Tandem Electrophilic Cyclization-[3+2] Cycloaddition-Rearrangement Reactions of 2-Alkynylbenzaldoxime, DMAD, and Br_2

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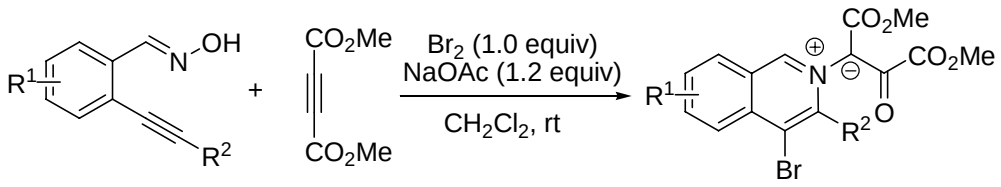
Supporting Information

General experimental methods:

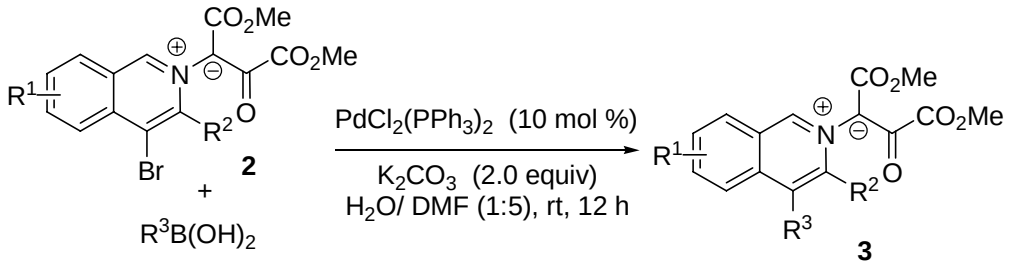
All reactions were performed in reaction tubes under nitrogen atmosphere. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated 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. 2-Alkynylbenzaldoxime was synthesized according to the literature method (Takao S.; Kondo Y.; Miura N.; Hayashi K.; Yamanaka H. *Heterocycles* **1986**, *24(8)*, 2311.).

General Procedure for Tandem Electrophilic Cyclization-[3+2] Cycloaddtion-Rearrangement Reactions of 2-Alkynylbenzaldoxime:

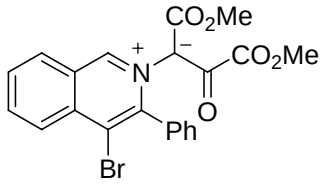


Br_2 (0.30 mmol, 1.0 equiv) in 2.0 mL of CH_2Cl_2 was added to a mixture of 2-alkynylbenzaldoxime **1** (0.30 mmol) and NaOAc (0.36 mmol, 1.2 equiv) in CH_2Cl_2 (2.0 mL). After 5 minutes, dimethyl acetylene dicarboxylate (DMAD) (0.60 mmol, 2.0 equiv) was added, and the reaction was stirred at room temperature. After completion of the reaction as indicated by TLC, the solvent was diluted with CH_2Cl_2 (10 mL), washed with saturated aqueous NaS_2O_3 (20 mL), dried by anhydrous Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding product **2**.

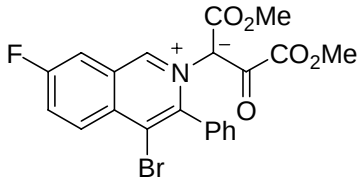
General Procedure for Synthesis of Functionalized Isoquinoline-Based Azomethine Ylides via Palladium-Catalyzed Suzuki Reactions:



A solution of compound **2** (0.25 mmol), $ArB(OH)_2$ (0.30 mmol, 1.2 equiv), K_2CO_3 (0.5 mmol, 2.0 equiv), $PdCl_2(PPh_3)_2$ (0.025 mmol, 10 mol %) in H_2O/DMF (1:5, 2.0 mL) was stirred at room temperature overnight. After completion of the reaction as indicated by TLC, the solvent was diluted with EtOAc (30 mL), washed with aqueous HCl (1.0 M, 10 mL), and dried by anhydrous Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding compound **3**.

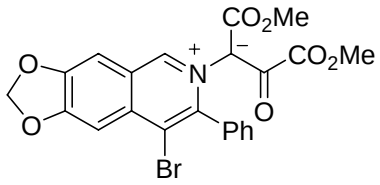


Compound **2a** yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.73 (s, 3H), 7.34 (d, *J* = 7.3 Hz, 1H), 7.44-7.48 (m, 1H), 7.49-7.53 (m, 3H), 7.97 (t, *J* = 8.3 Hz, 1H), 8.22 (t, *J* = 8.0 Hz, 1H), 8.28 (d, *J* = 7.8 Hz, 1H), 8.46 (d, *J* = 8.8 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.4, 51.8, 124.3, 127.3, 127.5, 127.6, 128.3, 128.4, 128.9, 130.4, 130.6, 131.0, 132.6, 137.9, 138.0, 150.0, 154.4, 167.9, 171.6; HRMS calcd for $C_{21}H_{17}BrNO_5^+$ $[M+H]^+$: 442.0290; Found: 442.0301.

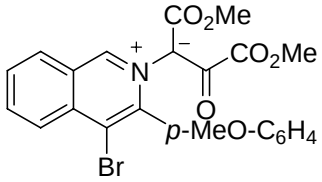


Compound **2b** yield: 63%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.73 (s, 3H), 7.32 (d, *J* = 7.3 Hz, 1H), 7.43-7.55 (m, 4H), 7.92-7.98 (m, 2H), 8.52 (dd, *J* = 4.9, 9.3

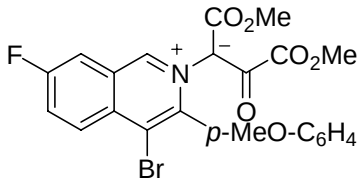
Hz, 1H), 9.34 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.5, 51.9, 113.7 (d, $^2J_{CF}$ = 22.9 Hz), 124.4, 127.6, 128.3 (d, $^2J_{CF}$ = 20.9 Hz), 128.4, 128.5, 128.9, 130.5, 131.0 (d, $^3J_{CF}$ = 8.6 Hz), 132.3, 135.2, 149.9, 153.6, 153.7, 162.6 (d, $^1J_{CF}$ = 256.5 Hz), 167.7, 171.5; HRMS calcd for $C_{21}H_{16}BrFNO_5^+$ $[M+H]^+$: 460.0196; Found: 460.0191.



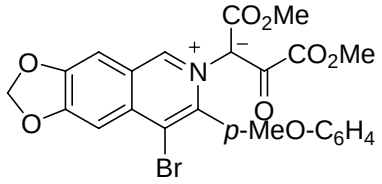
Compound **2c** yield: 42%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.72 (s, 3H), 6.35 (s, 2H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.38 (s, 1H), 7.41-7.46 (m, 1H), 7.47-7.52 (m, 3H), 7.72 (s, 1H), 8.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.5, 51.8, 103.8, 104.5, 104.8, 122.0, 125.3, 127.5, 128.3, 128.4, 128.8, 130.3, 132.9, 138.5, 149.4, 150.3, 151.2, 157.6, 168.1, 171.7; HRMS calcd for $C_{22}H_{17}BrNO_7^+$ $[M+H]^+$: 486.0188; Found: 486.0204.



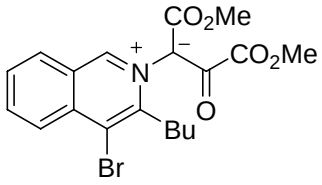
Compound **2d** yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.76 (s, 3H), 3.87 (s, 3H), 6.97 (dd, *J* = 2.0, 8.3 Hz, 1H), 7.01 (dd, *J* = 2.0, 8.8 Hz, 1H), 7.27 (d, *J* = 8.3 Hz, 1H), 7.45 (dd, *J* = 2.0, 8.0 Hz, 1H), 7.95 (t, *J* = 7.8 Hz, 1H), 8.20 (t, *J* = 7.3 Hz, 1H), 8.26 (d, *J* = 8.3 Hz, 1H), 8.44 (d, *J* = 8.8 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.4, 51.8, 55.1, 113.3, 113.4, 124.7, 124.8, 127.1, 127.6, 130.0, 130.5, 130.7, 130.8, 137.8, 137.9, 149.9, 154.3, 160.8, 168.0, 171.6; HRMS calcd for $C_{22}H_{19}BrNO_6^+$ $[M+H]^+$: 472.0395; Found: 472.0407.



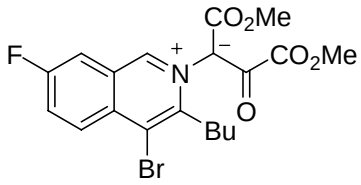
Compound **2e** yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.76 (s, 3H), 3.87 (s, 3H), 6.97 (dd, *J* = 2.4, 8.3 Hz, 1H), 7.01 (dd, *J* = 2.0, 8.3 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 1H), 7.43 (dd, *J* = 2.0, 8.3 Hz, 1H), 7.88-7.98 (m, 2H), 8.49 (dd, *J* = 4.4, 8.8 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.5, 51.8, 55.2, 113.7 (d, $^{2}J_{CF}$ = 22.9 Hz), 124.4, 127.6, 128.3 (d, $^{2}J_{CF}$ = 20.9 Hz), 128.4, 128.5, 128.9, 130.7, 131.0 (d, $^{3}J_{CF}$ = 8.6 Hz), 132.3, 135.2, 149.9, 153.5, 161.0, 162.5 (d, $^{1}J_{CF}$ = 256.5 Hz), 167.9, 171.5; HRMS calcd for $C_{22}H_{18}BrFNO_6^+$ $[M+H]^+$: 490.0302; Found: 490.0313.



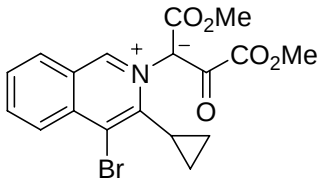
Compound **2f** yield: 52%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 3H), 3.75 (s, 3H), 3.86 (s, 3H), 6.33 (s, 2H), 6.94 (dd, *J* = 2.4, 8.3 Hz, 1H), 6.99 (dd, *J* = 2.0, 8.3 Hz, 1H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.39 (s, 1H), 7.43 (dd, *J* = 2.4, 8.8 Hz, 1H), 7.68 (s, 1H), 8.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.4, 51.8, 55.1, 103.8, 104.4, 104.7, 113.2, 113.3, 122.4, 125.1, 125.2, 129.9, 130.4, 138.3, 149.3, 150.2, 151.1, 157.5, 160.7, 168.1, 171.7; HRMS calcd for $C_{23}H_{19}BrNO_8^+$ $[M+H]^+$: 516.0294; Found: 516.0308.



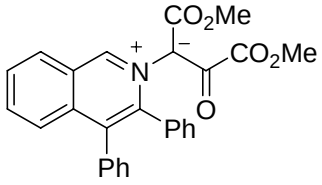
Compound **2g** yield: 66%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, 3H), 1.48-1.78 (m, 4H), 3.23-3.39 (m, 2H), 3.67 (s, 3H), 3.91 (s, 3H), 7.85 (d, *J* = 7.8 Hz, 1H), 8.11-8.17 (m, 2H), 8.34 (d, *J* = 8.8 Hz, 1H), 9.21(s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 22.7, 29.0, 33.5, 50.8, 51.9, 106.9, 123.1, 126.5, 126.8, 130.1, 130.2, 137.6, 138.0, 152.1, 154.7, 164.3, 168.1, 172.2; HRMS calcd for $C_{19}H_{21}BrNO_5^+$ $[M+H]^+$: 422.0603; Found: 422.0601.



Compound **2h** yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.3 Hz, 3H), 1.46-1.76 (m, 4H), 3.21-3.37 (m, 2H), 3.66 (s, 3H), 3.91 (s, 3H), 7.83 (dd, *J* = 2.0, 7.3 Hz, 1H), 7.89 (dt, *J* = 2.4, 8.3 Hz, 1H), 8.41 (dd, *J* = 4.9, 9.3 Hz, 1H), 9.22 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.3, 22.7, 29.0, 33.5, 50.9, 52.0, 107.3, 113.2 (d, $^{2}J_{CF}$ = 22.9 Hz), 123.2, 127. 5, 128.2, 130.2 (d, $^{3}J_{CF}$ = 8.6 Hz), 135.4, 152.1, 154.0, 162.0 (d, $^{1}J_{CF}$ = 255.5 Hz), 164.3, 168.0, 172.2; HRMS calcd for $C_{19}H_{20}BrFNO_5^+$ $[M+H]^+$: 440.0509; Found: 440.0519.

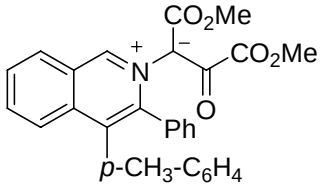


Compound **2i** yield: 75%; ^{1}H NMR (400 MHz, DMSO) δ 0.82-0.88 (m, 2H), 1.38-1.42 (m, 2H), 2.14-2.22 (m, 1H), 3.69 (s, 3H), 3.93 (s, 3H), 7.87 (t, *J* = 8.3 Hz, 1H), 8.11-8.15 (m, 2H), 8.44 (d, *J* = 8.8 Hz, 1H), 9.23(s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.5, 11.3, 15.0, 50.9, 52.0, 108.1, 126.1, 126.4, 126.9, 130.1, 130.3, 137.3, 137.9, 149.9, 153.9, 164.3, 168.3, 171.3; HRMS calcd for $C_{18}H_{17}BrNO_5^+$ $[M+H]^+$: 406.0290; Found: 406.0298.

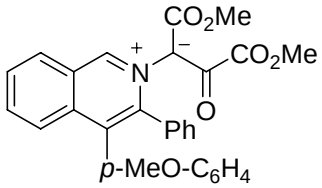


Compound **3a** yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.43 (s, 3H), 3.74 (s, 3H), 6.96 (br, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 7.20-7.32 (m, 5H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.43 (d, *J* = 7.3 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.89 (t, *J* = 7.3 Hz, 1H), 7.97 (t, *J* = 8.3 Hz, 1H), 8.28 (d, *J* = 7.8 Hz, 1H), 9.36 (s, 1H); ^{13}C NMR

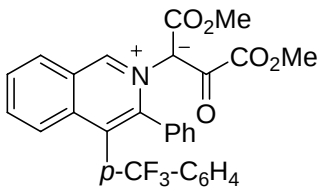
(100 MHz, $CDCl_3$) δ 50.3, 51.7, 126.4, 126.8, 127.4, 127.5, 128.0, 128.3, 128.5, 129.2, 129.4, 129.7, 130.0, 130.2, 131.3, 133.6, 136.3, 138.3, 138.4, 148.7, 153.9, 168.1, 171.8; HRMS calcd for $C_{27}H_{22}NO_5^+$ $[M+H]^+$: 440.1498; Found: 440.1508.



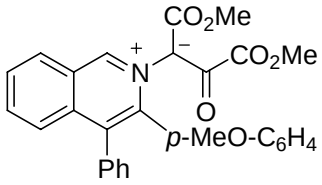
Compound **3b** yield: 77%; ^1H NMR (400 MHz, $CDCl_3$) δ 2.32 (s, 3H), 3.43 (s, 3H), 3.74 (s, 3H), 6.88 (d, *J* = 7.8 Hz, 1H), 6.98 (br, 1H), 7.04 (d, *J* = 7.8 Hz, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 7.17-7.30 (m, 4H), 7.43 (d, *J* = 7.3 Hz, 1H), 7.73 (d, *J* = 8.8 Hz, 1H), 7.87 (t, *J* = 8.3 Hz, 1H), 7.95 (t, *J* = 7.3 Hz, 1H), 8.26 (t, *J* = 7.8 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.1, 50.3, 51.7, 126.5, 126.8, 127.4, 127.5, 128.7, 129.1, 129.2, 129.5, 129.7, 129.9, 130.0, 130.1, 130.6, 131.4, 136.1, 138.2, 138.5, 148.7, 153.7, 168.2, 171.7; HRMS calcd for $C_{28}H_{24}NO_5^+$ $[M+H]^+$: 454.1654; Found: 454.1665.



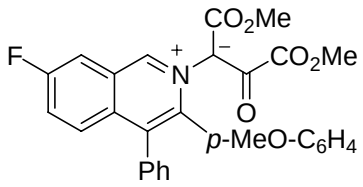
Compound **3c** yield: 70%; ^1H NMR (400 MHz, $CDCl_3$) δ 3.43 (s, 3H), 3.74 (s, 3H), 3.78 (s, 3H), 6.76 (dd, *J* = 2.4, 8.3 Hz, 1H), 6.91 (dt, *J* = 2.4, 8.3 Hz, 2H), 6.98 (br, 1H), 7.12 (t, *J* = 7.3 Hz, 1H), 7.19-7.30 (m, 3H), 7.43 (d, *J* = 7.3 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 1H), 7.88 (t, *J* = 7.3 Hz, 1H), 7.97 (t, *J* = 7.3 Hz, 1H), 8.26 (t, *J* = 8.3 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.2, 51.7, 55.1, 113.6, 113.7, 125.6, 126.4, 126.9, 127.3, 127.5, 129.1, 129.4, 129.6, 129.9, 131.4, 131.5, 136.1, 138.2, 138.6, 148.9, 153.6, 159.3, 168.2, 171.7; HRMS calcd for $C_{28}H_{24}NO_6^+$ $[M+H]^+$: 470.1604; Found: 470.1615.



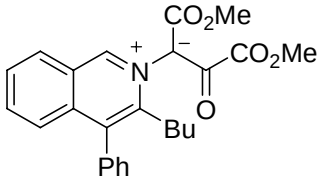
Compound **3d** yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.45 (s, 3H), 3.75 (s, 3H), 6.97 (br, 1H), 7.12 (t, *J* = 8.3 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 7.25-7.32 (m, 2H), 7.41-7.45 (m, 2H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.64 (t, *J* = 8.8 Hz, 2H), 7.93 (t, *J* = 7.3 Hz, 1H), 8.01 (t, *J* = 7.3 Hz, 1H), 8.31 (d, *J* = 8.3 Hz, 1H), 9.33 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.5, 51.8, 123.6 (q, $^{1}J_{CF}$ = 270.8 Hz), 125.3, 125.6, 125.9, 127.1, 127.4, 127.8, 129.4, 129.6, 129.7, 130.3, 130.5 (d, $^{2}J_{CF}$ = 32.0 Hz), 130.7, 130.8, 136.8, 136.9, 137.5, 137.9, 148.9, 154.7, 168.1, 171.8; HRMS calcd for $C_{28}H_{21}F_3NO_5^+$ $[M+H]^+$: 508.1372; Found: 508.1371.



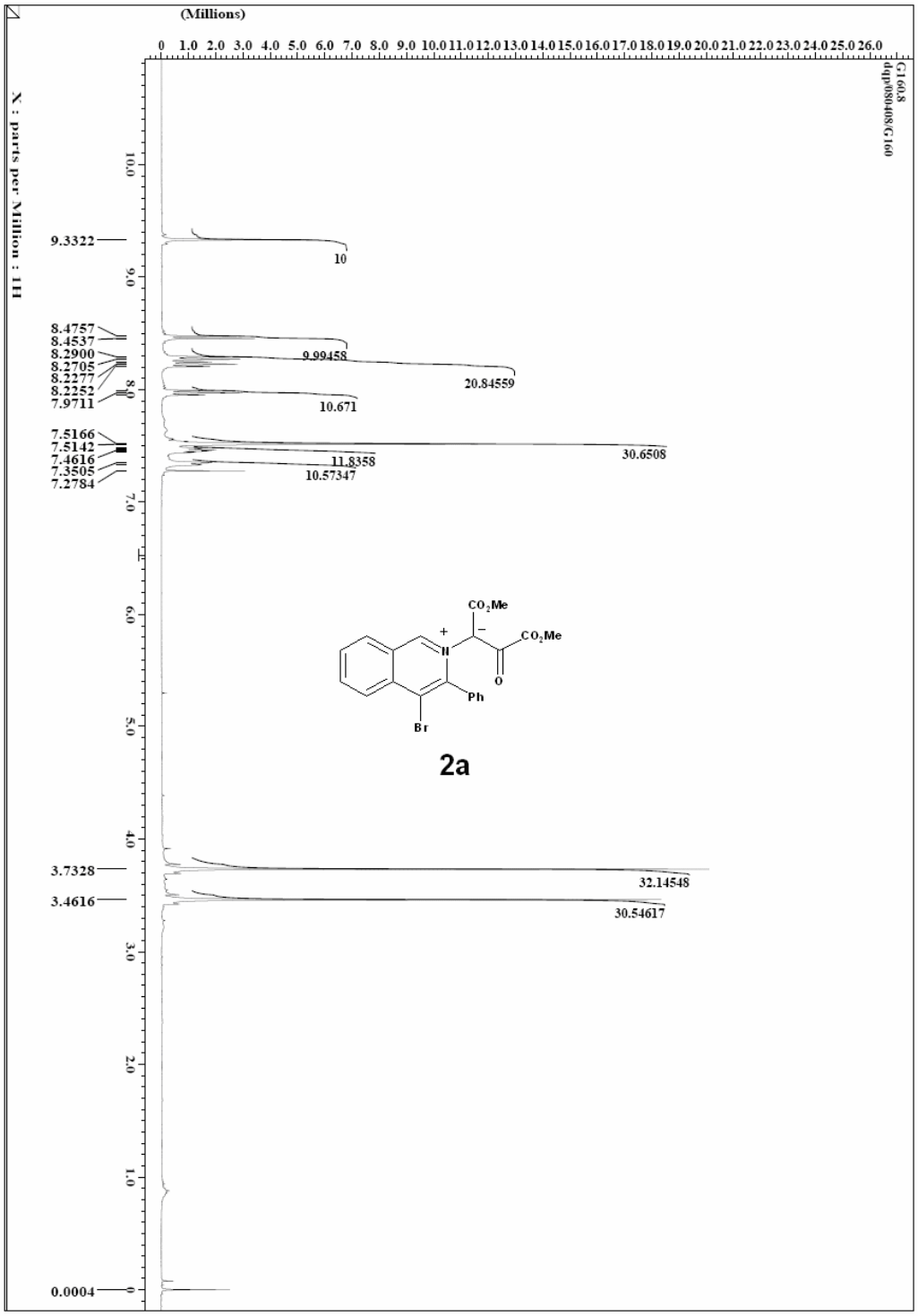
Compound **3e** yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.44 (s, 3H), 3.73 (s, 3H), 3.78 (s, 3H), 6.59 (dd, *J* = 2.4, 8.8 Hz, 1H), 6.80 (dd, *J* = 2.4, 8.8 Hz, 1H), 6.85 (br, 1H), 6.97 (d, *J* = 7.3 Hz, 1H), 7.23-7.42 (m, 5H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.86 (t, *J* = 7.8 Hz, 1H), 7.95 (t, *J* = 7.3 Hz, 1H), 8.25 (t, *J* = 8.3 Hz, 1H), 9.34 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.4, 51.8, 55.0, 112.6, 112.9, 123.6, 126.4, 127.3, 128.0, 128.3, 128.6, 129.8, 129.9, 130.1, 130.2, 131.0, 131.3, 133.9, 136.2, 138.4, 138.5, 148.8, 153.9, 159.9, 168.3, 171.8; HRMS calcd for $C_{28}H_{24}NO_6^+$ $[M+H]^+$: 470.1604; Found: 470.1617.



Compound **3f** yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.43 (s, 3H), 3.73 (s, 3H), 3.77 (s, 3H), 6.59 (dd, *J* = 2.4, 8.3 Hz, 1H), 6.80 (dd, *J* = 2.4, 8.3 Hz, 1H), 6.84 (br, 1H), 6.97 (d, *J* = 7.8 Hz, 1H), 7.24-7.42 (m, 5H), 7.67-7.75 (m, 2H), 7.87 (dd, *J* = 2.0, 7.3 Hz, 1H), 9.32 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.4, 51.9, 55.0, 112.7, 112.8 (d, $^{2}J_{CF}$ = 21.9 Hz), 113.0, 123.3, 126.6, 126.9, 128.2, 128.7 (d, $^{2}J_{CF}$ = 22.9 Hz), 129.7 (d, $^{3}J_{CF}$ = 8.6 Hz), 130.1, 131.0, 131.3, 133.7, 135.6, 138.9, 148.7, 152.9, 160.0, 162.0 (d, $^{1}J_{CF}$ = 254.6 Hz), 168.2, 171.7; HRMS calcd for $C_{28}H_{23}FNO_6^+$ $[M+H]^+$: 488.1509; Found: 488.1519.



Compound **3g** yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.72 (t, *J* = 7.3 Hz, 3H), 1.15-1.28 (m, 2H), 1.32-1.60 (m, 2H), 2.72-2.88 (m, 2H), 3.68 (s, 3H), 3.90 (s, 3H), 7.32-7.38 (m, 2H), 7.45 (d, *J* = 8.8 Hz, 1H), 7.53-7.62 (m, 3H), 7.78 (t, *J* = 7.3 Hz, 1H), 7.88 (t, *J* = 7.3 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 9.22(s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.0, 22.6, 29.5, 30.5, 50.7, 51.8, 106.3, 125.8, 126.5, 128.9, 129.0, 129.3, 129.4, 129.5, 134.1, 135.9, 137.4, 138.6, 151.0, 154.4, 164.7, 168.3, 172.3; HRMS calcd for $C_{25}H_{26}NO_5^+$ $[M+H]^+$: 420.1811; Found: 420.1825.


(Millions)
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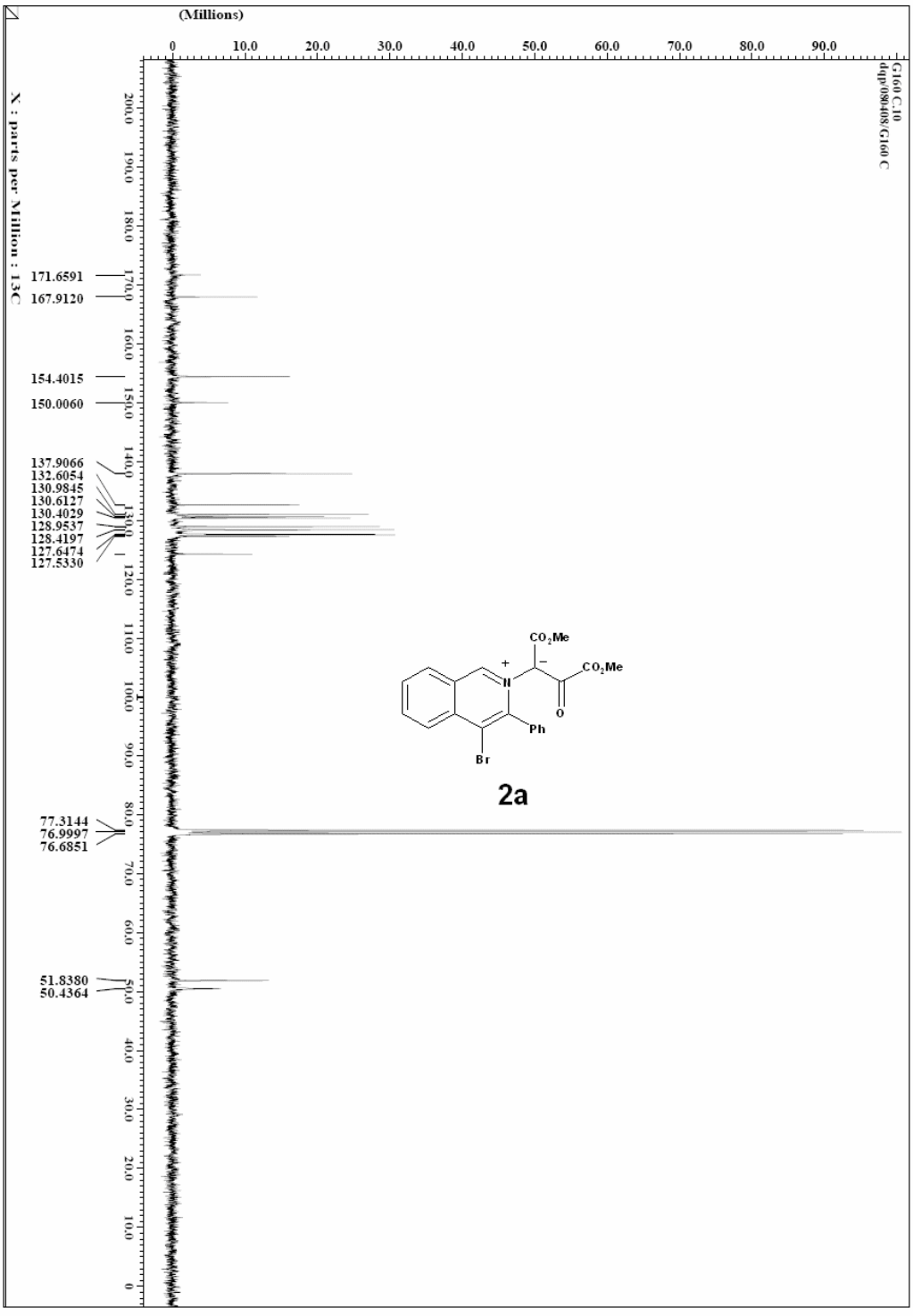
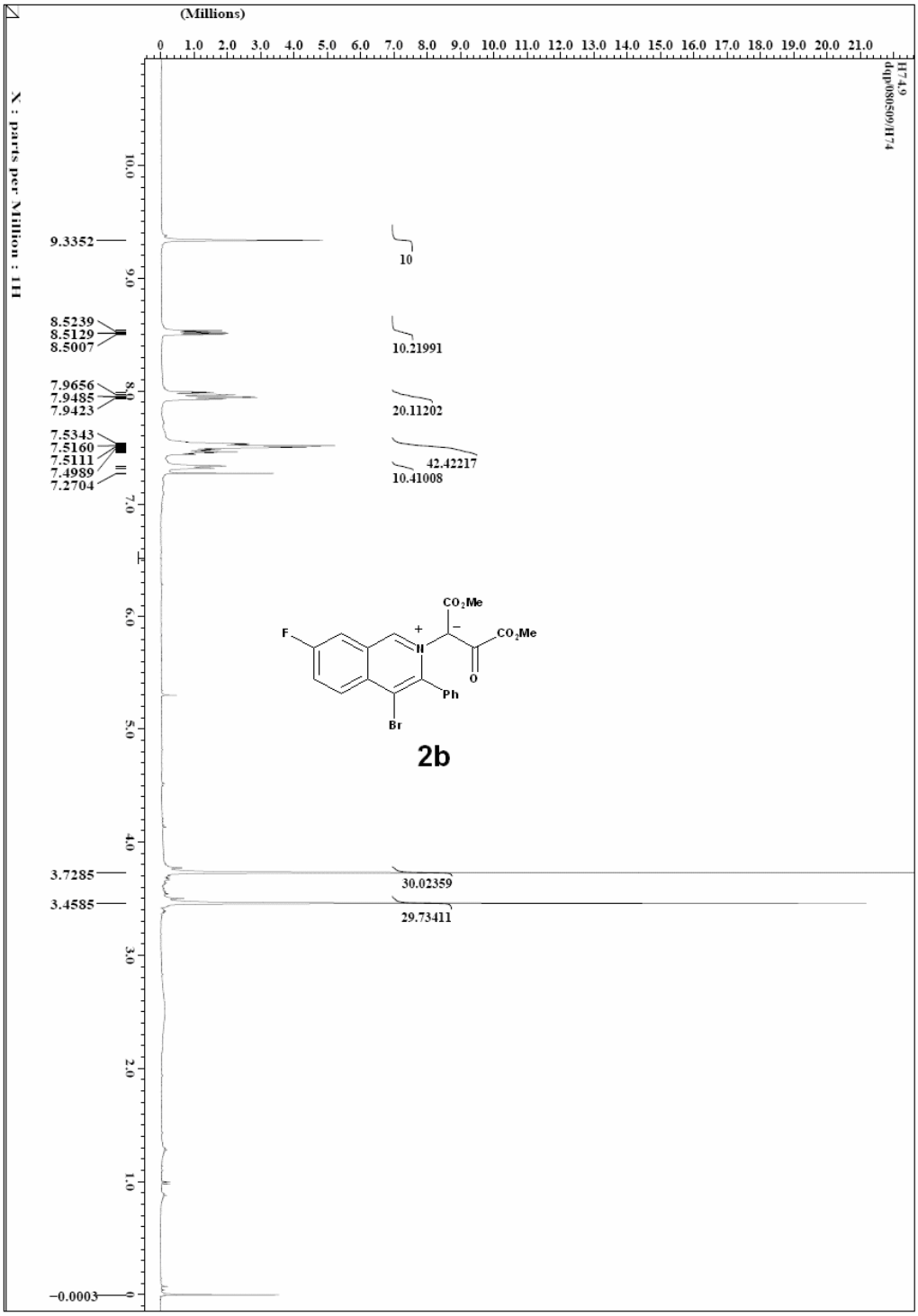
ddp080408\G160
9.3322
10
8.4757
8.4537
8.2900
8.2705
8.2277
8.2252
7.9711
9.99458
20.84559
10.671
7.5166
7.5142
7.4616
7.3505
7.2784
30.6508
11.8358
10.57347
3.7328
3.4616
32.14548
30.54617
0.0004
CO2Me
CO2Me
Ph
Br
2a


(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
X : parts per Million : 13C
G160 C.10
ddqp080408/G160 C
171.6591
167.9120
154.4015
150.0060
137.9066
132.6054
130.9845
130.6127
130.4029
128.9537
128.4197
127.6474
127.5330
77.3144
76.9997
76.6851
51.8380
50.4364
CO2Me
CO2Me
N
O
Ph
Br
2a


(Millions)
X : parts per Million : 1H
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dqfp080809H74
9.3352
8.5239
8.5129
8.5007
7.9656
7.9485
7.9423
7.5343
7.5160
7.5111
7.4989
7.2704
3.7285
3.4585
−0.0003
10
10.21991
20.11202
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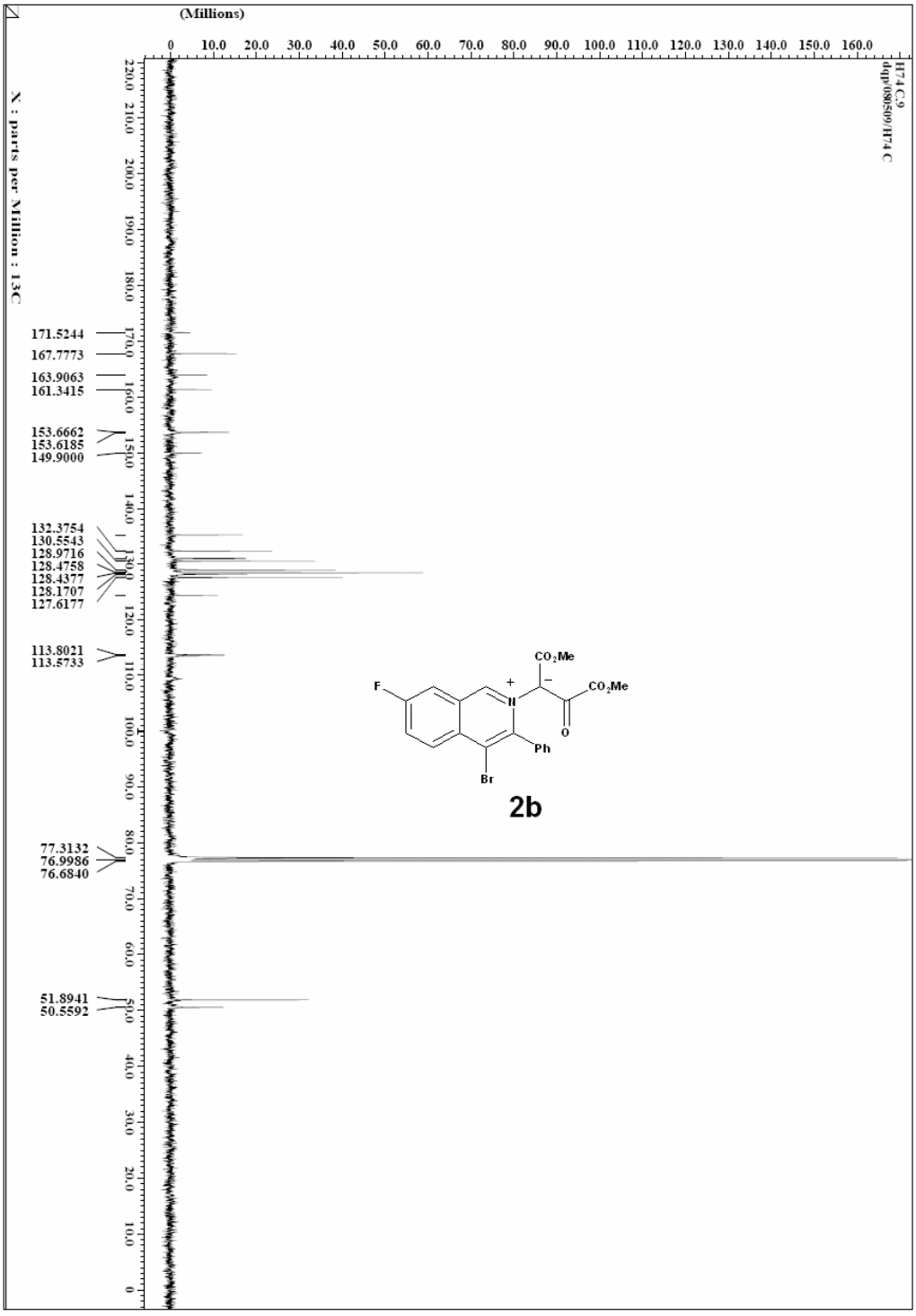
10.41008
30.02359
29.73411
CO2Me
CO2Me
F
N
O
Ph
Br

2b


(Millions)
X : parts per Million : 13C
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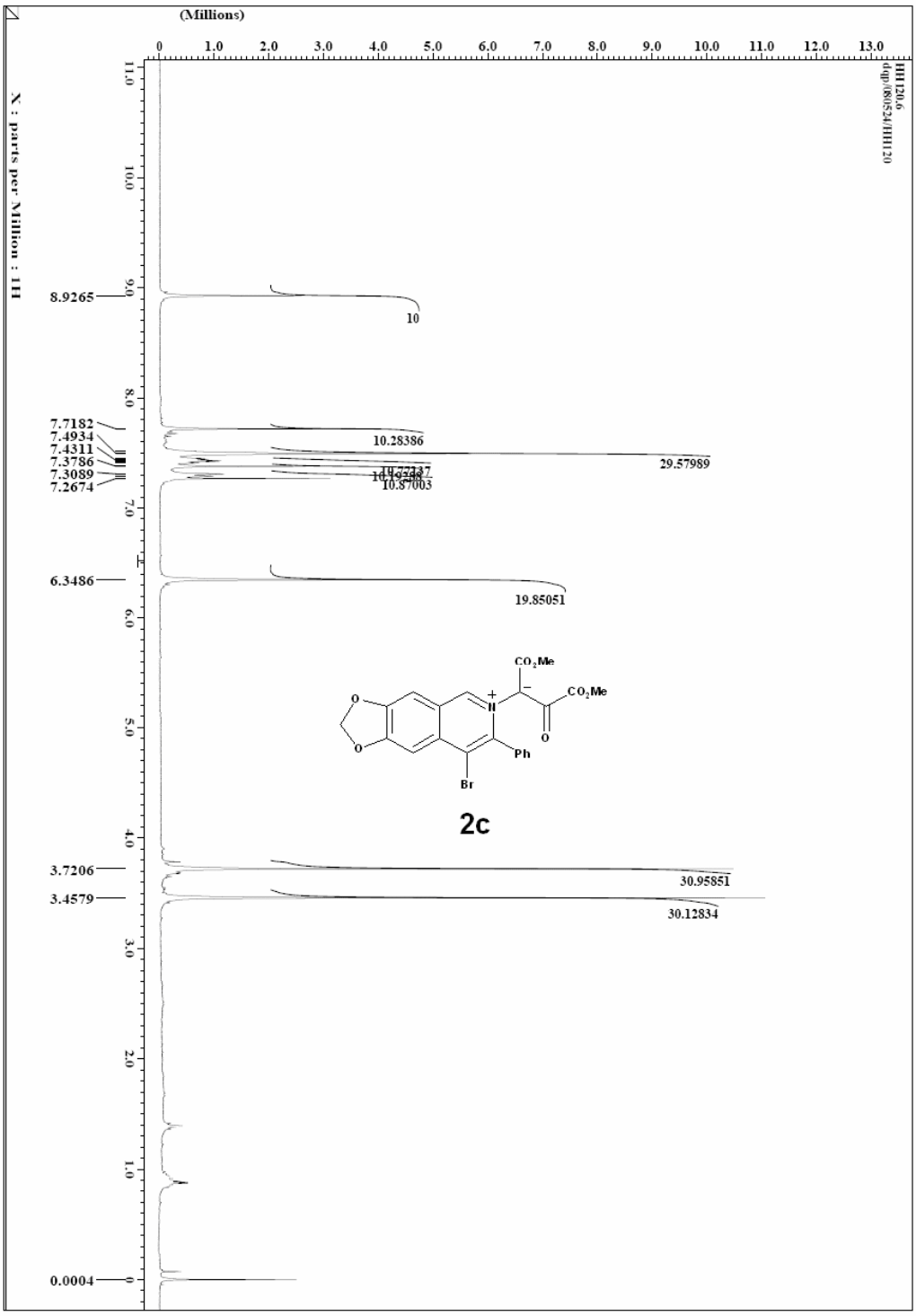
167.7773
163.9063
161.3415
153.6662
153.6185
149.9000
132.3754
130.5543
128.9716
128.4758
128.4377
128.1707
127.6177
113.8021
113.5733
77.3132
76.9986
76.6840
51.8941
50.5592
F
N
CO2Me
CO2Me
O
Ph
Br
2b


(Millions)
X : parts per Million : 1H
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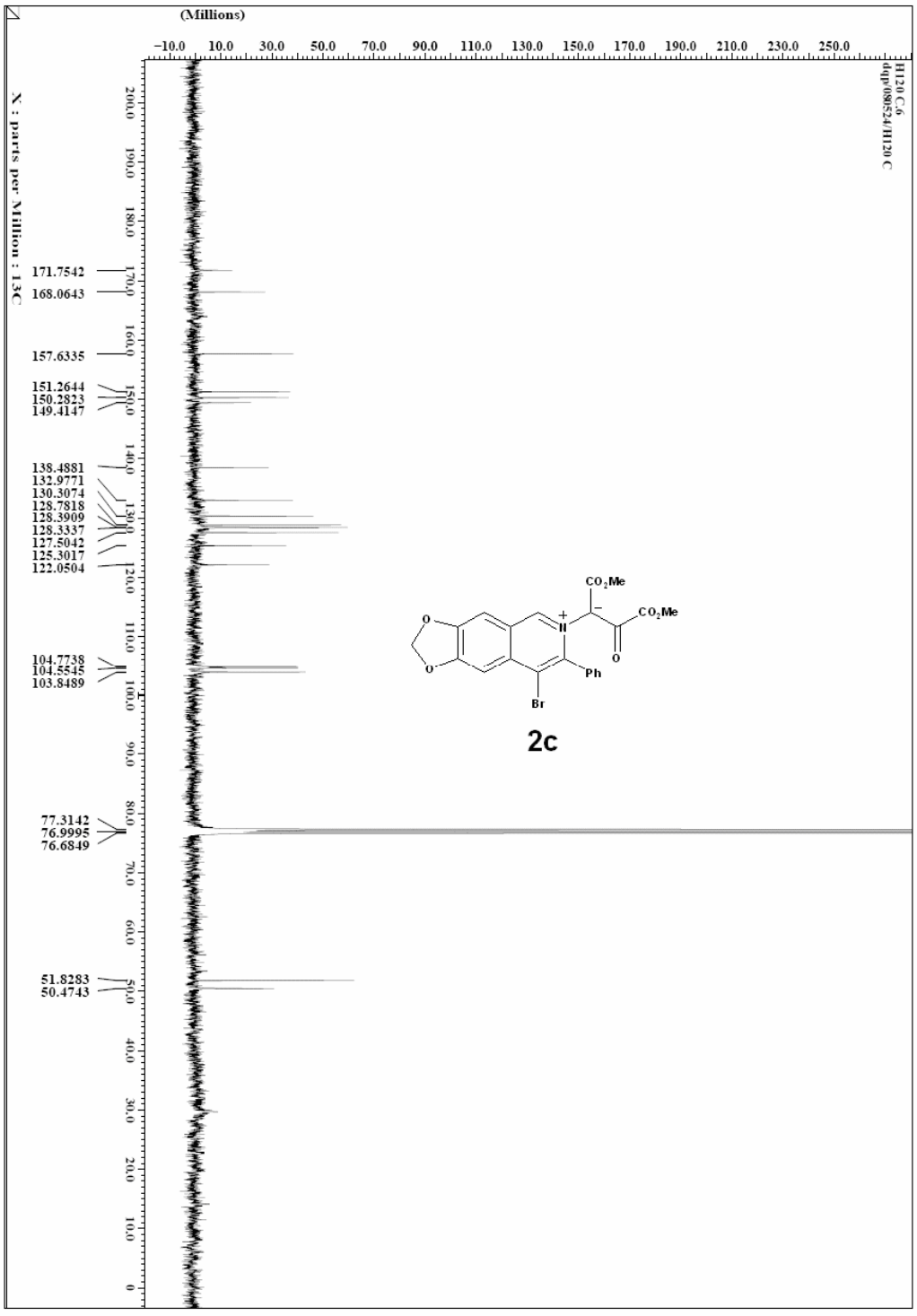
7.7182
7.4934
7.4311
7.3786
7.3089
7.2674
6.3486
3.7206
3.4579
0.0004
10
10.28386
29.57989
10.57003
19.85051
30.95851
30.12834
CO2Me
CO2Me
Ph
Br
2c


(Millions)
X : parts per Million : 13C
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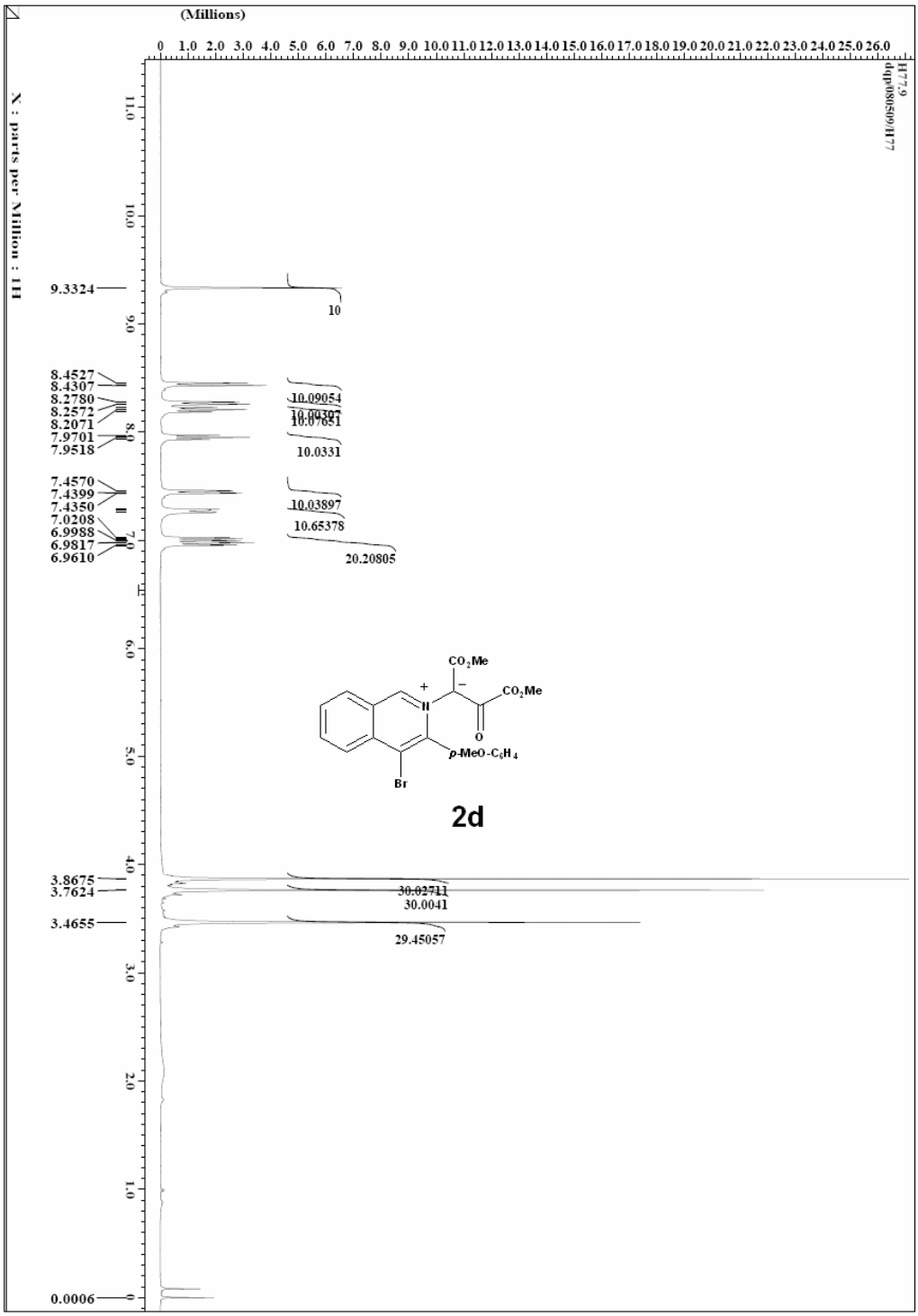
168.0643
157.6335
151.2644
150.2823
149.4147
138.4881
132.9771
130.3074
128.7818
128.3909
128.3337
127.5042
125.3017
122.0504
104.7738
104.5545
103.8489
77.3142
76.9995
76.6849
51.8283
50.4743
CO2Me
CO2Me
Ph
Br
2c


(Millions)
X : parts per Million : 1H
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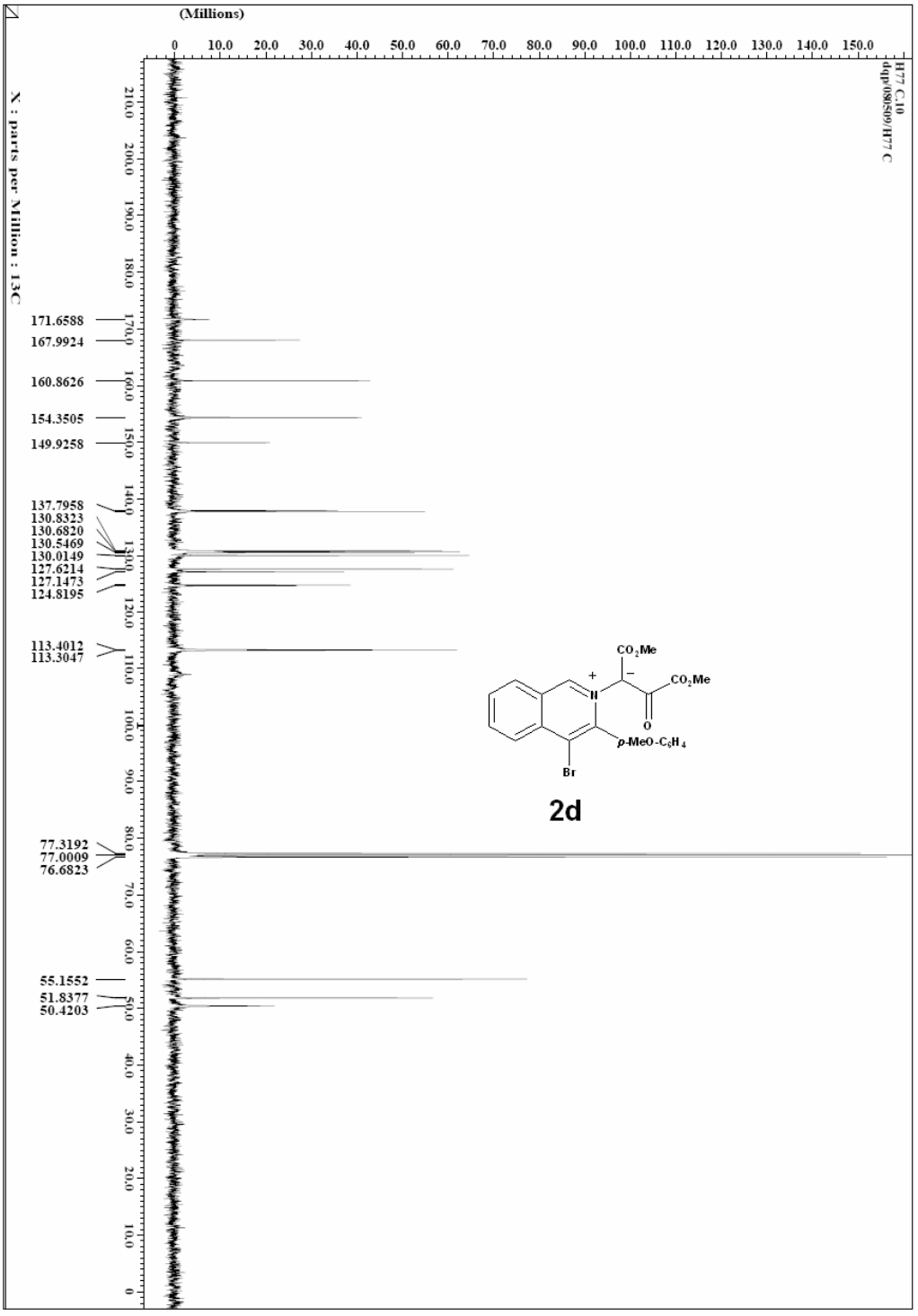
8.4307
8.2780
8.2572
8.2071
7.9701
7.9518
7.4570
7.4399
7.4350
7.0208
6.9988
6.9817
6.9610
3.8675
3.7624
3.4655
0.0006
10
10.09054
10.00397
10.07651
10.0331
10.03897
10.65378
20.20805
30.02711
30.0041
29.45057
CO2Me
CO2Me
p-MeO-C6H4
Br
2d


(Millions)
X : parts per Million : 13C
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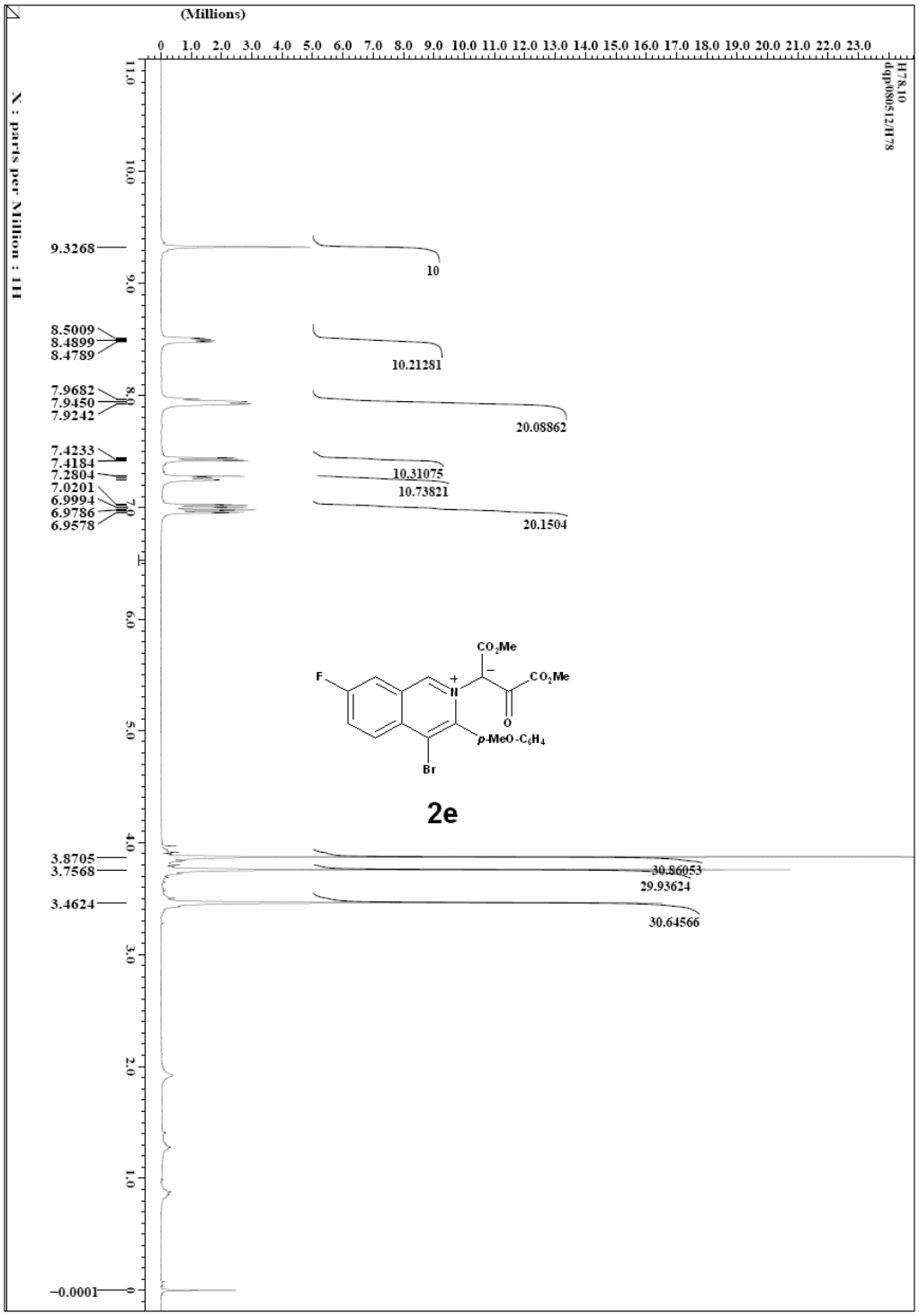
160.8626
154.3505
149.9258
137.7958
130.8323
130.6820
130.5469
130.0149
127.6214
127.1473
124.8195
113.4012
113.3047
77.3192
77.0009
76.6823
55.1552
51.8377
50.4203
CO2Me
CO2Me
p-MeO-C6H4
Br
2d


(Millions)
X : parts per Million : 1H
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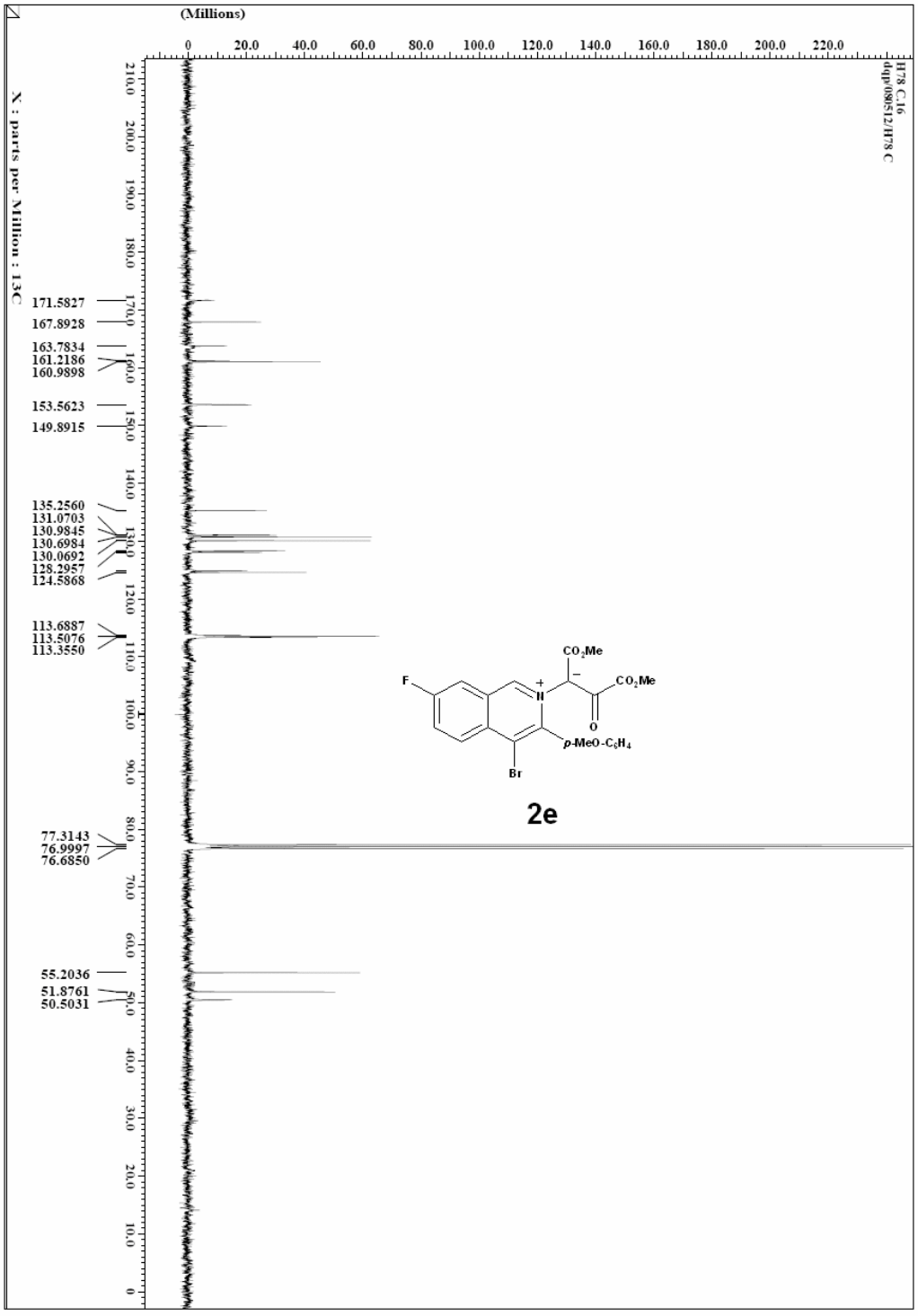
8.5009
8.4899
8.4789
7.9682
7.9450
7.9242
7.4233
7.4184
7.2804
7.0201
6.9994
6.9786
6.9578
3.8705
3.7568
3.4624
−0.0001
10
10.21281
20.08862
10.31075
10.73821
20.1504
30.86053
29.93624
30.64566
F
N
CO2Me
CO2Me
O
p-MeO-C6H4
Br
2e


(Millions)
X : parts per Million : 13C
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dqp/080512/H78 C
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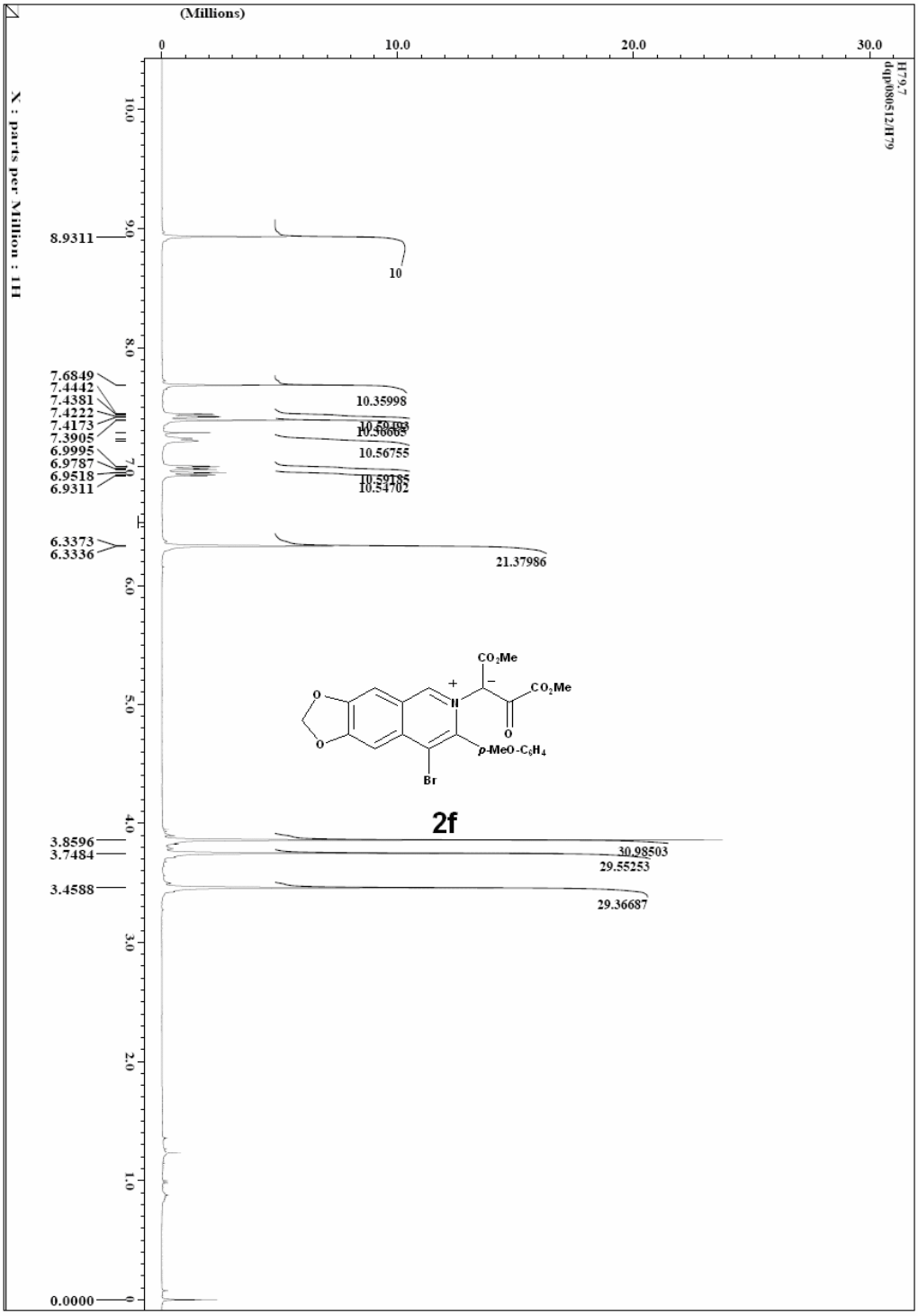
163.7834
161.2186
160.9898
153.5623
149.8915
135.2560
131.0703
130.9845
130.6984
130.0692
128.2957
124.5868
113.6887
113.5076
113.3550
77.3143
76.9997
76.6850
55.2036
51.8761
50.5031
CO2Me
CO2Me
F
N
O
p-MeO-C6H4
Br
2e


(Millions)
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30.0
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ddpt080512/H79
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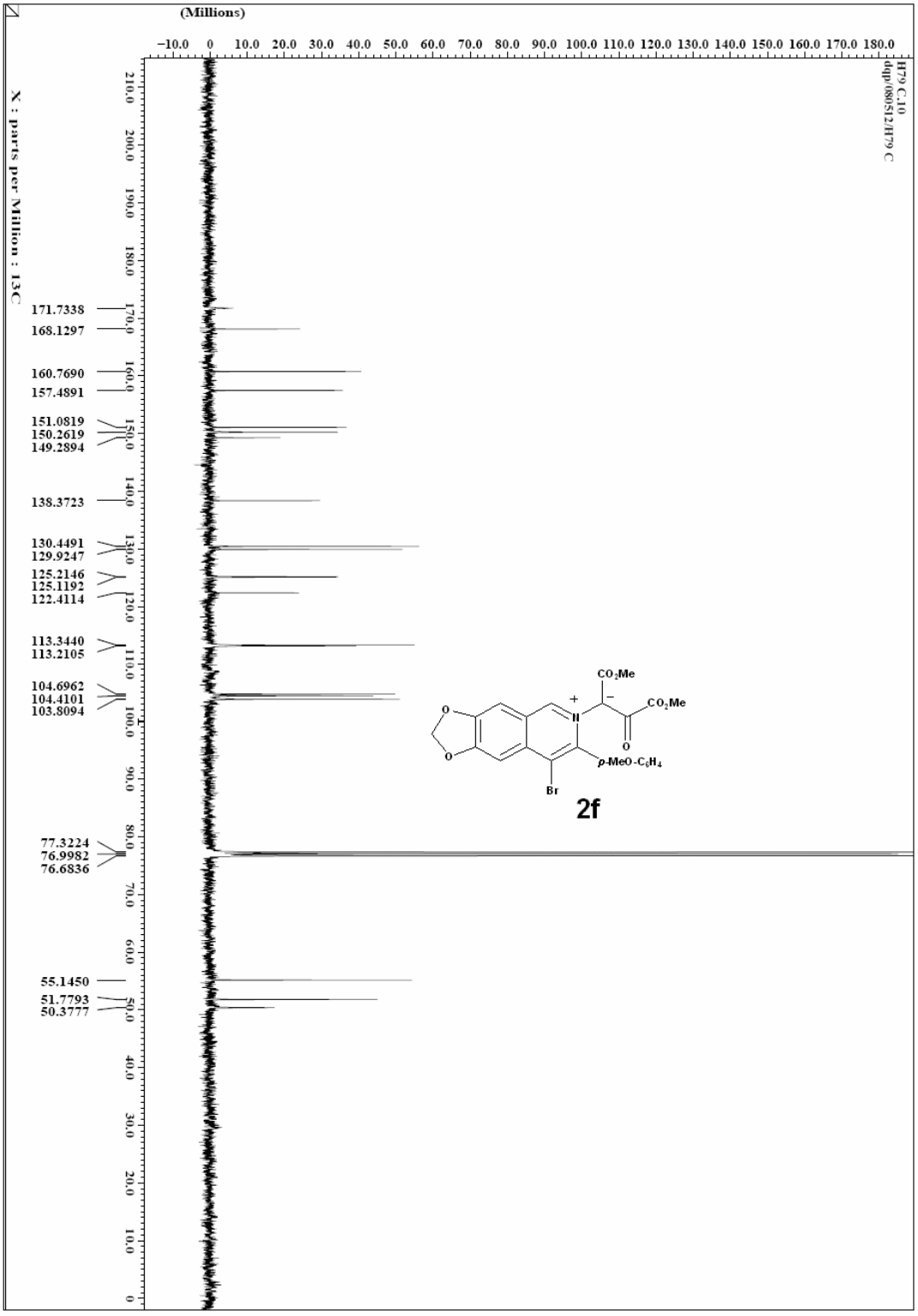
7.6849
7.4442
7.4381
7.4222
7.4173
7.3905
6.9995
6.9787
6.9518
6.9311
6.3373
6.3336
3.8596
3.7484
3.4588
0.0000
10
10.35998
10.56755
10.59185
10.54702
21.37986
30.98503
29.55253
29.36687
CO2Me
CO2Me
p-MeO-C6H4
Br
2f


(Millions)
X : parts per Million : 13C
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dqp/180512/H79 C
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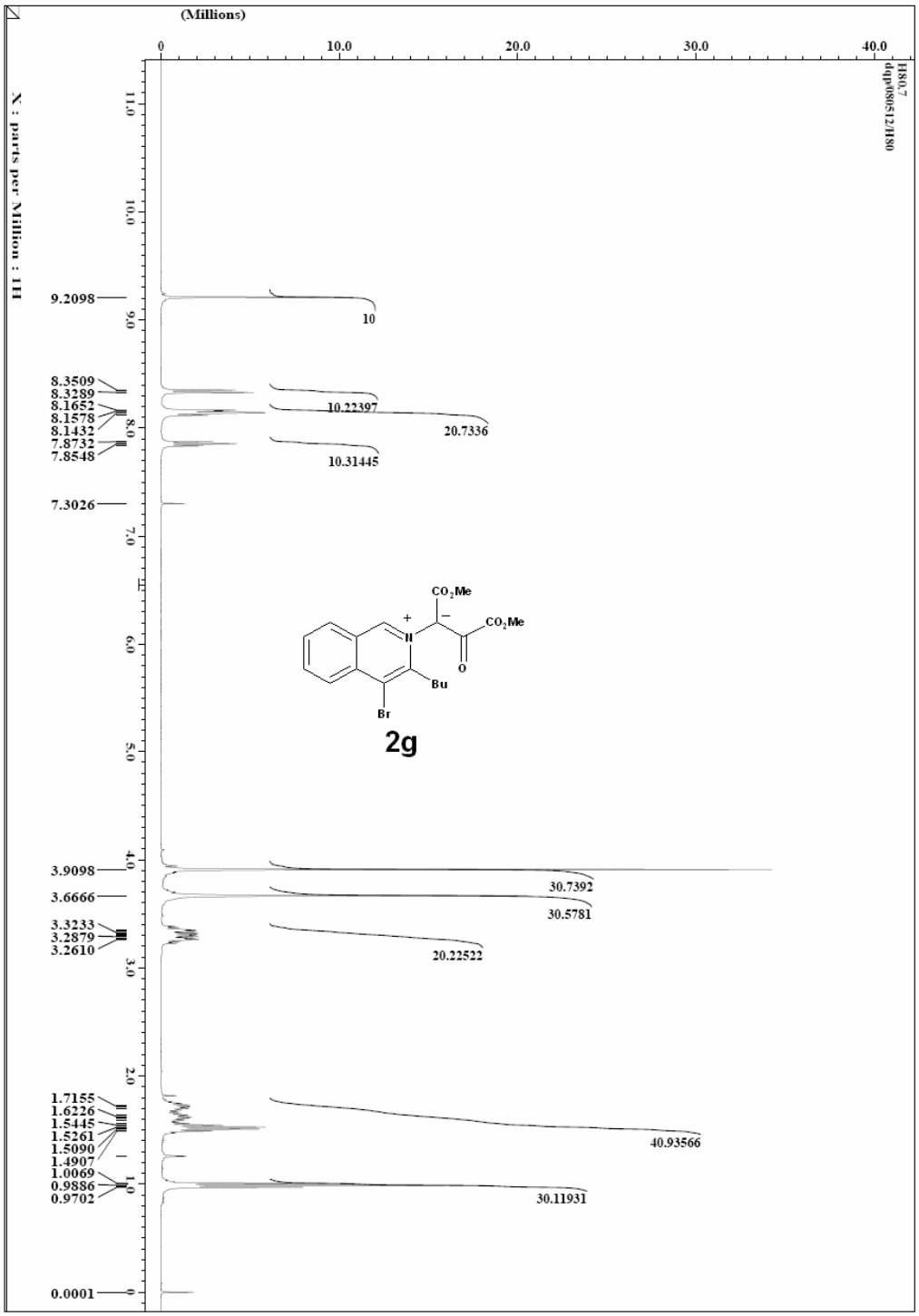
168.1297
160.7690
157.4891
151.0819
150.2619
149.2894
138.3723
130.4491
129.9247
125.2146
125.1192
122.4114
113.3440
113.2105
104.6962
104.4101
103.8094
77.3224
76.9982
76.6836
55.1450
51.7793
50.3777
CO2Me
CO2Me
p-MeO-C6H4
Br
2f


(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
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d4p080512/HS0
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10
8.3509
8.3289
8.1652
8.1578
8.1432
7.8732
7.8548
10.22397
20.7336
10.31445
7.3026
CO2Me
CO2Me
Bu
Br
2g
3.9098
3.6666
3.3233
3.2879
3.2610
30.7392
30.5781
20.22522
1.7155
1.6226
1.5445
1.5261
1.5090
1.4907
1.0069
0.9886
0.9702
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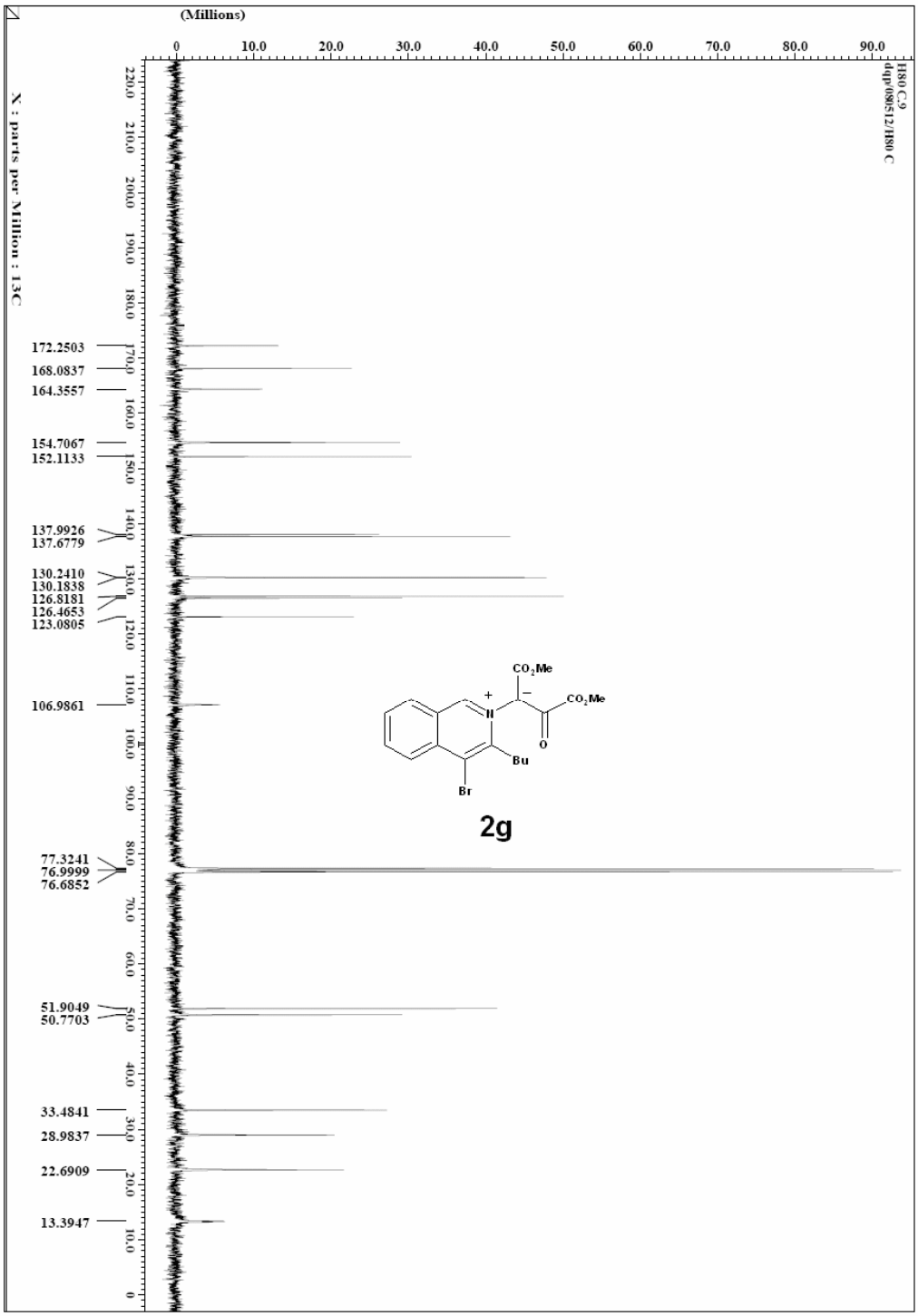
30.11931
0.0001


(Millions)
X : parts per Million : 13C
H80.C.9
dqp080512/H80 C
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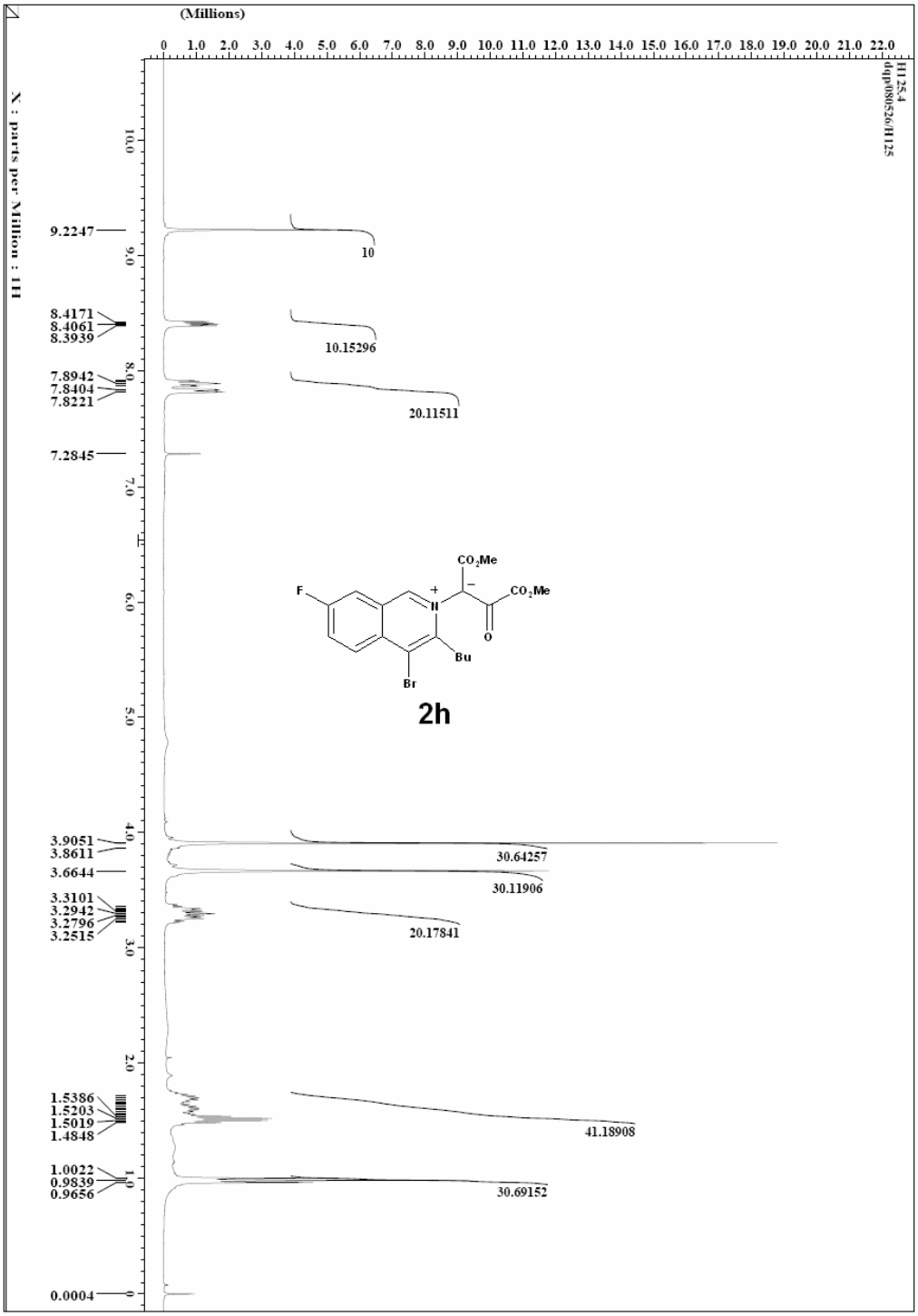
168.0837
164.3557
154.7067
152.1133
137.9926
137.6779
130.2410
130.1838
126.8181
126.4653
123.0805
106.9861
77.3241
76.9999
76.6852
51.9049
50.7703
33.4841
28.9837
22.6909
13.3947
CO2Me
CO2Me
Bu
Br
O
2g


(Millions)
X : parts per Million : 111
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8.3939
7.8942
7.8404
7.8221
7.2845
3.9051
3.8611
3.6644
3.3101
3.2942
3.2796
3.2515
1.5386
1.5203
1.5019
1.4848
1.0022
0.9839
0.9656
0.0004
10
10.15296
20.11511
30.64257
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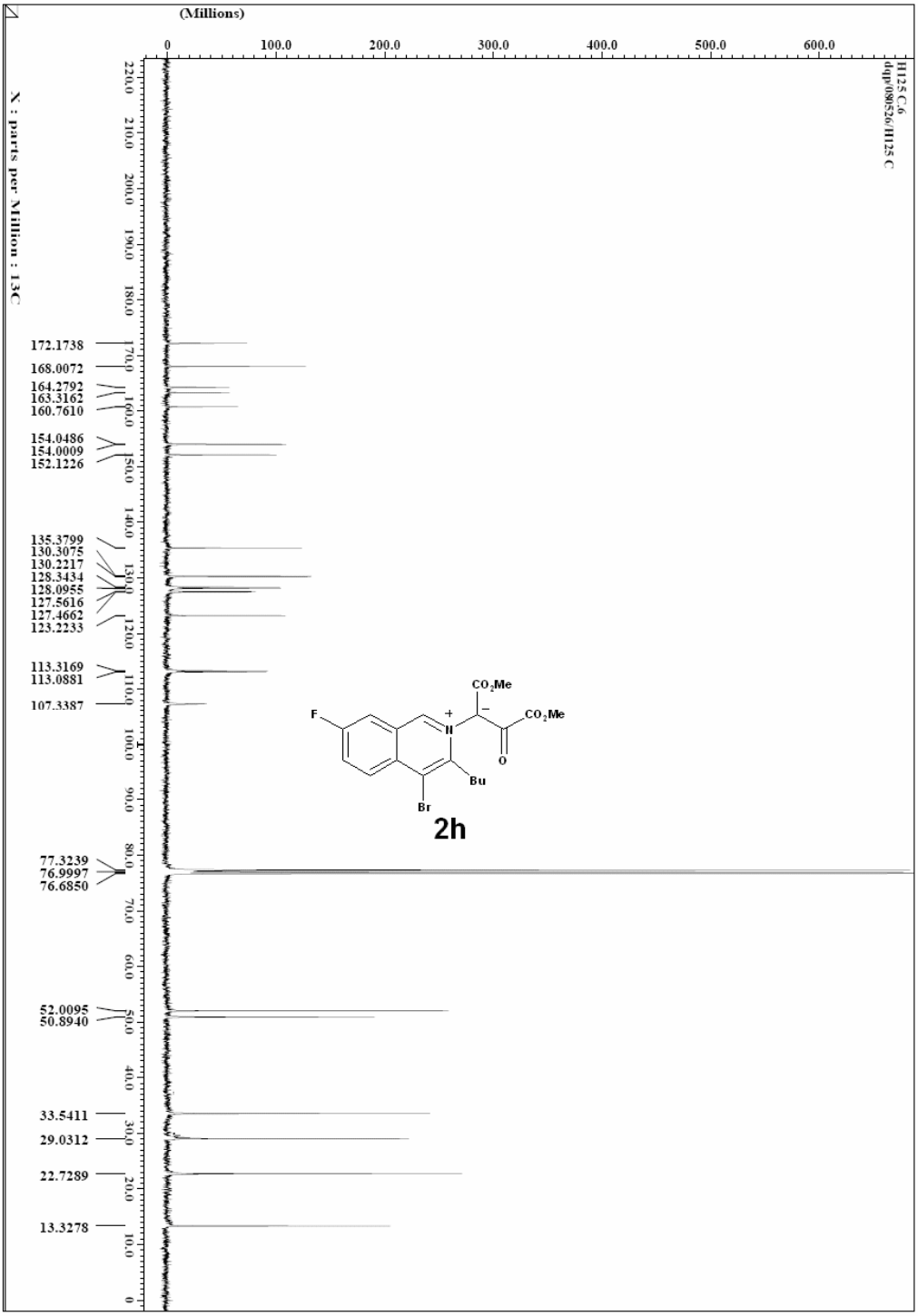
20.17841
41.18908
30.69152
F
CO2Me
CO2Me
N
O
Bu
Br
2h


(Millions)
X : parts per Million : 13C
H125 C 6
dqp090526 H125 C
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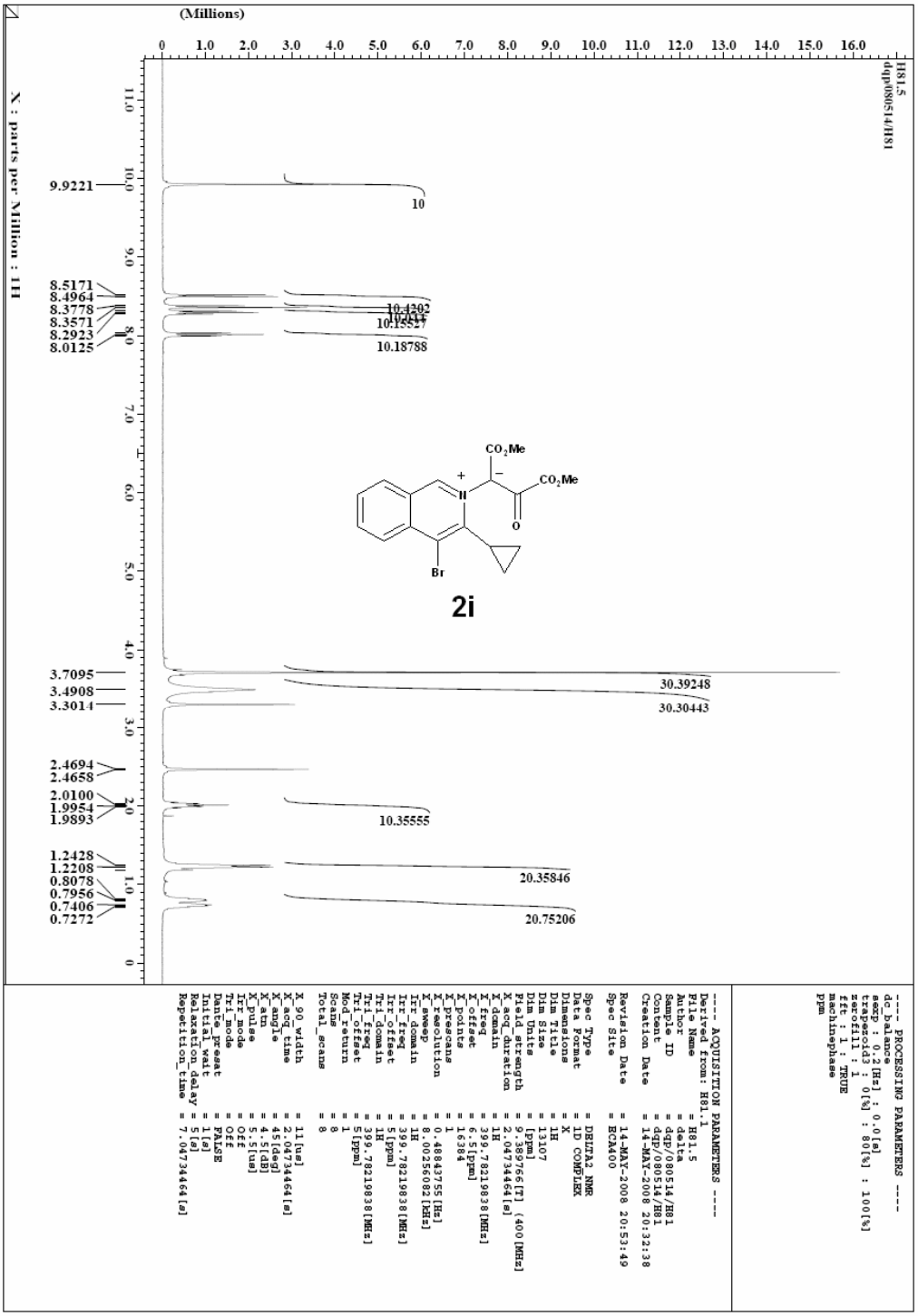
168.0072
164.2792
163.3162
160.7610
154.0486
154.0009
152.1226
135.3799
130.3075
130.2217
128.3434
128.0955
127.5616
127.4662
123.2233
113.3169
113.0881
107.3387
77.3239
76.9997
76.6850
52.0095
50.8940
33.5411
29.0312
22.7289
13.3278
F
CO2Me
CO2Me
N
O
Bu
Br
2h


(Millions)
X : parts per Million : 1H
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8.5171
8.4964
8.3778
8.3571
8.2923
8.0125
3.7095
3.4908
3.3014
2.4694
2.4658
2.0100
1.9954
1.9893
1.2428
1.2208
0.8078
0.7956
0.7406
0.7272
10
10.4202
10.4044
10.15527
10.18788
30.39248
30.30443
10.35555
20.35846
20.75206
CO2Me
CO2Me
N
O
Br
2i
H81.5
dqp/080514/H81
---- PROCESSING PARAMETERS ----
dc_balance : 0[s]
exp_mod : 0.3[Hz] ; 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
---- ACQUISITION PARAMETERS ----
Derived from: H81.1
File Name = H81.5
Author = delta
Sample_ID = dqp/080514/H81
Content = dqp/080514/H81
Creation_Date = 14-MAY-2008 20:32:38
Revision_Date = 14-MAY-2008 20:53:49
Spec_Site = HCA400
Spec_Type = DELTA2_NMR
Data_Format = 1D COMPLEX
Dimensions = X
Dim_Title = 1H
Dim_Size = 13107
Dim_Units = [ppm]
Field_strength = 9.397766[T] (400[MHz])
X_Acq_duration = 2.047344641[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 6.5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.48843755[Hz]
X_sweep = 8.002560821[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Mod_Preturn = 1
Scans = 8
Total_scans = 8
X_90_width = 11[us]
X_acq_time = 2.047344641[s]
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X_atn = 5.5[dB]
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Initial_wait = 1[s]
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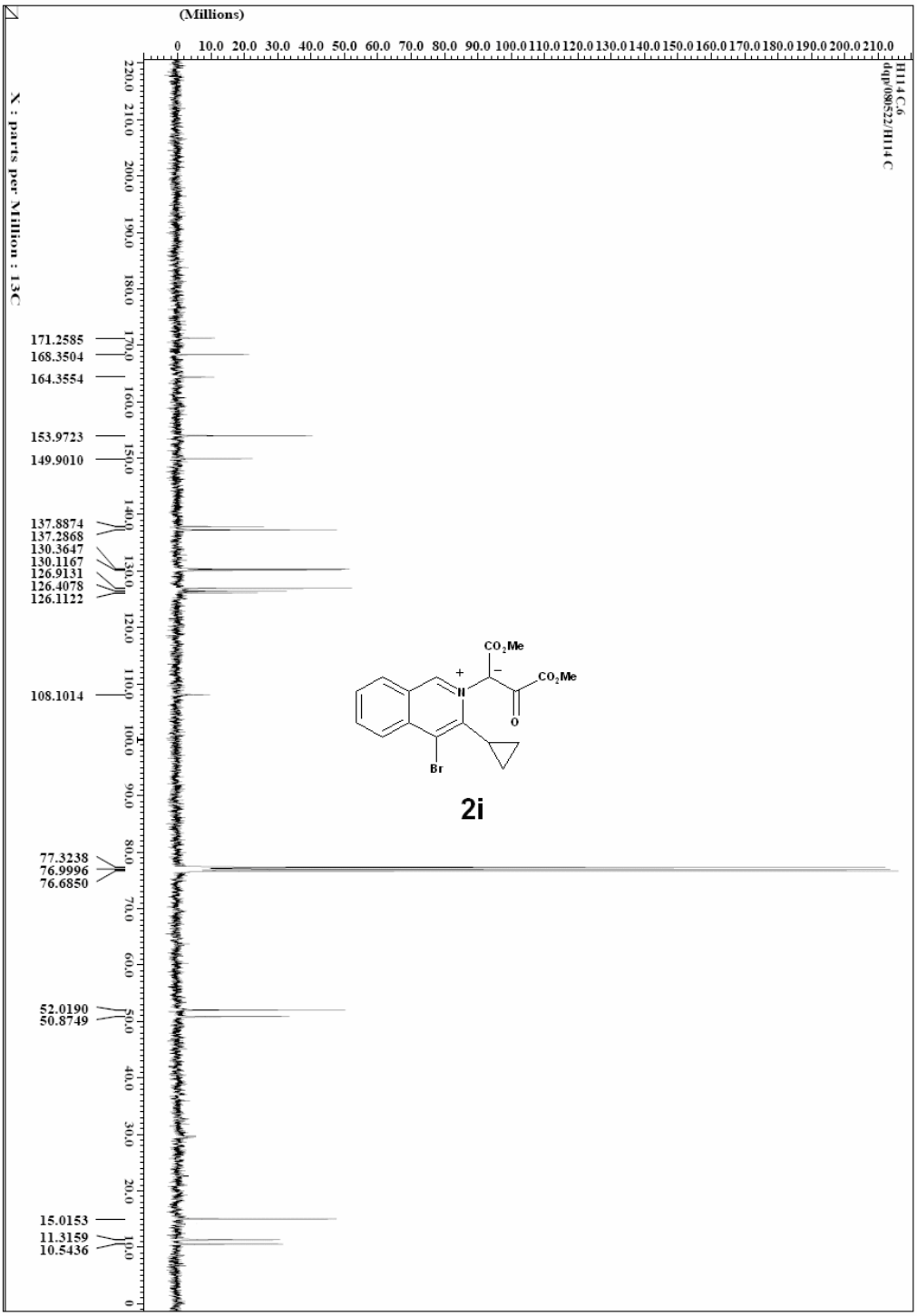
(Millions)
X : parts per Million : 13C
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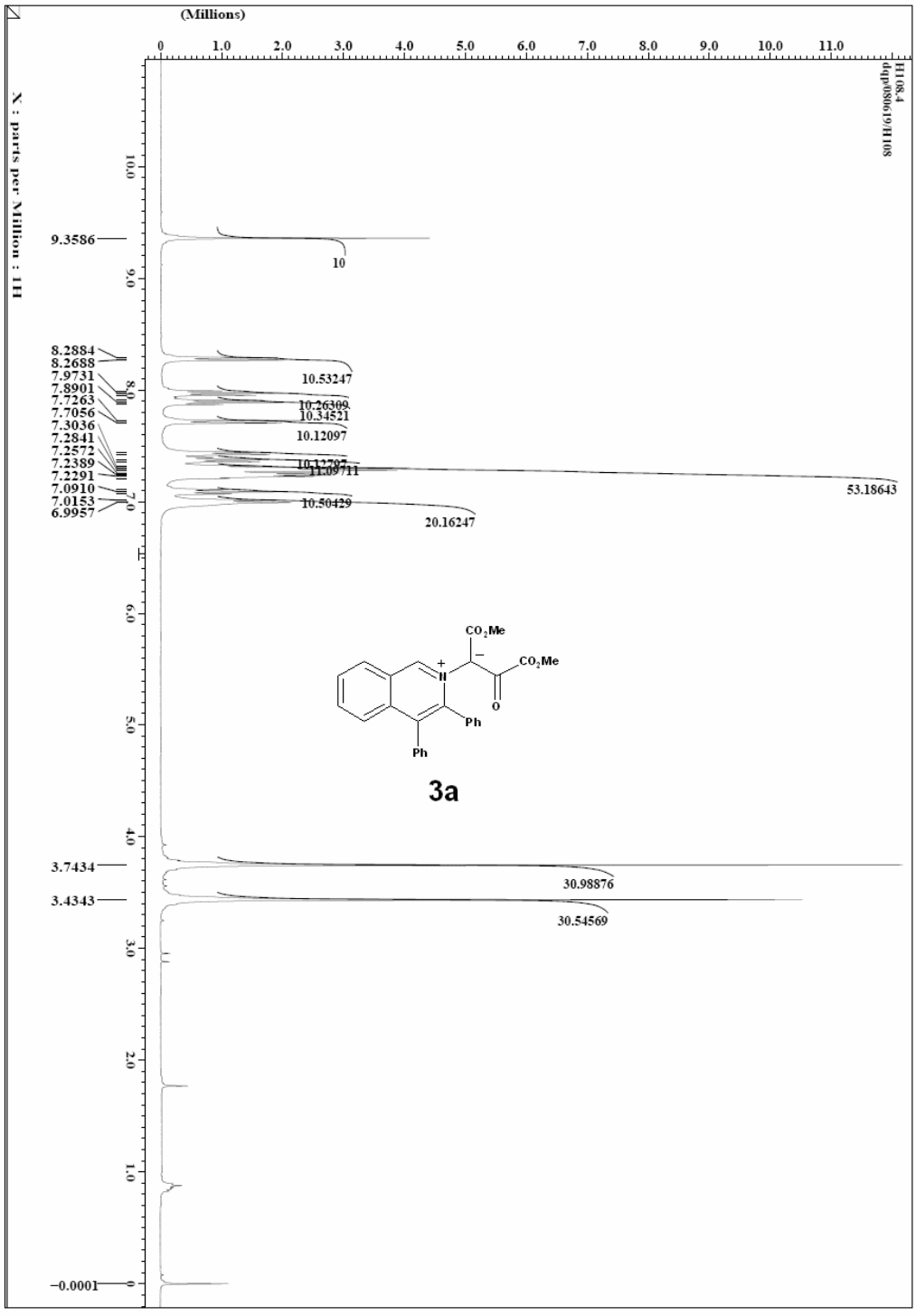
dqp090522/H114 C
171.2585
168.3504
164.3554
153.9723
149.9010
137.8874
137.2868
130.3647
130.1167
126.9131
126.4078
126.1122
108.1014
77.3238
76.9996
76.6850
52.0190
50.8749
15.0153
11.3159
10.5436
CO2Me
CO2Me
O
Br
2i


(Millions)
X : parts per Million : 1H1
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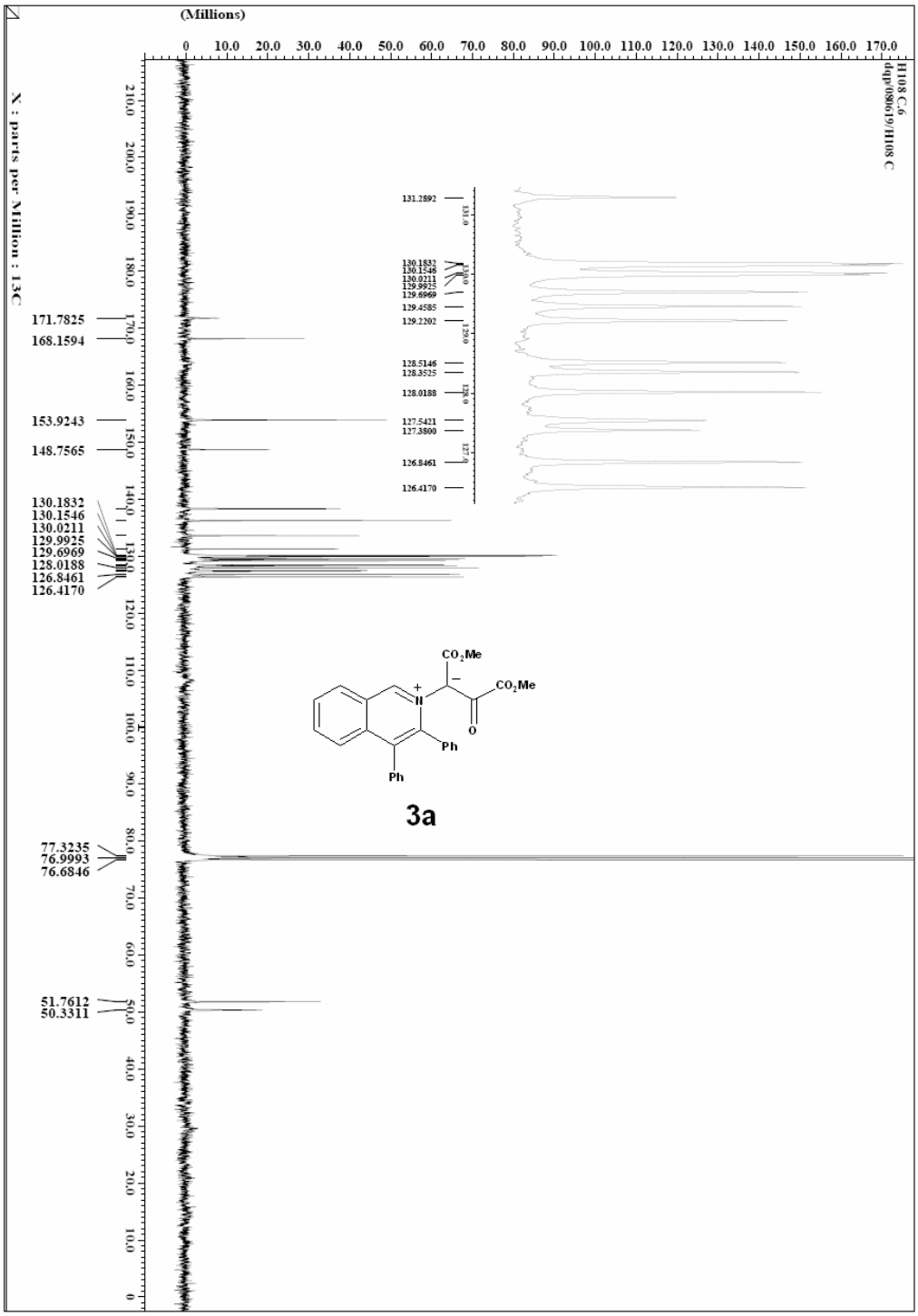
10
8.2884
8.2688
7.9731
7.8901
7.7263
7.7056
7.3036
7.2841
7.2572
7.2389
7.2291
7.0910
7.0153
6.9957
10.53247
10.26309
10.34521
10.12097
10.13797
11.09711
10.50429
20.16247
53.18643
3.7434
3.4343
30.98876
30.54569
−0.0001
CO2Me
CO2Me
O
Ph
Ph
3a


(Millions)
X : parts per Million : 13C
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dqp090619/H108 C
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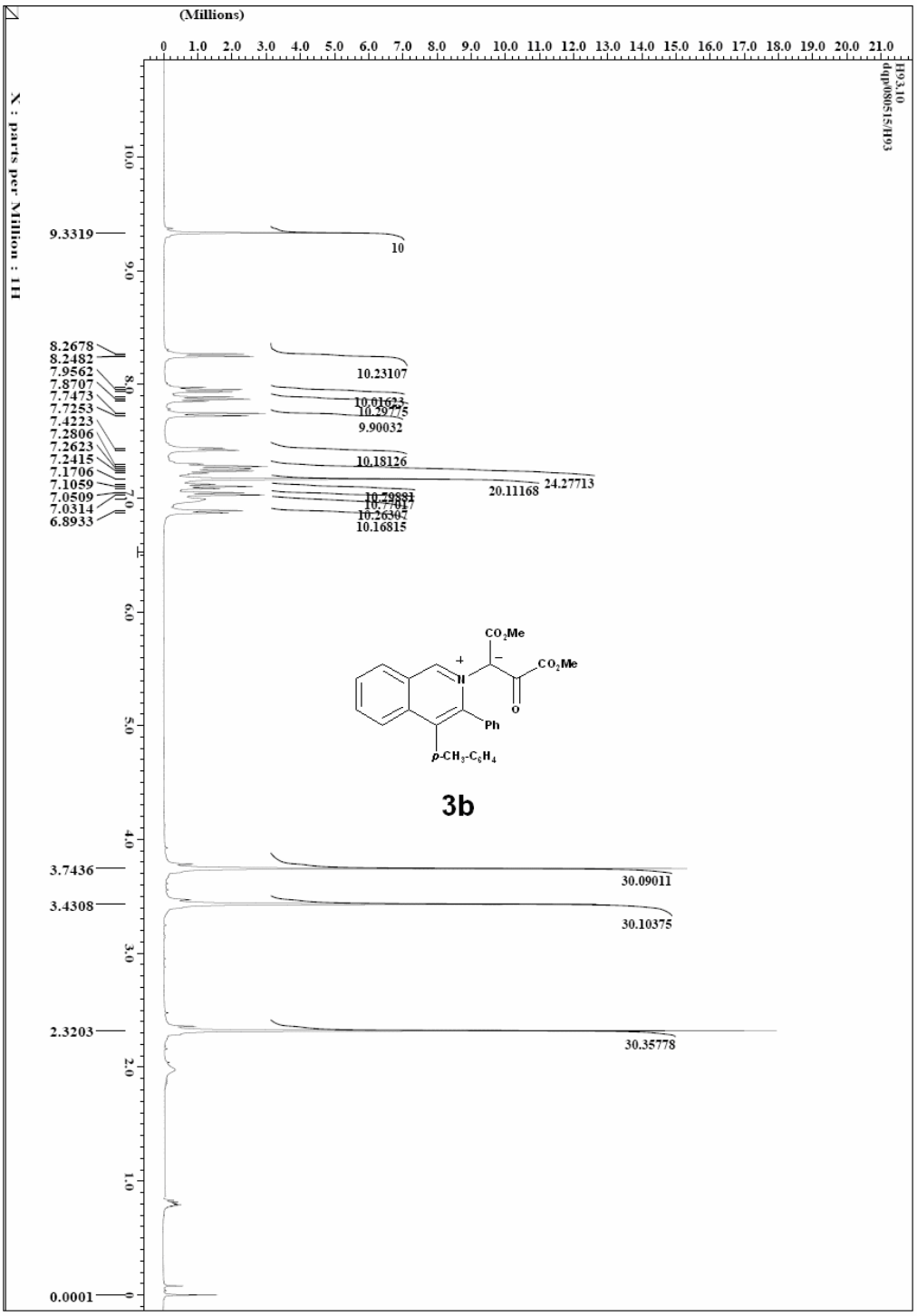
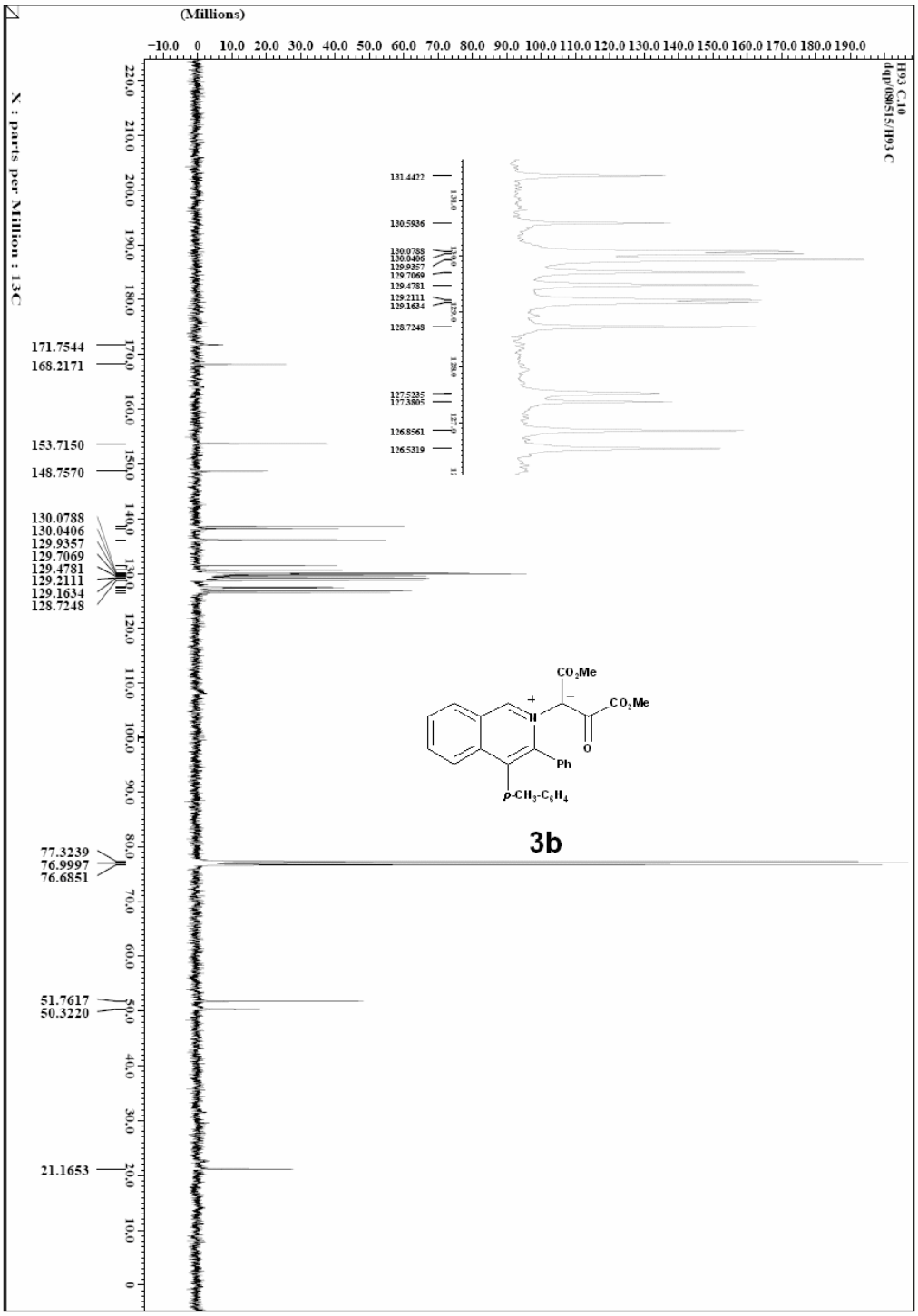
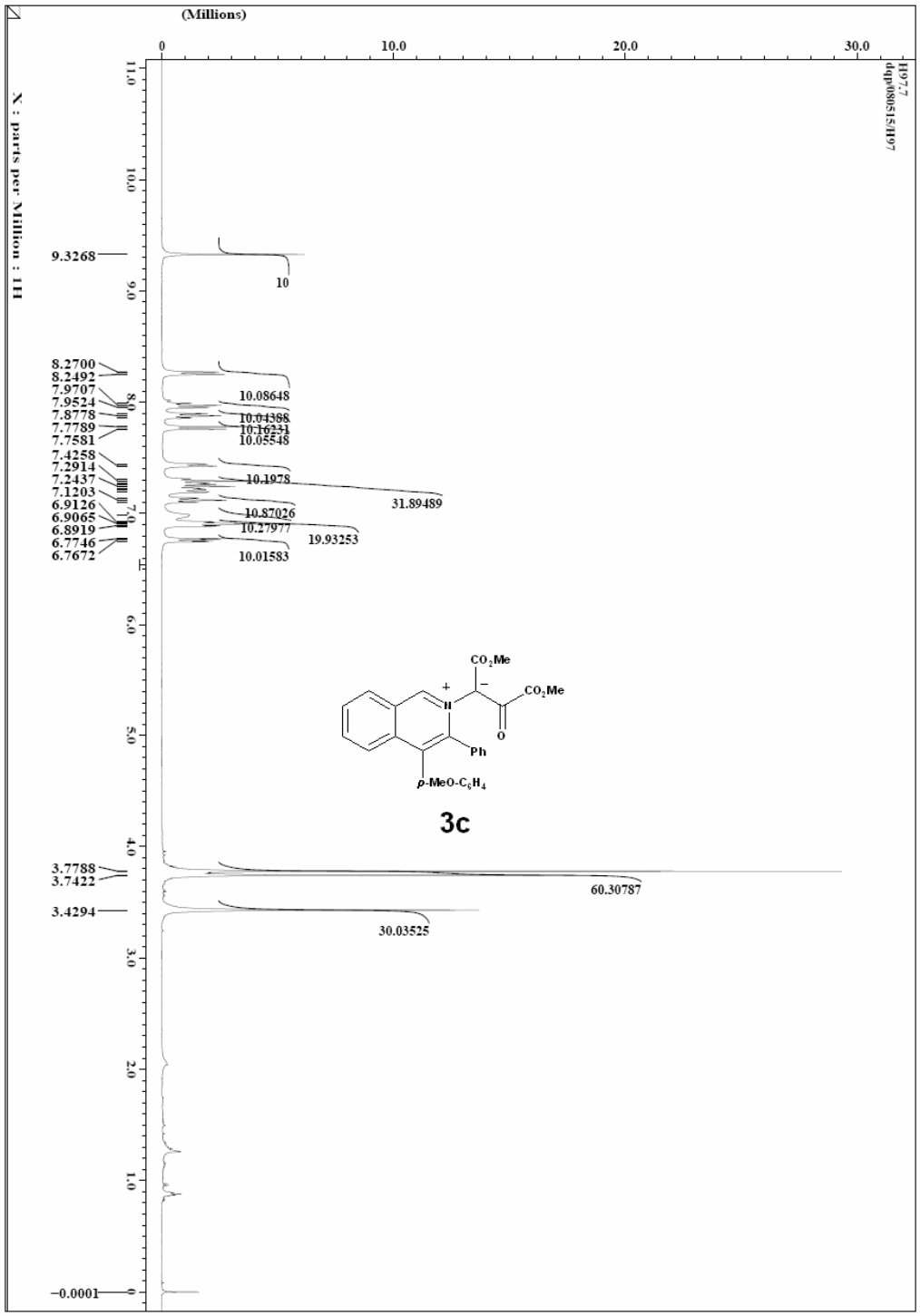
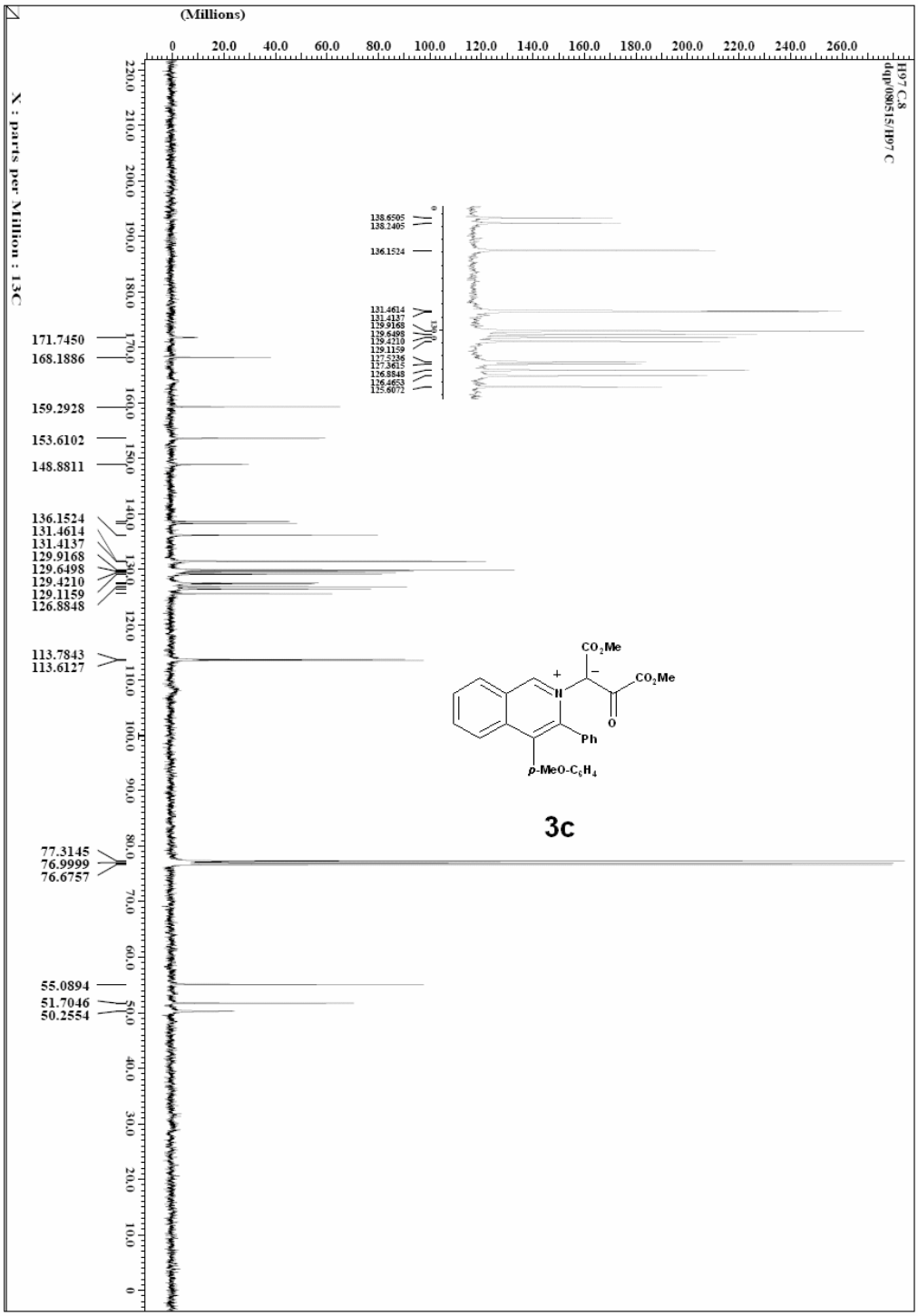
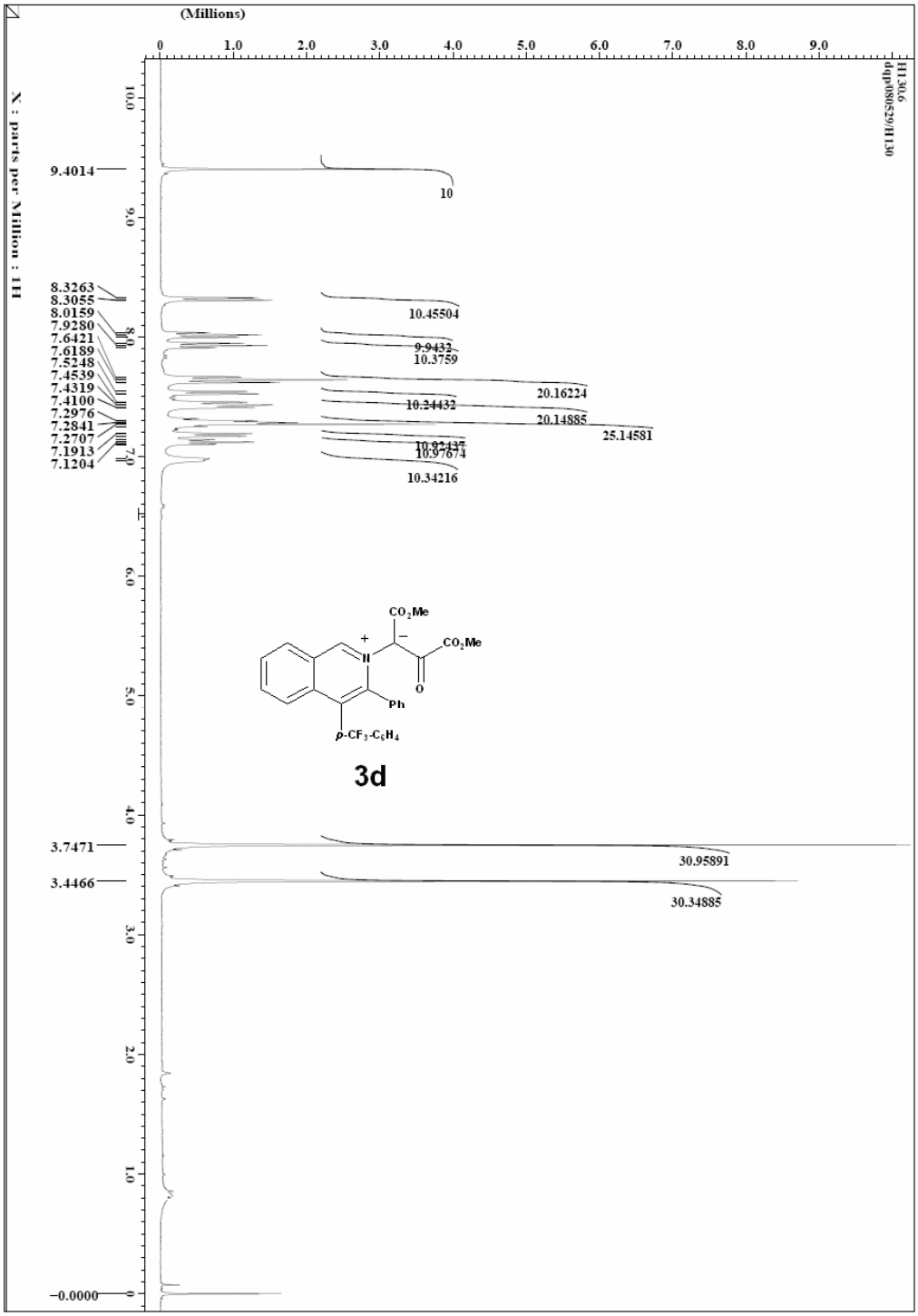
168.1594
153.9243
148.7565
130.1832
130.1546
130.0211
129.9925
129.6969
128.0188
126.8461
126.4170
77.3235
76.9993
76.6846
51.7612
50.3311
131.2892
130.1832
130.1546
130.0211
129.9925
129.6969
129.4585
129.2202
128.5146
128.3525
128.0188
127.5421
127.3800
126.8461
126.4170
CO2Me
CO2Me
O
Ph
Ph
3a

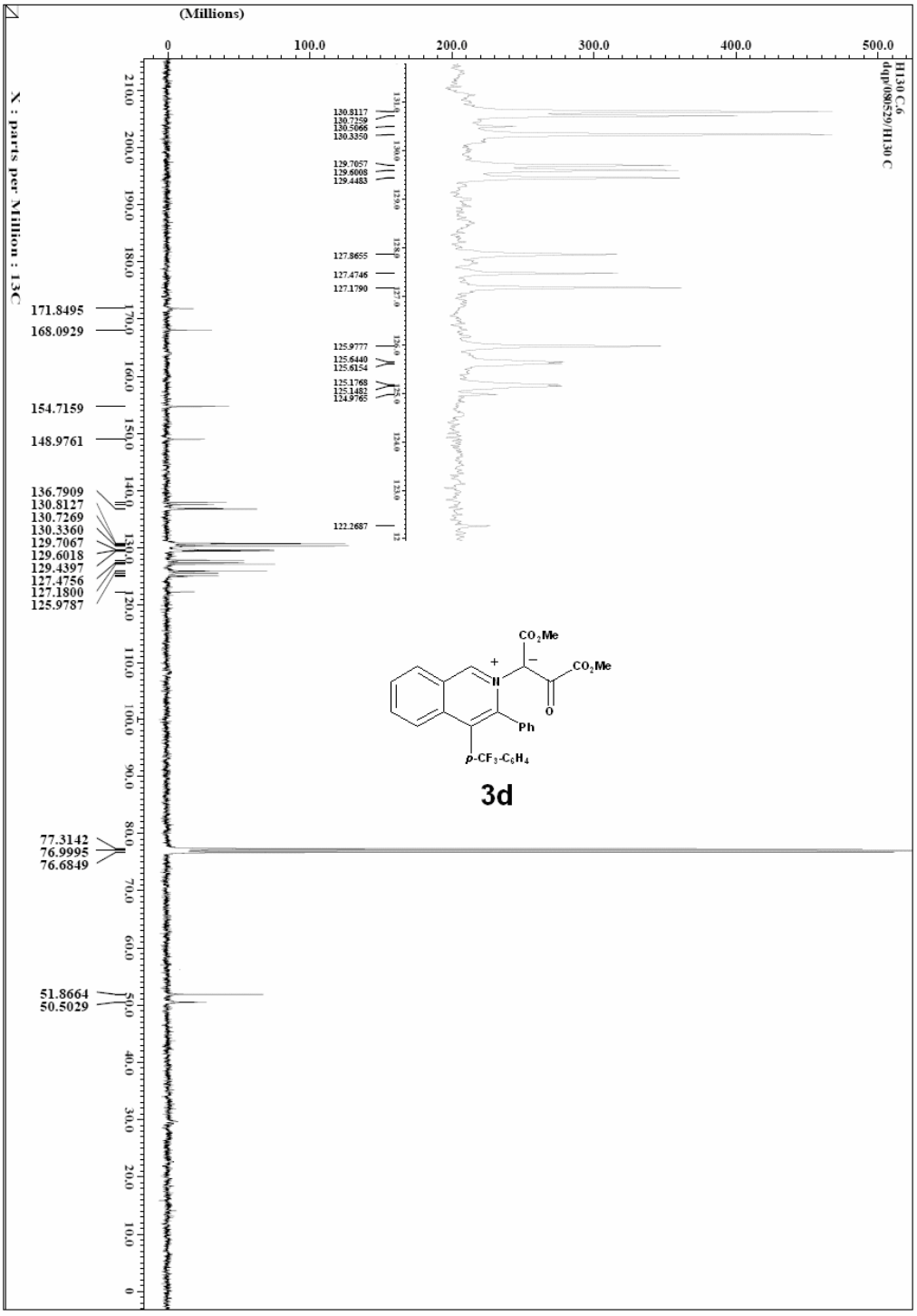

(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0
H93.10
dqp080515/H93
X : parts per Million : 1H
9.3319
8.2678
8.2482
7.9562
7.8707
7.7473
7.7253
7.4223
7.2806
7.2623
7.2415
7.1706
7.1059
7.0509
7.0314
6.8933
3.7436
3.4308
2.3203
0.0001
10
10.23107
10.01623
10.29775
9.90032
10.18126
20.11168
24.27713
10.79881
10.77017
10.26307
10.16815
30.09011
30.10375
30.35778
CO2Me
CO2Me
Ph
p-CH3-C6H4
3b

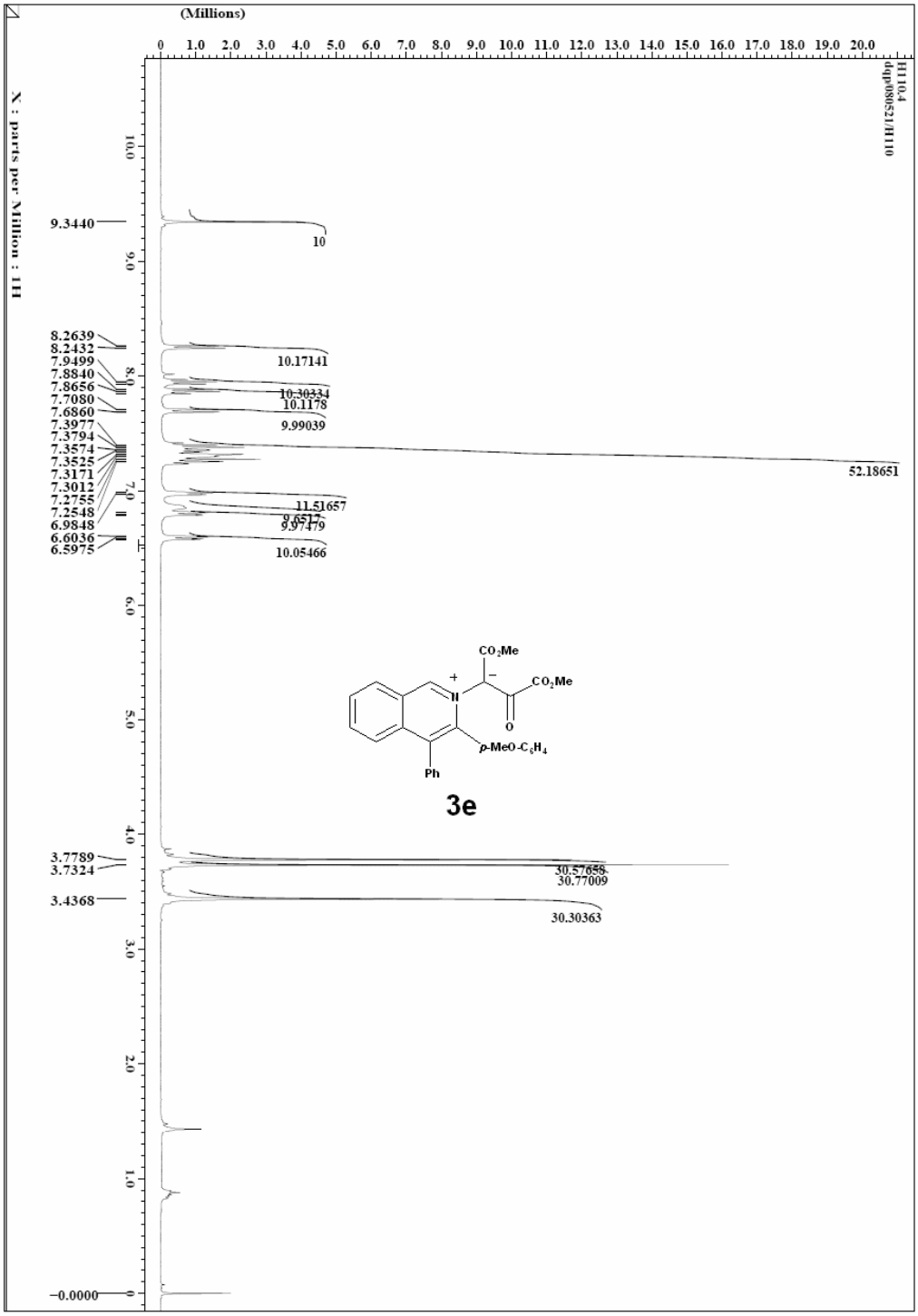

(Millions)
X : parts per Million : 13C
H93 C-10
dqp080515/H93 C
171.7544
168.2171
153.7150
148.7570
130.0788
130.0406
129.9357
129.7069
129.4781
129.2111
129.1634
128.7248
77.3239
76.9997
76.6851
51.7617
50.3220
21.1653
131.4422
130.5936
127.5285
127.3805
126.8561
126.5319
CO2Me
CO2Me
O
Ph
p-CH3-C6H4
3b

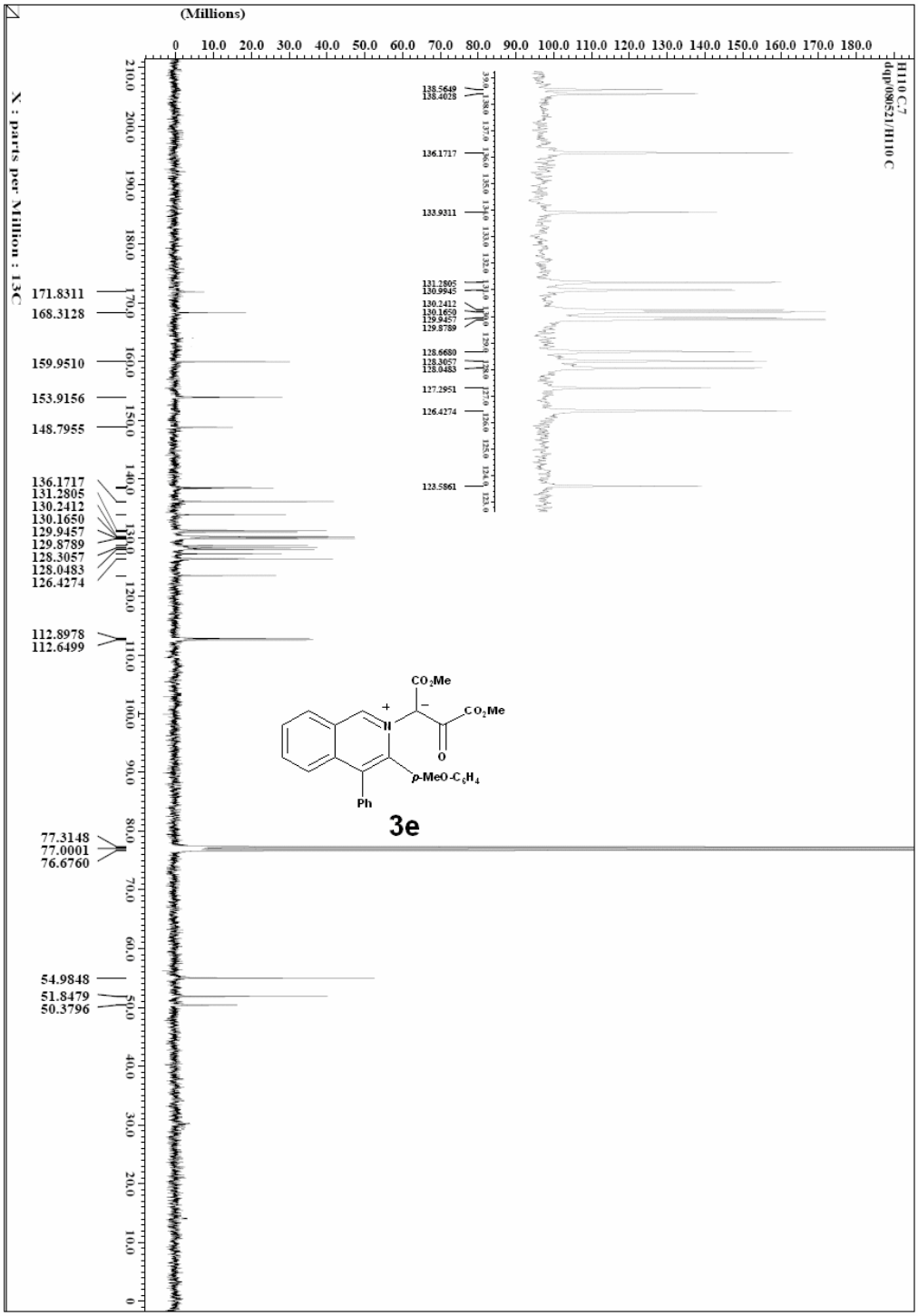

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
H97-7
dnp080515H97
9.3268
10
8.2700
8.2492
7.9707
7.9524
7.8778
7.7789
7.7581
7.4258
7.2914
7.2437
7.1203
6.9126
6.9065
6.8919
6.7746
6.7672
10.08648
10.04386
10.16231
10.05548
10.1978
31.89489
10.87026
10.27977
19.93253
10.01583
3.7788
3.7422
3.4294
60.30787
30.03525
−0.0001
CO2Me
CO2Me
Ph
p-MeO-C6H4
3c

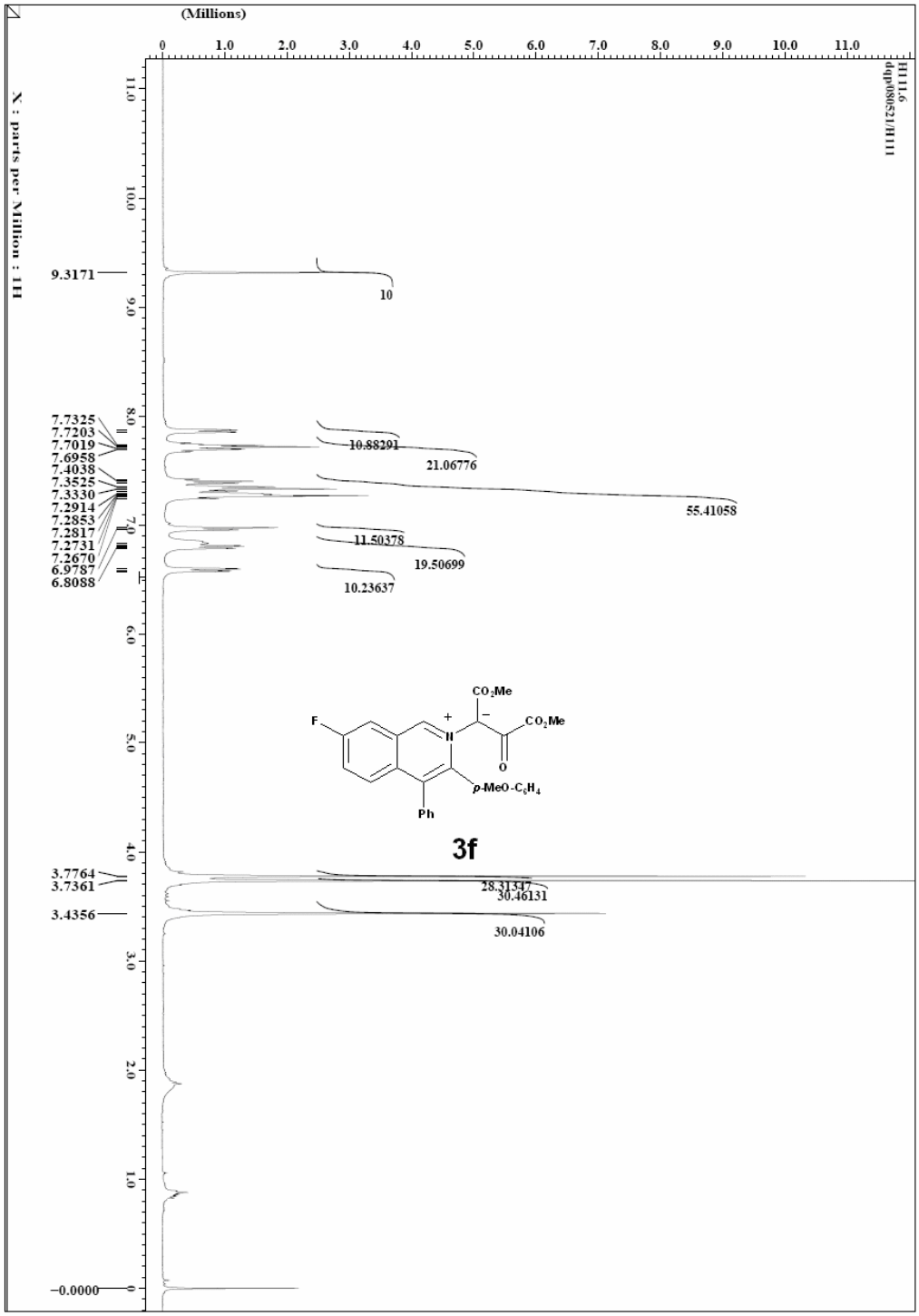

(Millions)
X : parts per Million : 13C
H97 C 8
dqtp080515 H97 C
171.7450
168.1886
159.2928
153.6102
148.8811
136.1524
131.4614
131.4137
129.9168
129.6498
129.4210
129.1159
126.8848
113.7843
113.6127
77.3145
76.9999
76.6757
55.0894
51.7046
50.2554
CO2Me
CO2Me
Ph
p-MeO-C6H4
3c

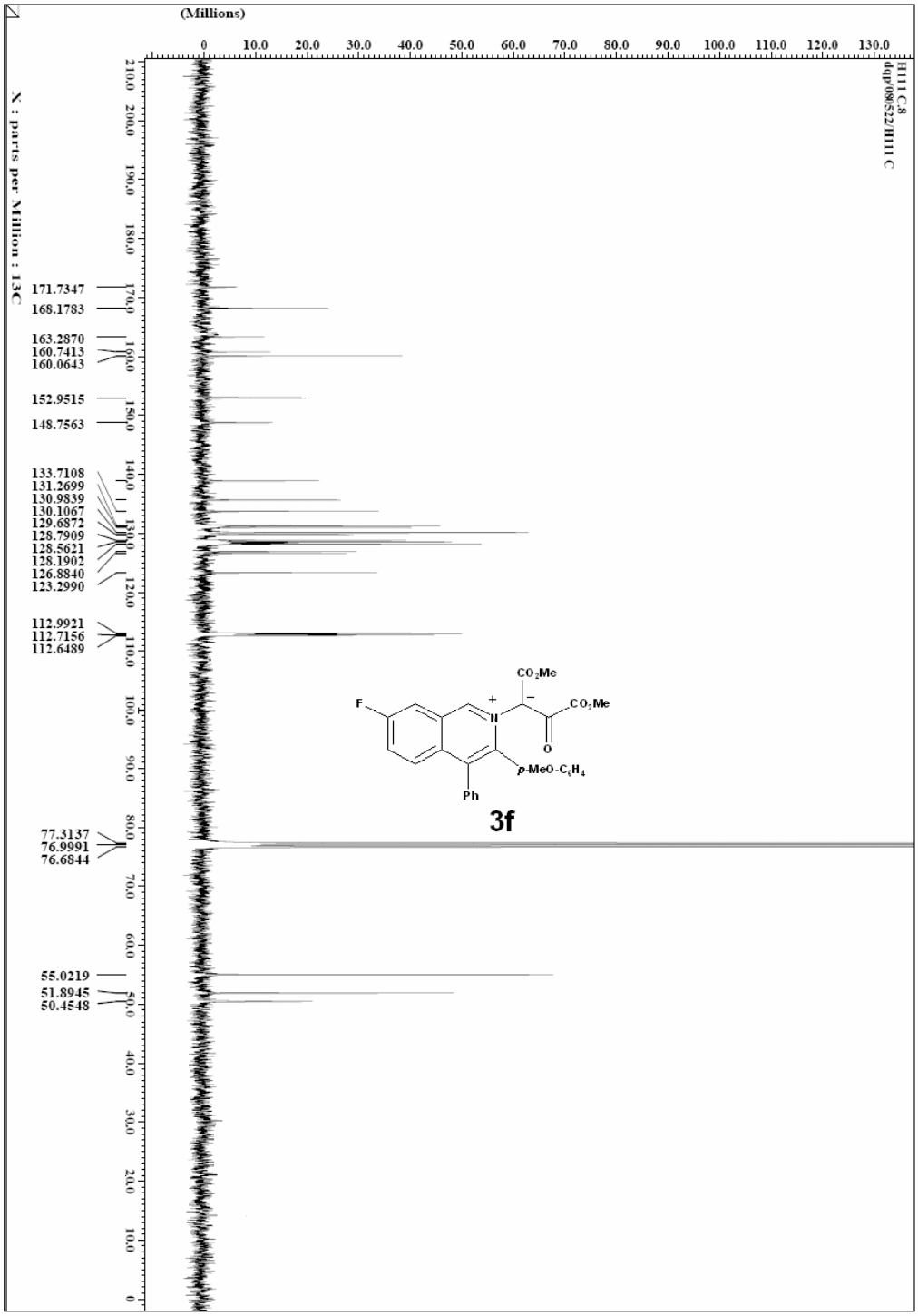

(Millions)
X : parts per Million : 1H
H1.30.6
dajp080529/H1p0
9.4014
8.3263
8.3055
8.0159
7.9280
7.6421
7.6189
7.5248
7.4539
7.4319
7.4100
7.2976
7.2841
7.2707
7.1913
7.1204
3.7471
3.4466
−0.0000
10
10.45504
9.9432
10.3759
20.16224
10.24432
20.14885
25.14581
10.82437
10.97674
10.34216
30.95891
30.34885
CO2Me
CO2Me
Ph
p-CF3-C6H4
3d

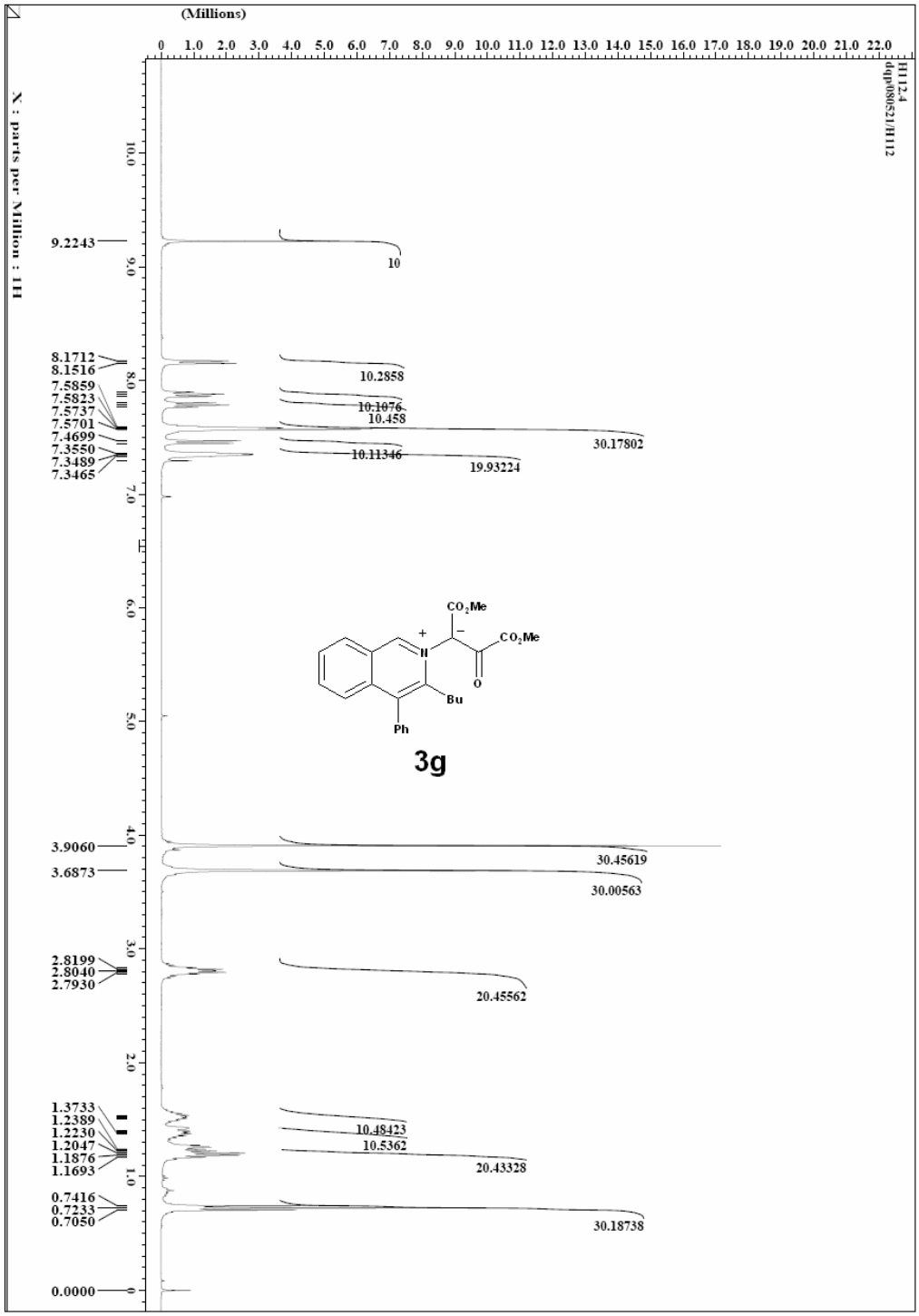

(Millions)
0
100.0
200.0
300.0
400.0
500.0
X : parts per Million : 13C
H130.C.6
dhp080529/H130.C
171.8495
168.0929
154.7159
148.9761
136.7909
130.8127
130.7269
130.3360
129.7067
129.6018
129.4397
127.4756
127.1800
125.9787
77.3142
76.9995
76.6849
51.8664
50.5029
130.8117
130.7259
130.5066
130.3350
129.7057
129.6008
129.4483
127.8655
127.4746
127.1790
125.9777
125.6440
125.6154
125.1768
125.1482
124.9765
122.2687
CO2Me
CO2Me
Ph
p-CF3-C6H4
3d

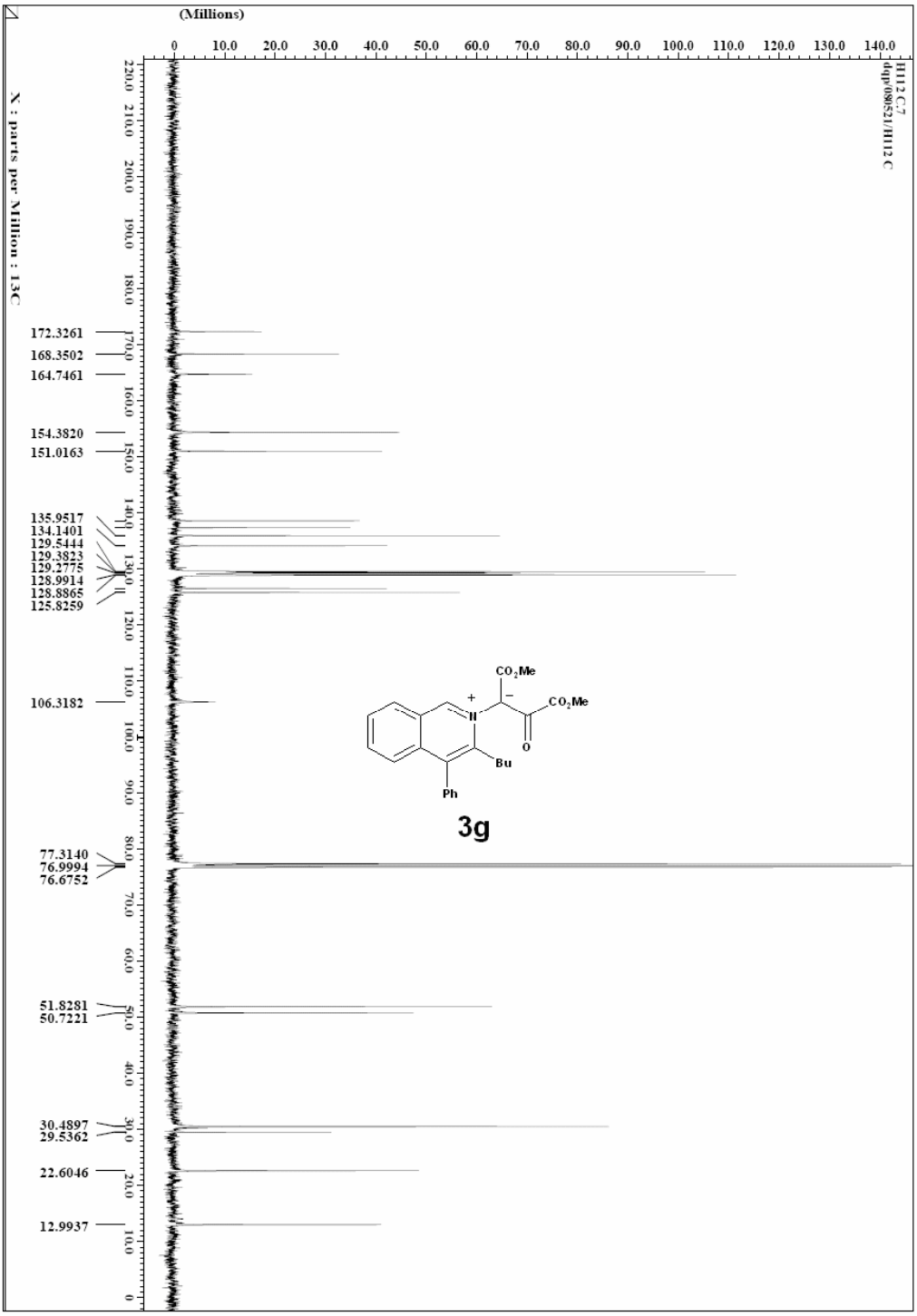

(Millions)
X : parts per Million : 1H
H110.4
dqp080521/H110
9.3440
8.2639
8.2432
7.9499
7.8840
7.8656
7.7080
7.6860
7.3977
7.3794
7.3574
7.3525
7.3171
7.3012
7.2755
7.2548
6.9848
6.6036
6.5975
3.7789
3.7324
3.4368
−0.0000
10
10.17141
10.30334
10.1178
9.99039
52.18651
11.51657
9.6517
9.97479
10.05466
30.57658
30.77009
30.30363
CO2Me
CO2Me
p-MeO-C6H4
Ph
3e

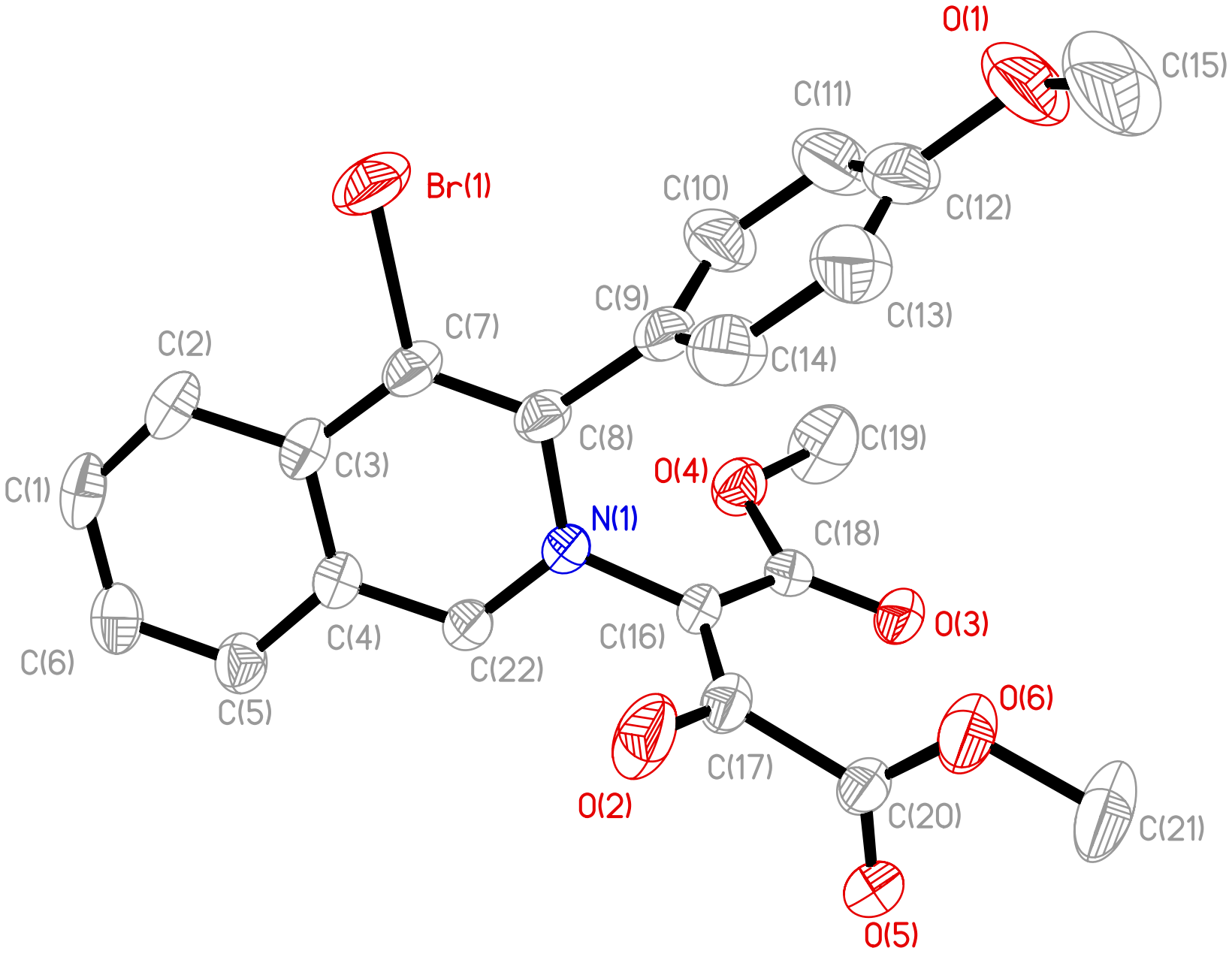

(Millions)
X : parts per Million : 13C
171.8311
168.3128
159.9510
153.9156
148.7955
136.1717
131.2805
130.2412
130.1650
129.9457
129.8789
128.3057
128.0483
126.4274
112.8978
112.6499
77.3148
77.0001
76.6760
54.9848
51.8479
50.3796
138.5649
138.4028
136.1717
133.9311
131.2805
130.9945
130.2412
130.1650
129.9457
129.8789
128.6680
128.3057
128.0483
127.2951
126.4274
123.5861
H110 C-7
dept080321/H110 C
CO2Me
CO2Me
O
p-MeO-C6H4
Ph
3e


(Millions)
X : parts per Million : 1H
9.3171
7.7325
7.7203
7.7019
7.6958
7.4038
7.3525
7.3330
7.2914
7.2853
7.2817
7.2731
7.2670
6.9787
6.8088
3.7764
3.7361
3.4356
−0.0000
10
10.88291
21.06776
55.41058
11.50378
19.50699
10.23637
28.31347
30.46131
30.04106
CO2Me
CO2Me
F
N
O
p-MeO-C6H4
Ph
3f


(Millions)
X : parts per Million : 13C
H111 C8
dqtp080522/H111 C
171.7347
168.1783
163.2870
160.7413
160.0643
152.9515
148.7563
133.7108
131.2699
130.9839
130.1067
129.6872
128.7909
128.5621
128.1902
126.8840
123.2990
112.9921
112.7156
112.6489
77.3137
76.9991
76.6844
55.0219
51.8945
50.4548
CO2Me
CO2Me
F
N
O
p-MeO-C6H4
Ph
3f


(Millions)
X : parts per Million : 1H
H112-4
dqp080821/H112
9.2243
8.1712
8.1516
7.5859
7.5823
7.5737
7.5701
7.4699
7.3550
7.3489
7.3465
3.9060
3.6873
2.8199
2.8040
2.7930
1.3733
1.2389
1.2230
1.2047
1.1876
1.1693
0.7416
0.7233
0.7050
0.0000
10
10.2858
10.1076
10.458
30.17802
10.11346
19.93224
30.45619
30.00563
20.45562
10.48423
10.5362
20.43328
30.18738
CO2Me
CO2Me
Bu
Ph
3g


(Millions)
X : parts per Million : 13C
H112 C7
dqp090521/H112 C
172.3261
168.3502
164.7461
154.3820
151.0163
135.9517
134.1401
129.5444
129.3823
129.2775
128.9914
128.8865
125.8259
106.3182
77.3140
76.9994
76.6752
51.8281
50.7221
30.4897
29.5362
22.6046
12.9937
CO2Me
CO2Me
Bu
Ph
3g


O(1)
C(15)
C(11)
Br(1)
C(10)
C(12)
C(9)
C(13)
C(7)
C(2)
C(14)
C(8)
C(19)
C(3)
O(4)
C(1)
N(1)
C(18)
O(3)
C(4)
C(16)
C(6)
C(22)
C(5)
O(6)
C(17)
O(2)
C(20)
C(21)
O(5)