Advanced Synthesis & Catalysis

Supporting Information

Access to Functionalized Isoquinoline-*N*-oxides via Sequential Cyclization-Coupling Reactions

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Supporting Information

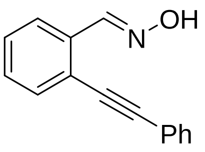
General Materials and Methods: NMR samples were run in $CDCl_3$ and ^1H NMR were referenced to TMS, ^{13}C NMR were referenced to $CDCl_3$ (δ = 77.0). All chemical shift values are quoted in ppm and coupling constants quoted in Hz.

General procedure for preparation of 2-alkynylbenzaldoxime 1*[1]*:

A solution of 2-alkynylbenzaldehyde (3.0 mmol), hydroxylamine hydrochloride (6 mmol, 2.0 equiv), pyridine (6.0 mmol, 2.0 equiv) in C_2H_5OH (15 mL) was stirred under refluxed for 2 hours. After completion of reaction as indicated by TLC, the solvent was evaporated and then quenched with water (10 mL), extracted with EtOAc (2×30 mL), dried by anhydrate Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding 2-alkynylbenzaldoxime **1**.

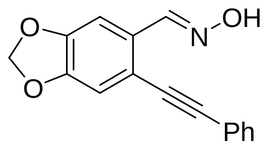
General procedure for electrophilic cyclization of 2-alkynylbenzldoxime 1 with various electrophiles:

The electrophile (1.2 equiv) in 2 mL of CH_2Cl_2 was added dropwise to a solution of 2-alkynylbenzaldoxime (0.30 mmol) in 4 mL of CH_2Cl_2. The reaction was stirred at room temperature for a period of time (10 min: Br_2, ICl, or NIS; 24 h: NBS and I_2). The reaction mixture was then diluted with CH_2Cl_2 (50 mL), washed with saturated aqueous $Na_2S_2O_3$ (25 mL), dried (Na_2SO_4) and filtered. The solvent was evaporated under reduced pressure and the product **2** was isolated by chromatography on a silica gel column.



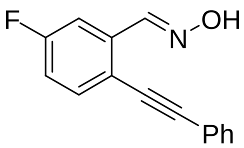
(*E*)-2-(2-Phenylethynyl)benzaldehyde oxime[2,3] (**1a**)

Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.38 (m, 5H), 7.50-7.57 (m, 3H), 7.87 (d, *J* = 7.3 Hz, 1H), 8.66 (s, 1H), 8.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 86.1, 95.0, 122.6, 123.1, 125.1, 128.4, 128.5, 128.6, 129.7, 131.6, 132.6, 132.8, 149.0.



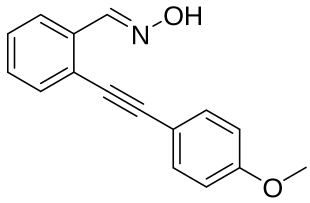
(*E*)-6-(2-Phenylethynyl)benzo[*d*][1,3]dioxole-5-carbaldehyde oxime (**1b**)

Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.02 (s, 2H), 6.96 (s, 1H), 7.30-7.37 (m, 4H), 7.50-7.54 (m, 2H), 7.86 (br, 1H), 8. 67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 86.1, 93.8, 101.8, 104.6, 111.4, 117.7, 122.8, 128.3, 128.4, 128.5, 131.5, 148.4, 148.9, 149.0; MS (ESI): *m/z* 266 (M^++H); HRMS calcd for $C_{16}H_{12}NO_3$ (M^++H): 266.0817, found: 266.0813.



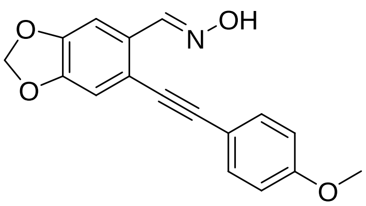
(*E*)-5-Fluoro-2-(2-phenylethynyl)benzaldehyde oxime (**1c**)

Yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (dt, *J* = 3.0, 8.8 Hz, 1H), 7.31-7.38 (m, 3H), 7.50-7.60 (m, 4H), 8.40 (br, 1H), 8.67-8.70 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 85.1, 94.6, 111.9 (d, $^{2}J_{CF}$ = 23.8 Hz), 117.3 (d, $^{2}J_{CF}$ = 22.9 Hz), 119.3, 122.5, 128.4, 128.7, 131.5, 134.4 (d, $^{3}J_{CF}$ = 7.6 Hz), 135.1 (d, $^{3}J_{CF}$ = 7.6 Hz), 148.3, 162.3 (d, $^{1}J_{CF}$ = 248.8 Hz); MS (ESI): *m/z* 240 (M^++H); HRMS calcd for $C_{15}H_{11}FNO$ (M^++H): 240.0825, found: 240.0815.

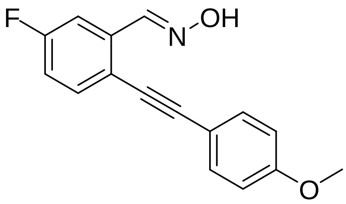


(*E*)-2-(2-(4-methoxyphenyl)ethynyl)benzaldehyde oxime (**1d**)

Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (s, 3H), 6.88 (d, *J* = 8.3 Hz, 2H), 7.28-7.36 (m, 2H), 7.44-7.54 (m, 3H), 7.85 (d, *J* = 7.3 Hz, 1H), 8.71 (br, 1H), 8.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 84.9, 95.1, 114.0, 114.7, 123.5, 125.1, 128.2, 129.6, 132.4, 132.6, 133.1, 149.1, 159.8; MS: *m/z* 274 $[M+Na]^+$; HRMS calcd for $C_{16}H_{13}NO_2$: 274.0844 $[M+Na]^+$, found: 274.0841.



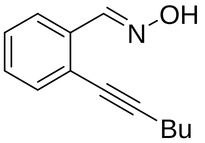
(*E*)-6-(2-(4-Methoxyphenyl)ethynyl)benzo[*d*][1,3]dioxole-5-carbaldehyde oxime (**1e**) Yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.83 (s, 3H), 6.01 (s, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.94 (s, 1H), 7.31 (s, 1H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.55(br, 1H), 8.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 84.8, 93.8, 101.7, 104.5, 106.1, 111.3, 114.0, 114.8, 128.0, 132.9, 148.1, 148.9, 149.0, 159.8; MS (ESI): *m/z* 296 (M^++H); HRMS calcd for $C_{17}H_{14}NO_4$ (M^++H): 296.0923, found: 296.0920.



(*E*)-5-Fluoro-2-(2-(4-methoxyphenyl)ethynyl)benzaldehyde oxime (**1f**)

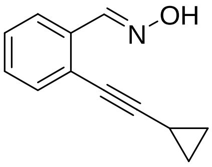
Yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.81 (s, 3H), 6.87 (d, *J* = 8.8 Hz, 2H), 7.04 (dt, *J* = 2.3, 8.2 Hz, 1H), 7.45-7.58 (m, 4H), 8.70 (s, 1H), 8.91 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 83.9, 94.7, 111.8 (d, $^2J_{CF}$ = 23.8 Hz), 114.0, 114.6, 117.2 (d, $^2J_{CF}$ = 22.9 Hz) , 119.7, 133.0, 134.1 (d, $^3J_{CF}$ = 7.6 Hz), 134.8 (d, $^3J_{CF}$ = 7.6

Hz), 148.4 (d, ${}^4J_{CF}$ = 1.9 Hz), 159.8, 162.0 (d, ${}^1J_{CF}$ = 248.8 Hz); MS (EI): *m/z* 270 (M^++H); HRMS calcd for $C_{16}H_{13}FNO_2$ (M^++H): 270.0930, found: 270.0926.



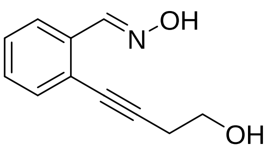
(*E*)-2-(Hex-1-ynyl)benzaldehyde oxime[2,3] (**1g**)

Yield: 79%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.95 (t, *J* = 6.8 Hz, 3H), 1.44-1.53 (m, 2H), 1.57-1.64 (m, 2H), 2.45 (t, *J* = 6.8 Hz, 2H), 7.24-7.31 (m, 2H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 8.67 (s, 1H), 9.32 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 19.2, 22.0, 30.6, 77.5, 96.5, 124.1, 124.9, 127.7, 129.5, 132.5, 132.6, 149.3.



(*E*)-2-(2-Cyclopropylethynyl)benzaldehyde oxime (**1h**)

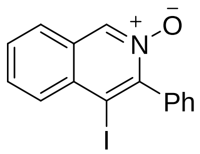
Yield: 69%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.80-0.93 (m, 4H), 1.45-1.52 (m, 1H), 7.24-7.30 (m, 2H), 7.38-7.41 (m, 1H),, 7.78 (d, *J* = 6.8 Hz, 1H), 8.63 (s, 1H), 9.08 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.3, 8.8, 72.6, 99.6, 123.9, 124.9, 127.7, 129.5, 132.6, 132.8, 149.3; MS (ESI): *m/z* 186 (M^++H); HRMS calcd for $C_{12}H_{12}NO$ (M^++H): 186.0919, found: 186.0909.



(*E*)-2-(4-hydroxybut-1-ynyl)benzaldehyde oxime (**1i**)

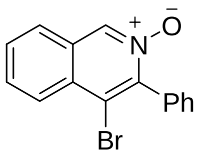
Yield: 36%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.74 (t, *J* = 6.0 Hz, 2H), 3.86 (t, *J* = 6.4 Hz, 2H), 7.28-7.32 (m, 2H), 7.42-7.46 (m, 1H), 7.66-7.70 (m, 1H), 8.54 (br, 2H); ^{13}C

NMR (100 MHz, $CDCl_3$) δ 24.1, 61.2, 80.5, 92.6, 122.6, 126.8, 128.3, 129.6, 132.9, 133.0, 149.8; MS: *m/z* 189 (M^+); HRMS calcd for $C_{11}H_{11}NO_2$: 189.0790, found:189.0788.



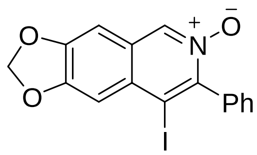
2a

Yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 6.8 Hz, 2H), 7.48-7.58 (m, 3H), 7.59-7.69 (m, 3H), 8.06 (d, *J* = 8.3 Hz, 1H), 8.85 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 122.1, 124.9, 127.3, 128.5, 128.9, 129.4, 129.7, 129.8, 129.9, 133.4, 136.0, 147.7; MS (ESI): *m/z* 348 (M^++H); Elemental analysis calcd (%) for $C_{15}H_{10}INO$: C 51.90, H 2.90, N 4.03; Found: C 51.64, H 2.88, N 3.98.



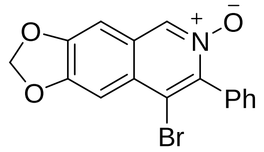
2b

Yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 6.8 Hz, 2H), 7.48-7.58 (m, 3H), 7.63-7.69 (m, 2H), 7.72 (t, *J* = 8.3 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 8.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 122.1, 124.9, 127.3, 128.5, 128.9, 129.4, 129.7, 129.8, 129.9, 133.4, 136.0, 147.7; MS (ESI): *m/z* 300 (M^++H); Elemental analysis calcd (%) for $C_{15}H_{10}BrNO$: C 60.02, H 3.36, N 4.67; Found: C 60.25, H 3.55, N 4.74.



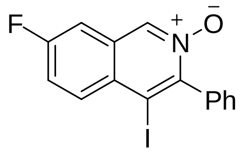
2c

Yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.16 (s, 2H), 6.92 (s, 1H), 7.37 (d, *J* = 6.8 Hz, 2H), 7.45 (s, 1H), 7.48-7.55 (m, 3H), 8.67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 99.7, 100.6, 102.5, 109.6, 125.7, 128.6, 129.3, 129.6, 130.1, 135.9, 137.6, 149.4, 150.3, 151.5; MS (ESI): *m/z* 392 (M^++H); Elemental analysis calcd (%) for $C_{16}H_{10}INO_3$: C 49.13, H 2.58, N 3.58; Found: C 48.86, H 2.62, N 3.51.



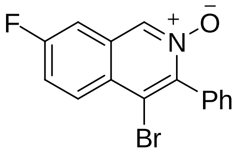
2d

Yield: 25%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.17 (s, 2H), 6.99 (s, 1H), 7.43 (d, J = 7.8 Hz, 2H), 7.46-7.56 (m, 4H), 8.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 100.7, 102.5, 104.3, 120.6, 126.1, 127.4, 128.5, 129.3, 129.7, 133.7, 135.4, 146.0, 150.5, 151.3; MS (ESI): *m/z* 366 (M^++Na); HRMS calcd for $C_{16}H_{10}BrNO_3$: 365.9742 $[M+Na]^+$; Found: 365.9757.



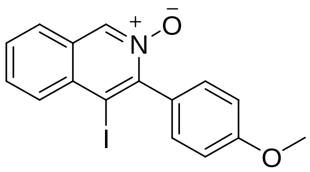
2e

Yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (dd, J = 2.4, 8.8 Hz, 1H), 7.35-7.41 (m, 3H), 7.48-7.58 (m, 3H), 8.10 (dd, J = 8.8, 9.3 Hz, 1H), 8.78 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 101.3, 108.2 (d, $^2J_{CF}$ = 22.9 Hz), 119.9 (d, $^2J_{CF}$ = 24.8 Hz), 128.4, 128.6, 129.1, 129.4, 129.5, 135.5 (d, $^3J_{CF}$ = 9.5 Hz), 135.7, 136.9, 150.7, 162.4 (d, $^1J_{CF}$ =251.7 Hz); MS (ESI): *m/z* 366 (M^++H)；Elemental analysis calcd (%) for $C_{15}H_9FINO$: C 49.34, H 2.48, N 3.84; Found: C 49.56, H 2.66, N 3.71.



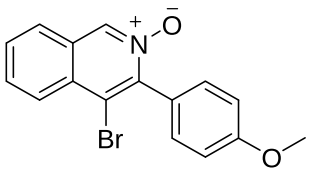
2f

Yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (dd, J = 2.4, 8.3 Hz, 1H), 7.38-7.46 (m, 3H), 7.50-7.58 (m, 3H), 8.18 (dd, J = 9.3, 9.3 Hz, 1H), 8.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 108.3 (d, $^2J_{CF}$ = 22.8 Hz), 119.8 (d, $^2J_{CF}$ = 22.8 Hz), 122.0, 125.8, 128.5, 129.4, 129.7, 130.3, 130.4, 133.1, 135.2, 147.3, 162.6 (d, $^1J_{CF}$ =251.7 Hz); MS (ESI): *m/z* 318 (M^++H); Elemental analysis calcd (%) for $C_{15}H_9BrFNO$: C 56.63, H 2.85, N 4.40; Found: C 56.84, H 3.11, N 4.25.



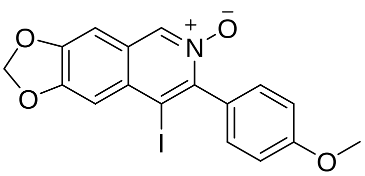
2g

Yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 (s, 3H), 7.07 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 7.60-7.67 (m, 3H), 8.06 (d, *J* = 8.3 Hz, 1H), 8.84 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.2, 102.3, 114.0, 125.0, 128.3, 129.4, 129.6, 130.1, 131.0, 131.6, 132.5, 136.6, 150.9, 160.2; MS (ESI): *m/z* 378 (M^++H); HRMS calcd for $C_{16}H_{13}INO_2$ (M^++H): 377.9991, Found: 377.9988.



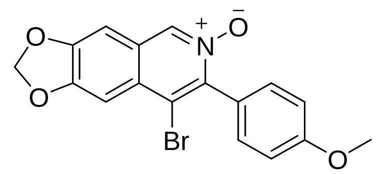
2h

Yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.88 (s, 3H), 7.06 (d, *J* = 8.8 Hz, 2H), 7.40 (d, *J* = 8.8 Hz, 2H), 7.61-7.72 (m, 3H), 8.15 (d, *J* = 8.3 Hz, 1H), 8.87 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.2, 113.9, 122.4, 124.8, 125.4, 127.2, 128.4, 128.9, 129.6, 129.7, 131.2, 135.9, 147.6, 160.2; MS (ESI): *m/z* 330 (M^++H); HRMS calcd for $C_{16}H_{13}BrNO_2$ (M^++H): 330.0130, Found: 330.0127.



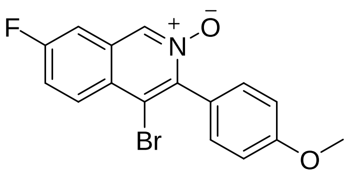
2i

Yield: 89%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.88 (s, 3H), 6.16 (s, 2H), 6.91 (s, 1H), 7.05 (d, *J* = 8.8 Hz, 2H), 7.31 (d, *J* = 8.8 Hz, 2H), 7.45 (s, 1H), 8.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.2, 100.3, 100.6, 102.4, 109.7, 114.0, 125.7, 129.9, 130.1, 131.1, 135.9, 149.3, 150.2, 151.4, 160.1; MS (ESI): *m/z* 422 (M^++H); Elemental analysis calcd (%) for $C_{17}H_{12}INO_4$: C 48.48, H 2.87, N 3.33; Found: C 48.44, H 2.75, N 3.37.



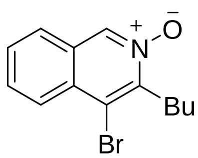
2j

Yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.87 (s, 3H), 6.15 (s, 2H), 6.94 (s, 1H), 7.05 (d, *J* = 8.8 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.45 (s, 1H), 8.68 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.2, 100.6, 102.4, 104.1, 113.9, 120.8, 125.8, 125.9, 127.2, 131.2, 135.2, 145.9, 150.3, 151.1, 160.1; MS (ESI): *m/z* 374 (M^{+}+H); Elemental analysis calcd (%) for $C_{17}H_{12}BrNO_4$: C 54.57, H 3.23, N 3.74; Found: C 54.18, H 3.29, N 3.62.



2k

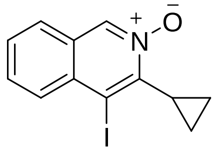
Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.88 (s, 3H), 7.06 (d, *J* = 8.3 Hz, 2H), 7.34-7.45 (m, 4H), 8.18 (dd, *J* = 9.3, 9.3 Hz, 1H), 8.82 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 108.3 (d, $^{2}J_{CF}$ = 22.9 Hz), 114.0, 119.7 (d, $^{2}J_{CF}$ = 25.7 Hz), 122.3, 125.1, 125.9, 129.4 (d, $^{3}J_{CF}$ = 10.5 Hz), 130.4 (d, (d, $^{3}J_{CF}$ = 9.5 Hz), 131.3, 135.1 (d, ^{4}J = 5.7 Hz), 147.2, 160.3, 162.6 (d, $^{1}J_{CF}$ =251.7 Hz); MS (ESI): *m/z* 348 (M^{+}+H); Elemental analysis calcd (%) for $C_{16}H_{11}BrFNO_2$: C 55.20, H 3.18, N 4.02; Found: C 55.16, H 3.15, N 4.01.



2l

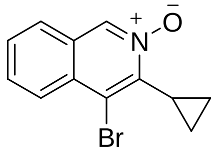
Yield: 85%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, 3H), 1.47-1.58 (m, 2H), 1.68-1.77 (m, 2H), 3.37 (t, *J* = 7.8 Hz, 2H), 7.57 (d, *J* = 6.8 Hz, 1H), 7.61-7.67 (m, 2H), 8.07 (d, *J* = 8.3 Hz, 1H), 8.80 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 22.8, 28.2, 30.9, 120.9, 124.8, 126.5, 127.5, 128.8, 128.9, 129.7, 135.7, 149.9; MS

(ESI): *m/z* 280 (M^++H); Elemental analysis calcd (%) for $C_{13}H_{14}BrNO$: C 55.73, H 5.04, N 5.00; Found: C 55.45, H 5.34, N 4.89.



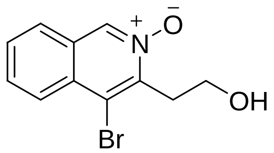
2m

Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.08-1.14 (m, 2H), 1.37-1.44 (m, 2H), 2.06-2.15 (m, 1H), 7.49-7.61 (m, 3H), 8.05 (d, *J* = 8.8 Hz, 1H), 8.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.1, 17.7, 103.5, 124.6, 127.1, 128.9, 129.8, 131.5, 131.6, 136.4, 150.9; MS (ESI): *m/z* 312 (M^++H); Elemental analysis calcd (%) for $C_{12}H_{10}INO$: C 46.33, H 3.24, N 4.50; Found: C 46.33, H 3.23, N 4.47.



2n

Yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.19-1.24 (m, 2H), 1.29-1.35 (m, 2H), 2.10-2.19 (m, 1H), 7.52-7.64 (m, 3H), 8.12 (d, *J* = 8.3 Hz, 1H), 8.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.6, 13.6, 123.5, 124.5, 126.5, 127.4, 128.9, 129.0, 129.4, 135.8, 148.3; MS (ESI): *m/z* 264 (M^++H); Elemental analysis calcd (%) for $C_{12}H_{10}BrNO$: C 54.57, H 3.82, N 5.30; Found: C 54.42, H 3.82, N 5.22.

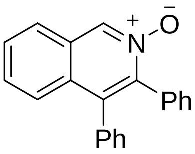


2o

Yield: 64%; ^{1}H NMR (400 MHz, DMSO) δ 3.43 (t, *J* = 6.8 Hz, 2H), 3.69 (t, *J* = 6.8 Hz, 2H), 7.60-7.75 (m, 2H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.94 (d, *J* = 7.8 Hz, 1H), 9.01 (s, 1H); ^{13}C NMR (100 MHz, DMSO) δ 34.9, 57.8, 121.8, 125.9, 126.5, 128.2, 128.3, 129.9, 130.8, 136.0, 146.5; MS: *m/z* 268 (M^++H); Elemental analysis calcd (%) for $C_{11}H_{10}BrNO_2$: C 49.28, H 3.76, N 5.22; Found: C 49.25, H 3.78, N 5.14.

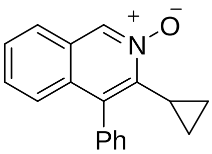
General procedure for synthesis of 3,4-disubstituted isoquinoline-N-oxide 3 via palladium-catalyzed Suzuki-Miyaura reaction.

A solution of 4-bromoisoquinoline-*N*-oxide **2** (0.30 mmol), sodium tetraphenylborate (0.15 mmol, 0.5 equiv) or arylboronic acid (0.45 mmol, 1.5 equiv), palladium catalyst (5 mol %), base (1.2 mmol, 4.0 equiv) in DMF/H_2O (2.0 mL) was stirred at 80 °C under N_2 for 24 h. After completion of reaction as indicated by TLC, the solvent was evaporated and then quenched with water (10 mL), extracted with EtOAc (2×10 mL), dried by anhydrate Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding 3,4-disubstituted isoquinoline-*N*-oxide **3**.



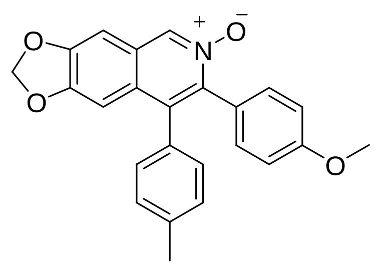
3a

Yield: 93% (for $NaBPh_4$) and 99% (for $PhB(OH)_2$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07-7.10 (m, 2H), 7.19-7.28 (m, 5H), 7.34-7.46 (m, 3H), 7.50 (dt, *J* = 1.5, 6.8 Hz, 1H), 7.60 (t, *J* = 6.8 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 1H), 7.96 (t, *J* = 6.8 Hz, 1H), 9.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 124.9, 126.2, 127.7, 127.8, 128.0, 128.3, 128.6, 129.0, 129.1, 129.7, 130.3, 130.7, 131.9, 134.7, 136.5, 137.0, 146.0; MS (ESI): *m/z* 298 (M^++H); HRMS calcd for $C_{21}H_{16}NO$ (M^++H): 298.1232, found: 298.1221



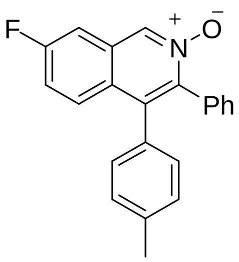
3b

Yield: 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.60-0.66 (m, 2H), 0.78-0.87 (m, 2H), 1.95-2.05 (m, 1H), 7.34 (dd, *J* =2.0, 7.8 Hz, 2H), 7.41 (d, *J* =4.4 Hz, 2H), 7.46-7.55 (m, 4H), 7.70 (d, *J* = 8.3 Hz, 1H), 8.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.9, 11.8, 124.4, 125.4, 127.8, 128.1, 128.2, 128.3, 128.4, 129.0, 130.2, 135.2, 135.9, 136.4, 146.8; MS (ESI): *m/z* 262 (M^++H); HRMS calcd for $C_{18}H_{16}NO$ (M^++H): 262.1232, found: 262.1227.



3c

Yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.32 (s, 3H), 3.75 (s, 3H), 6.05 (s, 2H), 6.72 (s, 1H), 6.76 (d, J = 8.8 Hz, 2H), 6.96 (d, J = 7.8 Hz, 2H), 7.00 (s, 1H), 7.08 (d, J = 7.8 Hz, 2H), 7.15 (d, J = 8.8 Hz, 2H), 8.77 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 55.1, 100.5, 101.9, 102.9, 113.2, 124.6, 126.2, 127.5, 128.9, 130.1, 132.0, 132.5, 135.1, 136.0, 137.3, 144.4, 149.6, 149.9, 159.0; MS (ESI): m/z 386 (M^++H); HRMS calcd for $C_{24}H_{20}NO_4$ (M^++H): 386.1392; Found: 386.1399.

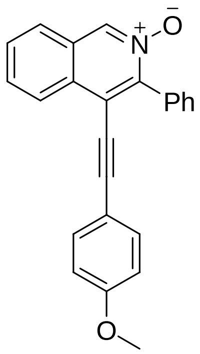


3d

Yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 3H), 6.99 (d, J = 8.3 Hz, 2H), 7.08 (d, J = 7.8 Hz, 2H), 7.16-7.28 (m, 6H), 7.37 (dd, J = 2.4, 8.8 Hz, 1H), 7.47 (dd, J = 9.3, 9.3 Hz, 1H), 8.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 107.9 (d, $^{2}J_{CF}$ = 21.9 Hz), 118.5 (d, $^{2}J_{CF}$ = 24.8 Hz), 126.3, 127.8, 128.3, 128.9, 129.3 (d, $^{3}J_{CF}$ = 9.5 Hz), 130.0 (d, $^{3}J_{CF}$ = 9.5 Hz), 130.1, 130.6, 131.6, 132.0, 135.0, 136.9, 137.7, 145.7, 162.1 (d, $^{1}J_{CF}$ = 249.8 Hz); MS (ESI): m/z 330 (M^++H); HRMS calcd for $C_{22}H_{17}FNO$ (M^++H): 330.1294; found: 330.1297.

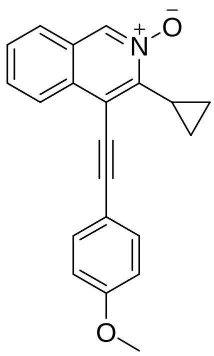
General procedure for synthesis of 3,4-disubstituted isoquinoline-N-oxide 4 via palladium-catalyzed Sonogashira reaction.

A solution of 4-iodoisoquinoline-*N*-oxide **2** (0.30 mmol), 4-methoxyphenylacetylene (0.45 mmol, 1.5 equiv), $PdCl_2(PPh_3)_2$ (3 mol %), CuI (3 mol %) in Et_3N (2.0 mL) was stirred at 80 °C under N_2 for 24 h. After completion of reaction as indicated by TLC, the solvent was evaporated and then quenched with water (10 mL), extracted with EtOAc (2×10 mL), dried by anhydrate Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding 3,4-disubstituted isoquinoline-*N*-oxide **4**.



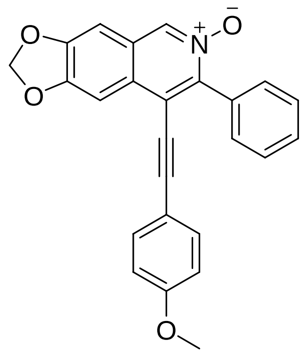
4a

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.81 (s, 3H), 6.83 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 2H), 7.48-7.57 (m, 3H), 7.61-7.75 (m, 5H), 8.30 (d, *J* = 8.8 Hz, 1H), 8.87 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 82.2, 101.4, 114.1, 120.0, 124.7, 125.9, 127.9, 128.5, 129.2, 129.3, 129.4, 130.6, 132.2, 133.2, 136.3, 148.9, 160.4; MS (ESI): *m/z* 352 (M^++H); HRMS calcd for $C_{24}H_{18}NO_2$ (M^++H): 352.1338; Found: 352.1339.



4b

Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.23-1.29 (m, 2H), 1.52-1.56 (m, 2H), 2.51-2.59 (m, 1H), 3.88 (s, 3H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.54-7.67 (m, 5H), 8.26 (d, *J* = 8.3 Hz, 1H), 8.78 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.8, 12.2, 55.4, 81.3 102.4, 114.3, 114.4, 118.7, 124.6, 125.2, 127.4, 128.7, 129.1, 129.6, 133.1, 136.2, 150.9, 160.4; MS (ESI): *m/z* 316 (M^++H); HRMS calcd for $C_{21}H_{18}NO_2$ (M^++H): 316.1338; Found: 316.1331.

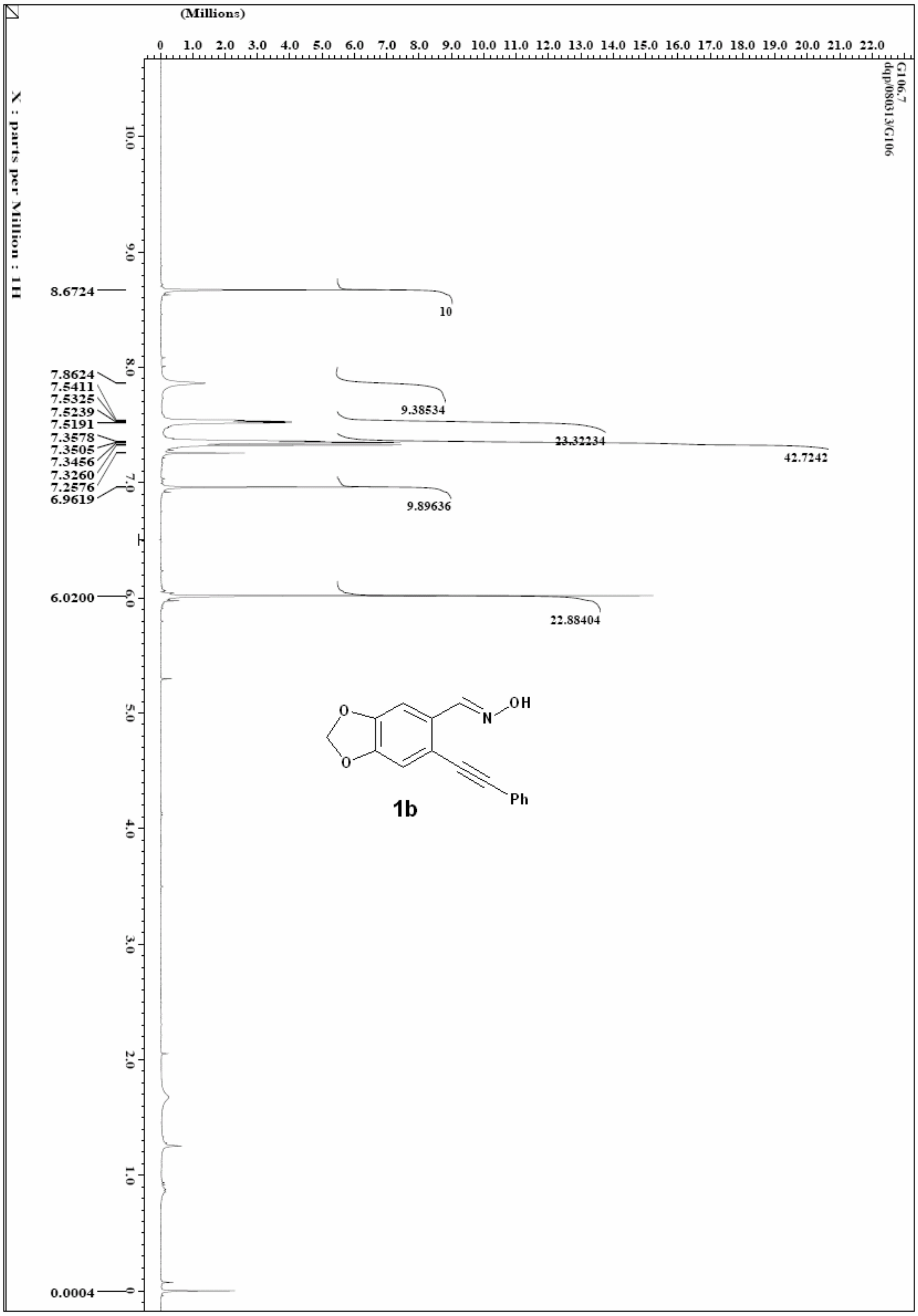


4c

Yield: 71%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.80 (s, 3H), 6.15 (s, 2H), 6.81 (d, J = 8.8 Hz, 2H), 6.97 (s, 1H), 7.18 (d, J = 8.8 Hz, 2H), 7.48-7.54 (m, 3H), 7.57 (s, 1H), 7.68 (d, J = 6.8 Hz, 2H), 8.68 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 82.5, 100.7, 102.2, 102.7, 114.1, 118.8, 125.0, 125.9, 127.8, 127.9, 129.1, 130.6, 132.3, 133.1, 135.6, 147.1, 150.2, 150.8, 160.3; MS (ESI): *m/z* 396 (M^++H); HRMS calcd for $C_{25}H_{18}NO_4$ (M^++H): 396.1236; Found: 396.1233.

Reference :

1. Yang, S. H.; Chang, S. *Org. Lett.* **2001**, 3, 4209.
2. Takao, S.; Kondo, Y.; Miura, N.; Hayashi, K.; Yamanaka, H. *Heterocycles* **1986**, 24, 2311.
3. Yeom, H.S.; Kim, S.; Shin, S. *Synlett* **2008**, 924.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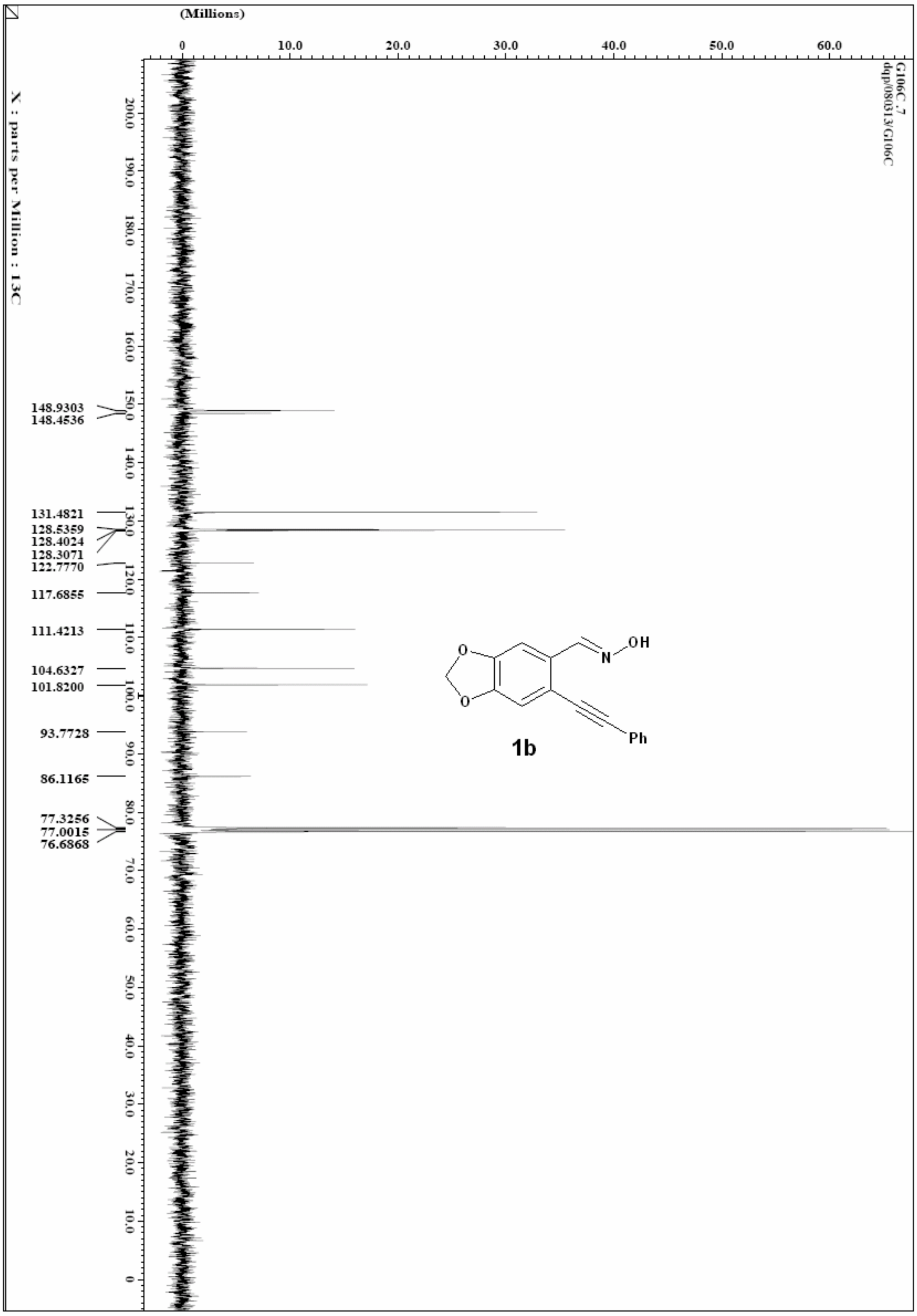
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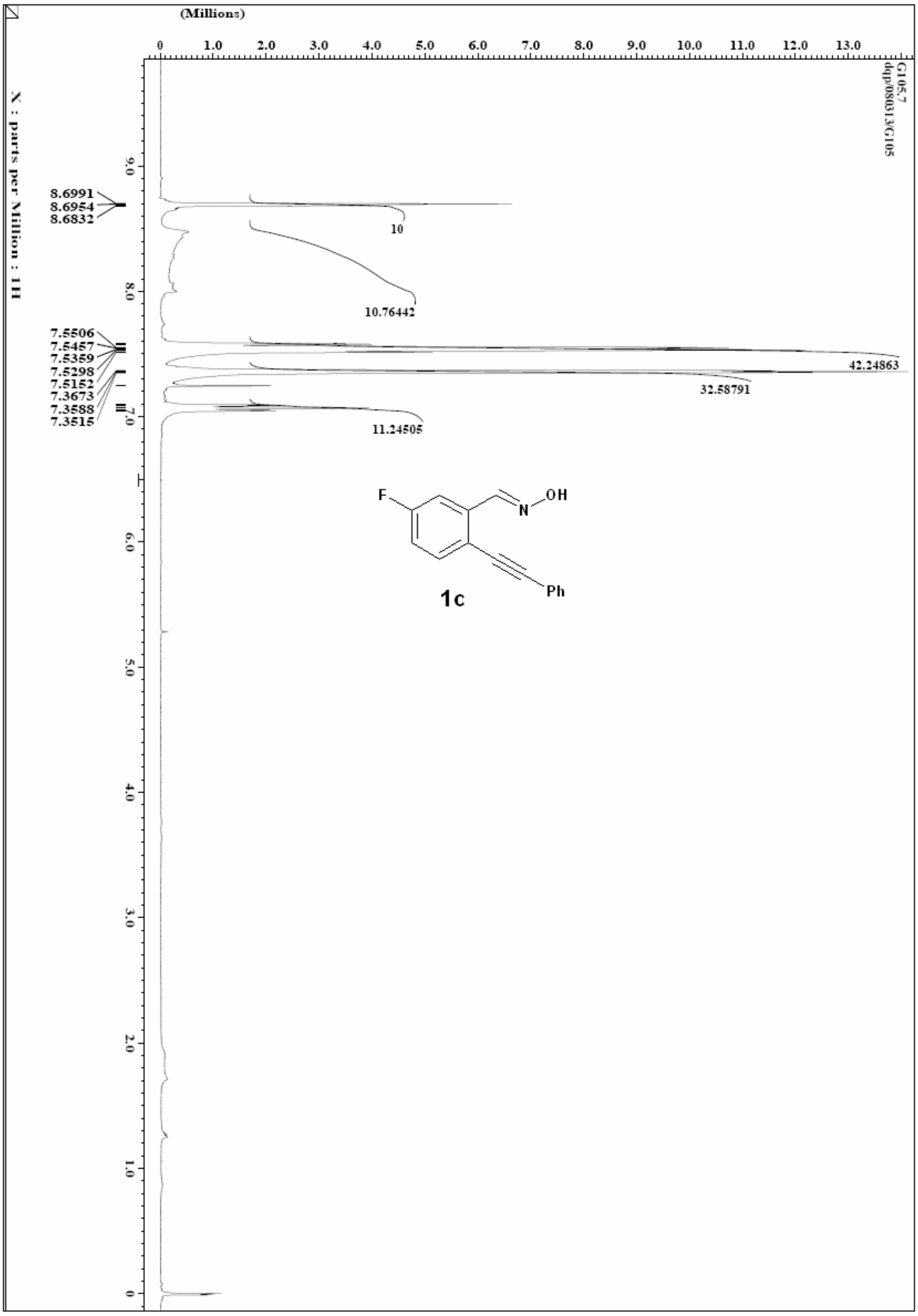
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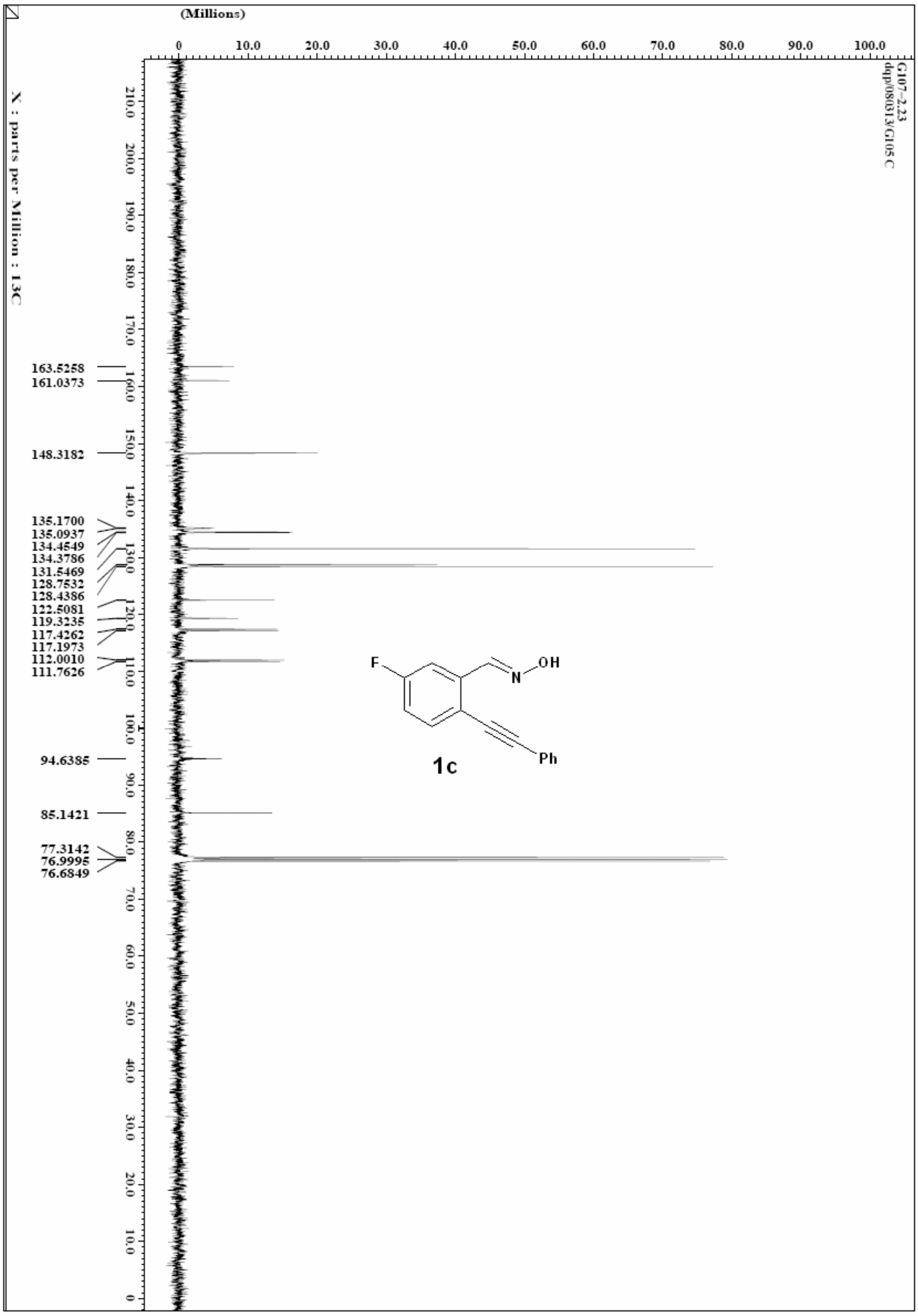
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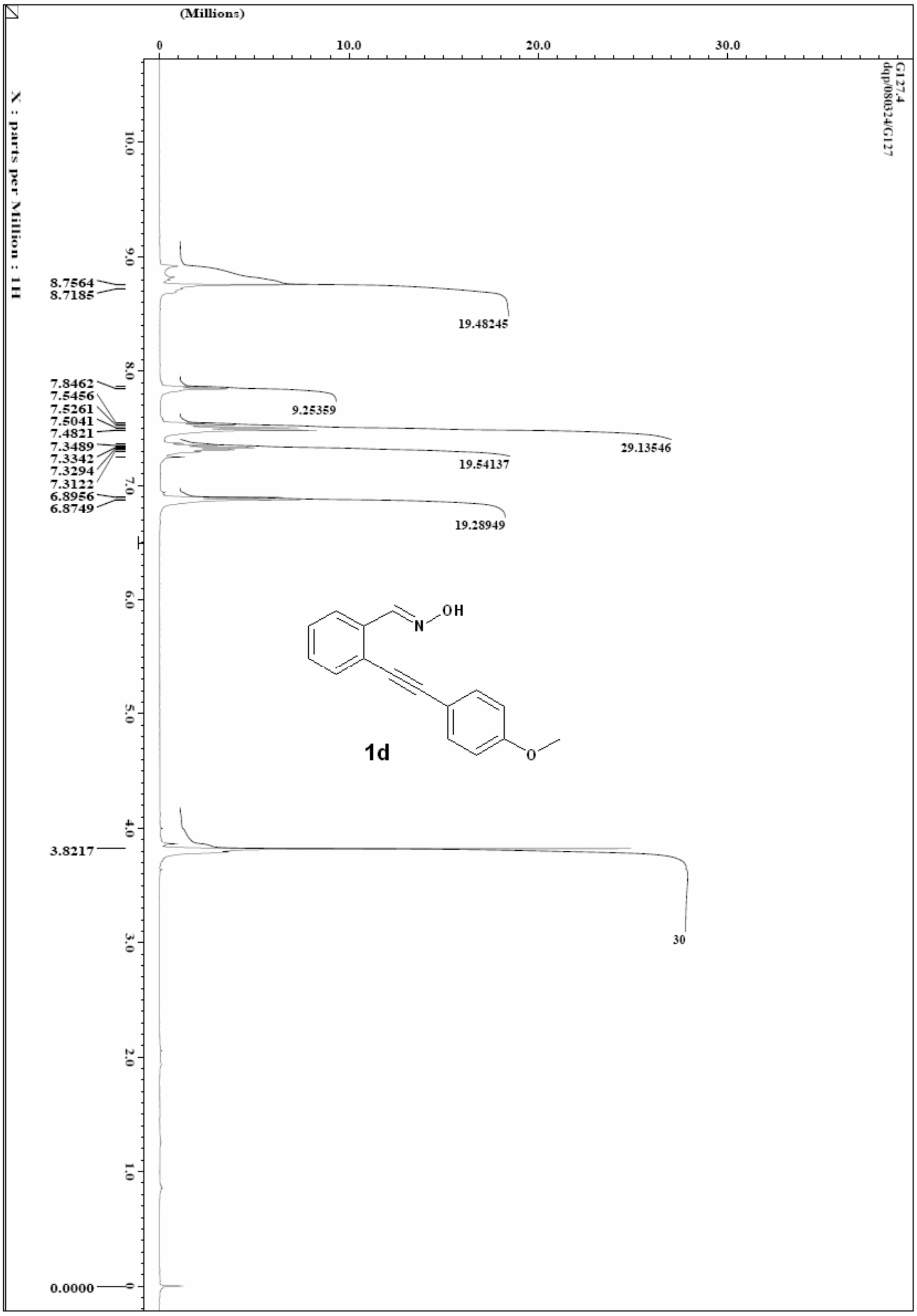
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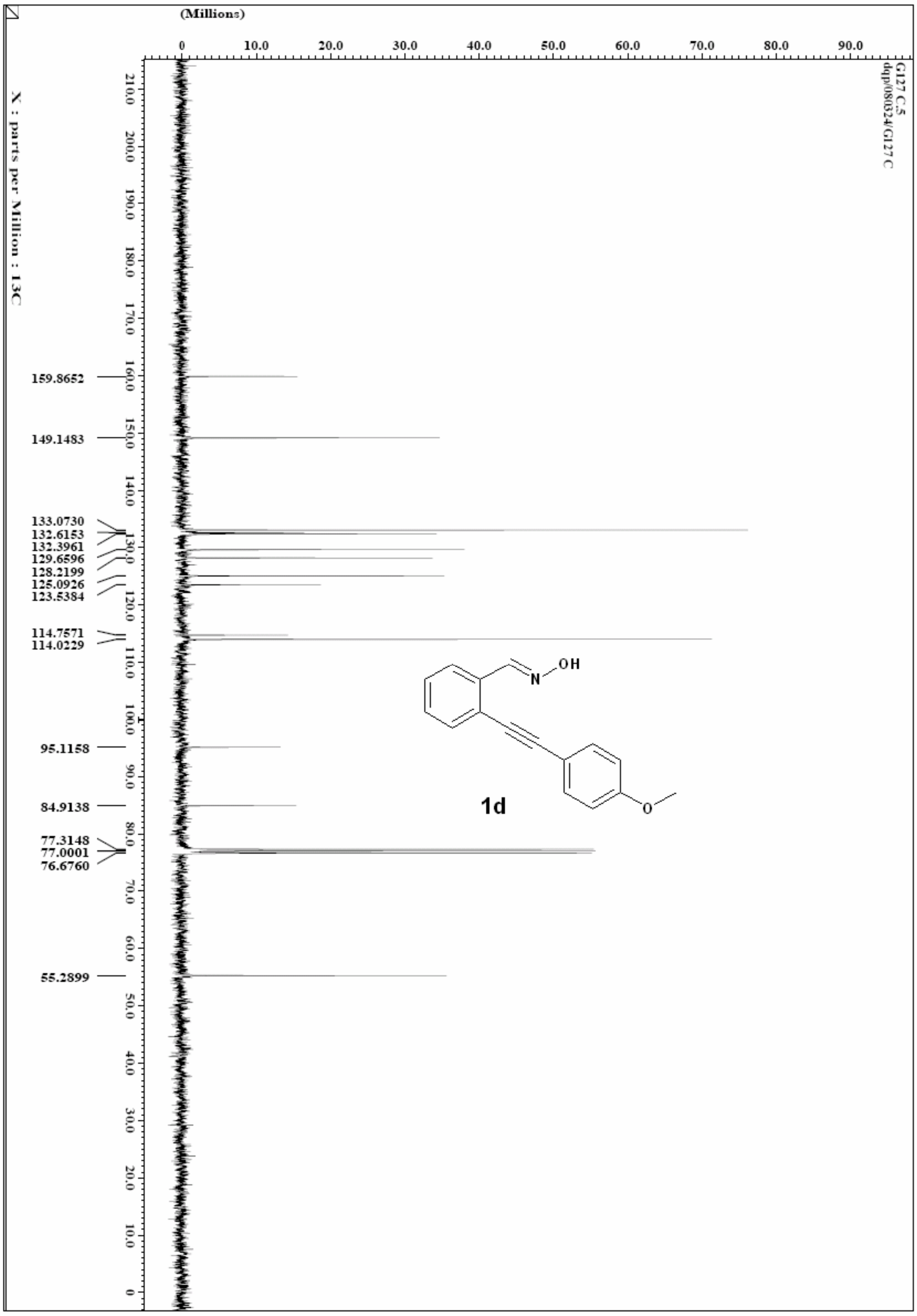
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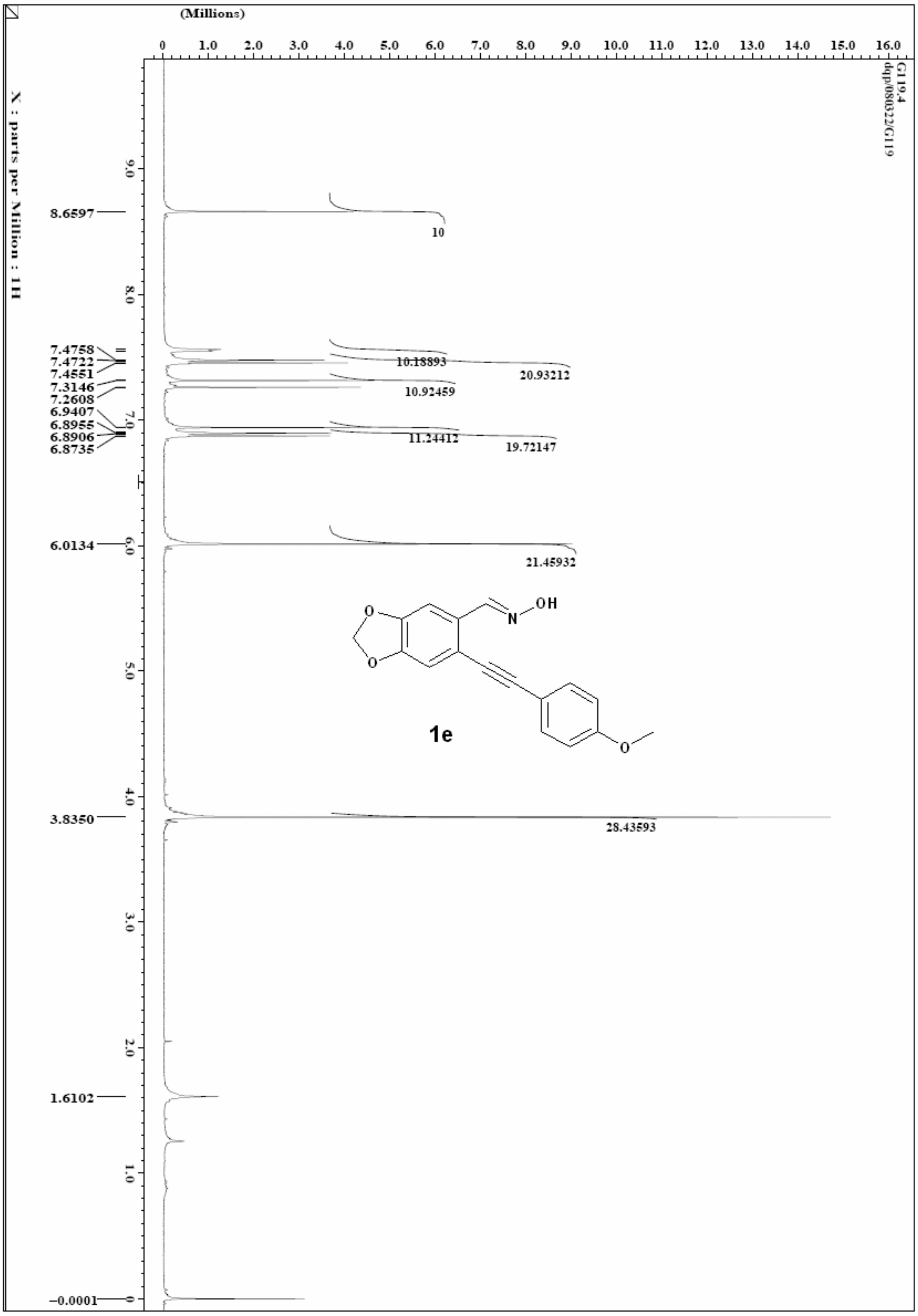
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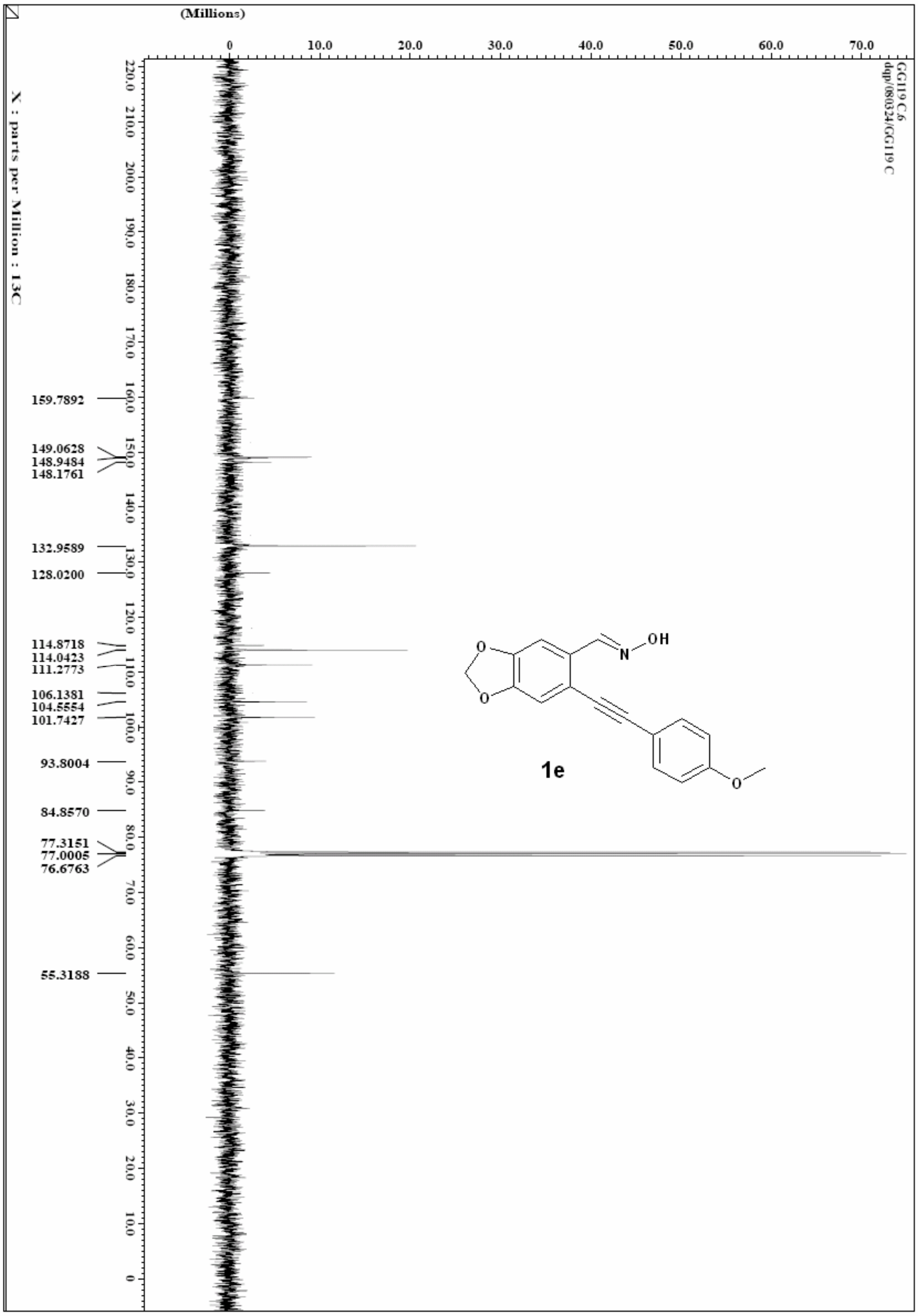
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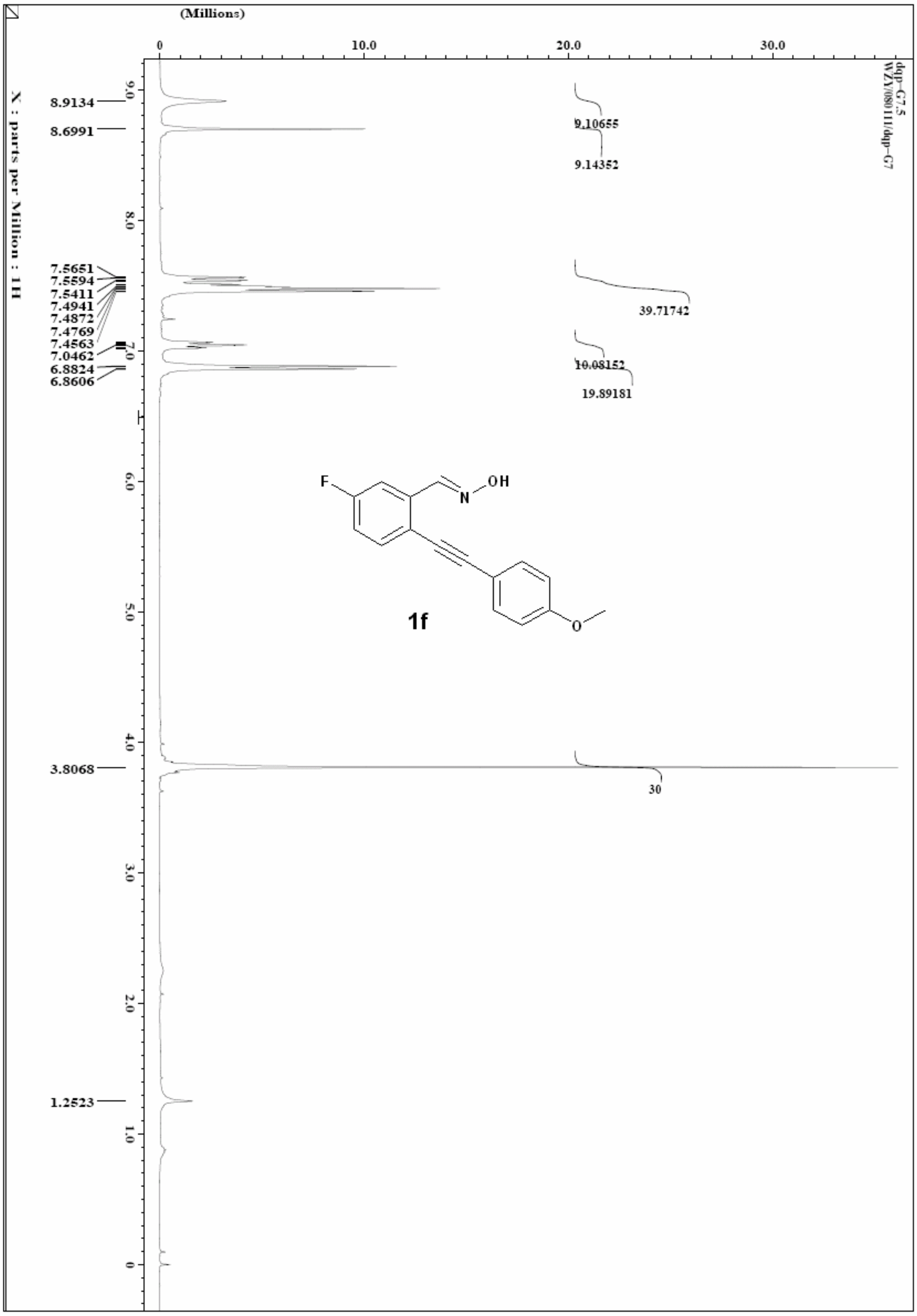
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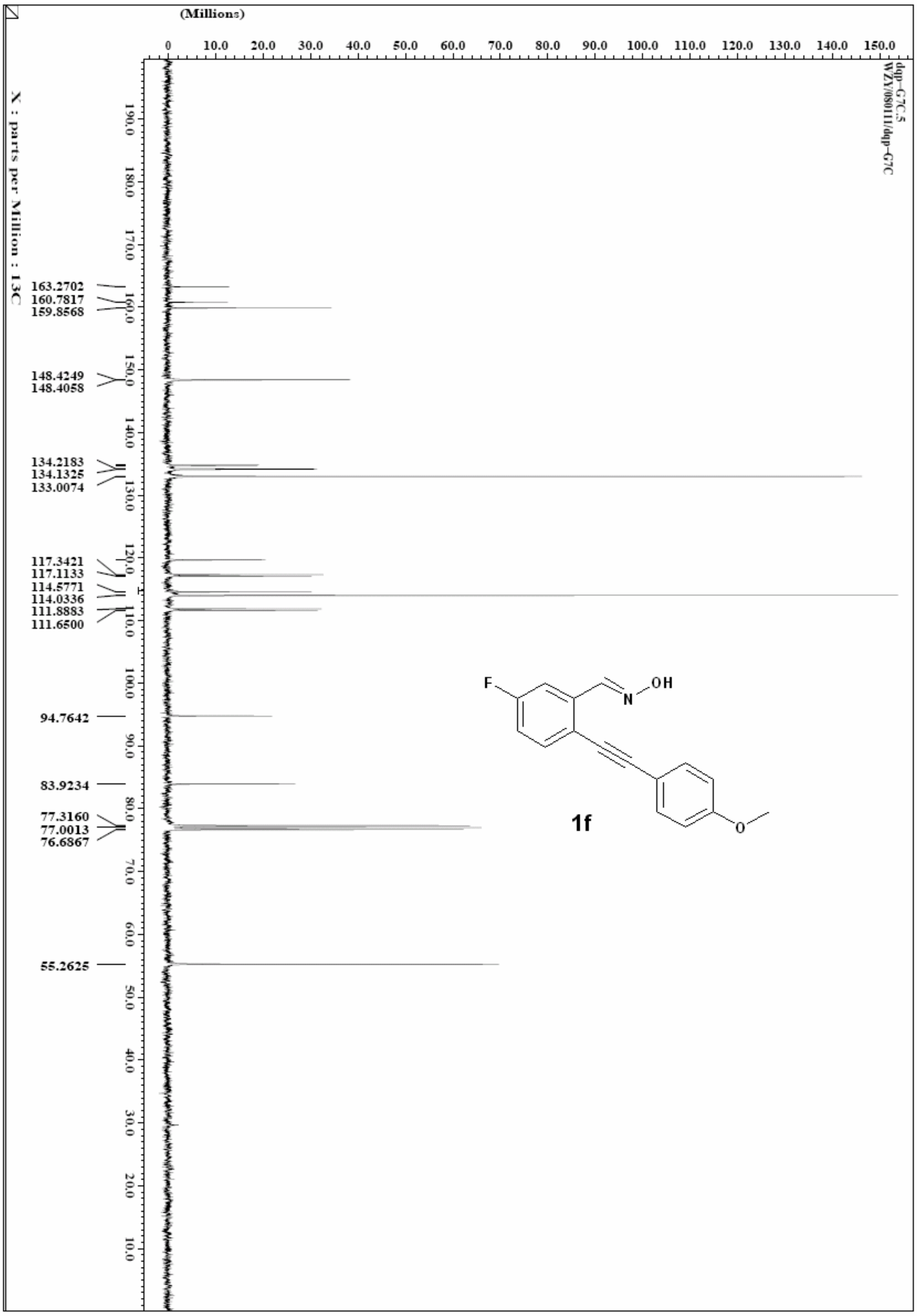

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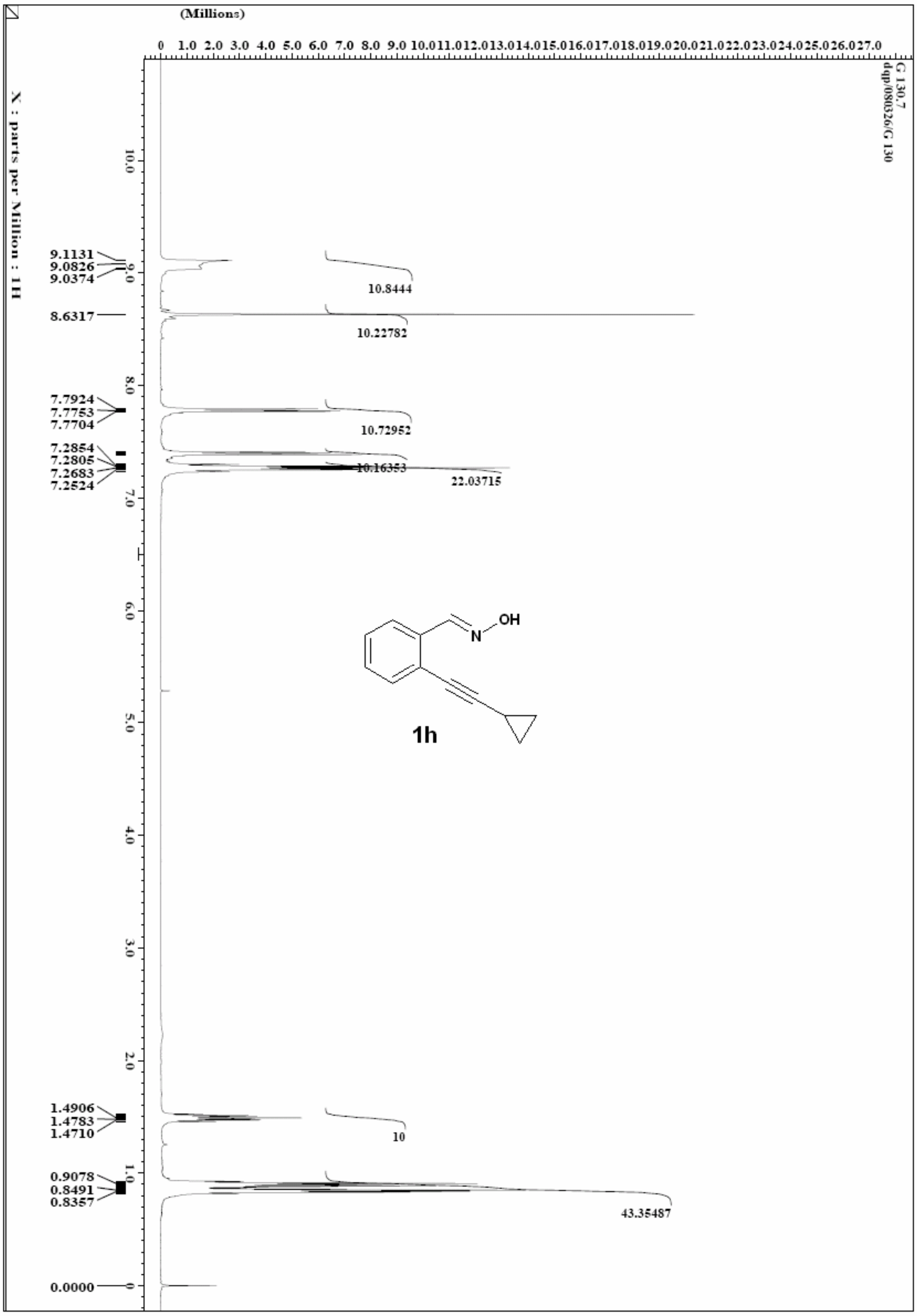
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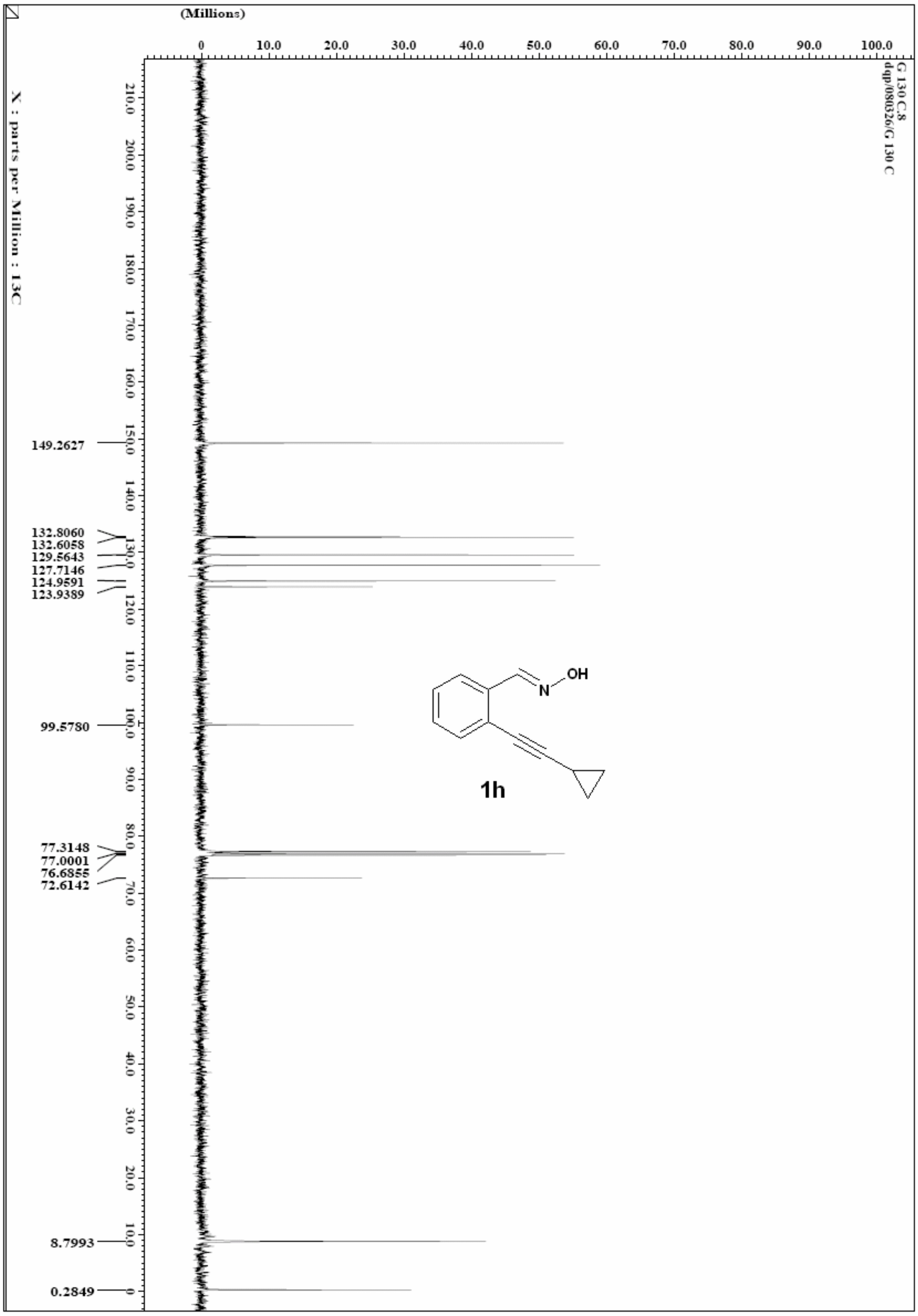
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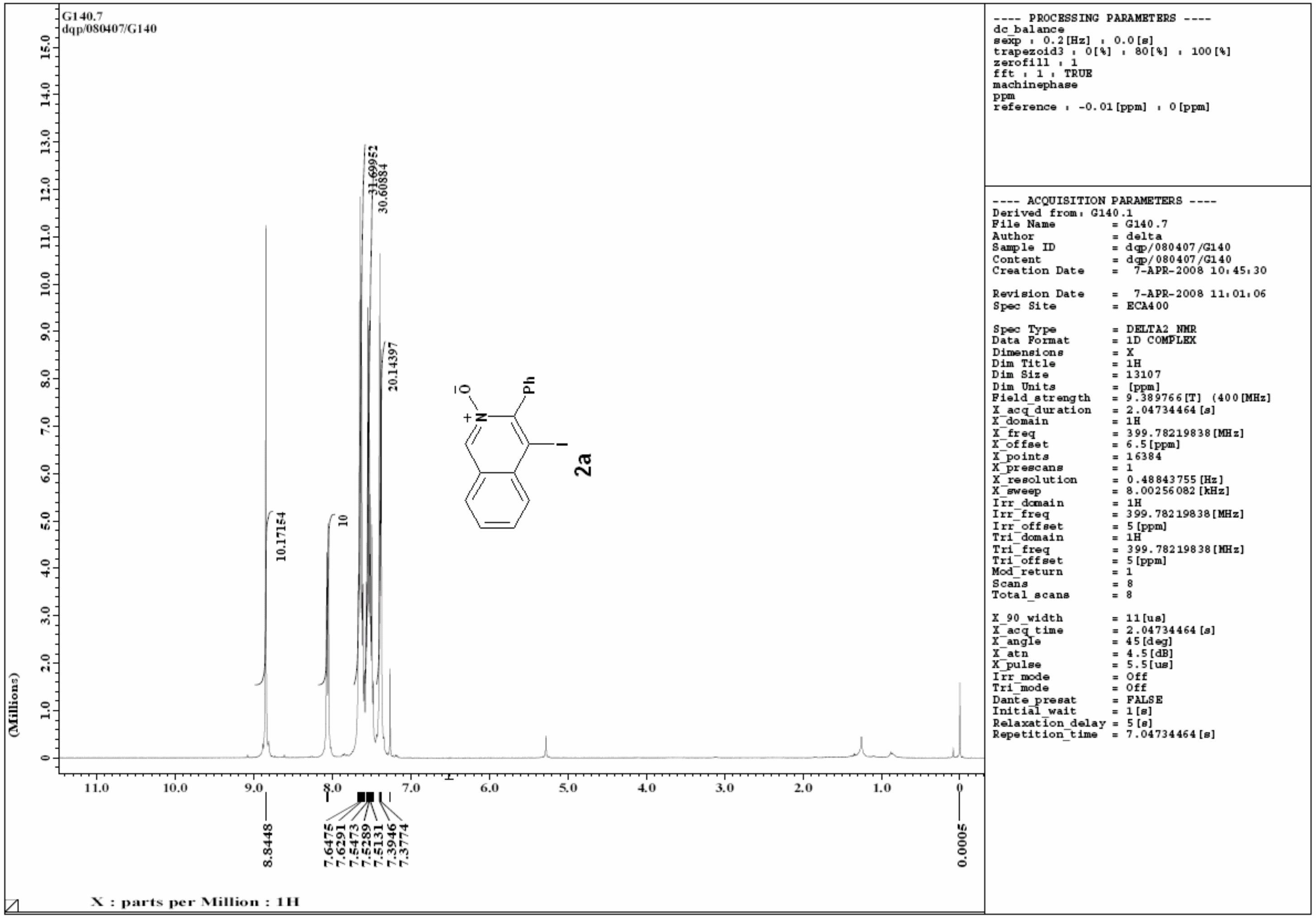
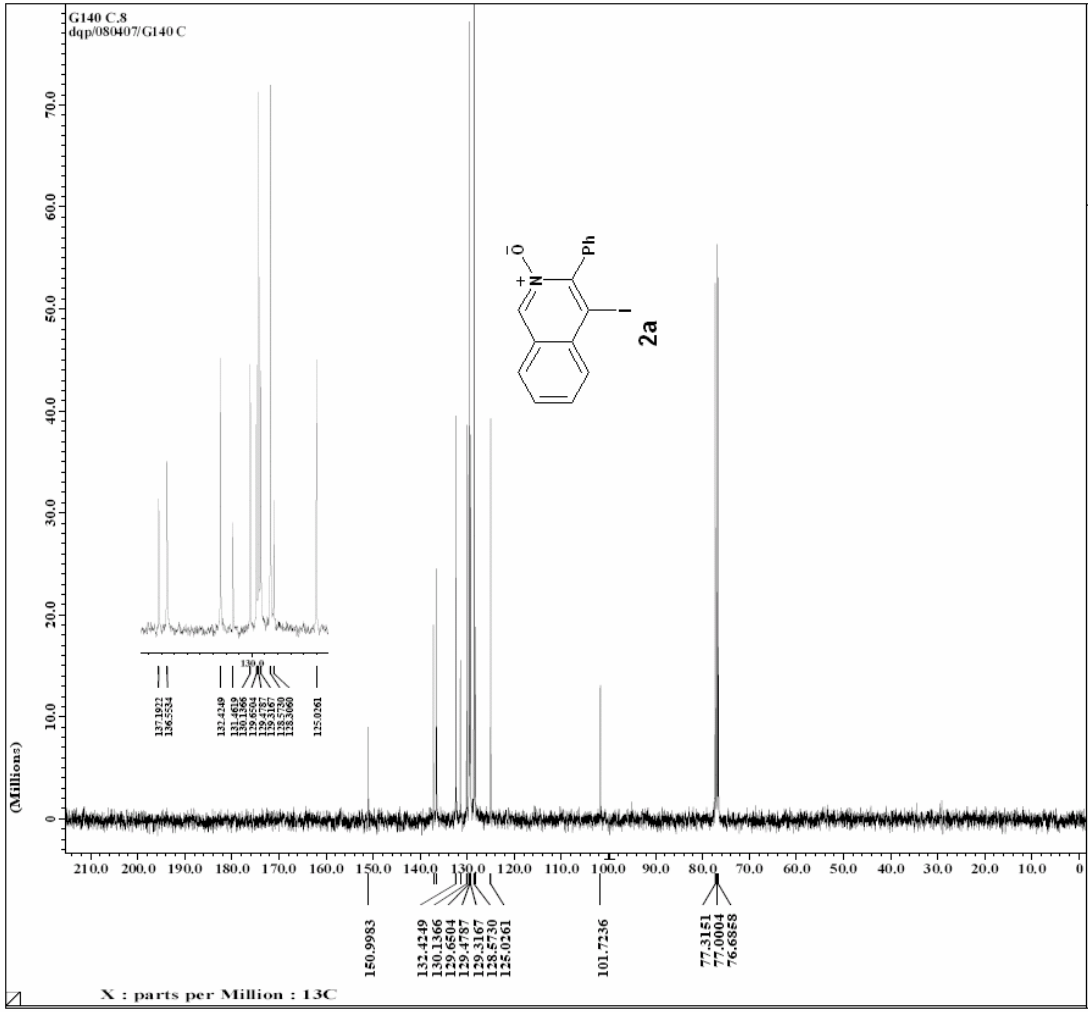
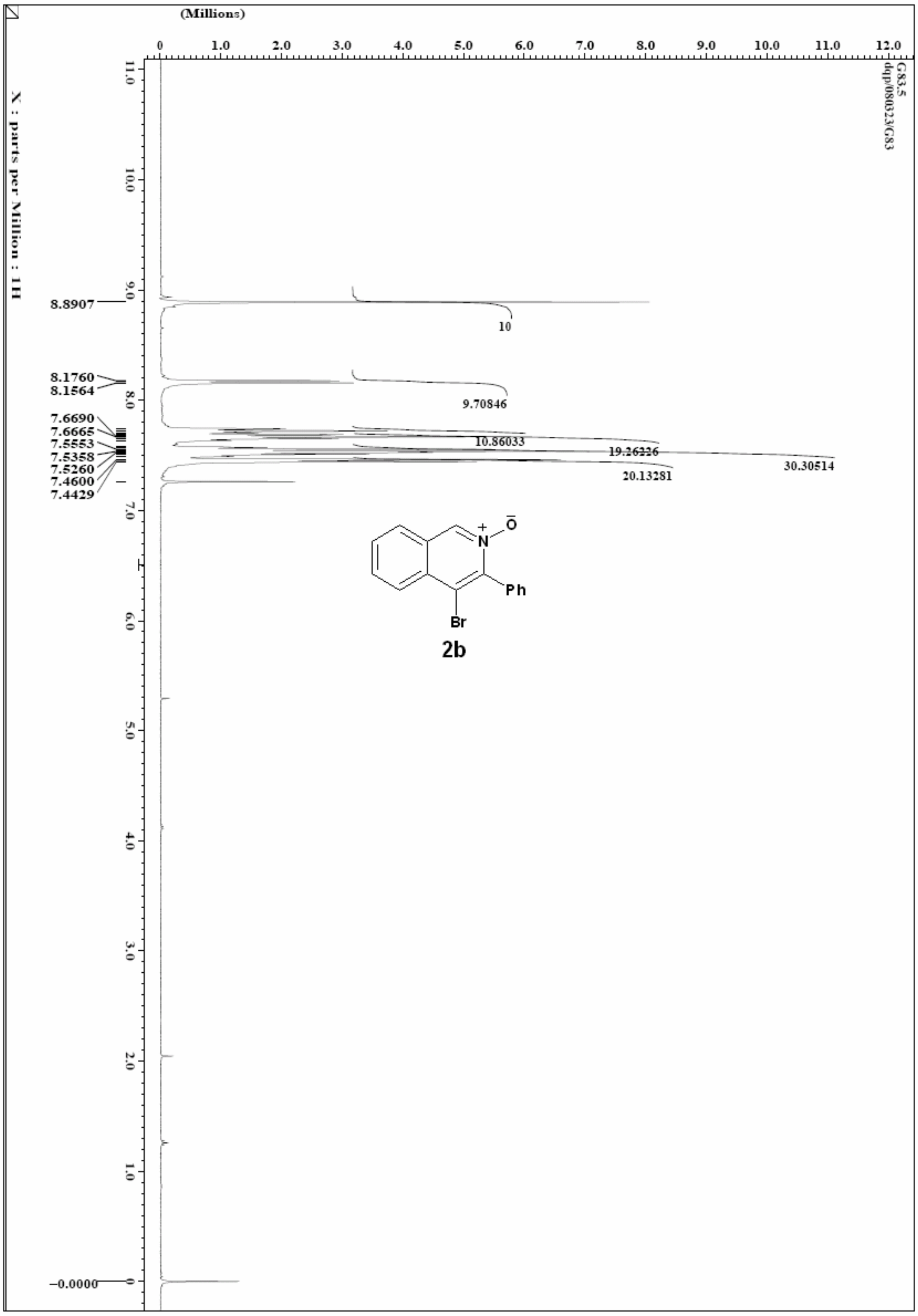
N
1h

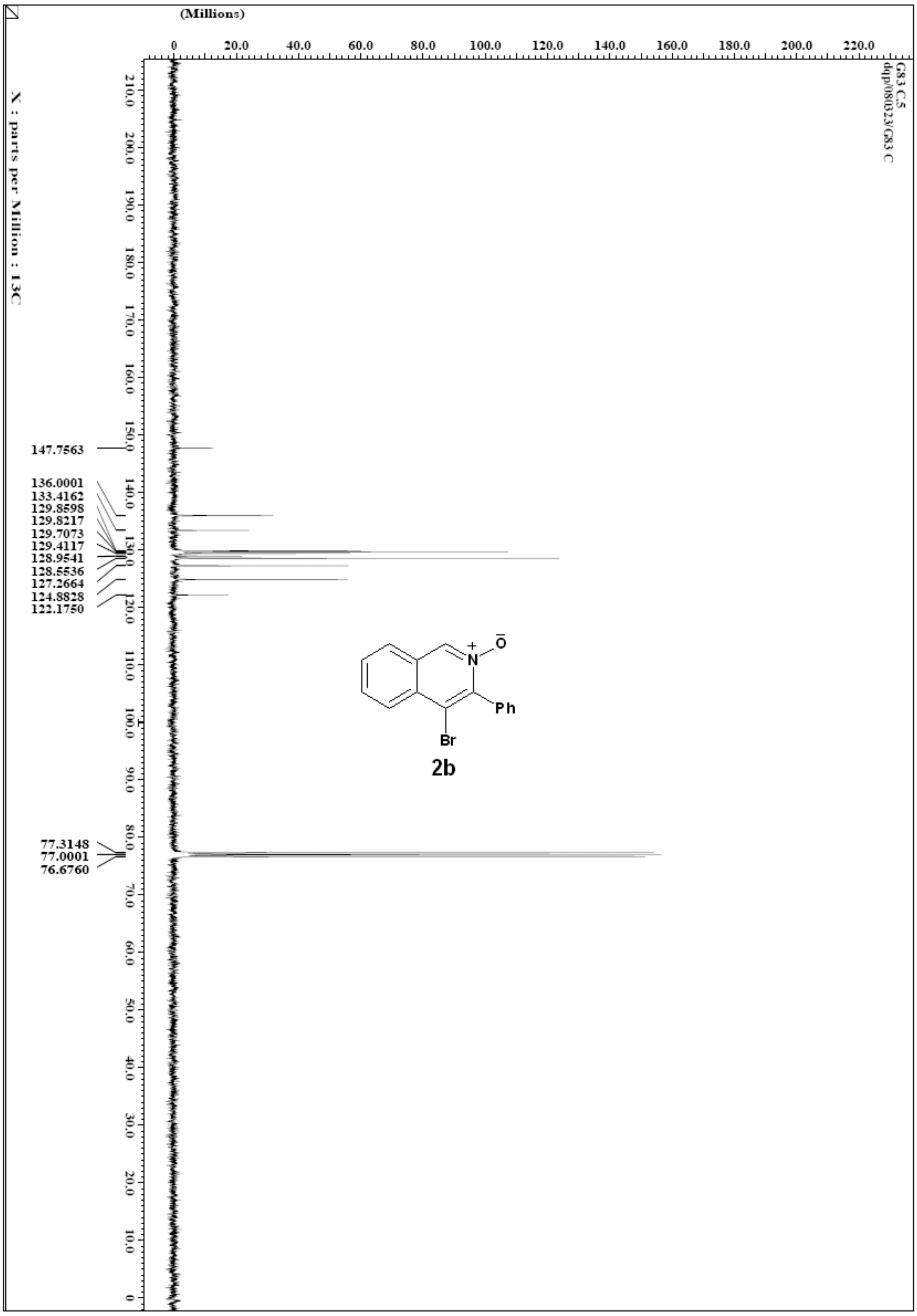

G140.7
dqp/080407/G140
(Millions)
X : parts per Million : 1H
8.8448
7.6475
7.6291
7.5473
7.5289
7.5131
7.3946
7.3774
0.0005
10.17154
10
31.69952
30.60884
20.14397
Ph
2a
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : -0.01[ppm] : 0[ppm]
---- ACQUISITION PARAMETERS ----
Derived from: G140.1
File Name = G140.7
Author = delta
Sample ID = dqp/080407/G140
Content = dqp/080407/G140
Creation Date = 7-APR-2008 10:45:30
Revision Date = 7-APR-2008 11:01:06
Spec Site = ECA400
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 = 2.04734464[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 = 8.00256082[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_return = 1
Scans = 8
Total_scans = 8
X_90_width = 11[us]
X_acq_time = 2.04734464[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 7.04734464[s]

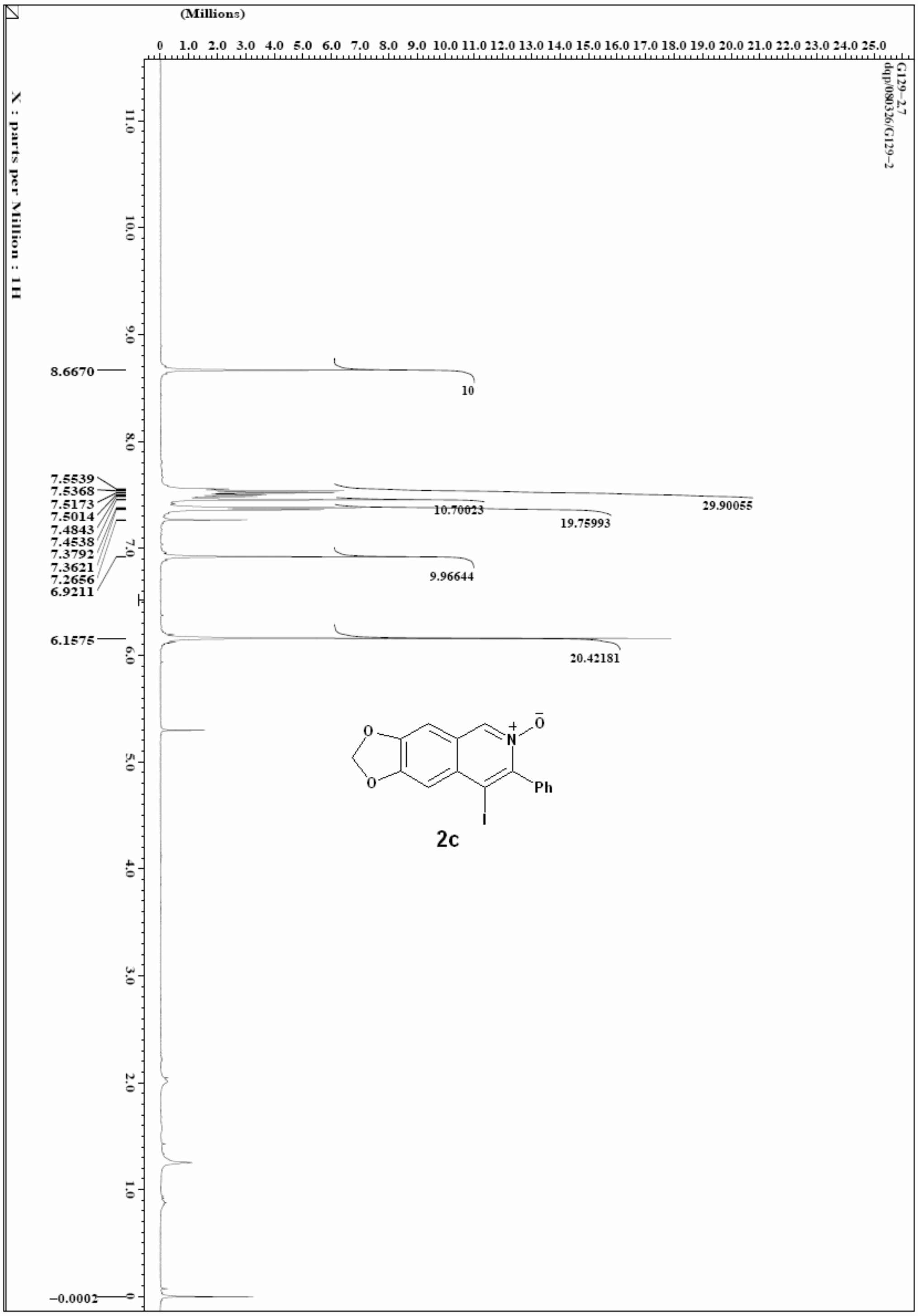



```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 77.231[ppm] : 77[ppm]
```

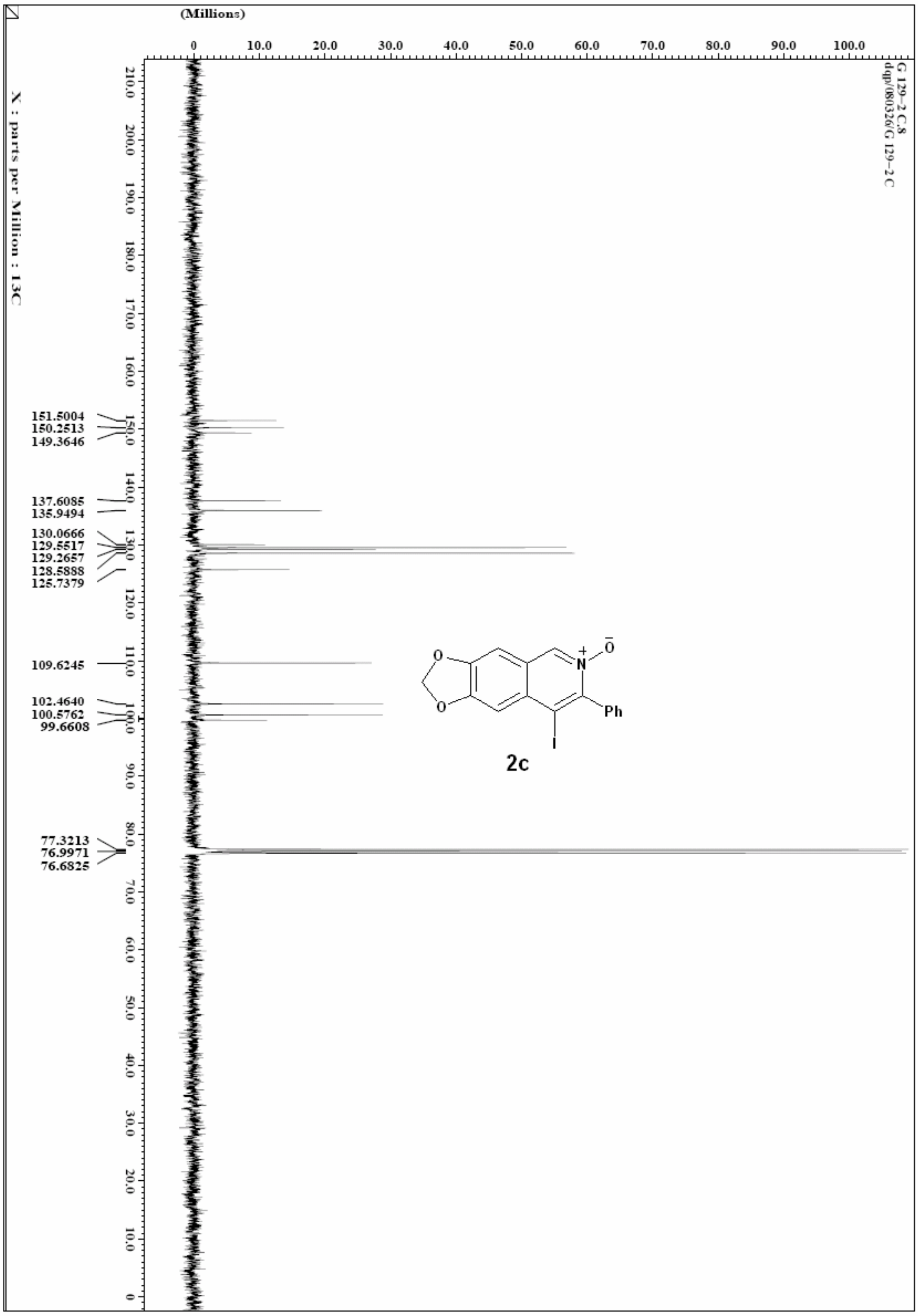
```
---- ACQUISITION PARAMETERS ----
Derived from: G140 C.1
File Name          = G140 C.8
Author             = delta
Sample ID          = dqp/080407/G140 C
Content            = dqp/080407/G140 C
Creation Date      =  7-APR-2008 10:50:50

Revision Date      =  7-APR-2008 11:09:43
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         = -41[ppm]
Mod_return         = 1
Scans              = 82
Total_scans        = 82

X_90_width         = 8.15[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 7[dB]
X_pulse            = 2.71666667[us]
Irr_atn_dec        = 24.8861[dB]
Irr_atn_noe        = 24.8861[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
G83.5
dqp080323G83
8.8907
8.1760
8.1564
7.6690
7.6665
7.5553
7.5358
7.5260
7.4600
7.4429
-0.0000
10
9.70846
10.86033
19.26226
30.30514
20.13281
Ph
Br
2b

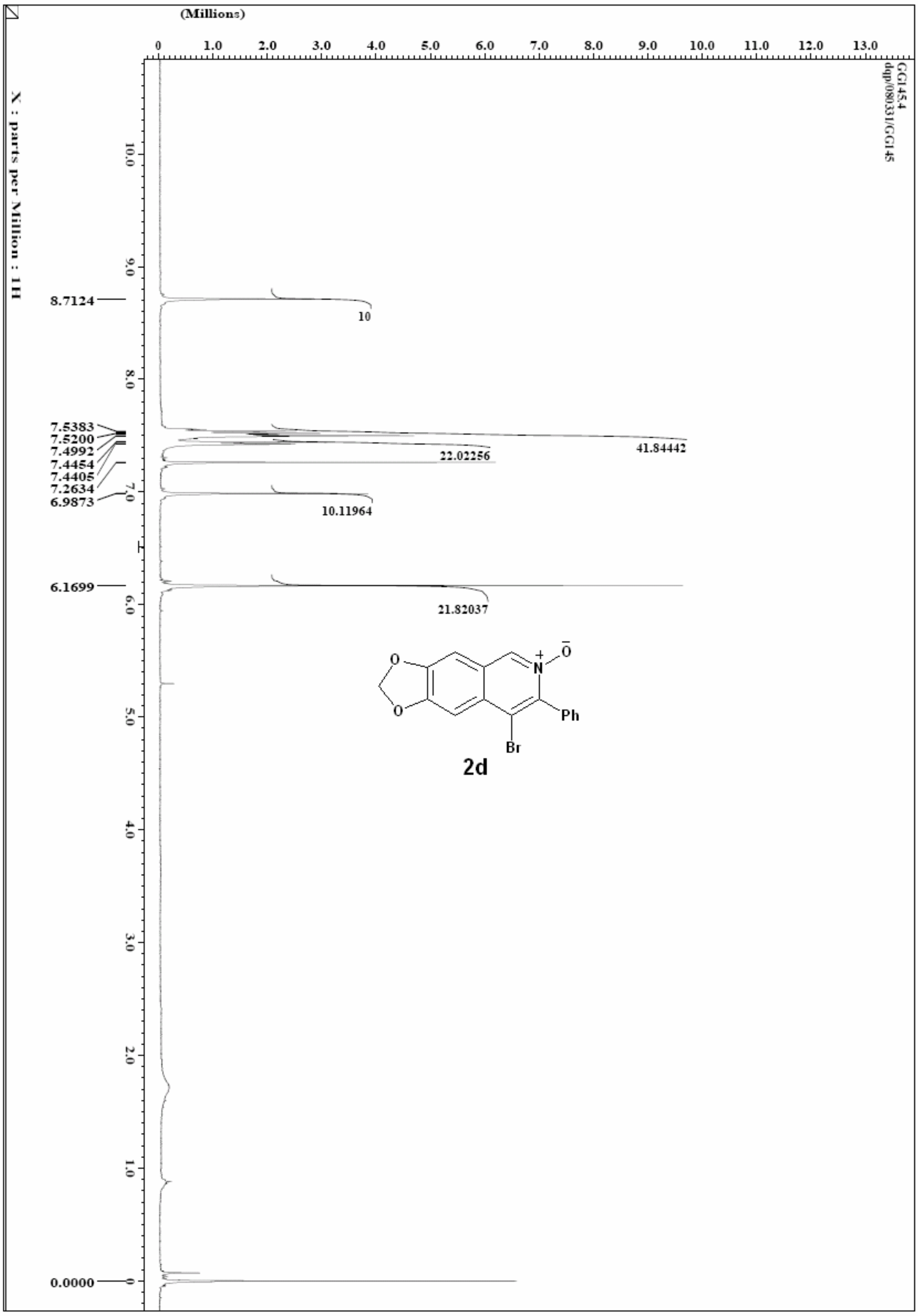

(Millions)
X : parts per Million : 13C
G83 C.5
dqp/080323/G83 C
147.7563
136.0001
133.4162
129.8598
129.8217
129.7073
129.4117
128.9541
128.5536
127.2664
124.8828
122.1750
77.3148
77.0001
76.6760
Ph
Br
2b

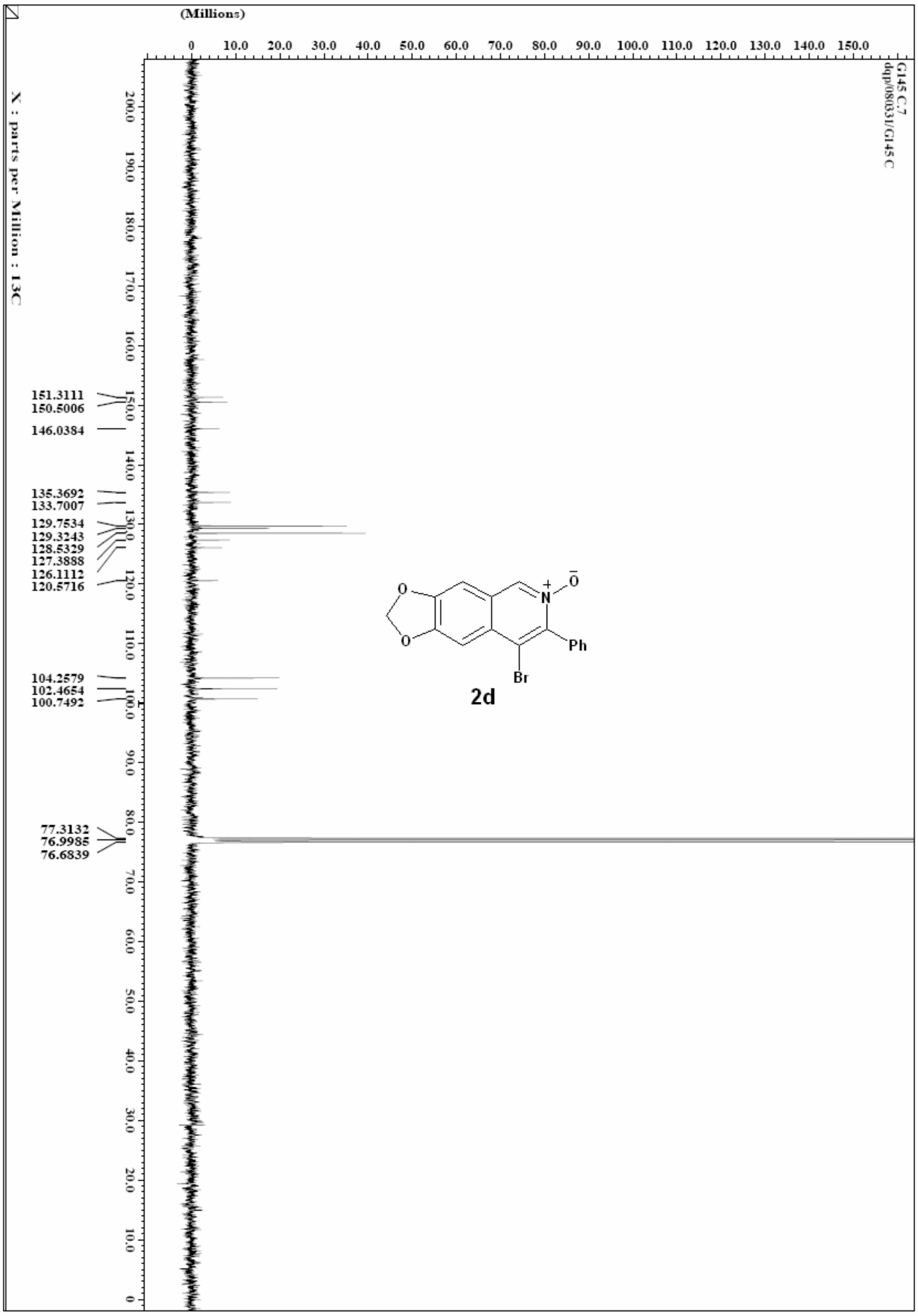


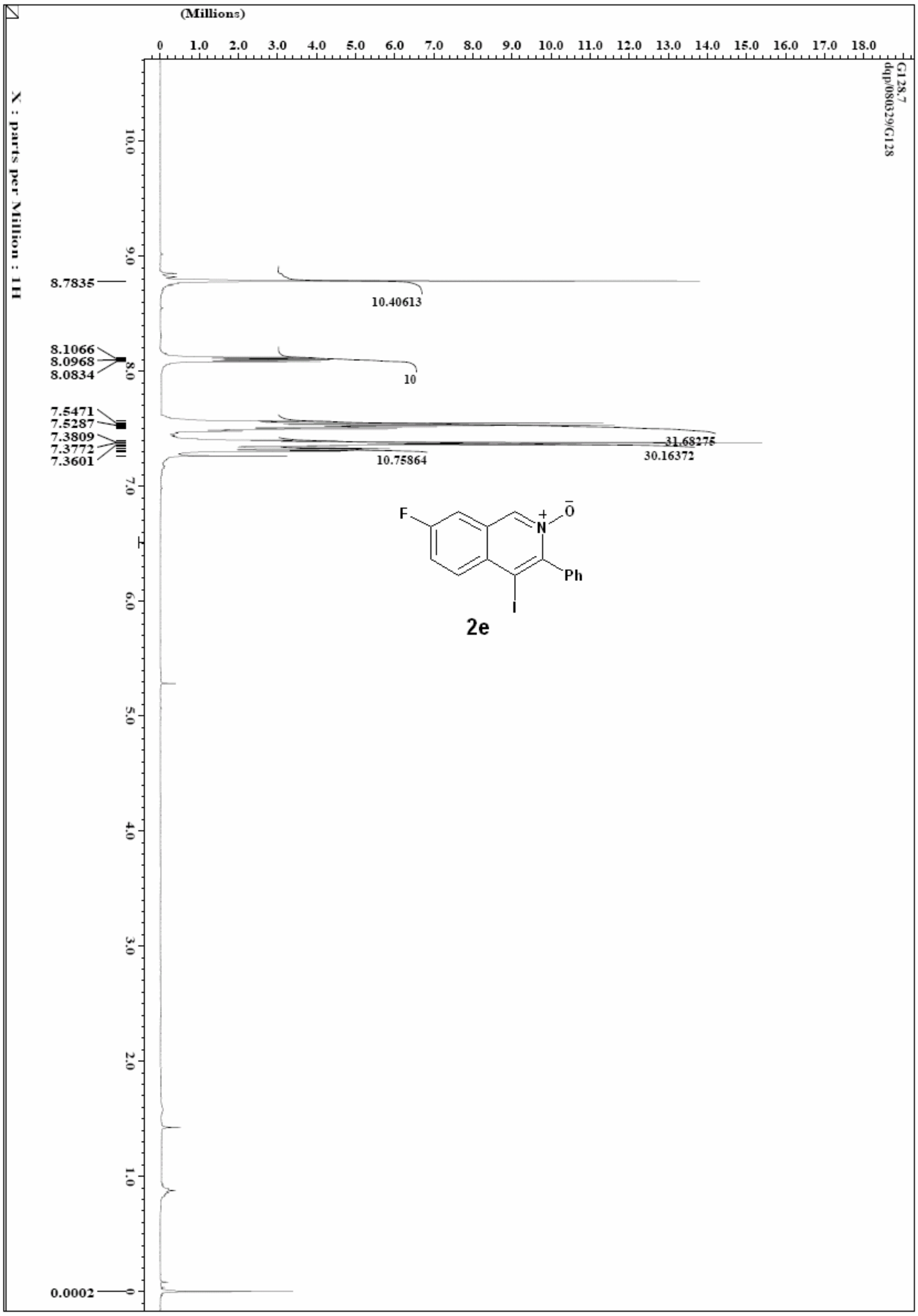
(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0
X : parts per Million : 1H
G129--2.7
dqp/080326/G129--2
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.6670
7.5539
7.5368
7.5173
7.5014
7.4843
7.4538
7.3792
7.3621
7.2656
6.9211
6.1575
-0.0002
10
10.70023
19.75993
29.90055
9.96644
20.42181
O
O
N
O
Ph
I
2c

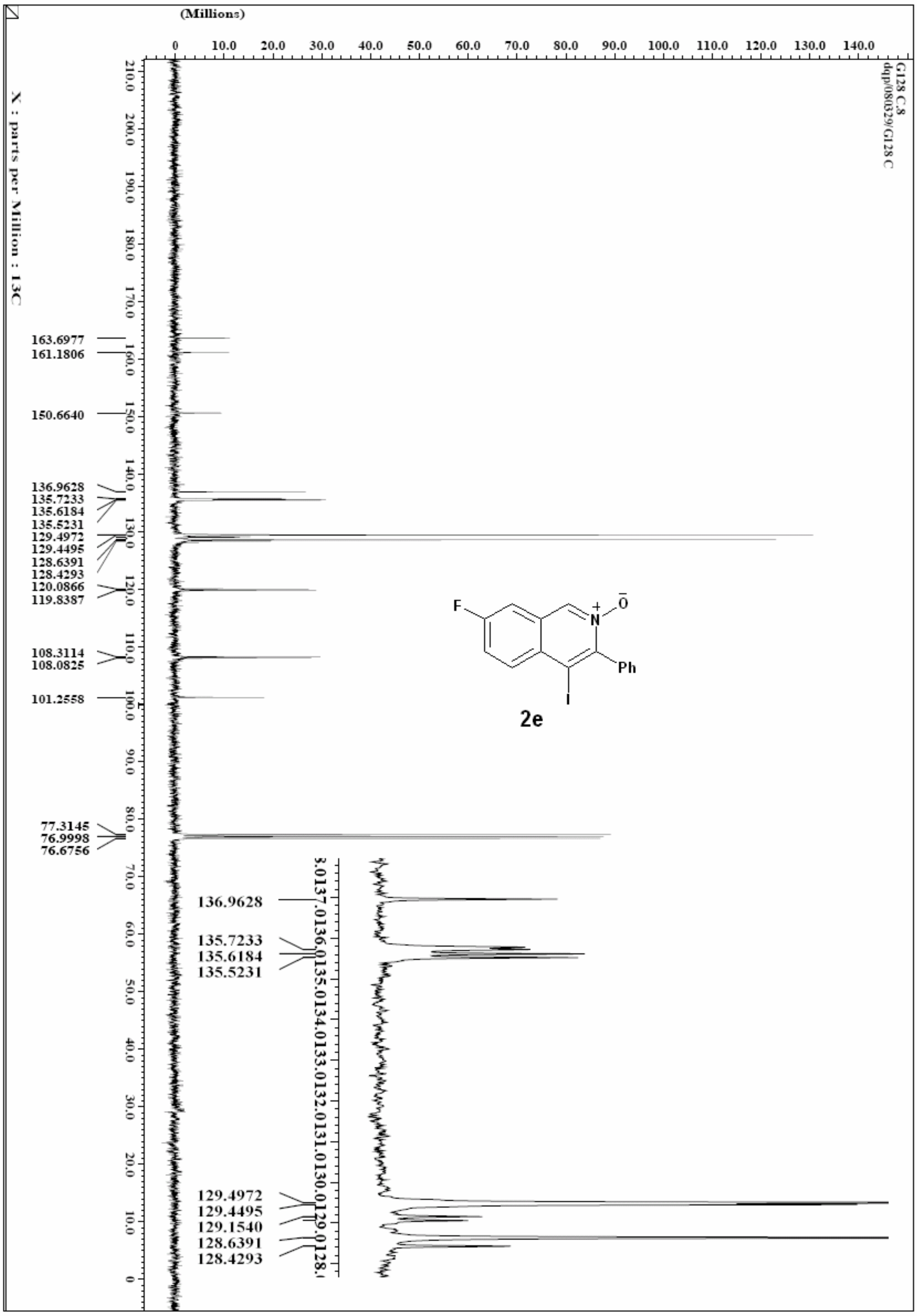


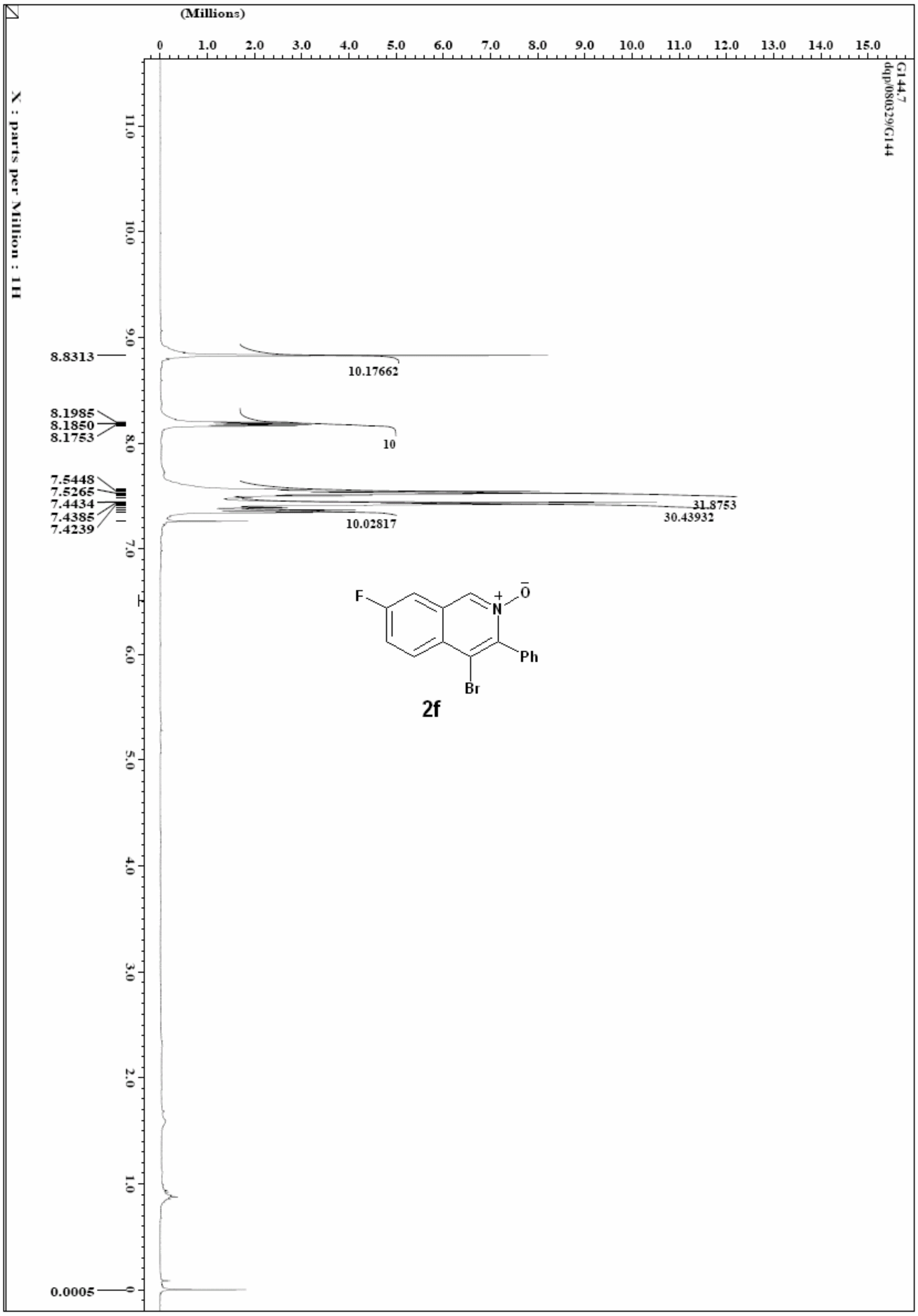
(Millions)
0 10.0 20.0 30.0 40.0 50.0 60.0 70.0 80.0 90.0 100.0
G 129-2 C.8
dqp/080326/G 129-2C
X : parts per Million : 13C
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.5004
150.2513
149.3646
137.6085
135.9494
130.0666
129.5517
129.2657
128.5888
125.7379
109.6245
102.4640
100.5762
99.6608
77.3213
76.9971
76.6825
Ph
I
2c

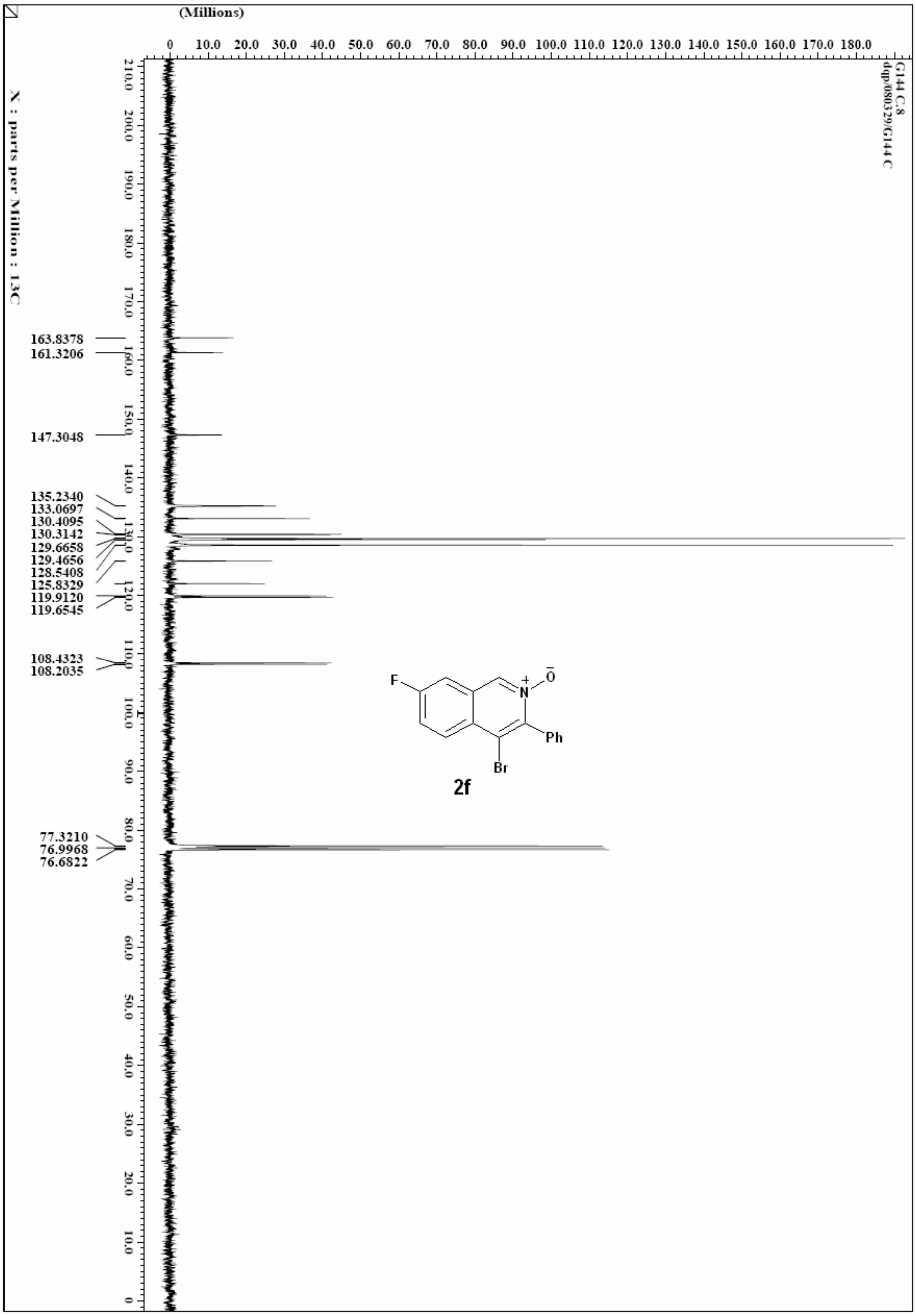

(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0
GG145.4
dup/080331/GG145
X : parts per Million : 1H
8.7124
7.5383
7.5200
7.4992
7.4454
7.4405
7.2634
6.9873
6.1699
0.0000
10
41.84442
22.02256
10.11964
21.82037
O
O
N
O
Ph
Br
2d


(Millions)
151.3111
150.5006
146.0384
135.3692
133.7007
129.7534
129.3243
128.5329
127.3888
126.1112
120.5716
104.2579
102.4654
100.7492
77.3132
76.9985
76.6839
X : parts per Million : 13C
G145 C.7
dqp/080331/G145C
Ph
Br
2d

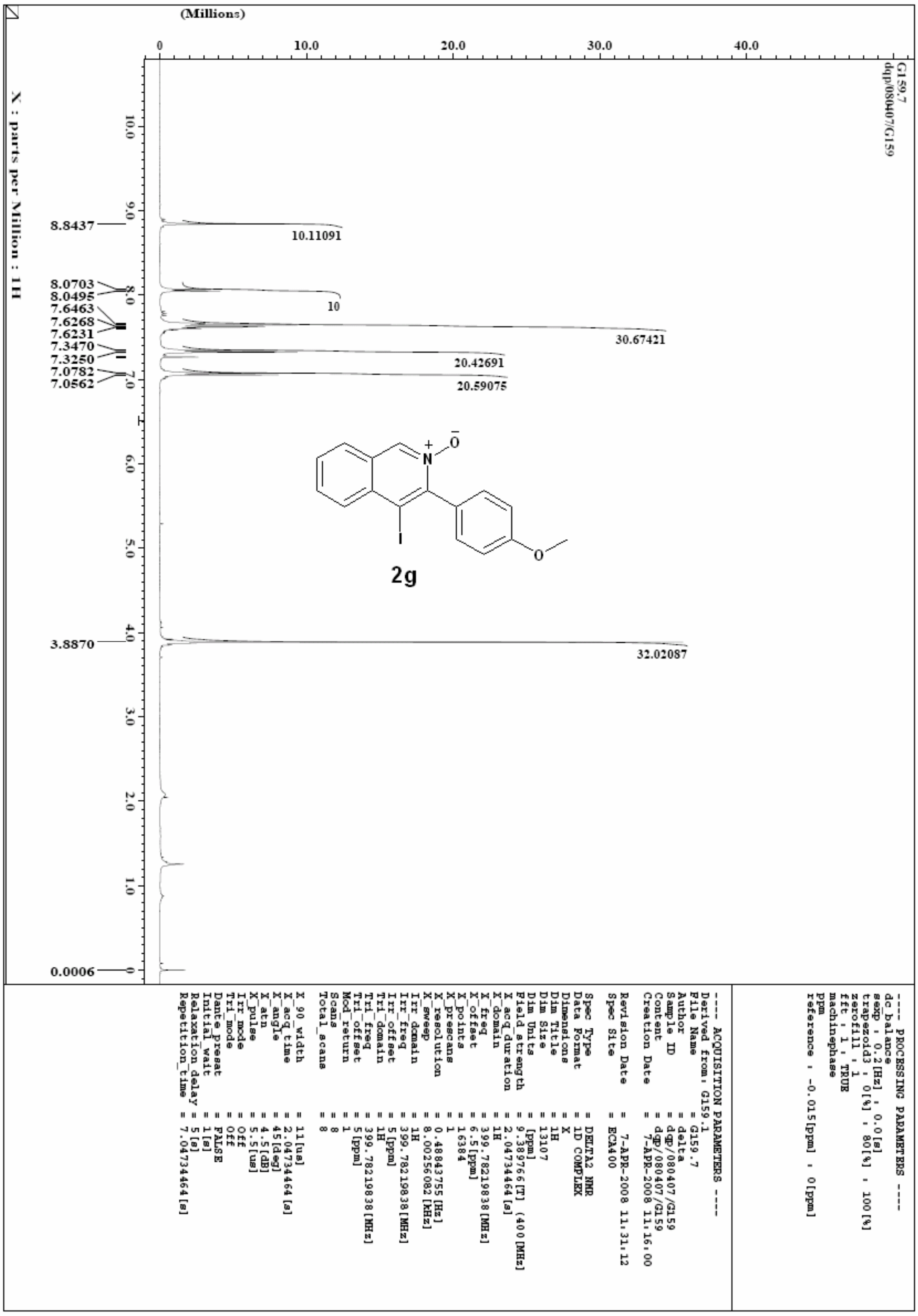

(Millions)
X : parts per Million : 1H
G128.7
dqp/080329/G128
8.7835
8.1066
8.0968
8.0834
7.5471
7.5287
7.3809
7.3772
7.3601
0.0002
10.40613
10
31.68275
30.16372
10.75864
Ph
I
F
2e

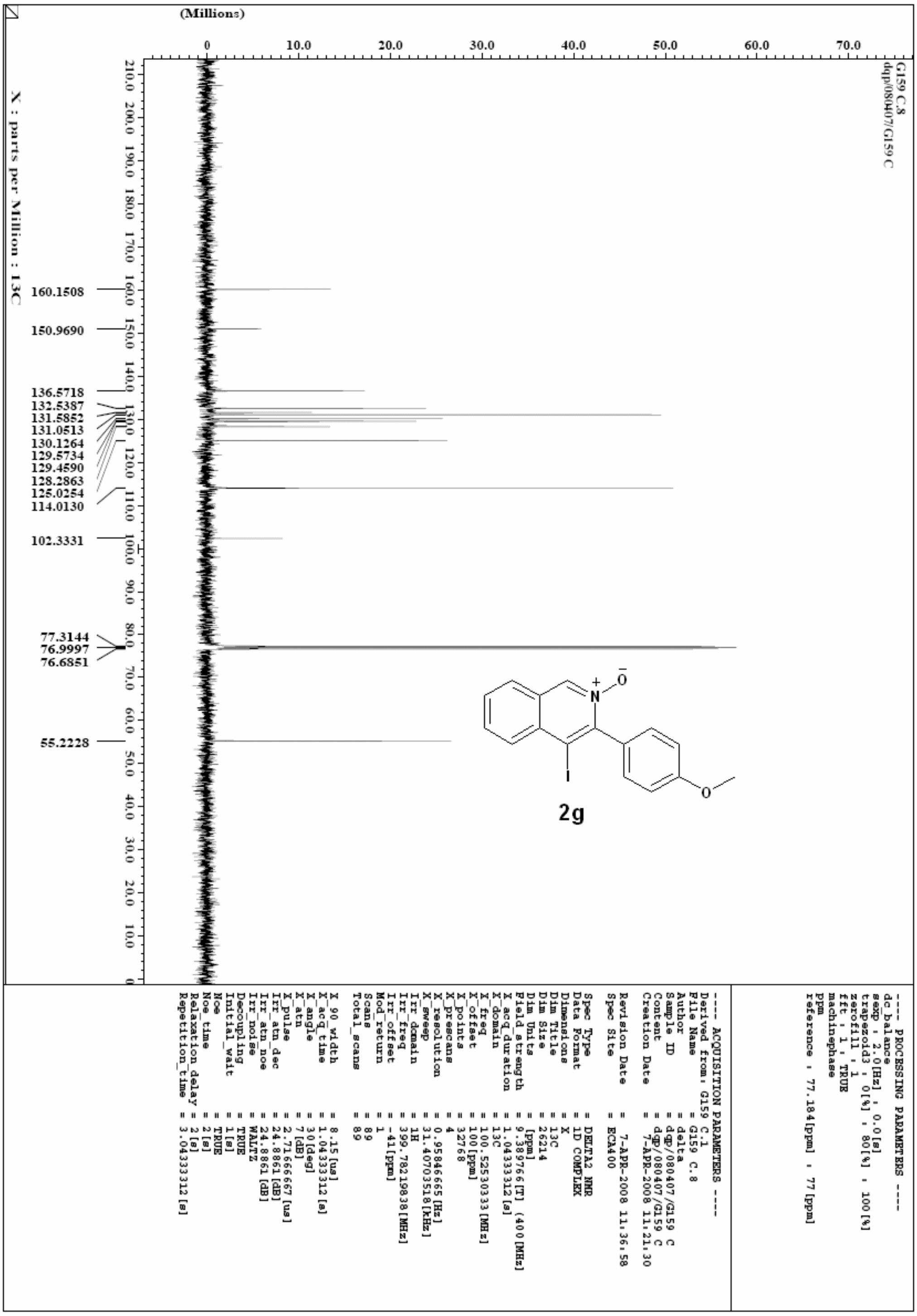

(Millions)
G128_C.8
dqp/080329/G128 C
X : parts per Million : 13C
163.6977
161.1806
150.6640
136.9628
135.7233
135.6184
135.5231
129.4972
129.4495
128.6391
128.4293
120.0866
119.8387
108.3114
108.0825
101.2558
77.3145
76.9998
76.6756
129.1540
F
Ph
I
2e

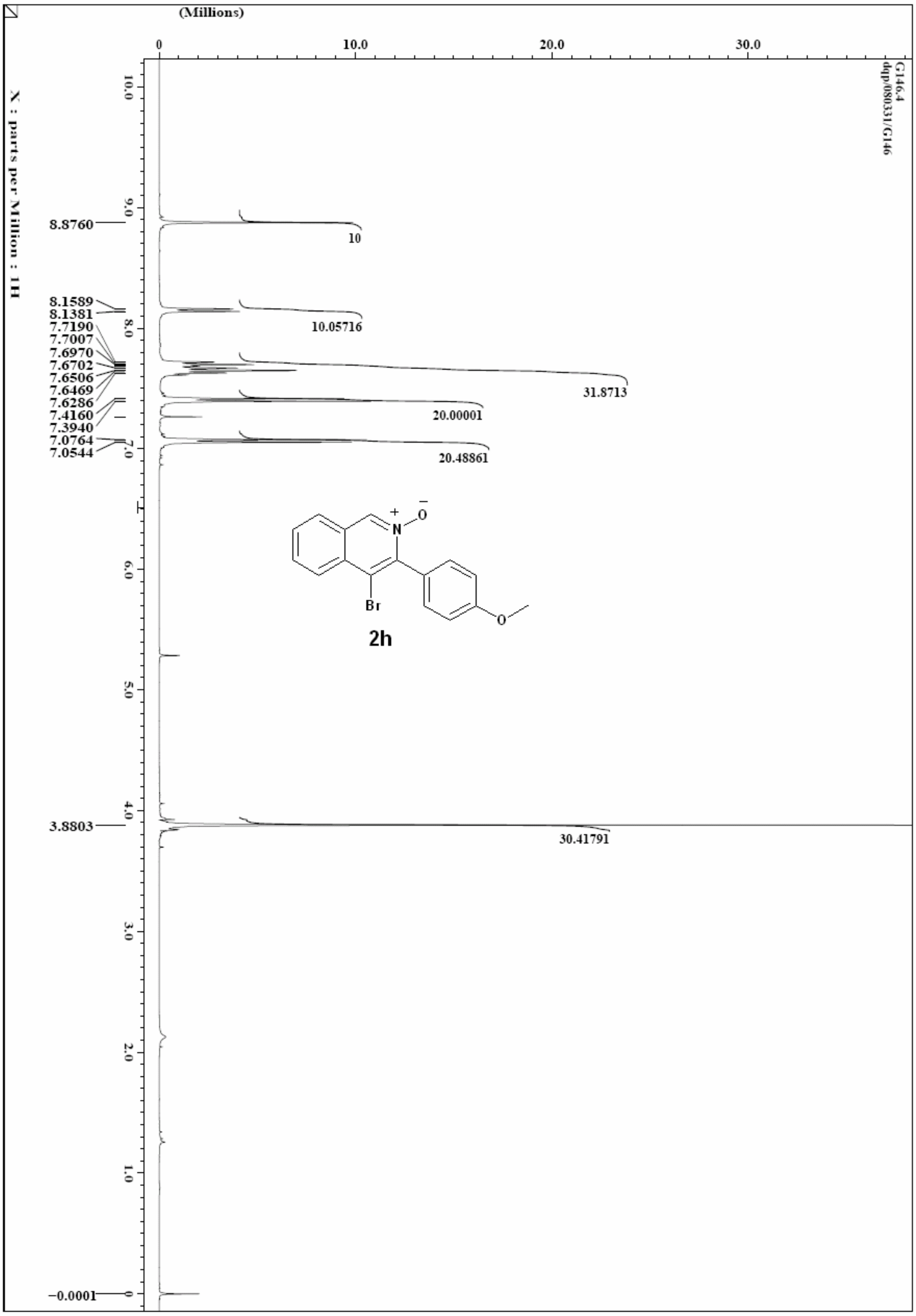

(Millions)
X : parts per Million : 1H
G144.7
dqp080329G144
8.8313
8.1985
8.1850
8.1753
7.5448
7.5265
7.4434
7.4385
7.4239
0.0005
10.17662
10
31.8753
30.43932
10.02817
F
N
O
Ph
Br
2f

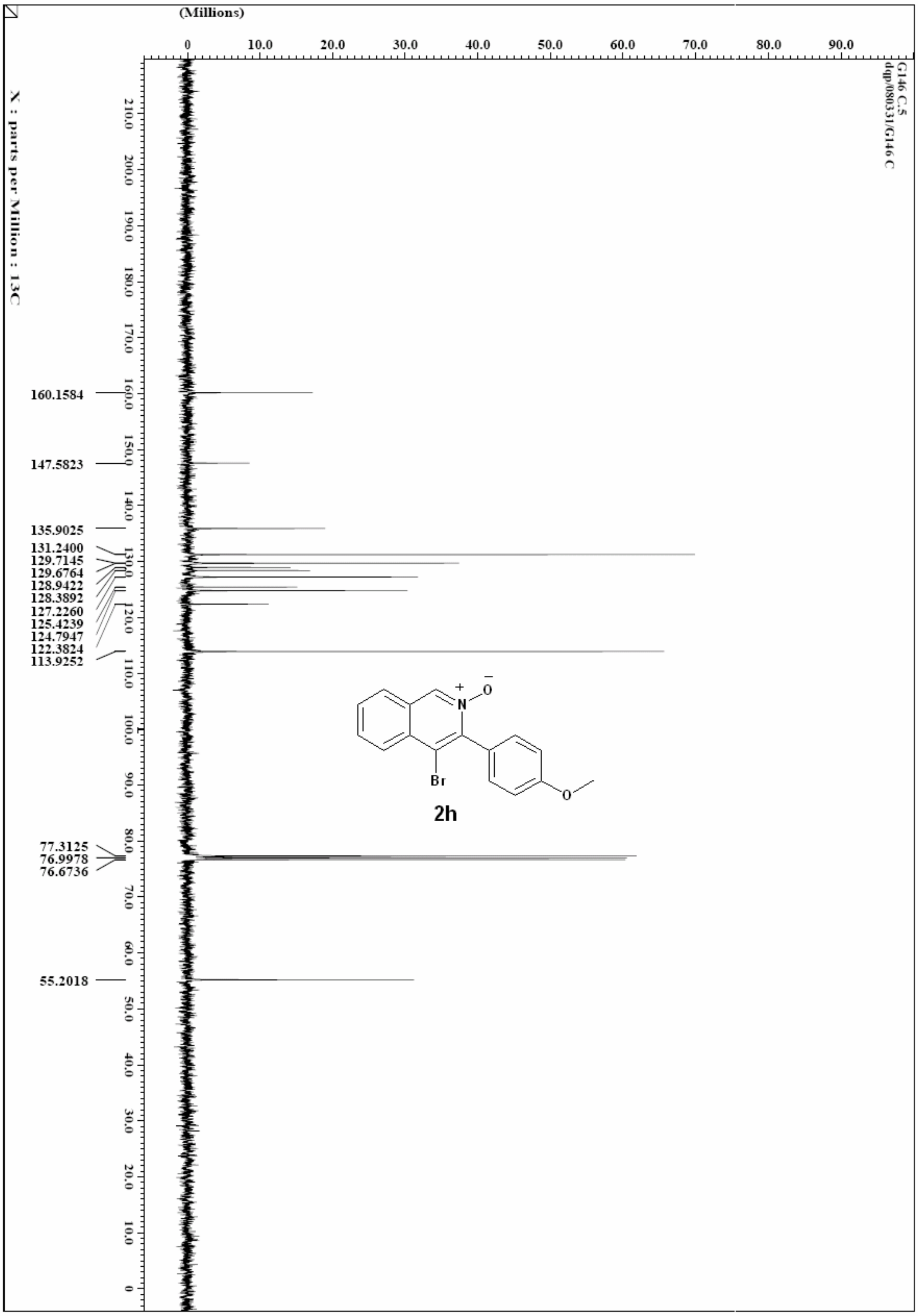


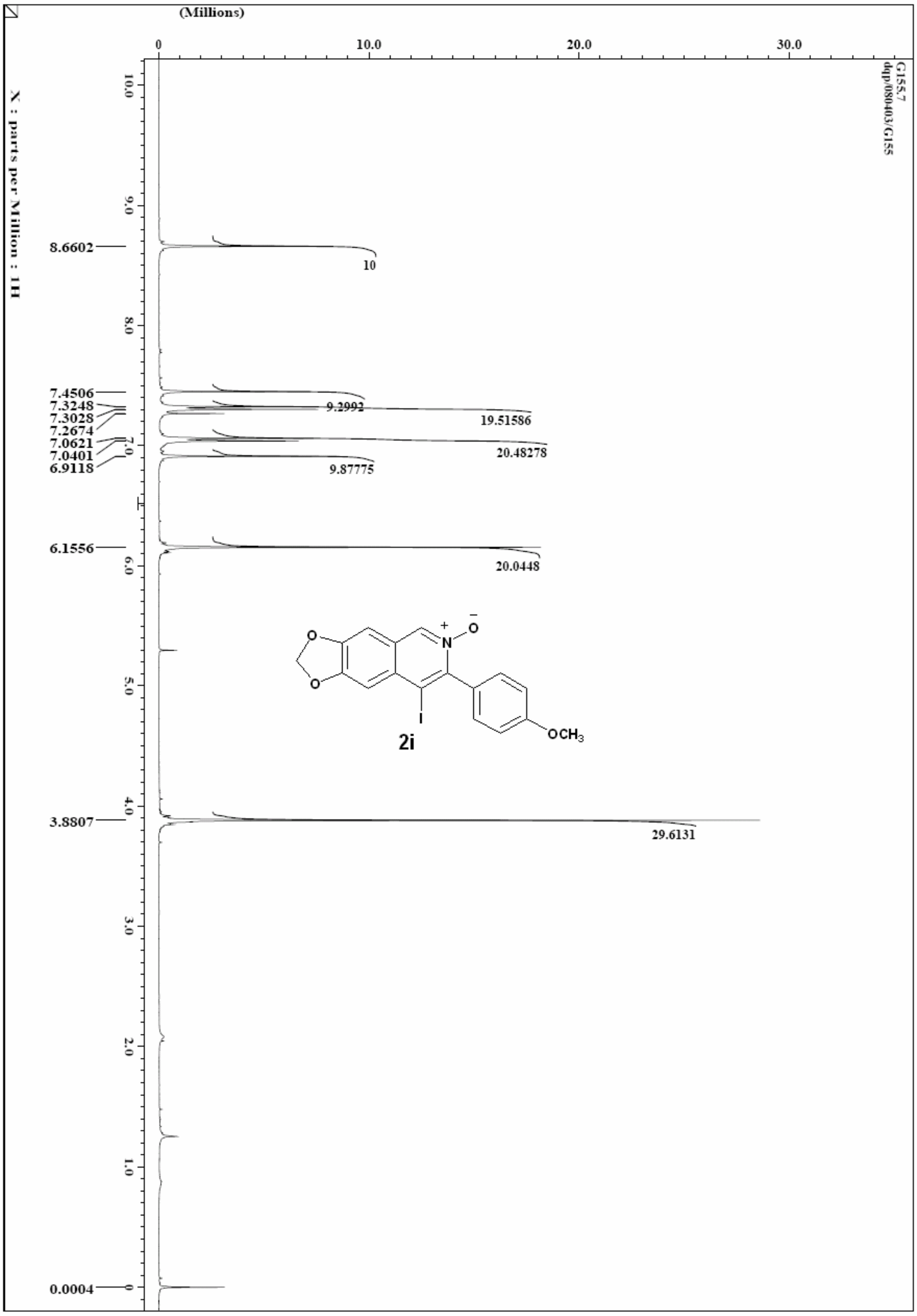
(Millions)
X : parts per Million : 13C
G144_C.8
dqp/080329/G144 C
163.8378
161.3206
147.3048
135.2340
133.0697
130.4095
130.3142
129.6658
129.4656
128.5408
125.8329
119.9120
119.6545
108.4323
108.2035
77.3210
76.9968
76.6822
F
N
O
Ph
Br
2f

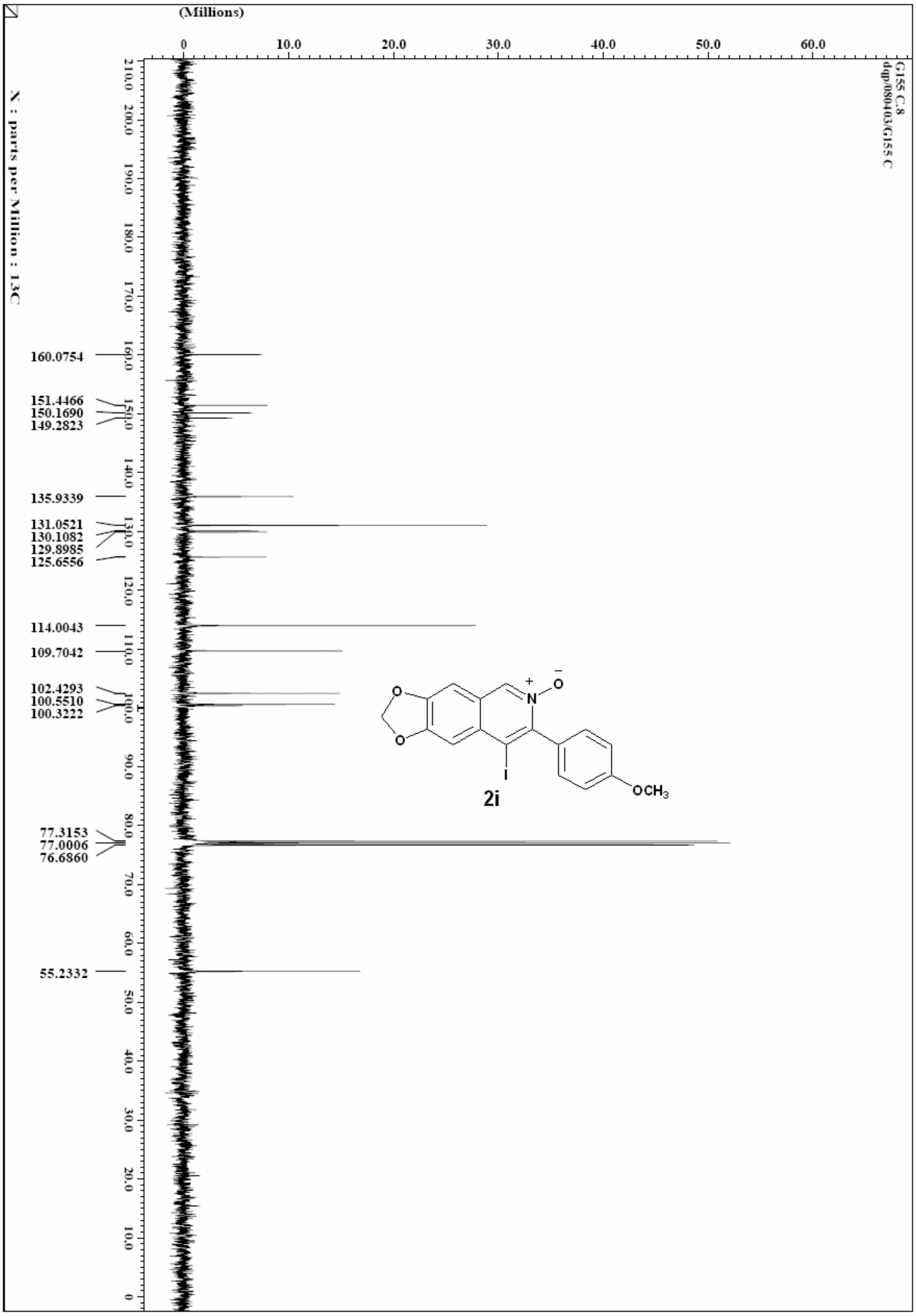

G159.7
dqp/080407/G159
(Millions)
0
10.0
20.0
30.0
40.0
10.11091
10
30.67421
20.42691
20.59075
32.02087
2g
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.8437
8.0703
8.0495
7.6463
7.6268
7.6231
7.3470
7.3250
7.0782
7.0562
3.8870
0.0006
X : parts per Million : 1H
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : -0.015[ppm] : 0[ppm]
---- ACQUISITION PARAMETERS ----
Derived from: G159.1
File Name = G159.7
Author = delta
Sample ID = dqp/080407/G159
Content = dqp/080407/G159
Creation Date = 7-APR-2008 11:16:00
Revision Date = 7-APR-2008 11:31:12
Spec Site = ECA400
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 = 2.04734464[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 = 8.00256082[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_return = 1
Scans = 8
Total_scans = 8
X_90_width = 11[us]
X_acq_time = 2.04734464[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 7.04734464[s]

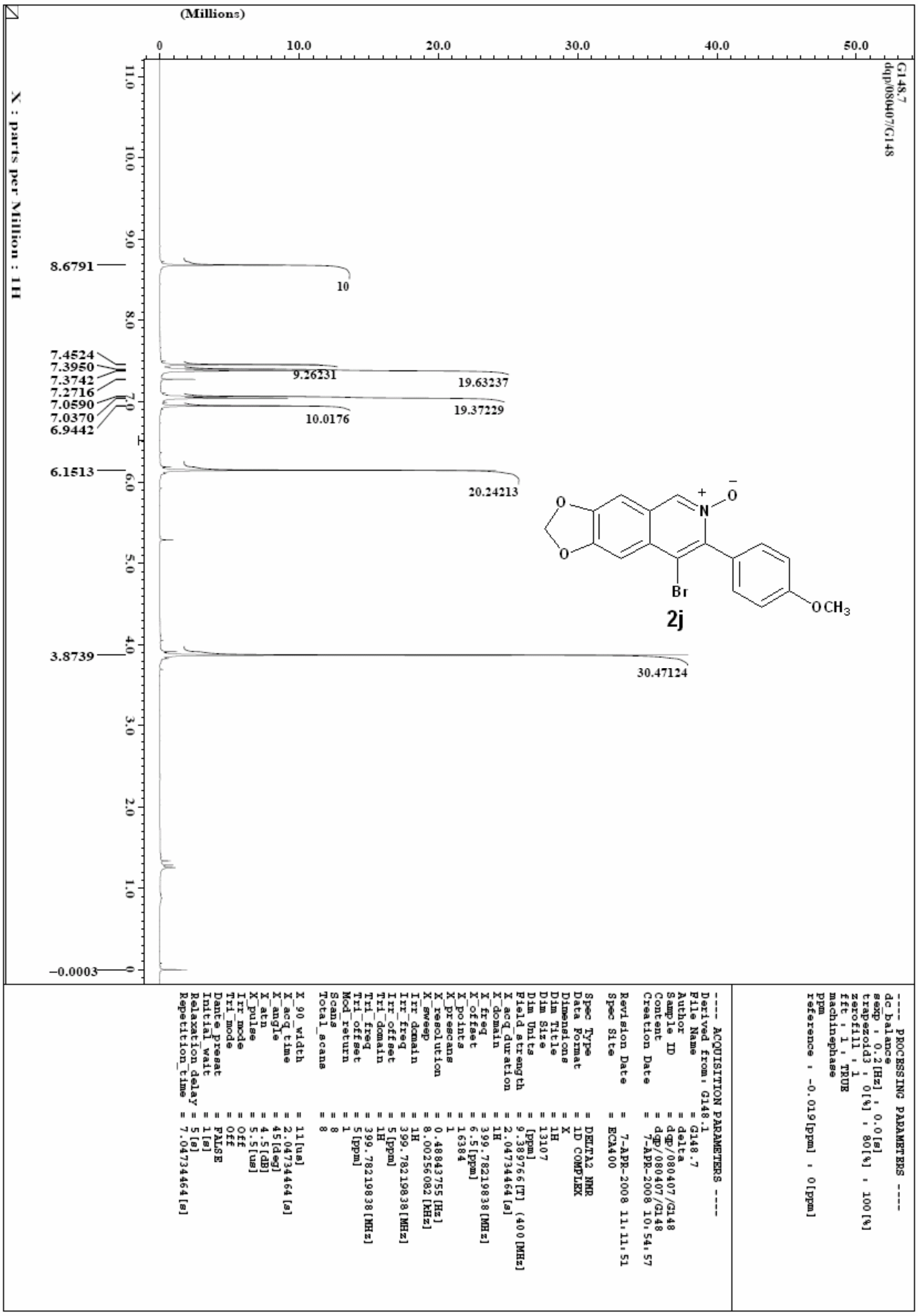

G159 C.8
dqp/080407/G159 C
(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
X : parts per Million : 13C
160.1508
150.9690
136.5718
132.5387
131.5852
131.0513
130.1264
129.5734
129.4590
128.2863
125.0254
114.0130
102.3331
77.3144
76.9997
76.6851
55.2228
2g
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 77.184[ppm] : 77 [ppm]
---- ACQUISITION PARAMETERS ----
Derived from: G159 C.1
File Name = G159 C.8
Author = delta
Sample ID = dqp/080407/G159 C
Content = dqp/080407/G159 C
Creation Date = 7-APR-2008 11:21:30
Revision Date = 7-APR-2008 11:36:58
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 = -41[ppm]
Mod_return = 1
Scans = 89
Total_scans = 89
X_90_width = 8.15[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 7[dB]
X_pulse = 2.71666667[us]
Irr_atn_dec = 24.8861[dB]
Irr_atn_noe = 24.8861[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]

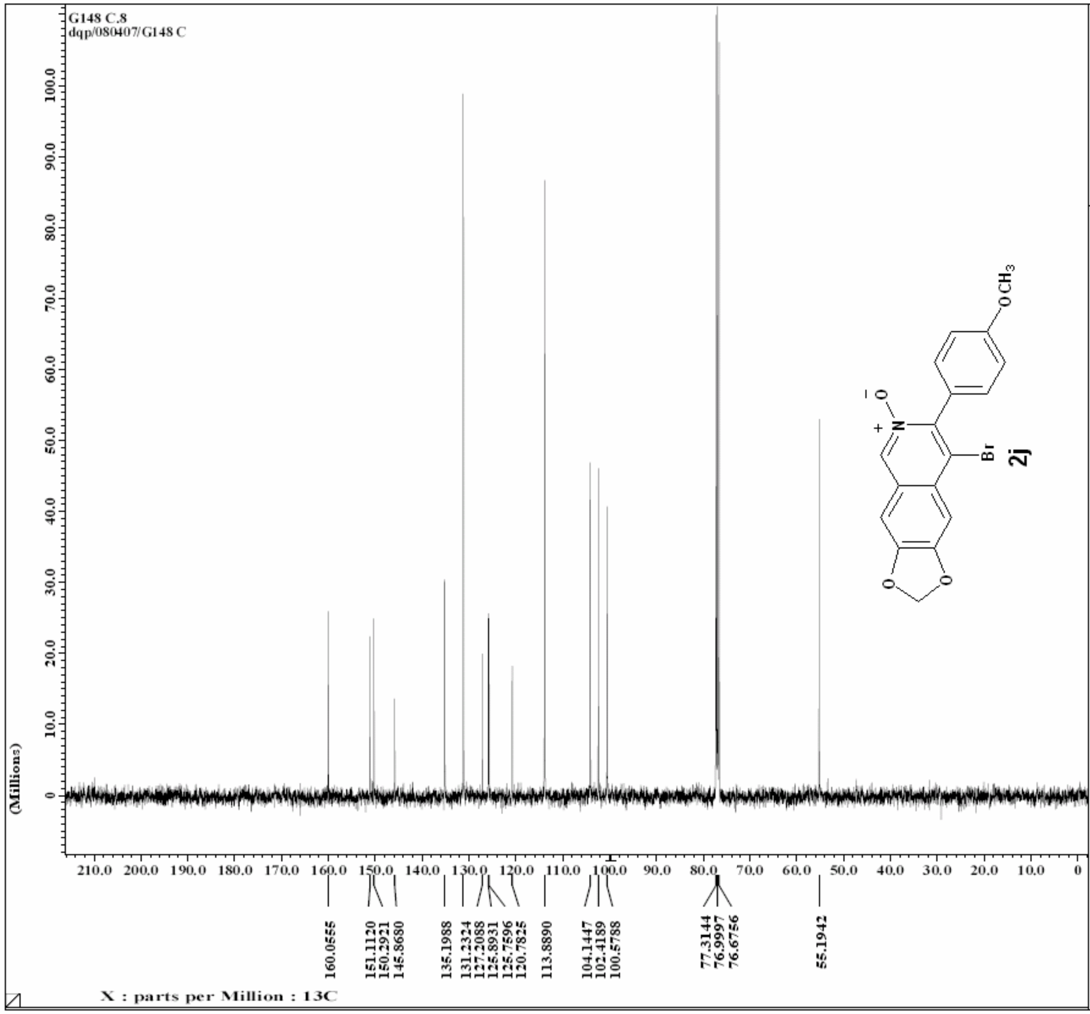

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
G146.4
dup/080331/G146
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.8760
8.1589
8.1381
7.7190
7.7007
7.6970
7.6702
7.6506
7.6469
7.6286
7.4160
7.3940
7.0764
7.0544
3.8803
-0.0001
10
10.05716
31.8713
20.00001
20.48861
30.41791
Br
N
O
2h


(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
G146 C.5
dup/080331/G146 C
160.1584
147.5823
135.9025
131.2400
129.7145
129.6764
128.9422
128.3892
127.2260
125.4239
124.7947
122.3824
113.9252
77.3125
76.9978
76.6736
55.2018
Br
2h


(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
G155.7
dup/080403/G155
8.6602
7.4506
7.3248
7.3028
7.2674
7.0621
7.0401
6.9118
6.1556
3.8807
0.0004
10
9.2992
19.51586
20.48278
9.87775
20.0448
29.6131
2i
OCH3
I

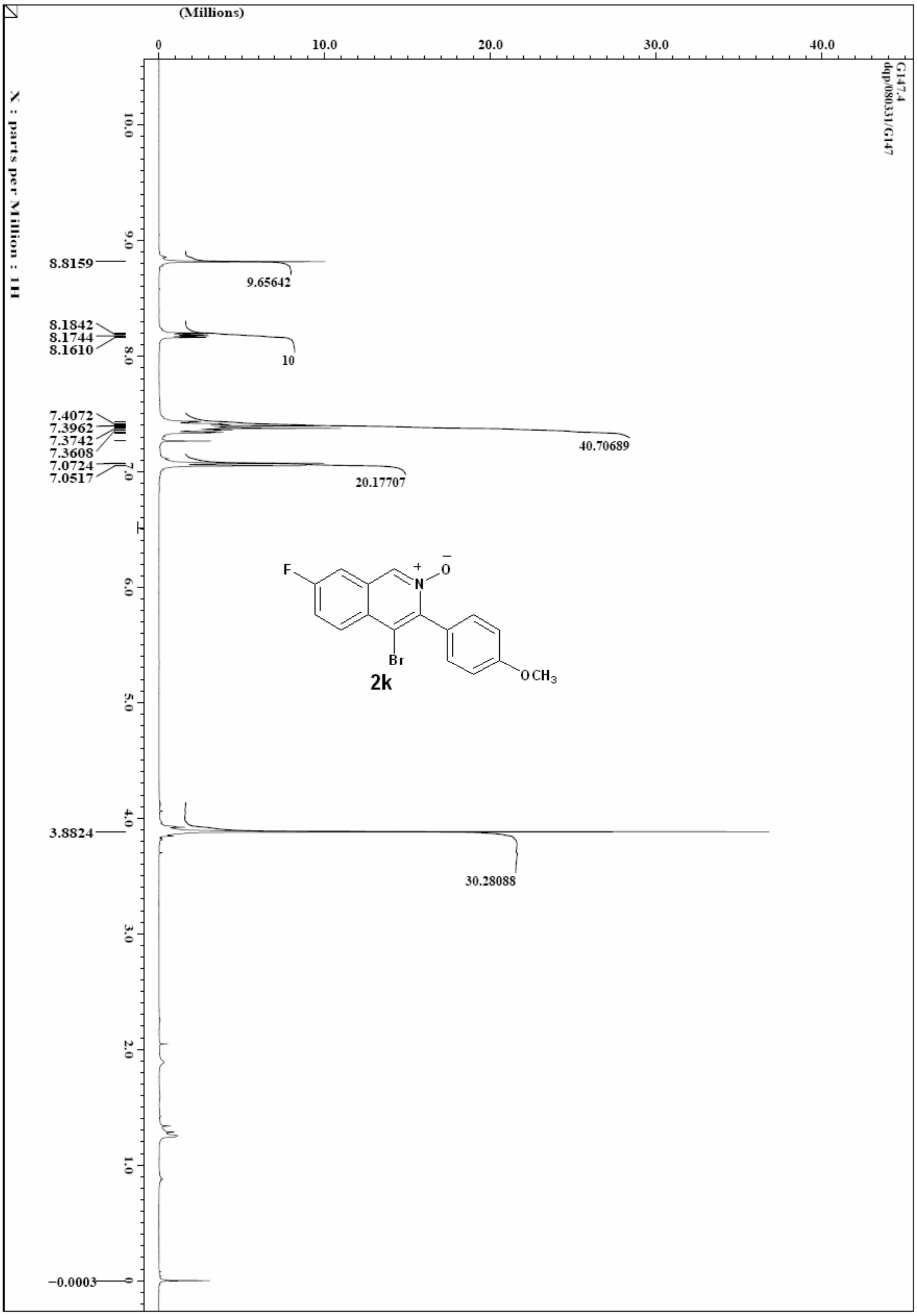

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
G155 C.8
dqp/080403/G155 C
X : parts per Million : 13C
160.0754
151.4466
150.1690
149.2823
135.9339
131.0521
130.1082
129.8985
125.6556
114.0043
109.7042
102.4293
100.5510
100.3222
77.3153
77.0006
76.6860
55.2332
OCH3
I
2i

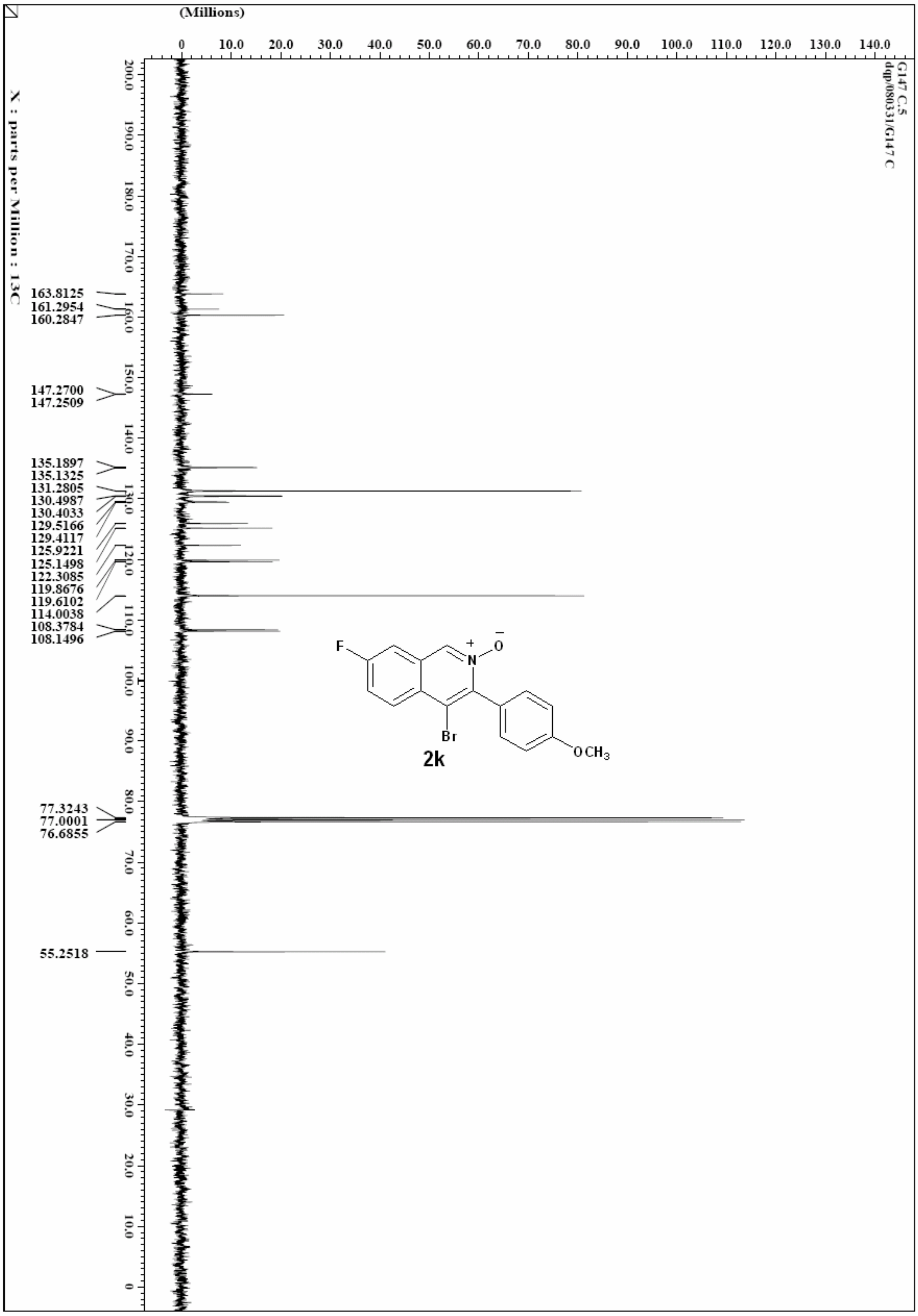

G148.7
dqp/080407/G148
(Millions)
X : parts per Million : 1H
8.6791
7.4524
7.3950
7.3742
7.2716
7.0590
7.0370
6.9442
6.1513
3.8739
-0.0003
10
9.26231
19.63237
19.37229
10.0176
20.24213
30.47124
Br
OCH3
2j
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : -0.019[ppm] : 0[ppm]
---- ACQUISITION PARAMETERS ----
Derived from: G148.1
File Name = G148.7
Author = delta
Sample ID = dqp/080407/G148
Content = dqp/080407/G148
Creation Date = 7-APR-2008 10:54:57
Revision Date = 7-APR-2008 11:11:51
Spec Site = ECA400
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 = 2.04734464[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 = 8.00256082[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_return = 1
Scans = 8
Total_scans = 8
X_90_width = 11[us]
X_acq_time = 2.04734464[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 7.04734464[s]

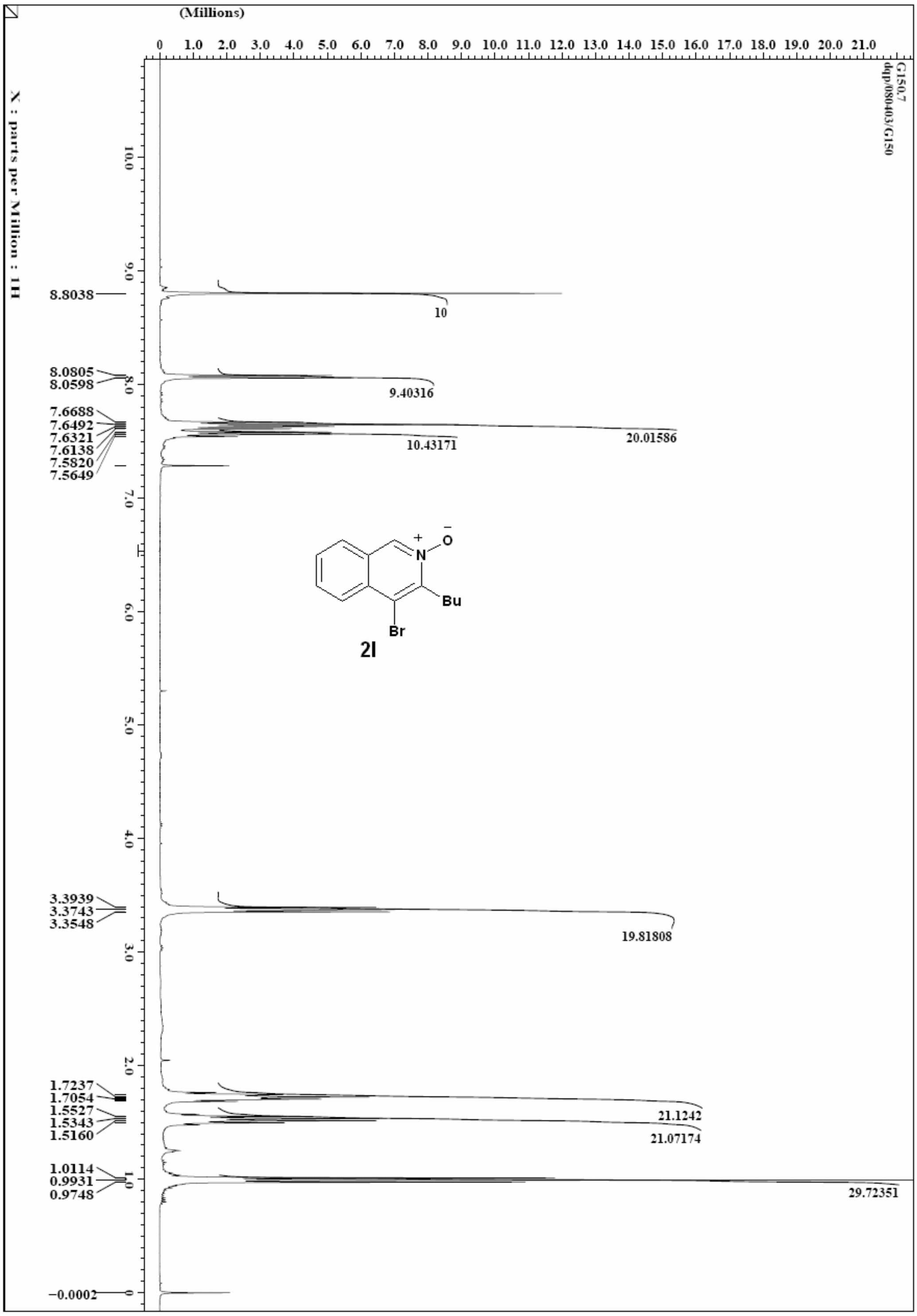


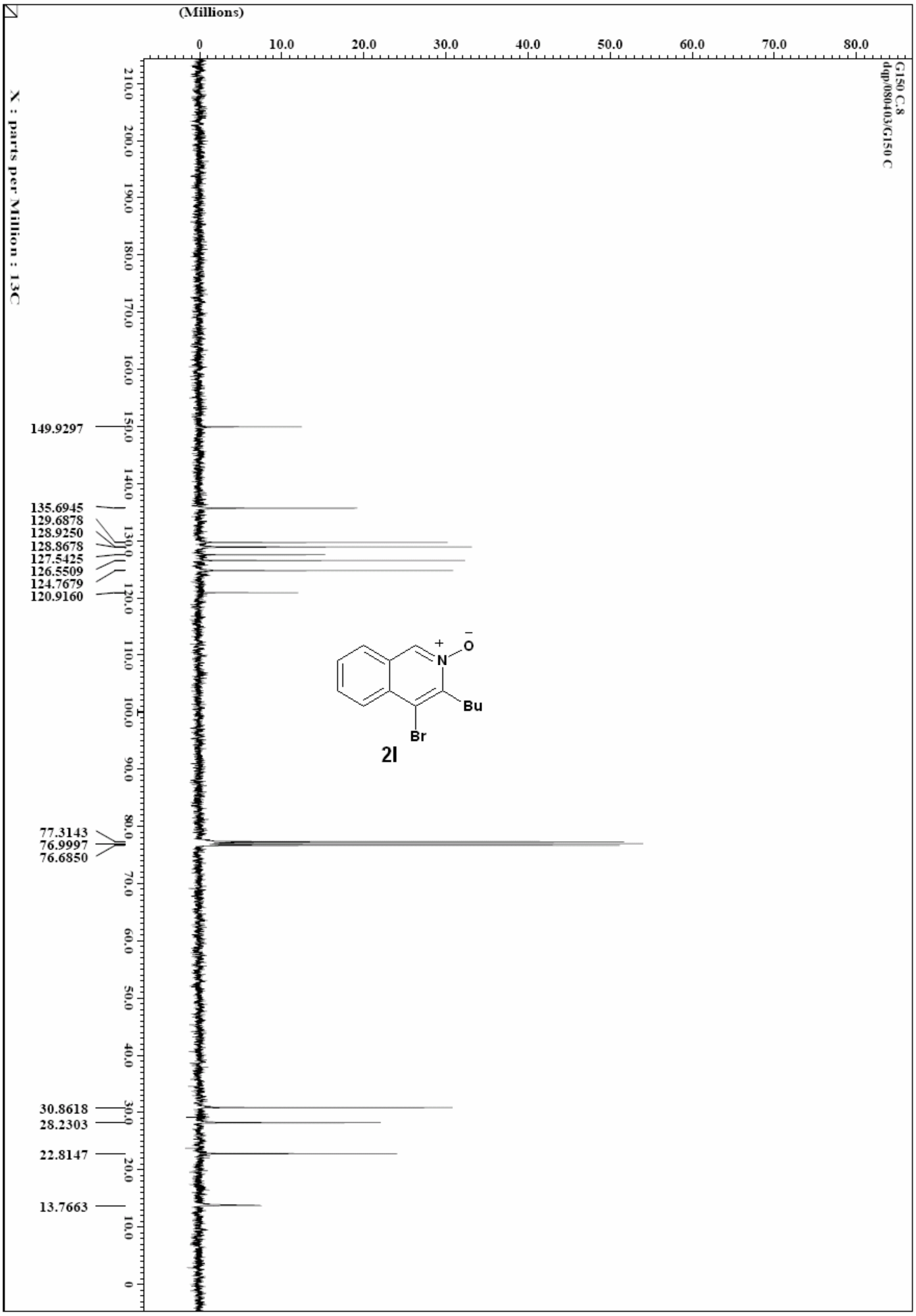

```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 77.184[ppm] : 77[ppm]
```

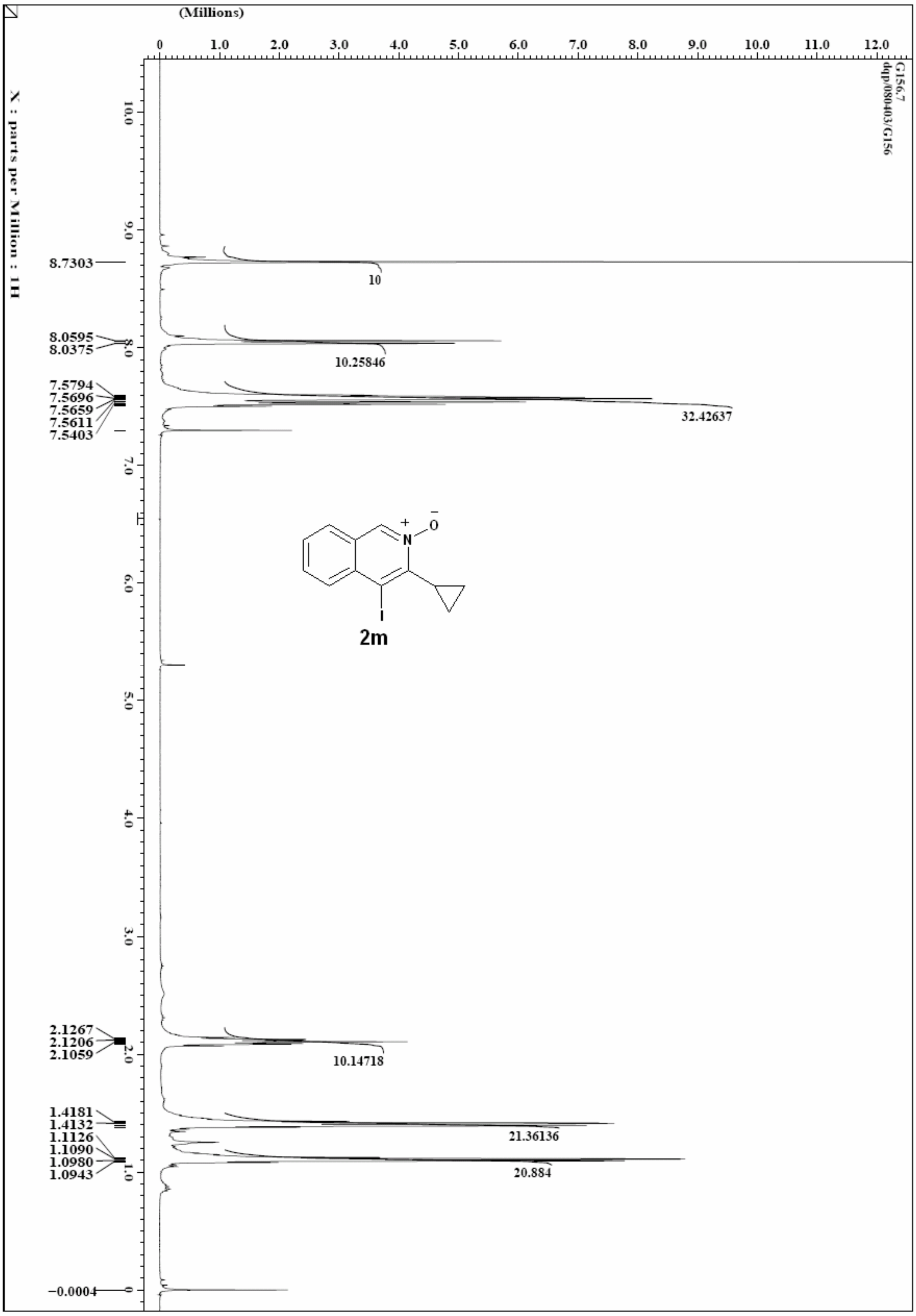
```
---- ACQUISITION PARAMETERS ----
Derived from: G148 C.1
File Name          = G148 C.8
Author             = delta
Sample ID          = dqp/080407/G148 C
Content            = dqp/080407/G148 C
Creation Date      =  7-APR-2008 11:04:17

Revision Date      =  7-APR-2008 11:20:22
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         = -41[ppm]
Mod_return         = 1
Scans              = 166
Total_scans        = 166

X_90_width         = 8.15[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 7[dB]
X_pulse            = 2.71666667[us]
Irr_atn_dec        = 24.8861[dB]
Irr_atn_noe        = 24.8861[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]
```

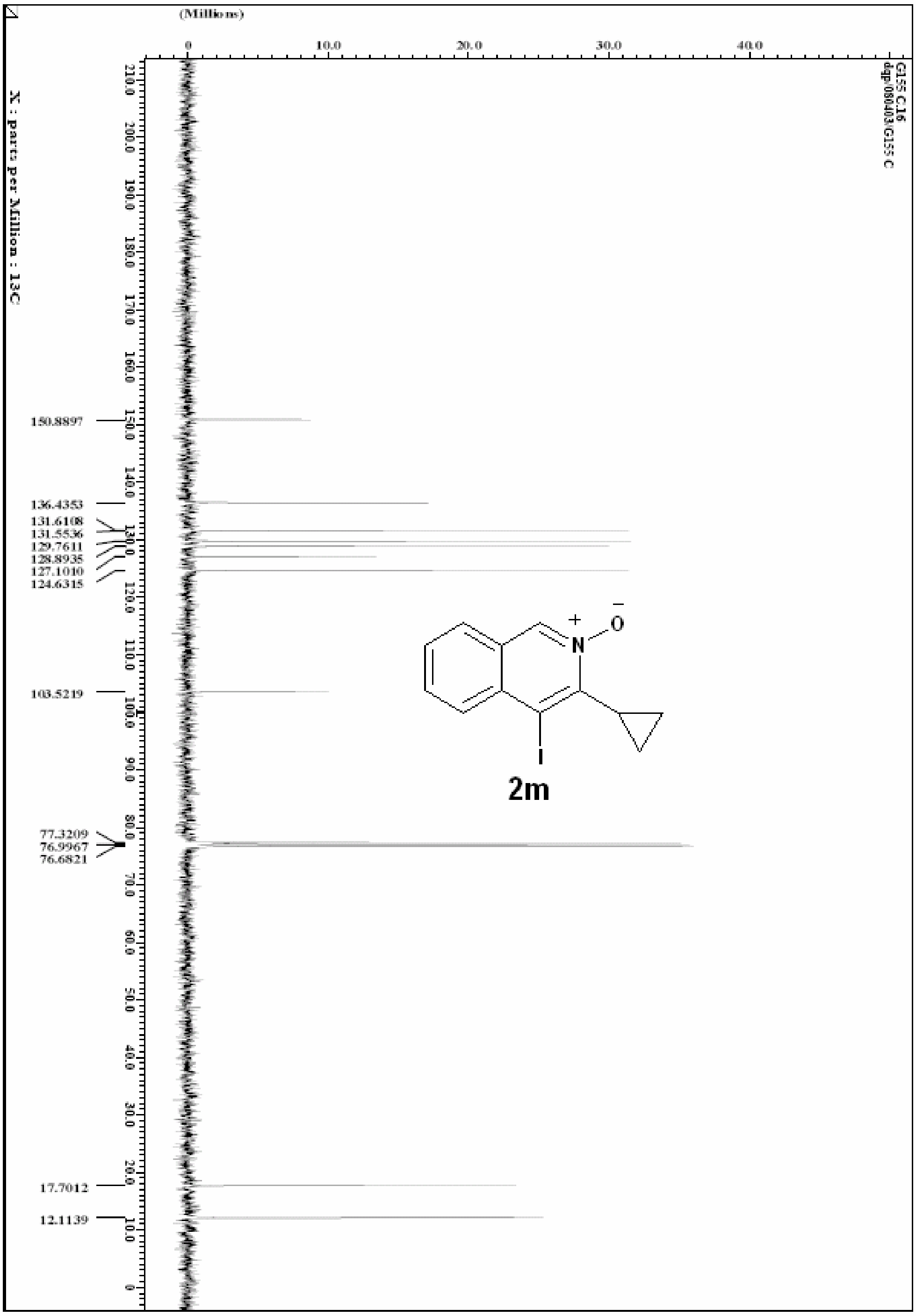

(Millions)
X : parts per Million : 1H
G147.4
dqp/080331/G147
8.8159
8.1842
8.1744
8.1610
7.4072
7.3962
7.3742
7.3608
7.0724
7.0517
3.8824
-0.0003
9.65642
10
40.70689
20.17707
30.28088
Br
OCH3
2k

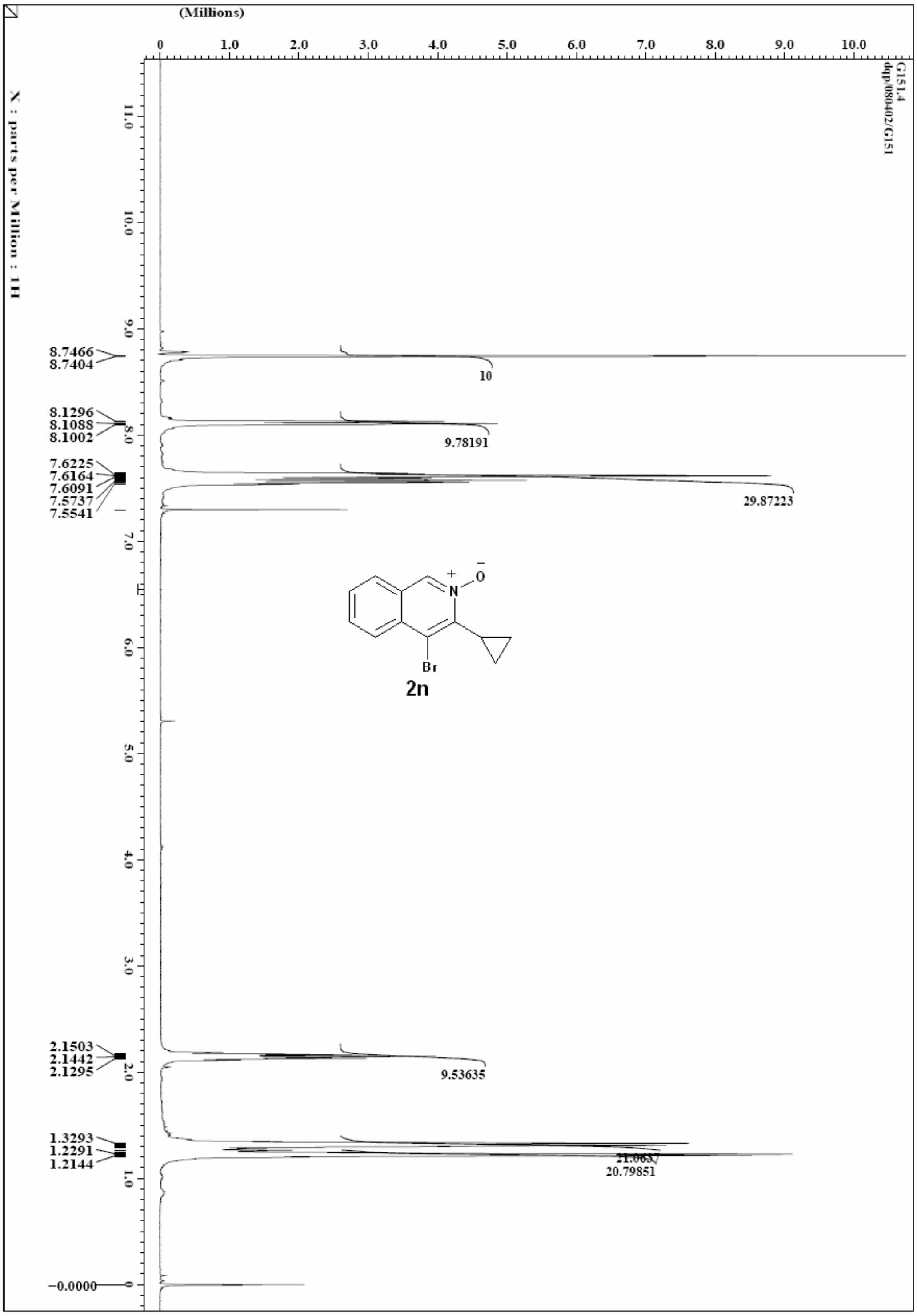

(Millions)
X : parts per Million : 13C
G147 C.5
dqp/08031G147 C
163.8125
161.2954
160.2847
147.2700
147.2509
135.1897
135.1325
131.2805
130.4987
130.4033
129.5166
129.4117
125.9221
125.1498
122.3085
119.8676
119.6102
114.0038
108.3784
108.1496
77.3243
77.0001
76.6855
55.2518
2k

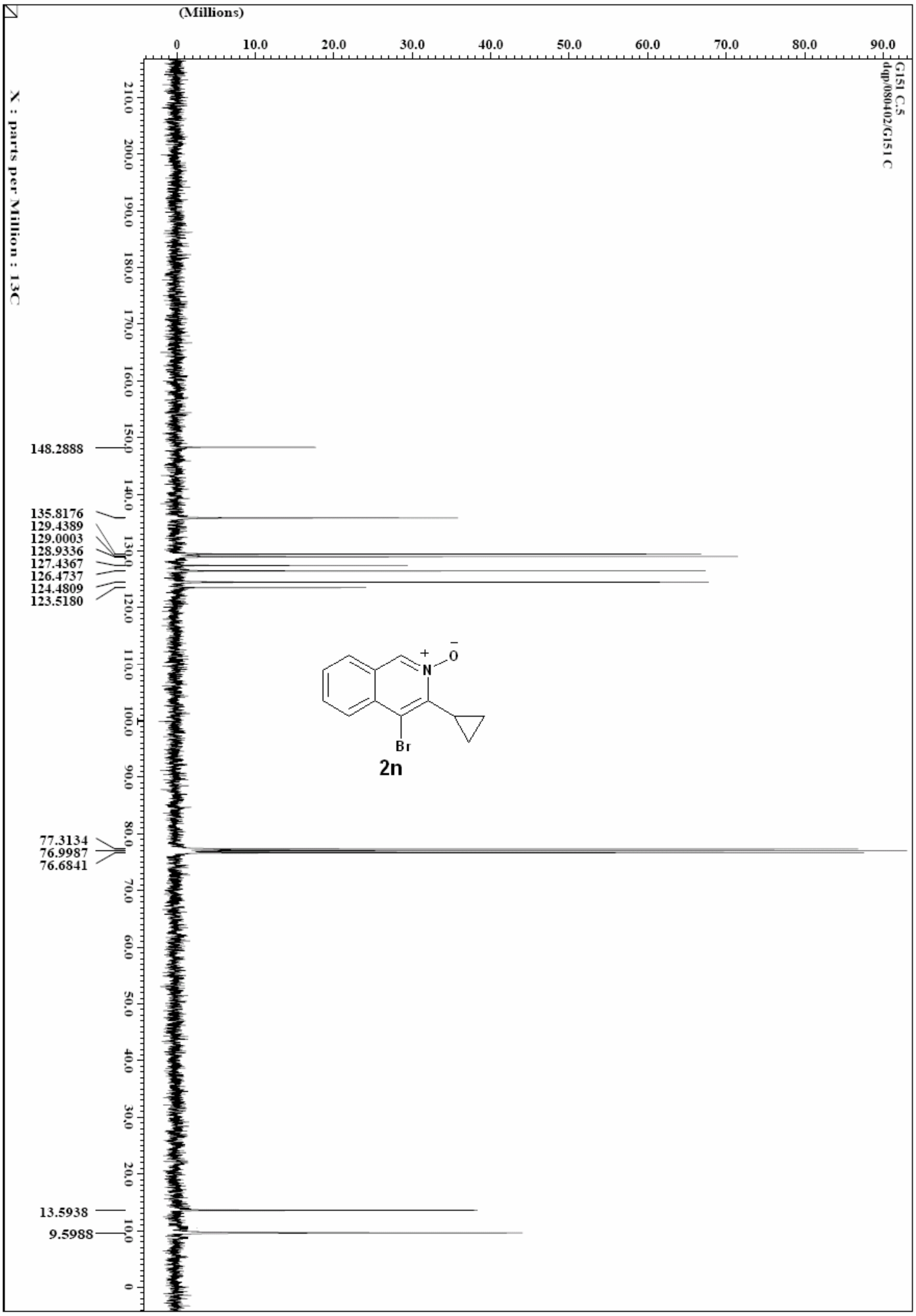

(Millions)
X : parts per Million : 1H
G150.7
dtp/080403/G150
8.8038
8.0805
8.0598
7.6688
7.6492
7.6321
7.6138
7.5820
7.5649
3.3939
3.3743
3.3548
1.7237
1.7054
1.5527
1.5343
1.5160
1.0114
0.9931
0.9748
−0.0002
10
9.40316
10.43171
20.01586
19.81808
21.1242
21.07174
29.72351
Bu
Br
2l

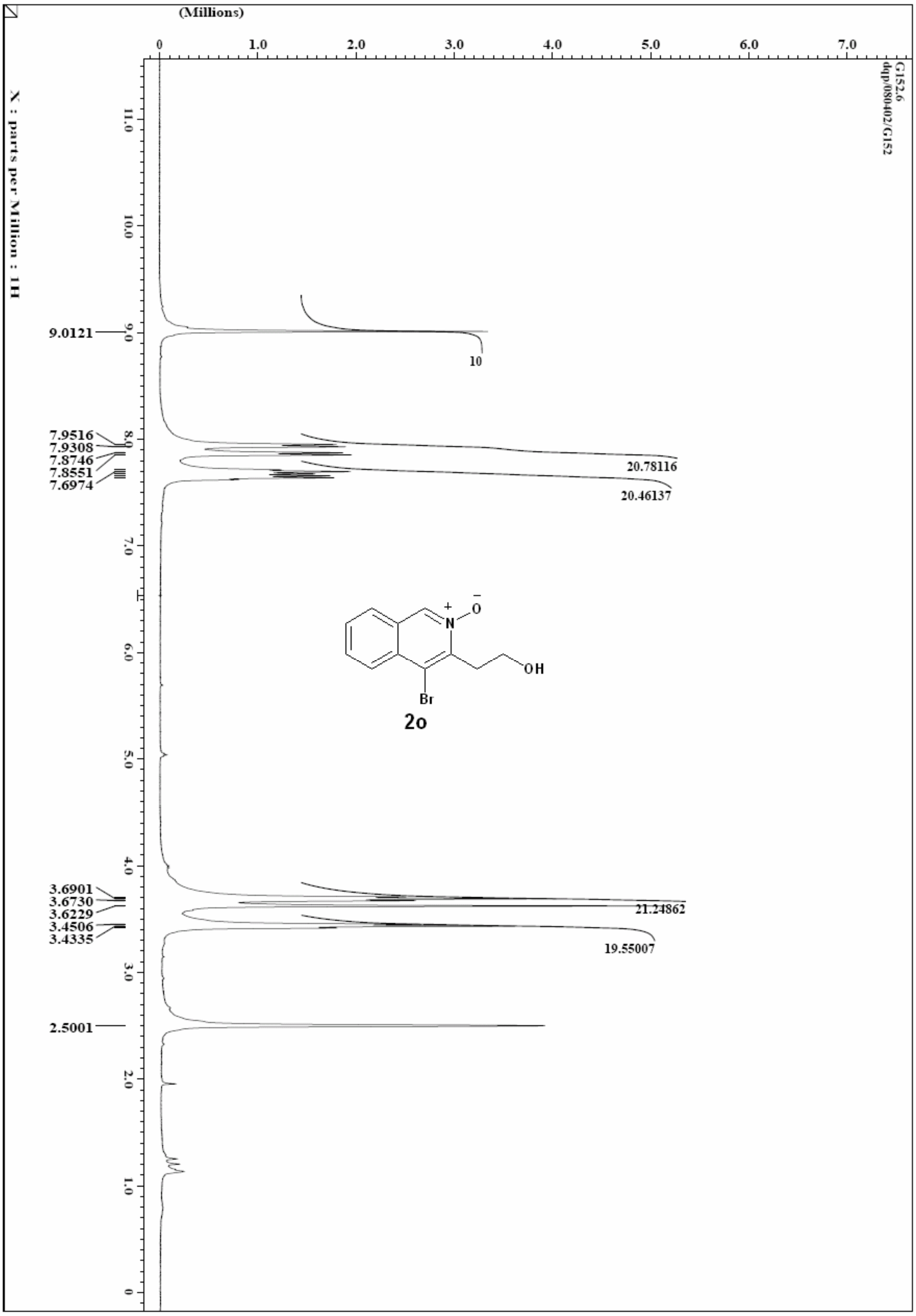

(Millions)
X : parts per Million : 13C
G150 C.8
dup/080403/G150 C
149.9297
135.6945
129.6878
128.9250
128.8678
127.5425
126.5509
124.7679
120.9160
77.3143
76.9997
76.6850
30.8618
28.2303
22.8147
13.7663
Bu
Br
2l


(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
G156.7
dup/080403/G156
X : parts per Million : 1H
8.7303
8.0595
8.0375
7.5794
7.5696
7.5659
7.5611
7.5403
2.1267
2.1206
2.1059
1.4181
1.4132
1.1126
1.1090
1.0980
1.0943
-0.0004
10
10.25846
32.42637
10.14718
21.36136
20.884
2m

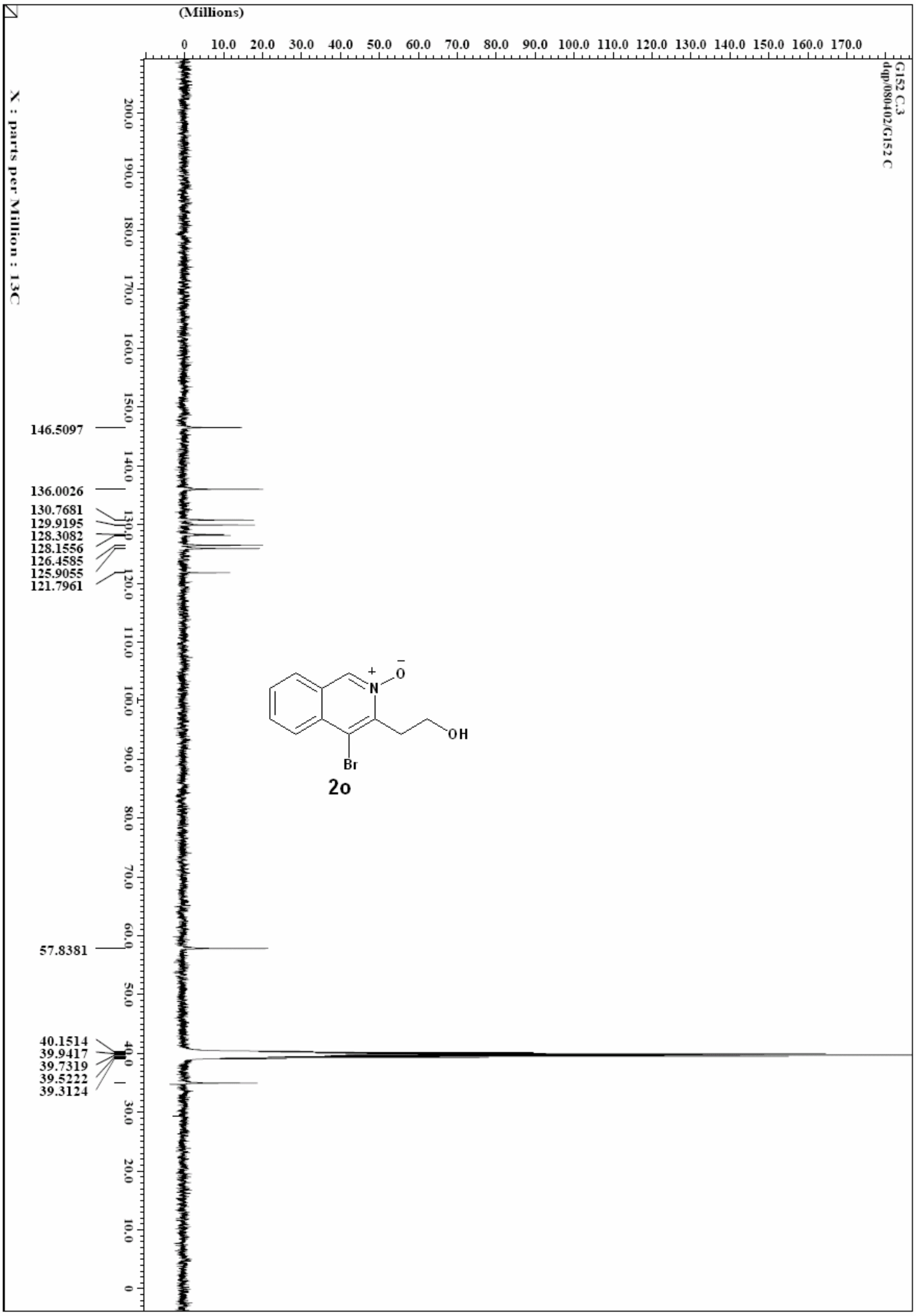

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 13C
150.8897
136.4353
131.6108
131.5536
129.7611
128.8935
127.1010
124.6315
103.5219
77.3209
76.9967
76.6821
17.7012
12.1139
2m

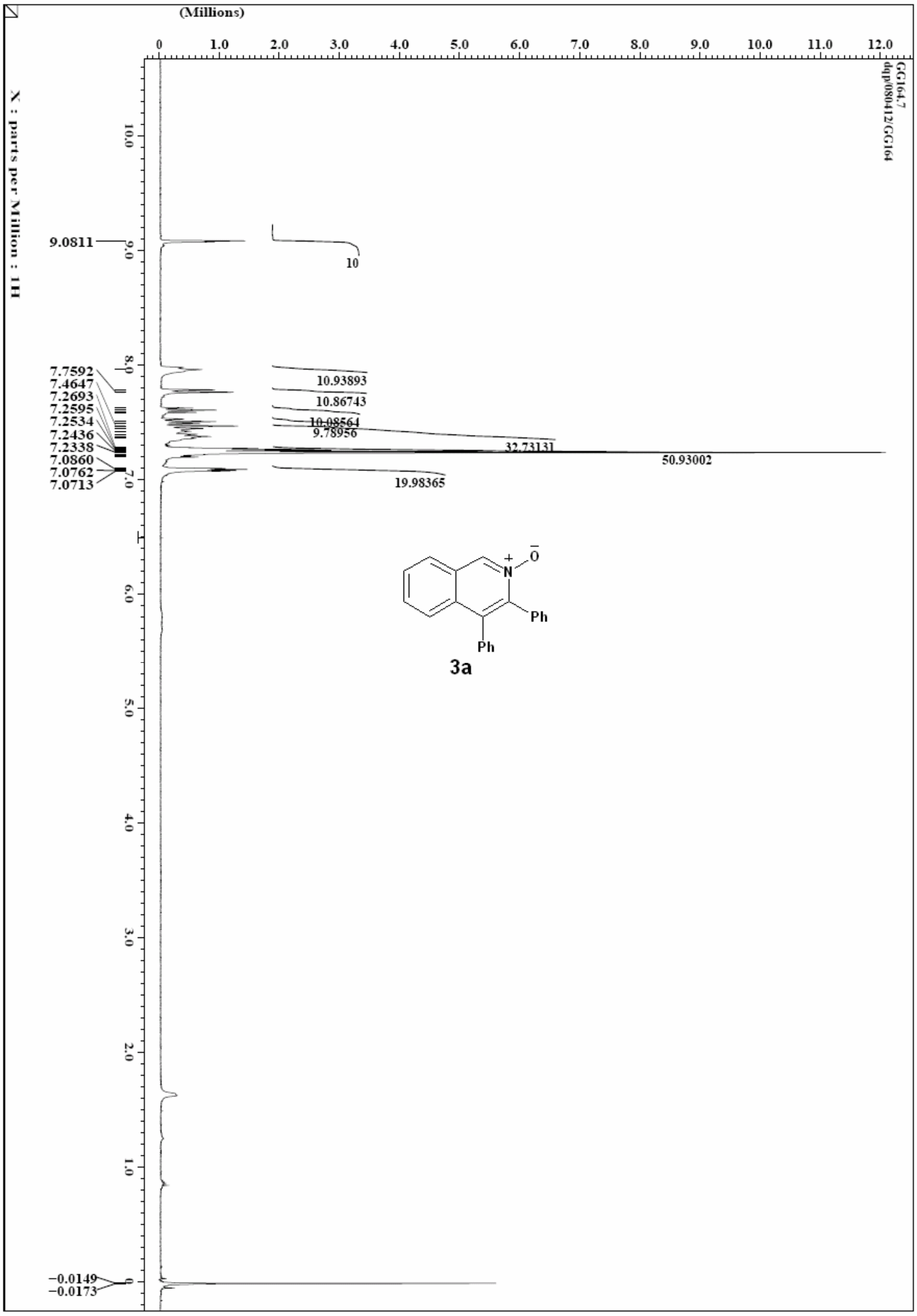

(Millions)
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
G151.4
dup/080402/G151
X : parts per Million : 1H
11.0
10.0
9.0
8.7466
8.7404
10
8.1296
8.1088
8.1002
8.0
9.78191
7.6225
7.6164
7.6091
7.5737
7.5541
29.87223
7.0
6.0
5.0
4.0
3.0
2.1503
2.1442
2.1295
2.0
9.53635
1.3293
1.2291
1.2144
21.063
20.79851
1.0
-0.0000
0
Br
2n

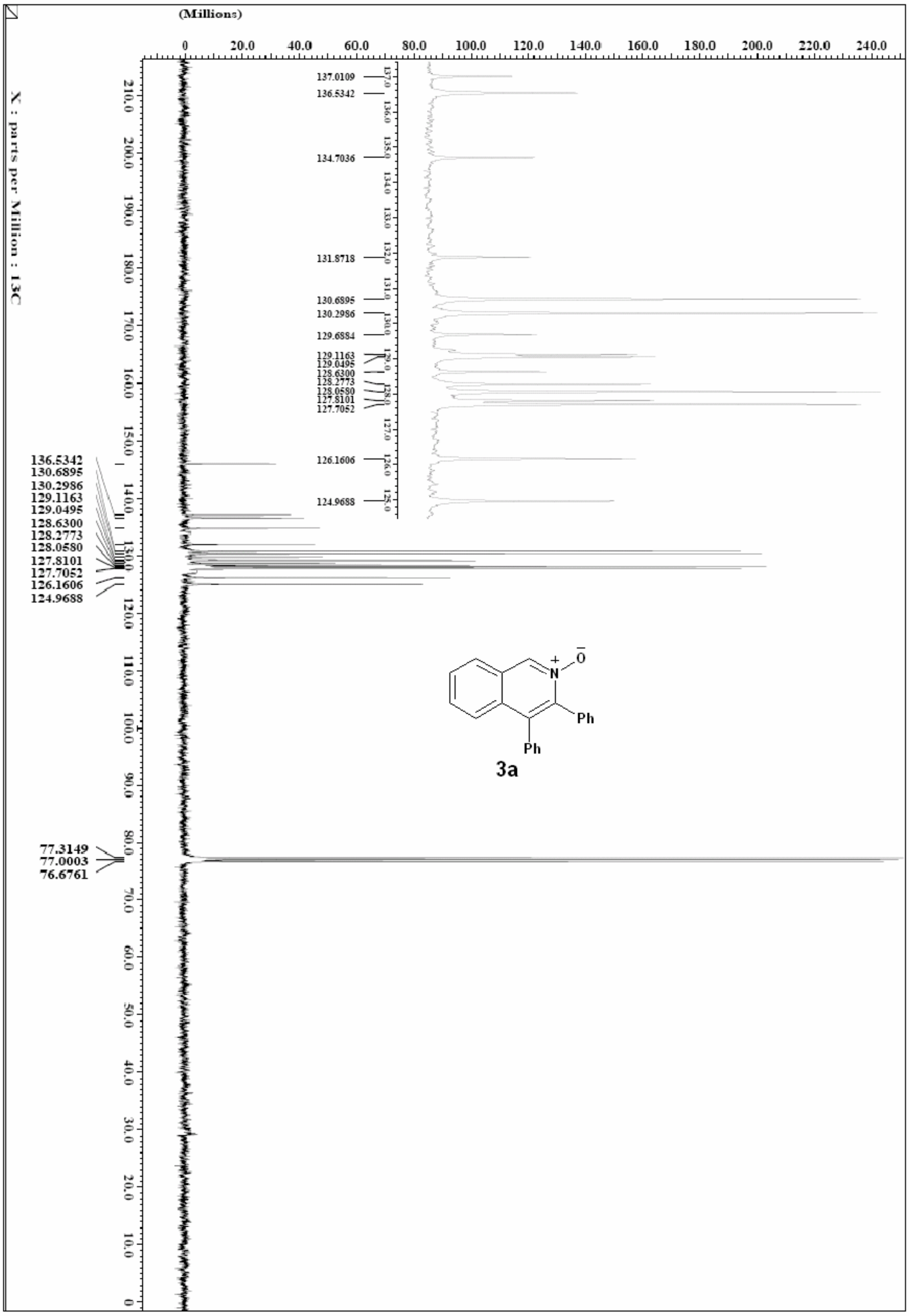

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
G151 C.5
dup/080402/G151 C
X : parts per Million : 13C
148.2888
135.8176
129.4389
129.0003
128.9336
127.4367
126.4737
124.4809
123.5180
77.3134
76.9987
76.6841
13.5938
9.5988
Br
2n

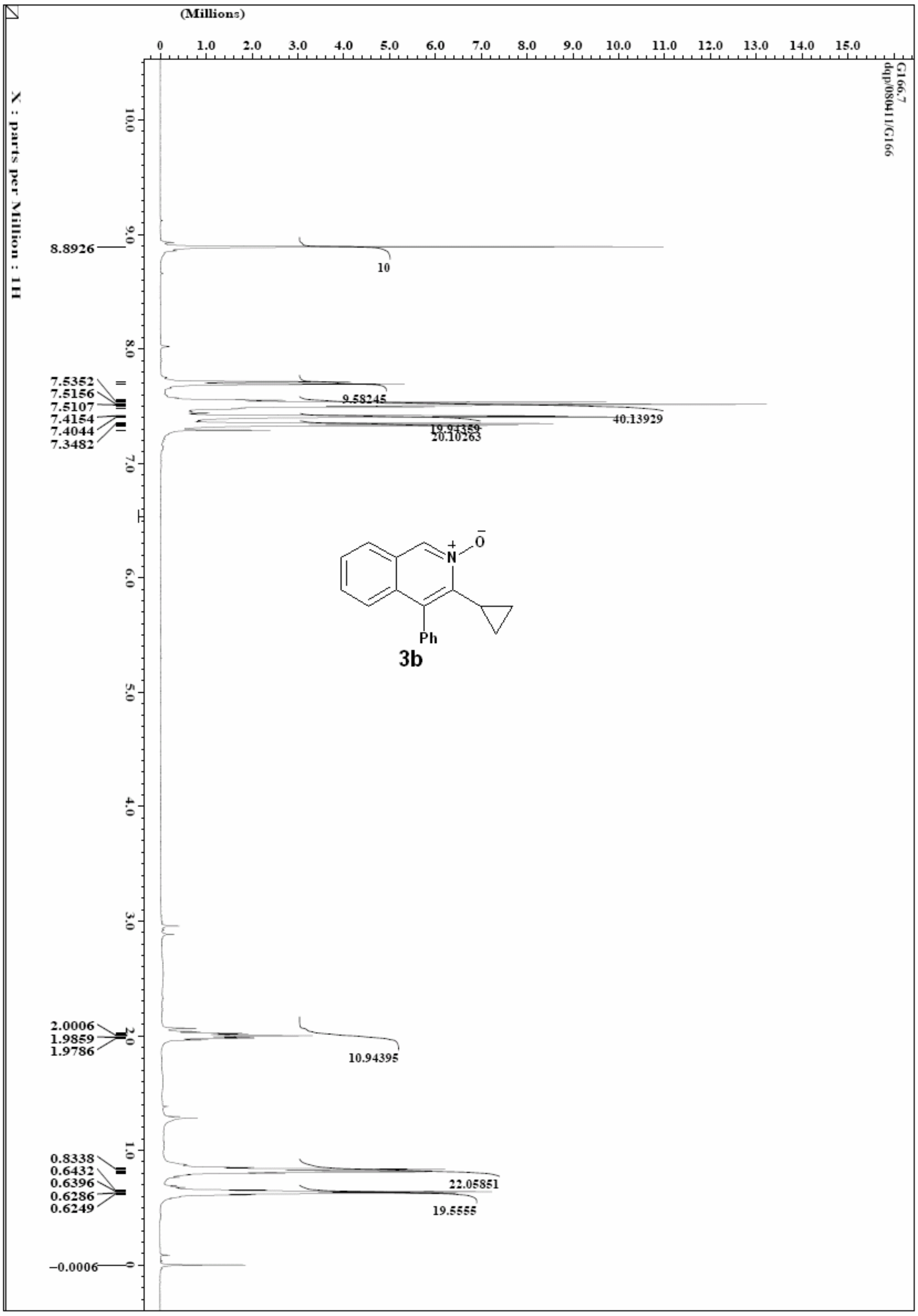


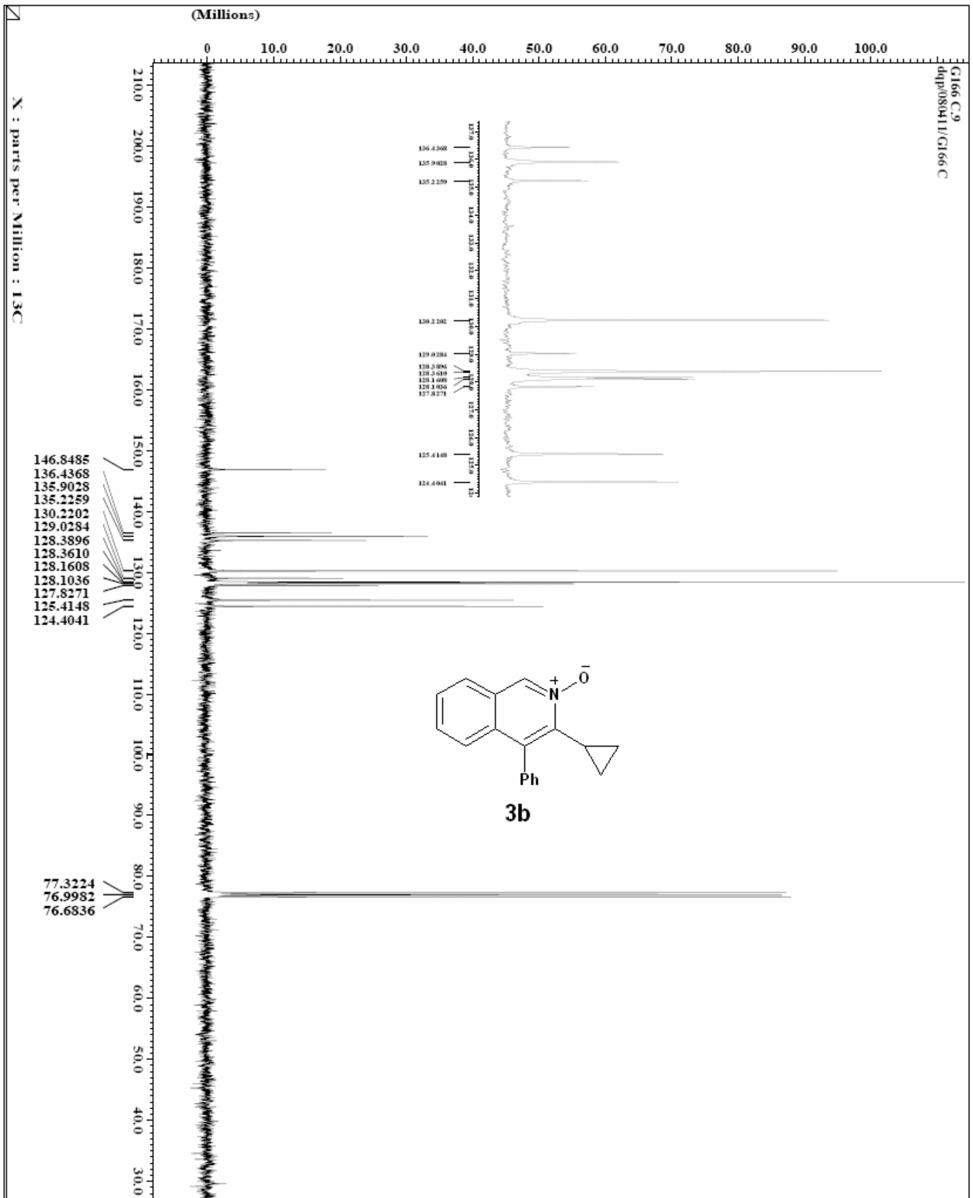
(Millions)
X : parts per Million : 1H
G152.6
dap/080402/G152
9.0121
7.9516
7.9308
7.8746
7.8551
7.6974
3.6901
3.6730
3.6229
3.4506
3.4335
2.5001
10
20.78116
20.46137
21.24862
19.55007
Br
OH
2o

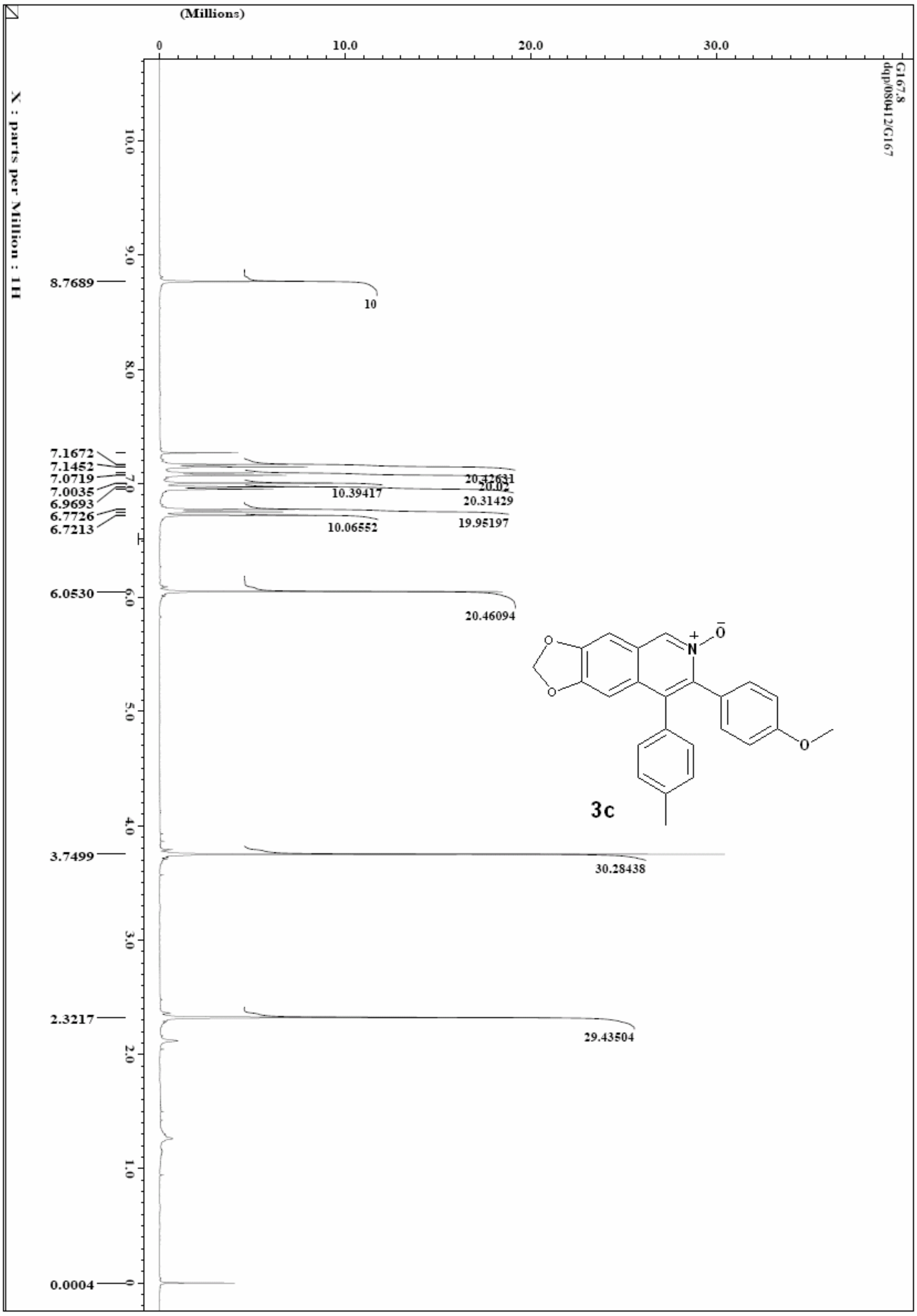

(Millions)
X : parts per Million : 13C
G152 C.3
dqp/080402/G152 C
146.5097
136.0026
130.7681
129.9195
128.3082
128.1556
126.4585
125.9055
121.7961
57.8381
40.1514
39.9417
39.7319
39.5222
39.3124
Br
OH
2o

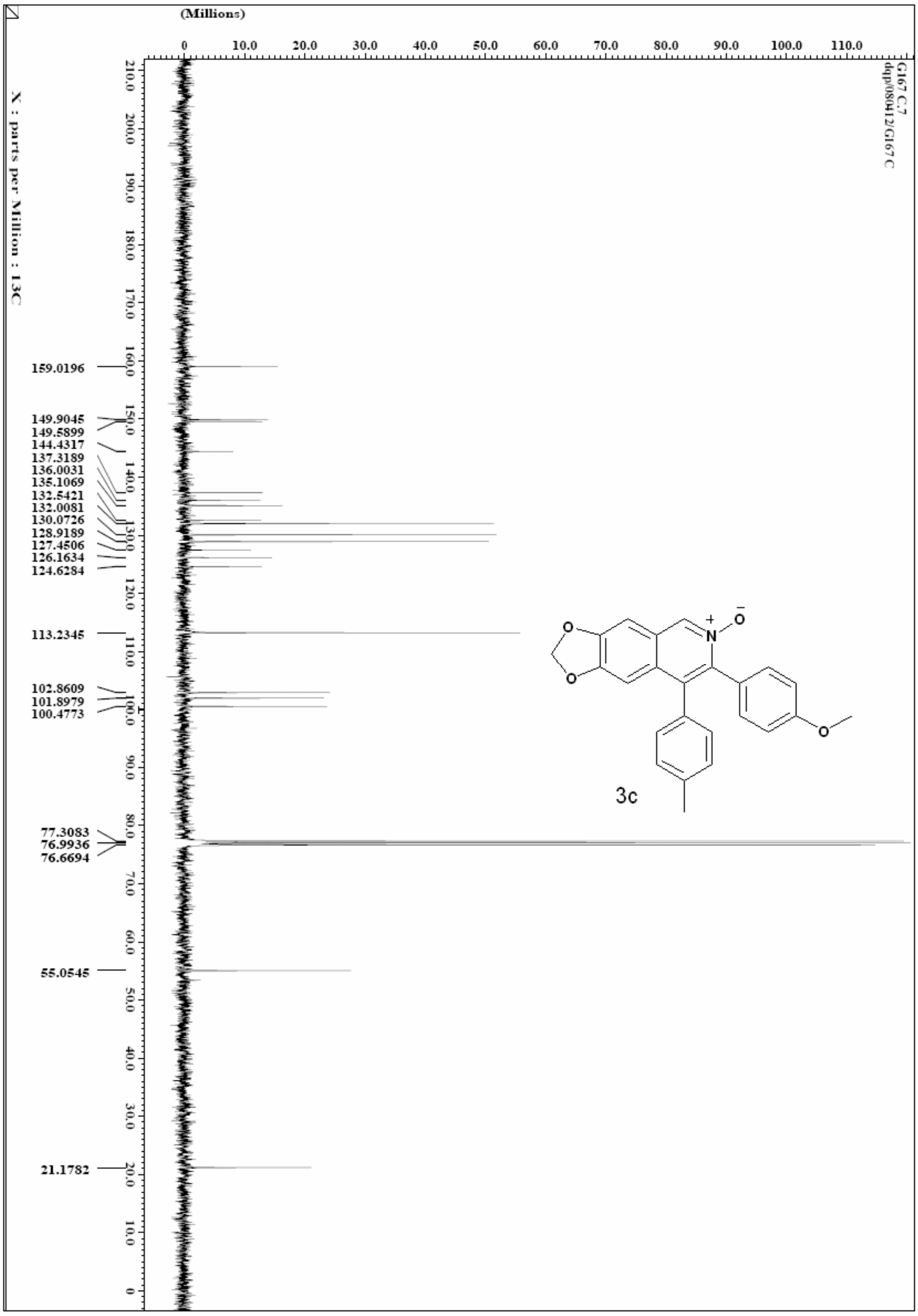

(Millions)
X : parts per Million : 1H
GG164.7
dqp/080412/GG164
9.0811
7.7592
7.4647
7.2693
7.2595
7.2534
7.2436
7.2338
7.0860
7.0762
7.0713
-0.0149
-0.0173
10
10.93893
10.86743
10.08564
9.78956
32.73131
50.93002
19.98365
3a


(Millions)
X : parts per Million : 13C
136.5342
130.6895
130.2986
129.1163
129.0495
128.6300
128.2773
128.0580
127.8101
127.7052
126.1606
124.9688
77.3149
77.0003
76.6761
137.0109
134.7036
131.8718
129.6884
Ph
Ph
3a

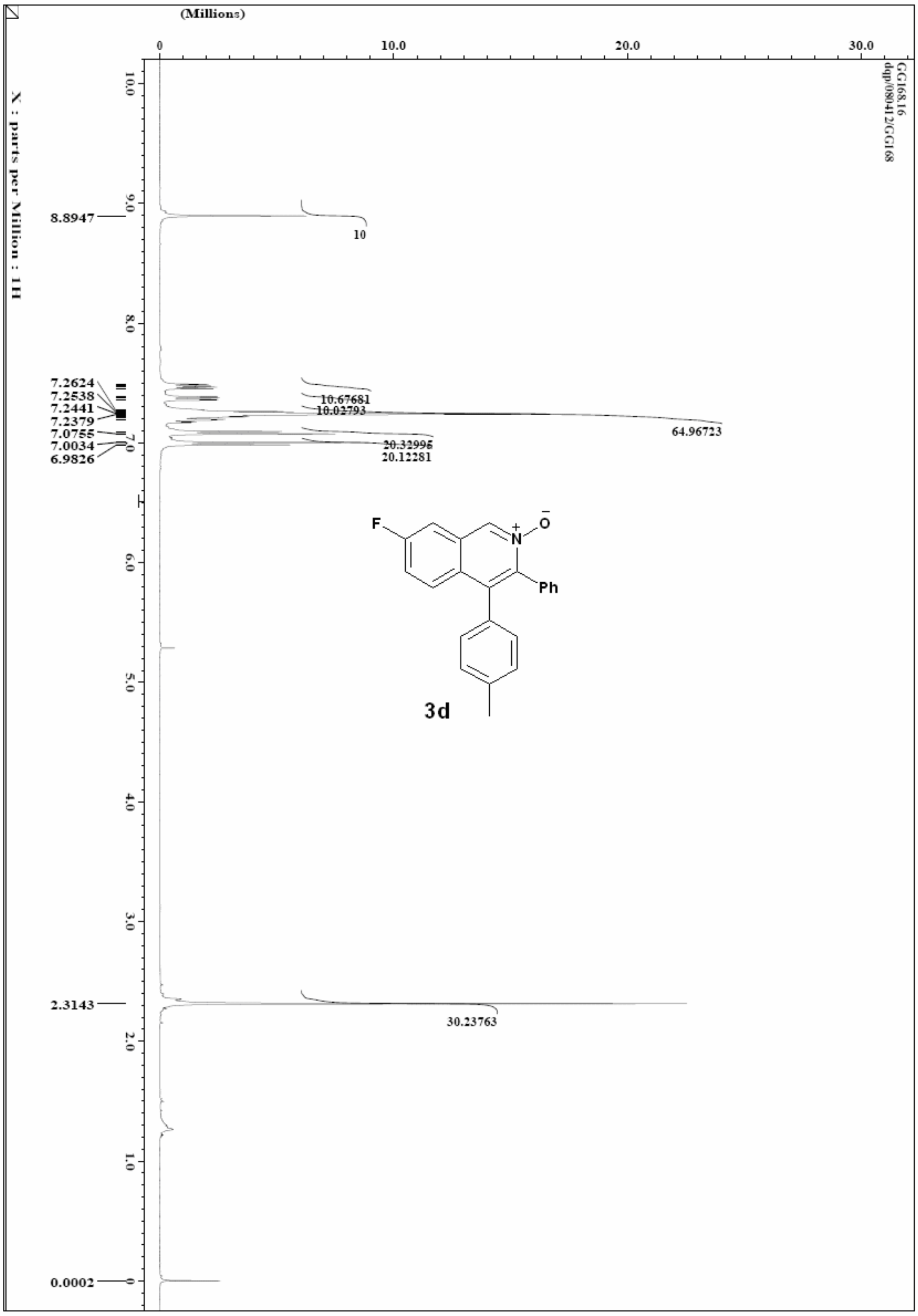

(Millions)
X : parts per Million : 1H
G166.7
dqp/08041/G166
8.8926
7.5352
7.5156
7.5107
7.4154
7.4044
7.3482
2.0006
1.9859
1.9786
0.8338
0.6432
0.6396
0.6286
0.6249
−0.0006
10
9.58245
40.13929
19.94359
20.10263
10.94395
22.05851
19.5555
Ph
3b

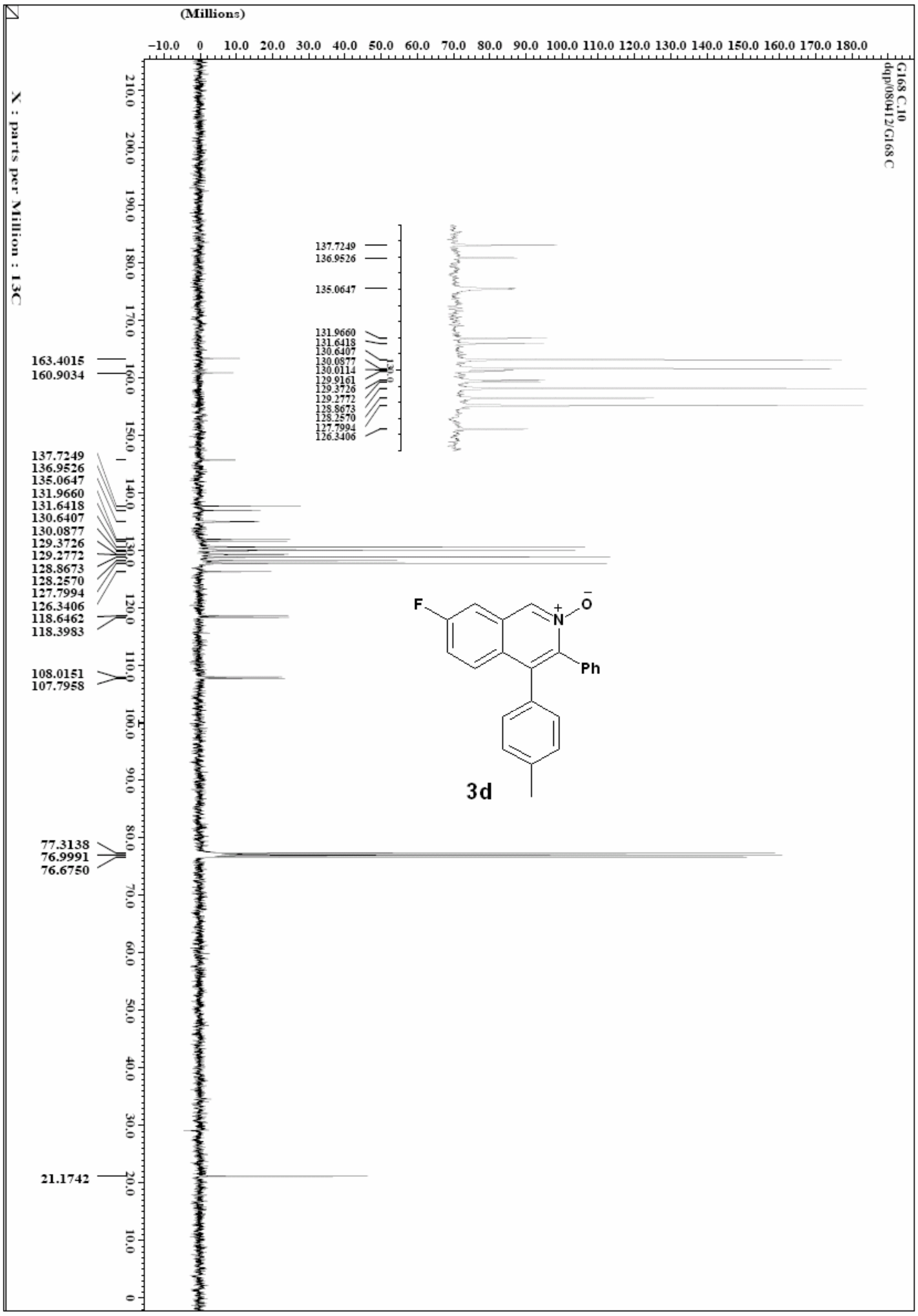

(Millions)
X : parts per Million : 13C
G166_C.9
dqp08041/G166C
146.8485
136.4368
135.9028
135.2259
130.2202
129.0284
128.3896
128.3610
128.1608
128.1036
127.8271
125.4148
124.4041
77.3224
76.9982
76.6836
11.7722
7.9679
Ph
3b

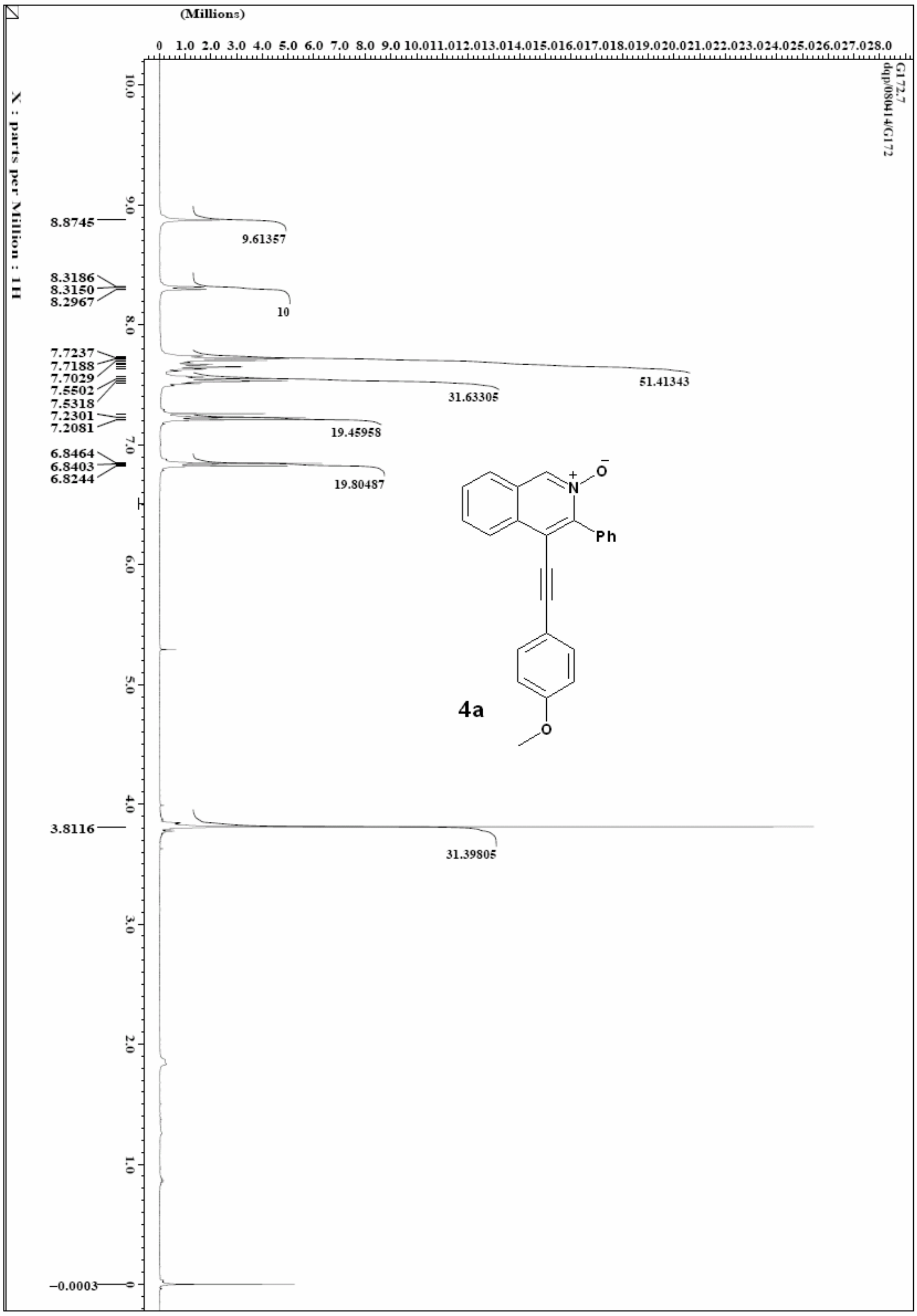

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
G167.8
dqp080412G167
8.7689
7.1672
7.1452
7.0719
7.0035
6.9693
6.7726
6.7213
6.0530
3.7499
2.3217
0.0004
10
20.32631
20.02
10.39417
20.31429
10.06552
19.95197
20.46094
30.28438
29.43504
3c

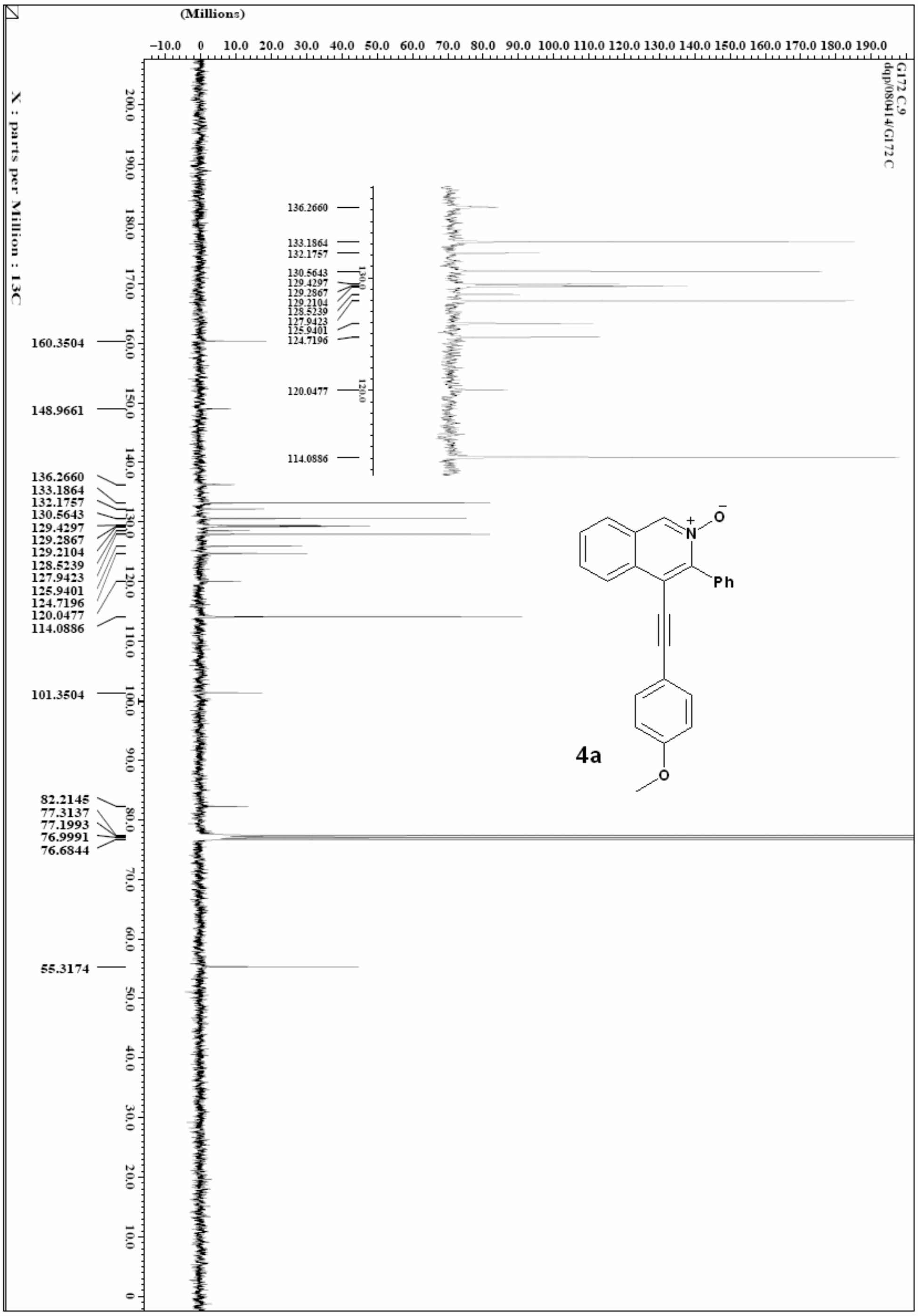


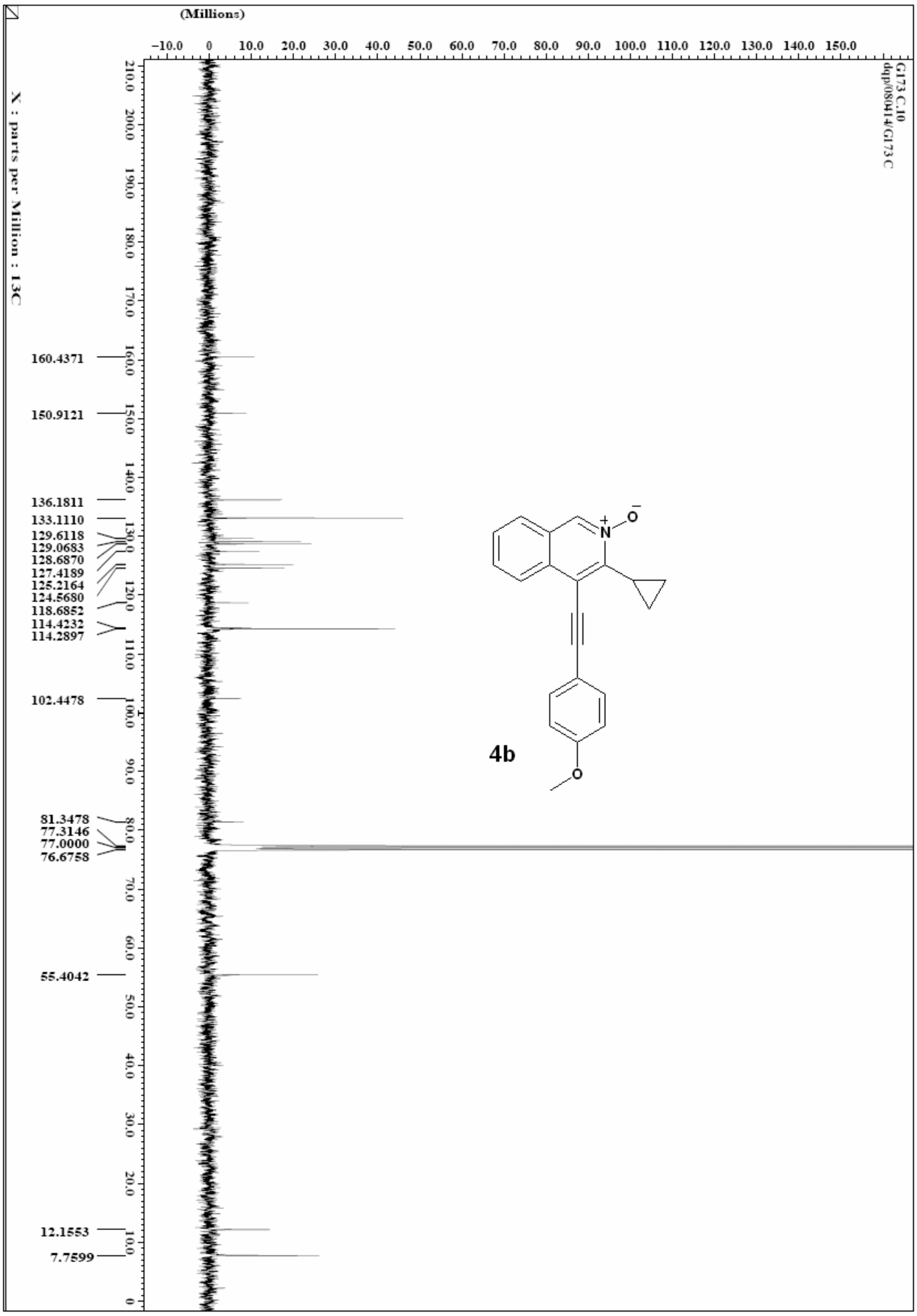
(Millions)
X : parts per Million : 13C
G167.C.7
dqp/080412/G167C
159.0196
149.9045
149.5899
144.4317
137.3189
136.0031
135.1069
132.5421
132.0081
130.0726
128.9189
127.4506
126.1634
124.6284
113.2345
102.8609
101.8979
100.4773
77.3083
76.9936
76.6694
55.0545
21.1782
3c

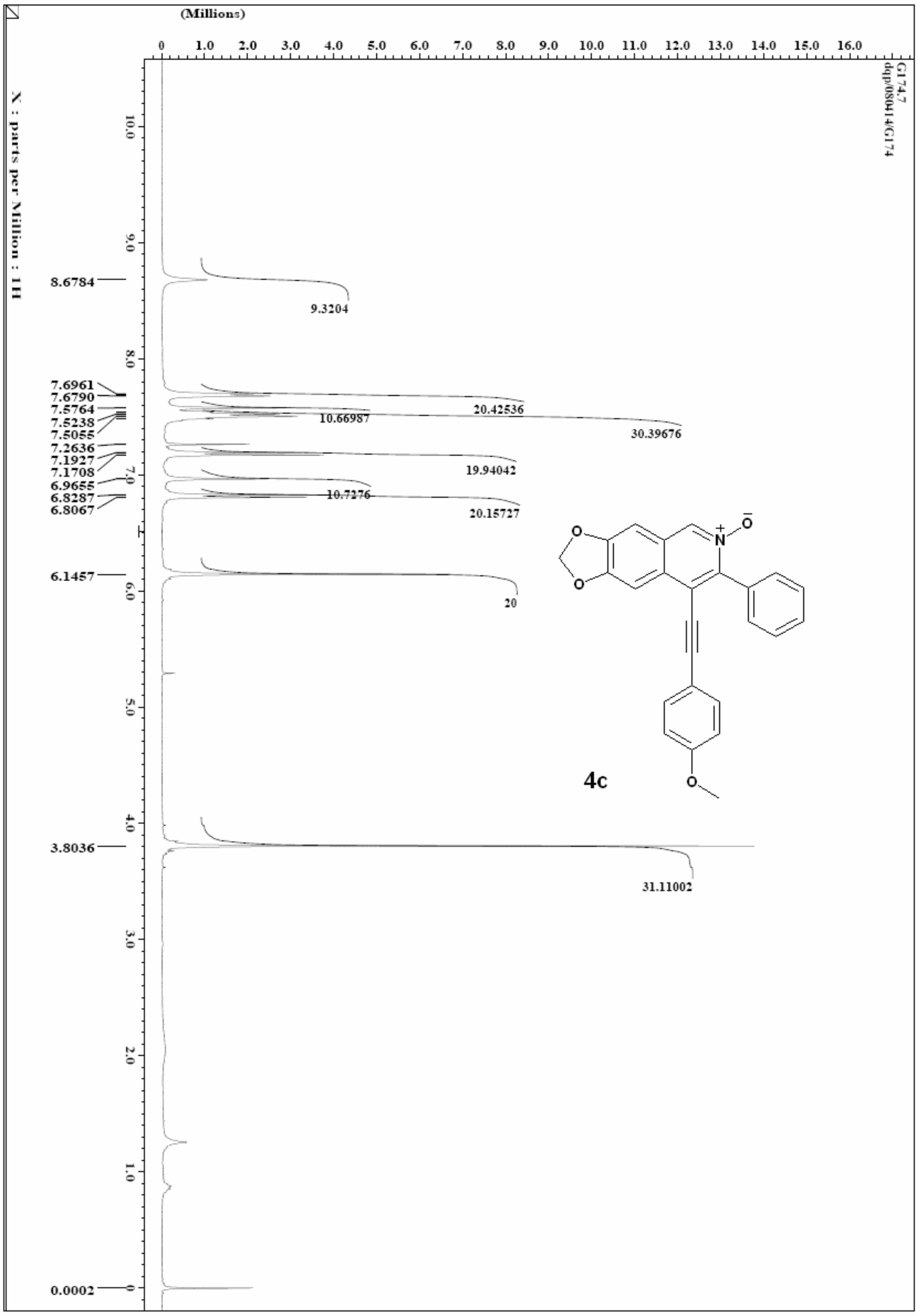

(Millions)
0
10.0
20.0
30.0
GG16816
dqp/080412/GG168
X : parts per Million : 1H
8.8947
7.2624
7.2538
7.2441
7.2379
7.0755
7.0034
6.9826
2.3143
0.0002
10
10.67681
10.02793
64.96723
20.32995
20.12281
30.23763
F
N
O
Ph
3d

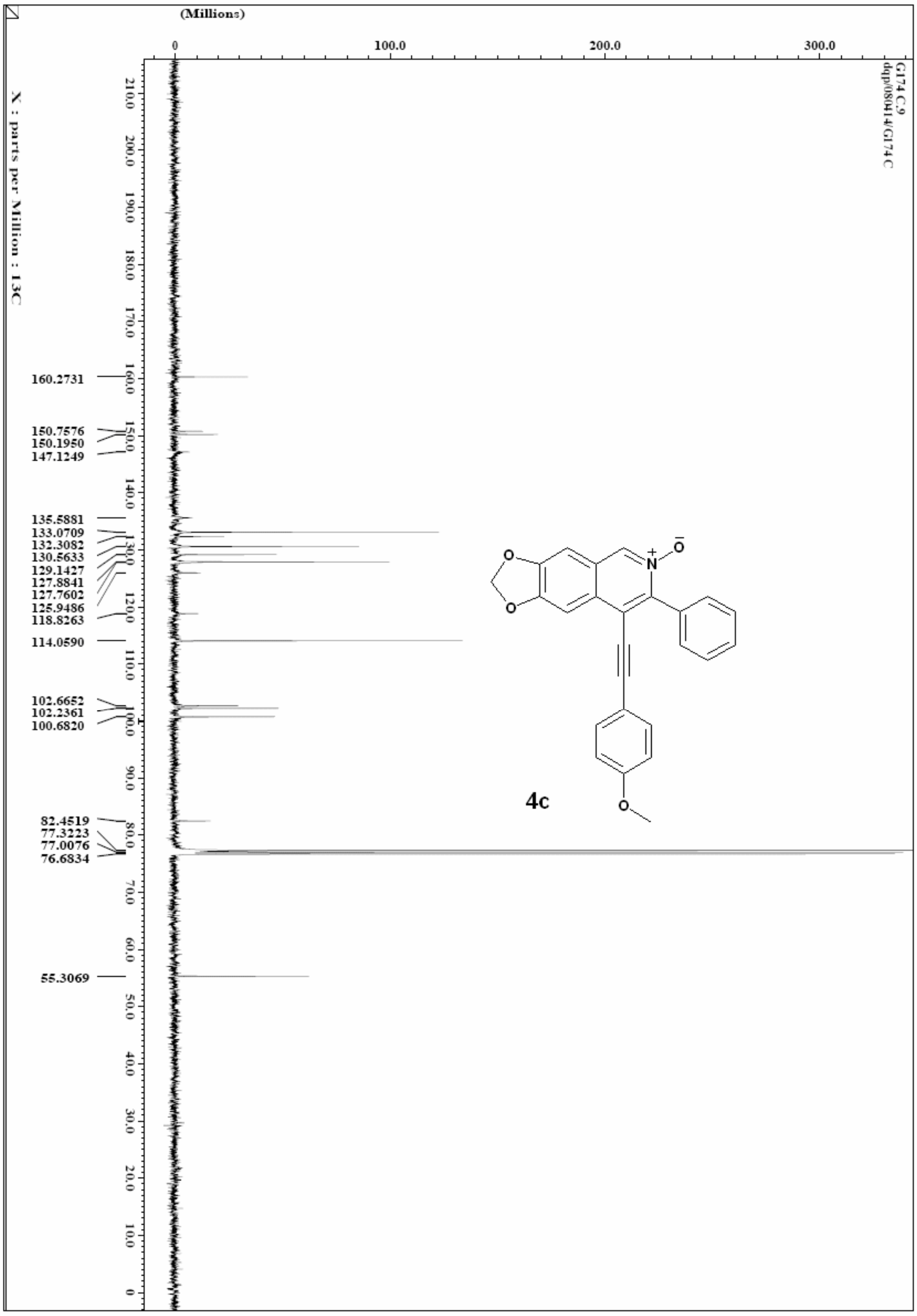

(Millions)
X : parts per Million : 13C
G168 C.10
dqp080412/G168 C
163.4015
160.9034
137.7249
136.9526
135.0647
131.9660
131.6418
130.6407
130.0877
129.3726
129.2772
128.8673
128.2570
127.7994
126.3406
118.6462
118.3983
108.0151
107.7958
77.3138
76.9991
76.6750
21.1742
130.0114
129.9161
F
Ph
3d


(Millions)
X : parts per Million : 1H
G172.7
dqp/080414G172
8.8745
8.3186
8.3150
8.2967
7.7237
7.7188
7.7029
7.5502
7.5318
7.2301
7.2081
6.8464
6.8403
6.8244
3.8116
-0.0003
9.61357
10
51.41343
31.63305
19.45958
19.80487
31.39805
Ph
4a


(Millions)
X : parts per Million : 13C
G172.C.9
dqp08041-G172C
160.3504
148.9661
136.2660
133.1864
132.1757
130.5643
129.4297
129.2867
129.2104
128.5239
127.9423
125.9401
124.7196
120.0477
114.0886
101.3504
82.2145
77.3137
77.1993
76.9991
76.6844
55.3174
Ph
4a


(Millions)
X : parts per Million : 13C
G173C.10
dnp/080414/G173C
160.4371
150.9121
136.1811
133.1110
129.6118
129.0683
128.6870
127.4189
125.2164
124.5680
118.6852
114.4232
114.2897
102.4478
81.3478
77.3146
77.0000
76.6758
55.4042
12.1553
7.7599
4b


(Millions)
X : parts per Million : 1H
8.6784
7.6961
7.6790
7.5764
7.5238
7.5055
7.2636
7.1927
7.1708
6.9655
6.8287
6.8067
6.1457
3.8036
0.0002
9.3204
20.42536
10.66987
30.39676
19.94042
10.7276
20.15727
20
31.11002
4c


(Millions)
0
100.0
200.0
300.0
X : parts per Million : 13C
G174 C.9
dpp08414/G174C
160.2731
150.7576
150.1950
147.1249
135.5881
133.0709
132.3082
130.5633
129.1427
127.8841
127.7602
125.9486
118.8263
114.0590
102.6652
102.2361
100.6820
82.4519
77.3223
77.0076
76.6834
55.3069
4c