316, 1250, 1174, 1029; *m/z* (ESI): 409 (M^++H); HRMS calcd. for $C_{22}H_{22}BrN_2O$ (M+H) 409.0916, found 409.0913.



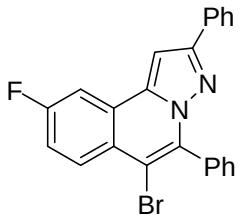
6g. 6-bromo-5-butyl-2-cyclopropylpyrazolo[5,1-a]isoquinoline

Yield: 61%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.89-0.94 (m, 2H), 0.99 (t, *J* = 7.36 Hz, 3H), 1.03-1.07 (m, 2H), 1.47-1.56 (m, 2H), 1.74-1.81 (m, 2H), 2.14-2.19 (m, 1H), 3.48 (t, *J* = 7.36 Hz, 2H), 6.64 (s, 1H), 7.46-7.57 (m, 2H), 7.94-7.97 (m, 1H), 8.06-8.08 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.15, 9.77, 14.0, 22.8, 28.9, 31.3, 94.1, 106.7, 122.9, 123.5, 127.0, 127.2, 128.3, 128.4, 138.6, 139.3, 157.1. IR (cm^{-1}): 2956, 2926, 2847, 1618, 1598, 1541, 1496, 1449, 1392, 1316, 1070; *m/z* (ESI): 343 (M^++H); HRMS calcd. for $C_{18}H_{20}BrN_2$ (M+H) 343.0810, found 343.0819.



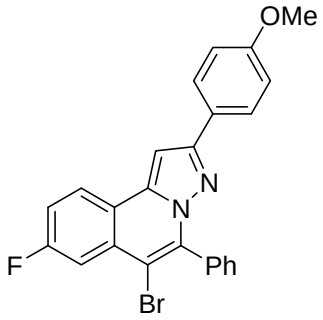
6h. 6-bromo-9-fluoro-2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.82 (s, 3H), 6.89-6.92 (m, 2H), 7.24 (s, 1H), 7.33-7.37 (m, 1H), 7.54-7.60 (m, 5H), 7.75-7.81 (m, 3H), 8.20-8.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 95.2, 107.8, 108.9 (d, $^{2}J_{CF}$ = 22.9 Hz), 114.1, 117.1 (d, $^{2}J_{CF}$ = 22.9 Hz), 125.1, 125.2, 125.5, 127.8, 128.4, 129.4, 130.6, 130.7, 133.9, 137.3, 138.9, 152.7, 160.0, 162.2 (d, $^{1}J_{CF}$ = 248.9 Hz). IR (cm^{-1}): 2960, 2924, 2842, 1603, 1516, 1485, 1458, 1434, 1372, 1250, 1178, 1034; *m/z* (ESI): 447 (M^{+}+H); HRMS calcd for $C_{24}H_{17}BrFN_2O$ (M+H) 447.0508, found 447.0512.



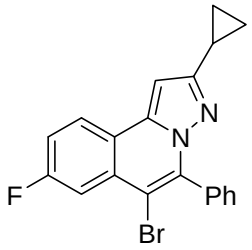
6i. 6-bromo-9-fluoro-2,5-diphenylpyrazolo[5,1-a]isoquinoline

Yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$): 7.35-7.54 (m, 7H), 7.59-7.65 (m, 1H), 7.70-7.73 (m, 4H), 8.14-8.17 (m, 1H), 8.38-8.41 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 86.1, 95.5, 110.9(d, $^{2}J_{CF}$ = 21.9 Hz), 118.1, 119.2, 121.8, 122.4(d, $^{2}J_{CF}$ = 25.7 Hz), 124.4, 128.1, 128.6, 129.7, 129.9, 132.3, 133.6, 140.3, 142.6, 147.2, 152.5, 161.9 (d, $^{1}J_{CF}$ = 250.8 Hz). IR (cm^{-1}): 3057, 2960, 2919, 2842, 1618, 1546, 1493, 1439, 1419, 1393, 1301, 1170; *m/z* (ESI): 317 (M^{+}+H); HRMS calcd for $C_{23}H_{15}BrFN_2$ (M+H) 417.0403, found 417.0413.



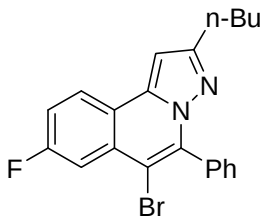
6j. 6-bromo-8-fluoro-2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

Yield: 65%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.80 (s, 3H), 6.87-6.89 (m, 2H), 7.18 (s, 1H), 7.29-7.34 (m, 1H), 7.54-7.58 (m, 5H), 7.73-7.76 (m, 2H), 7.85-7.89(m, 1H), 8.06-8.10(m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 94.3, 107.2, 113.4 (d, $^{2}J_{CF}$ = 24.8 Hz), 114.0, 116.8 (d, $^{2}J_{CF}$ = 23.8 Hz), 120.4, 125.5, 126.1, 127.7, 128.3, 129.4, 130.6, 130.7, 133.9, 138.9, 139.3, 152.8, 159.9, 162.6 (d, $^{1}J_{CF}$ = 246.9 Hz). IR (cm^{-1}): 3057, 2924, 2831, 1614, 1518, 1477, 1451, 1437, 1380, 1250, 1171, 1026; *m/z* (ESI): 447 (M^{+}+H); HRMS calcd for $C_{24}H_{17}BrFN_2O$ (M+H) 447.0508, found 447.0515.



6k. 6-bromo-2-cyclopropyl-8-fluoro-5-phenylpyrazolo[5,1-a]isoquinoline

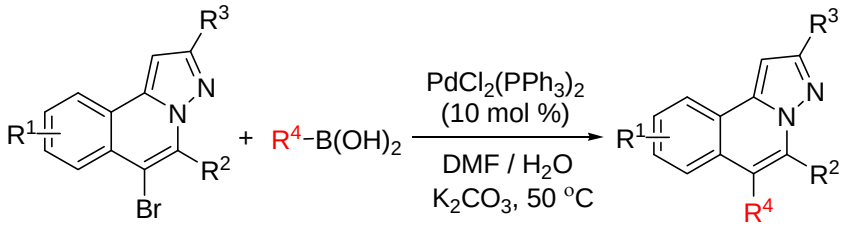
Yield: 65%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.74-0.79 (m, 2H), 0.96-0.99 (m, 2H), 2.04-2.08 (m, 1H), 6.56 (s, 1H), 7.27-7.32 (m, 1H), 7.51-7.59 (m, 5H), 7.84-7.87 (m, 1H), 7.99-8.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.33, 9.83, 93.1, 113.3 (d, $^{2}J_{CF}$ = 24.8 Hz), 116.7 (d, $^{2}J_{CF}$ = 23.8 Hz), 120.2, 125.9, 126.1, 128.5, 129.5, 130.3, 130.6, 134.0, 138.7, 138.8, 158.3, 162.5 (d, $^{1}J_{CF}$ = 246.0 Hz). IR (cm^{-1}): 3052, 2961, 2909, 2852, 1621, 1544, 1499, 1445, 1398, 1378, 1268, 1166; *m/z* (ESI): 381 (M^{+}+H); HRMS calcd. for $C_{20}H_{15}BrFN_2$ (M+H) 381.0403, found 381.0412.



6l. 6-bromo-2-butyl-8-fluoro-5-phenylpyrazolo[5,1-a]isoquinoline

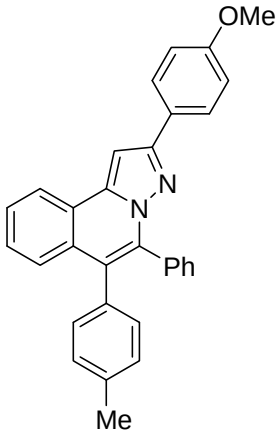
Yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.92 (t, J = 7.32 Hz, 3H), 1.34-1.41 (m, 2H), 1.64-1.68 (m, 2H), 2.74 (t, J = 7.80 Hz, 2H), 6.81 (s, 1H), 7.29-7.34 (m, 1H), 7.50-7.56 (m, 5H), 7.85-7.88 (m, 1H), 8.03-8.07 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 22.6, 28.3, 31.9, 96.5, 106.7, 113.3 (d, $^{2}J_{CF}$ = 24.8 Hz), 116.7(d, $^{2}J_{CF}$ = 23.8 Hz), 120.4, 126.1, 128.5, 129.5, 130.4, 130.5, 130.6, 134.1, 138.7, 156.3, 162.5 (d, $^{1}J_{CF}$ = 246.9 Hz). IR (cm^{-1}): 3057, 2955, 2927, 2858, 1621, 1541, 1484, 1444, 1398, 1378, 1311, 1269, 1166; *m/z* (ESI): 397 (M^{+}+H); HRMS calcd. for $C_{21}H_{19}BrFN_2$ (M+H) 397.0716, found 397.0715.

General procedure for palladium-catalyzed Suzuki couplings of 6-bromoH-pyrazolo[5,1-a]isoquinoline 6



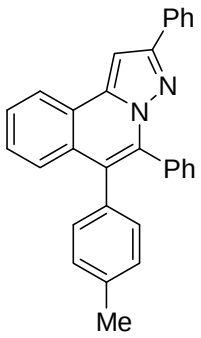
6 **7**

A mixture of 6-bromo*H*-pyrazolo[5,1-a]isoquinoline **6** (0.12 mmol), arylboronic acid (1.2 equiv), $PdCl_2(PPh_3)_2$ (10 mol %) and K_2CO_3 (2.0 equiv) in 1.0 mL of DMF/H_2O (5:1, v/v) was stirred under N_2 atmosphere at 50~60 °C. After completion of reaction as indicated by TLC, the mixture was cooled to room temperature and water (10 mL) was added. The mixture was extracted with ethyl acetate (5.0 mL × 3) and the organic layer was combined, which was then washed with brine, dried over Na_2SO_4, and purified by column chromatography on silica gel to afford the desired product **7**.



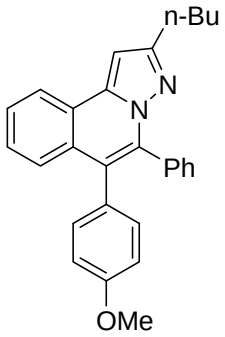
7a. 2-(4-methoxyphenyl)-5-phenyl-6-p-tolylpyrazolo[5,1-a]isoquinoline

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): 2.32 (s, 3H), 3.81 (s, 3H), 6.88-6.92 (m, 2H), 7.05-7.09 (m, 4H), 7.26-7.29 (m, 3H), 7.32 (s, 1H), 7.38-7.44 (m, 4H), 7.52-7.55 (m, 1H), 7.82-7.85 (m, 2H), 8.18 (d J = 8.24 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 55.4, 94.1, 114.0, 123.5, 123.6, 123.9, 126.3, 126.9, 127.2, 127.5, 127.6, 127.7, 128.0, 128.8, 130.3, 131.5, 131.6, 133.3, 133.4, 136.4, 136.7, 139.9, 152.1, 159.7. IR (cm^{-1}): 2945, 2914, 2837, 1608, 1526, 1458, 1429, 1248, 1163, 1024; *m/z* (ESI): 441 (M^++H); HRMS calcd. for $C_{31}H_{25}N_2O$ (M+H) 441.1967, found 441.1980.



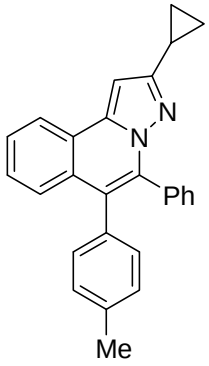
7b. 2,5-diphenyl-6-p-tolylpyrazolo[5,1-a]isoquinoline

Yield: 83%; ^{1}H NMR (400 MHz, $CDCl_3$): 2.33 (s, 3H), 7.06-7.11 (m, 4H), 7.24-7.32 (m, 4H), 7.36-7.44 (m, 7H), 7.53-7.59 (m, 1H), 7.90-7.93 (m, 2H), 8.20-8.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 94.6, 123.6, 123.9, 124.0, 126.5, 126.9, 127.3, 127.6, 127.7, 128.0, 128.1, 128.6, 128.8, 130.3, 131.5, 131.6, 133.3, 133.5, 136.5, 136.8, 139.9, 152.2. IR (cm^{-1}): 3052, 3016, 2914, 2858, 1588, 1536, 1506, 1456, 1378, 1342, 1178, 1081, 1014; *m/z* (ESI): 411 (M^++H); HRMS calcd. for $C_{30}H_{23}N_2$ (M+H) 411.1861, found 411.1873.



7c. 2-butyl-6-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

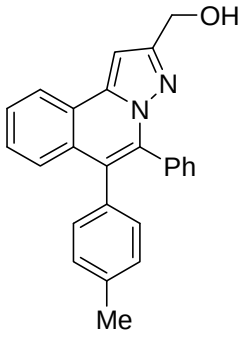
Yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.94 (t, J = 7.32 Hz, 3H), 1.39-1.45 (m, 2H), 1.68-1.74 (m, 2H), 2.80 (t, J = 7.80 Hz, 2H), 3.78 (s, 3H), 6.77-6.81 (m, 2H), 6.91 (s, 1H), 7.04-7.08 (m, 2H), 7.24-7.29 (m, 3H), 7.31-7.34 (m, 2H), 7.38-7.41 (m, 2H), 7.48-7.54 (m, 1H), 8.13 (d, J = 7.76 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.7, 28.5, 32.1, 55.2, 96.1, 113.5, 122.6, 123.5, 123.8, 126.7, 127.0, 127.4, 127.8, 128.1, 128.7, 130.3, 131.2, 132.8, 133.5, 136.4, 139.2, 155.4, 158.5. IR (cm^{-1}): 3052, 2955, 2929, 2858, 1603, 1512, 1475, 1460, 1337, 1245, 1168, 1034; *m/z* (ESI): 407 (M^{+}+H); HRMS calcd. for $C_{28}H_{27}N_2O$ (M+H) 407.2123, found 407.2141.



7d. 2-cyclopropyl-5-phenyl-6-p-tolylpyrazolo[5,1-a]isoquinoline

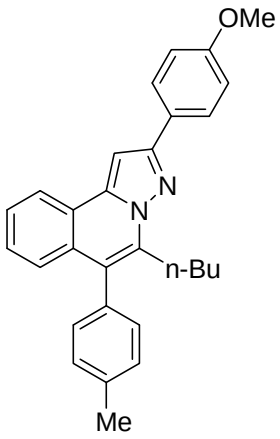
Yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.80-0.84 (m, 2H), 0.96-1.04 (m, 2H), 2.10-2.14 (m, 1H), 2.31 (s, 3H), 6.65 (s, 1H), 7.02-7.07 (m, 4H), 7.25-7.28 (m, 3H), 7.31-7.36 (m, 2H), 7.37-7.40 (m, 2H), 7.48-7.51 (m, 1H), 8.07 (d, J = 8.28 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.28, 9.96, 21.3, 92.6, 122.8, 123.4, 123.7, 126.7, 127.0, 127.5, 127.7, 128.1, 128.7, 130.2, 131.3, 131.6, 133.3, 133.4, 136.1, 136.6, 139.4, 157.4. IR (cm^{-1}): 3042, 2955, 2923, 2852, 1536, 1513, 1493, 1449, 1398, 1342, 1301, 1178, 1075; *m/z* (ESI): 375 (M^{+}+H); HRMS calcd. for $C_{27}H_{23}N_2$ (M+H)

375.1861, found 375.1878.



7e. (5-phenyl-6-p-tolylpyrazolo[5,1-a]isoquinolin-2-yl)methanol

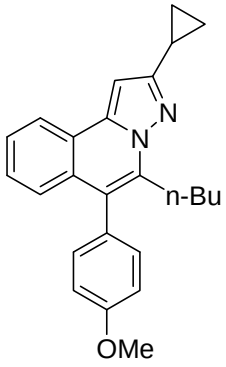
Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$)): 2.32 (s, 3H), 4.84 (s, 2H), 7.04-7.09 (m, 5H), 7.27-7.33 (m, 5H), 7.41-7.43 (m, 2H), 7.54-7.57 (m, 1H), 8.15 (d, *J* = 8.24 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 59.6, 95.9, 123.6, 123.9, 124.0, 126.9, 127.4, 127.9, 128.3, 128.8, 130.2, 131.2, 131.5, 133.1, 133.2, 136.2, 136.8, 139.6, 153.7. IR (cm^{-1}): 3354, 3052, 2920, 2847, 1593, 1536, 1513, 1490, 1454, 1388, 1332, 1033; *m/z* (ESI): 365 (M^{+}+H); HRMS calcd for $C_{25}H_{21}N_2O$ (M+H) 365.1654, found 365.1665.



7f. 5-butyl-2-(4-methoxyphenyl)-6-p-tolylpyrazolo[5,1-a]isoquinoline

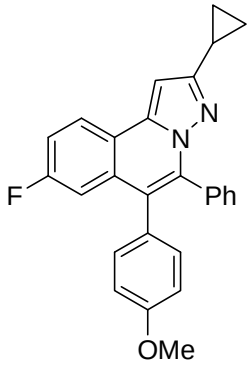
Yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.86 (t, *J* = 7.32 Hz, 3H), 1.31-1.38 (m, 2H), 1.74-1.79 (m, 2H), 2.48 (s, 3H), 3.03 (t, *J* = 7.80 Hz, 2H), 3.87 (s, 3H), 6.98-7.03 (m, 2H), 7.18-7.25 (m, 4H), 7.31-7.38 (m, 3H), 7.45-7.49 (m, 1H), 7.97-8.02 (m, 2H), 8.13 (d, *J* = 7.80 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 21.4, 22.9, 28.9, 30.3, 55.4, 93.9, 114.2, 121.9, 123.2, 123.4, 126.3, 126.4, 126.7, 127.4, 127.7, 129.3, 130.4, 130.9, 134.0, 137.3, 137.8, 139.6, 151.8, 159.8; IR (cm^{-1}): 2957, 2929, 2868, 1612,

1527, 1510, 1482, 1460, 1438, 1248, 1173, 1106, 1034; *m/z* (ESI): 421 (M^++H); HRMS calcd. for $C_{29}H_{29}N_2O$ (M+H) 421.2280, found 421.2296.



7g. 5-butyl-2-cyclopropyl-6-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline

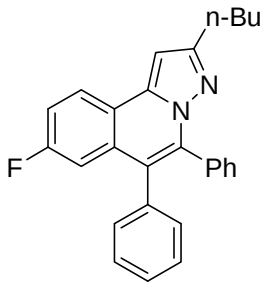
Yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.81 (t, *J* = 7.32 Hz, 3H), 0.92-0.95 (m, 2H), 1.04-1.09 (m, 2H), 1.28-1.30 (m, 2H), 1.67-1.71 (m, 2H), 2.17-2.22 (m, 1H), 2.95 (t, *J* = 7.80 Hz, 2H), 3.90 (s, 3H), 6.68 (s, 1H), 7.02-7.05 (m, 2H), 7.17-7.23 (m, 3H), 7.41-7.48 (m, 2H), 8.02 (d, *J* = 7.80 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.10, 9.86, 13.9, 22.8, 28.9, 30.2, 55.4, 93.3, 113.9, 114.2, 122.9, 123.3, 126.1, 126.2, 127.3, 127.8, 129.3, 130.6, 132.2, 137.9, 156.6, 159.0; IR (cm^{-1}): 2956, 2927, 2852, 1607, 1510, 1456, 1342, 1276, 1245, 1173, 1106, 1040; *m/z* (ESI): 371 (M^++H); HRMS calcd. for $C_{25}H_{27}N_2O$ (M+H) 371.2123, found 371.2135.



7h.2-cyclopropyl-8-fluoro-6-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

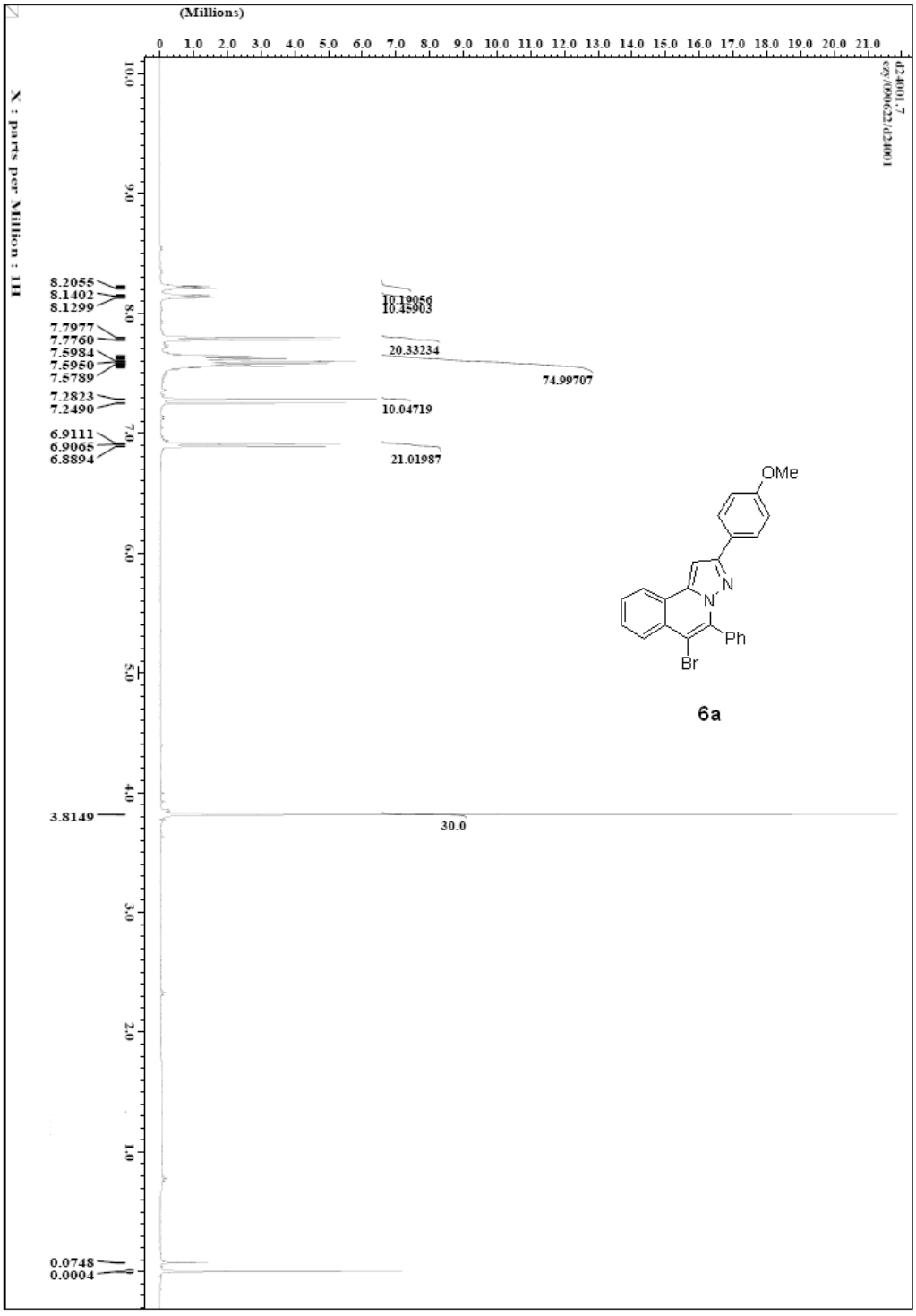
Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.79-0.82 (m, 2H), 0.98-1.02 (m, 2H), 2.09-2.14 (m, 1H), 3.79 (s, 3H), 6.60 (s, 1H), 6.77-6.82 (m, 2H), 7.02-7.07 (m, 3H), 7.21-7.35 (m, 6H), 8.04-8.07 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.29, 9.95, 55.2, 92.4, 111.8 (d, $^2J_{CF}$ = 22.9 Hz), 113.7, 115.7 (d, $^2J_{CF}$ = 23.8 Hz), 120.3, 121.8, 125.7, 127.8, 128.1, 128.3, 131.1, 132.4, 132.7, 133.2, 137.3, 139.1, 157.8, 158.7, 161.9 (d, $^1J_{CF}$ = 245.0 Hz); IR (cm^{-1}): 2924, 2852, 1608, 1512, 1500, 1449, 1403, 1342, 1285,

1246, 1175, 1024; *m/z* (ESI): 409 (M^++H); HRMS calcd. for $C_{27}H_{22}FN_2O$ (M+H) 409.1716, found 409.1710.



7i. 2-butyl-8-fluoro-5,6-diphenylpyrazolo[5,1-a]isoquinoline

Yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.94 (t, J = 7.32 Hz, 3H), 1.37-1.45 (m, 2H), 1.67-1.75 (m, 2H), 2.79 (t, J = 7.80 Hz, 2H), 6.86 (s, 1H), 6.97-7.08 (m, 1H), 7.15-7.20 (m, 2H), 7.24-7.31 (m, 9H), 8.09-8.12 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.7, 28.5, 32.0, 95.9, 111.7 (d, $^2J_{CF}$ = 22.9 Hz), 115.7 (d, $^2J_{CF}$ = 23.8 Hz), 120.5, 122.3, 125.7, 127.3, 127.8, 128.2, 128.4, 131.1, 131.7, 132.0, 133.0, 136.1, 137.3, 138.9, 155.9, 161.9 (d, $^1J_{CF}$ = 245.0 Hz); IR (cm^{-1}): 3062, 2955, 2921, 2847, 1620, 1540, 1486, 1464, 1443, 1400, 1342, 1321, 1265, 1191; *m/z* (ESI): 395 (M^++H); HRMS calcd. for $C_{27}H_{24}FN_2$ (M+H) 395.1924, found 395.1945.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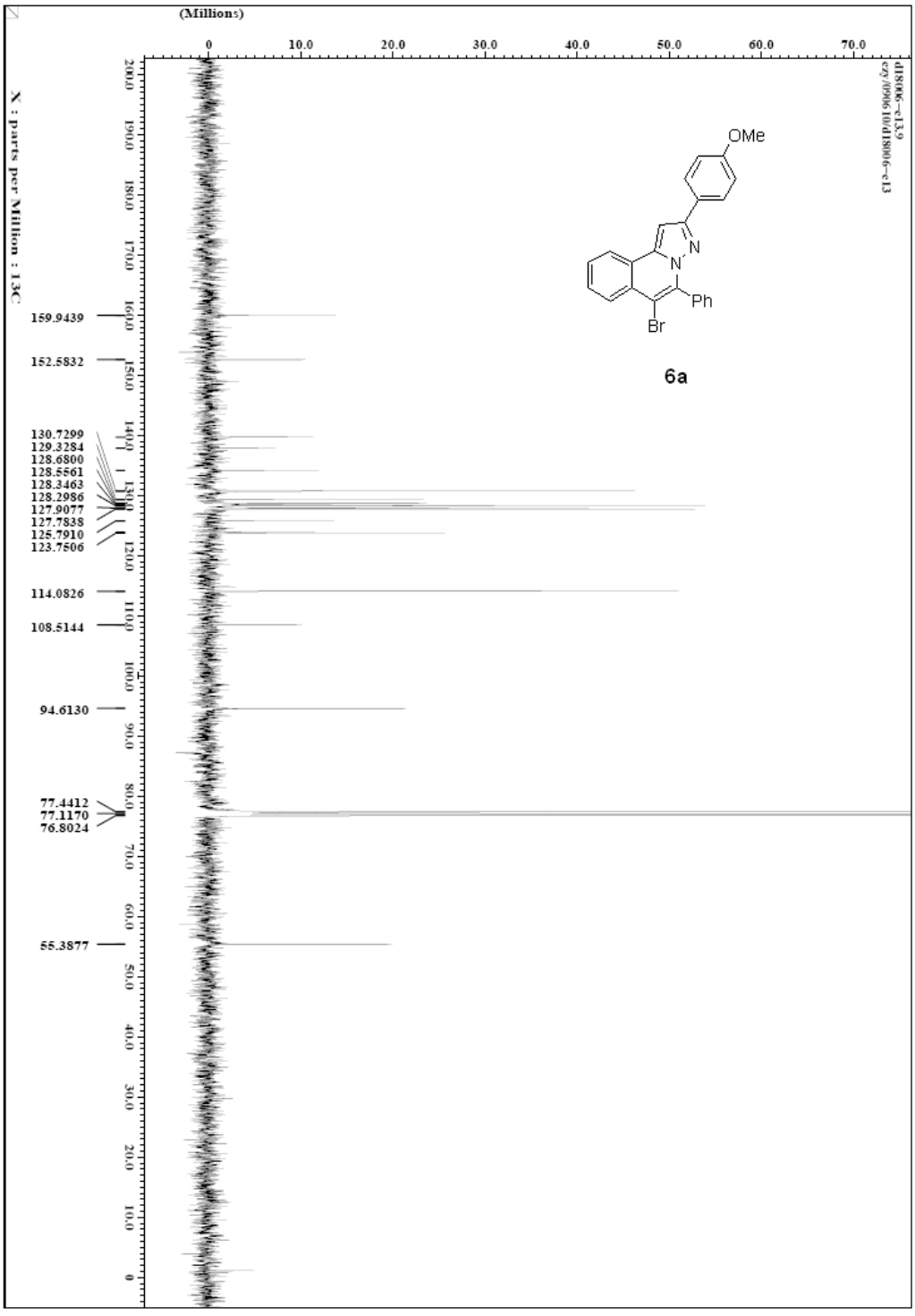
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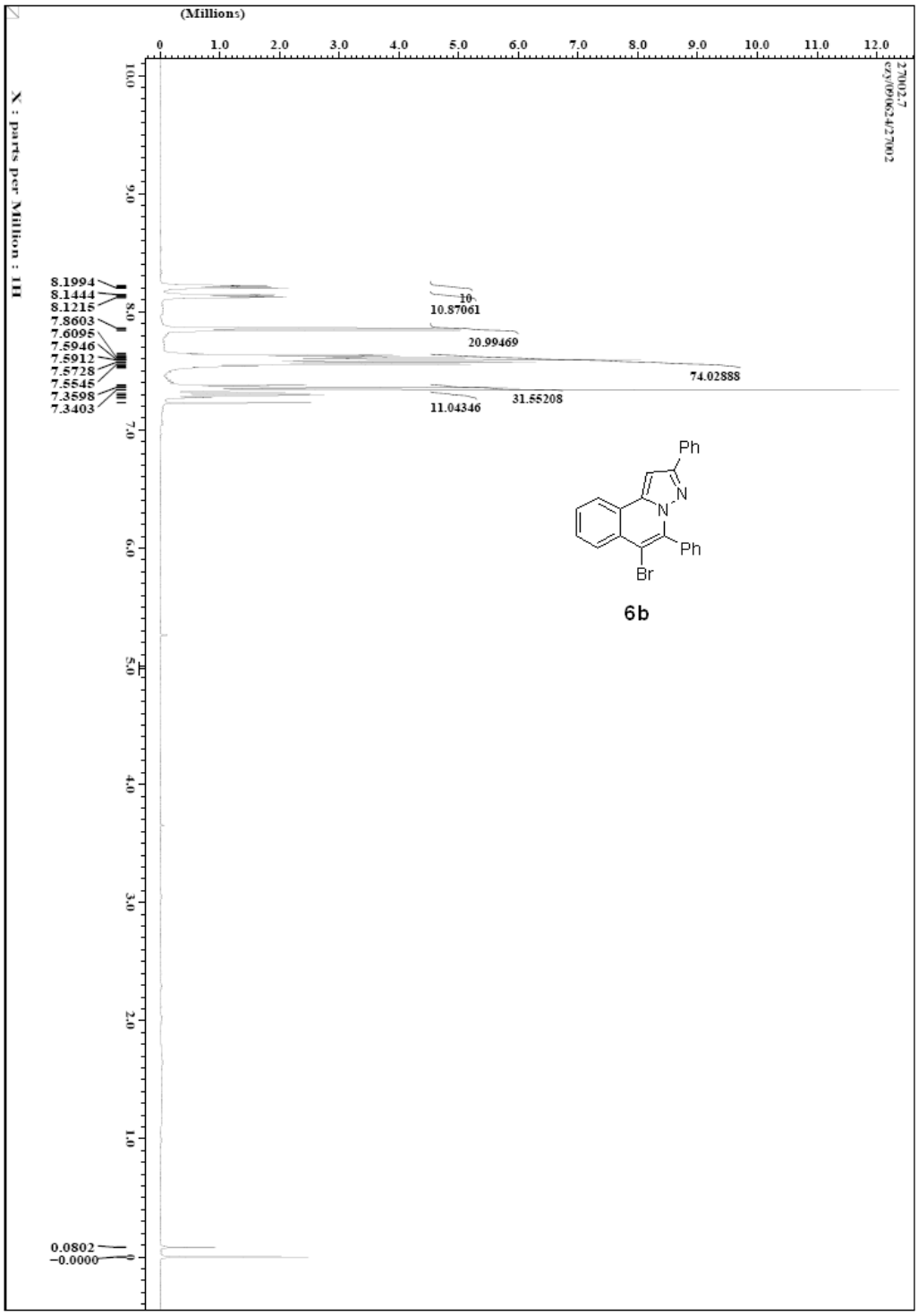
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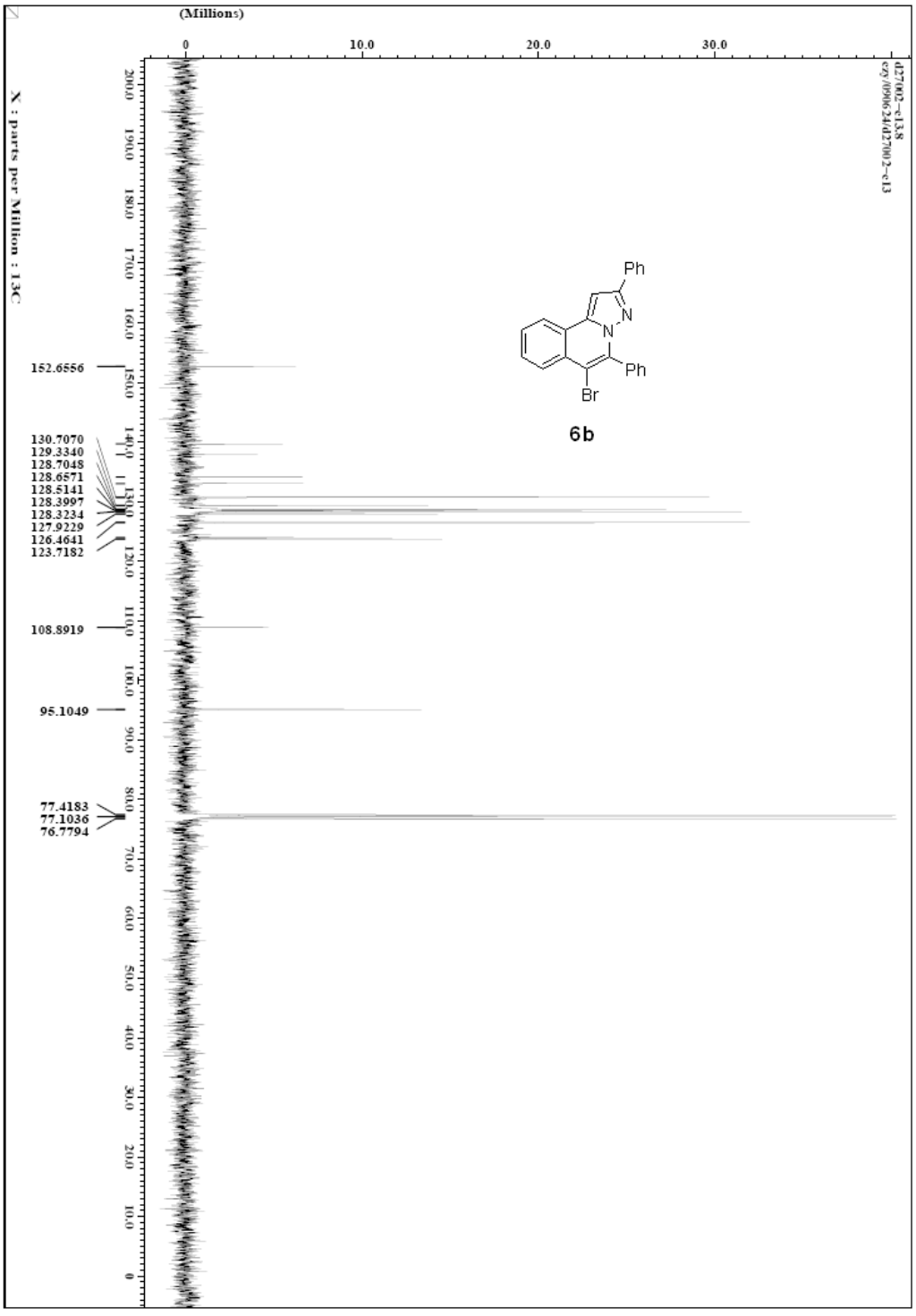
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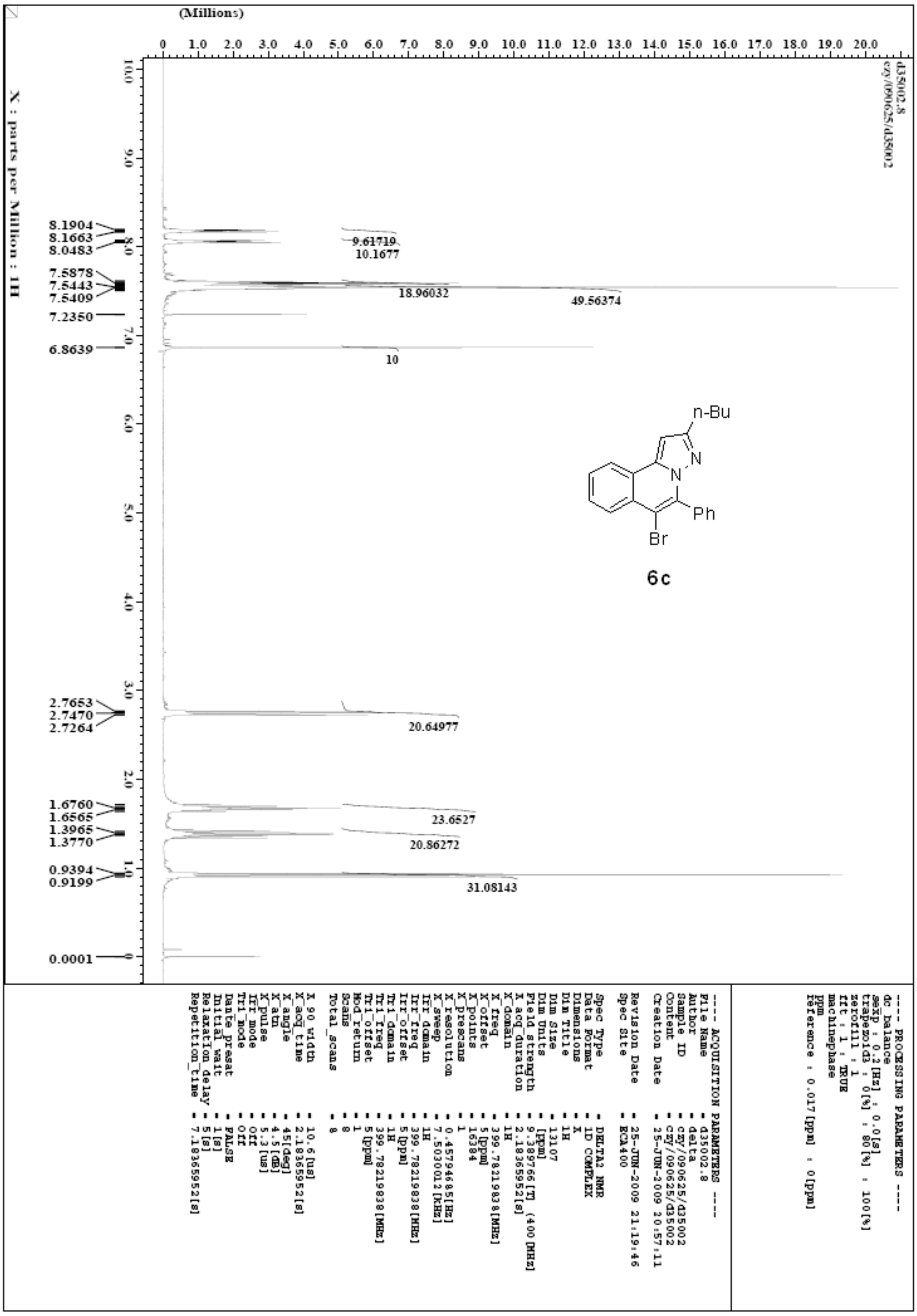
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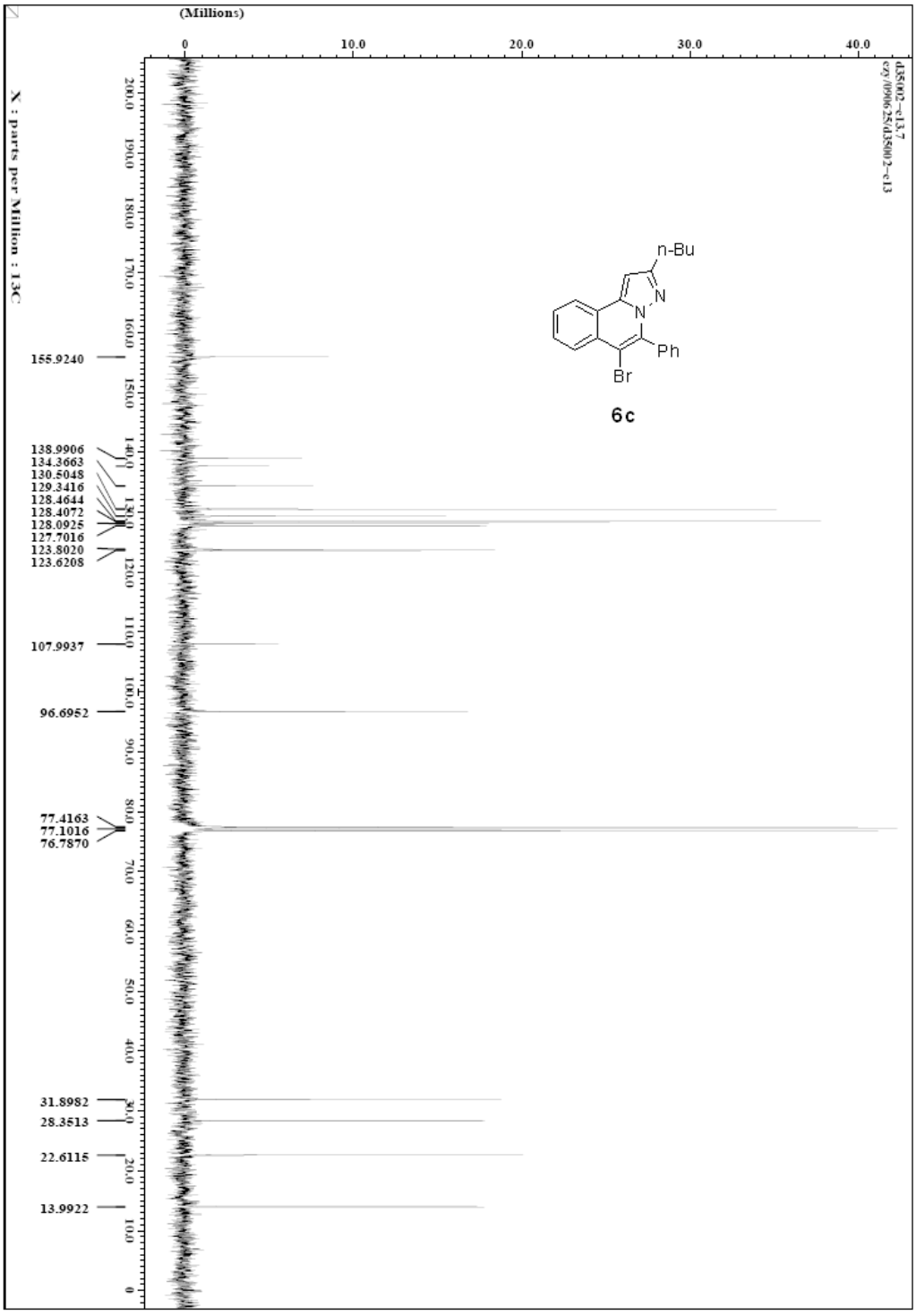
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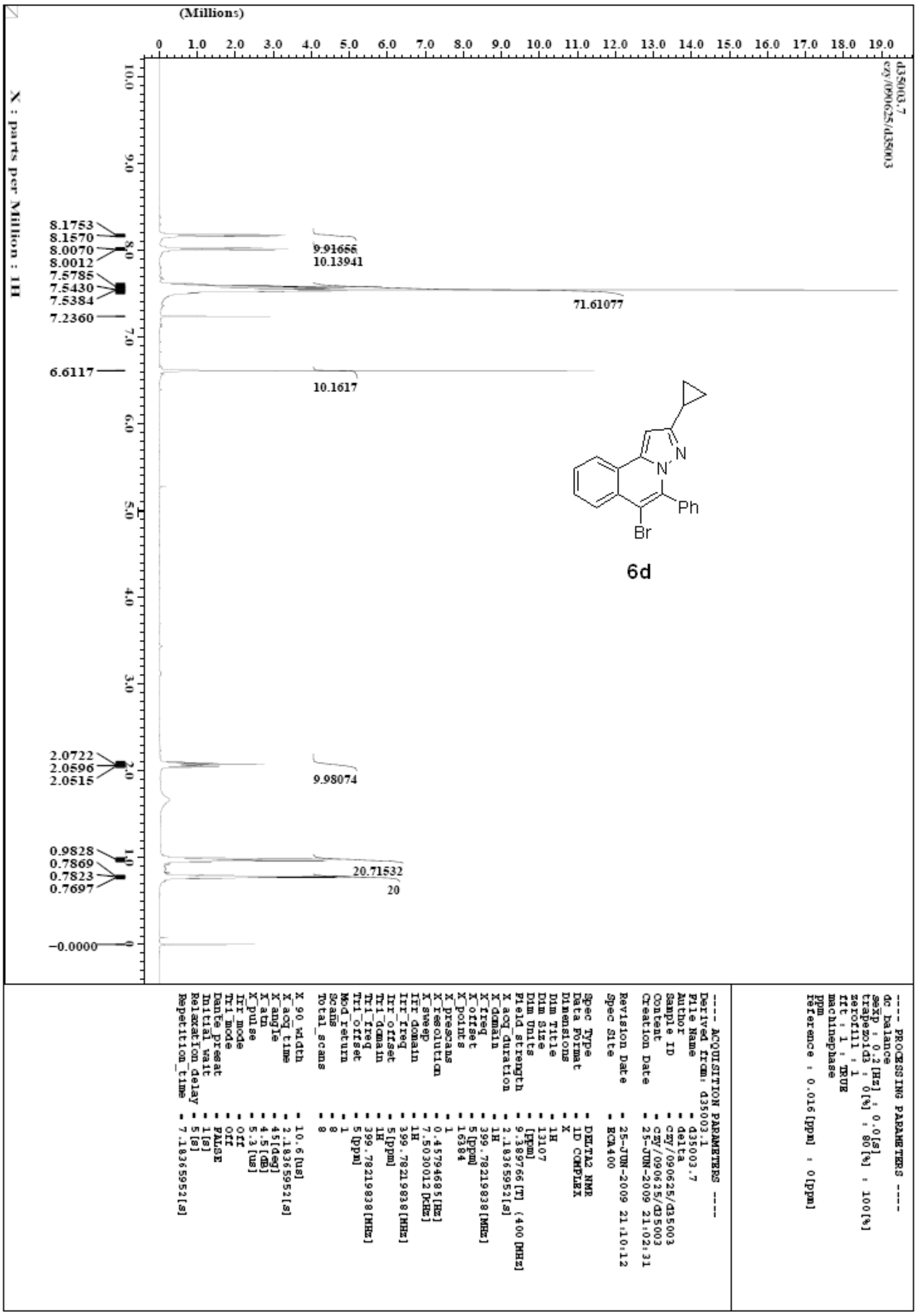

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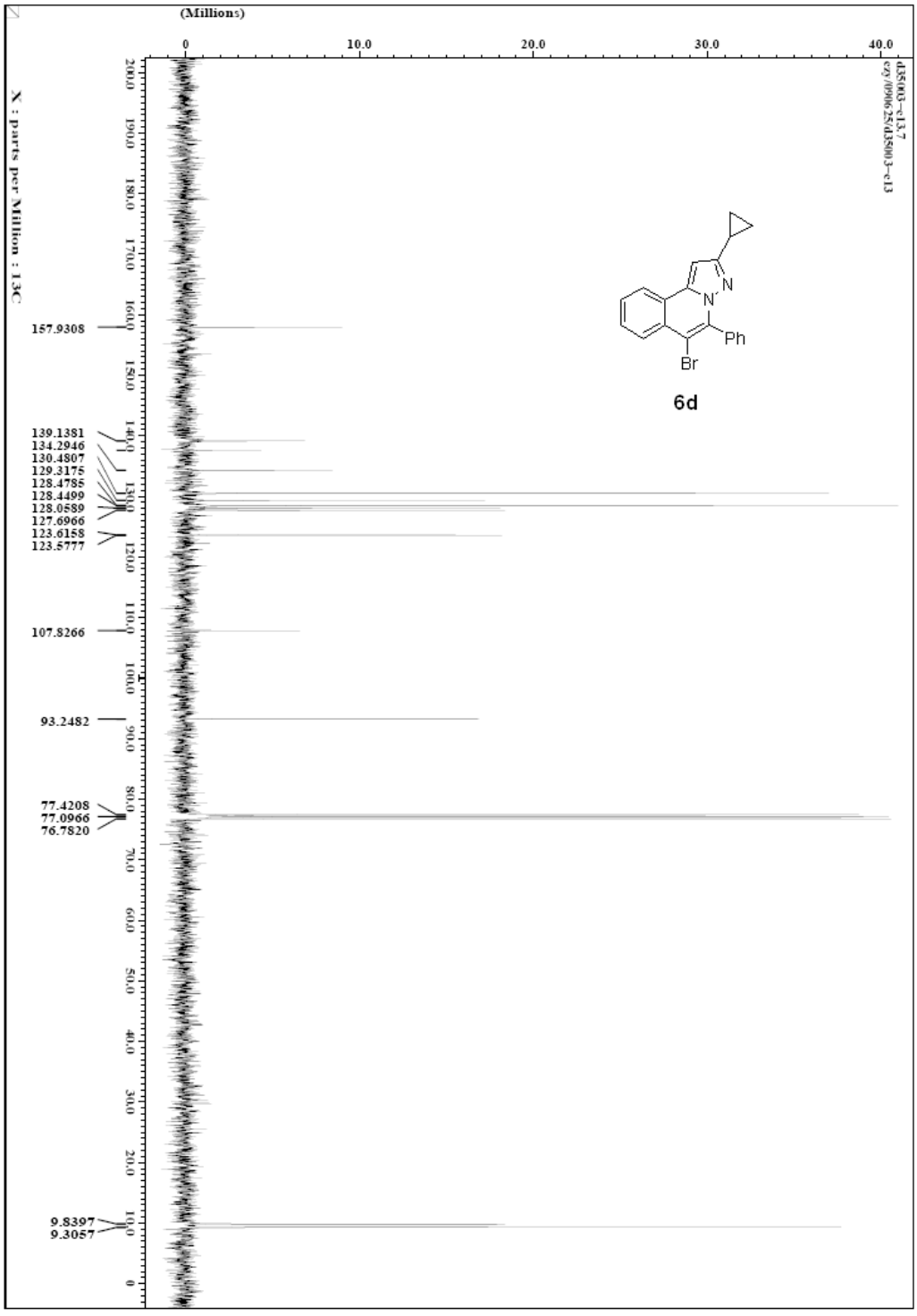
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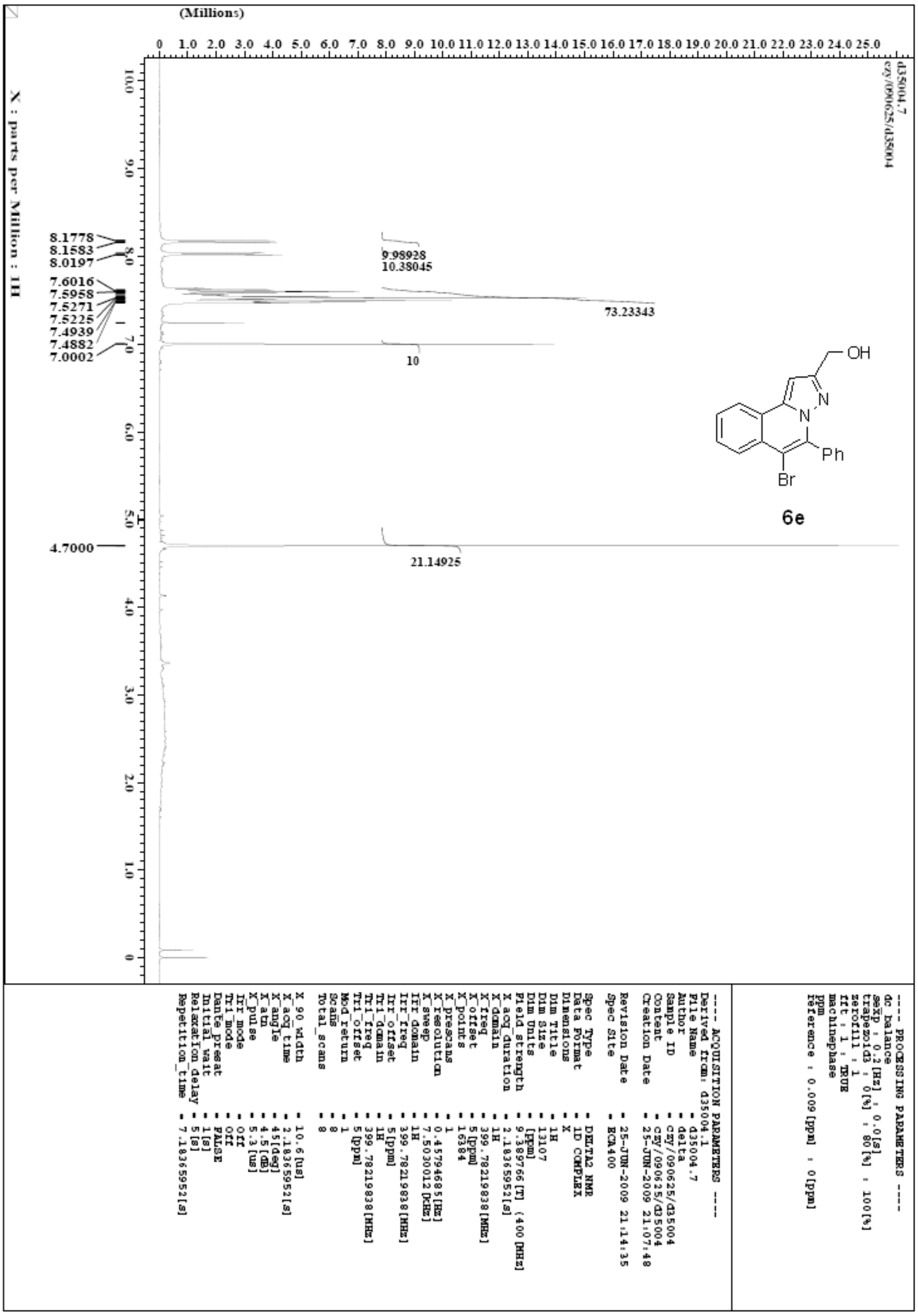

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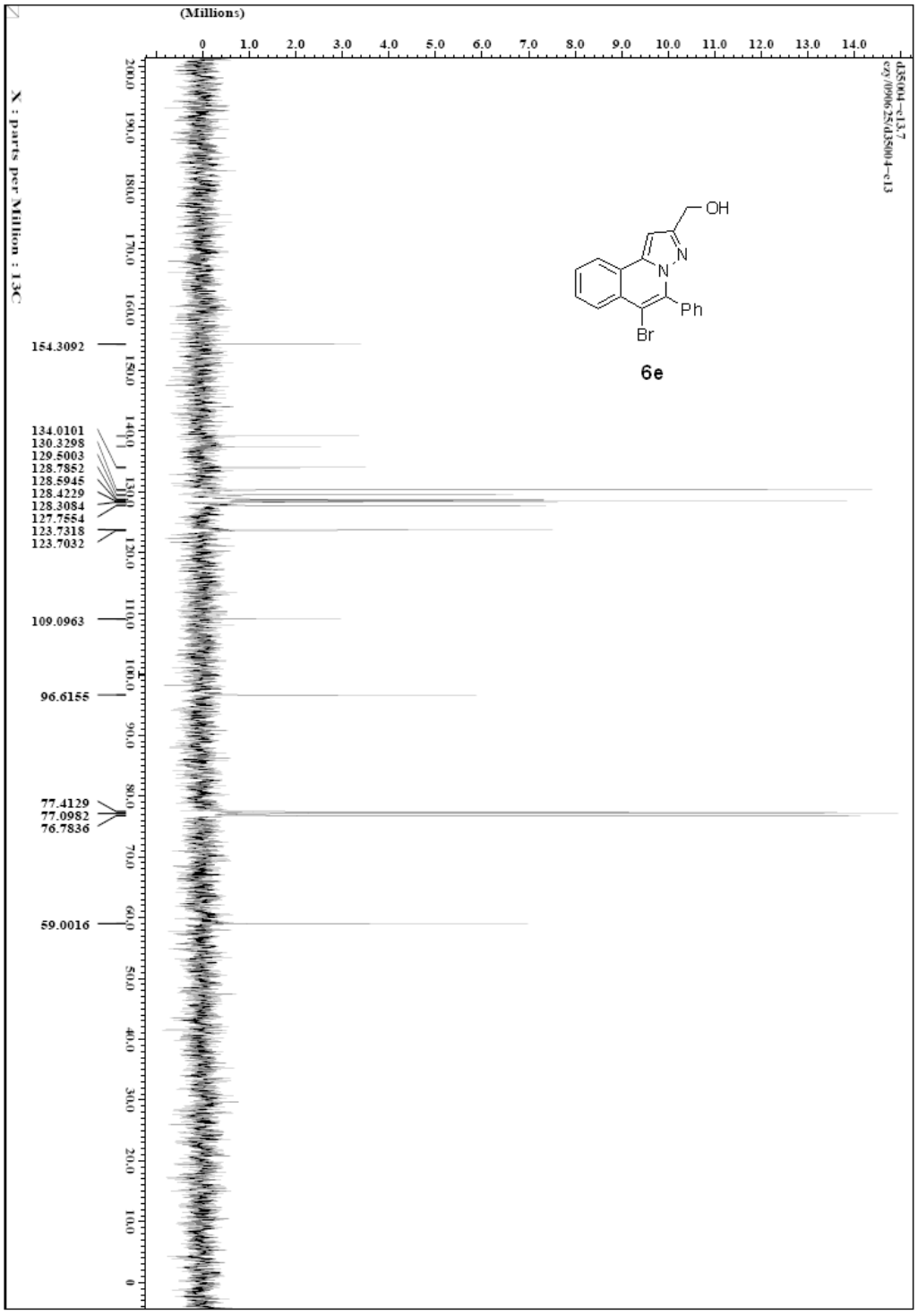
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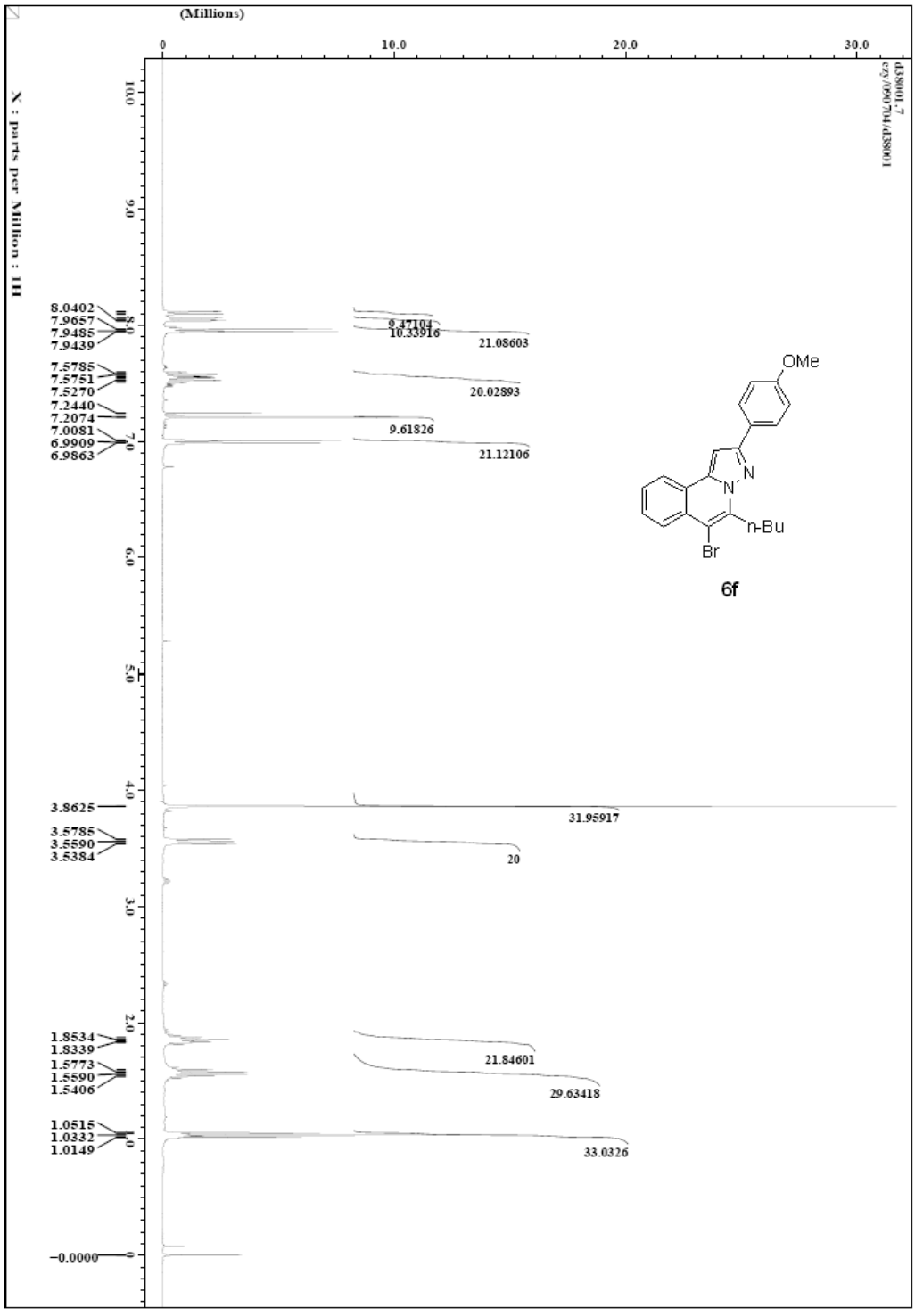
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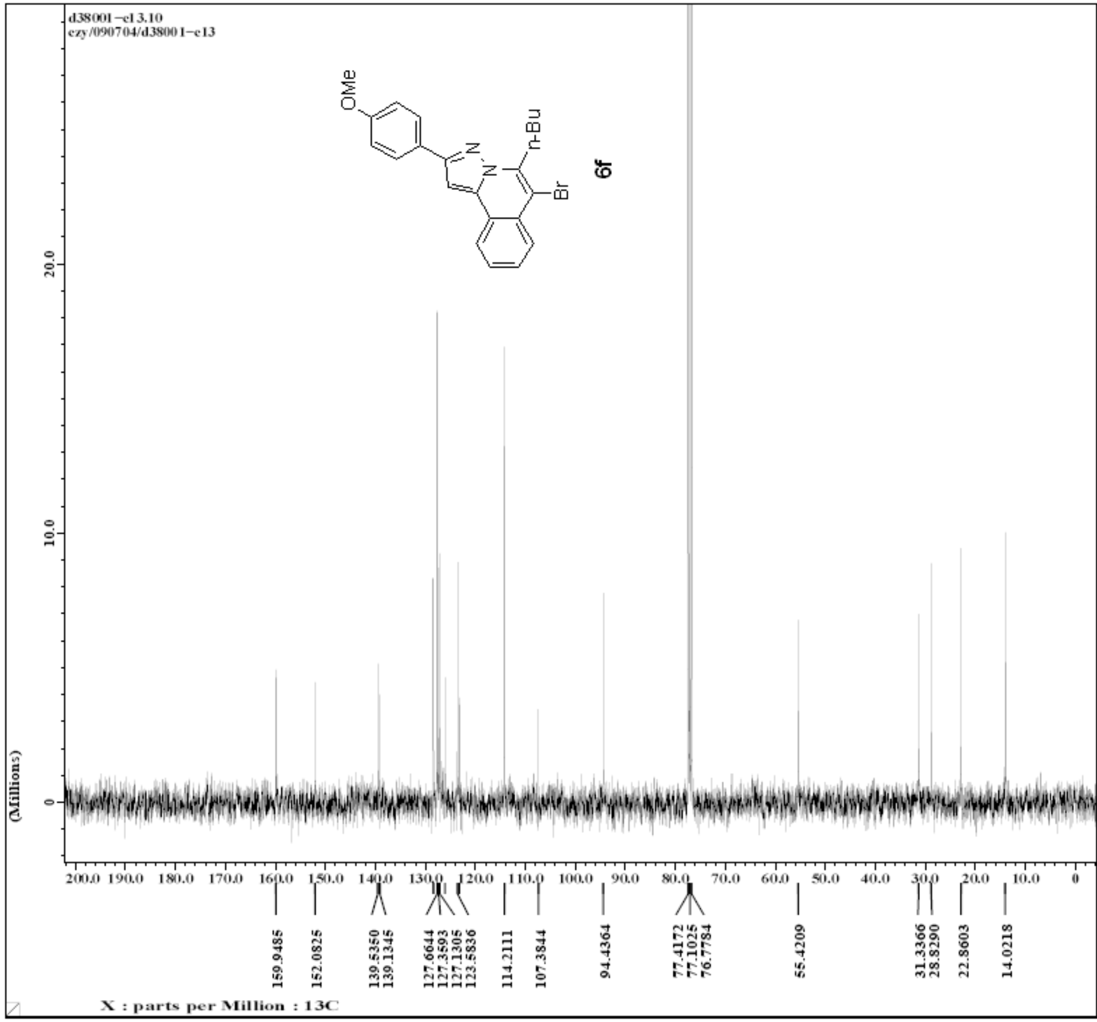


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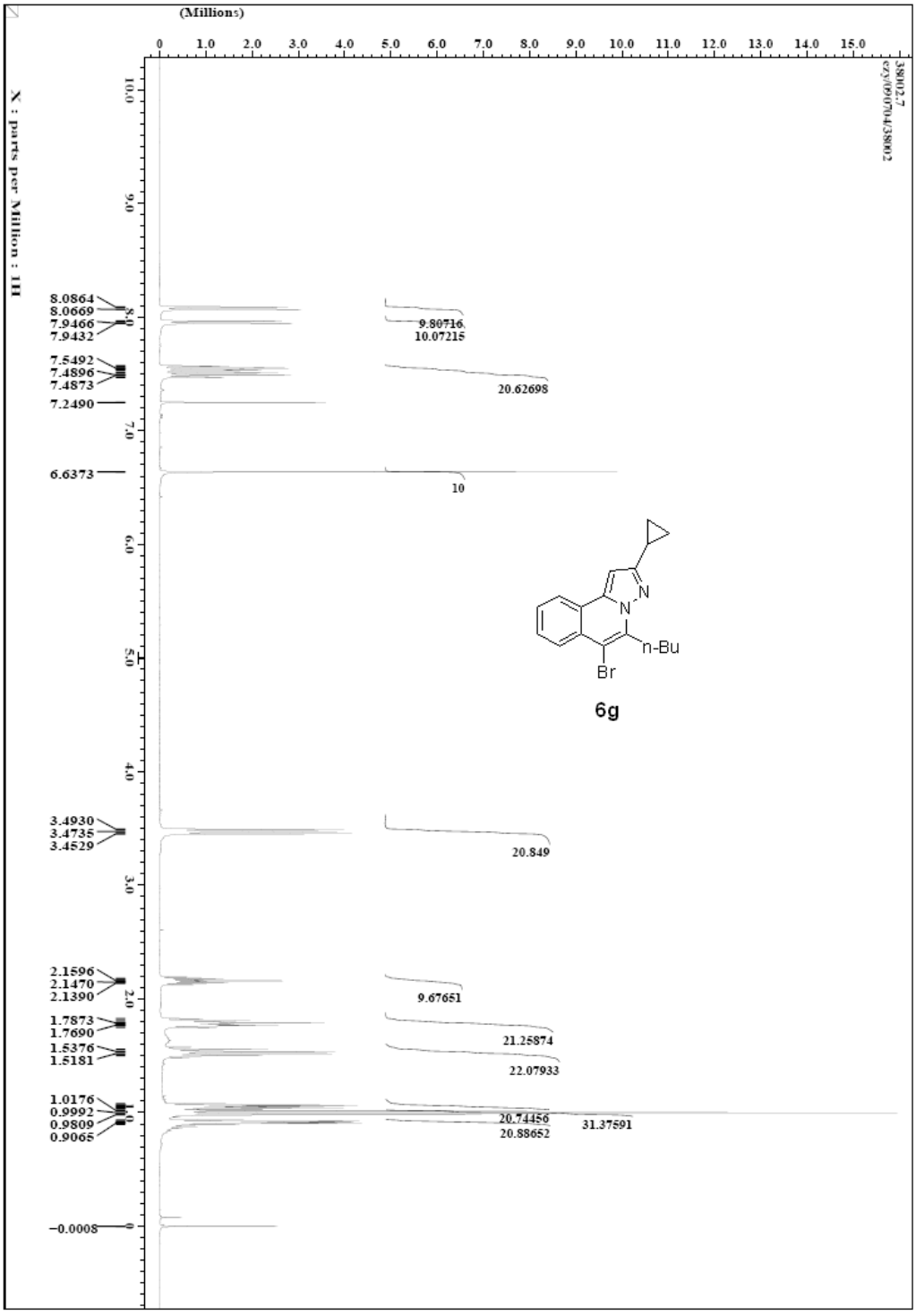
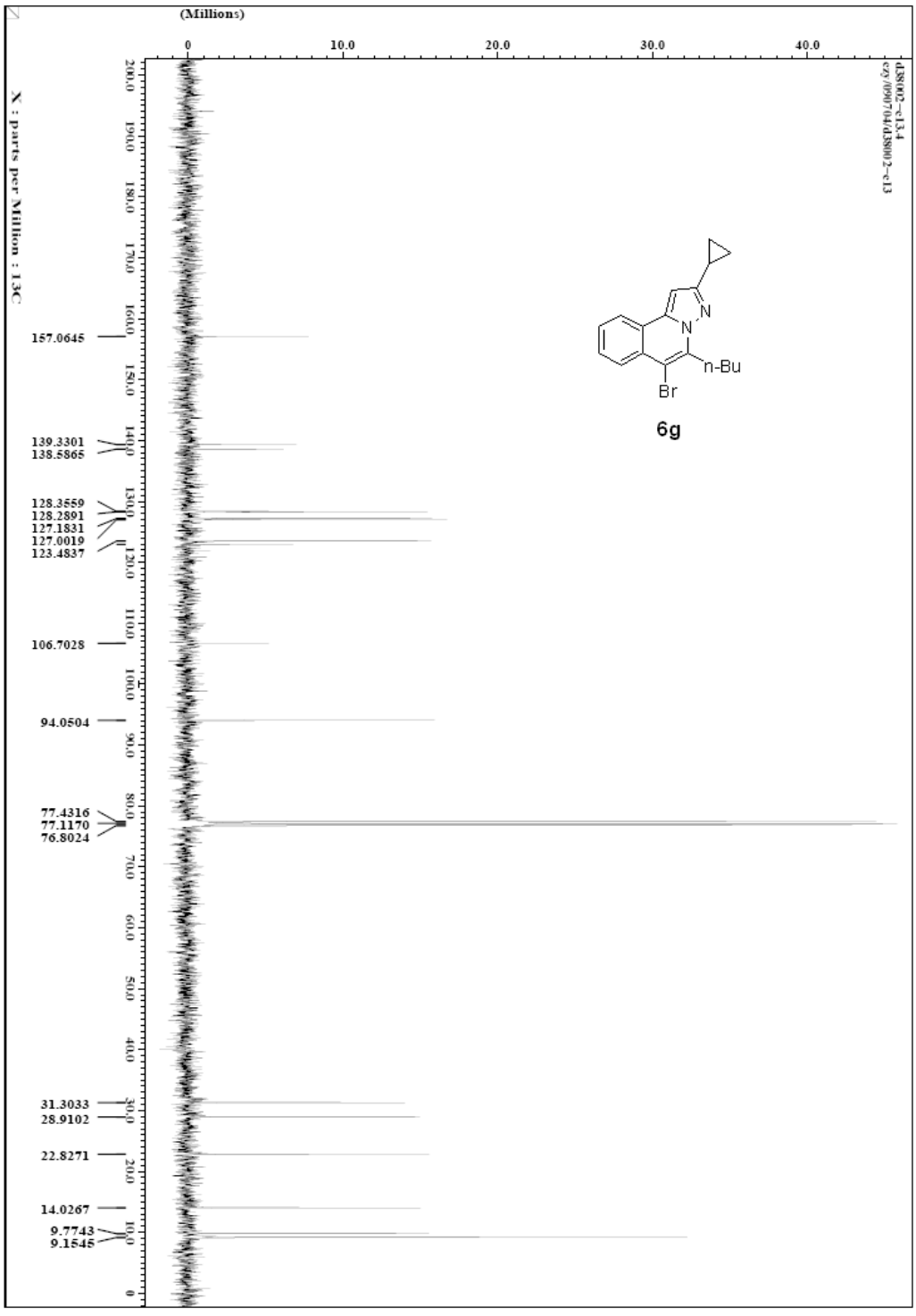
Creation Date      =  4-JUL-2009 15:15:30

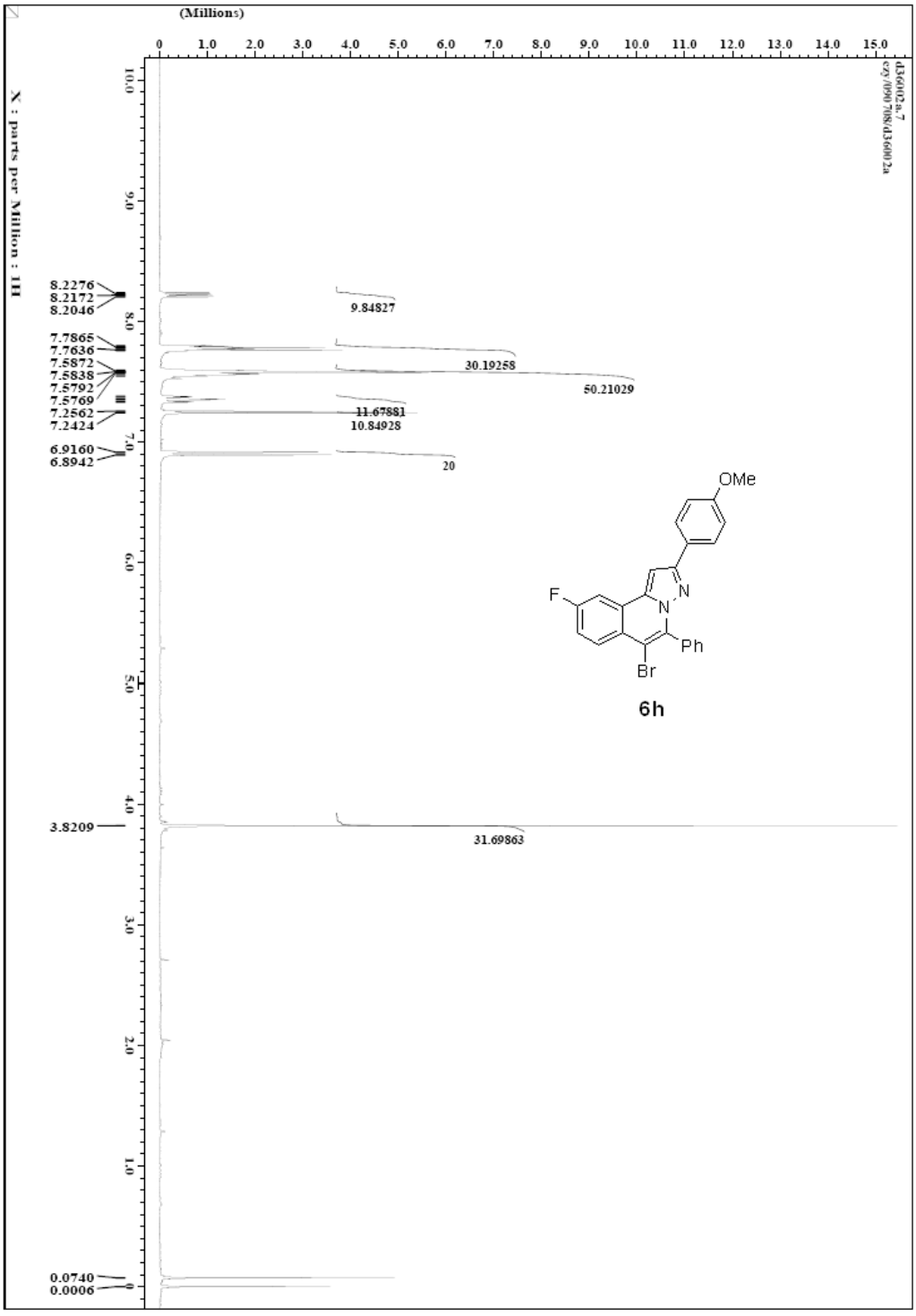
Revision Date      =  9-JUL-2009 14:34:06
Spec Site          = ECA400

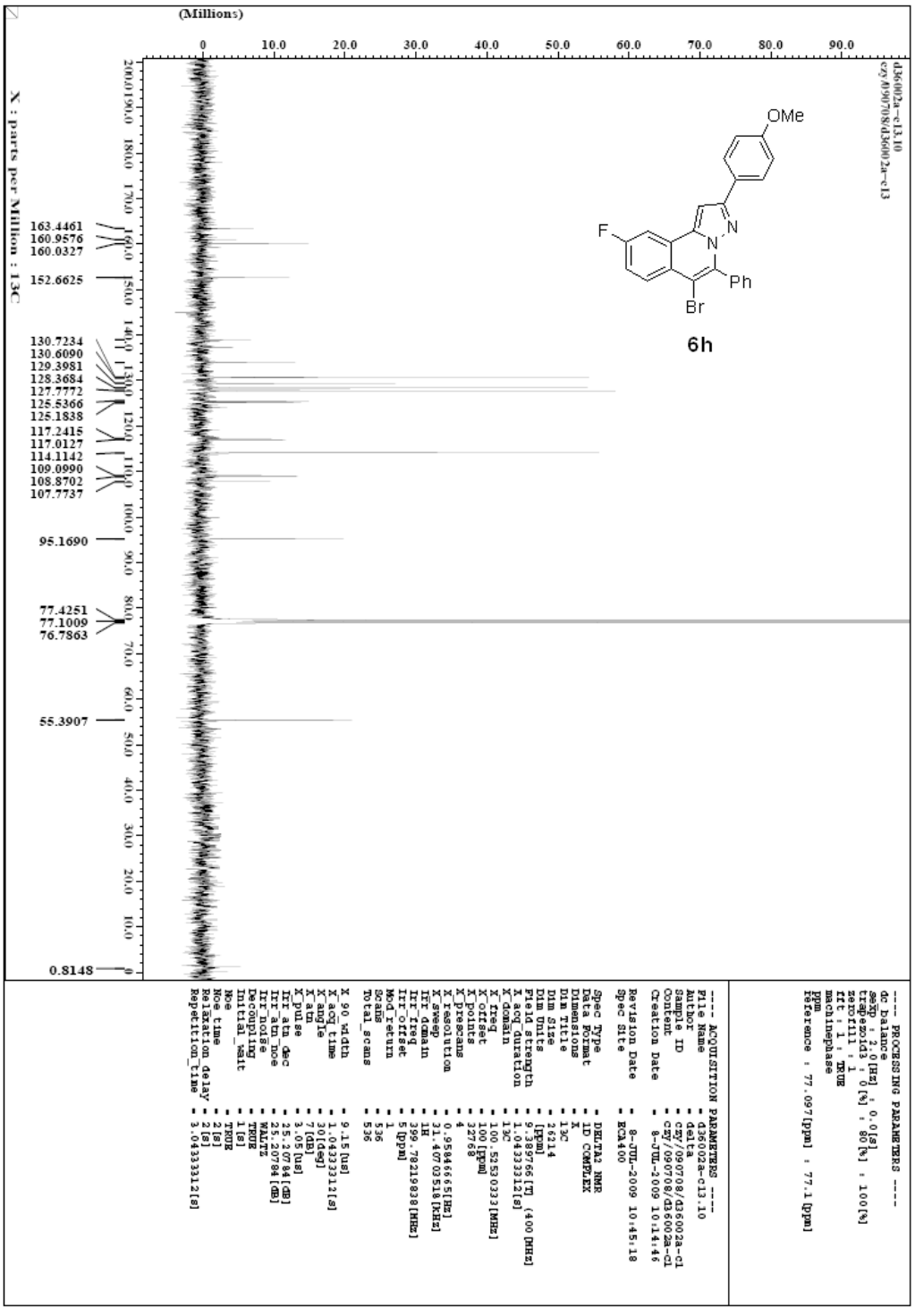
Spec Type          = DELTA2 NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 13C
Dim Size           = 26214
Dim Units          = [ppm]
Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Mod_return         = 1
Scans              = 54
Total_scans        = 54

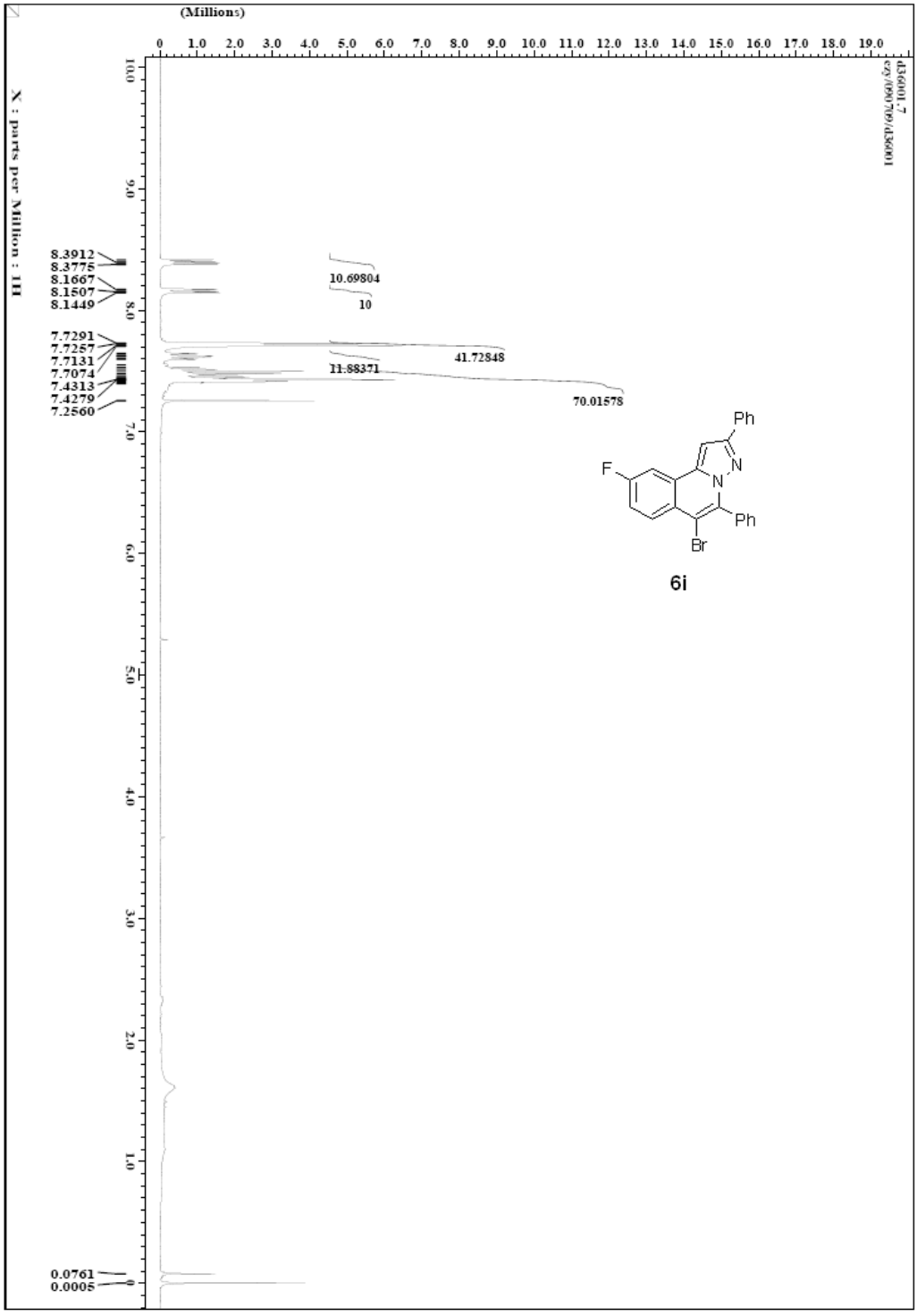
X_90_width         = 9.15[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 7[dB]
X_pulse            = 3.05[us]
Irr_atn_dec        = 25.20784[dB]
Irr_atn_noe        = 25.20784[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Relaxation_delay   = 2[s]
Repetition_time    = 3.04333312[s]
```

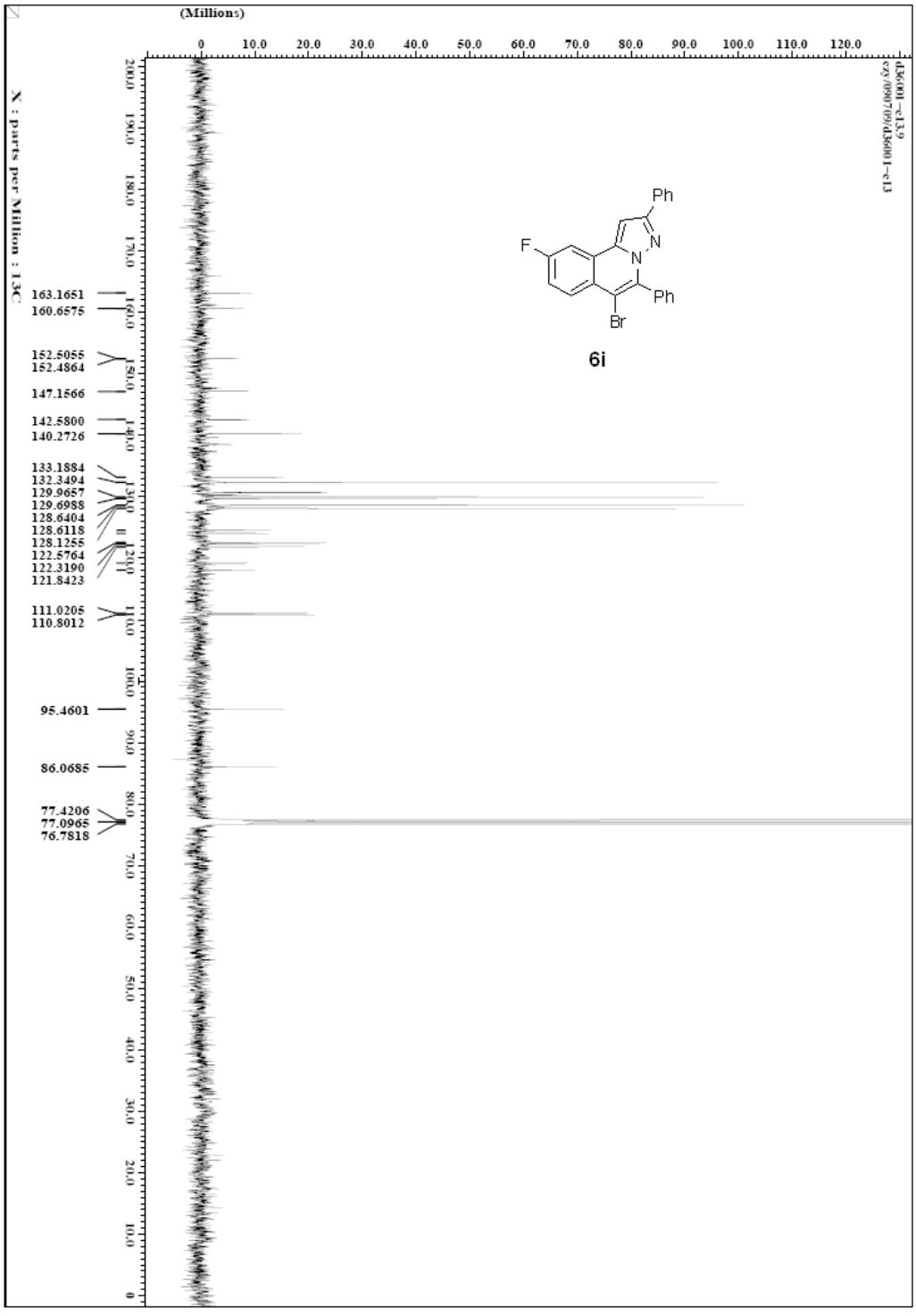

(Millions)
X : parts per Million : 1H
8.0864
8.0669
7.9466
7.9432
7.5492
7.4896
7.4873
7.2490
6.6373
3.4930
3.4735
3.4529
2.1596
2.1470
2.1390
1.7873
1.7690
1.5376
1.5181
1.0176
0.9992
0.9809
0.9065
−0.0008
9.80716
10.07215
20.62698
10
20.849
9.67651
21.25874
22.07933
20.74456
20.88652
31.37591
n-Bu
Br
6g

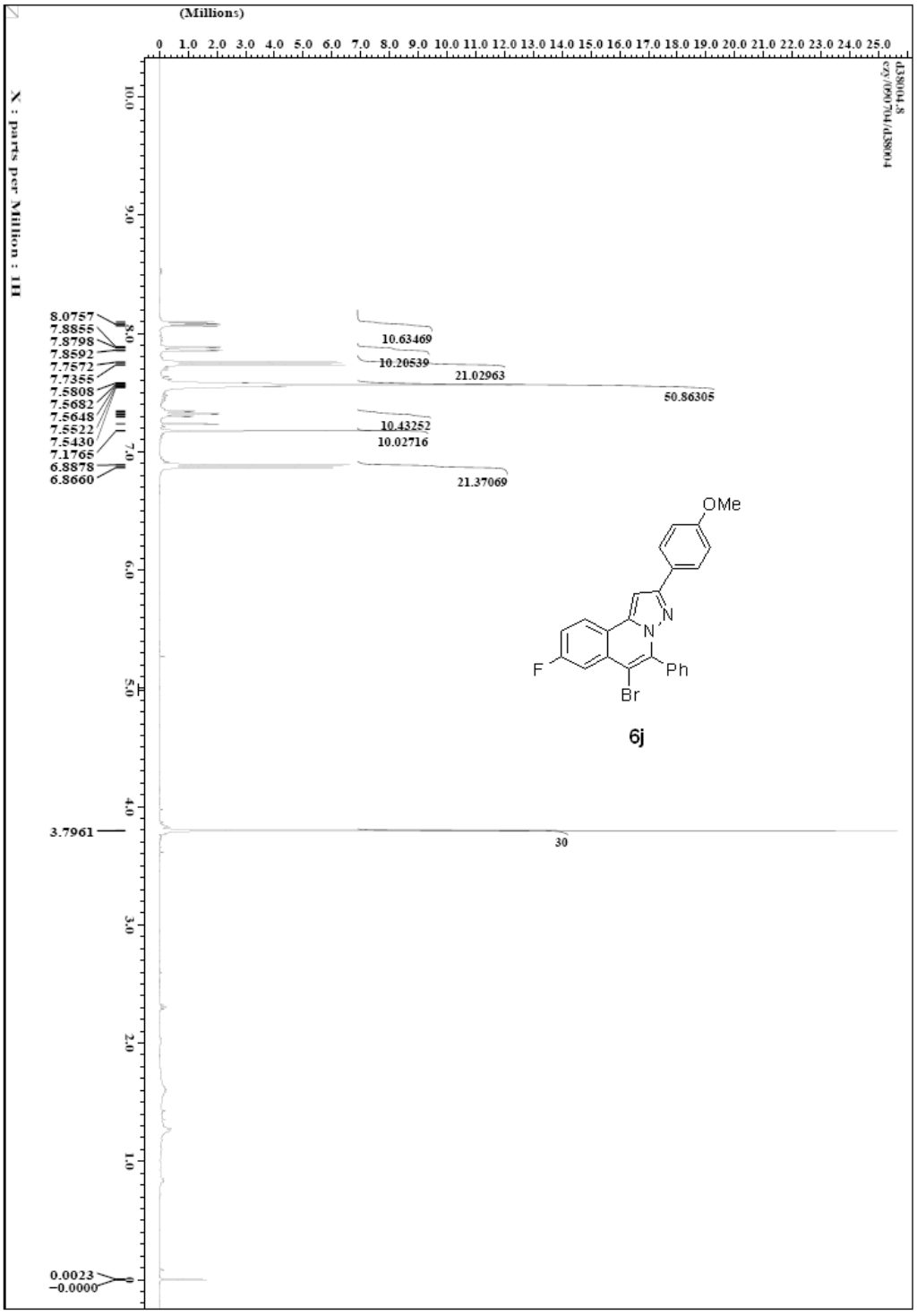

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 13C
d38002-c13-4
czy1090704d38002-c13
157.0645
139.3301
138.5865
128.3559
128.2891
127.1831
127.0019
123.4837
106.7028
94.0504
77.4316
77.1170
76.8024
31.3033
28.9102
22.8271
14.0267
9.7743
9.1545
N
N
n-Bu
Br
6g

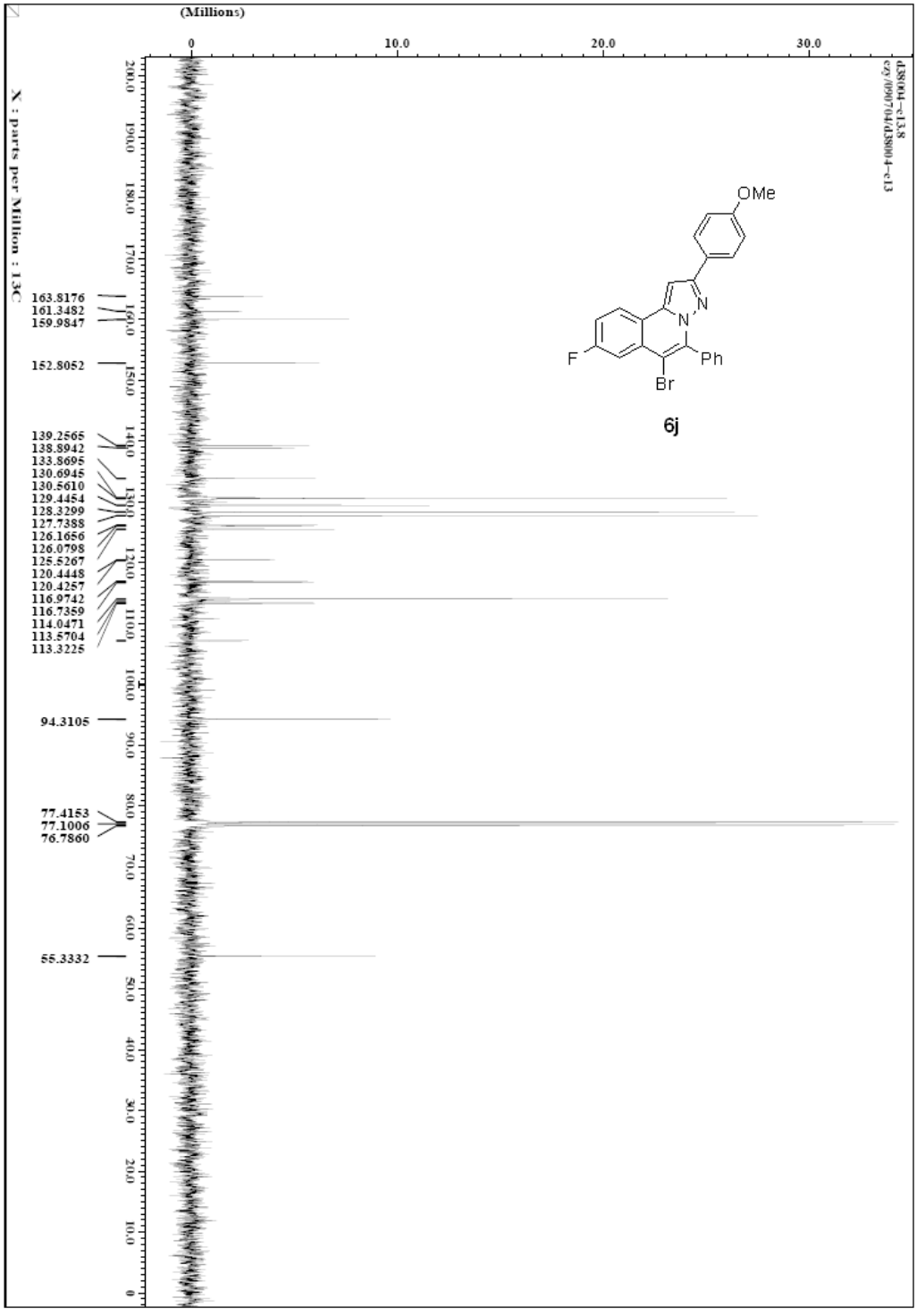

(Millions)
X : parts per Million : 1H
d3600 2a.7
czy/090708/d3600 2a
8.2276
8.2172
8.2046
7.7865
7.7636
7.5872
7.5838
7.5792
7.5769
7.2562
7.2424
6.9160
6.8942
3.8209
0.0740
0.0006
9.84827
30.19258
50.21029
11.67881
10.84928
20
31.69863
OMe
F
N
N
Ph
Br
6h

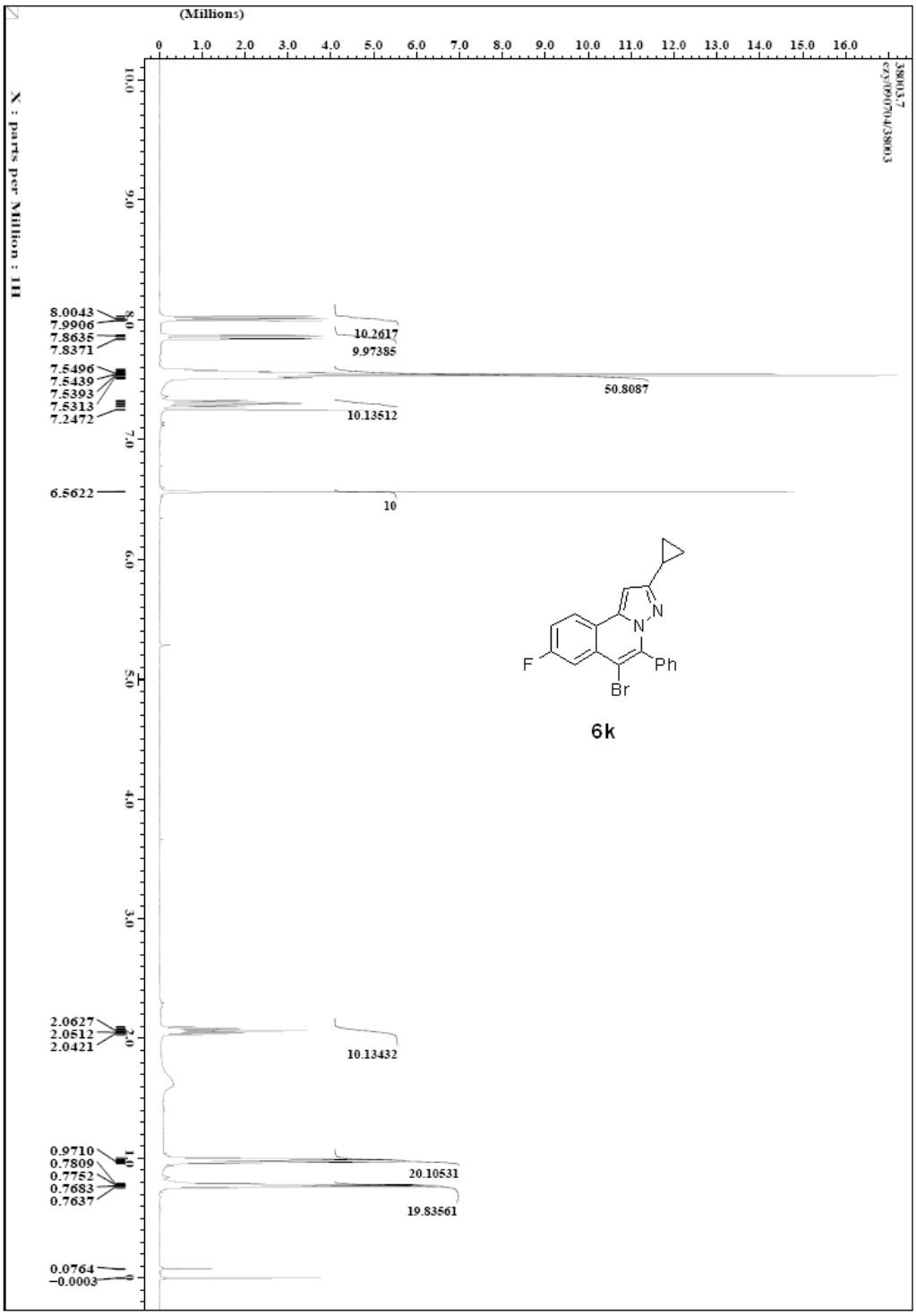

(Millions)
X : parts per Million : 13C
d360602a-c13.10
czy/090708/d360602a-c13
163.4461
160.9576
160.0327
152.6625
130.7234
130.6090
129.3981
128.3684
127.7772
125.5366
125.1838
117.2415
117.0127
114.1142
109.0990
108.8702
107.7737
95.1690
77.4251
77.1009
76.7863
55.3907
0.8148
OMe
F
N
N
Ph
Br
6h
---- PROCESSING PARAMETERS ----
dc_balance : 2.0[Hz] , 0.0[%]
exp_weight : 0[%] , 80[%] , 100[%]
line_broad : 1
zero_fill : 1
fft : 1
machinephase
ppm
Reference : 77.097[ppm] , 77.1[ppm]
---- ACQUISITION PARAMETERS ----
File Name = d360602a-c13.10
Author = delta
Sample ID = czy/090708/d360602a-c1
Content = czy/090708/d360602a-c1
Creation Date = 8-JUL-2009 10:14:46
Revision Date = 8-JUL-2009 10:45:18
Spec Site = ECA400
Spec Type = DELTA2 NMR
Data Format = 1D COMPLEX
Dim Title = 13C
Dim Size = 24214
Dim Units = [ppm]
Field_strength = 9.389766[T] (400 [MHz]
X_acq_duration = 1.043333312[s]
X_domain = 13C
X_freq = 100.5530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.958466651[Hz]
X_sweep = 31.407351B[kHz]
Irr_domain = 1H
Irr_freq = 399.7821903B[MHz]
Irr_offset = 5[ppm]
Mod_return = 1
Scans = 536
Total_scans = 536
X_90_width = 9.15[us]
X_acq_time = 1.043333312[s]
X_angle = 30[deg]
X_atn = 7[dB]
X_pulse = 3.05[us]
Irr_atn_dec = 25.20784[dB]
Irr_atn_mode = WALTZ
Irr_noise = TPPM
Decoupling = 1[s]
Initial_wait = TPPM
Mode = 2[s]
Mode time = 2[s]
Relaxation delay = 3.043333312[s]
Repetition_time

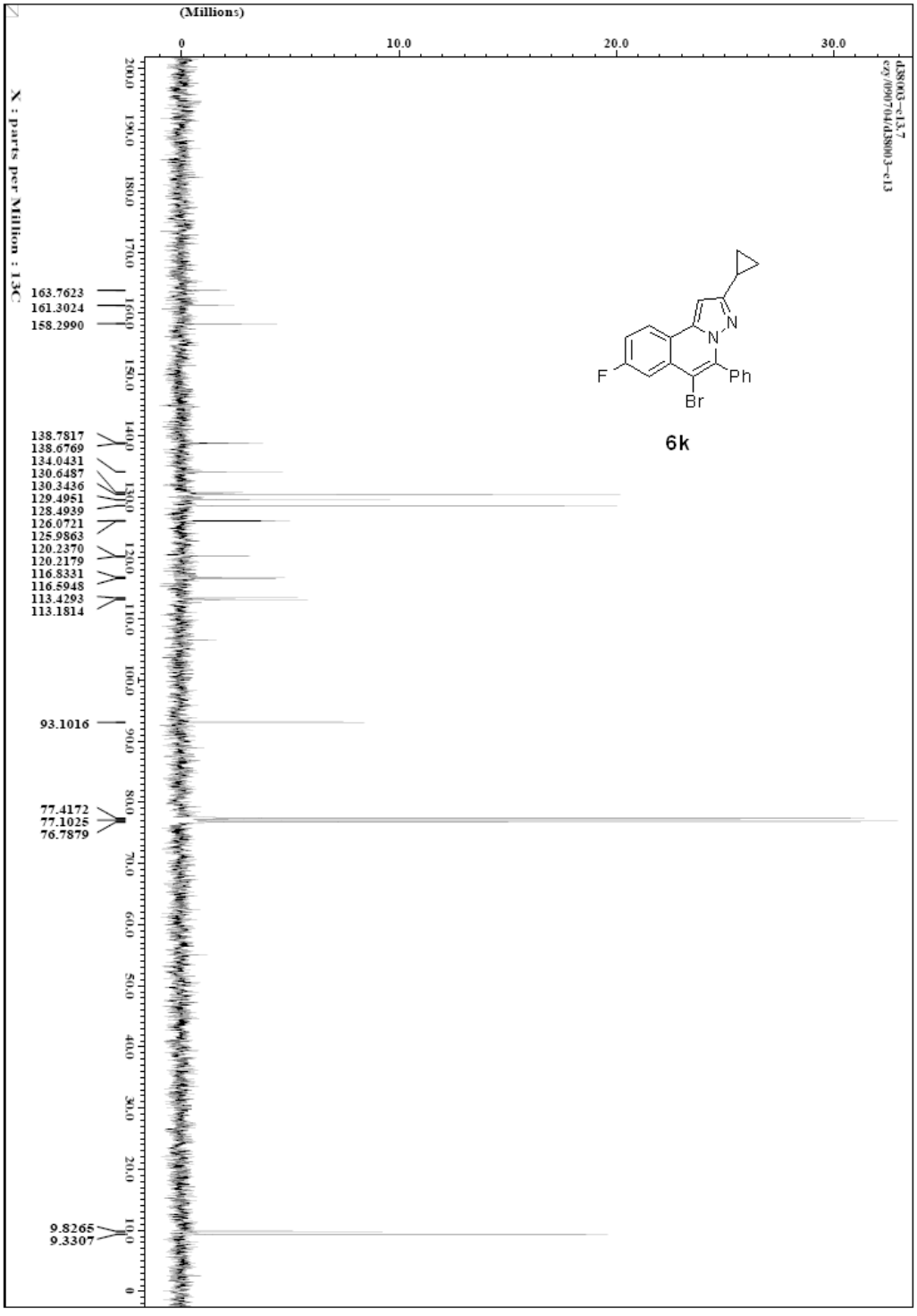

(Millions)
X : parts per Million : 1H
8.3912
8.3775
8.1667
8.1507
8.1449
7.7291
7.7257
7.7131
7.7074
7.4313
7.4279
7.2560
0.0761
0.0005
10.69804
10
41.72848
11.88371
70.01578
Ph
F
N
N
Ph
Br
6i

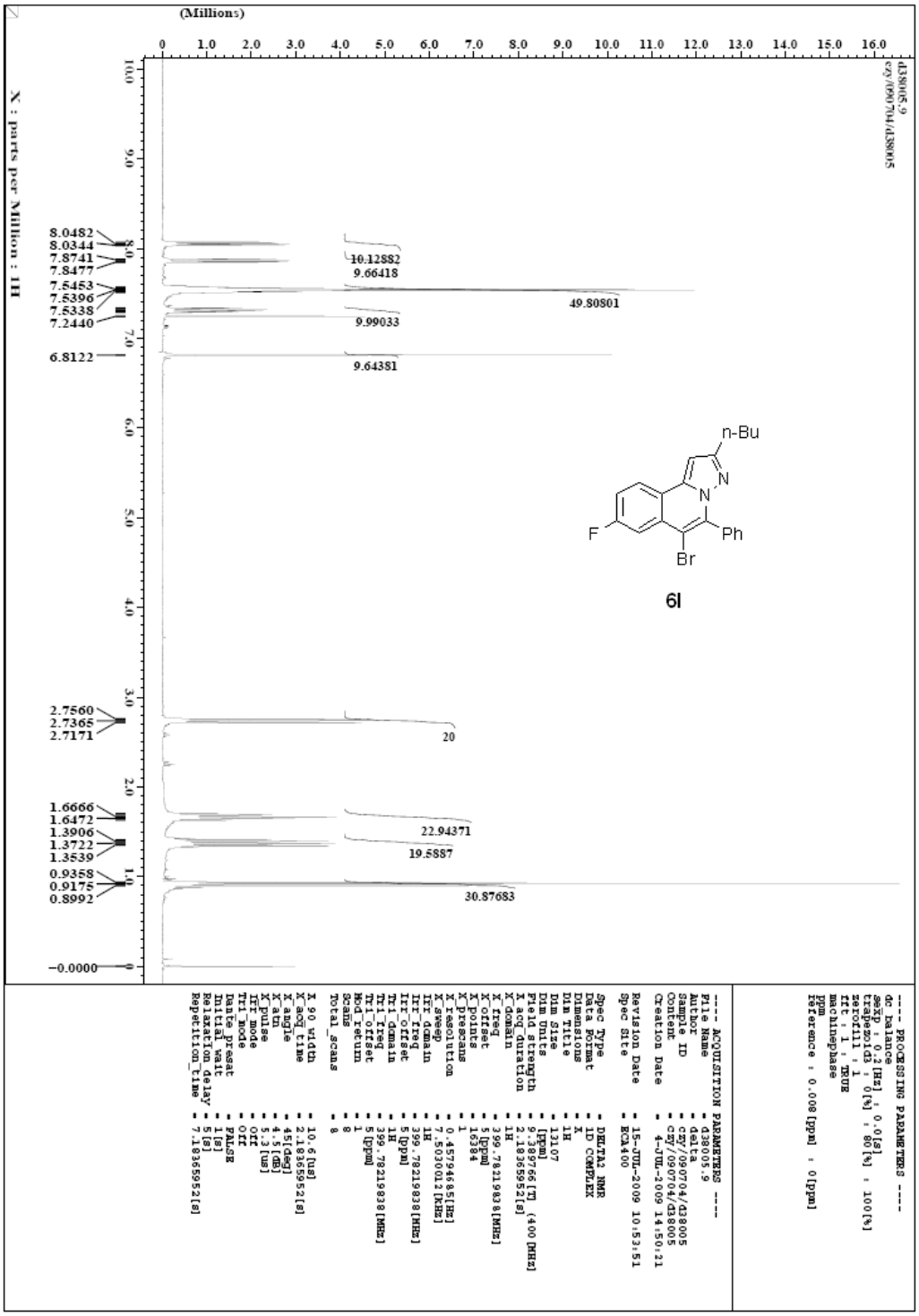

(Millions)
X : parts per Million : 13C
163.1651
160.6575
152.5055
152.4864
147.1566
142.5800
140.2726
133.1884
132.3494
129.9657
129.6988
128.6404
128.6118
128.1255
122.5764
122.3190
121.8423
111.0205
110.8012
95.4601
86.0685
77.4206
77.0965
76.7818
Ph
F
N
N
Ph
Br
6i

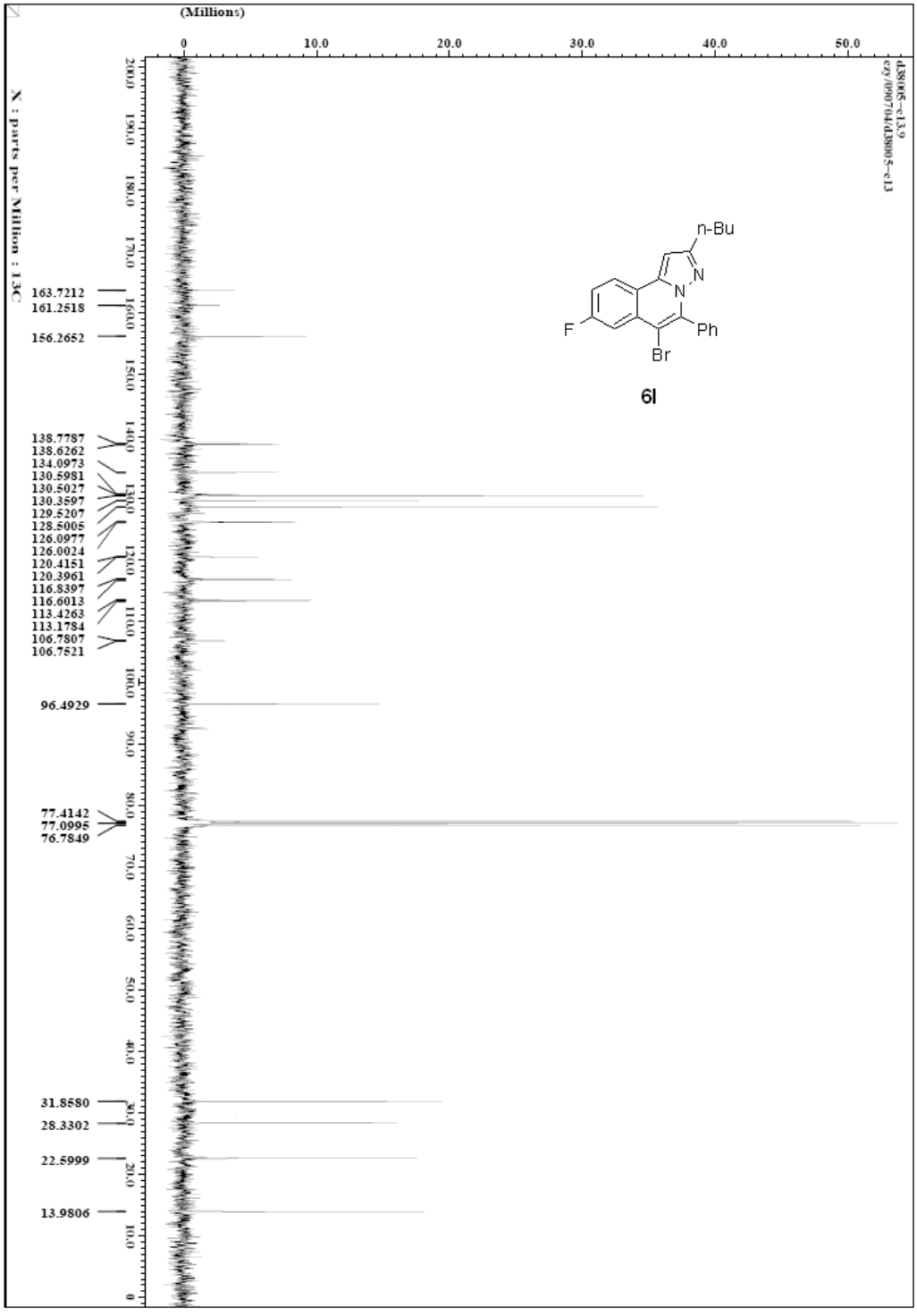

(Millions)
X : parts per Million : 1H
8.0757
7.8855
7.8798
7.8592
7.7572
7.7355
7.5808
7.5682
7.5648
7.5522
7.5430
7.1765
6.8878
6.8660
3.7961
0.0023
−0.0000
10.63469
10.20539
21.02963
50.86305
10.43252
10.02716
21.37069
30
OMe
N
N
F
Ph
Br
6j

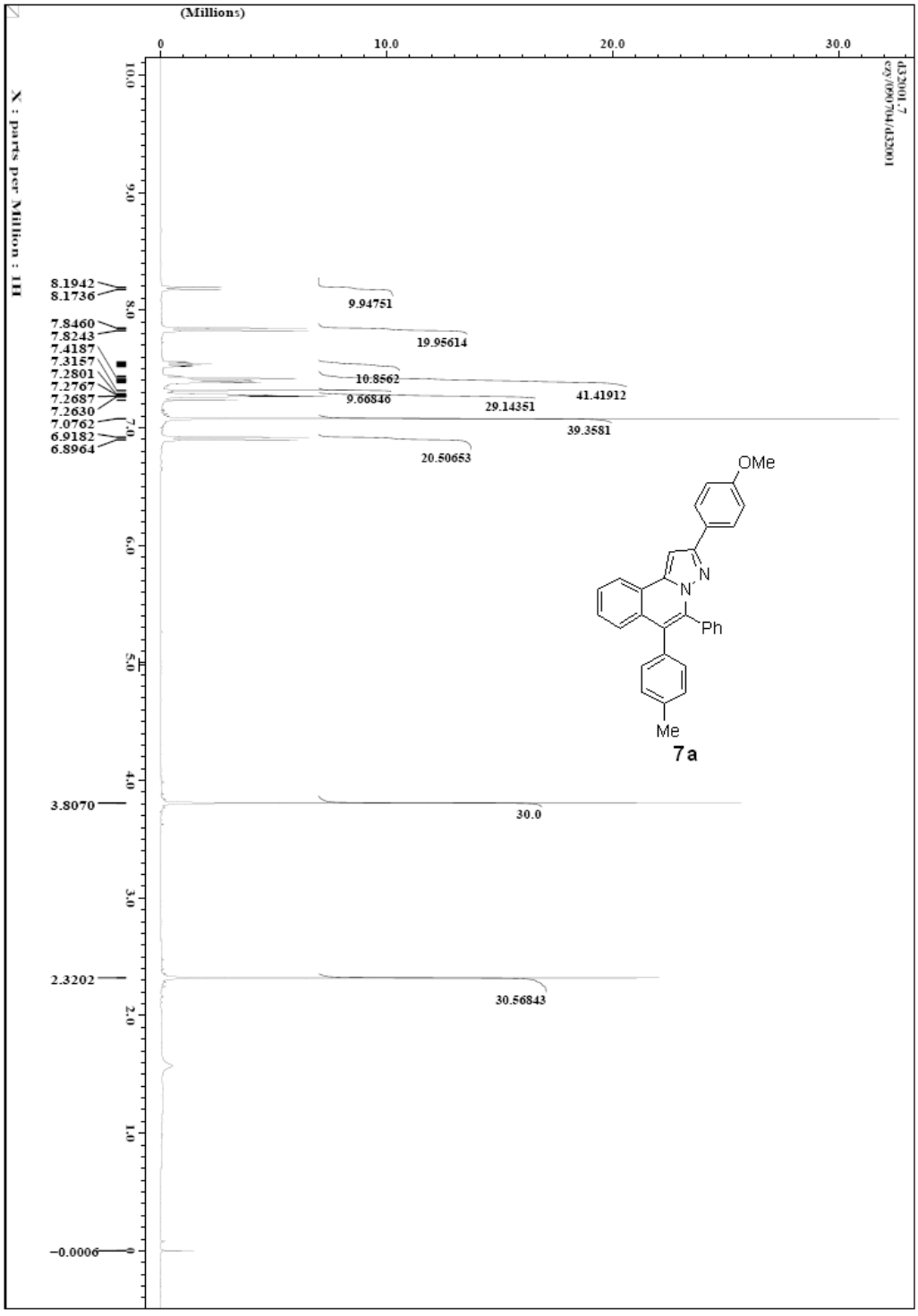

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
163.8176
161.3482
159.9847
152.8052
139.2565
138.8942
133.8695
130.6945
130.5610
129.4454
128.3299
127.7388
126.1656
126.0798
125.5267
120.4448
120.4257
116.9742
116.7359
114.0471
113.5704
113.3225
94.3105
77.4153
77.1006
76.7860
55.3332
OMe
N
N
F
Ph
Br
6j

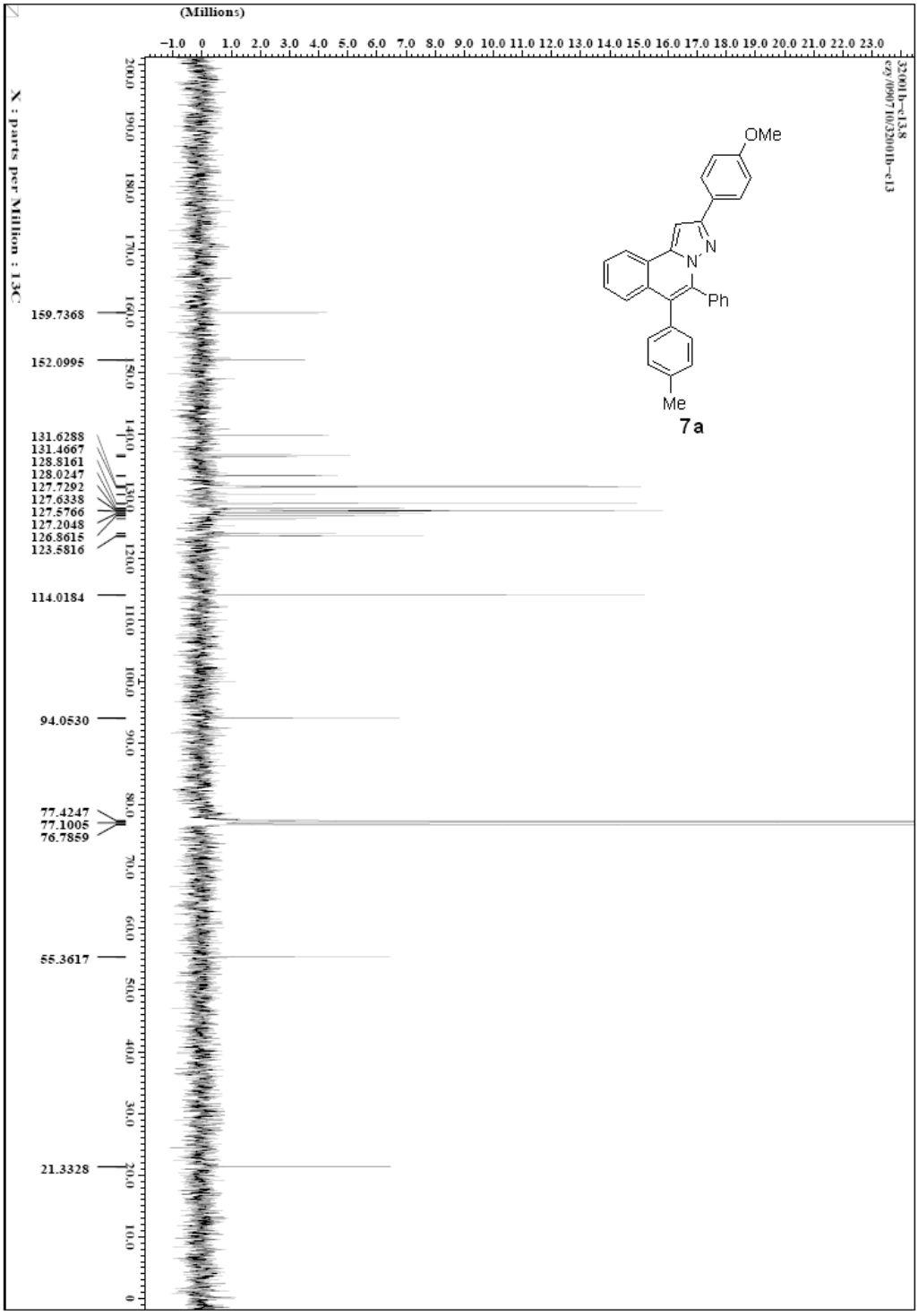

(Millions)
X : parts per Million : 1H
8.0043
7.9906
7.8635
7.8371
7.5496
7.5439
7.5393
7.5313
7.2472
6.5622
2.0627
2.0512
2.0421
0.9710
0.7809
0.7752
0.7683
0.7637
0.0764
−0.0003
10.2617
9.97385
50.8087
10.13512
10
10.13432
20.10531
19.83561
F
N
N
Ph
Br
6k

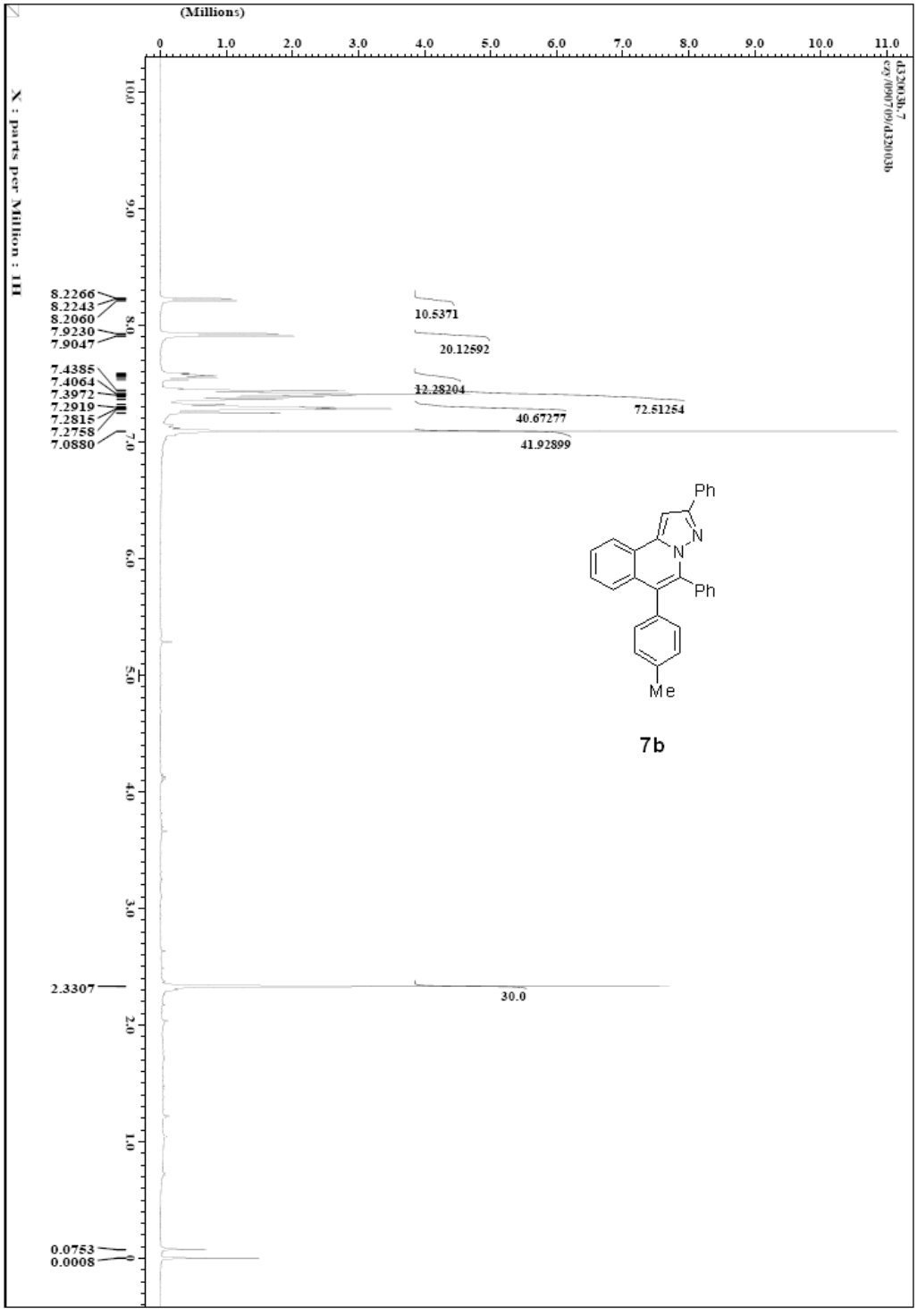

(Millions)
0
10.0
20.0
30.0
d38003-c13.7
czy/999/704/d38003-c13
X : parts per Million : 13C
163.7623
161.3024
158.2990
138.7817
138.6769
134.0431
130.6487
130.3436
129.4951
128.4939
126.0721
125.9863
120.2370
120.2179
116.8331
116.5948
113.4293
113.1814
93.1016
77.4172
77.1025
76.7879
9.8265
9.3307
F
N
N
Ph
Br
6k

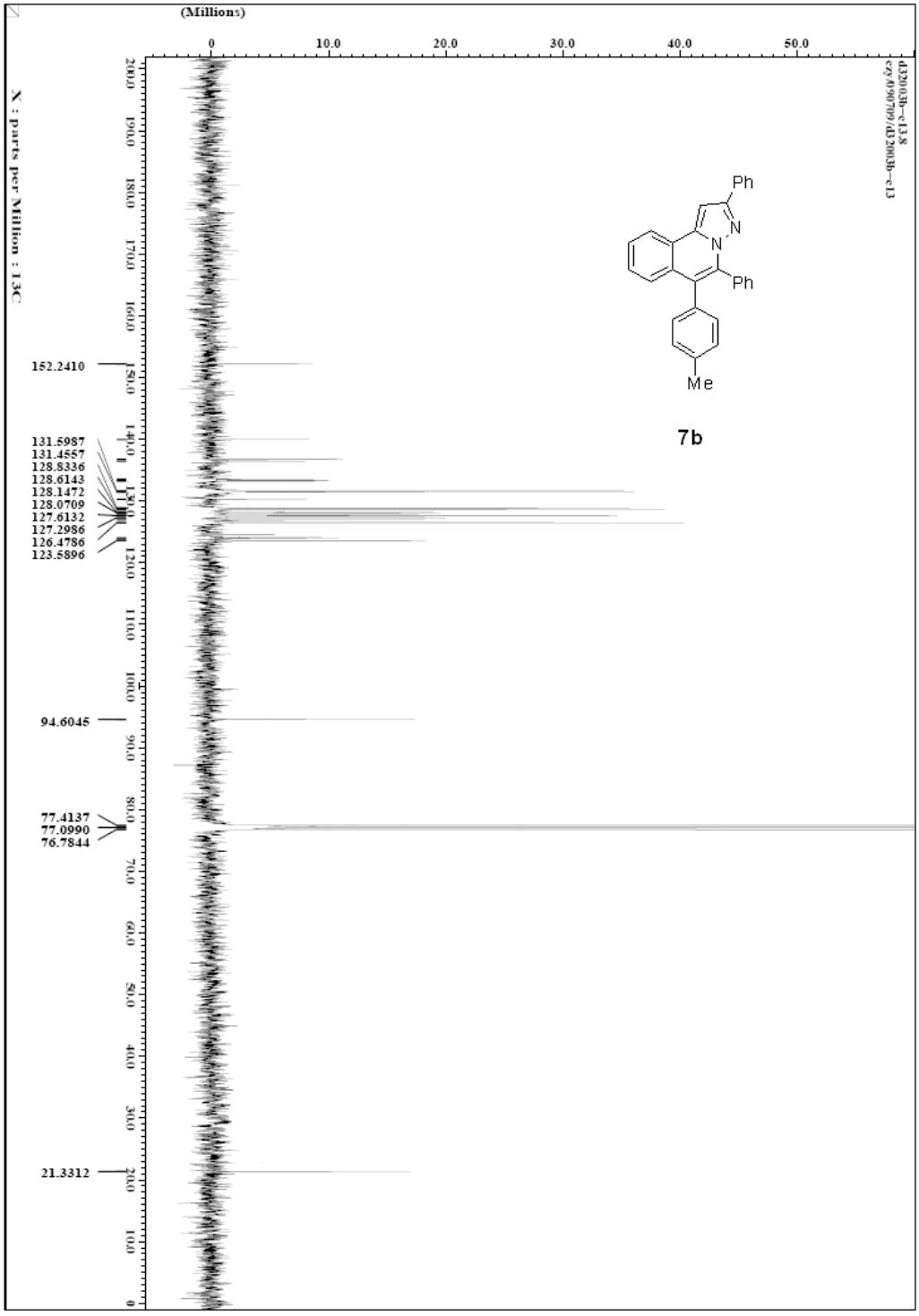

(Millions)
X : parts per Million : 1H
8.0482
8.0344
7.8741
7.8477
7.5453
7.5396
7.5338
7.2440
6.8122
2.7560
2.7365
2.7171
1.6666
1.6472
1.3906
1.3722
1.3539
0.9358
0.9175
0.8992
−0.0000
10.12882
9.66418
49.80801
9.99033
9.64381
20
22.94371
19.5887
30.87683
n-Bu
N
N
F
Ph
Br
6l

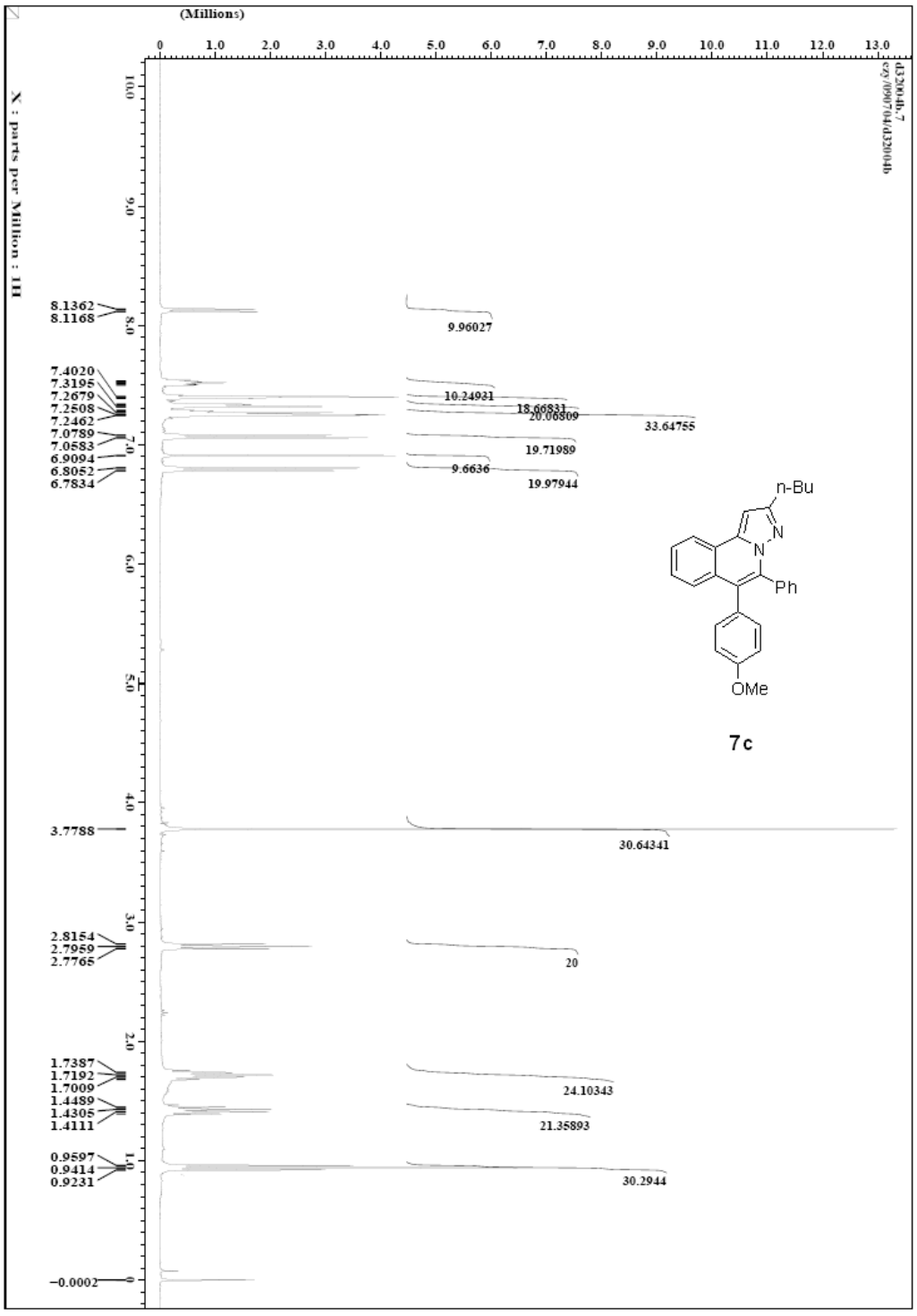

(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 13C
d38005-c13-9
czy1990704d38005-c13
n-Bu
N
N
F
Ph
Br
6l
163.7212
161.2518
156.2652
138.7787
138.6262
134.0973
130.5981
130.5027
130.3597
129.5207
128.5005
126.0977
126.0024
120.4151
120.3961
116.8397
116.6013
113.4263
113.1784
106.7807
106.7521
96.4929
77.4142
77.0995
76.7849
31.8580
28.3302
22.5999
13.9806

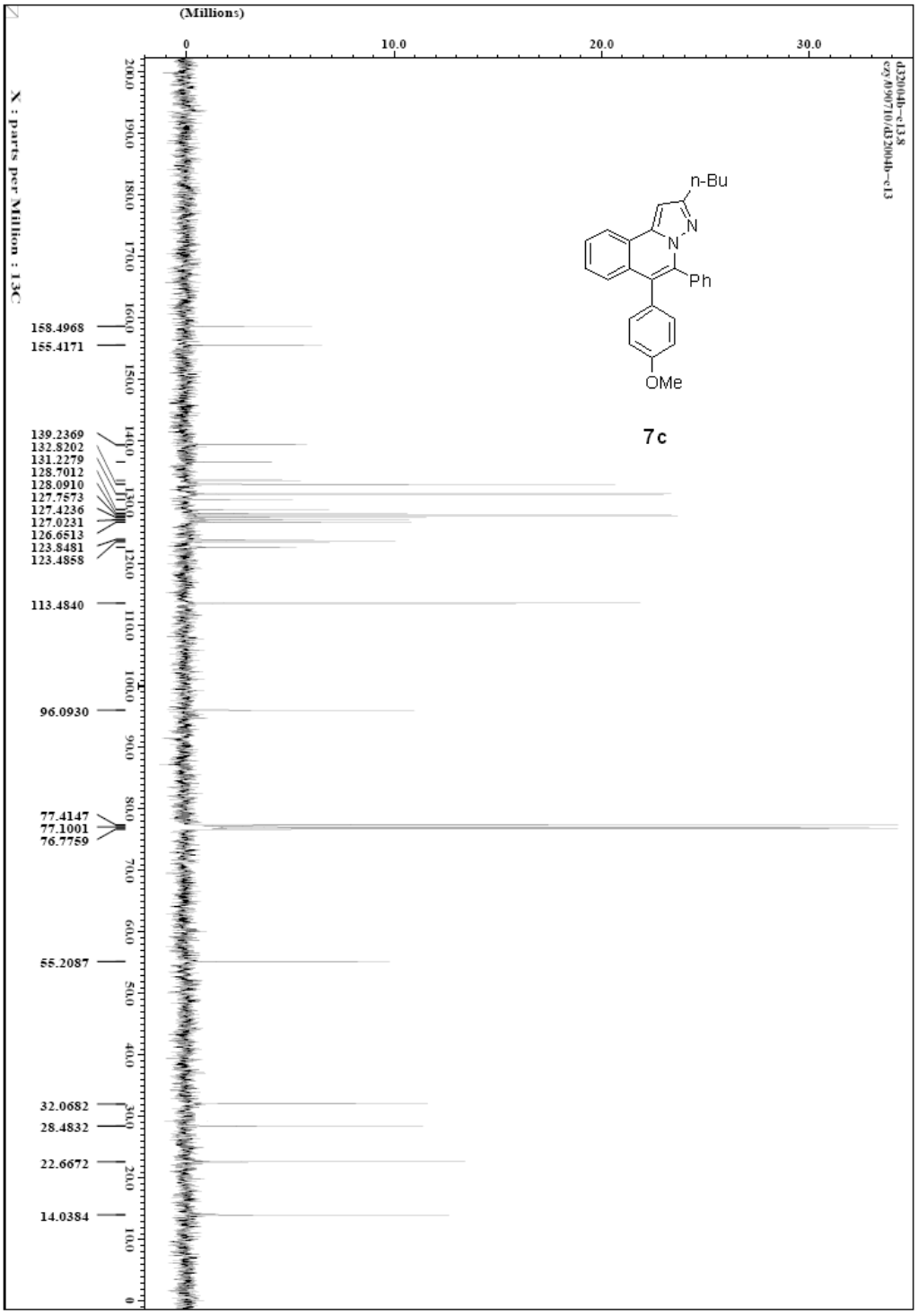

(Millions)
X : parts per Million : 1H
8.1942
8.1736
7.8460
7.8243
7.4187
7.3157
7.2801
7.2767
7.2687
7.2630
7.0762
6.9182
6.8964
3.8070
2.3202
−0.0006
9.94751
19.95614
10.8562
9.66846
41.41912
29.14351
39.3581
20.50653
30.0
30.56843
OMe
N
N
Ph
Me
7a

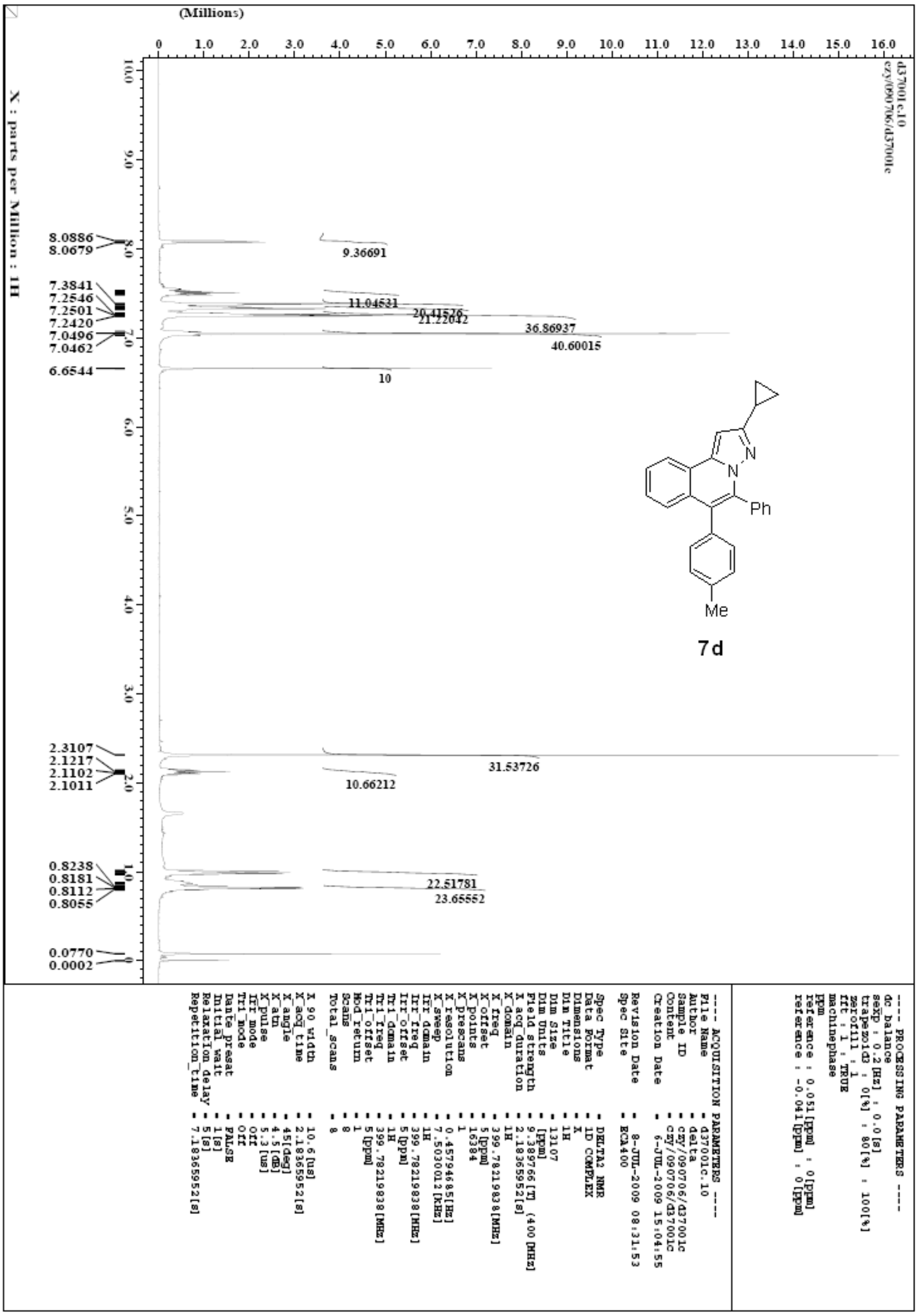

(Millions)
X : parts per Million : 13C
159.7368
152.0995
131.6288
131.4667
128.8161
128.0247
127.7292
127.6338
127.5766
127.2048
126.8615
123.5816
114.0184
94.0530
77.4247
77.1005
76.7859
55.3617
21.3328
OMe
N
N
Ph
Me
7a


(Millions)
X : parts per Million : 1H
8.2266
8.2243
8.2060
7.9230
7.9047
7.4385
7.4064
7.3972
7.2919
7.2815
7.2758
7.0880
2.3307
0.0753
0.0008
10.5371
20.12592
12.28204
72.51254
40.67277
41.92899
30.0
Ph
Ph
Me
7b

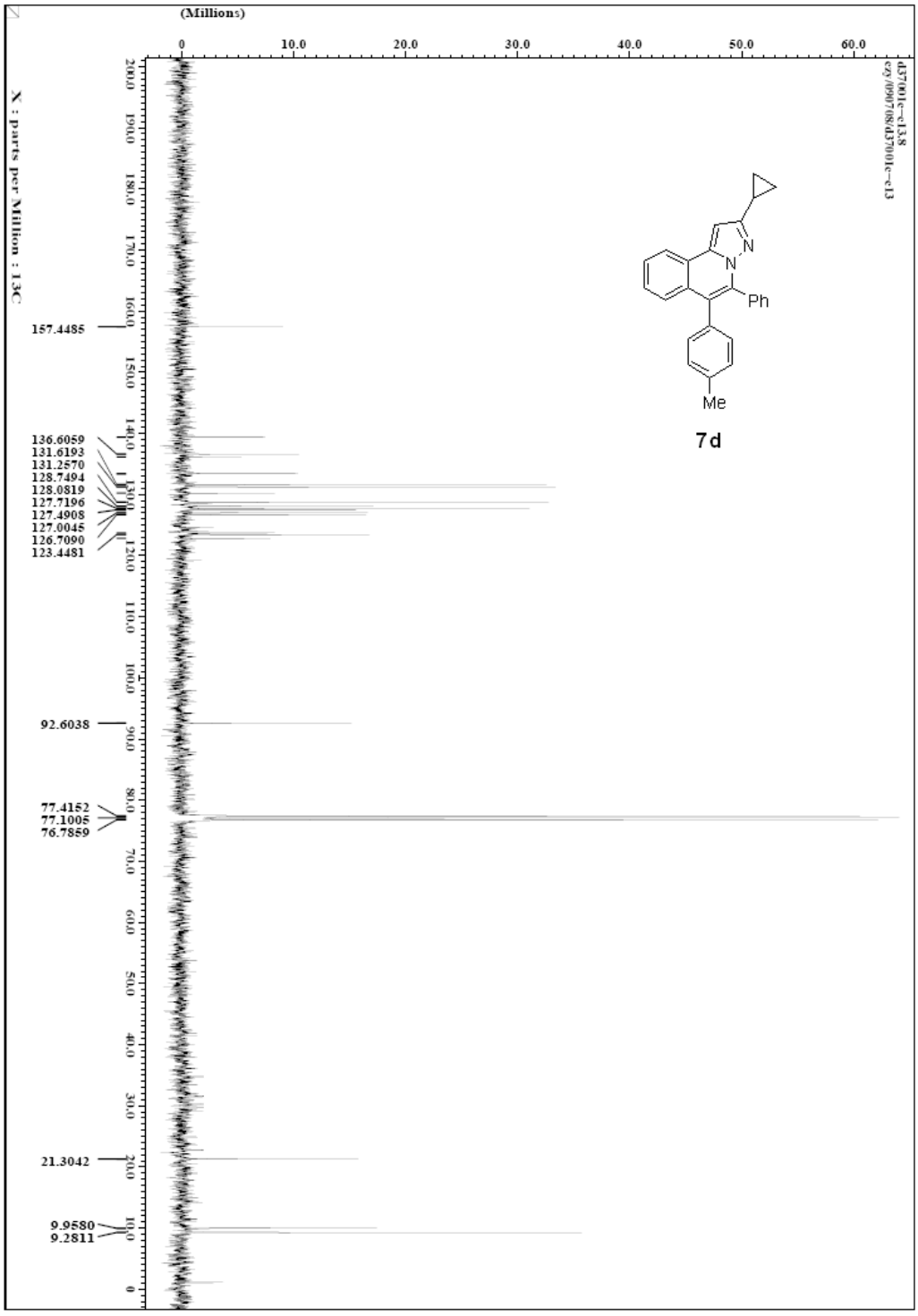

(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 13C
152.2410
131.5987
131.4557
128.8336
128.6143
128.1472
128.0709
127.6132
127.2986
126.4786
123.5896
94.6045
77.4137
77.0990
76.7844
21.3312
Ph
N
N
Ph
Me
7b

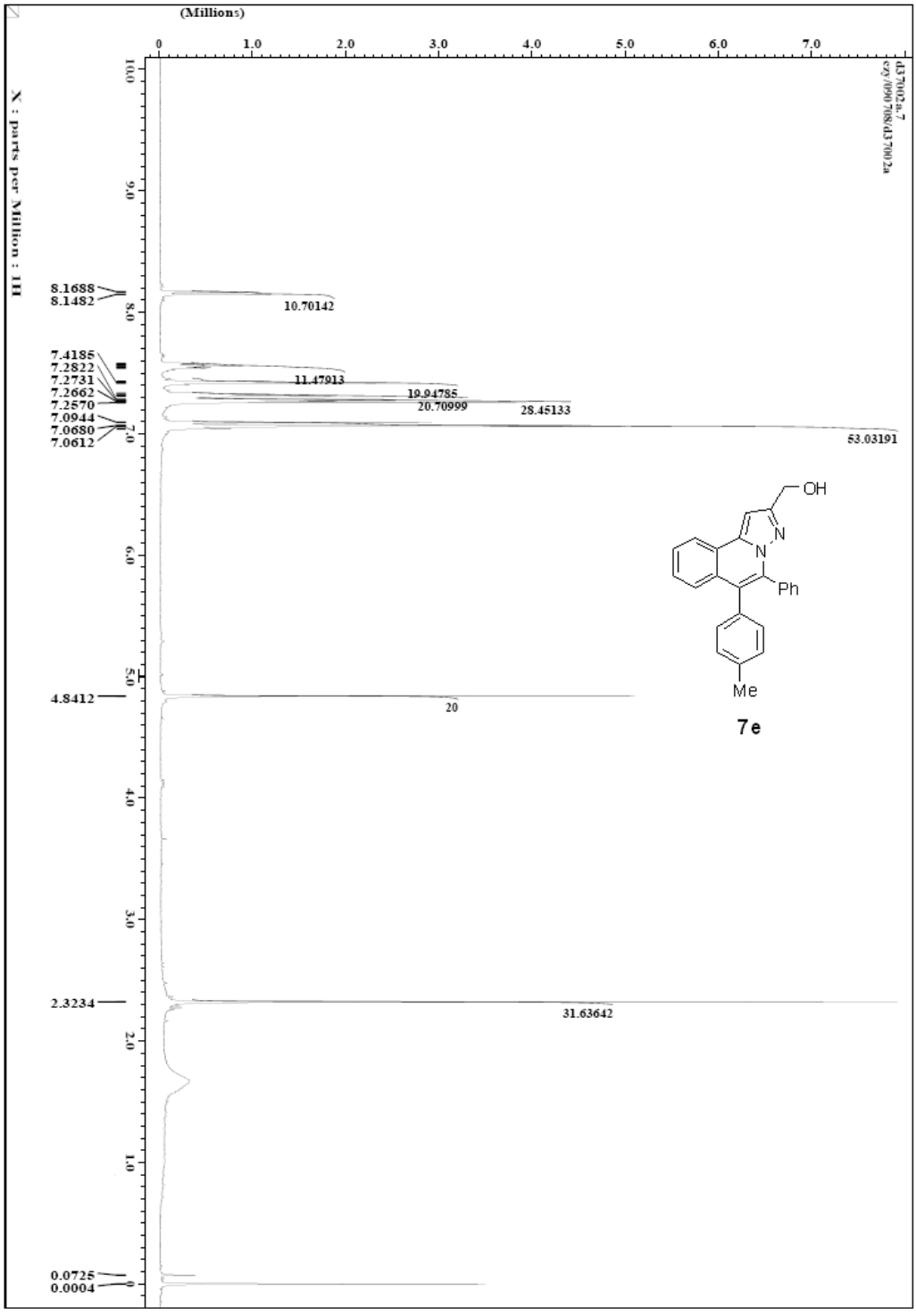

(Millions)
X : parts per Million : 1H
8.1362
8.1168
7.4020
7.3195
7.2679
7.2508
7.2462
7.0789
7.0583
6.9094
6.8052
6.7834
3.7788
2.8164
2.7959
2.7765
1.7387
1.7192
1.7009
1.4489
1.4305
1.4111
0.9597
0.9414
0.9231
−0.0002
9.96027
10.24931
18.66831
20.06809
33.64755
19.71989
9.6636
19.97944
30.64341
20
24.10343
21.35893
30.2944
n-Bu
N
N
Ph
OMe
7c

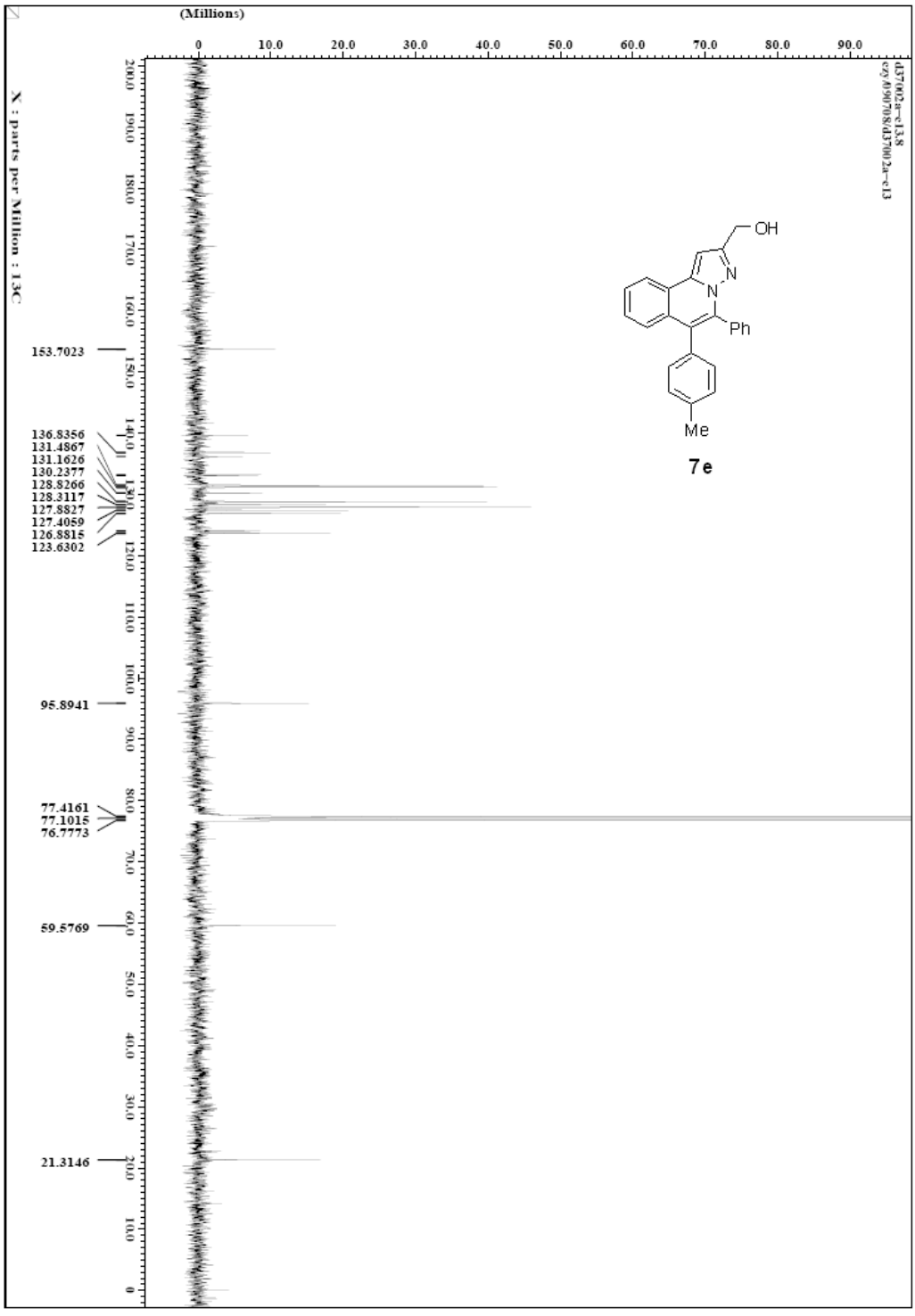

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
158.4968
155.4171
139.2369
132.8202
131.2279
128.7012
128.0910
127.7573
127.4236
127.0231
126.6513
123.8481
123.4858
113.4840
96.0930
77.4147
77.1001
76.7759
55.2087
32.0682
28.4832
22.6672
14.0384
n-Bu
N
N
Ph
OMe
7c

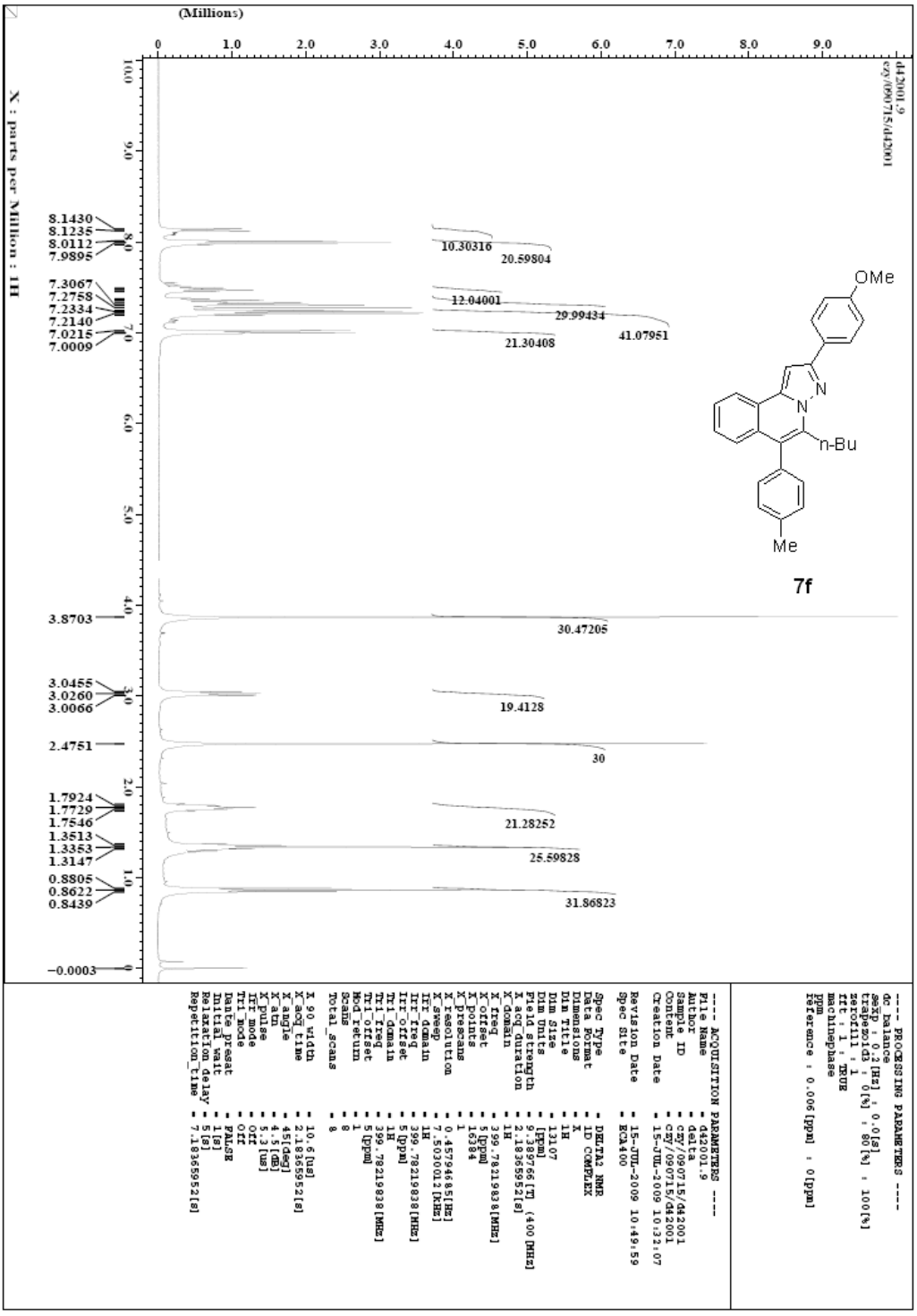


(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0
X : parts per Million : 1H
8.0886
8.0679
7.3841
7.2546
7.2501
7.2420
7.0496
7.0462
6.6544
2.3107
2.1217
2.1102
2.1011
0.8238
0.8181
0.8112
0.8055
0.0770
0.0002
9.36691
11.04531
20.41526
21.22042
36.86937
40.60015
10
31.53726
10.66212
22.51781
23.65552
Ph
Me
7d


(Millions)
X : parts per Million : 13C
157.4485
136.6059
131.6193
131.2570
128.7494
128.0819
127.7196
127.4908
127.0045
126.7090
123.4481
92.6038
77.4152
77.1005
76.7859
21.3042
9.9580
9.2811
Ph
Me
7d


(Millions)
X : parts per Million : 1H
8.1688
8.1482
7.4185
7.2822
7.2731
7.2662
7.2570
7.0944
7.0680
7.0612
4.8412
2.3234
0.0725
0.0004
10.70142
11.47913
19.94785
20.70999
28.45133
53.03191
20
31.63642
OH
N
N
Ph
Me
7e


(Millions)
X : parts per Million : 13C
153.7023
136.8356
131.4867
131.1626
130.2377
128.8266
128.3117
127.8827
127.4059
126.8815
123.6302
95.8941
77.4161
77.1015
76.7773
59.5769
21.3146
OH
N
N
Ph
Me
7e

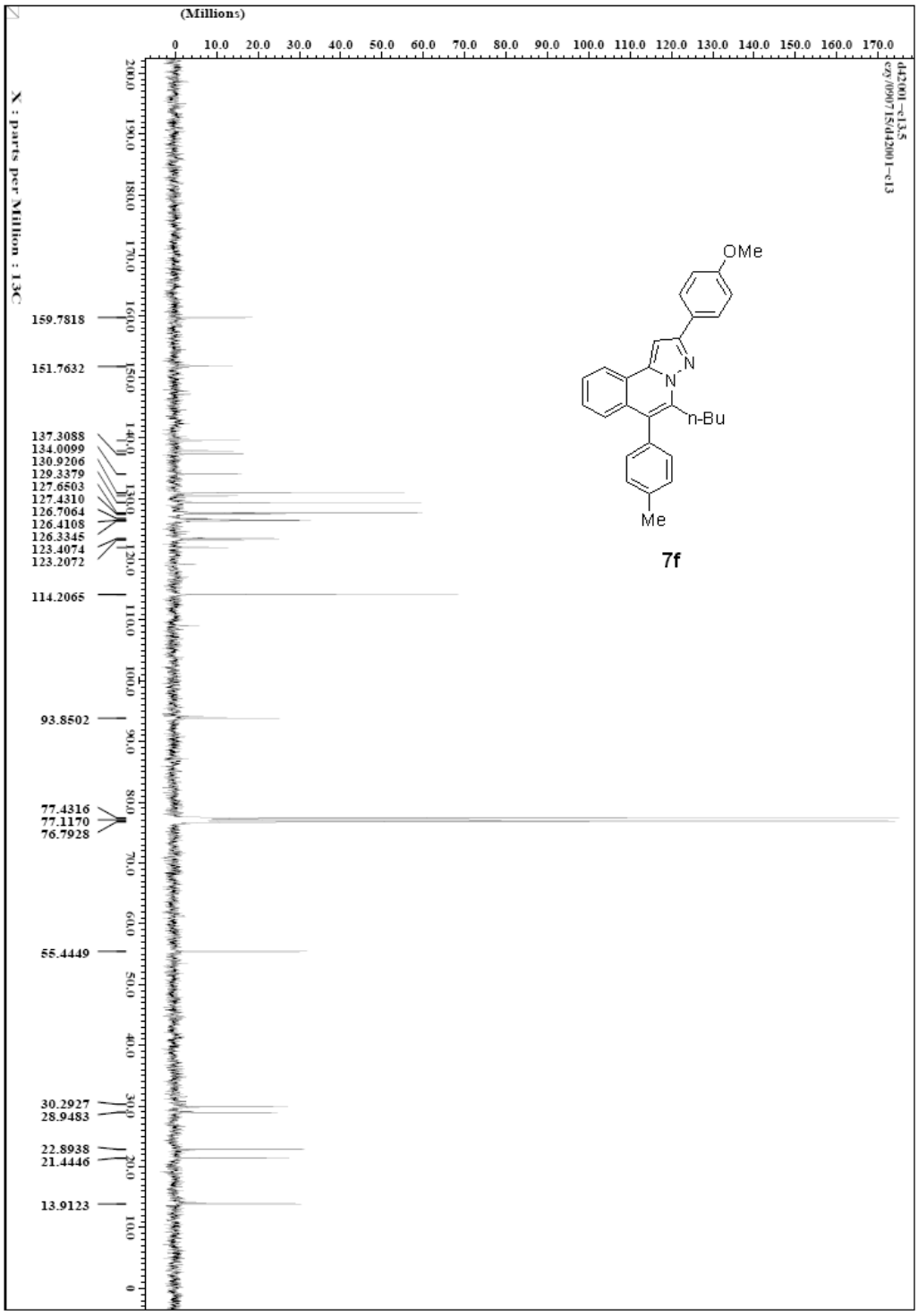


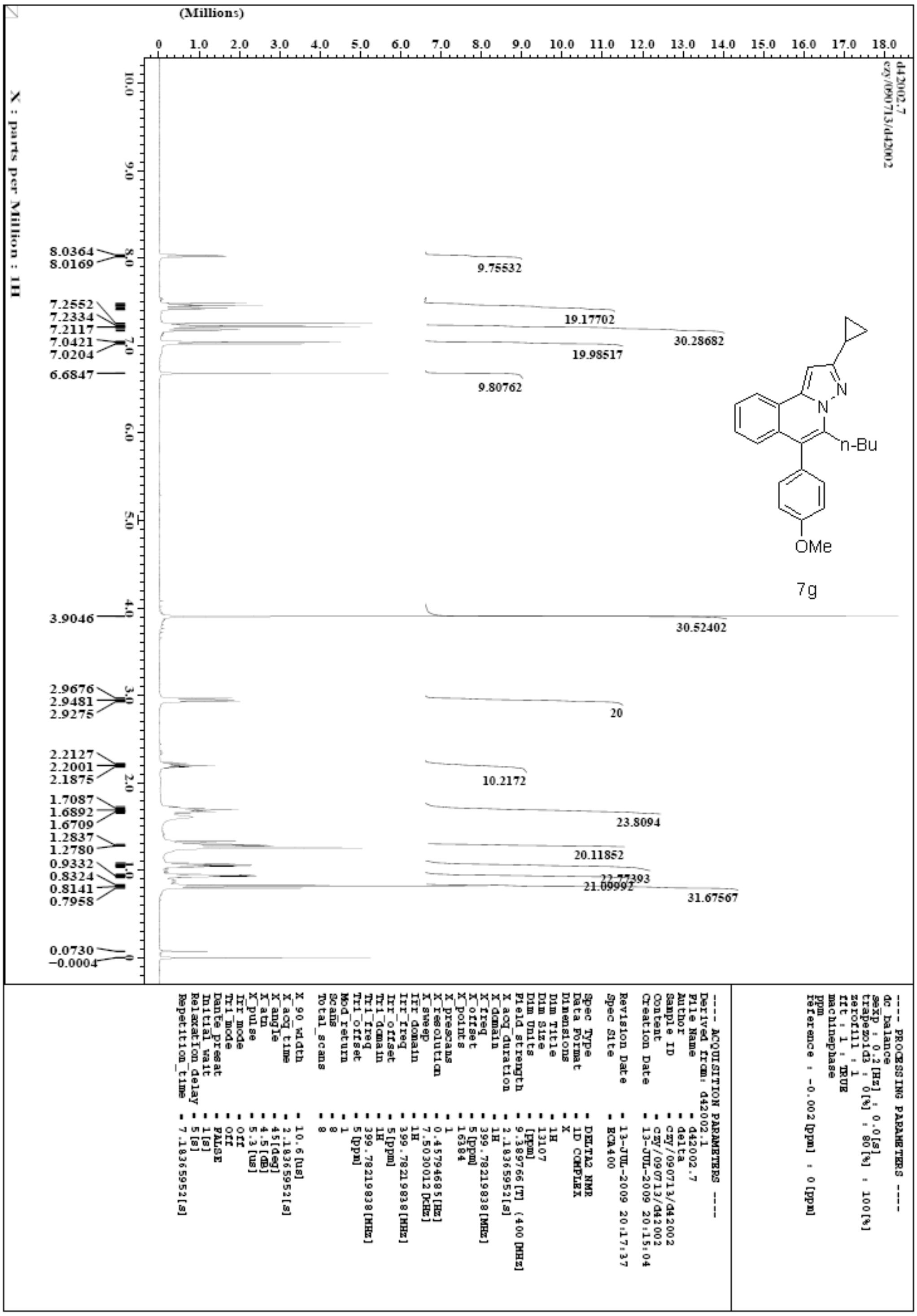

```
--- PROCESSING PARAMETERS ----
dc_flag : 0
sexp_flag : 0[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 0.006[ppm] : 0[ppm]
```

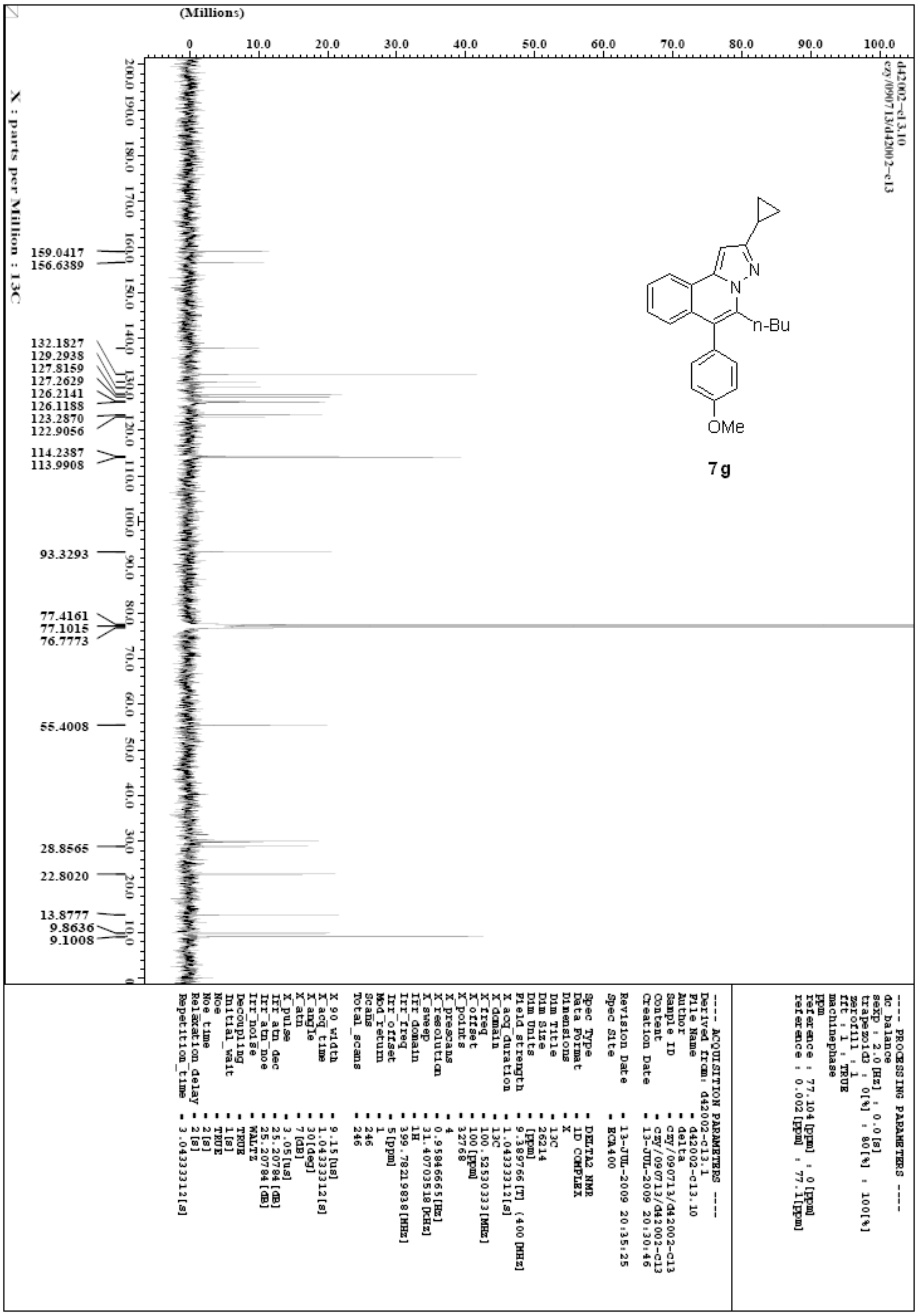
```
---- ACQUISITION PARAMETERS ----
File Name          = d42001.9
Author             = delta
Sample ID          = czy/090715/d42001
Content            = czy/090715/d42001
Creation Date      = 15-JUL-2009 10:32:07
Revision Date      = 15-JUL-2009 10:49:59
Spec Site          = EC2400

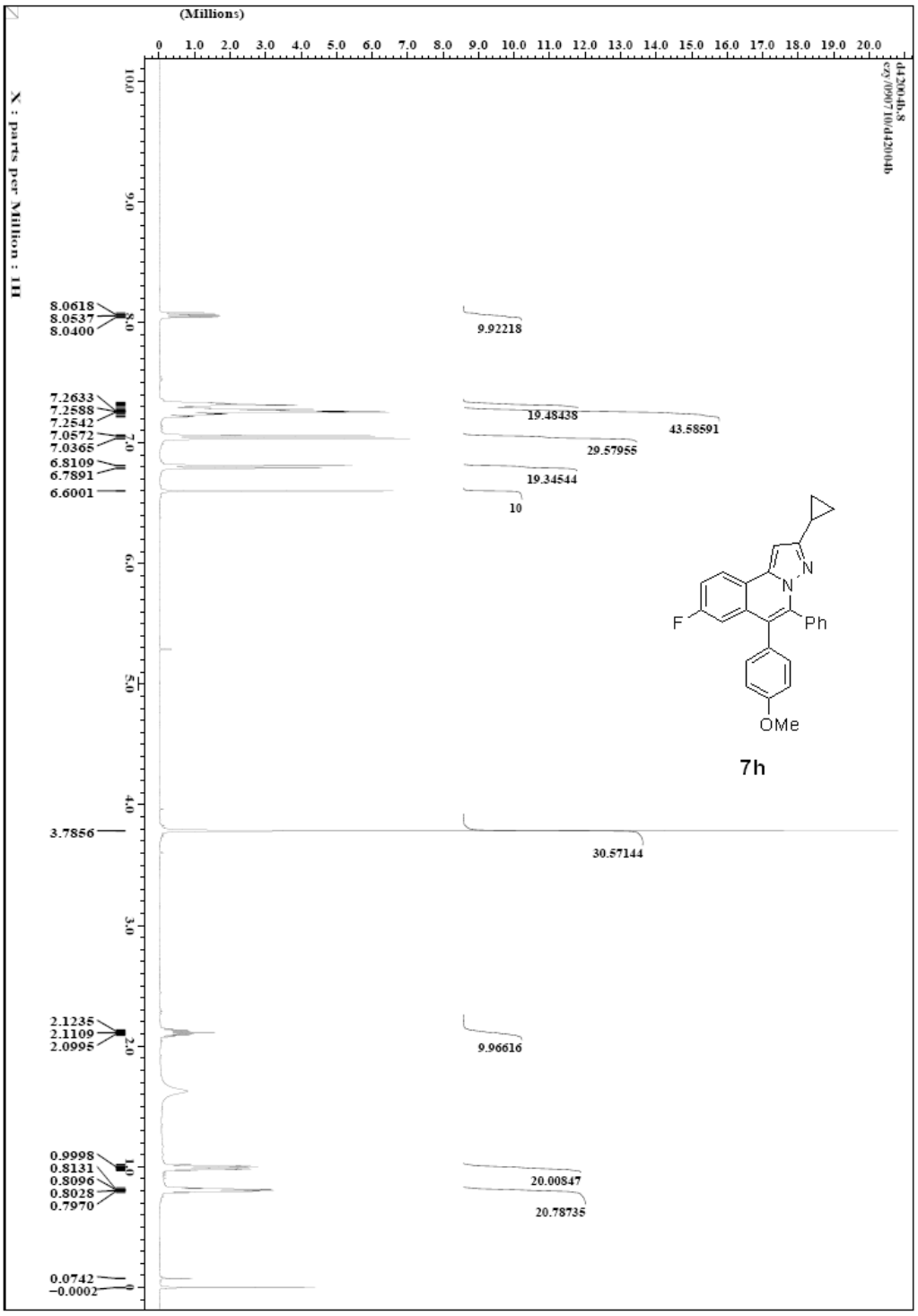
Spec Type          = DELTA2 NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 1H
Dim Size           = 13107
Dim Units          = [ppm]
Field strength     = 9.389766[T] (400 [MHz])
X_acq_duration     = 2.1836559520[s]
X_domain           = 1H
X_freq             = 399.7821983[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 0
X_resolution       = 0.4579468[Hz]
X_sweep            = 7.5030013[kHz]
IRr_domain         = 1H
IRr_freq           = 399.7821983[MHz]
IRr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 399.7821983[MHz]
Tri_offset         = 5[ppm]
Mod_return         = 1
Scans              = 8
Total_scans        = 8

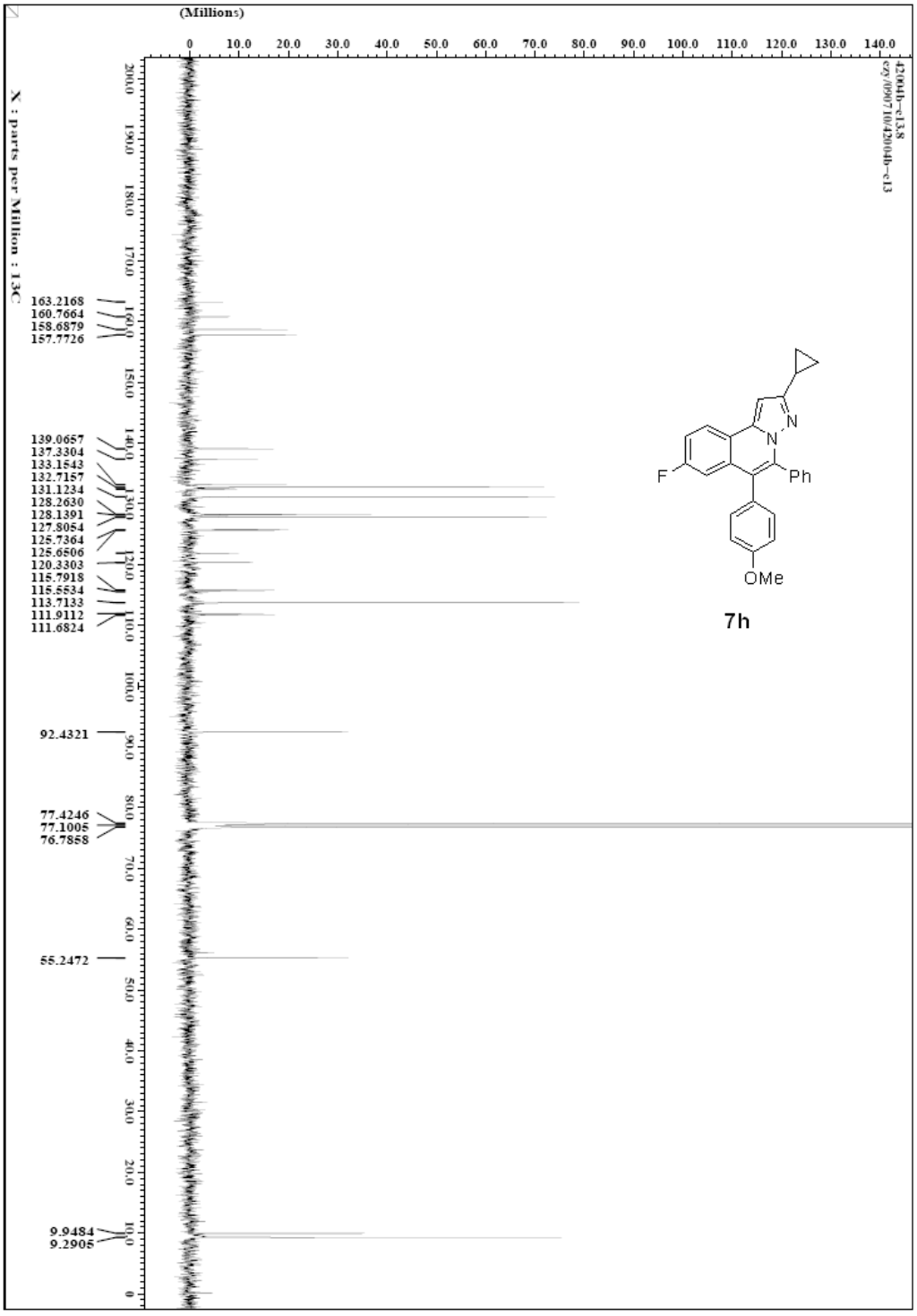
X_90_width         = 10.6[us]
X_acq_time         = 2.1836559520[s]
X_angle            = 45[deg]
X_atn              = 4.5[dB]
X_pulse            = 5.3[us]
IRr_mode           = off
Tri_mode           = off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Relaxation_delay   = 5[s]
Repetition_time    = 7.1836559520[s]
```


(Millions)
X : parts per Million : 13C
159.7818
151.7632
137.3088
134.0099
130.9206
129.3379
127.6503
127.4310
126.7064
126.4108
126.3345
123.4074
123.2072
114.2065
93.8502
77.4316
77.1170
76.7928
55.4449
30.2927
28.9483
22.8938
21.4446
13.9123
OMe
N
N
n-Bu
Me
7f

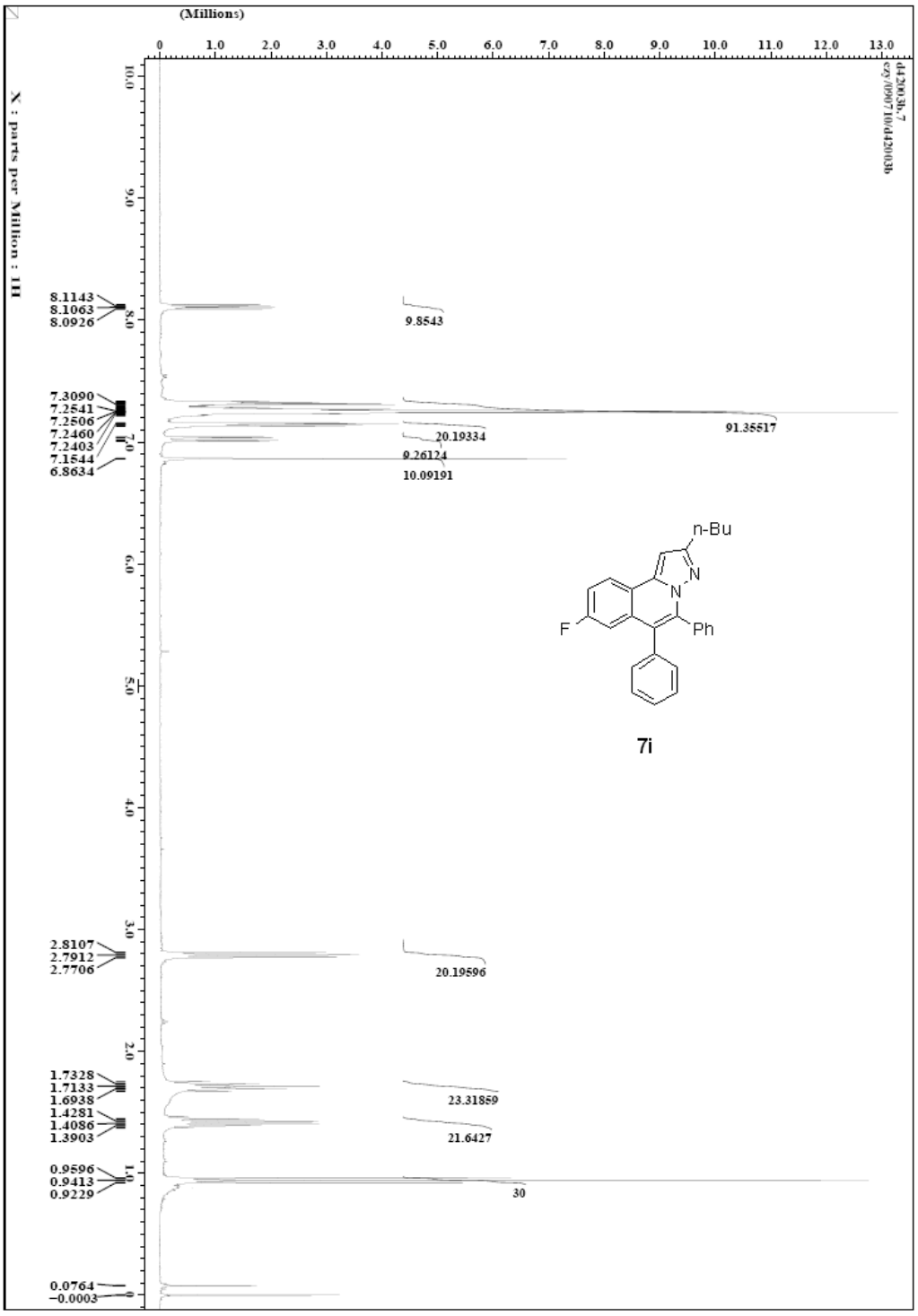

8.0364
8.0169
7.2552
7.2334
7.2117
7.0421
7.0204
6.6847
3.9046
2.9676
2.9481
2.9275
2.2127
2.2001
2.1875
1.7087
1.6892
1.6709
1.2837
1.2780
0.9332
0.8324
0.8141
0.7958
0.0730
−0.0004
9.75532
19.17702
30.28682
19.98517
9.80762
30.52402
20
10.2172
23.8094
20.11852
22.77393
21.09992
31.67567
n-Bu
OMe
7g

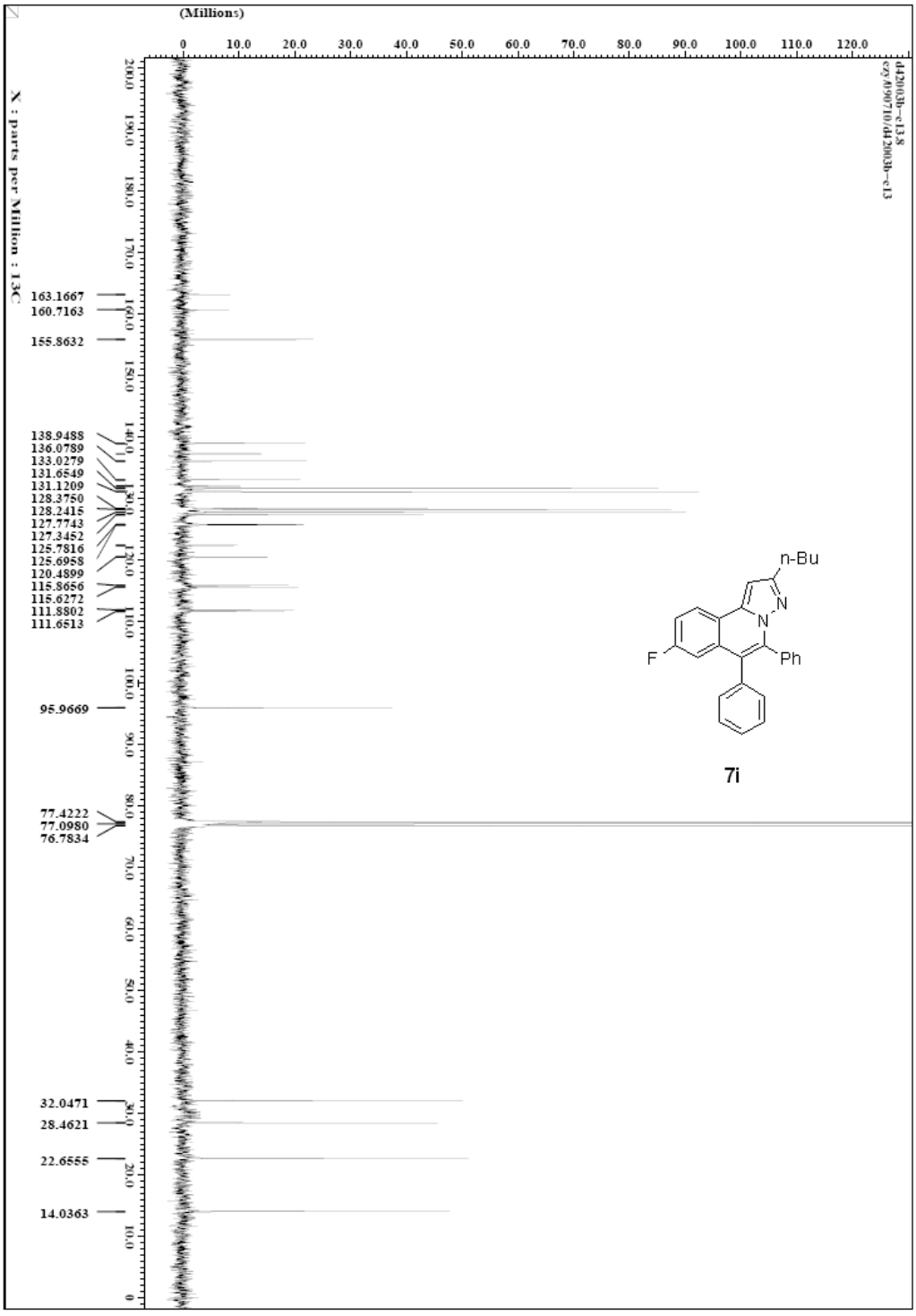

X : parts per Million : 13C
(Millions)
159.0417
156.6389
132.1827
129.2938
127.8159
127.2629
126.2141
126.1188
123.2870
122.9056
114.2387
113.9908
93.3293
77.4161
77.1015
76.7773
55.4008
28.8565
22.8020
13.8777
9.8636
9.1008
n-Bu
OMe
7g


(Millions)
X : parts per Million : 1H
8.0618
8.0537
8.0400
7.2633
7.2588
7.2542
7.0572
7.0365
6.8109
6.7891
6.6001
3.7856
2.1235
2.1109
2.0995
0.9998
0.8131
0.8096
0.8028
0.7970
0.0742
-0.0002
9.92218
19.48438
43.58591
29.57955
19.34544
10
30.57144
9.96616
20.00847
20.78735
F
N
N
Ph
OMe
7h


(Millions)
X : parts per Million : 13C
163.2168
160.7664
158.6879
157.7726
139.0657
137.3304
133.1543
132.7157
131.1234
128.2630
128.1391
127.8054
125.7364
125.6506
120.3303
115.7918
115.5534
113.7133
111.9112
111.6824
92.4321
77.4246
77.1005
76.7858
55.2472
9.9484
9.2905
F
N
N
Ph
OMe

7h


(Millions)
X : parts per Million : 1H
8.1143
8.1063
8.0926
7.3090
7.2541
7.2506
7.2460
7.2403
7.1544
6.8634
2.8107
2.7912
2.7706
1.7328
1.7133
1.6938
1.4281
1.4086
1.3903
0.9596
0.9413
0.9229
0.0764
−0.0003
9.8543
91.35517
20.19334
9.26124
10.09191
20.19596
23.31859
21.6427
30
n-Bu
N
N
F
Ph
7i


(Millions)
X : parts per Million : 13C
163.1667
160.7163
155.8632
138.9488
136.0789
133.0279
131.6549
131.1209
128.3750
128.2415
127.7743
127.3452
125.7816
125.6958
120.4899
115.8656
115.6272
111.8802
111.6513
95.9669
77.4222
77.0980
76.7834
32.0471
28.4621
22.6555
14.0363
n-Bu
N
N
F
Ph

7i