Lewis Acid and Organocatalyst Co-catalyzed Multicomponent Reactions of 2-Alkynylbenzaldehydes, Amines, and Ketones

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Supporting Information

1. General experimental methods (S2).

2. Characterization data of compound **4** (S2-S12).

3. Copies of spectra of compound **4** (S13-S74).

General Materials and Methods: All reactions were preformed under nitrogen atmosphere. 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 one-pot reaction of 2-alkynylbenzaldehyde 1, amine 2, and ketone 3 catalyzed by silver triflate and proline: A solution of 2-alkynyl benzaldehyde **1** (0.30 mmol), amine **2** (0.30 mmol, 1.0 equiv), ketone **3** (1.50 mmol, 5.0 equiv), AgOTf (0.03 mmol, 10 mol %), proline (0.03 mmol, 10 mol %) in C_2H_5OH (1.5 mL) was stirred at 50~60 °C under N_2 for a period of time. 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 product **4**.

2-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)cyclohexanone (**4a**)

Yield: 65%; ^1H NMR (400 MHz, $CDCl_3$) δ 1.22-1.30 (m, 1H), 1.36-1.50 (m, 2H), 1.68-1.75 (m, 2H), 1.98-2.07 (m, 1H), 2.31-2.43 (m, 1H), 2.50-2.58 (m, 1H), 3.02-3.08 (m, 1H), 3.62 (s, 3H), 5.24 (d, J = 9.3 Hz, 1H), 6.61 (d, J = 8.8 Hz, 2H), 6.72 (s, 1H), 6.90 (d, J = 7.8 Hz, 1H), 7.11 (dt, J = 1.5, 7.3 Hz, 1H), 7.18-7.26 (m, 7H), 7.50 (d, J =7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 29.0, 32.1, 43.1, 52.0, 55.2, 64.1, 112.0, 113.7, 124.1, 124.4, 126.0, 127.0, 127.2, 127.3, 127.9, 128.3, 129.0, 132.2, 137.7, 140.8, 142.1, 155.0, 213.0; MS *m/z* 410.2 (M^++1); Elemental analysis calcd (%) for $C_{28}H_{27}NO_2$: C 82.12, H 6.65, N 3.42; Found: C 82.25, H 6.61, N 3.50. HPLC: OD, *i*-propanol/hexane= 6:94, flow rate 0.5mL/min, λ = 254 nm): t =8.3 min, t '=11.6 min, racemate.

Diastereoisomer (**4a'**)

Yield: 15%; ^{1}H NMR (400 MHz, $CDCl_3$) δ1.56-1.66 (m, 1H), 1.70-1.82 (m, 1H), 1.85-2.00 (m, 2H), 2.02-2.10 (m, 1H), 2.16-2.25 (m, 1H), 2.33-2.38 (m, 1H), 2.58-2.66 (m, 1H), 2.76-2.85 (m, 1H), 3.62 (s, 3H), 5.34 (d, *J* = 9.3 Hz, 1H), 6.60 (d, *J* = 9.3 Hz, 2H), 6.75 (s, 1H), 6.80 (d, *J* = 8.8 Hz, 2H), 7.09 (dt, *J* = 1.4, 7.3 Hz, 1H), 7.16-7.27 (m, 7H), 7.59 (dd, *J* = 1.5, 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.2, 28.8, 32.2, 43.3, 51.1, 51.3, 63.5, 111.6, 113.8, 123.8, 124.4, 126.2, 126.9, 127.3, 127.5, 127.8, 128.3, 131.5, 131.6, 136.7, 141.2, 141.3, 155.1, 212.0; MS *m/z* 410.2 (M^++1)；Elemental analysis calcd (%) for $C_{28}H_{27}NO_2$: C 82.12, H 6.65, N 3.42; Found: C 82.03, H 6.80, N 3.62. HPLC: OD, *i*-propanol/hexane= 6:94, flow rate 0.5mL/min, λ = 254 nm): *t* =8.8 min, *t* '=13.7 min, racemate.

2-(2,3-diphenyl-1,2-dihydroisoquinolin-1-yl)cyclohexanone (**4b**)

Yield: 61%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.21-1.30 (m, 1H), 1.38-1.50 (m, 2H), 1.66-1.80 (m, 2H), 1.98-2.08 (m, 1H), 2.34-2.42 (m, 1H), 2.48-2.54 (m, 1H), 3.02-3.10 (m, 1H), 5.40 (d, *J* = 8.8 Hz, 1H), 6.77 (s, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 6.93 (d, *J* = 7.8 Hz, 1H), 7.06 (t, *J* = 7.8 Hz, 2H), 7.11 (t, *J* = 7.3 Hz, 1H), 7.17-7.28 (m, 7H), 7.50 (d, *J* =7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.1, 28.9, 32.1, 43.1, 52.0, 63.3, 113.2, 121.8, 122.5, 124.3, 126.2, 126.9, 127.1, 127.3, 128.0, 128.4, 128.5, 129.7, 132.1, 137.6, 141.5, 147.1, 212.8; MS *m/z* 380.2 (M^++1)；Elemental analysis calcd (%) for $C_{27}H_{25}NO$: C 85.45, H 6.64, N 3.69; Found: C 85.34, H 6.58, N 3.63.

Diastereoisomer (**4b'**)

Yield: 25%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.56-1.65 (m, 1H), 1.70-1.83 (m, 2H), 1.85-2.00 (m, 2H), 2.04-2.10 (m, 1H), 2.17-2.25 (m, 1H), 2.32-2.40 (m, 1H), 2.58-2.65 (m, 1H), 2.79-2.88 (m, 1H), 5.50 (d, *J* = 9.3 Hz, 1H), 6.80-6.87 (m, 4H), 7.03-7.15 (m, 3H), 7.18-7.30 (m, 6H), 7.58 (d, *J* =8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 28.9, 32.4, 43.4, 55.0, 62.9, 112.5, 122.0,

122.8, 123.9, 126.3, 127.0, 127.3, 127.4, 127.9, 128.3, 128.5, 131.4, 132.2, 136.5, 140.7, 147.6, 212.0; MS *m/z* 380.2 (M^++1); Elemental analysis calcd (%) for $C_{27}H_{25}NO$: C 85.45, H 6.64, N 3.69; Found: C 85.23, H 6.78, N 3.58.

2-(2-(4-fluorophenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)cyclohexanone (**4c**)

Yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.21-1.30 (m, 1H), 1.38-1.50 (m, 2H), 1.62-1.75 (m, 2H), 2.02-2.08 (m, 1H), 2.34-2.42 (m, 1H), 2.48-2.53 (m, 1H), 3.03-3.09 (m, 1H), 5.25 (d, *J* = 9.2 Hz, 1H), 6.72-6.77 (m, 3H), 6.91 (d, *J* = 7.3 Hz, 1H), 7.13 (dt, *J* = 1.5, 7.3 Hz, 1H), 7.18-7.28 (m, 7H), 7.48 (d, *J* =7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.2, 29.0, 32.3, 43.2, 51.7, 64.0, 112.9, 115.0 ($^2J_{CF}$ =21.9 Hz), 124.2 ($^3J_{CF}$ =7.6 Hz), 124.3, 126.3, 126.9, 127.2, 127.4, 128.1, 128.4, 129.2, 132.0, 137.4, 141.6, 143.4, 158.3 ($^1J_{CF}$ =240.3 Hz), 213.1; MS *m/z* 398.2 (M^++1); Elemental analysis calcd (%) for $C_{27}H_{24}FNO$: C 81.59, H 6.09, N 3.52; Found: C 81.42, H 6.23, N 3.61.

Diastereoisomer (**4c'**)

Yield: 27%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.57-1.65 (m, 1H), 1.70-1.78 (m, 1H), 1.81-1.98 (m, 2H), 2.02-2.10 (m, 1H), 2.17-2.25 (m, 1H), 2.31-2.39 (m, 1H), 2.56-2.62 (m, 1H), 2.76-2.82 (m, 1H), 5.38 (d, *J* = 9.3 Hz, 1H), 6.72-6.77 (m, 2H), 6.80 (s, 1H), 6.81-6.85 (m, 2H), 6.91 (d, *J* = 7.3 Hz, 1H), 7.12 (dt, *J* = 2.0, 7.3 Hz, 1H), 7.18-7.28 (m, 7H), 7.57 (dd, *J* =1.5, 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 28.8, 32.2, 43.3, 55.3, 64.4, 112.5, 115.2 ($^2J_{CF}$ =21.9 Hz), 124.0, 124.3 ($^3J_{CF}$ =8.6 Hz), 126.5, 127.1, 127.3, 127.4, 128.0, 128.4, 131.4, 131.7, 136.3, 141.0, 143.9, 158.3 ($^1J_{CF}$ =241.2 Hz), 211.9; MS *m/z* 398.2 (M^++1); Elemental analysis calcd (%) for $C_{27}H_{24}FNO$: C 81.59, H 6.09, N 3.52; Found: C 81.35, H 6.37, N 3.45.

1-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4d**)

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.3 Hz, 3H), 2.20-2.45 (m, 2H), 2.48 (dd, *J*

= 4.9, 16.6 Hz, 1H), 3.20 (dd, *J* = 9.3, 16.1 Hz, 1H), 3.62 (s, 3H), 5.44 (dd, *J* = 4.9, 9.3 Hz, 1H), 6.61 (d, *J* = 8.8 Hz, 2H), 6.72 (s, 1H), 6.95-7.00 (m, 3H), 7.12 (dt, *J* = 1.4, 6.8 Hz, 1H), 7.16-7.26 (m, 5H), 7.49 (dd, *J* =1.5, 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.4, 37.9, 46.3, 55.2, 61.7, 111.3, 113.9, 124.0, 124.3, 125.2, 126.7, 127.2, 127.3, 127.9, 128.3, 131.6, 131.7, 137.4, 140.6, 140.9, 155.1, 210.2; MS *m/z* 384.2 (M^++1)；Elemental analysis calcd (%) for $C_{26}H_{25}NO_2$: C 81.43, H 6.57, N 3.65; Found: C 81.31, H 6.52, N 3.60.

1-(3-phenyl-2-p-tolyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4e**)

Yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.3 Hz, 3H), 2.15 (s, 3H), 2.24-2.43 (m, 2H), 2.53 (dd, *J* = 4.8, 16.0 Hz, 1H), 3.20 (dd, *J* = 9.3, 16.0 Hz, 1H), 5.52 (dd, *J* = 4.9, 9.3 Hz, 1H), 6.72 (s, 1H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 6.98-7.03 (m, 1H), 7.12 (t, *J* = 6.8 Hz, 1H), 7.16-7.27 (m, 5H), 7.49 (dd, *J* =1.5, 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.4, 20.4, 37.9, 46.2, 61.2, 111.8, 112.3, 124.3, 125.2, 126.6, 127.1, 127.3, 127.9, 128.3, 129.2, 131.5, 131.6, 132.0, 137.4, 140.6, 144.6, 210.1; MS *m/z* 368.2 (M^++1)；Elemental analysis calcd (%) for $C_{26}H_{25}NO$: C 84.98, H 6.86, N 3.81; Found: C 84.66, H 6.68, N 3.70.

1-(2,3-diphenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4f**)

Yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (t, *J* = 7.8 Hz, 3H), 2.22-2.42 (m, 2H), 2.55 (dd, *J* = 5.2, 16.4 Hz, 1H), 3.21 (dd, *J* = 8.8, 16.4 Hz, 1H), 5.58 (dd, *J* = 4.9, 8.8 Hz, 1H), 6.77 (s, 1H), 6.79-6.82 (m, 1H), 7.01-7.09 (m, 5H), 7.12 (dt, *J* = 1.4, 7.6 Hz, 1H), 7.16-7.27 (m, 5H), 7.48 (d, *J* =6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.4, 37.8, 46.1, 60.9, 112.3, 122.0, 122.3, 124.4, 125.2, 126.8, 127.0, 127.4, 128.0, 128.4, 128.5, 131.5, 132.2, 137.3, 140.4, 146.9, 210.0; MS *m/z* 354.2 (M^++1)；Elemental analysis calcd (%) for $C_{25}H_{23}NO$: C 84.95, H 6.56, N 3.96; Found: C 84.80, H 6.38, N 3.87.

1-(2-(4-chlorophenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4g**)

Yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.2 Hz, 3H), 2.20-2.45 (m, 2H), 2.48 (dd, *J*

= 4.8, 16.8 Hz, 1H), 3.22 (dd, *J* = 8.8, 16.4 Hz, 1H), 5.51 (dd, *J* = 4.8, 9.2 Hz, 1H), 6.79 (s, 1H), 6.97-7.06 (m, 5H), 7.14 (dt, *J* = 1.6, 7.6 Hz, 1H), 7.19-7.28 (m, 5H), 7.45 (d, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 37.7, 45.9, 60.9, 112.9, 123.5, 124.6, 125.1, 126.9, 127.0, 127.2, 127.5, 128.2, 128.5, 128.6, 131.4, 132.1, 136.9, 139.9, 145.6, 210.0; MS *m/z* 388.1 (M^++1); Elemental analysis calcd (%) for $C_{25}H_{22}ClNO$: C 77.41, H 5.72, N 3.61; Found: C 77.60, H 5.75, N 3.50.

1-(2-(4-fluorophenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4h**)

Yield: 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.3 Hz, 3H), 2.31-2.40 (m, 2H), 2.46 (dd, *J* = 4.8, 16.8 Hz, 1H), 3.23 (dd, *J* = 9.3, 16.6 Hz, 1H), 5.46 (dd, *J* = 4.9, 9.3 Hz, 1H), 6.72-6.77 (m, 3H), 7.00-7.05 (m, 3H), 7.14 (dt, *J* = 1.5, 6.8 Hz, 1H), 7.19-7.27 (m, 5H), 7.46 (dd, *J* = 2.0, 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.4, 37.8, 46.1, 61.4, 112.2, 115.2 ($^2J_{CF}$ = 22.9 Hz), 123.9 ($^3J_{CF}$ = 8.6 Hz), 124.5, 125.1, 126.9, 127.1, 127.5, 128.1, 128.4, 131.5, 131.8, 137.0 140.4, 143.2, 158.4 ($^1J_{CF}$ = 240.2 Hz), 210.1; MS *m/z* 372.2 (M^++1); Elemental analysis calcd (%) for $C_{25}H_{22}FNO$: C 80.84, H 5.97, N 3.77; Found: C 80.65, H 5.88, N 3.71.

1-(2-(3-nitrophenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4i**)

Yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07 (t, *J* = 7.3 Hz, 3H), 2.34-2.40 (m, 2H), 2.51 (dd, *J* = 4.4, 16.4 Hz, 1H), 3.31 (dd, *J* = 9.7, 16.8 Hz, 1H), 5.62 (dd, *J* = 4.4, 9.7 Hz, 1H), 6.89 (s, 1H), 7.06 (d, *J* = 7.8 Hz, 1H), 7.18 (dt, *J* =1.5, 7.2 Hz, 1H), 7.20-7.32 (m, 6H), 7.45 (dd, *J* = 1.5, 7.8 Hz, 2H), 7.53 (dd, *J* = 2.4, 7.8 Hz, 1H), 7.64 (dd, *J* = 1.6, 8.3 Hz, 1H), 7.93 (t, *J* = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 37.4, 45.6, 60.3, 114.4, 116.4, 116.5, 125.0, 125.1, 126.8, 127.5, 127.6, 127.8, 128.5, 128.8, 129.2, 131.1, 132.5, 136.3, 139.1, 148.0, 148.5, 209.6; MS *m/z* 399.2 (M^++1); Elemental analysis calcd (%) for $C_{25}H_{22}N_2O_3$: C 75.36, H 5.57, N 7.03; Found: C 75.25, H 5.47, N 7.15.

1-(3-phenyl-2-(3-(trifluoromethyl)phenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4j**)

Yield: 68%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (t, *J* = 7.3 Hz, 3H), 2.28-2.40 (m, 2H), 2.53 (dd, *J* = 4.4, 16.4 Hz, 1H), 3.26 (dd, *J* = 9.3, 16.6 Hz, 1H), 5.60 (dd, *J* = 4.8, 9.2 Hz, 1H), 6.83 (s, 1H), 7.04 (t, *J* = 7.8 Hz, 2H), 7.16 (t, *J* = 7.8 Hz, 2H), 7.21-7.30 (m, 6H), 7.35 (s, 1H), 7.44 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 37.6, 45.8, 60.4, 113.6, 118.3, 118.6, 123.9 (q, $^{1}J_{CF}$ = 270.8 Hz), 124.8, 125.0, 125.1, 126.9, 127.2, 127.6, 128.3, 128.6, 129.0, 130.9 (q, $^{2}J_{CF}$ = 31.5 Hz), 131.3, 132.4, 136.7, 139.6, 147.3, 209.7; MS *m/z* 422.2 (M^{+}+1)；Elemental analysis calcd (%) for $C_{26}H_{22}F_3NO$: C 74.10, H 5.26, N 3.32; Found: C 74.25, H 5.40, N 3.36.

1-(2-benzyl-3-phenyl-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4k**)

Yield: 32%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00 (t, *J* = 7.3 Hz, 3H), 2.15-2.38 (m, 2H), 2.42 (dd, *J* = 5.4, 16.4 Hz, 1H), 3.02 (dd, *J* = 5.3, 15.6 Hz, 1H), 4.11 (q, *J* = 15.2 Hz, 2H), 4.88 (dd, *J* = 4.9, 8.8 Hz, 1H), 6.19 (s, 1H), 6.82 (d, *J* =7.3 Hz, 1H), 7.00-7.16 (m, 8H), 7.33-7.42 (m, 3H), 7.60 (d, *J* =6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.6, 37.0, 46.9, 55.7, 56.4, 107.7, 123.6, 125.1, 126.0, 126.9, 127.0, 127.8, 127.9, 128.0, 128.5, 130.8, 132.5, 137.6, 139.1, 145.1, 210.0; MS *m/z* 368.2 (M^{+}+1)；Elemental analysis calcd (%) for $C_{26}H_{25}NO$: C 84.98, H 6.86, N 3.81; Found: C 84.79, H 6.99, N 3.75.

1-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (**4l**)

Yield: 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.09 (s, 3H), 2.50 (dd, *J* = 4.9, 16.6 Hz, 1H), 3.23 (dd, *J* = 8.8, 16.6 Hz, 1H), 3.60 (s, 3H), 5.42 (dd, *J* = 4.9, 8.8 Hz, 1H), 6.59 (d, *J* = 8.8 Hz, 2H), 6.72 (s, 1H), 6.98-7.00 (m, 3H), 7.11 (dt, *J* = 1.4, 7.3 Hz, 1H), 7.17-7.26 (m, 5H), 7.51 (dd, *J* =1.5, 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.8, 47.4, 55.1, 61.5, 111.3, 113.8, 124.0, 124.3, 125.2, 126.6, 127.2, 127.3, 127.9, 128.3, 131.5, 131.7, 137.4, 140.5, 140.9, 155.1, 207.5; MS *m/z* 370.2 (M^{+}+1)；Elemental analysis calcd (%) for $C_{25}H_{23}NO_2$: C 81.27, H 6.27, N 3.79; Found: C 81.45, H 6.35, N 3.64; HPLC: OD, *i*-propanol/hexane= 6:94, flow rate 0.5mL/min, λ = 254 nm): *t* =13.2

min, *t* ’=23.0 min, racemate.

1-(2,3-diphenyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (**4m**)[1]

Yield: 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.09 (s, 3H), 2.58 (dd, *J* = 5.0, 16.5 Hz, 1H), 3.23 (dd, *J* = 8.7, 16.5 Hz, 1H), 5.56 (dd, *J* = 5.0, 8.3 Hz, 1H), 6.76 (s, 1H), 6.75-6.82 (m, 1H), 7.01-7.08 (m, 5H), 7.12 (t, *J* = 7.3 Hz, 1H), 7.17-7.27 (m, 5H), 7.50 (d, *J* =6.8 Hz, 2H).

1-(2-(4-fluorophenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)propan-2-one (**4n**)

Yield: 93%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.11 (s, 3H), 2.49 (dd, *J* = 4.4, 16.6 Hz, 1H), 3.26 (dd, *J* = 9.3, 16.6 Hz, 1H), 5.45 (dd, *J* = 4.4, 9.3 Hz, 1H), 6.72-6.76 (m, 3H), 7.02-7.06 (m, 3H), 7.14 (t, *J* = 6.8 Hz, 1H), 7.20-7.28 (m, 5H), 7.48 (d, *J* =7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.5, 47.2, 61.2, 112.3, 115.2 ($^{2}J_{CF}$ = 22.9 Hz), 123.9 ($^{3}J_{CF}$ = 7.6 Hz), 124.5, 125.1, 126.9, 127.1, 127.5, 128.1, 128.4, 131.5, 131.7, 137.0, 140.5, 143.2, 158.4 ($^{1}J_{CF}$ = 241.2 Hz), 207.3; MS *m/z* 358.2 (M^{+}+1)；Elemental analysis calcd (%) for $C_{24}H_{20}FNO$: C 80.65, H 5.64, N 3.92; Found: C 80.70, H 5.70, N 3.85.

1-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)pentan-2-one (**4o**)

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.3 Hz, 3H), 1.53-1.61 (m, 2H), 2.20-2.38 (m, 2H), 2.44 (dd, *J* = 4.9, 16.6 Hz, 1H), 3.20 (dd, *J* = 9.3, 16.6 Hz, 1H), 3.59 (s, 3H), 5.44 (dd, *J* = 4.9, 8.9 Hz, 1H), 6.59 (d, *J* = 9.3 Hz, 2H), 6.72 (s, 1H), 6.95-7.00 (m, 3H), 7.12 (dt, *J* = 1.4, 6.8 Hz, 1H), 7.16-7.26 (m, 5H), 7.49 (d, *J* =6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 16.8，46.5, 46.6, 55.1, 61.6, 111.3, 113.8, 124.0, 124.3, 125.2, 126.6, 127.2, 127.3, 127.9, 128.3, 131.6, 131.7, 137.3, 140.6, 140.9, 155.1, 209.7; MS *m/z* 398.2 (M^{+}+1)；Elemental analysis calcd (%) for $C_{27}H_{27}NO_2$: C 81.58, H 6.85, N 3.52; Found: C 81.42, H 6.63, N 3.61.

1-(4-methoxyphenyl)-2-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)ethanone (**4p**)

Yield: 61%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.84 (dd, *J* = 4.4, 15.6 Hz, 1H), 3.63 (s, 3H), 3.81 (s,

3H), 3.84 (dd, *J* = 9.2, 16.0 Hz, 1H), 5.62 (dd, *J* = 4.4, 9.3 Hz, 1H), 6.59 (d, *J* = 9.2 Hz, 2H), 6.73 (s, 1H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 9.2 Hz, 2H), 7.08-7.15 (m, 5H), 7.21-7.29 (m, 2H), 7.33-7.35 (m, 2H), 7.91 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.2, 55.2, 55.4，62.7, 111.2, 113.7, 113.8，124.1, 124.2, 125.3, 126.6, 127.3, 127.5, 127.7, 128.0, 130.6，130.7，131.7, 131.8, 137.3, 140.6, 141.3, 155.0, 163.5，197.7; MS *m/z* 462.2 (M^++1)；Elemental analysis calcd (%) for $C_{31}H_{27}NO_3$: C 80.67, H 5.90, N 3.03; Found: C 80.55, H 5.81, N 3.25.

(Z)-4-hydroxy-5-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)pent-3-en-2-one (**4q**)

Yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.04 (s, 3H), 2.41 (dd, *J* = 6.0, 14.6 Hz, 1H), 2.86 (dd, *J* = 9.2, 16.1 Hz, 1H), 3.63 (s, 3H), 5.38 (dd, *J* = 4.6, 10.6 Hz, 1H), 5.45 (s, 1H), 6.59 (d, *J* = 8.4 Hz, 2H), 6.71 (s, 1H), 6.86 (d, *J* = 9.2 Hz, 2H), 7.03 (d, *J* = 7.6 Hz, 1H), 7.12 (dt, *J* = 1.5, 6.8 Hz, 1H), 7.19-7.29 (m, 5H), 7.55 (dd, *J* =1.6, 7.6 Hz, 2H), 15.76 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.4, 42.7, 55.2, 63.4, 102.6, 110.9, 113.9, 124.0, 124.3, 125.1, 126.5, 127.4, 127.6, 127.9, 128.1, 131.3, 131.6, 137.2, 140.6, 140.7, 155.1, 189.1, 193.4; MS *m/z* 412.2 (M^++1)；Elemental analysis calcd (%) for $C_{27}H_{25}NO_3$: C 78.81, H 6.12, N 3.40; Found: C 78.95, H 6.30, N 3.55.

2-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)pentan-3-one (**4r**)

Yield: 30%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (d, *J* = 6.8 Hz, 3H), 1.07 (t, *J* = 6.8 Hz, 3H), 2.37-2.54 (m, 2H), 3.12-3.21 (m, 1H), 3.63 (s, 3H), 4.94 (d, *J* = 10.8 Hz, 1H), 6.59 (d, *J* = 9.3 Hz, 2H), 6.86 (s, 1H), 6.93-6.96 (m, 3H), 7.14 (t, *J* = 6.8 Hz, 1H), 7.18-7.30 (m, 5H), 7.47 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.2, 15.3, 37.6, 46.3, 55.2, 68.5, 111.5, 113.8, 124.1, 124.3, 126.1, 127.1, 127.2, 127.4, 128.0, 128.3, 129.3, 132.0, 136.7, 140.6, 141.1, 155.3, 215.9; MS *m/z* 398.2 (M^++1)；Elemental analysis calcd (%) for $C_{27}H_{27}NO_2$: C 81.58, H 6.85, N 3.52; Found: C 81.32, H 6.65, N 3.32.

Diastereoisomer (**4r'**)

Yield: 20%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (t, *J* = 7.9 Hz, 3H), 1.49 (d, *J* = 6.8 Hz, 3H), 2.00-2.08 (m, 2H), 3.20-3.23 (m, 1H), 3.65 (s, 3H), 4.87 (d, *J* = 10.2 Hz, 1H), 6.63 (d, *J* = 9.3 Hz, 2H), 6.73 (s, 1H), 6.79 (d, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 12.2 Hz, 2H), 7.05 (t, *J* = 7.8 Hz, 1H), 7.18-7.28 (m, 5H), 7.60 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.1, 15.1, 38.2, 47.5, 55.3, 67.9, 111.3, 113.9, 124.2, 124.3, 126.2, 126.7, 127.3, 127.5, 128.0, 128.3, 130.2, 131.7, 137.1, 140.7, 141.1, 155.2, 214.9; MS *m/z* 398.2 (M^++1); Elemental analysis calcd (%) for $C_{27}H_{27}NO_2$: C 81.58, H 6.85, N 3.52; Found: C 81.38, H 7.05, N 3.28.

1-(2,3-bis(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4s**)

Yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.2 Hz, 3H), 2.27-2.42 (m, 2H), 2.46 (dd, *J* = 4.8, 16.0 Hz, 1H), 3.20 (dd, *J* = 9.2, 16.0 Hz, 1H), 3.64 (s, 3H), 3.74 (s, 3H), 5.41 (dd, *J* = 4.8, 9.6 Hz, 1H), 6.61 (d, *J* = 9.2 Hz, 2H), 6.64 (s, 1H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.96-6.99 (m, 3H), 7.10 (dt, *J* = 1.2, 6.8 Hz, 1H), 7.19-7.24 (m, 2H), 7.42 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 37.9, 46.2, 55.1, 55.2, 61.8, 109.9, 113.7, 113.8, 124.1, 125.2, 126.3, 127.2, 127.3, 128.5, 129.8, 131.5, 132.0, 137.4, 140.6, 140.7, 155.1, 159.4, 210.4; MS *m/z* 414.2 (M^++1); Elemental analysis calcd (%) for $C_{27}H_{27}NO_3$: C 78.42, H 6.58, N 3.39; Found: C 78.24, H 6.50, N 3.55.

1-(2-(4-fluorophenyl)-3-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4t**)

Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (t, *J* = 7.6 Hz, 3H), 2.26-2.42 (m, 2H), 2.45 (dd, *J* = 4.8, 16.8 Hz, 1H), 3.23 (dd, *J* = 9.6, 16.4 Hz, 1H), 3.75 (s, 3H), 5.44 (dd, *J* = 4.4, 9.2 Hz, 1H), 6.68 (s, 1H), 6.73-6.79 (m, 4H), 6.99-7.05 (m, 3H), 7.12 (dt, *J* = 2.0, 7.2 Hz, 1H), 7.20-7.25 (m, 2H), 7.39 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 37.8, 46.0, 55.1, 61.5, 110.7, 113.8, 115.2 ($^{2}J_{CF}$ = 22.2 Hz), 124.0 ($^{3}J_{CF}$ = 7.9 Hz), 124.3, 125.1, 126.6, 127.4, 128.4, 129.5,

131.6, 131.8, 140.2, 143.4, 158.4 ($^1J_{CF}$ = 242.3 Hz), 159.6, 210.3; MS *m/z* 402.2 (M^++1); Elemental analysis calcd (%) for $C_{26}H_{24}FNO_2$: C 77.78, H 6.03, N 3.49; Found: C 77.57, H 6.15, N 3.42.

1-(3-cyclopropyl-2-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4u**)

Yield: 30%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.63-0.76 (m, 4H), 0.99 (t, *J* = 7.3 Hz, 3H), 1.26-1.34 (m,1H), 2.18-2.28 (m, 1H), 2.34-2.45 (m, 1H), 2.54 (dd, *J* = 5.9, 14.6 Hz, 1H), 2.99 (dd, *J* = 8.3, 15.2 Hz, 1H), 3.75 (s, 3H), 5.18 (t, *J* = 7.8 Hz, 1H), 5.82 (s, 1H), 6.78 (d, *J* = 8.3 Hz, 2H), 6.90 (d, *J* = 8.0 Hz, 1H), 6.98-7.02 (m, 2H), 7.08 (d, *J* = 8.3 Hz, 2H), 7.13 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 7.7, 10.1, 14.1, 38.0, 46.4, 55.4, 62.1, 102.4, 113.9, 122.9, 125.1, 125.2, 125.3, 127.2, 129.7, 131.9, 140.0, 145.3, 155.9, 210.5; MS *m/z* 348.2 (M^++1); Elemental analysis calcd (%) for $C_{23}H_{25}NO_2$: C 79.51, H 7.25, N 4.03; Found: C 79.35, H 7.17, N 4.10.

1-(3-cyclopropyl-2-(4-fluorophenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4v**)

Yield: 35%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.64-0.82 (m, 4H), 1.01 (t, *J* = 7.3 Hz, 3H), 1.29-1.35 (m, 1H), 2.23-2.32 (m, 1H), 2.35-2.45 (m, 1H), 2.49 (dd, *J* = 5.4, 14.6 Hz, 1H), 3.04 (dd, *J* = 8.3, 15.6 Hz, 1H), 5.21 (dd, *J* = 5.4, 8.3 Hz, 1H), 5.92 (s, 1H), 6.89-6.94 (m, 3H), 7.02 (d, *J* = 7.3 Hz, 2H), 7.10-7.16 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5, 7.9, 10.1, 14.1, 38.0, 46.3, 61.8, 104.4, 115.3 ($^2J_{CF}$ = 21.9 Hz), 123.2, 124.8 ($^3J_{CF}$ = 7.6 Hz), 125.0, 125.6, 127.3, 130.1, 131.6, 142.8, 144.7, 159.0 ($^1J_{CF}$ = 241.2 Hz), 210.4; MS *m/z* 336.2 (M^++1); Elemental analysis calcd (%) for $C_{22}H_{22}FNO$: C 78.78, H 6.61, N 4.18; Found: C 78.98, H 6.57, N 4.05.

1-(3-butyl-2-(4-methoxyphenyl)-1,2-dihydroisoquinolin-1-yl)butan-2-one (**4w**)

Yield: 28%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.84 (t, *J* = 7.3 Hz, 3H), 0.99 (t, *J* = 7.3 Hz, 3H), 1.22-1.45 (m, 4H), 2.07-2.40 (m, 4H), 2.57 (dd, *J* = 6.3, 16.1 Hz, 1H), 3.01 (dd, *J* = 7.8, 16.1 Hz, 1H), 3.75 (s, 3H), 5.44 (dd, *J* = 5.9, 7.3 Hz, 1H), 6.05 (s, 1H), 6.77 (d, *J* = 8.3 Hz, 2H), 6.89 (d, *J* = 7.3 Hz, 1H), 7.00-7.06 (m, 4H), 7.15 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.5,

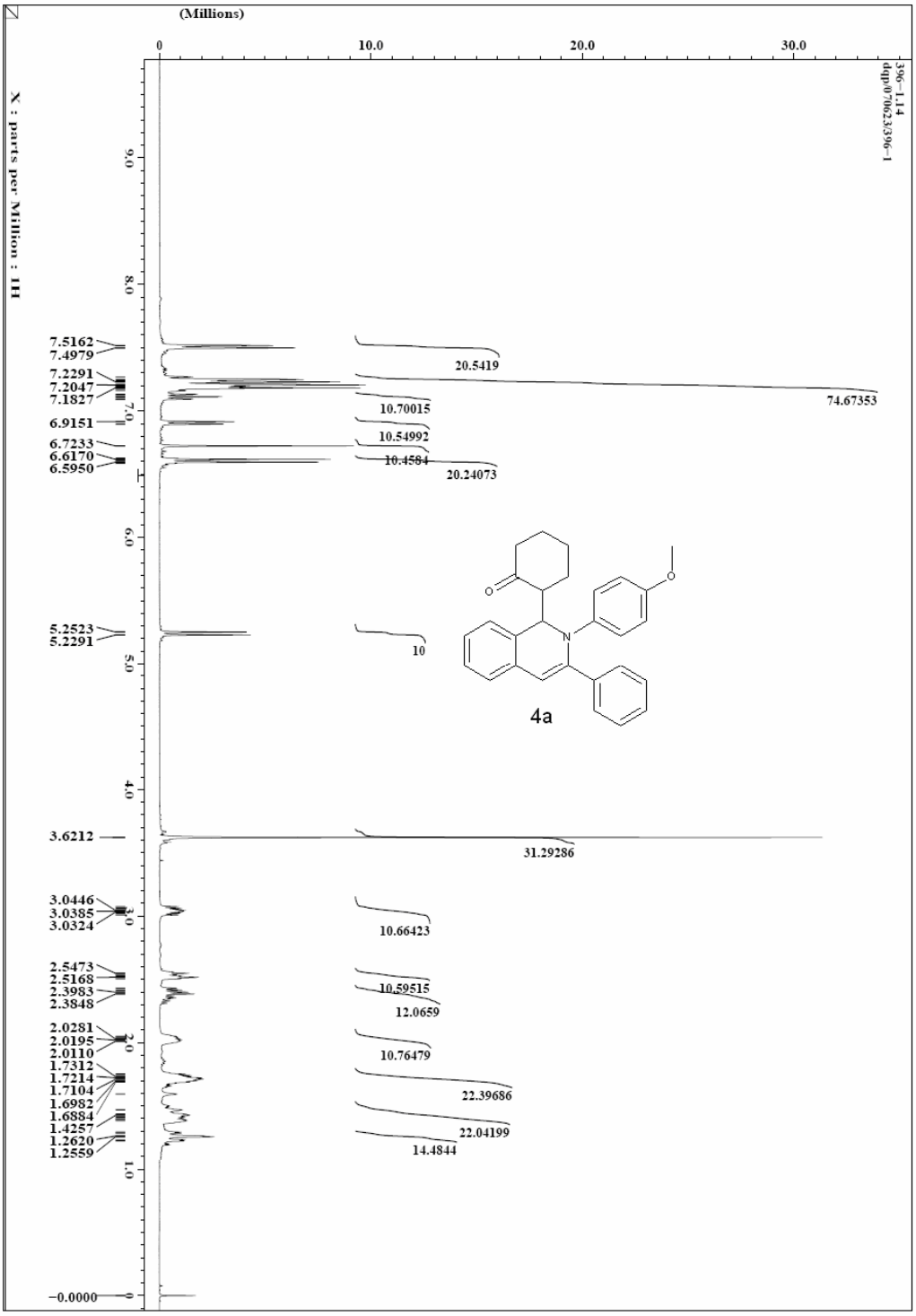
13.8, 22.3, 30.6, 32.7, 37.7, 46.8, 55.4, 61.7, 108.2, 114.0, 123.1, 125.1, 125.4, 125.5, 127.1, 129.7, 131.8, 139.9, 143.6, 156.0, 210.0; MS *m/z* 364.2 (M^++1); Elemental analysis calcd (%) for $C_{24}H_{29}NO_2$: C 79.30, H 8.04, N 3.85; Found: C 79.56, H 8.20, N 3.75.

2-(2-(4-methoxyphenyl)-3-phenyl-1,2-dihydroisoquinolin-1-yl)pentan-3-one (**4x**)

Yield: 12%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.8 Hz, 3H), 1.98-2.05 (m, 1H), 2.23-2.28 (m, 1H), 2.68 (dd, *J* = 3.9, 15.2 Hz, 1H), 3.10 (dd, *J* = 8.8, 15.2 Hz, 1H), 3.78 (s, 3H), 5.51-5.55 (m, 1H), 5.82 (d, *J* = 7.3 Hz, 1H), 6.54 (d, *J* = 7.8 Hz, 1H), 6.88 (d, *J* = 8.9 Hz, 2H), 7.00-7.10 (m, 5H), 7.17 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.3, 37.9, 44.7, 55.6, 56.6, 103.7, 114.7, 117.6, 123.3, 125.6, 125.8, 127.5, 129.6, 129.7, 131.2, 138.6, 154.6, 210.1; MS *m/z* 308.1 (M^++1); Elemental analysis calcd (%) for $C_{20}H_{21}NO_2$: C 78.15, H 6.89, N 4.56; Found: C 77.85, H 6.95, N 4.36

Reference:

1. Asao, N.; Yudha S., S.; Nogami, T.; Yamamoto, Y. *Angew. Chem. Int. Ed.* **2005**, *44*, 5526-5528.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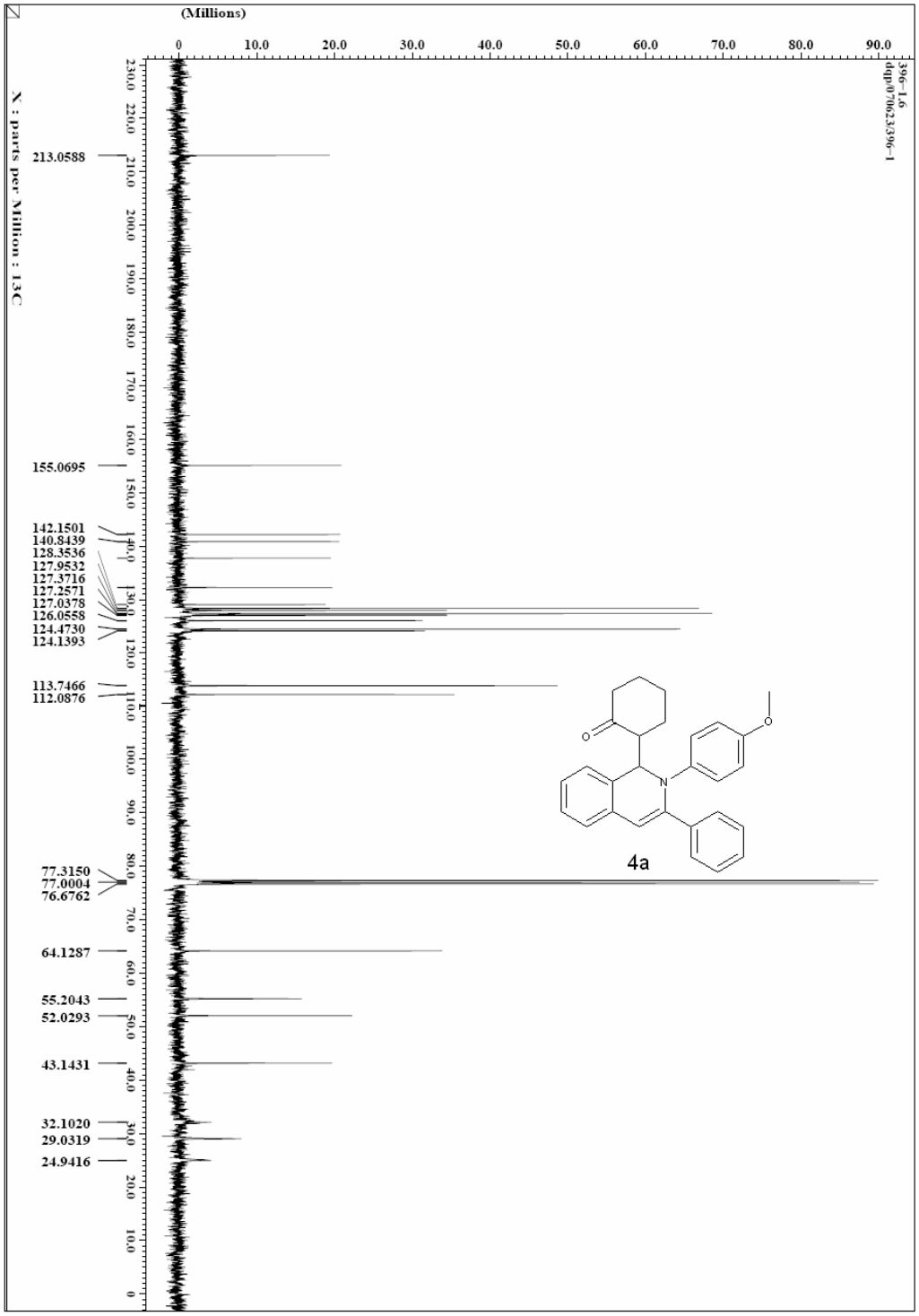
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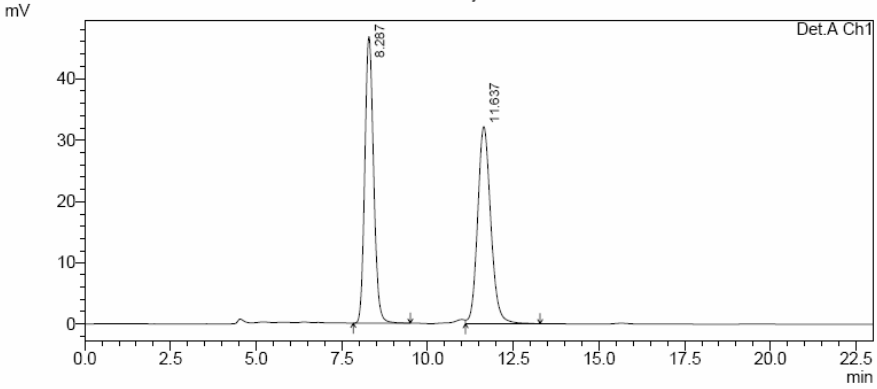

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Detector A Ch1 254nm

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4a sample

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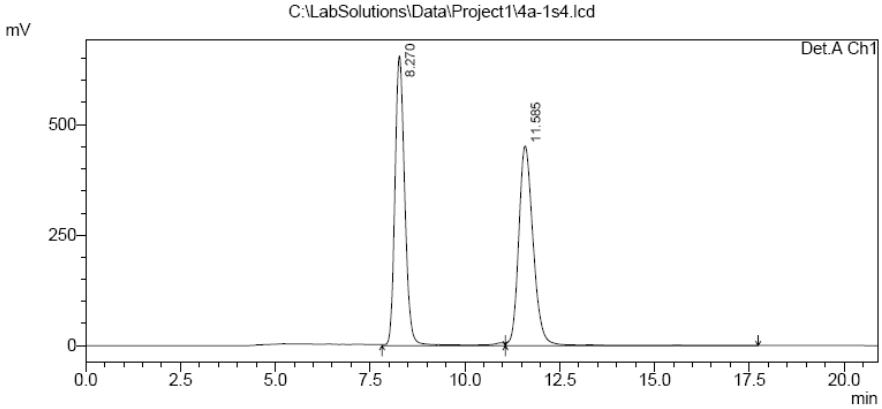

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PeakTable

Detector A Ch1 254nm

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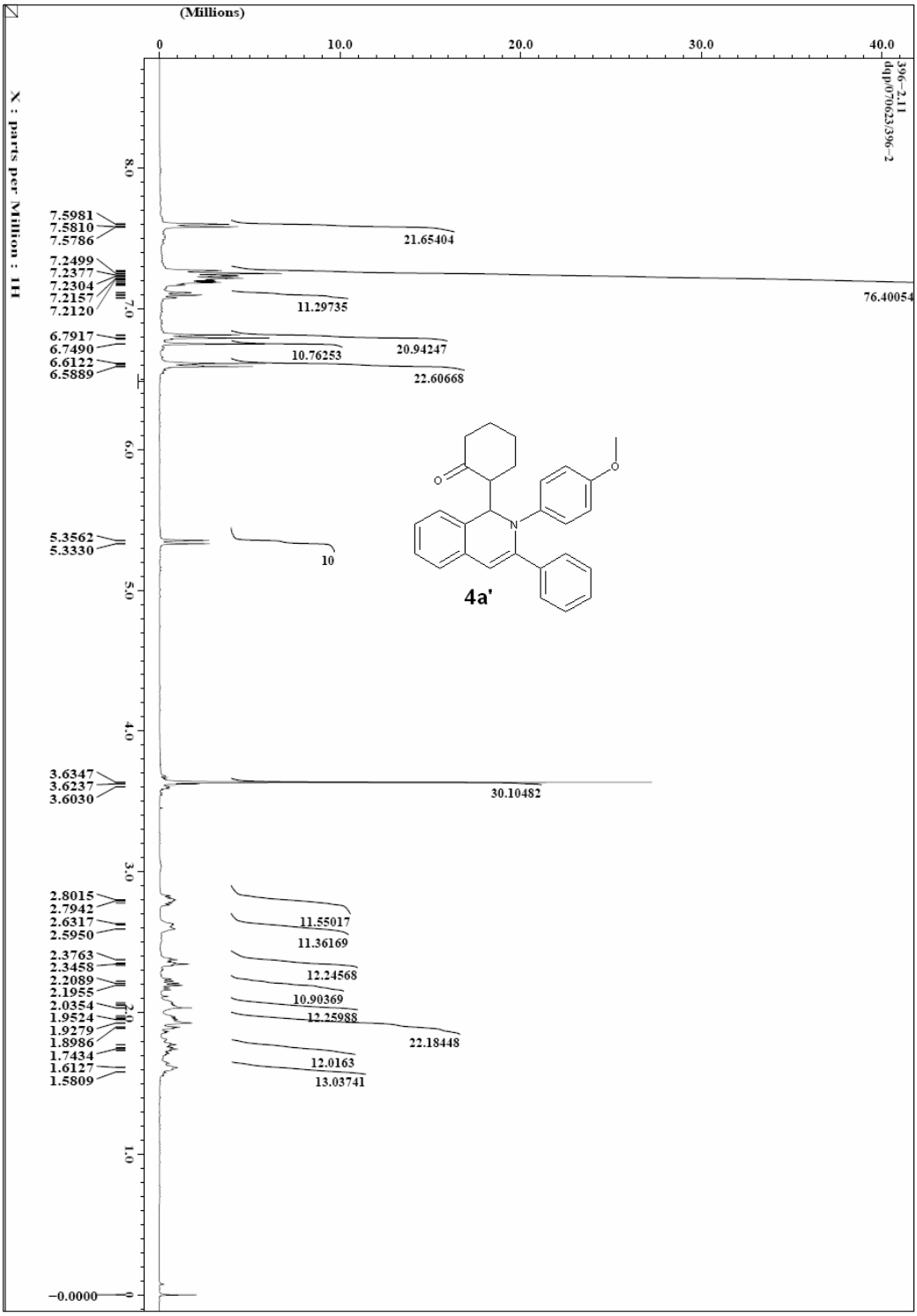
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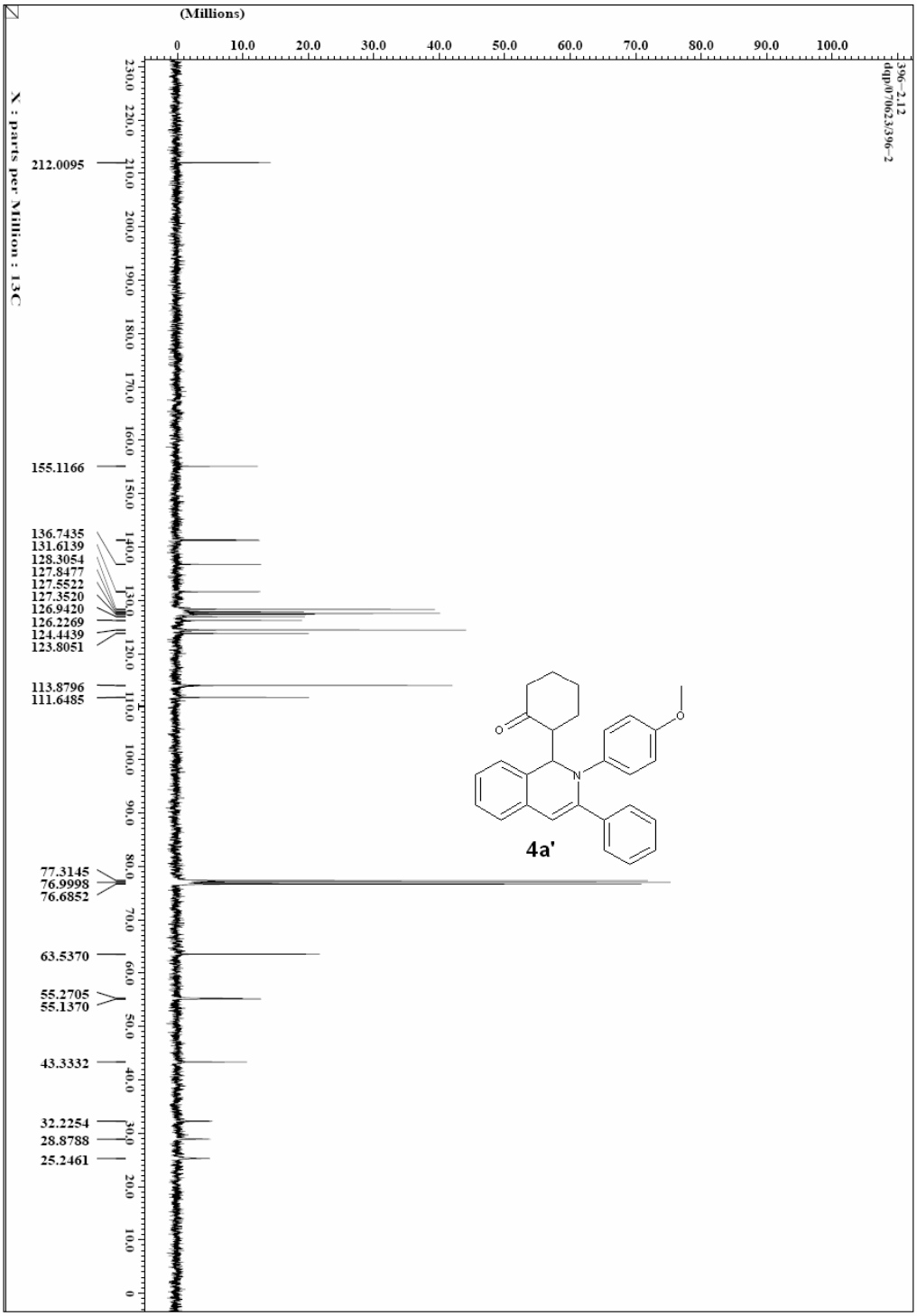
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32.2254
28.8788
25.2461
4a'

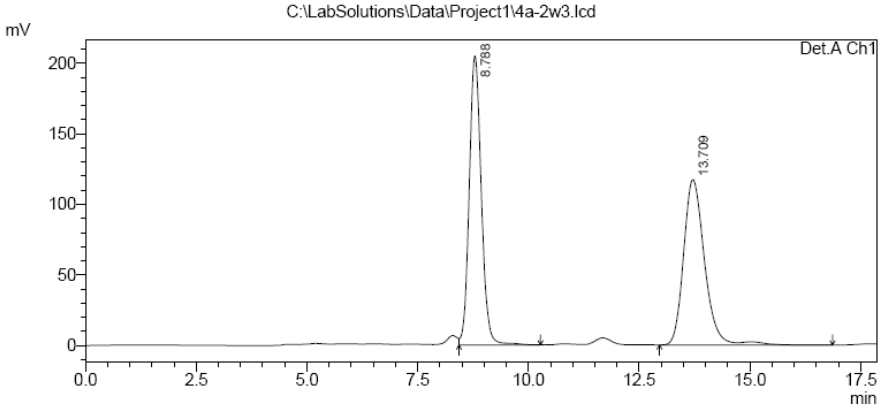
4a' racemic

C:\LabSolutions\Data\Project1\4a-2w3.lcd

Acquired by	: Admin
Sample Name	: dqp
Sample ID	:
Vail #	:
Injection Volume	: 10 uL
Data File Name	: 4a-2w3.lcd
Method File Name	: 4a-1.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2007-10-7 21:32:26
Data Processed	: 2007-10-7 21:57:46

<Chromatogram>

C:\LabSolutions\Data\Project1\4a-2w3.lcd



1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.788	3882734	204915	50.237	63.604
2	13.709	3846074	117257	49.763	36.396
Total		7728808	322172	100.000	100.000

C:\LabSolutions\Data\Project1\4a-2w3.lcd

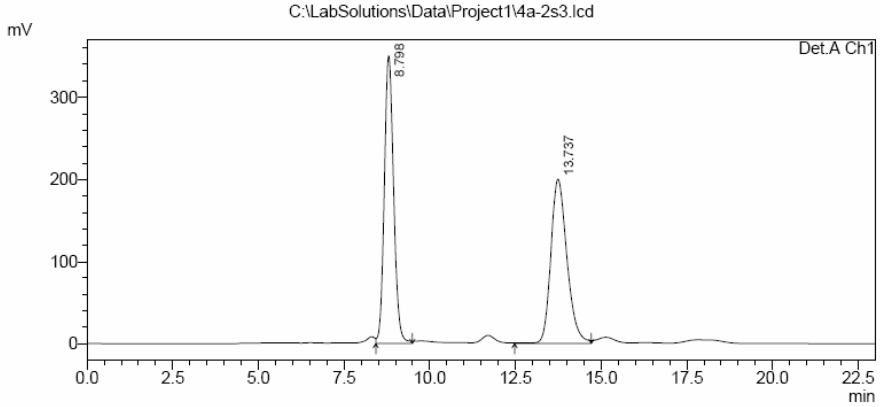
4a' sample

C:\LabSolutions\Data\Project1\4a-2s3.lcd

Acquired by	: Admin
Sample Name	: dqp
Sample ID	:
Vail #	:
Injection Volume	: 10 uL
Data File Name	: 4a-2s3.lcd
Method File Name	: 4a-1.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2007-10-7 23:09:45
Data Processed	: 2007-10-7 23:35:59

<Chromatogram>

C:\LabSolutions\Data\Project1\4a-2s3.lcd



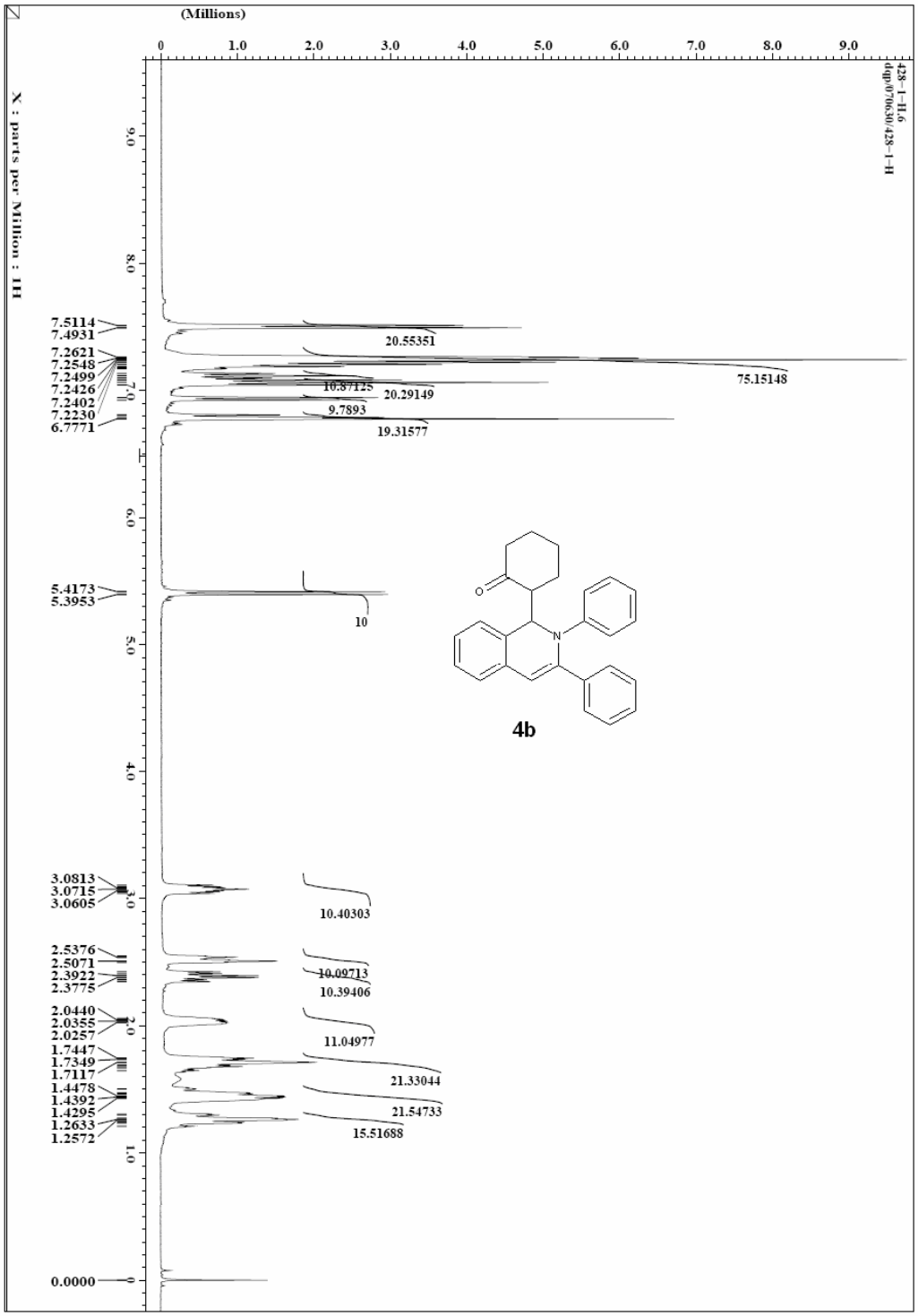
1 Det.A Ch1/254nm

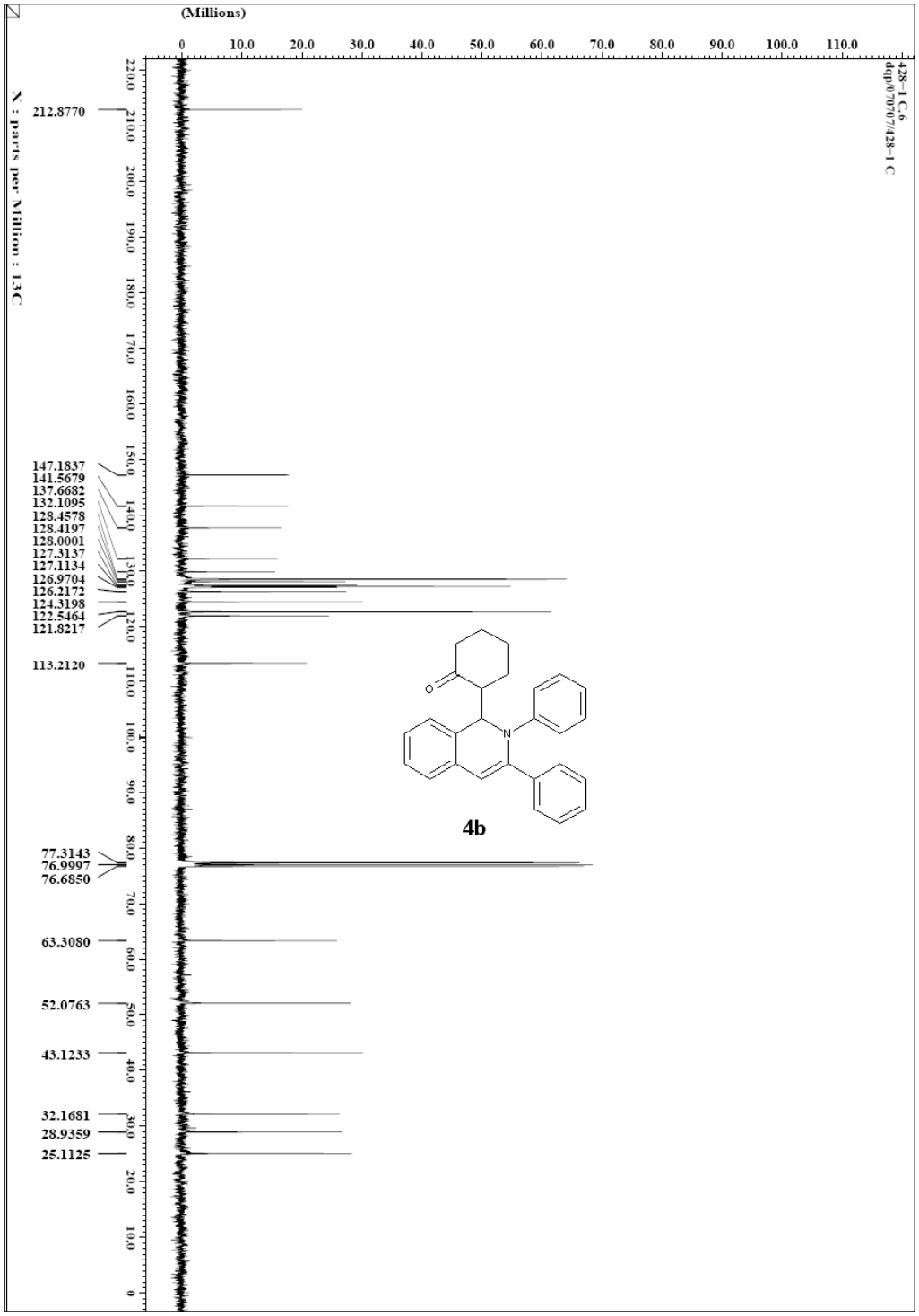
PeakTable

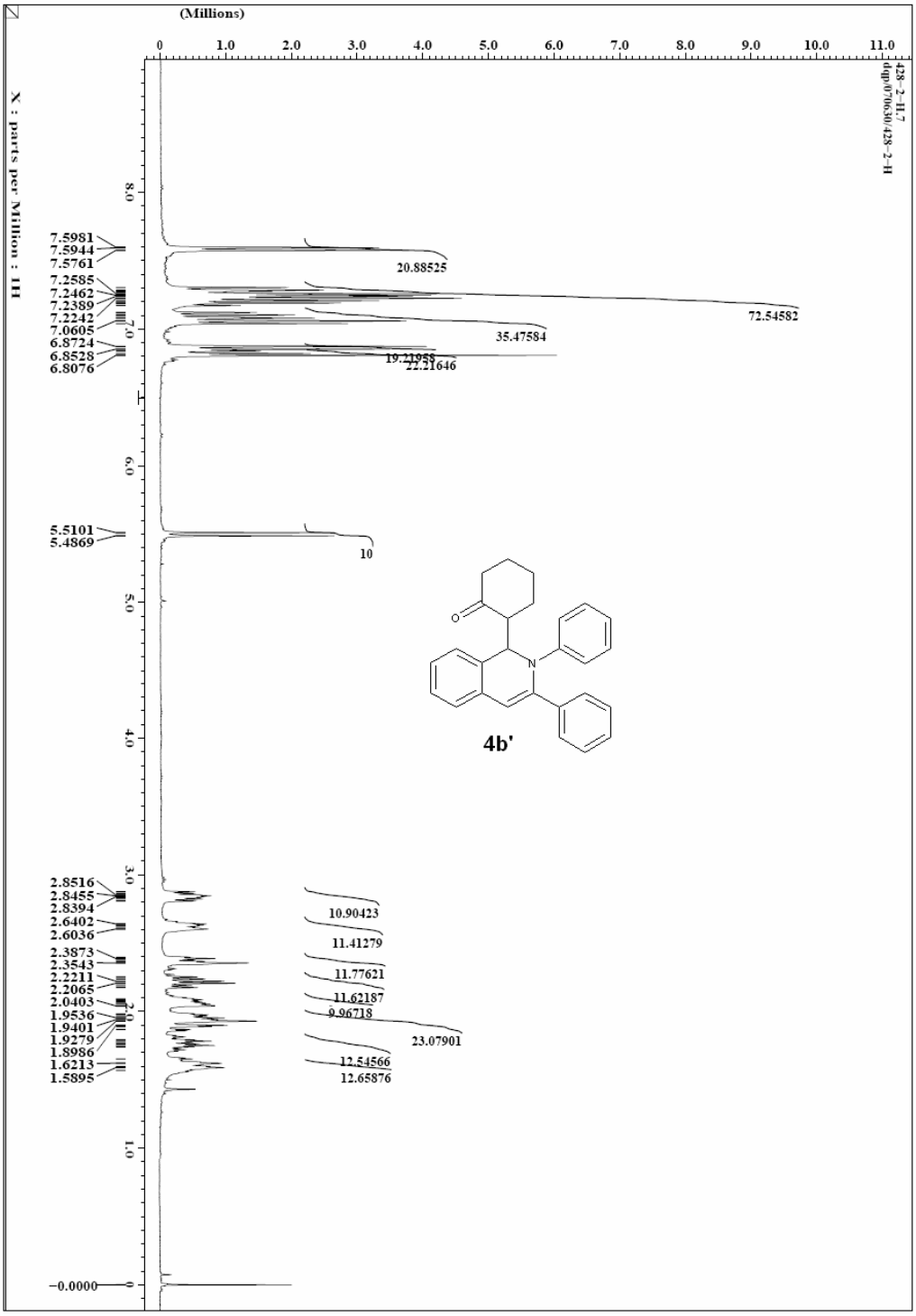
Detector A Ch1 254nm

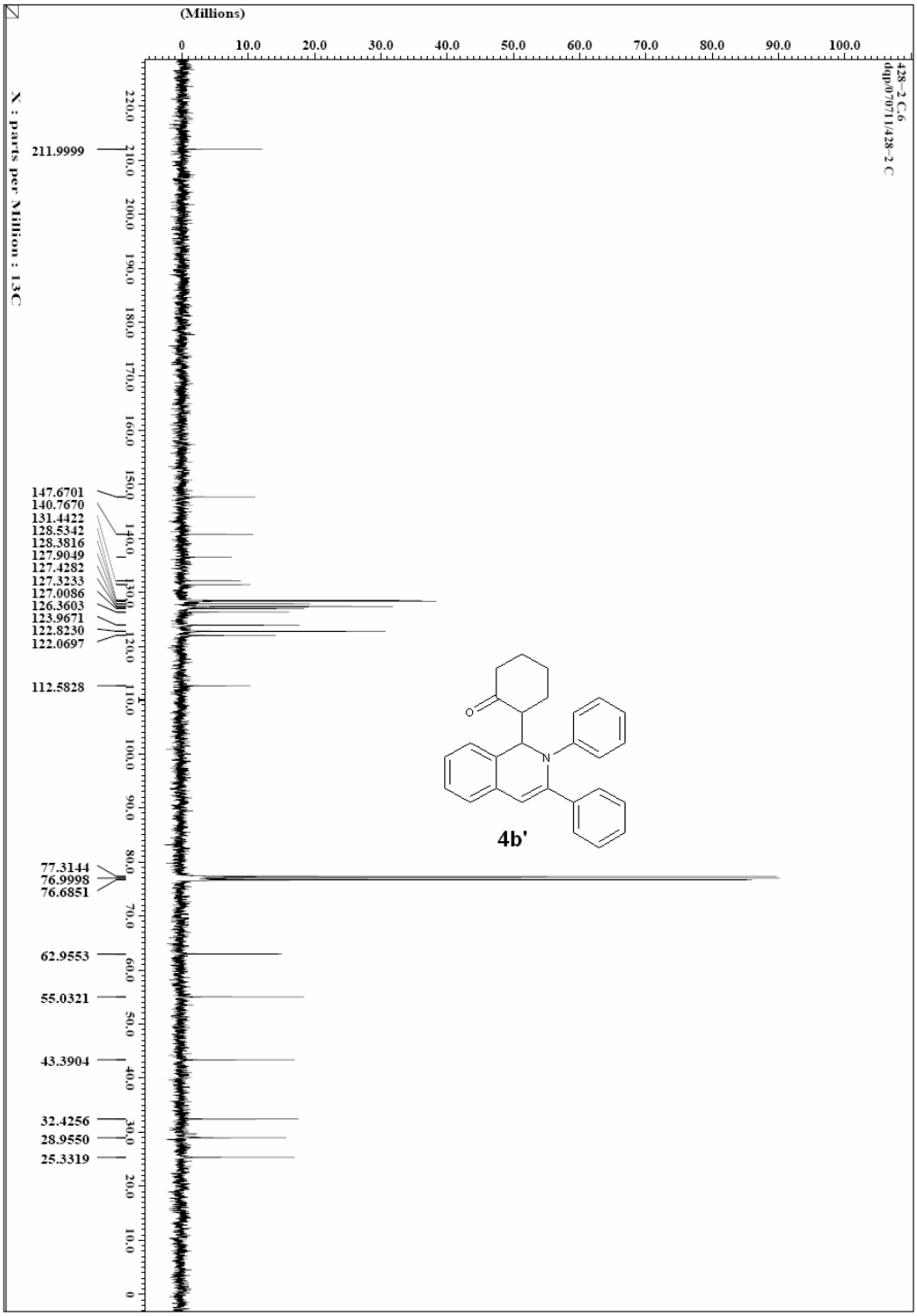
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.798	6630078	350436	50.164	63.580
2	13.737	6586655	200739	49.836	36.420
Total		13216734	551176	100.000	100.000

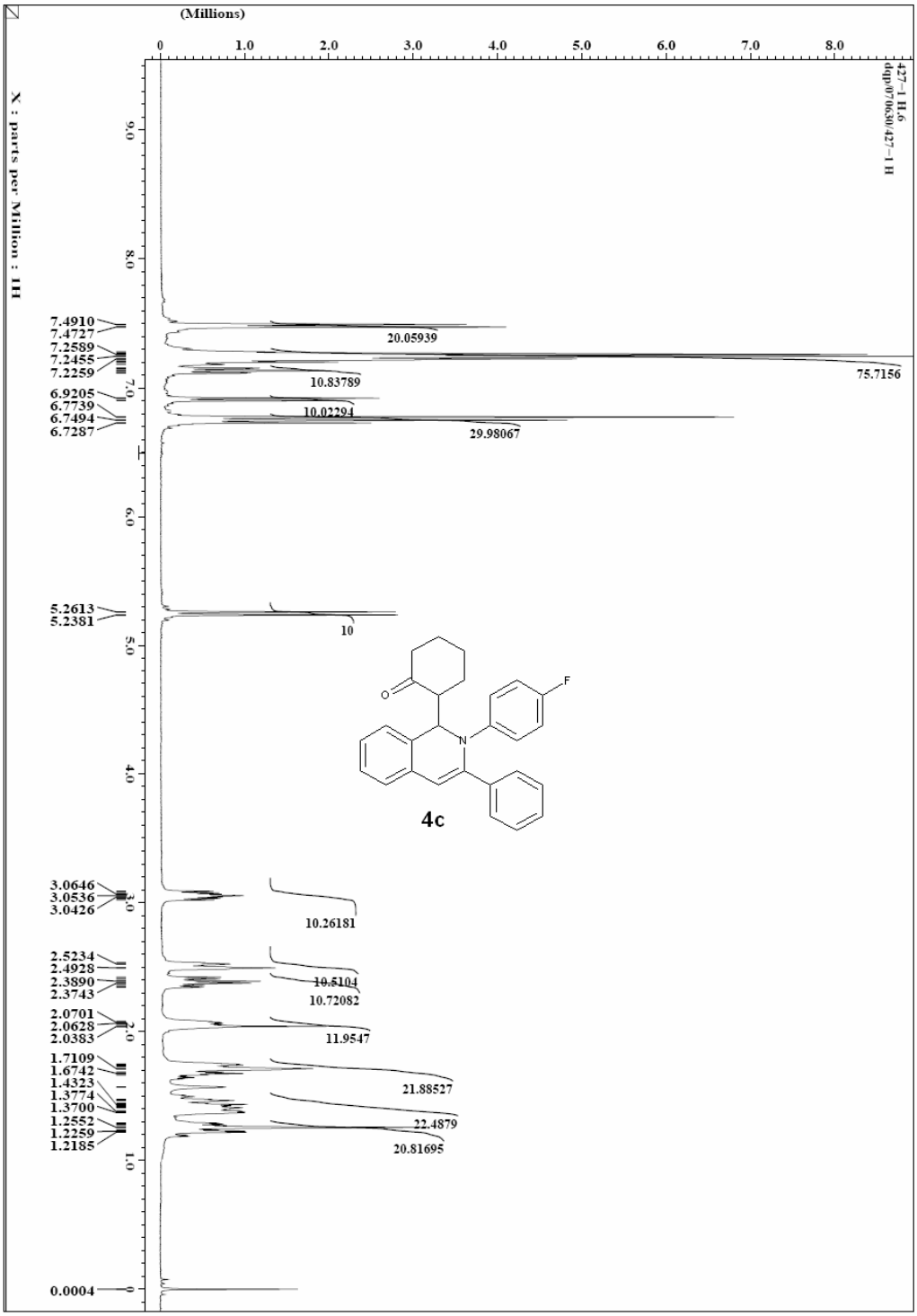
C:\LabSolutions\Data\Project1\4a-2s3.lcd

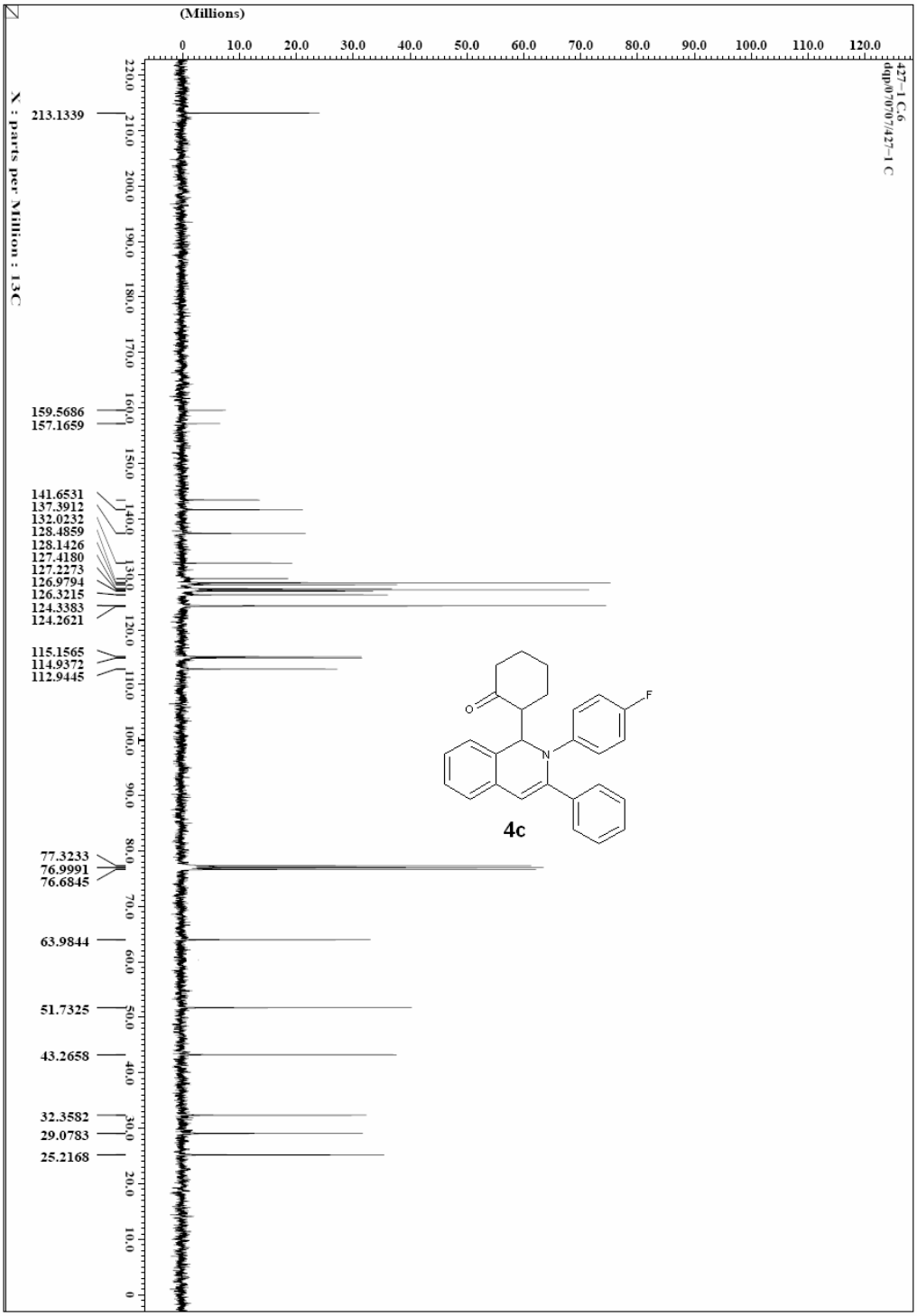

(Millions)
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
X : parts per Million : 1H
428-1-H.6
dqp,0706,30,428-1-H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.5114
7.4931
7.2621
7.2548
7.2499
7.2426
7.2402
7.2230
6.7771
5.4173
5.3953
3.0813
3.0715
3.0605
2.5376
2.5071
2.3922
2.3775
2.0440
2.0355
2.0257
1.7447
1.7349
1.7117
1.4478
1.4392
1.4295
1.2633
1.2572
0.0000
20.55351
75.15148
10.87125
20.29149
9.7893
19.31577
10
10.40303
10.09713
10.39406
11.04977
21.33044
21.54733
15.51688
O
N
4b

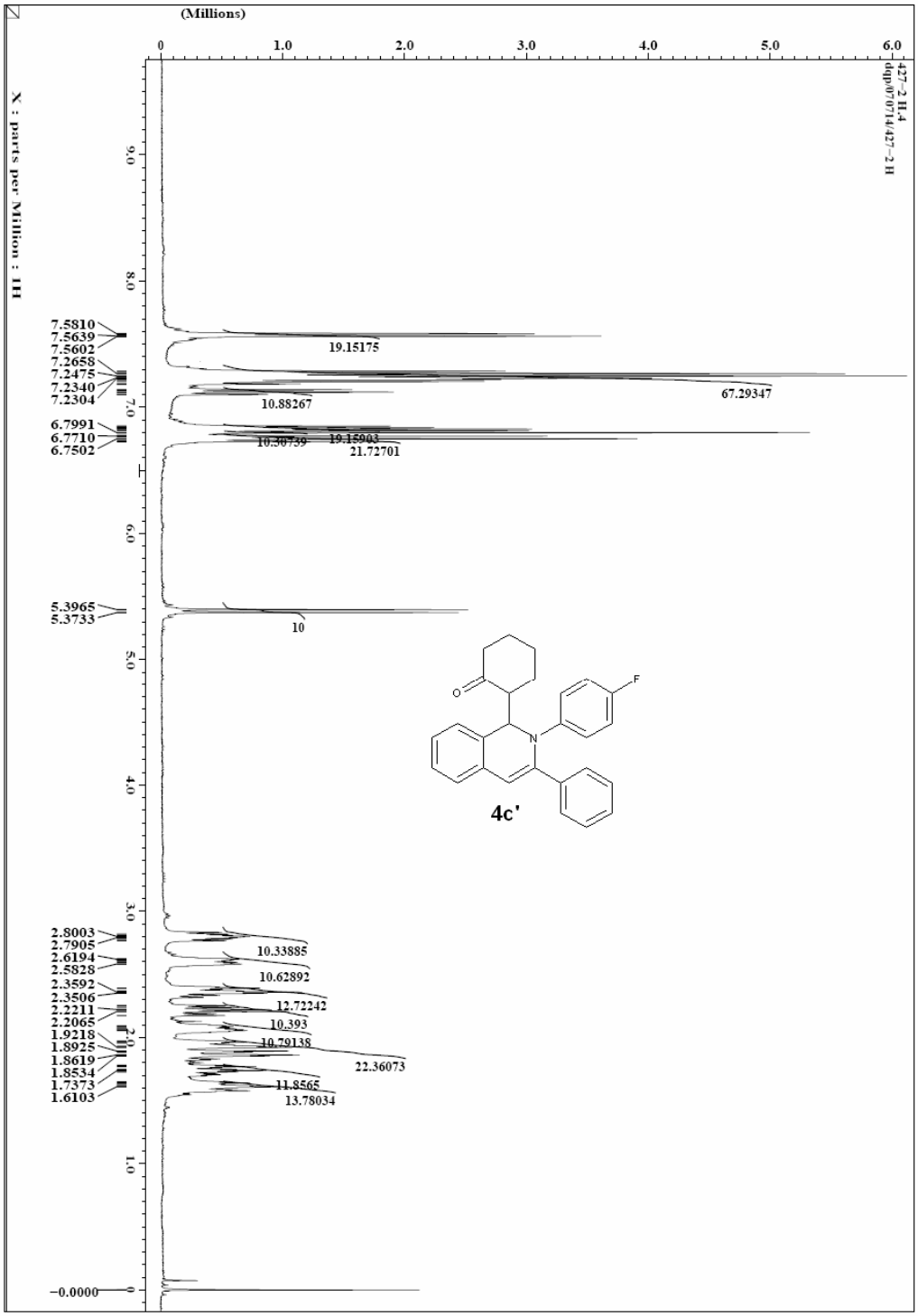

(Millions)
0 10.0 20.0 30.0 40.0 50.0 60.0 70.0 80.0 90.0 100.0 110.0
428-1 C-6
dqp/070707/428-1 C
X : parts per Million : 13C
212.8770
147.1837
141.5679
137.6682
132.1095
128.4578
128.4197
128.0001
127.3137
127.1134
126.9704
126.2172
124.3198
122.5464
121.8217
113.2120
77.3143
76.9997
76.6850
63.3080
52.0763
43.1233
32.1681
28.9359
25.1125
O
N
4b

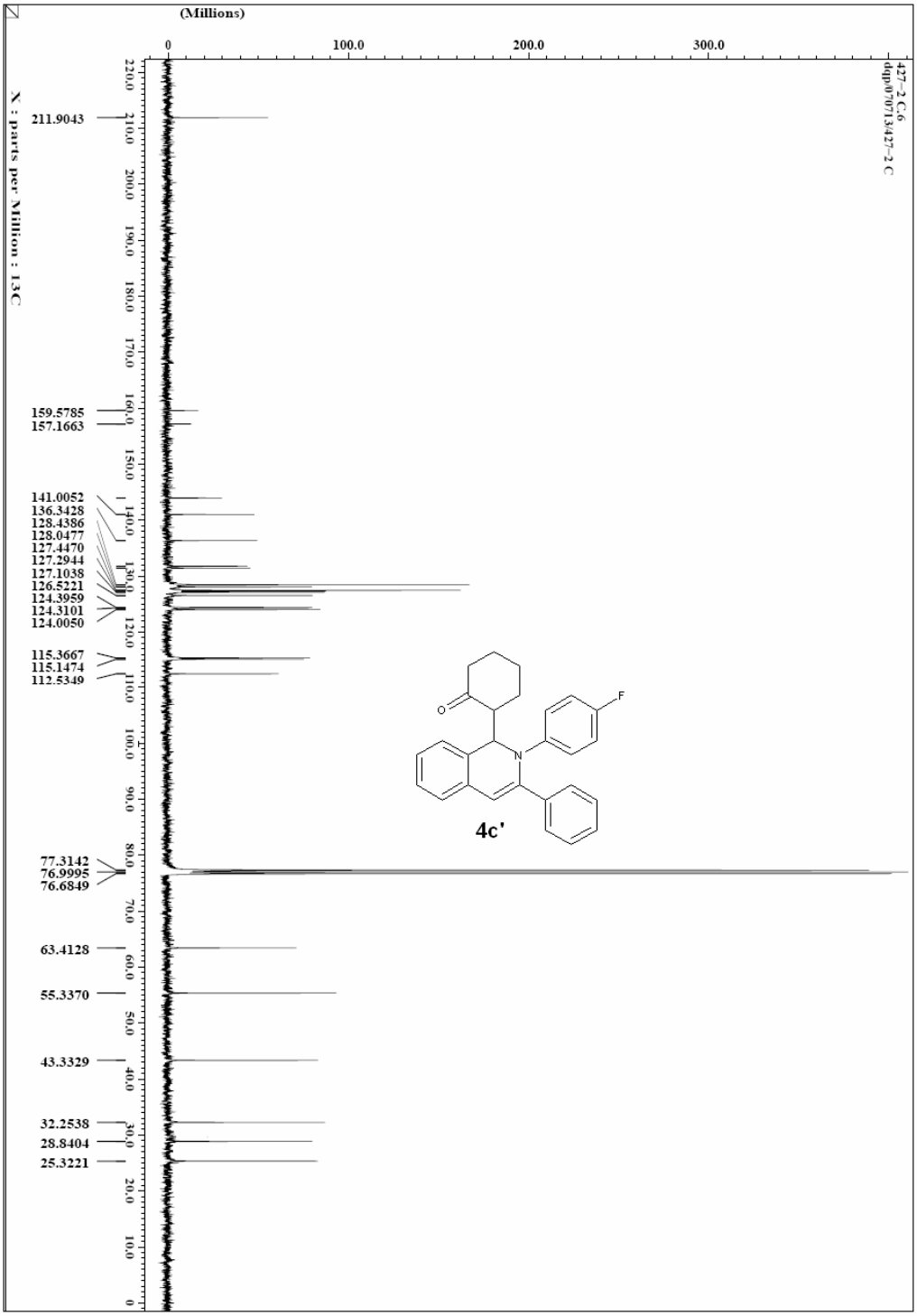

(Millions)
X : parts per Million : 1H
428-2-H.7
dqp/070630/428-2-H
7.5981
7.5944
7.5761
7.2585
7.2462
7.2389
7.2242
7.0605
6.8724
6.8528
6.8076
5.5101
5.4869
2.8516
2.8455
2.8394
2.6402
2.6036
2.3873
2.3543
2.2211
2.2065
2.0403
1.9536
1.9401
1.9279
1.8986
1.6213
1.5895
-0.0000
20.88525
72.54582
35.47584
19.21955
22.21646
10
10.90423
11.41279
11.77621
11.62187
9.96718
23.07901
12.54566
12.65876
O
N
4b'

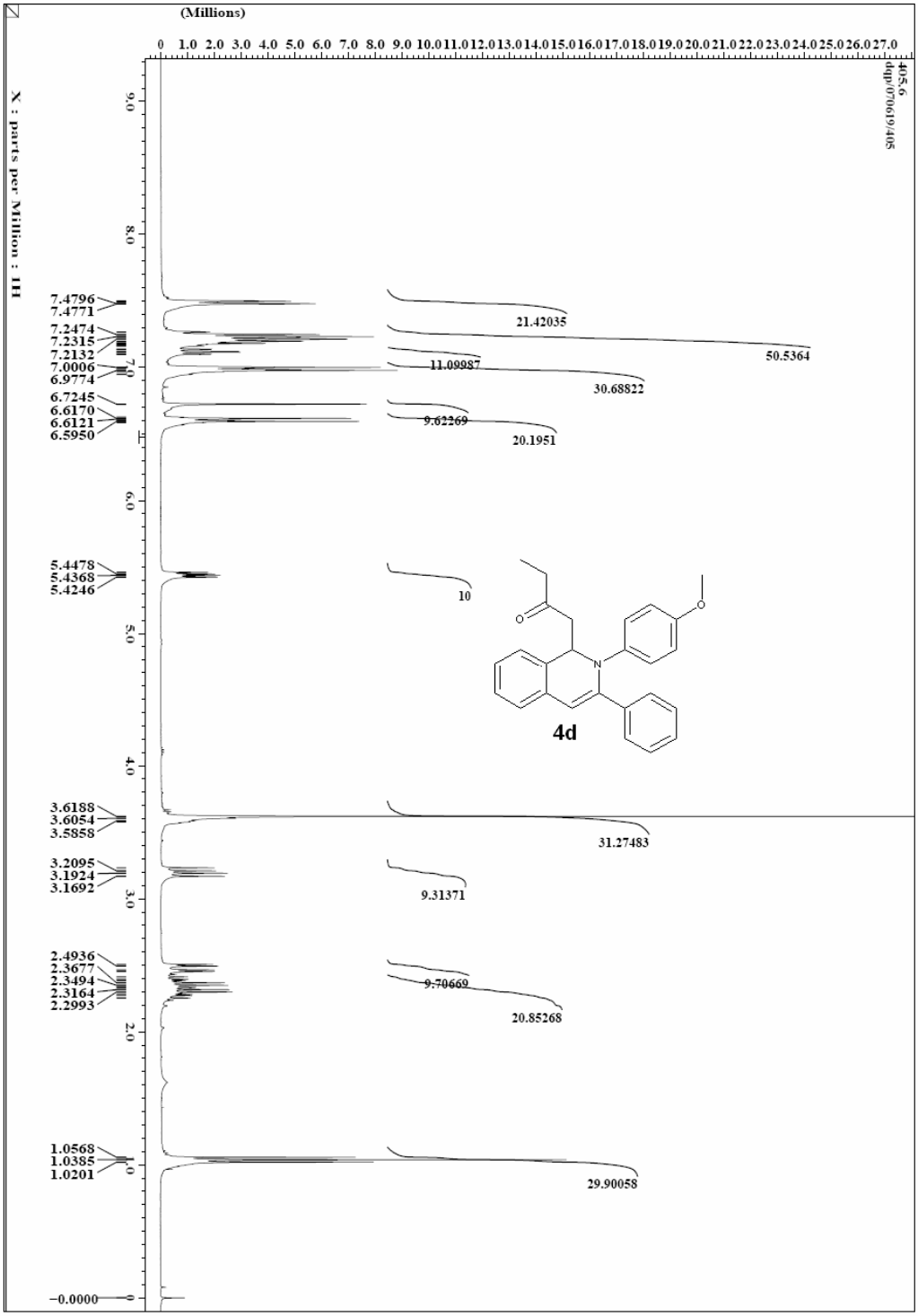

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
428-2 C.6
dqp/07071/428-2 C
X : parts per Million : 13C
211.9999
147.6701
140.7670
131.4422
128.5342
128.3816
127.9049
127.4282
127.3233
127.0086
126.3603
123.9671
122.8230
122.0697
112.5828
77.3144
76.9998
76.6851
62.9553
55.0321
43.3904
32.4256
28.9550
25.3319
O
N
4b'

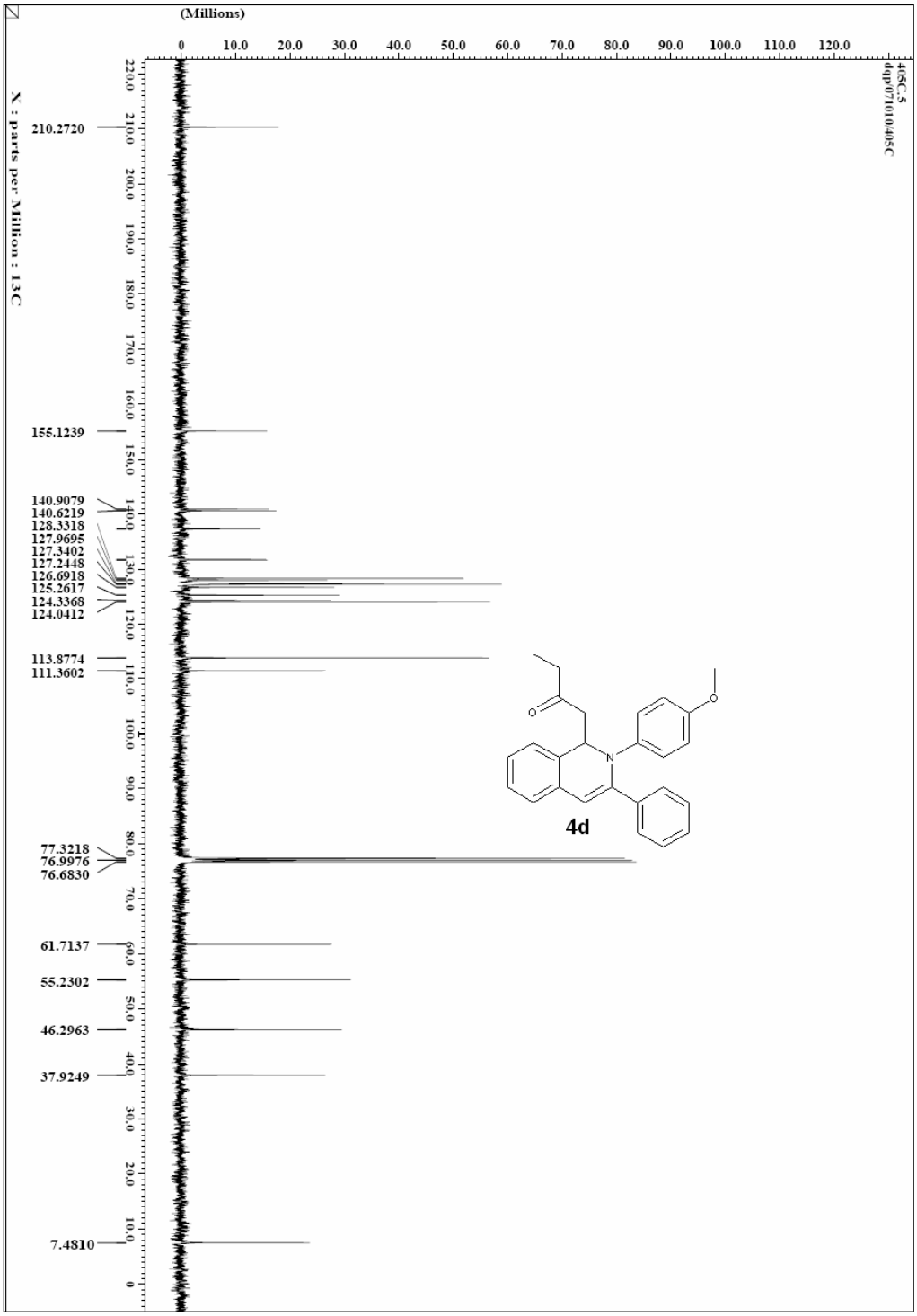

(Millions)
X : parts per Million : 1H
427-1 H 6
dqp070630427-1 H
7.4910
7.4727
7.2589
7.2455
7.2259
6.9205
6.7739
6.7494
6.7287
5.2613
5.2381
3.0646
3.0536
3.0426
2.5234
2.4928
2.3890
2.3743
2.0701
2.0628
2.0383
1.7109
1.6742
1.4323
1.3774
1.3700
1.2552
1.2259
1.2185
0.0004
20.05939
75.7156
10.83789
10.02294
29.98067
10
10.26181
10.5104
10.72082
11.9547
21.88527
22.4879
20.81695
F
O
N
4c

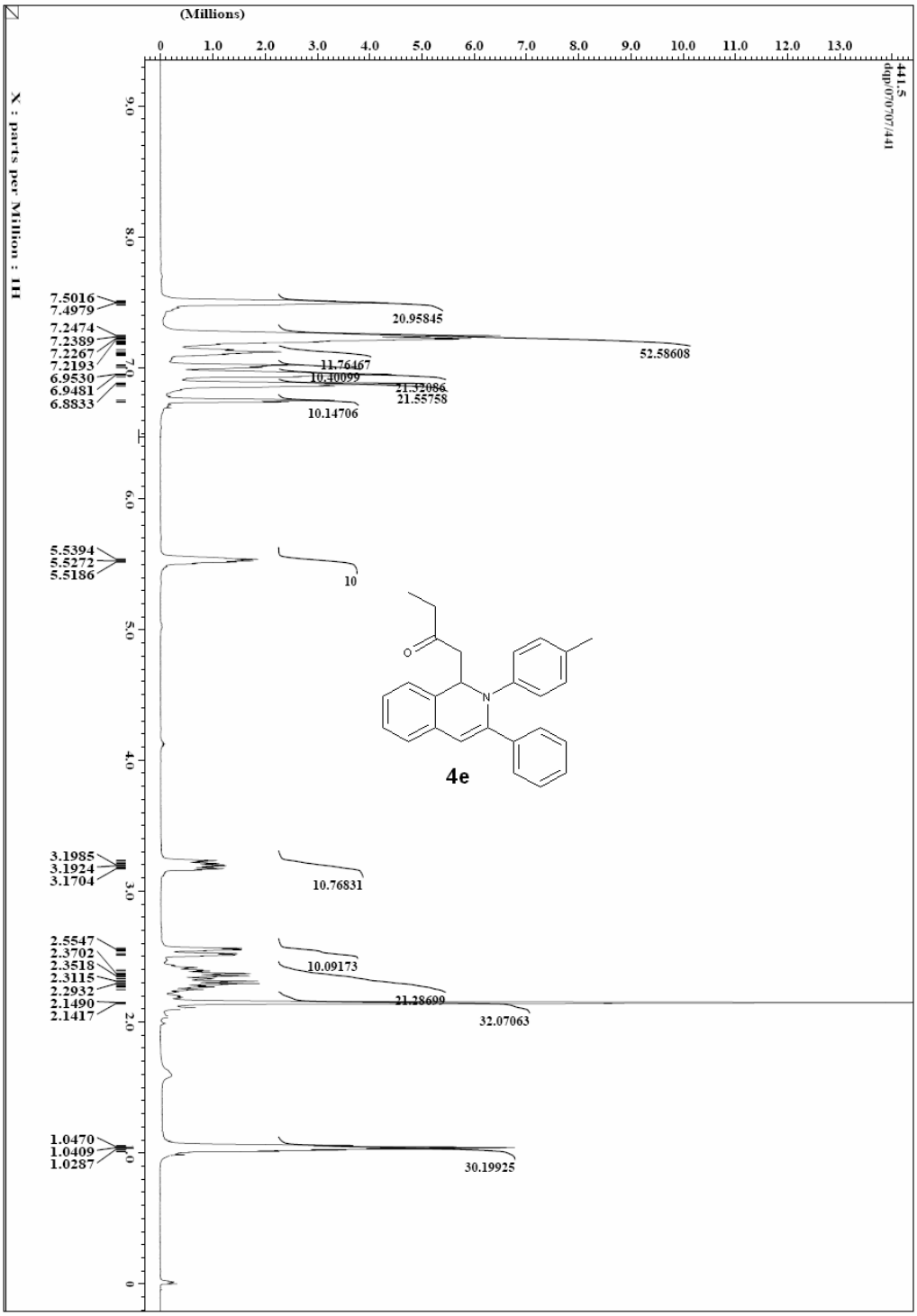

(Millions)
X : parts per Million : 13C
213.1339
159.5686
157.1659
141.6531
137.3912
132.0232
128.4859
128.1426
127.4180
127.2273
126.9794
126.3215
124.3383
124.2621
115.1565
114.9372
112.9445
77.3233
76.9991
76.6845
63.9844
51.7325
43.2658
32.3582
29.0783
25.2168
427-1 C.6
dqp/07/07/427-1 C
O
N
F
4c

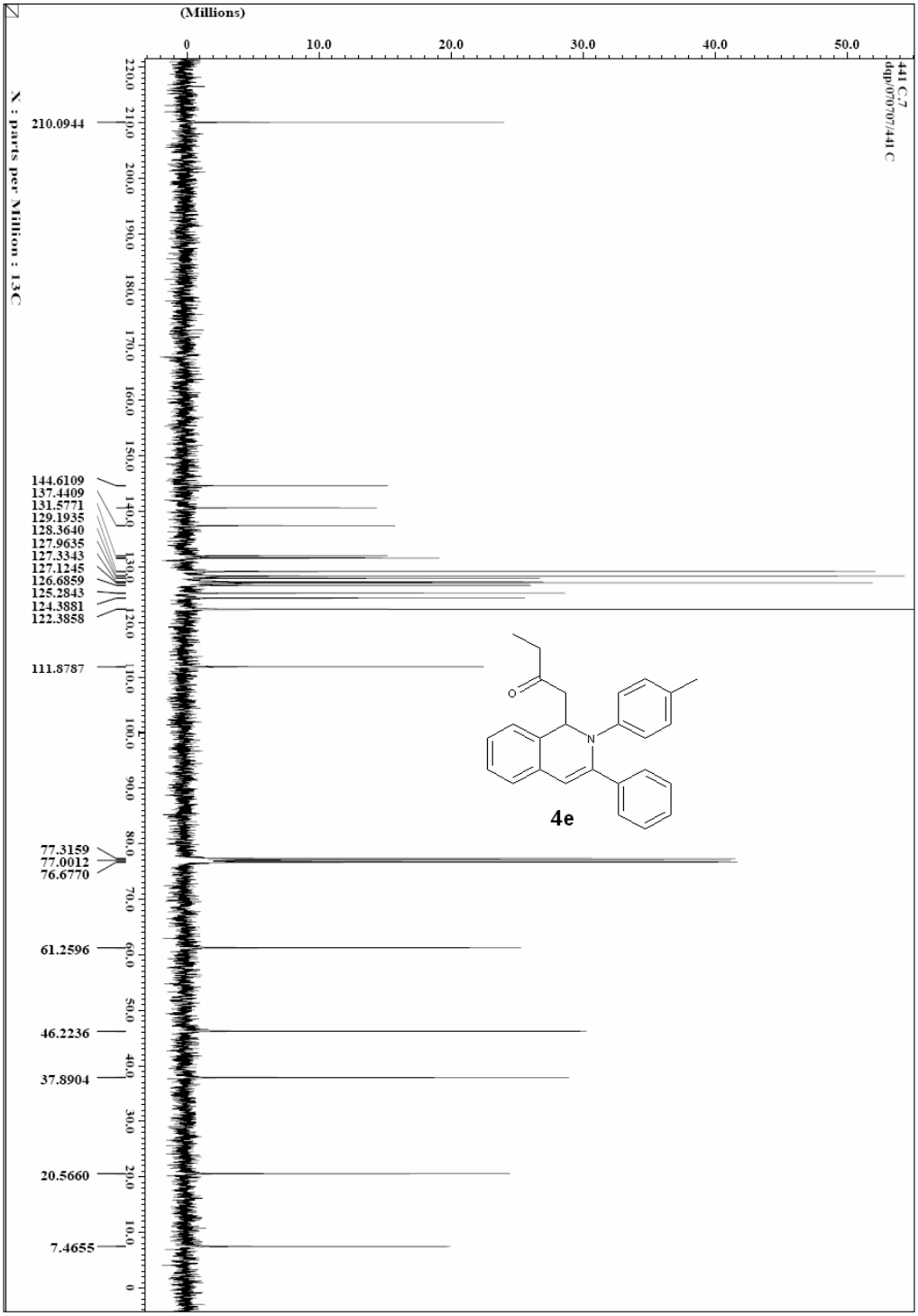

(Millions)
X : parts per Million : 1H
427-2 H-4
dqp/070714/427-2 H
7.5810
7.5639
7.5602
7.2658
7.2475
7.2340
7.2304
6.7991
6.7710
6.7502
5.3965
5.3733
2.8003
2.7905
2.6194
2.5828
2.3592
2.3506
2.2211
2.2065
1.9218
1.8925
1.8619
1.8534
1.7373
1.6103
19.15175
67.29347
10.88267
10.30739
19.15903
21.72701
10
10.33885
10.62892
12.72242
10.393
10.79138
22.36073
11.8565
13.78034
F
O
N
4c'

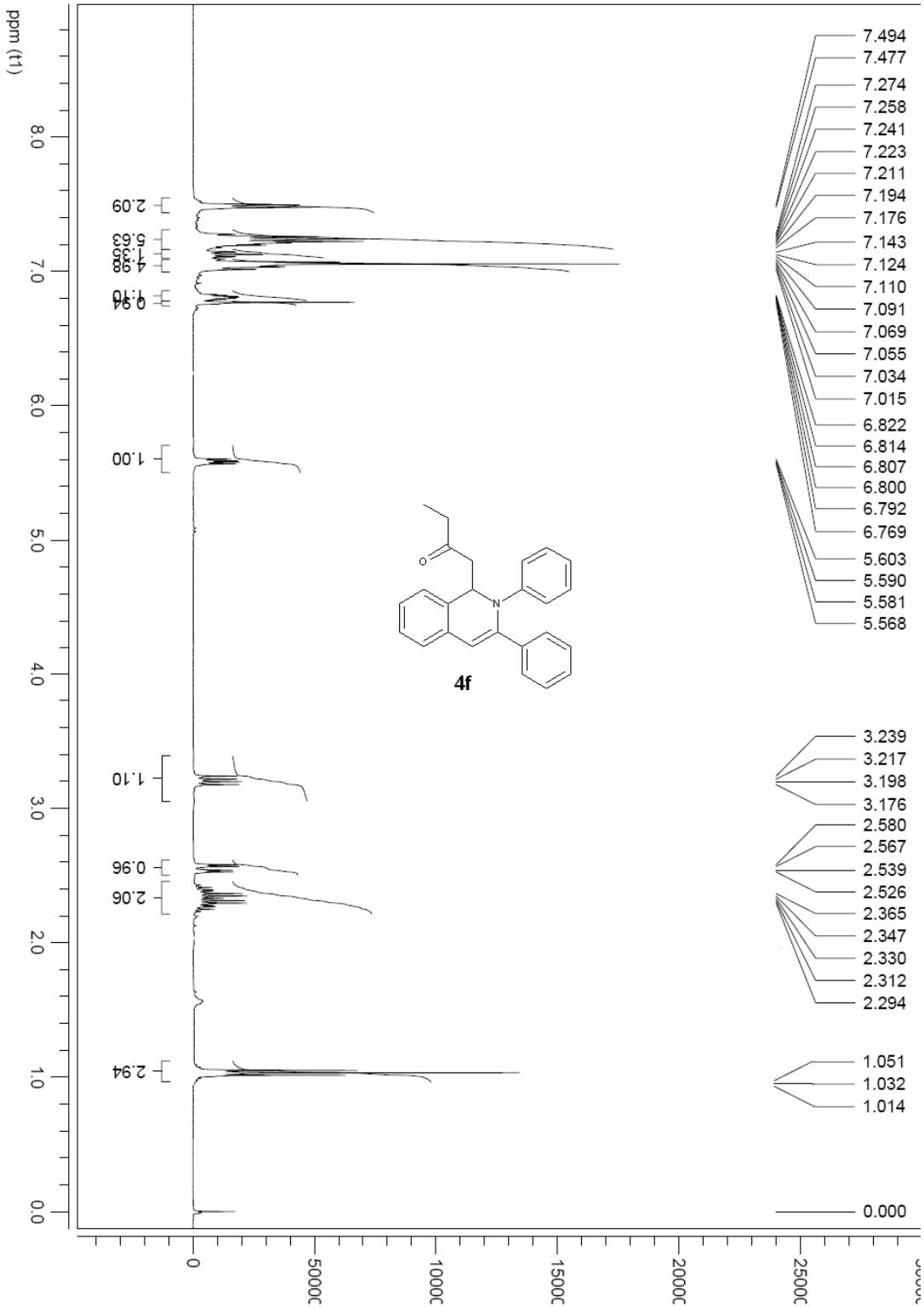

(Millions)
0
100.0
200.0
300.0
427-2 C.6
dup/070713/427-2 C
N : parts per Million : 13C
211.9043
159.5785
157.1663
141.0052
136.3428
128.4386
128.0477
127.4470
127.2944
127.1038
126.5221
124.3959
124.3101
124.0050
115.3667
115.1474
112.5349
77.3142
76.9995
76.6849
63.4128
55.3370
43.3329
32.2538
28.8404
25.3221
O
N
F
4c'

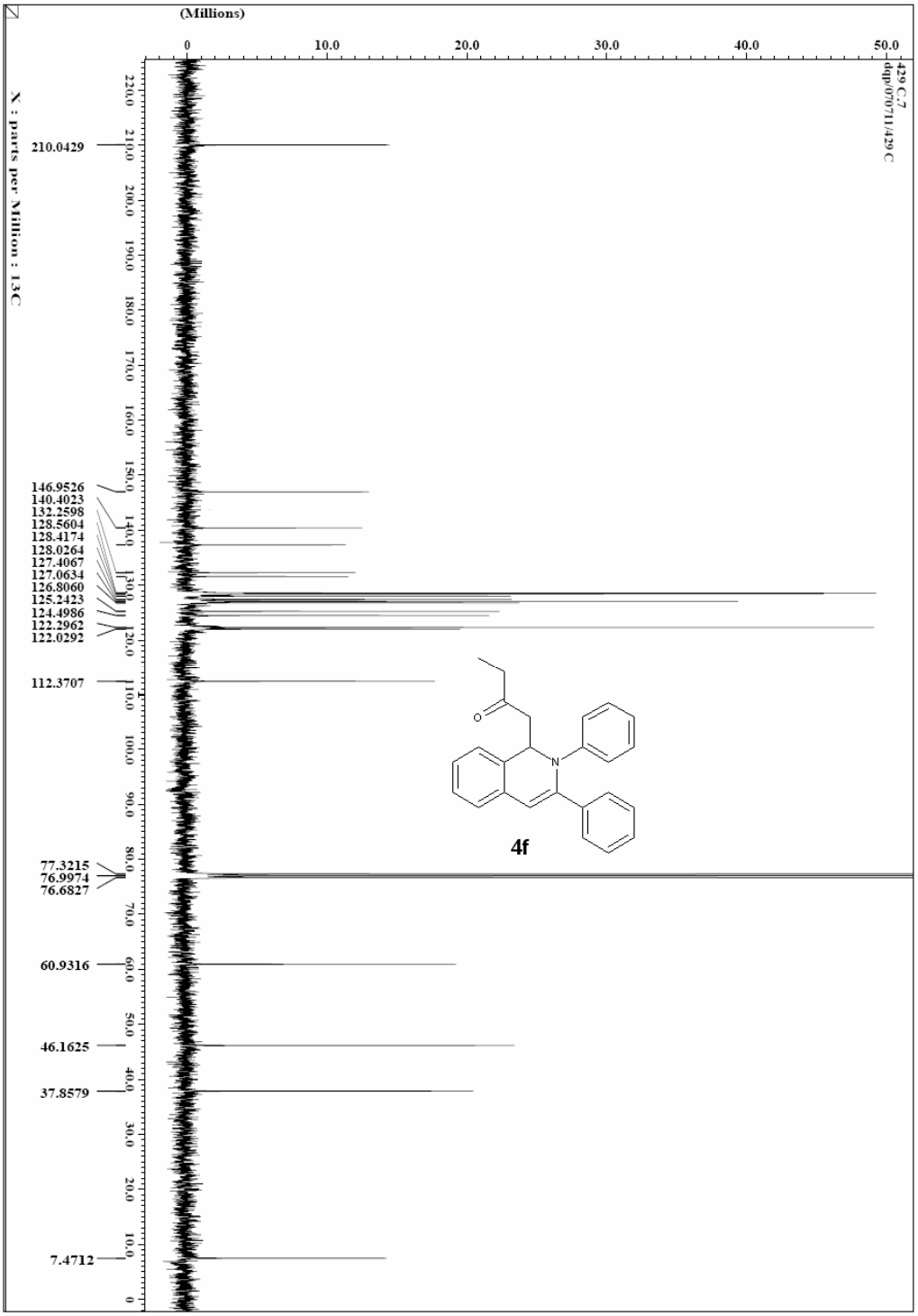

(Millions)
X : parts per Million : 1H
405.6
dqp/070619/405
7.4796
7.4771
7.2474
7.2315
7.2132
7.0006
6.9774
6.7245
6.6170
6.6121
6.5950
5.4478
5.4368
5.4246
3.6188
3.6054
3.5858
3.2095
3.1924
3.1692
2.4936
2.3677
2.3494
2.3164
2.2993
1.0568
1.0385
1.0201
−0.0000
21.42035
50.5364
11.09987
30.68822
9.62269
20.1951
10
31.27483
9.31371
9.70669
20.85268
29.90058
4d

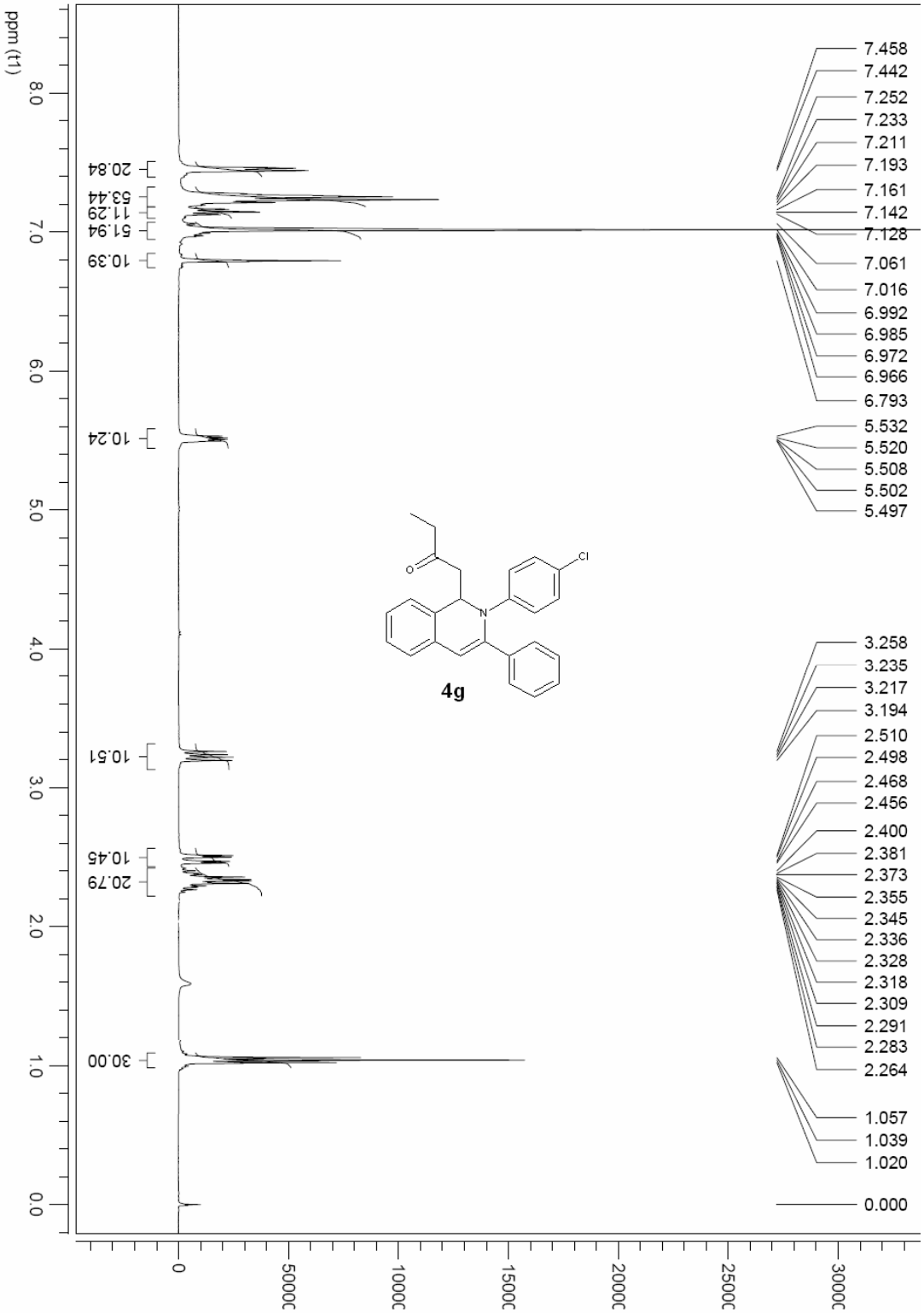

(Millions)
N : parts per Million : 13C
405C.5
dqp070101405C
210.2720
155.1239
140.9079
140.6219
128.3318
127.9695
127.3402
127.2448
126.6918
125.2617
124.3368
124.0412
113.8774
111.3602
77.3218
76.9976
76.6830
61.7137
55.2302
46.2963
37.9249
7.4810
4d

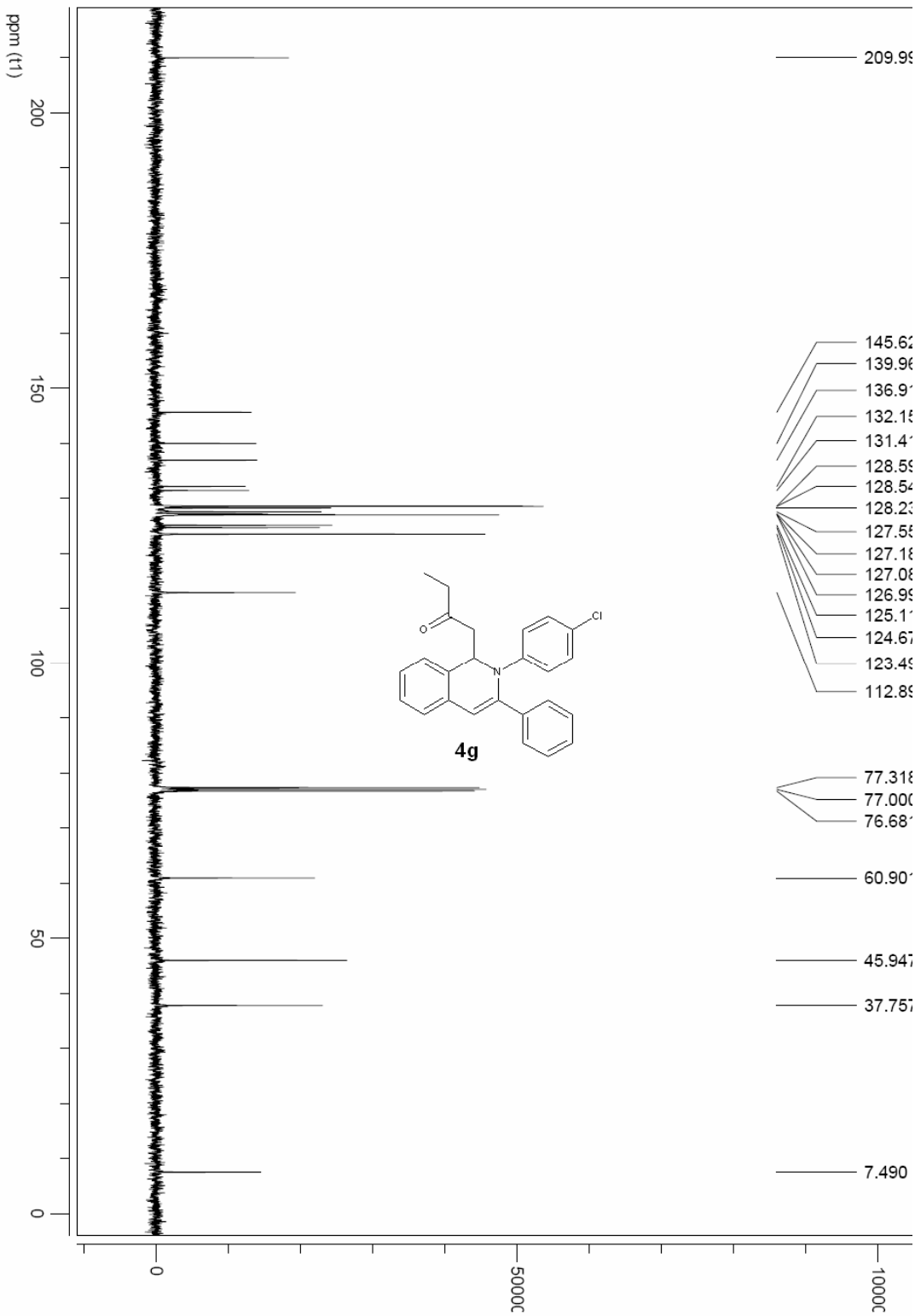

X : parts per Million : 1H
(Millions)
441.5
dap/070707/441
7.5016
7.4979
7.2474
7.2389
7.2267
7.2193
6.9530
6.9481
6.8833
5.5394
5.5272
5.5186
3.1985
3.1924
3.1704
2.5547
2.3702
2.3518
2.3115
2.2932
2.1490
2.1417
1.0470
1.0409
1.0287
20.95845
52.58608
11.76467
10.40099
21.32086
21.55758
10.14706
10
10.76831
10.09173
21.28699
32.07063
30.19925
4e

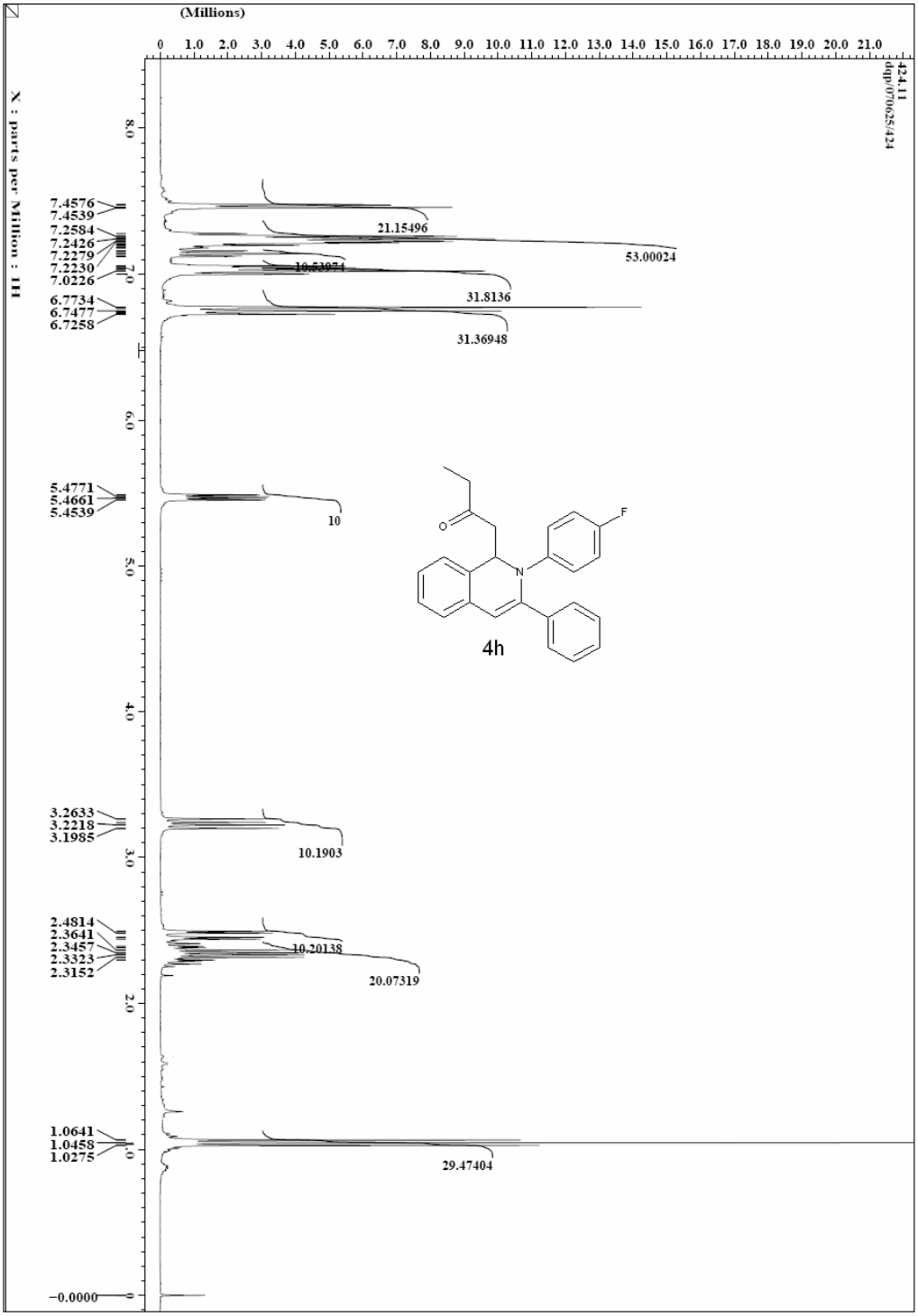

(Millions)
0
10.0
20.0
30.0
40.0
50.0
441 C.7
dup/070707/441 C
N : parts per Million : 13C
210.0944
144.6109
137.4409
131.5771
129.1935
128.3640
127.9635
127.3343
127.1245
126.6859
125.2843
124.3881
122.3858
111.8787
77.3159
77.0012
76.6770
61.2596
46.2236
37.8904
20.5660
7.4655
4e

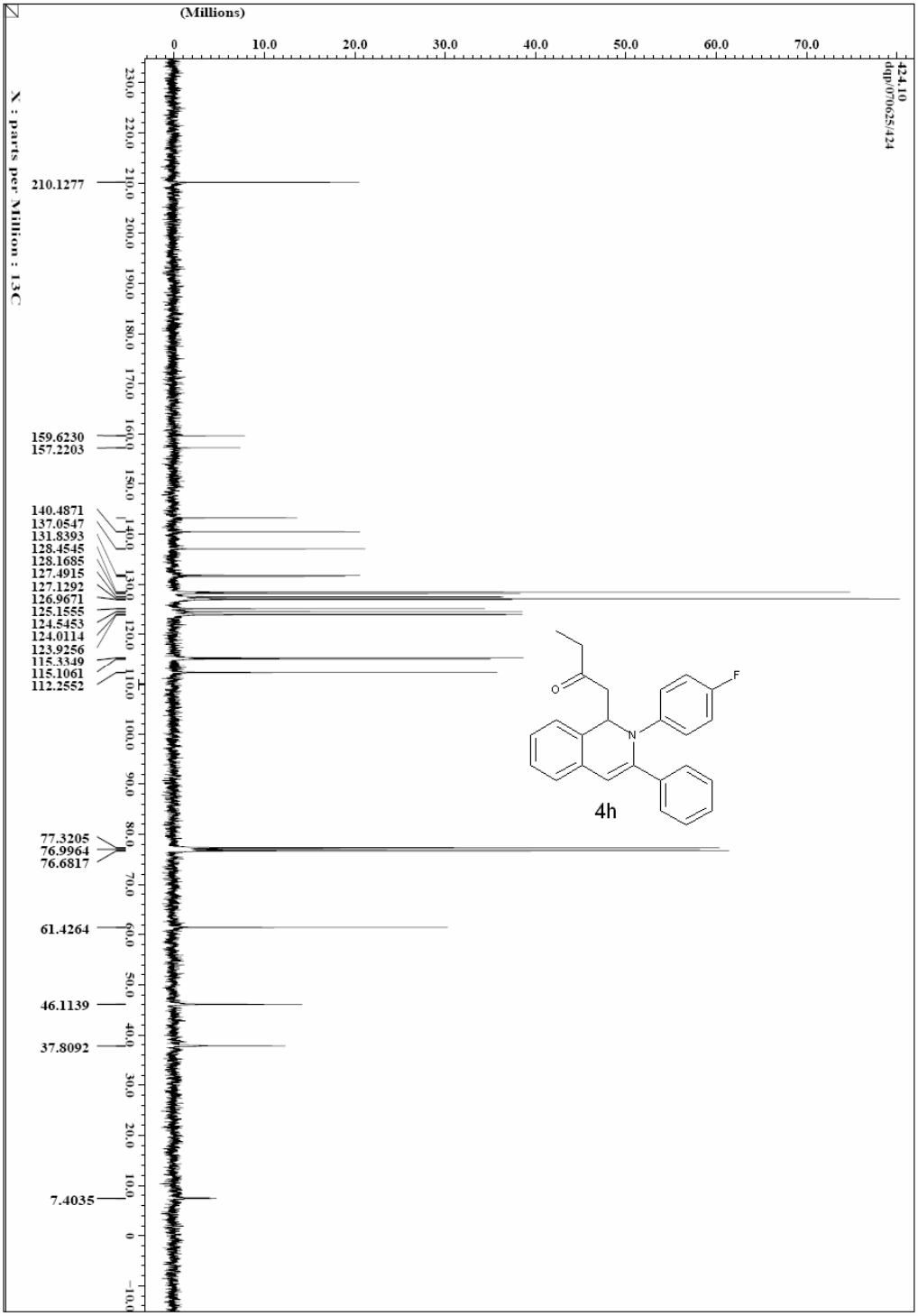

ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
2.09
5.63
1.35
4.98
0.94
1.10
1.00
1.10
0.96
2.06
2.94
7.494
7.477
7.274
7.258
7.241
7.223
7.211
7.194
7.176
7.143
7.124
7.110
7.091
7.069
7.055
7.034
7.015
6.822
6.814
6.807
6.800
6.792
6.769
5.603
5.590
5.581
5.568
3.239
3.217
3.198
3.176
2.580
2.567
2.539
2.526
2.365
2.347
2.330
2.312
2.294
1.051
1.032
1.014
0.000
O
N
4f
0
5000
10000
15000
20000
25000

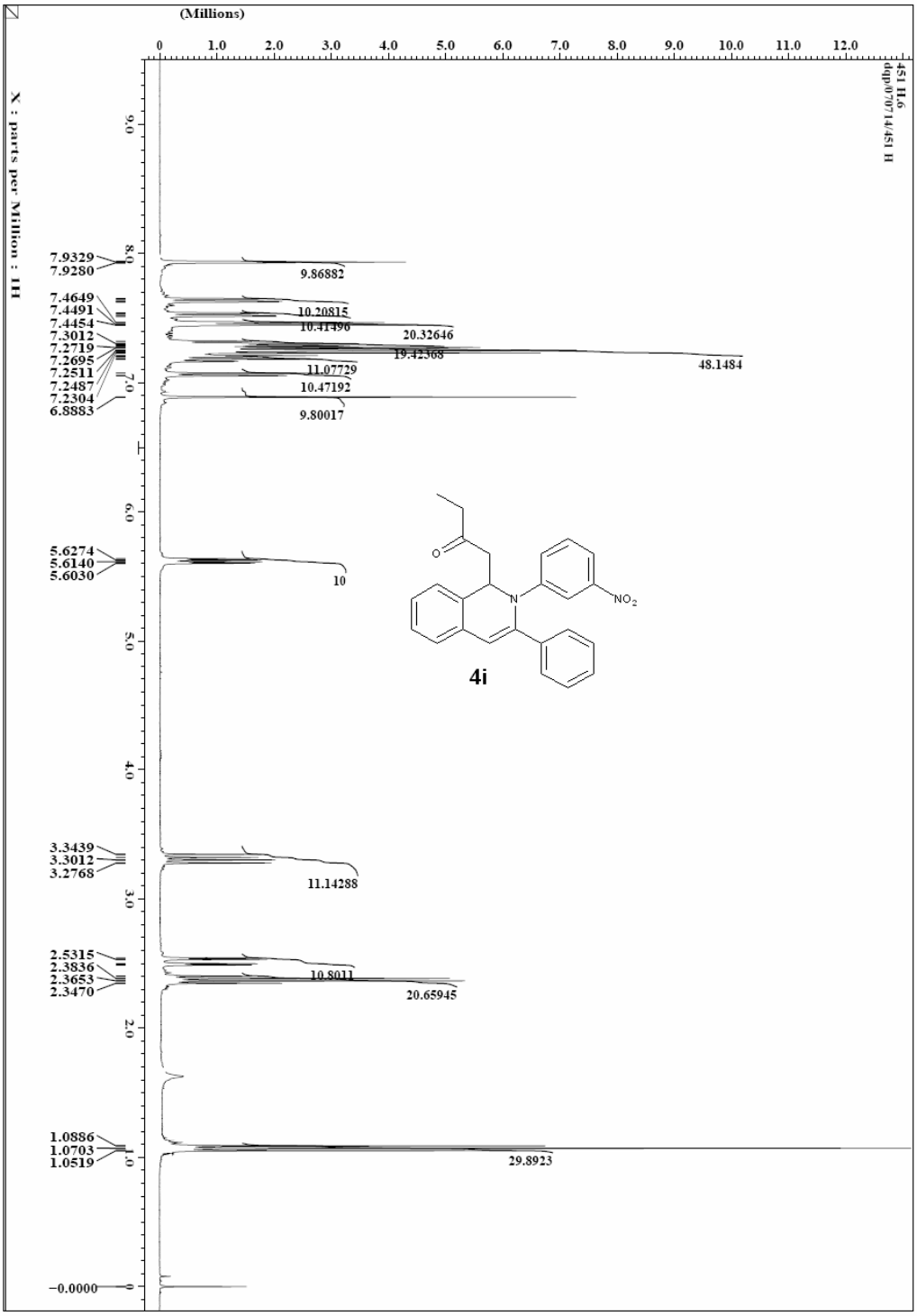

(Millions)
0
10.0
20.0
30.0
40.0
50.0
429 C.7
dqp/070711/429 C
X : parts per Million : 13C
210.0429
146.9526
140.4023
132.2598
128.5604
128.4174
128.0264
127.4067
127.0634
126.8060
125.2423
124.4986
122.2962
122.0292
112.3707
77.3215
76.9974
76.6827
60.9316
46.1625
37.8579
7.4712
O
N
4f

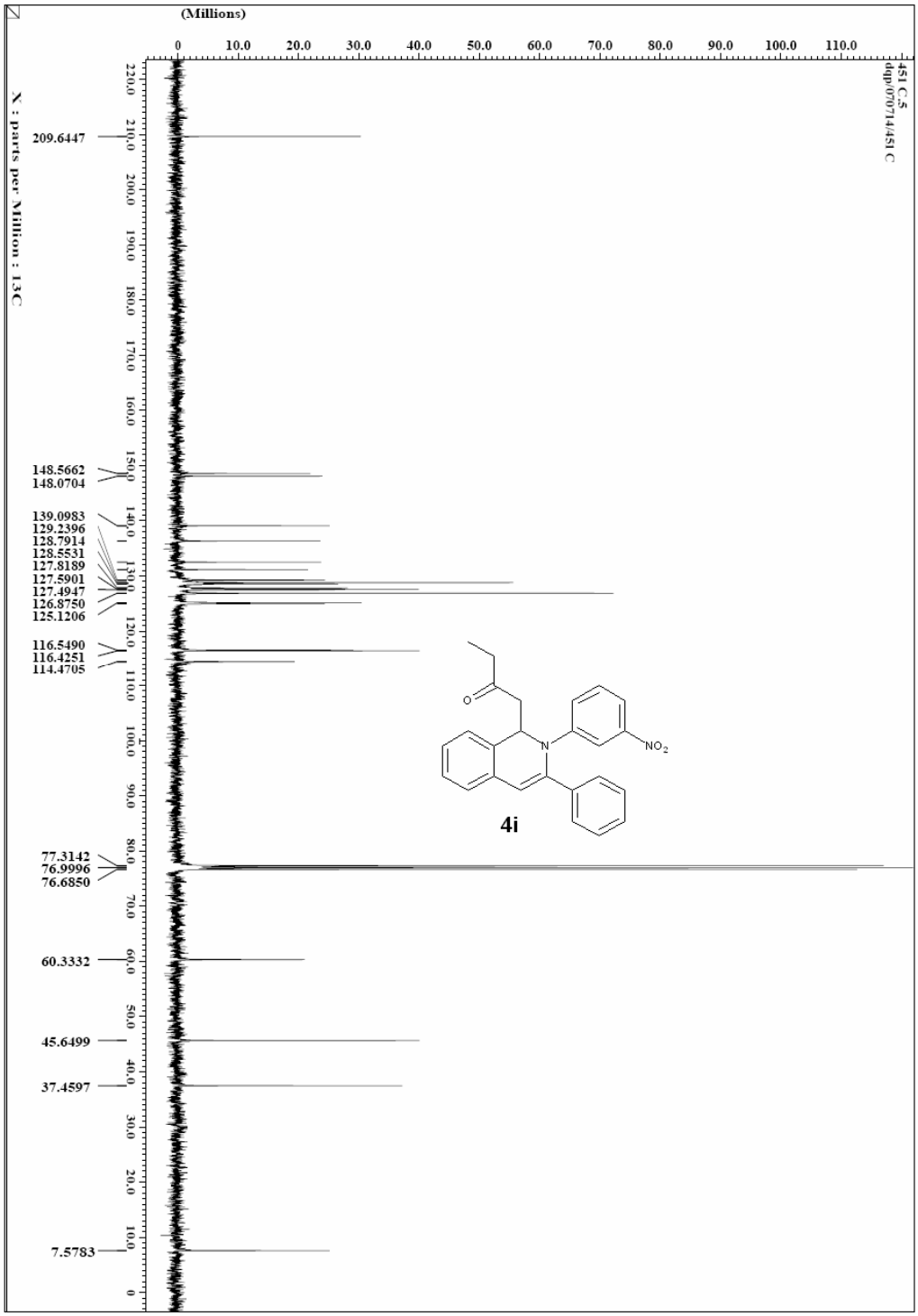

7.458
7.442
7.252
7.233
7.211
7.193
7.161
7.142
7.128
7.061
7.016
6.992
6.985
6.972
6.966
6.793
5.532
5.520
5.508
5.502
5.497
3.258
3.235
3.217
3.194
2.510
2.498
2.468
2.456
2.400
2.381
2.373
2.355
2.345
2.336
2.328
2.318
2.309
2.291
2.283
2.264
1.057
1.039
1.020
0.000
20.84
53.44
11.29
51.94
10.39
10.24
10.51
10.45
20.79
30.00
Cl
N
O
4g
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
5000
10000
15000
20000
25000
30000

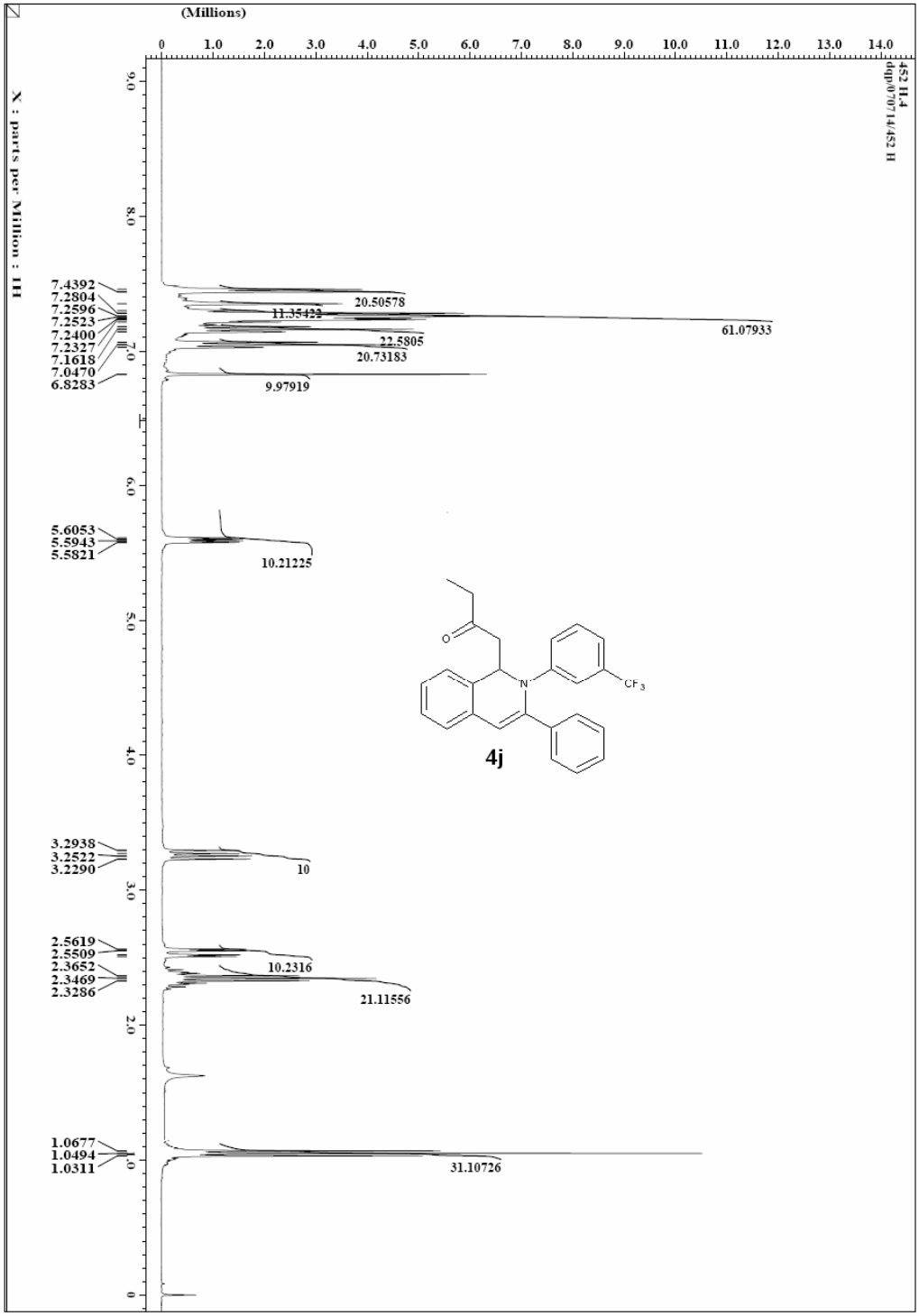

ppm (t1)
200
150
100
50
0
209.99
145.62
139.96
136.91
132.15
131.41
128.59
128.54
128.23
127.55
127.18
127.08
126.99
125.11
124.67
123.49
112.89
77.318
77.000
76.681
60.901
45.947
37.757
7.490
4g
Cl
N
O
0
50000
100000

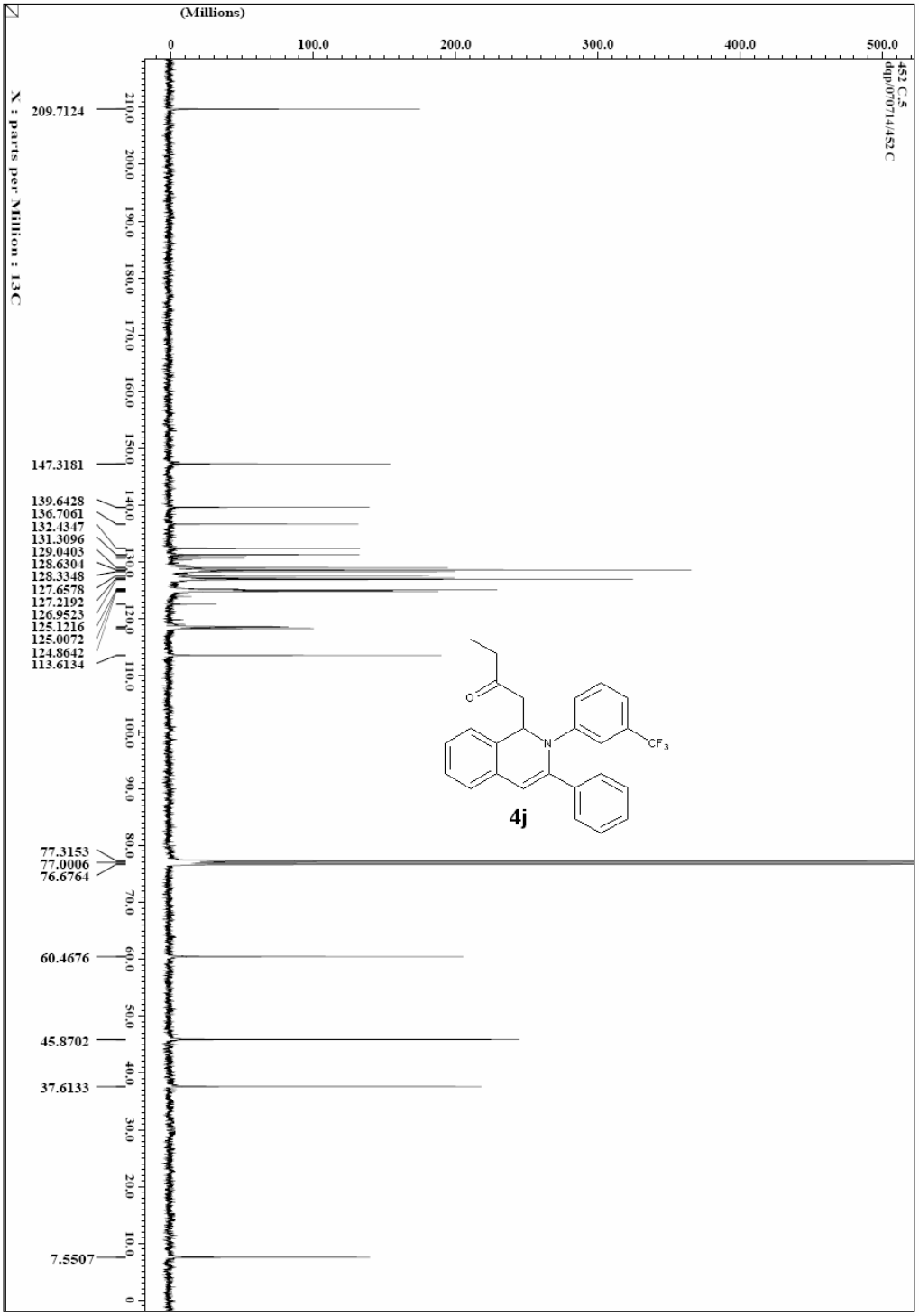

(Millions)
X : parts per Million : 1H
424.11
ddp/070625/424
7.4576
7.4539
7.2584
7.2426
7.2279
7.2230
7.0226
6.7734
6.7477
6.7258
5.4771
5.4661
5.4539
3.2633
3.2218
3.1985
2.4814
2.3641
2.3457
2.3323
2.3152
1.0641
1.0458
1.0275
−0.0000
21.15496
53.00024
10.53974
31.8136
31.36948
10
10.1903
10.20138
20.07319
29.47404
O
N
F
4h

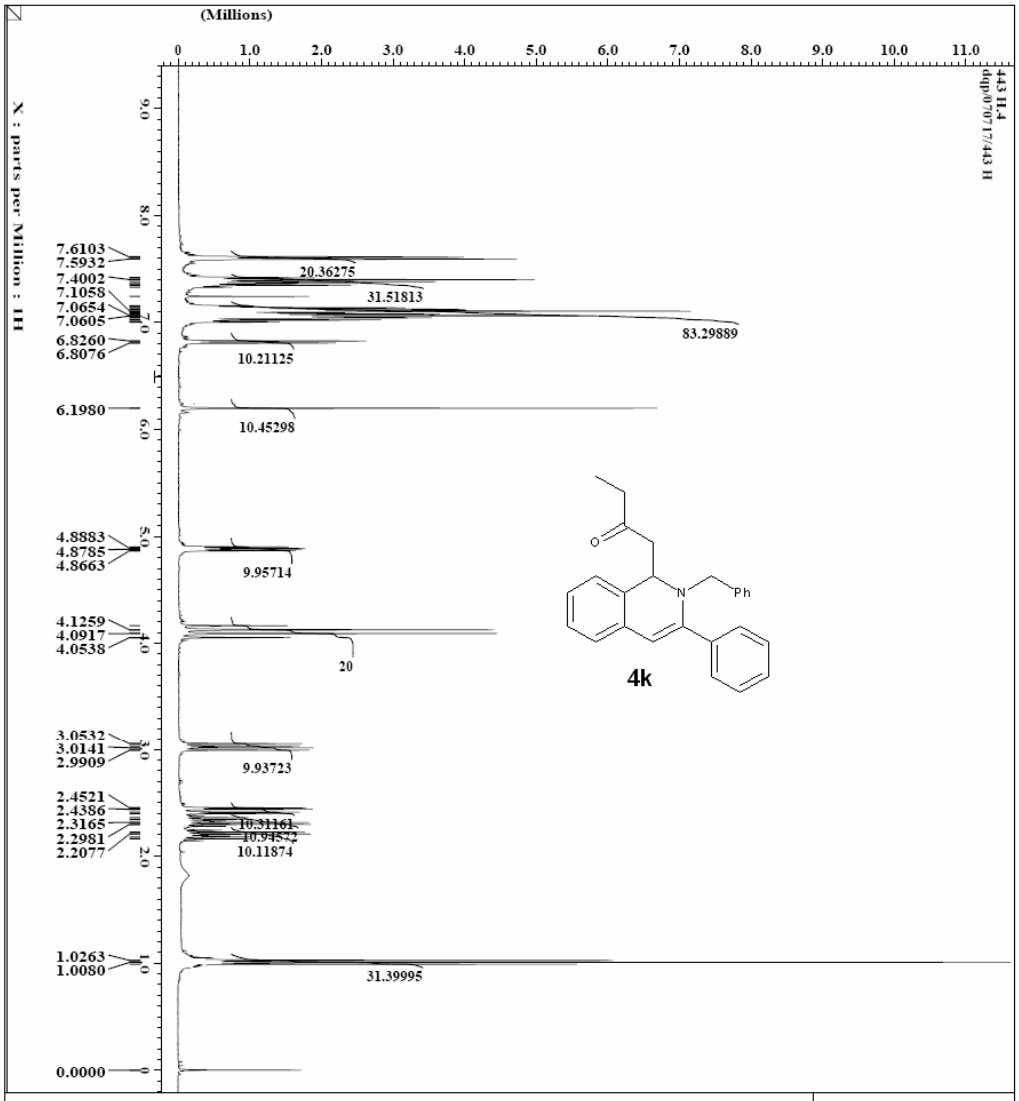

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
424.10
dap/070625/424
X : parts per Million : 13C
210.1277
159.6230
157.2203
140.4871
137.0547
131.8393
128.4545
128.1685
127.4915
127.1292
126.9671
125.1555
124.5453
124.0114
123.9256
115.3349
115.1061
112.2552
77.3205
76.9964
76.6817
61.4264
46.1139
37.8092
7.4035
O
N
F
4h


(Millions)
X : parts per Million : 1H
7.9329
7.9280
7.4649
7.4491
7.4454
7.3012
7.2719
7.2695
7.2511
7.2487
7.2304
6.8883
5.6274
5.6140
5.6030
3.3439
3.3012
3.2768
2.5315
2.3836
2.3653
2.3470
1.0886
1.0703
1.0519
−0.0000
9.86882
10.20815
10.41496
20.32646
19.42368
48.1484
11.07729
10.47192
9.80017
10
11.14288
10.8011
20.65945
29.8923
4i
N
O
NO2

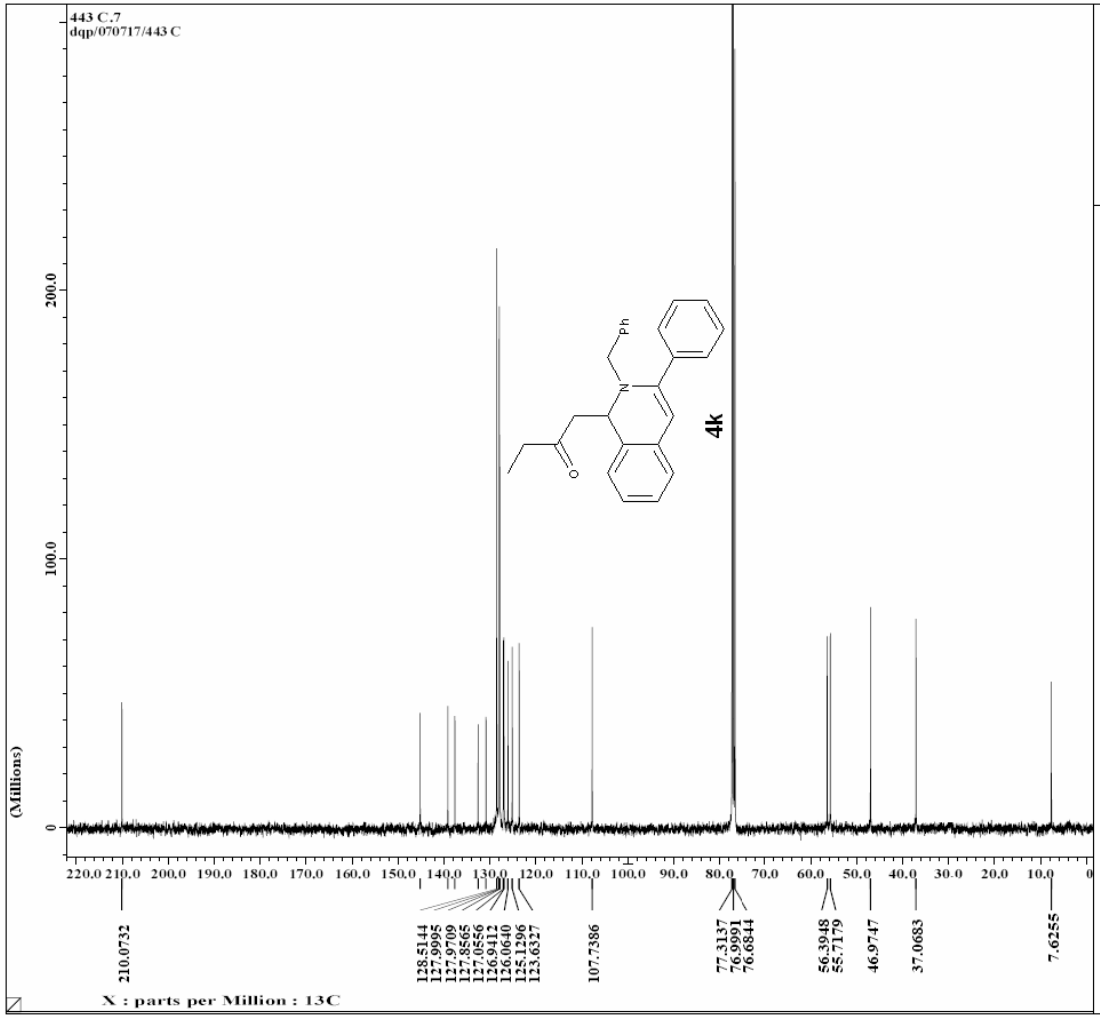

(Millions)
X : parts per Million : 13C
451 C.5
dup/070714/451 C
209.6447
148.5662
148.0704
139.0983
129.2396
128.7914
128.5531
127.8189
127.5901
127.4947
126.8750
125.1206
116.5490
116.4251
114.4705
77.3142
76.9996
76.6850
60.3332
45.6499
37.4597
7.5783
4i


(Millions)
X : parts per Million : 1H
452 H-4
ddp/070714/452 H
7.4392
7.2804
7.2596
7.2523
7.2400
7.2327
7.1618
7.0470
6.8283
20.50578
11.35422
61.07933
22.5805
20.73183
9.97919
5.6053
5.5943
5.5821
10.21225
3.2938
3.2522
3.2290
10
2.5619
2.5509
2.3652
2.3469
2.3286
10.2316
21.11556
1.0677
1.0494
1.0311
31.10726
O
N
CF3
4j


(Millions)
0
100.0
200.0
300.0
400.0
500.0
452 C,5
ddp/07/14/452 C
X : parts per Million : 13C
209.7124
147.3181
139.6428
136.7061
132.4347
131.3096
129.0403
128.6304
128.3348
127.6578
127.2192
126.9523
125.1216
125.0072
124.8642
113.6134
77.3153
77.0006
76.6764
60.4676
45.8702
37.6133
7.5507
O
N
CF3
4j

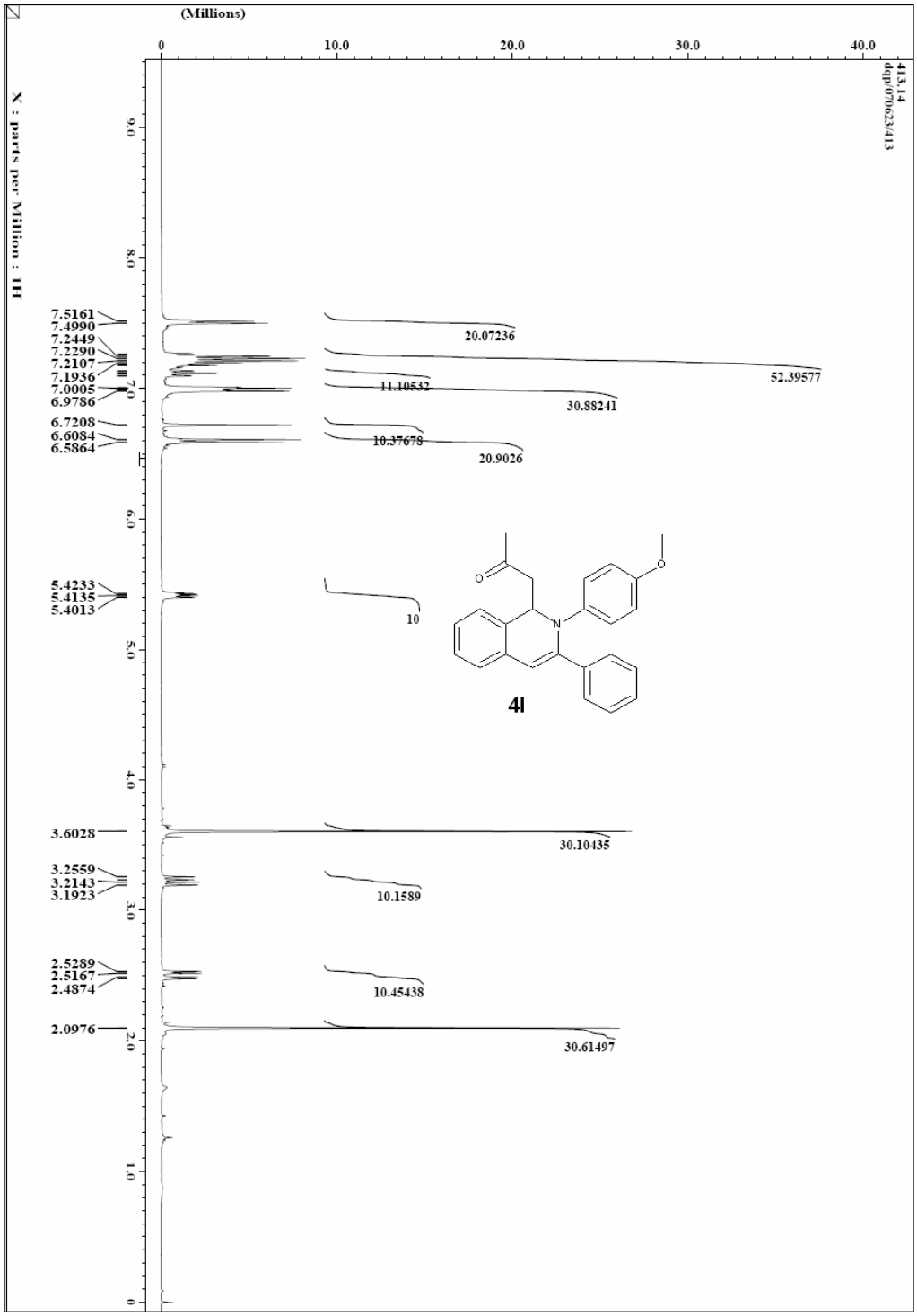


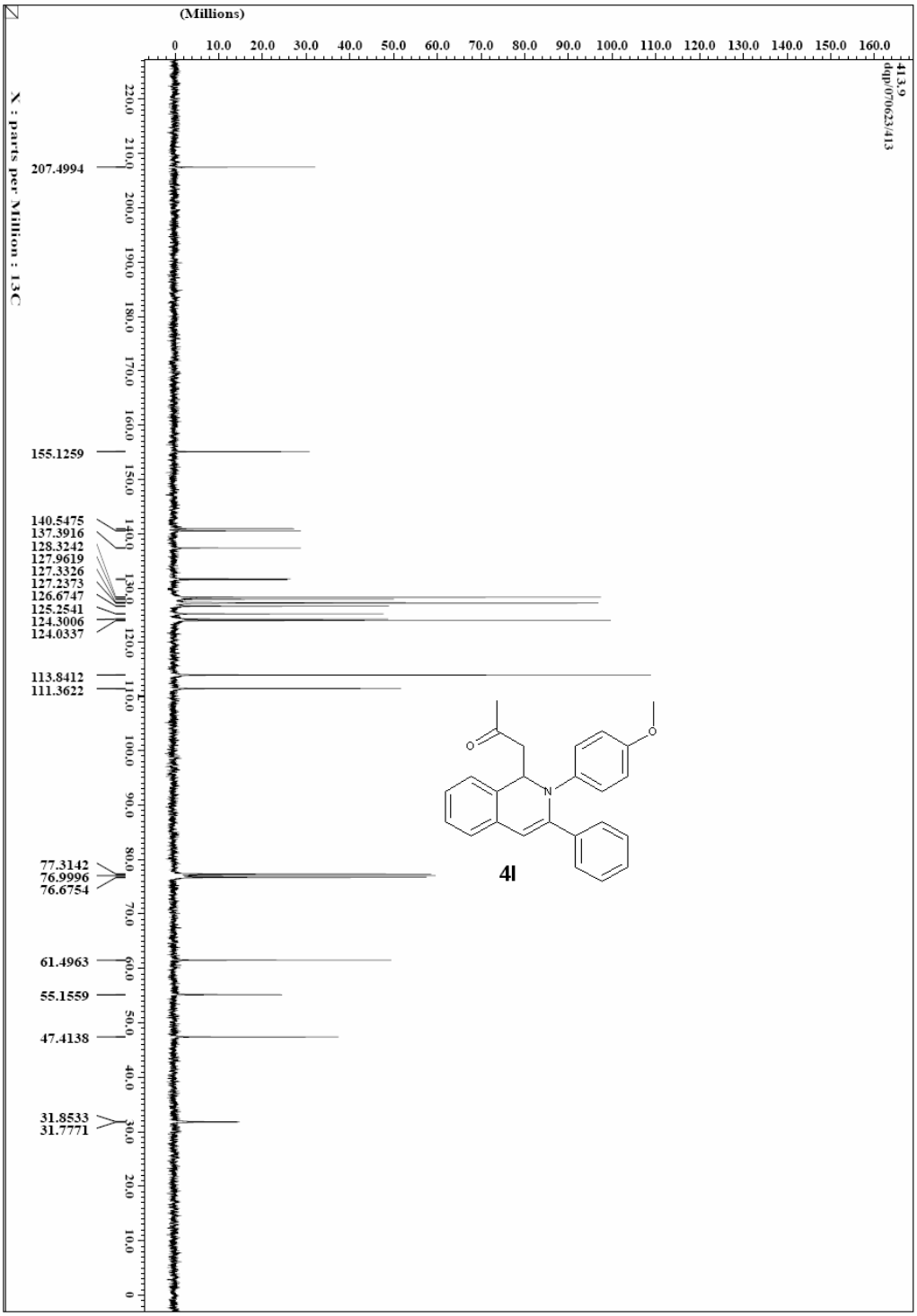

```
---- PROCESSING PARAMETERS ----
dc_balance
exp : 0.2[Hz] : 0.0[s]
trapezoid3 1 0[%] : 80[%] : 100[%]
zerofill : 1
ft_cfill : TRUE
machinephase
ppm

---- ACQUISITION PARAMETERS ----
Derivative from: 443 H.1
File Name = 443 H.4
Author = delta
Sample_ID = dqp/070717/443 H
Content = dqp/070717/443 H
Creation_Date = 17-JUL-2007 10:31:35
Revision_Date = 17-JUL-2007 11:00:08
Spec_Site = ECA400

Spec_Type = DELTA2_NMR
Data_Format = 1D COMPLEX
Dim_number = 1
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_atten = 4.5[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = 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 : 77.137[ppm] : 77[ppm]
```

```
---- ACQUISITION PARAMETERS ----
Derived from: 443 C.1
File Name          = 443 C.7
Author             = delta
Sample ID          = dqp/070717/443 C
Content            = dqp/070717/443 C
Creation Date      = 17-JUL-2007 10:54:04

Revision Date      = 17-JUL-2007 10:58:21
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         = -44.5[ppm]
Mod_return         = 1
Scans              = 417
Total_scans        = 417

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
40.0
413.14
dqp/070623/413
X : parts per Million : 1H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.5161
7.4990
7.2449
7.2290
7.2107
7.1936
7.0005
6.9786
6.7208
6.6084
6.5864
5.4233
5.4135
5.4013
3.6028
3.2559
3.2143
3.1923
2.5289
2.5167
2.4874
2.0976
20.07236
52.39577
11.10532
30.88241
10.37678
20.9026
10
30.10435
10.1589
10.45438
30.61497
O
O
N
4l


(Millions)
0 10.0 20.0 30.0 40.0 50.0 60.0 70.0 80.0 90.0 100.0 110.0 120.0 130.0 140.0 150.0 160.0
413.9
ddp/070623/413
N : parts per Million : 13C
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
207.4994
155.1259
140.5475
137.3916
128.3242
127.9619
127.3326
127.2373
126.6747
125.2541
124.3006
124.0337
113.8412
111.3622
77.3142
76.9996
76.6754
61.4963
55.1559
47.4138
31.8533
31.7771
O
N
O
4l

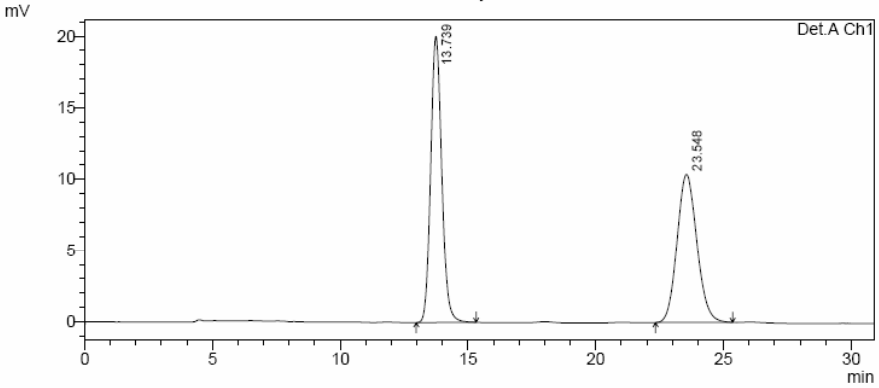
4l racemic

C:\LabSolutions\Data\Project1\4l-w5.lcd

Acquired by	: Admin
Sample Name	: dqp
Sample ID	:
Vail #	:
Injection Volume	: 10 uL
Data File Name	: 4l-w5.lcd
Method File Name	: 4l.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2007-10-13 16:00:48
Data Processed	: 2007-10-13 16:31:43

<Chromatogram>

C:\LabSolutions\Data\Project1\4l-w5.lcd



1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.739	602563	20030	51.735	65.838
2	23.548	562138	10393	48.265	34.162
Total		1164701	30424	100.000	100.000

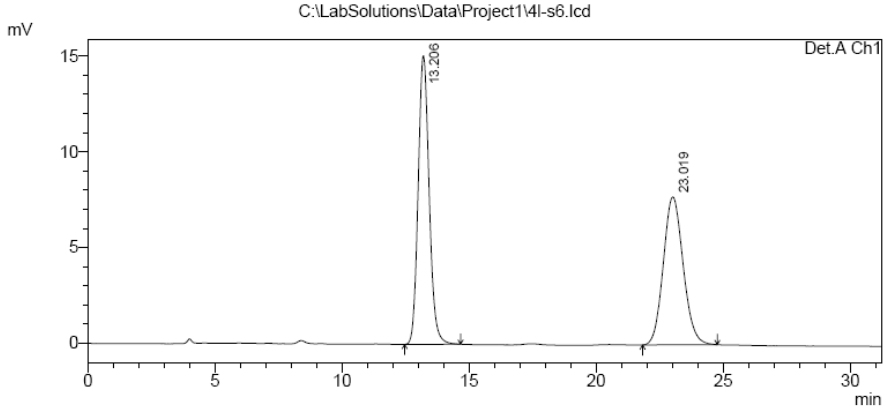
4l sample

C:\LabSolutions\Data\Project1\4l-s6.lcd

Acquired by	: Admin
Sample Name	: dqp
Sample ID	:
Vail #	:
Injection Volume	: 10 uL
Data File Name	: 4l-s6.lcd
Method File Name	: 4l.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2007-10-13 17:11:18
Data Processed	: 2007-10-13 17:42:35

<Chromatogram>

C:\LabSolutions\Data\Project1\4l-s6.lcd

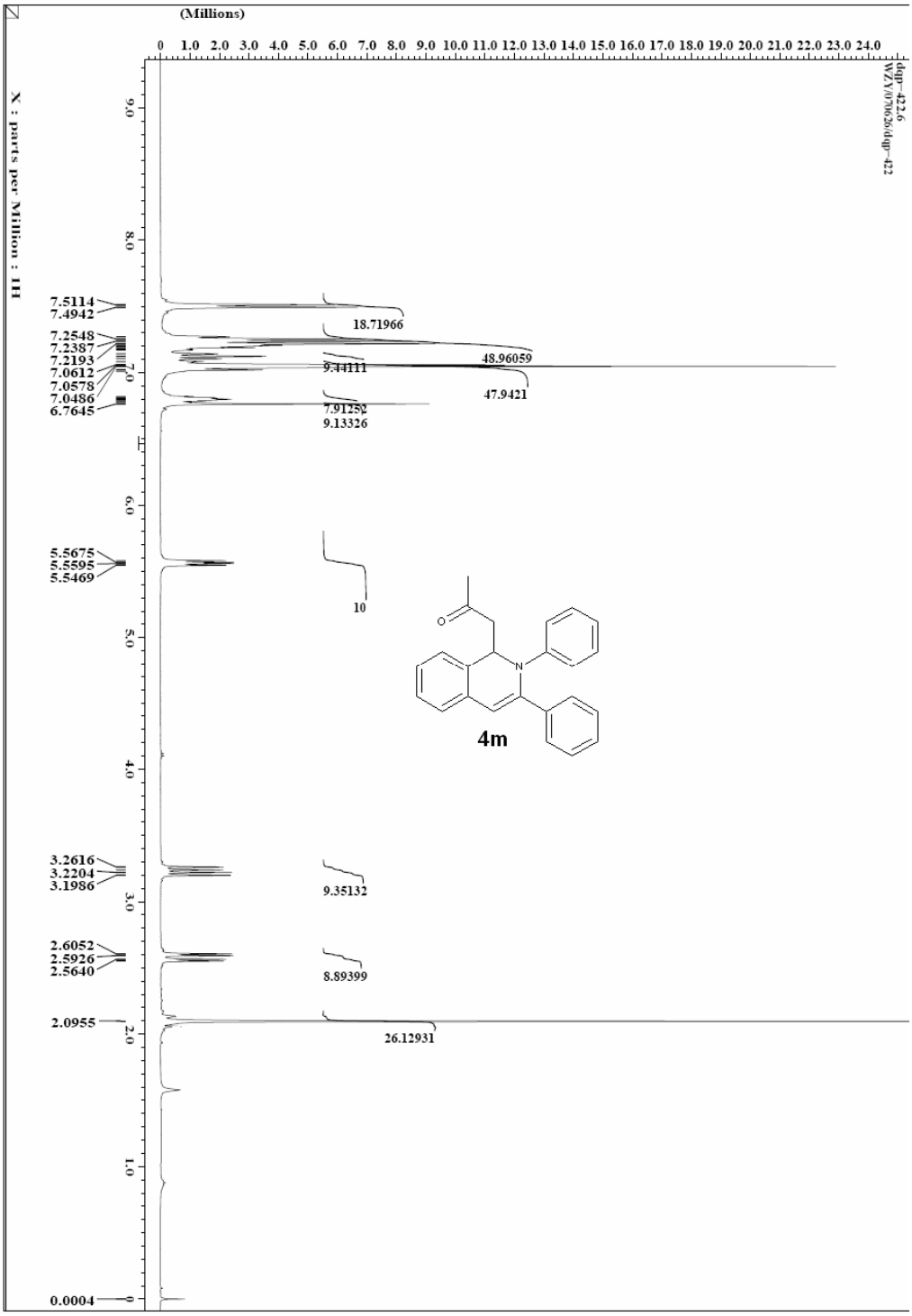


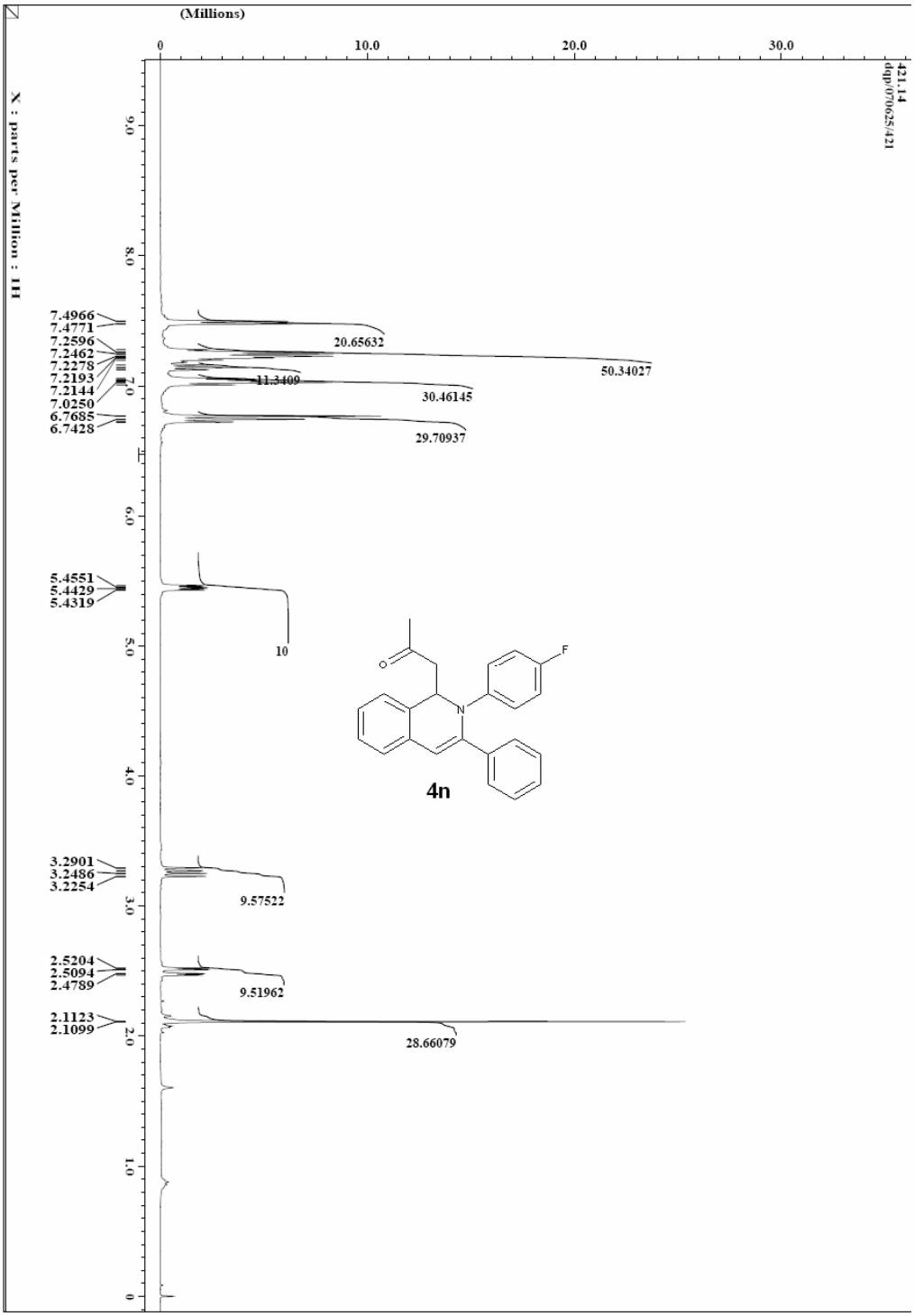
1 Det.A Ch1/254nm

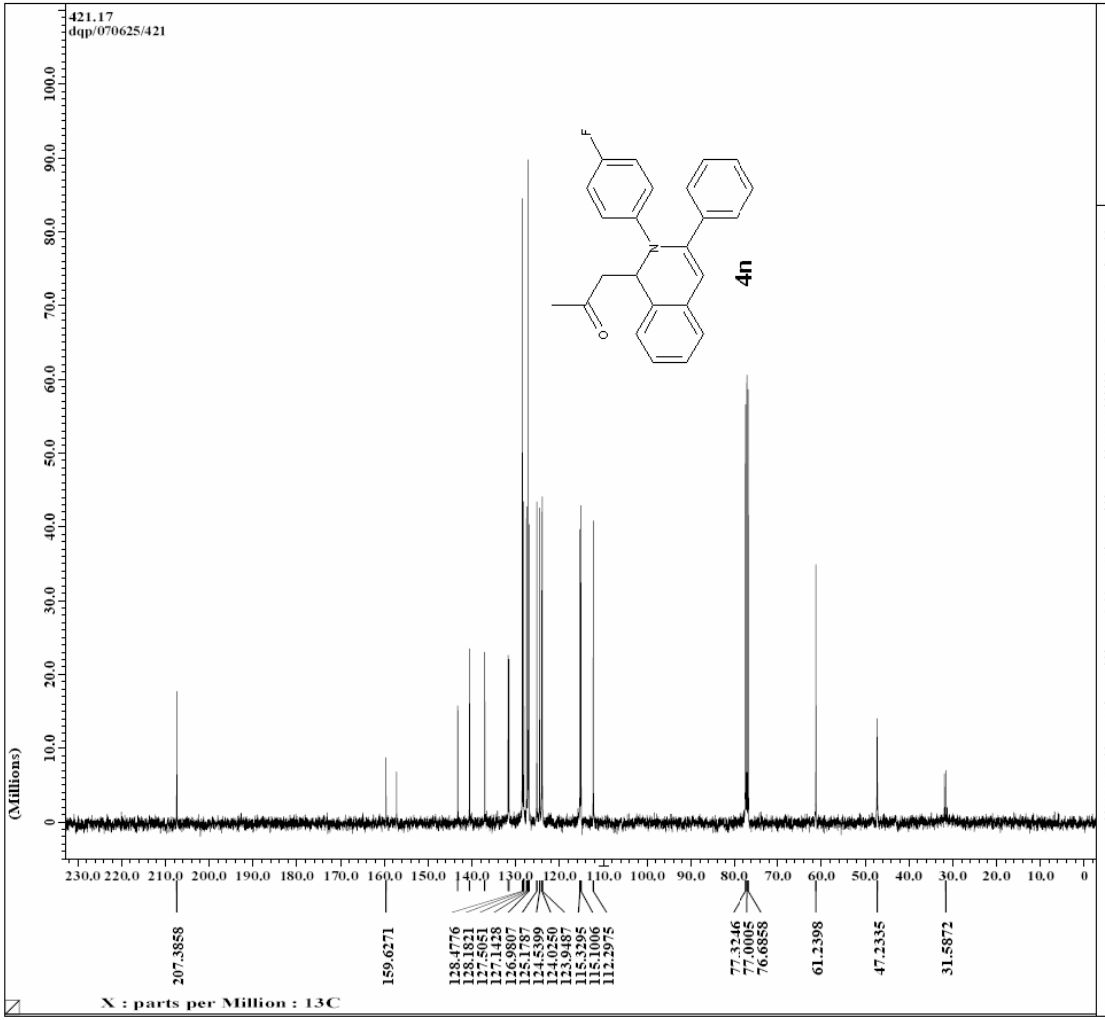
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.206	452678	15061	51.924	66.102
2	23.019	419125	7724	48.076	33.898
Total		871803	22785	100.000	100.000

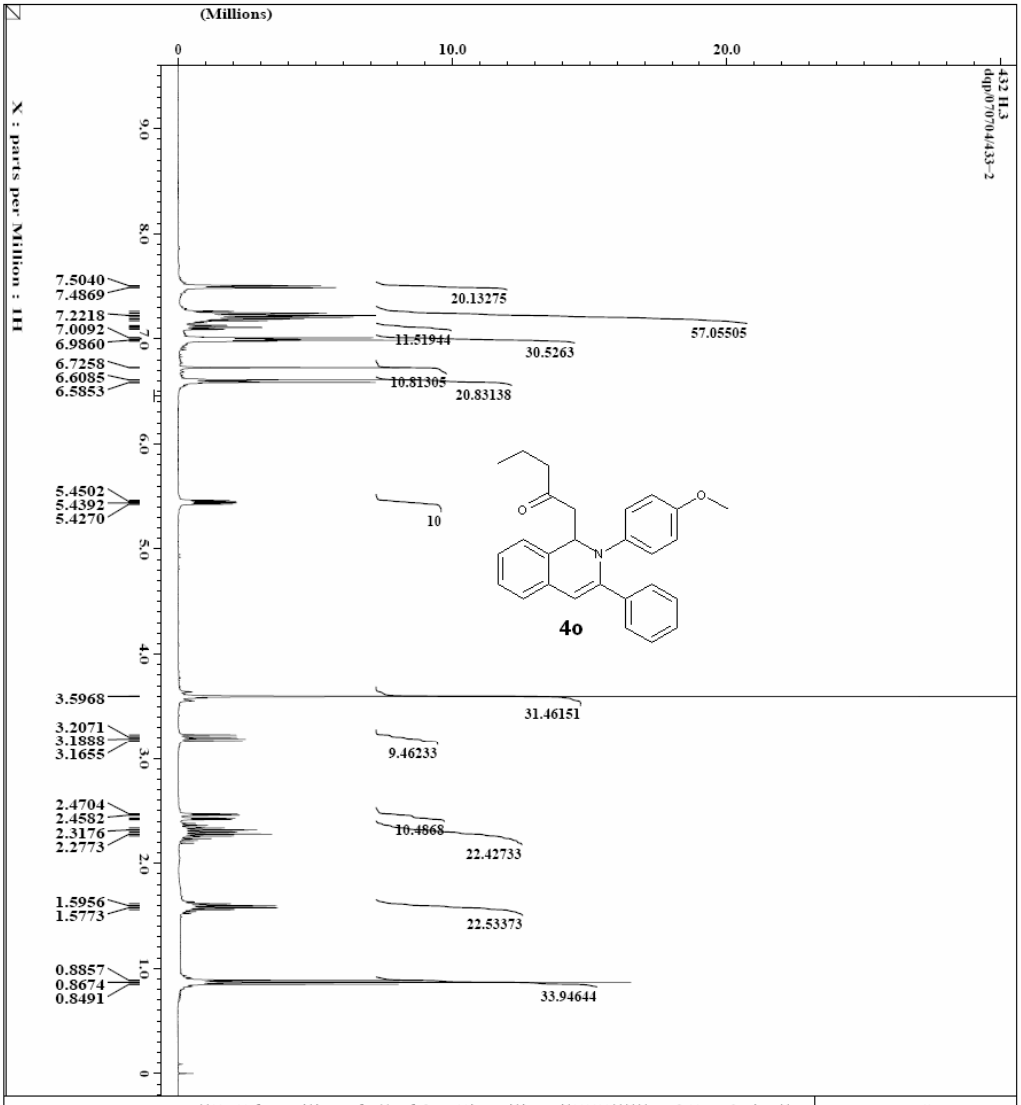

(Millions)
X : parts per Million : 1H
7.5114
7.4942
7.2548
7.2387
7.2193
7.0612
7.0578
7.0486
6.7645
5.5675
5.5595
5.5469
3.2616
3.2204
3.1986
2.6052
2.5926
2.5640
2.0955
0.0004
18.71966
48.96059
9.44111
47.9421
7.91252
9.13326
10
9.35132
8.89399
26.12931
4m
N
O


(Millions)
0
10.0
20.0
30.0
421.14
dup/070625/421
X : parts per Million : 1H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4966
7.4771
7.2596
7.2462
7.2278
7.2193
7.2144
7.0250
6.7685
6.7428
5.4551
5.4429
5.4319
3.2901
3.2486
3.2254
2.5204
2.5094
2.4789
2.1123
2.1099
20.65632
50.34027
11.3409
30.46145
29.70937
10
9.57522
9.51962
28.66079
F
N
O
4n

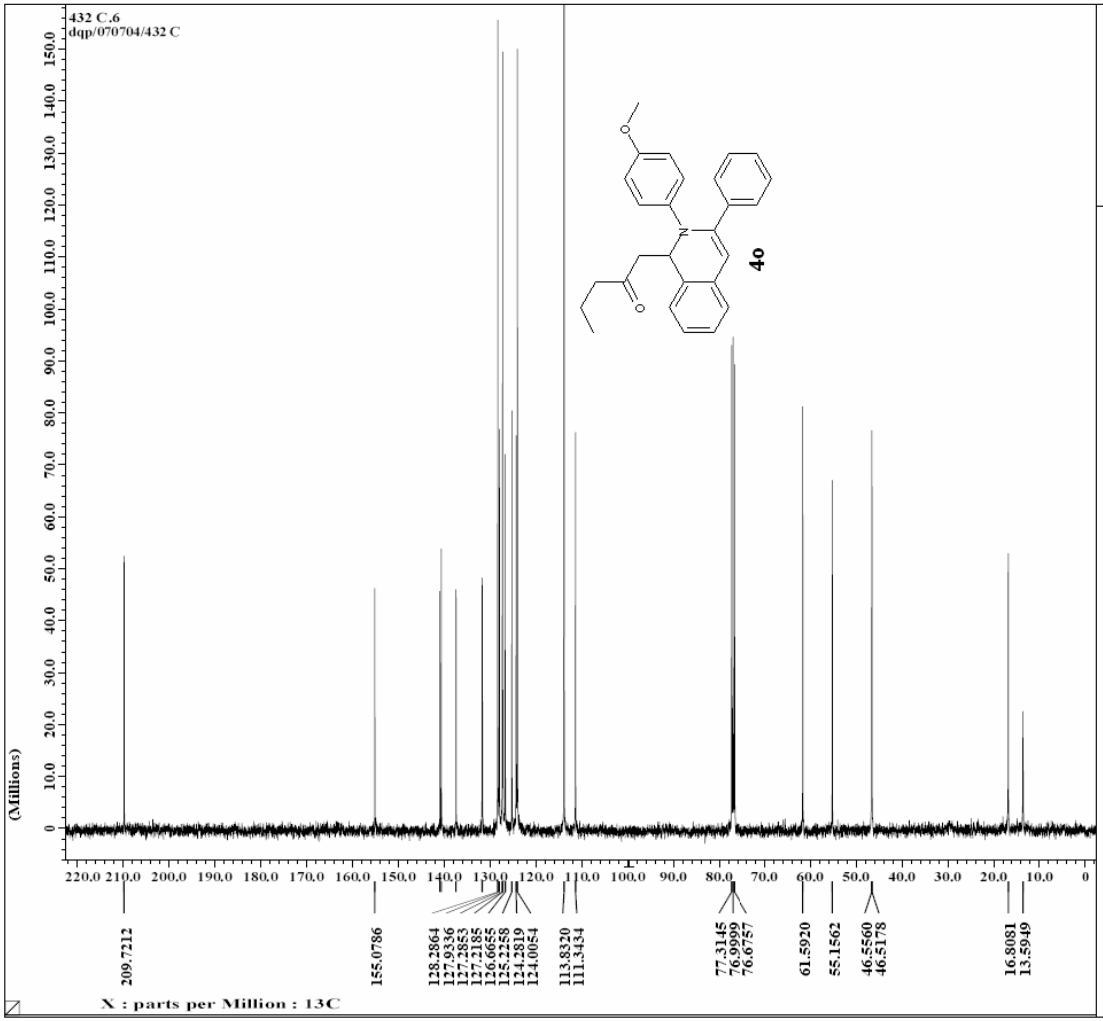



```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 77.174[ppm] : 0[ppm]
reference : 77.174[ppm] : 77[ppm]
reference : -0.176[ppm] : 77[ppm]
```

```
---- ACQUISITION PARAMETERS ----
Derived from: 421.17
File Name          = 421.17
Author             = delta
Sample ID          = dqp/070625/421
Content            = dqp/070625/421
Creation Date      = 25-JUN-2007 20:47:38

Revision Date      = 17-JUL-2007 10:24:19
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           = 110[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         = -40[ppm]
Mod_return         = 1
Scans              = 142
Total_scans        = 142

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]
```

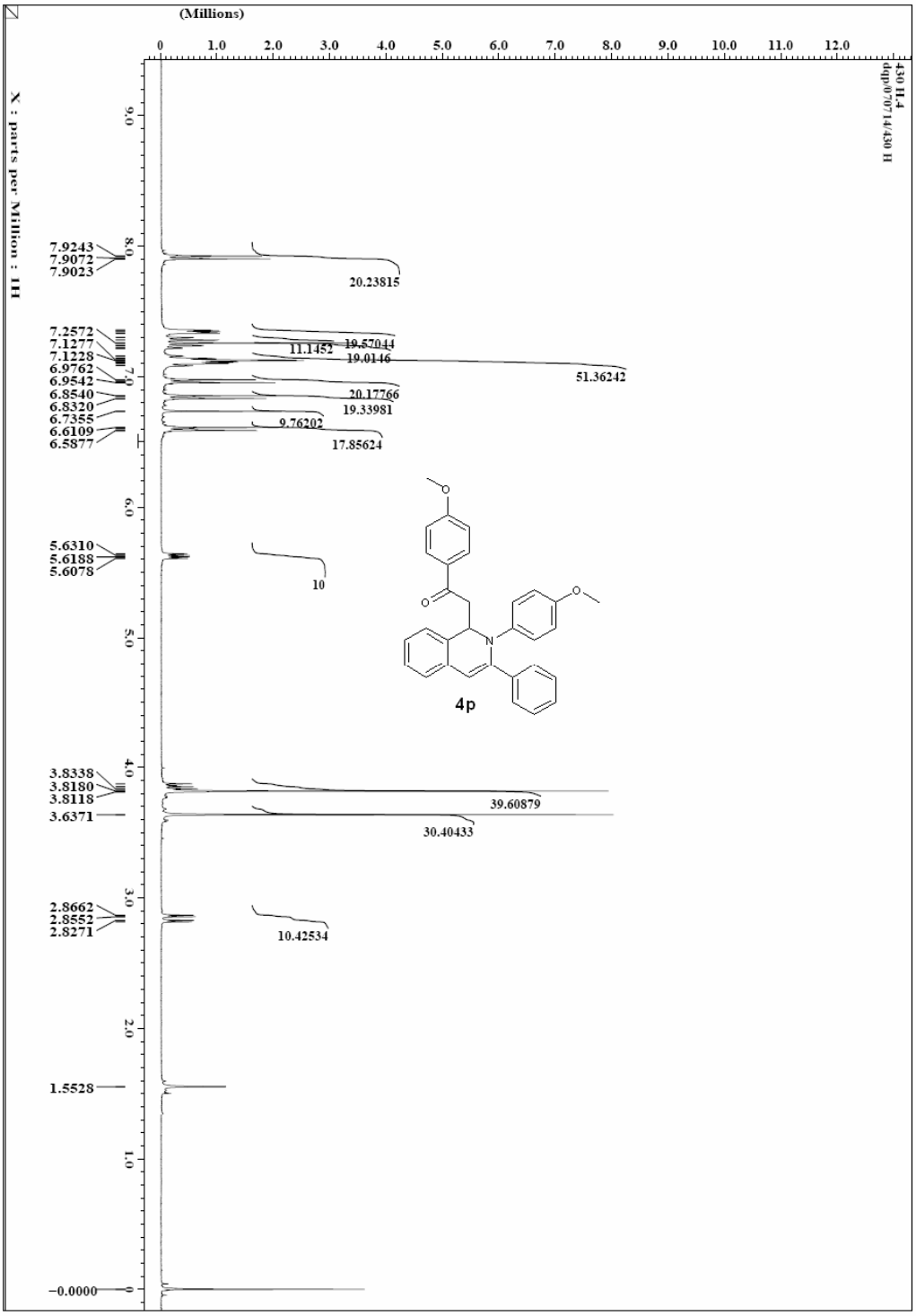


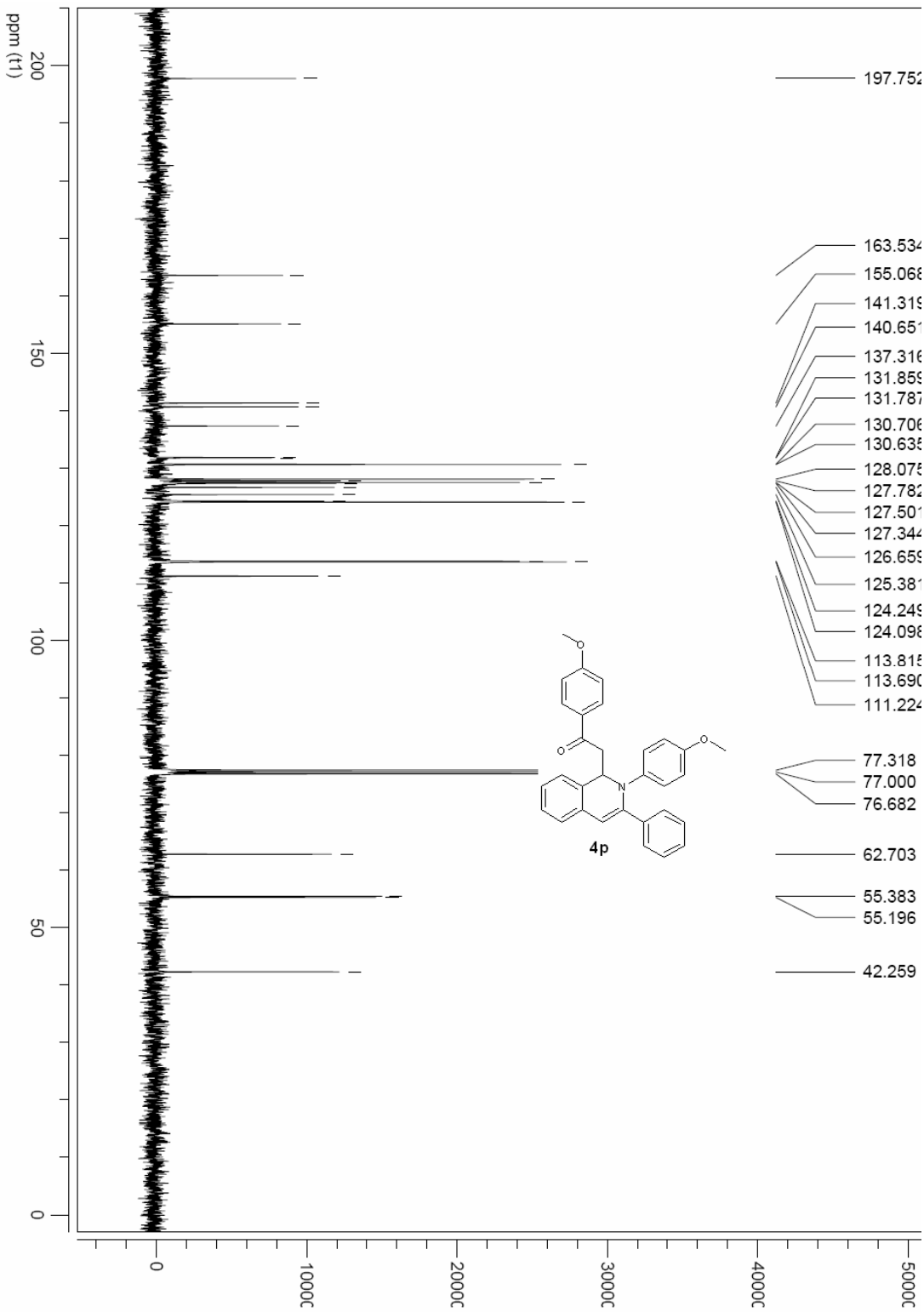

```
---- PROCESSING PARAMETERS ----
dc_balance
exp : 0.2[Hz] ; 0.0[s]
temperature : 0[°] ; 80[%] ; 100[%]
rg : 1
ff : 1 ; TRUE
machinephase
ppm

---- ACQUISITION PARAMETERS ----
Derived from: 432 H.2
File Name = 432 H.3
Author = delta
Sample ID = dqp/070704/433-2
Content = dqp/070704/433-2
Creation Date = 4-JUL-2007 22:27:07
Revision Date = 17-JUL-2007 10:45:14
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 = 2.047344464[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 = 2.047344464[s]
X_angle = 45[deg]
X_atten = 5.5[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 = 7.047344464[s]
```

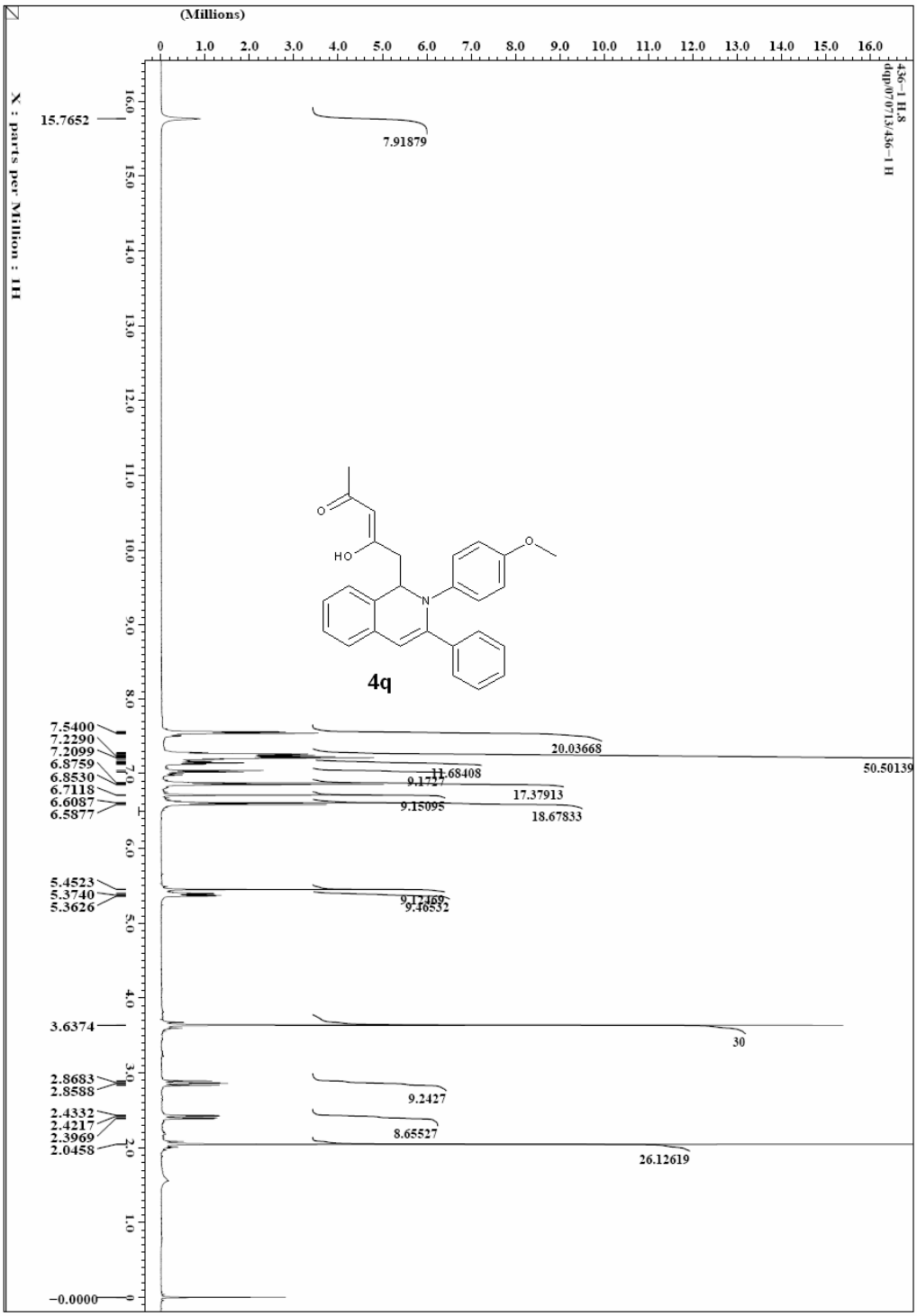


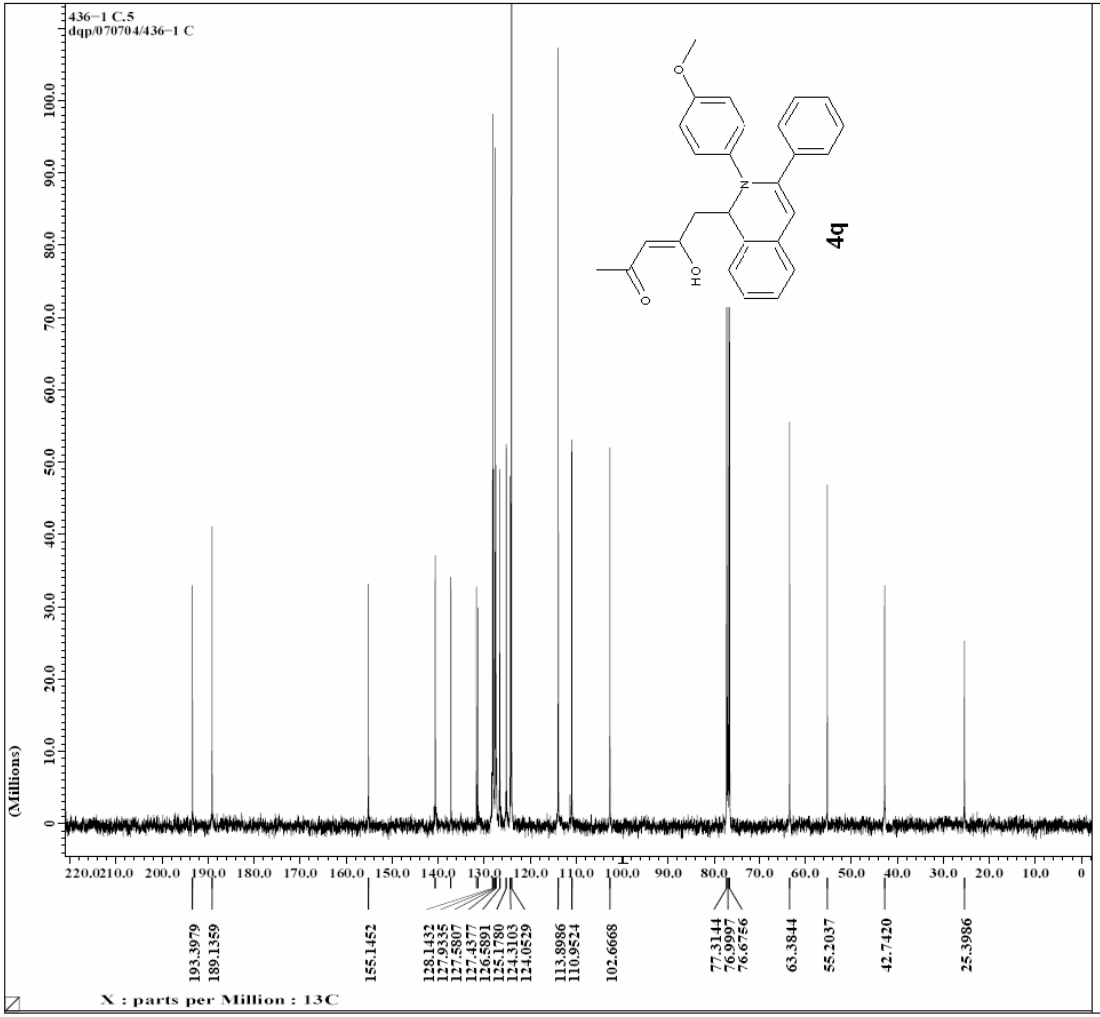

```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
reference : 77.222[ppm] : 77[ppm]
```

```
---- ACQUISITION PARAMETERS ----
Derived from: 432 C.6
File Name          = 432 C.6
Author             = delta
Sample ID          = dqp/070704/432 C
Content            = dqp/070704/432 C
Creation Date      =  4-JUL-2007 22:35:49

Revision Date      = 13-JUL-2007 10:35:37
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         = -42[ppm]
Mod_return         = 1
Scans              = 151
Total_scans        = 151

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]
```

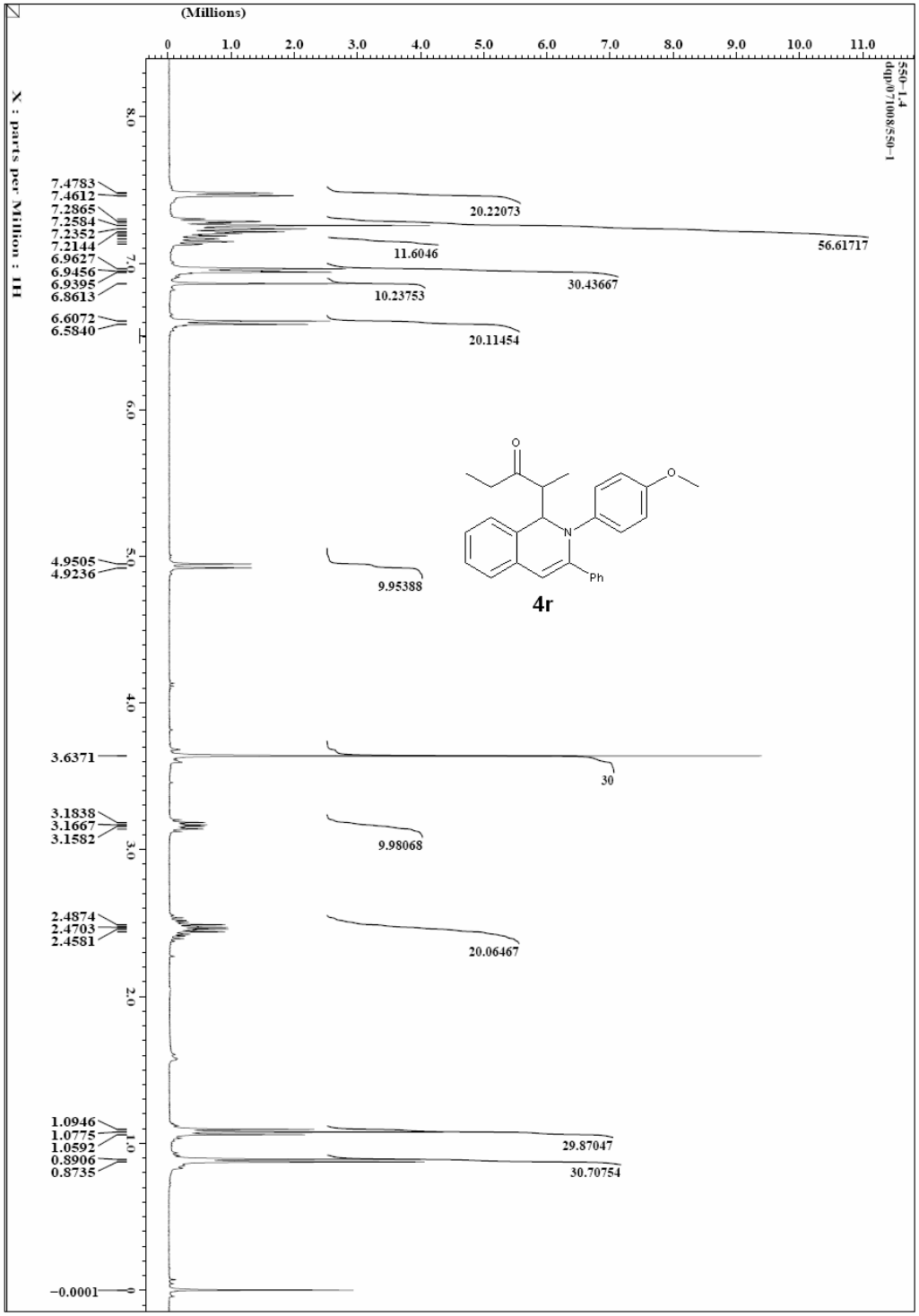

(Millions)
X : parts per Million : 1H
7.9243
7.9072
7.9023
20.23815
7.2572
7.1277
7.1228
6.9762
6.9542
6.8540
6.8320
6.7355
6.6109
6.5877
11.1452
19.57044
19.0146
51.36242
20.17766
19.33981
9.76202
17.85624
5.6310
5.6188
5.6078
10
4p
3.8338
3.8180
3.8118
3.6371
39.60879
30.40433
2.8662
2.8552
2.8271
10.42534
1.5528
−0.0000

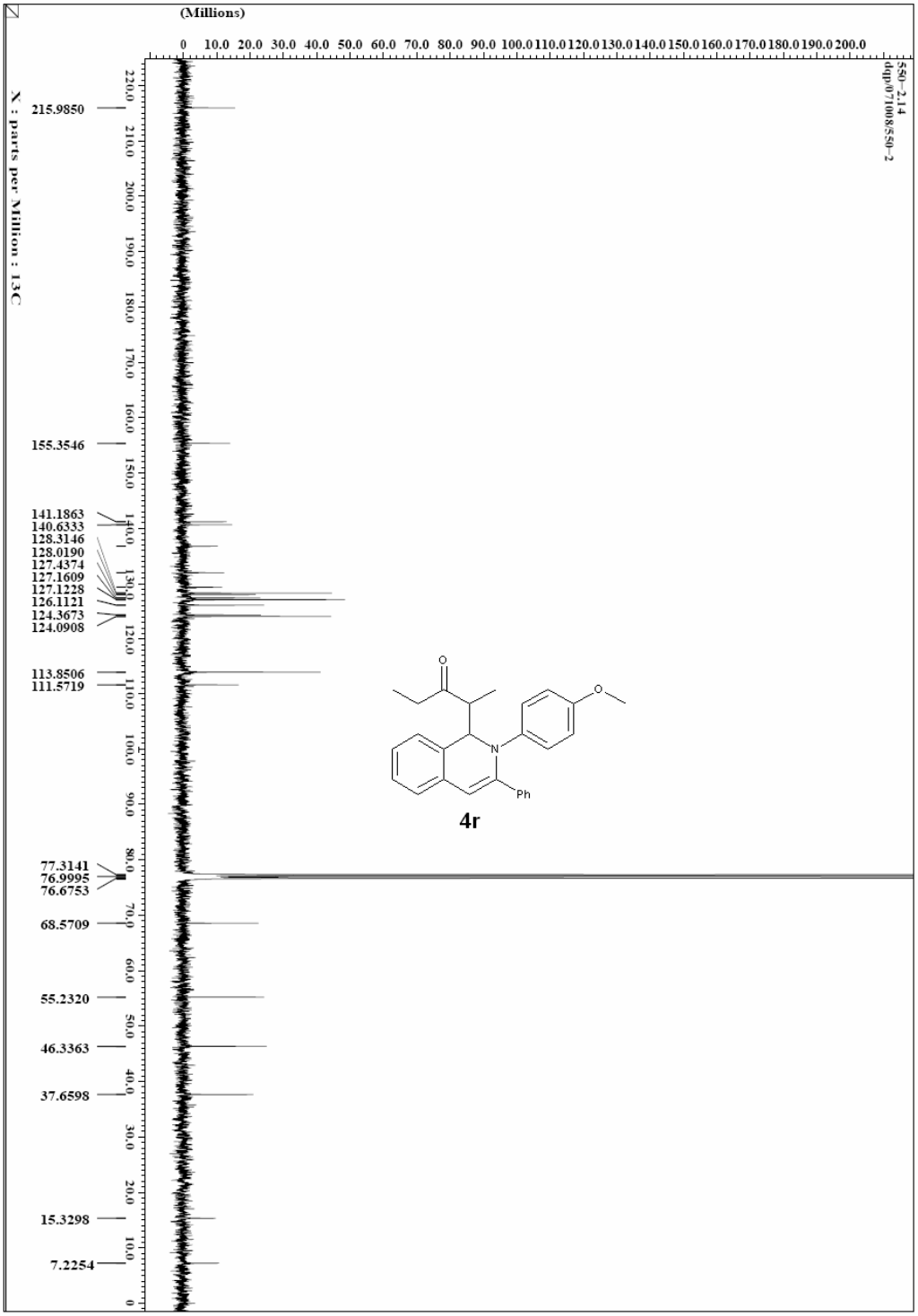

ppm (t1)
200
150
100
50
0
197.752
163.534
155.068
141.319
140.651
137.316
131.859
131.787
130.706
130.635
128.075
127.782
127.501
127.344
126.659
125.381
124.249
124.098
113.815
113.690
111.224
77.318
77.000
76.682
62.703
55.383
55.196
42.259
4p
0
10000
20000
30000
40000
50000

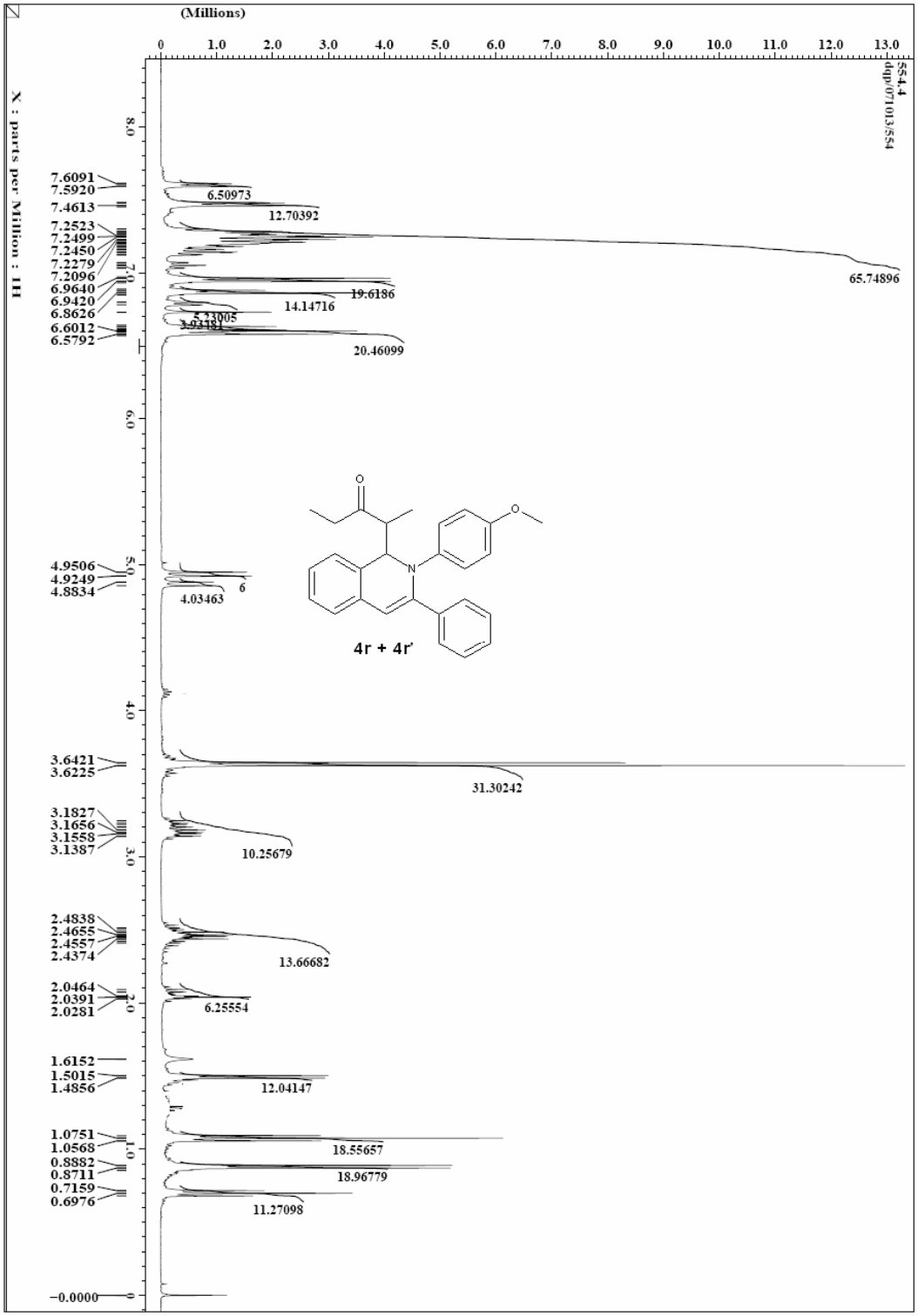

(Millions)
X : parts per Million : 1H
436-1 H.8
ddp/07/13/436-1 H
15.7652
7.91879
7.5400
7.2290
7.2099
6.8759
6.8530
6.7118
6.6087
6.5877
20.03668
50.50139
11.68408
9.1727
17.37913
9.15095
18.67833
5.4523
5.3740
5.3626
9.12469
9.46532
3.6374
30
2.8683
2.8588
9.2427
2.4332
2.4217
2.3969
2.0458
8.65527
26.12619
-0.0000
O
HO
N
O
4q

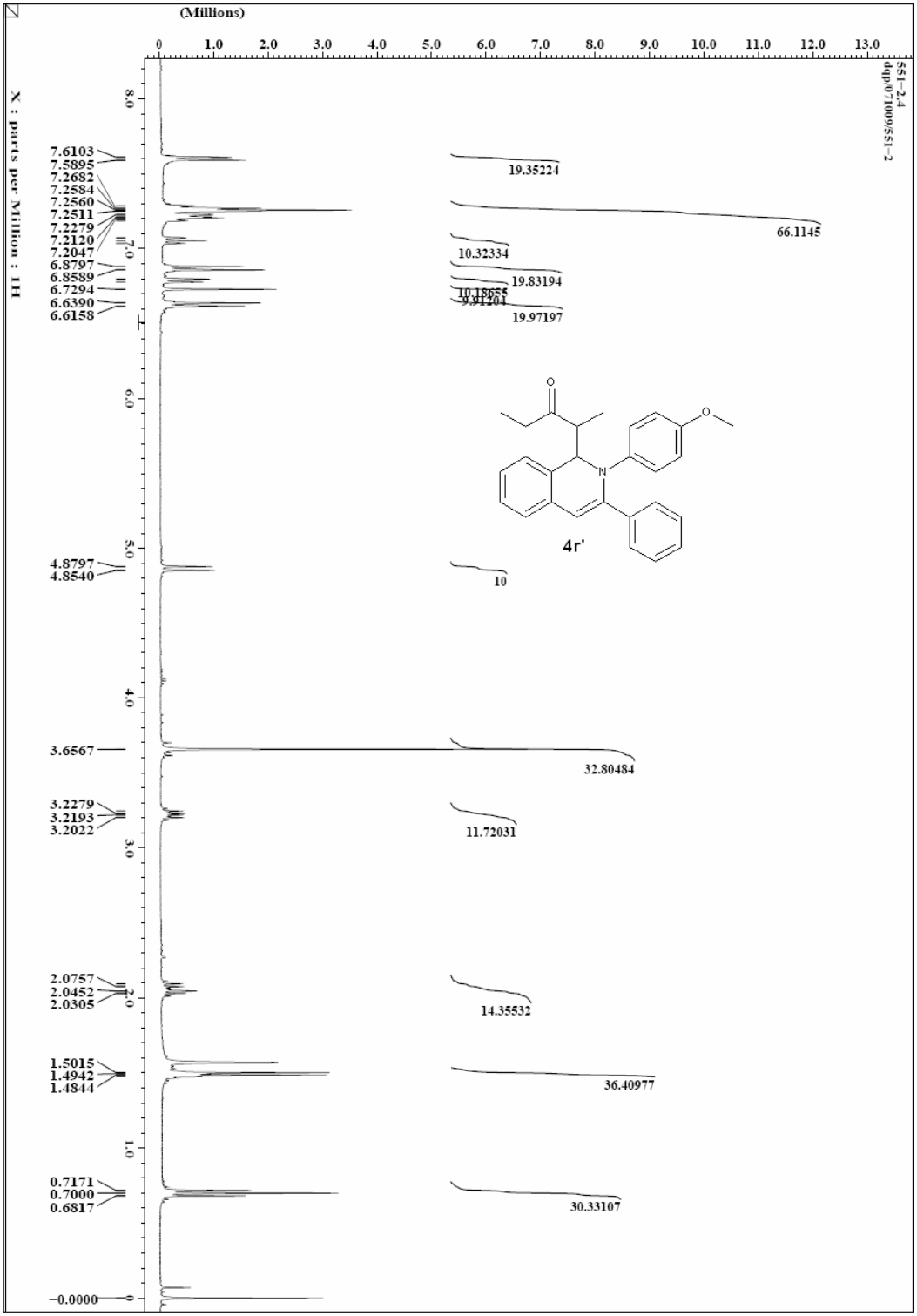


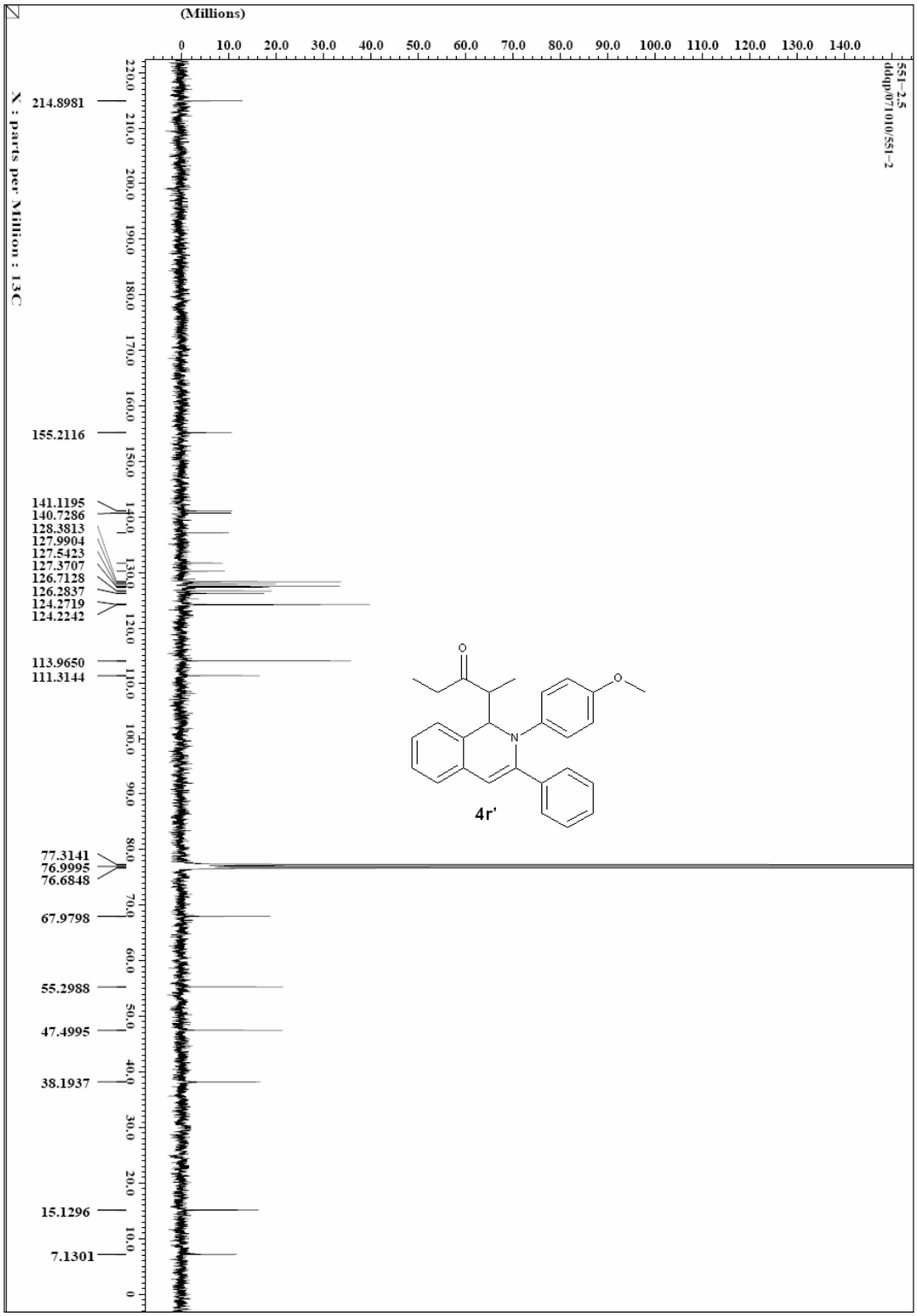

```
---- ACQUISITION PARAMETERS ----
Derived from: 436-1 C.5
File Name          = 436-1 C.5
Author             = delta
Sample ID          = dqp/070704/436-1 C
Content            = dqp/070704/436-1 C
Creation Date      =  4-JUL-2007 21:14:50

Revision Date      =  4-JUL-2007 21:23:05
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         = -42[ppm]
Mod_return         = 1
Scans              = 211
Total_scans        = 211

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]
```

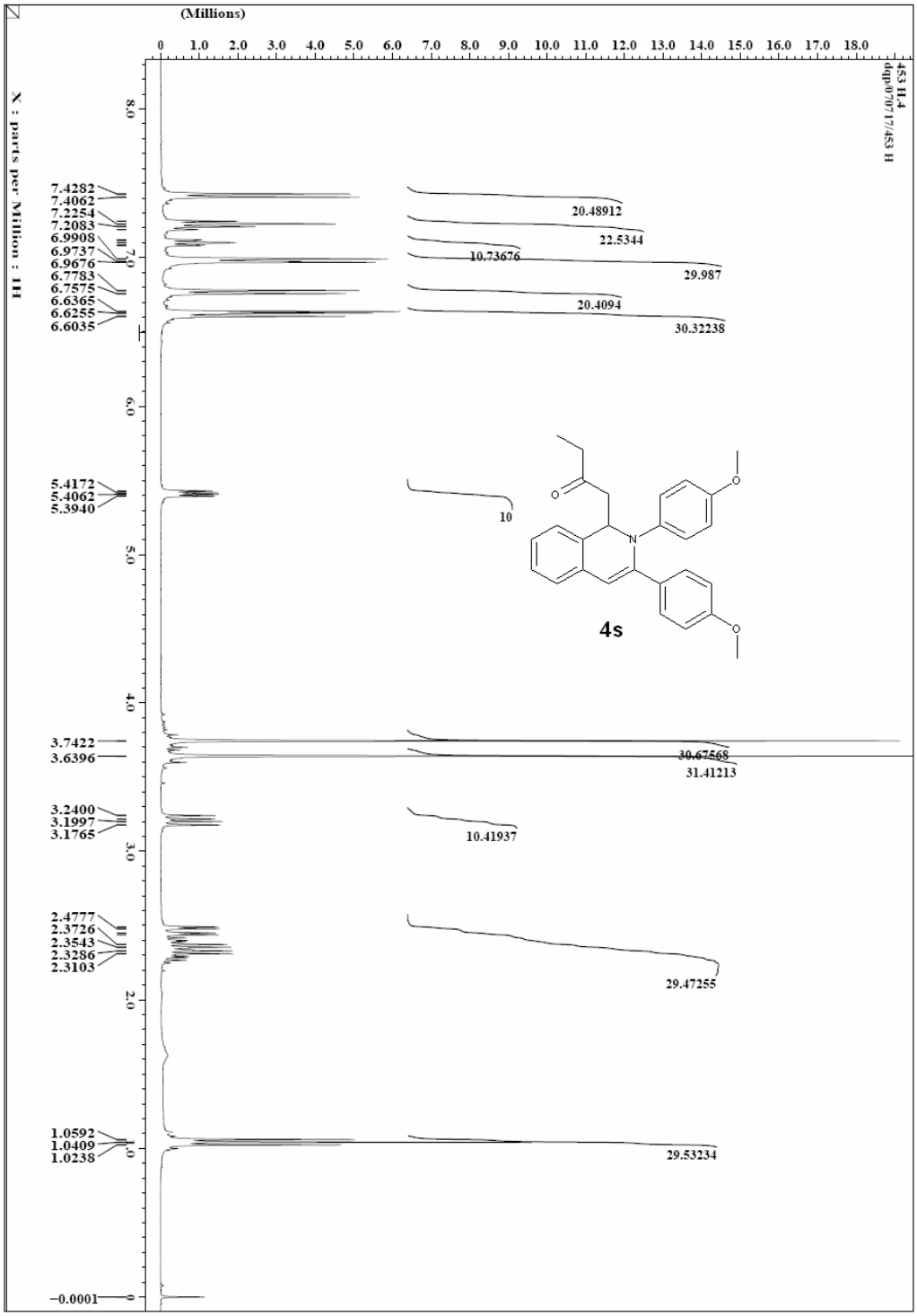

(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0
550-1.4
dqp071008/550-1
X : parts per Million : 1H
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4783
7.4612
7.2865
7.2584
7.2352
7.2144
6.9627
6.9456
6.9395
6.8613
6.6072
6.5840
4.9505
4.9236
3.6371
3.1838
3.1667
3.1582
2.4874
2.4703
2.4581
1.0946
1.0775
1.0592
0.8906
0.8735
−0.0001
20.22073
56.61717
11.6046
30.43667
10.23753
20.11454
9.95388
30
9.98068
20.06467
29.87047
30.70754
O
O
N
Ph
4r

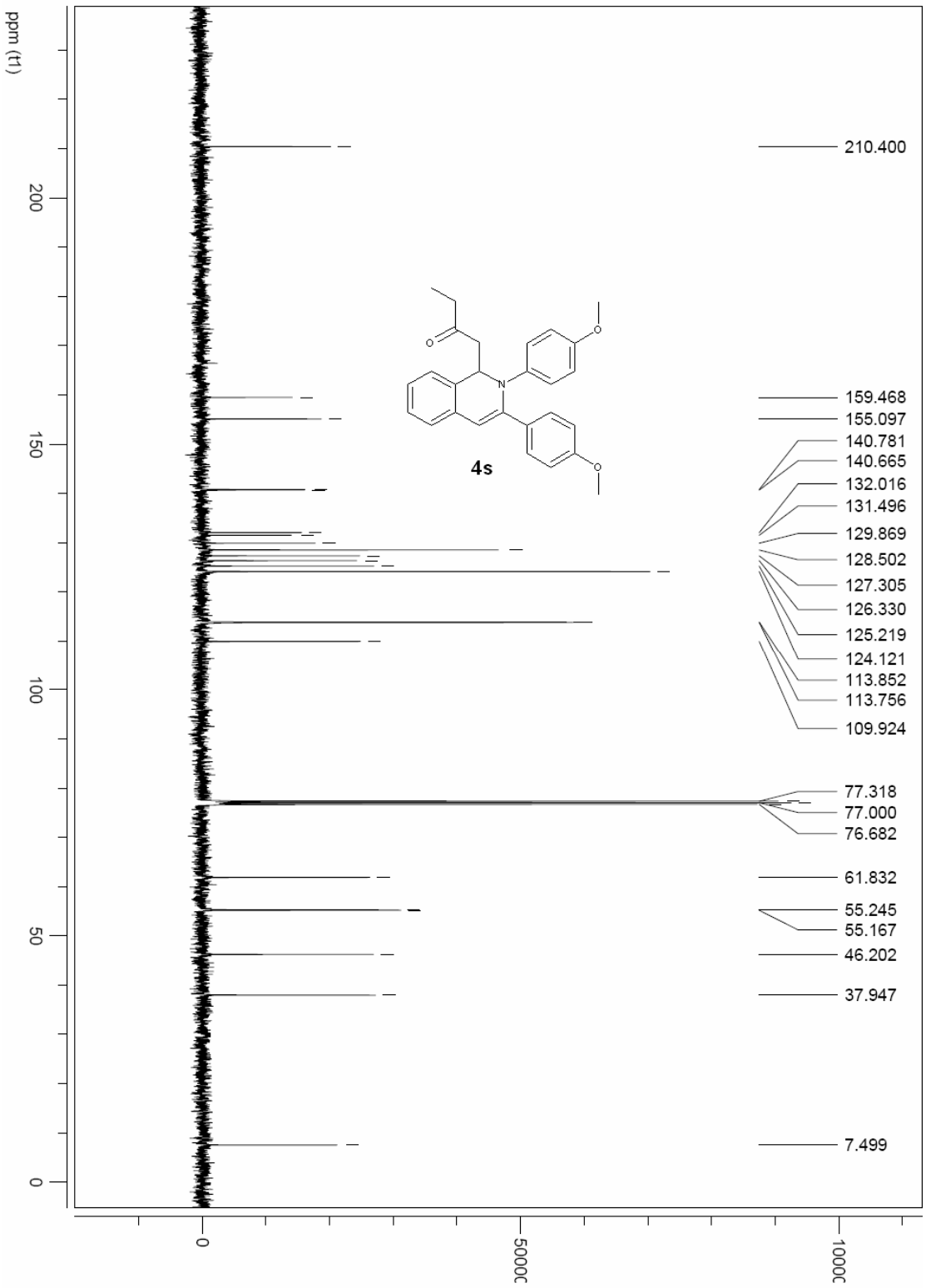

(Millions)
X : parts per Million : 13C
215.9850
155.3546
141.1863
140.6333
128.3146
128.0190
127.4374
127.1609
127.1228
126.1121
124.3673
124.0908
113.8506
111.5719
77.3141
76.9995
76.6753
68.5709
55.2320
46.3363
37.6598
15.3298
7.2254
O
N
Ph
O
4r

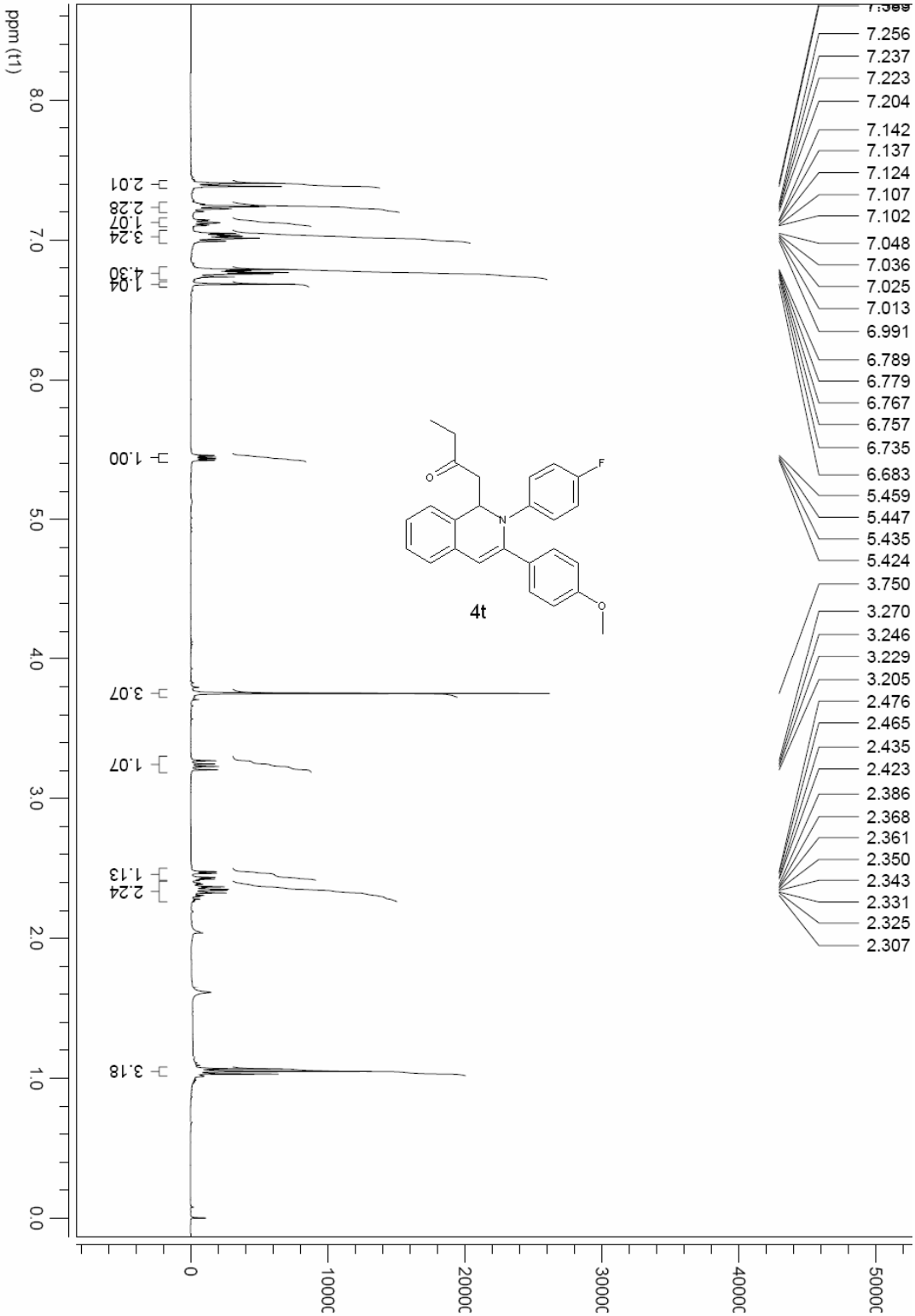

(Millions)
X : parts per Million : 1H
554.4
dqp/071013/554
7.6091
7.5920
7.4613
7.2523
7.2499
7.2450
7.2279
7.2096
6.9640
6.9420
6.8626
6.6012
6.5792
4.9506
4.9249
4.8834
3.6421
3.6225
3.1827
3.1656
3.1558
3.1387
2.4838
2.4655
2.4557
2.4374
2.0464
2.0391
2.0281
1.6152
1.5015
1.4856
1.0751
1.0568
0.8882
0.8711
0.7159
0.6976
−0.0000
6.50973
12.70392
65.74896
19.6186
14.14716
5.23005
3.93181
20.46099
4.03463
6
31.30242
10.25679
13.66682
6.25554
12.04147
18.55657
18.96779
11.27098
4r + 4r'

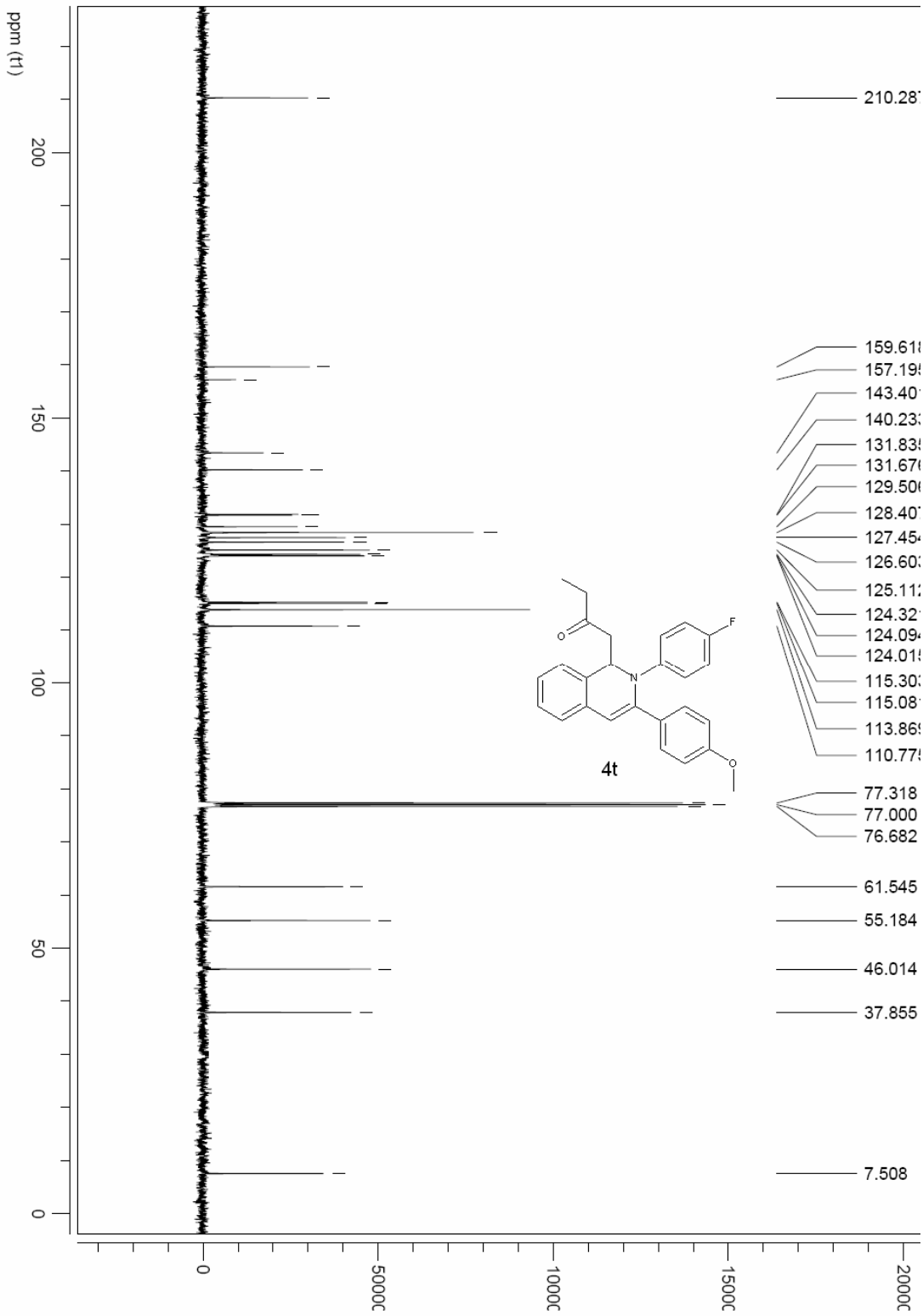

(Millions)
X : parts per Million : 1H
551-2,4
dqp/071009/551-2
7.6103
7.5895
7.2682
7.2584
7.2560
7.2511
7.2279
7.2120
7.2047
6.8797
6.8589
6.7294
6.6390
6.6158
4.8797
4.8540
3.6567
3.2279
3.2193
3.2022
2.0757
2.0452
2.0305
1.5015
1.4942
1.4844
0.7171
0.7000
0.6817
-0.0000
19.35224
66.1145
10.32334
19.83194
19.97197
10
32.80484
11.72031
14.35532
36.40977
30.33107
4r'

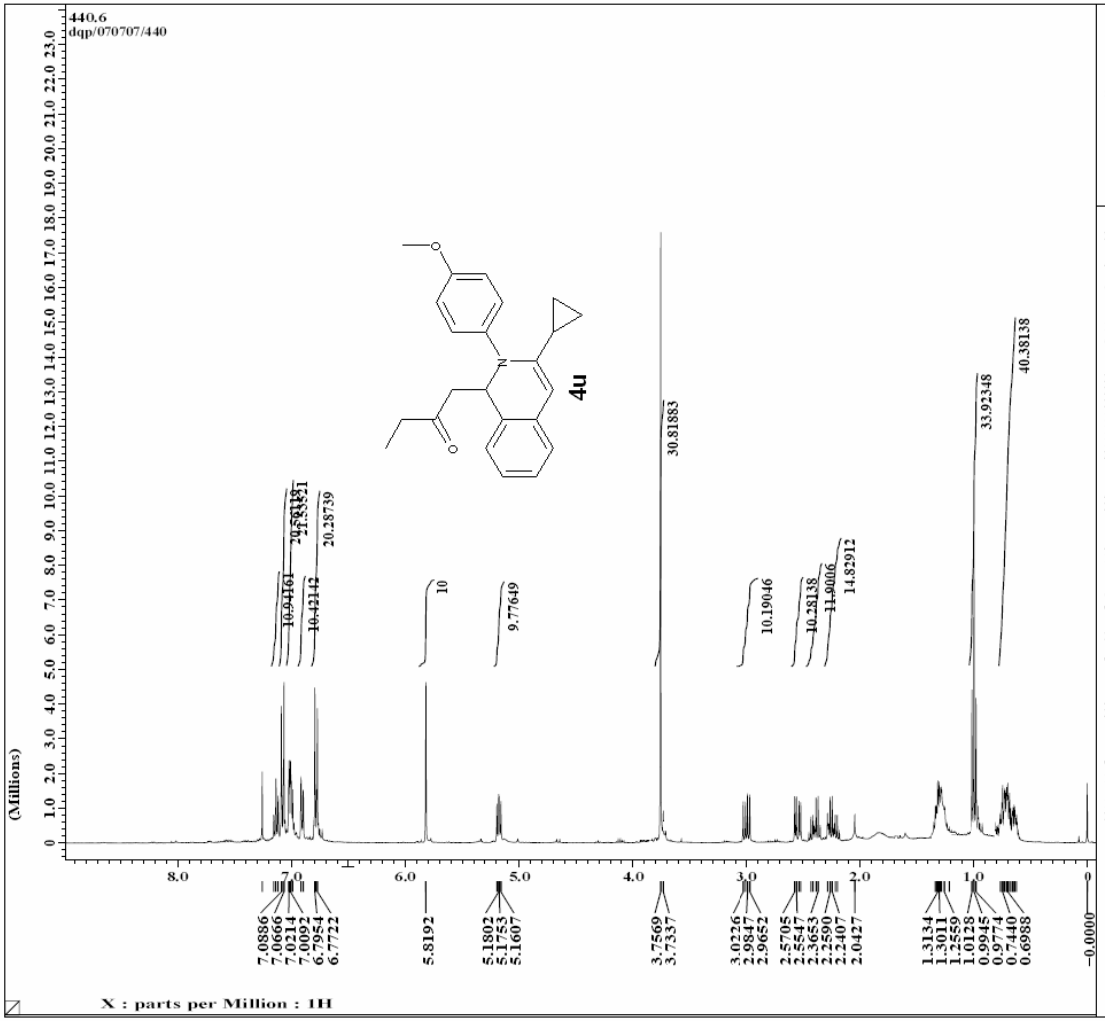

(Millions)
0 10.0 20.0 30.0 40.0 50.0 60.0 70.0 80.0 90.0 100.0 110.0 120.0 130.0 140.0
551-2.5
ddqp/071010/551-2
X : parts per Million : 13C
214.8981
155.2116
141.1195
140.7286
128.3813
127.9904
127.5423
127.3707
126.7128
126.2837
124.2719
124.2242
113.9650
111.3144
77.3141
76.9995
76.6848
67.9798
55.2988
47.4995
38.1937
15.1296
7.1301
4r'


(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
X : parts per Million : 1H
453 H-4
ddp/0/07/17/453 H
7.4282
7.4062
7.2254
7.2083
6.9908
6.9737
6.9676
6.7783
6.7575
6.6365
6.6255
6.6035
5.4172
5.4062
5.3940
3.7422
3.6396
3.2400
3.1997
3.1765
2.4777
2.3726
2.3543
2.3286
2.3103
1.0592
1.0409
1.0238
−0.0001
20.48912
22.5344
10.73676
29.987
20.4094
30.32238
10
30.67568
31.41213
10.41937
29.47255
29.53234
4s


ppm (t1)
200
150
100
50
0
0
5000
10000
210.400
159.468
155.097
140.781
140.665
132.016
131.496
129.869
128.502
127.305
126.330
125.219
124.121
113.852
113.756
109.924
77.318
77.000
76.682
61.832
55.245
55.167
46.202
37.947
7.499
4s

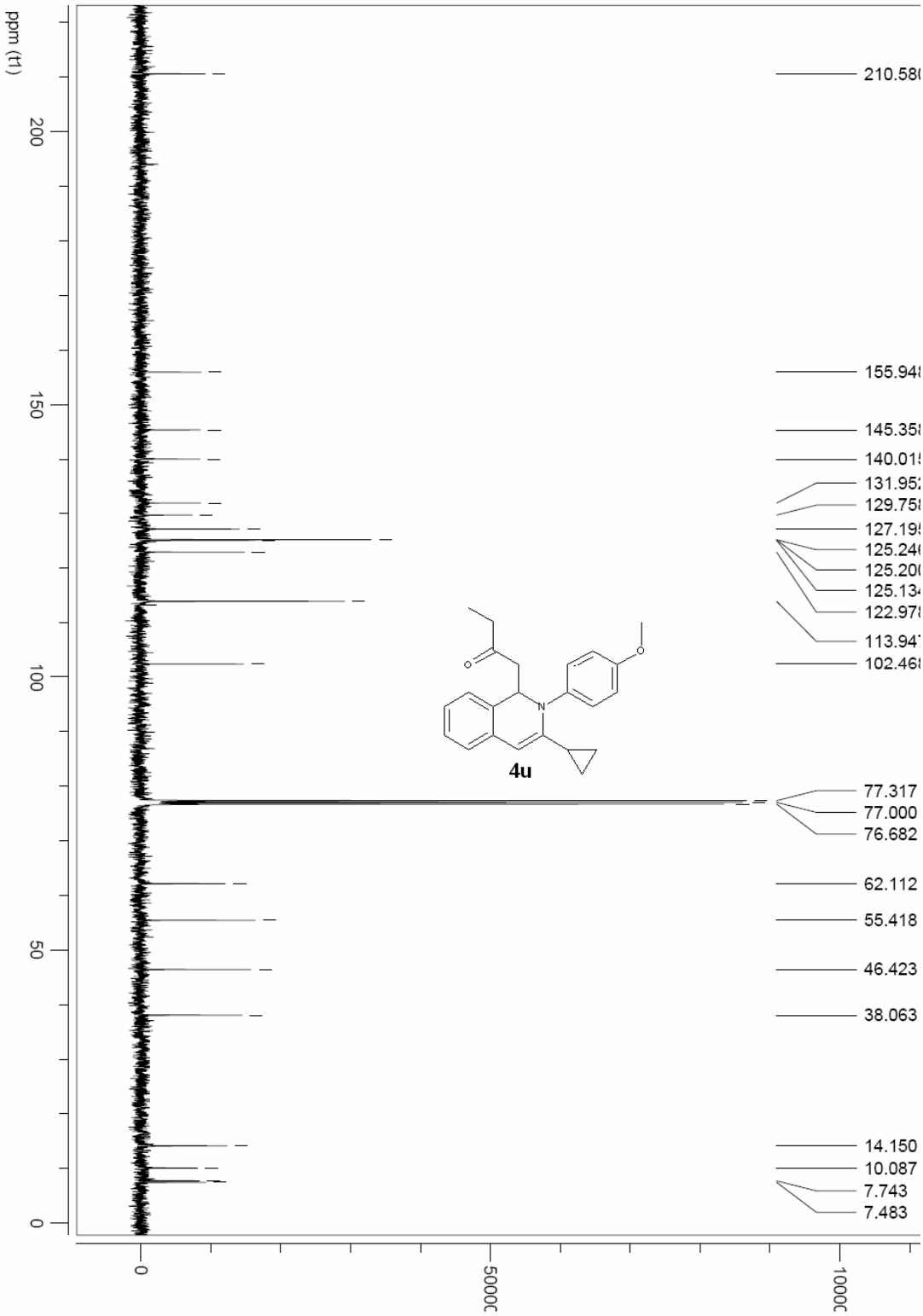

7.256
7.237
7.223
7.204
7.142
7.137
7.124
7.107
7.102
7.048
7.036
7.025
7.013
6.991
6.789
6.779
6.767
6.757
6.735
6.683
5.459
5.447
5.435
5.424
3.750
3.270
3.246
3.229
3.205
2.476
2.465
2.435
2.423
2.386
2.368
2.361
2.350
2.343
2.331
2.325
2.307
3.18
1.13
2.24
1.07
3.07
1.00
4.30
1.04
3.24
1.07
2.28
2.01
4t
F
O
N
O
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10000
20000
30000
40000
50000

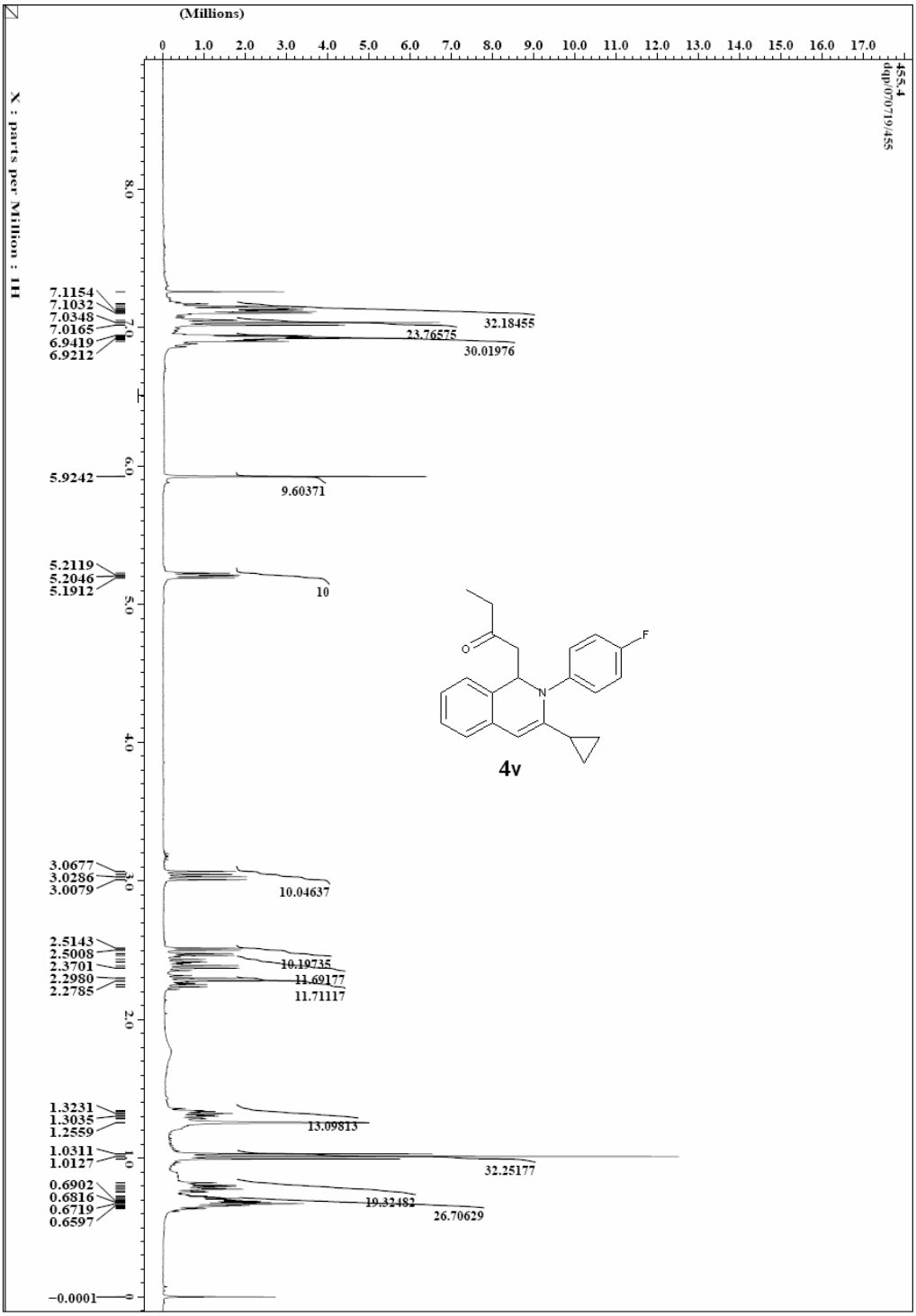

ppm (t1)
210.287
159.618
157.195
143.401
140.233
131.835
131.676
129.506
128.407
127.454
126.603
125.112
124.321
124.094
124.015
115.303
115.081
113.869
110.775
77.318
77.000
76.682
61.545
55.184
46.014
37.855
7.508
4t

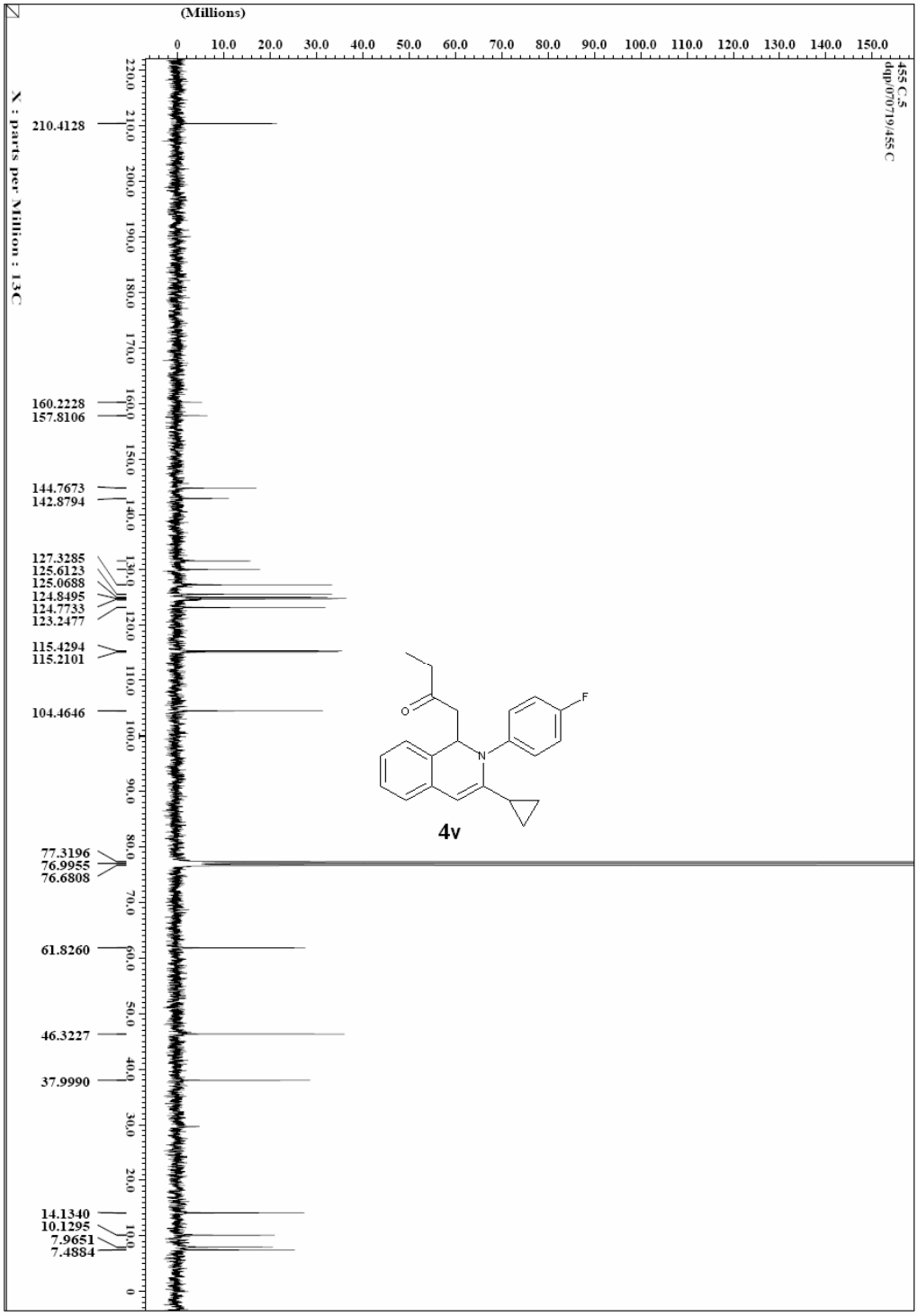


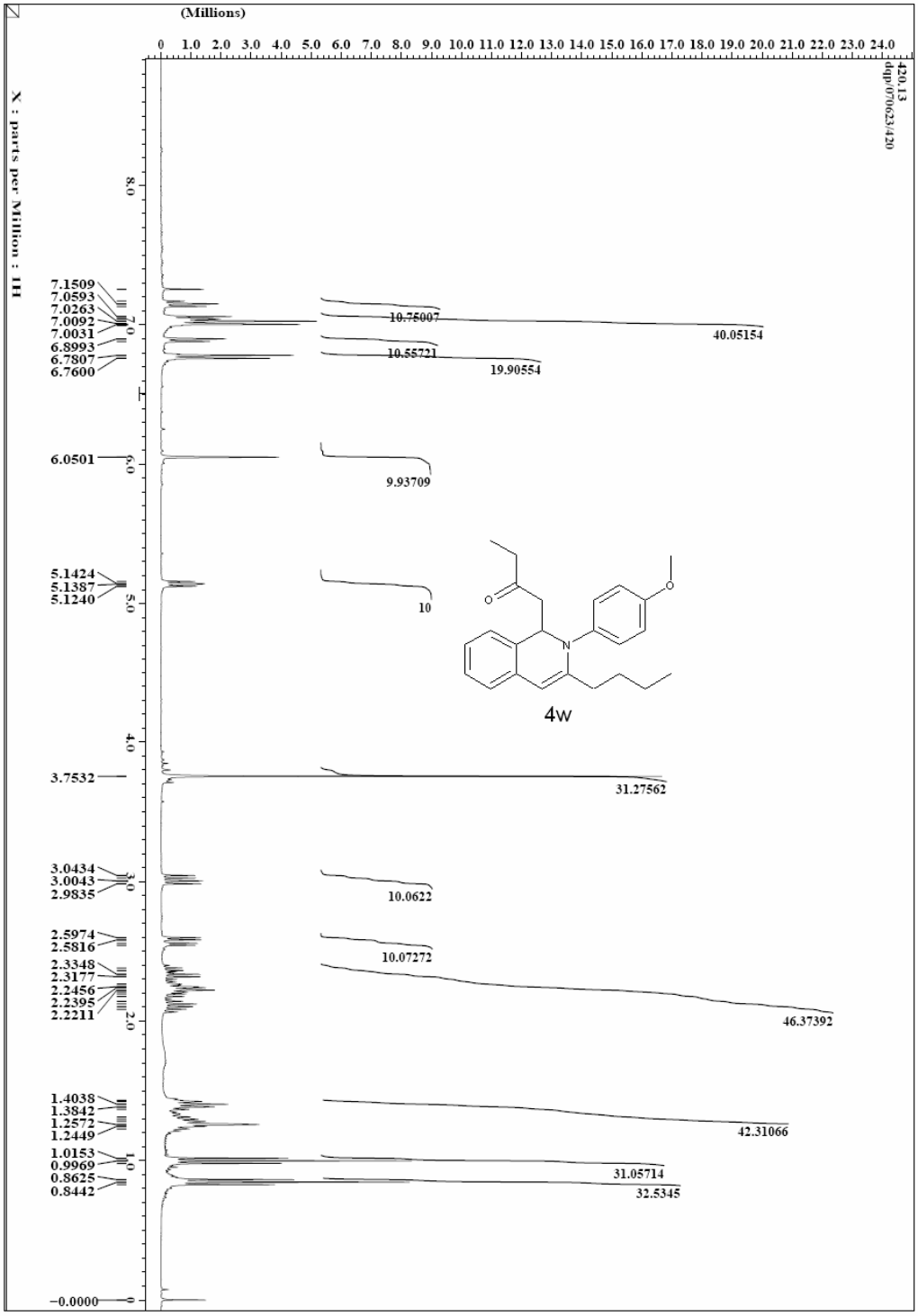

```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE
machinephase
ppm
```

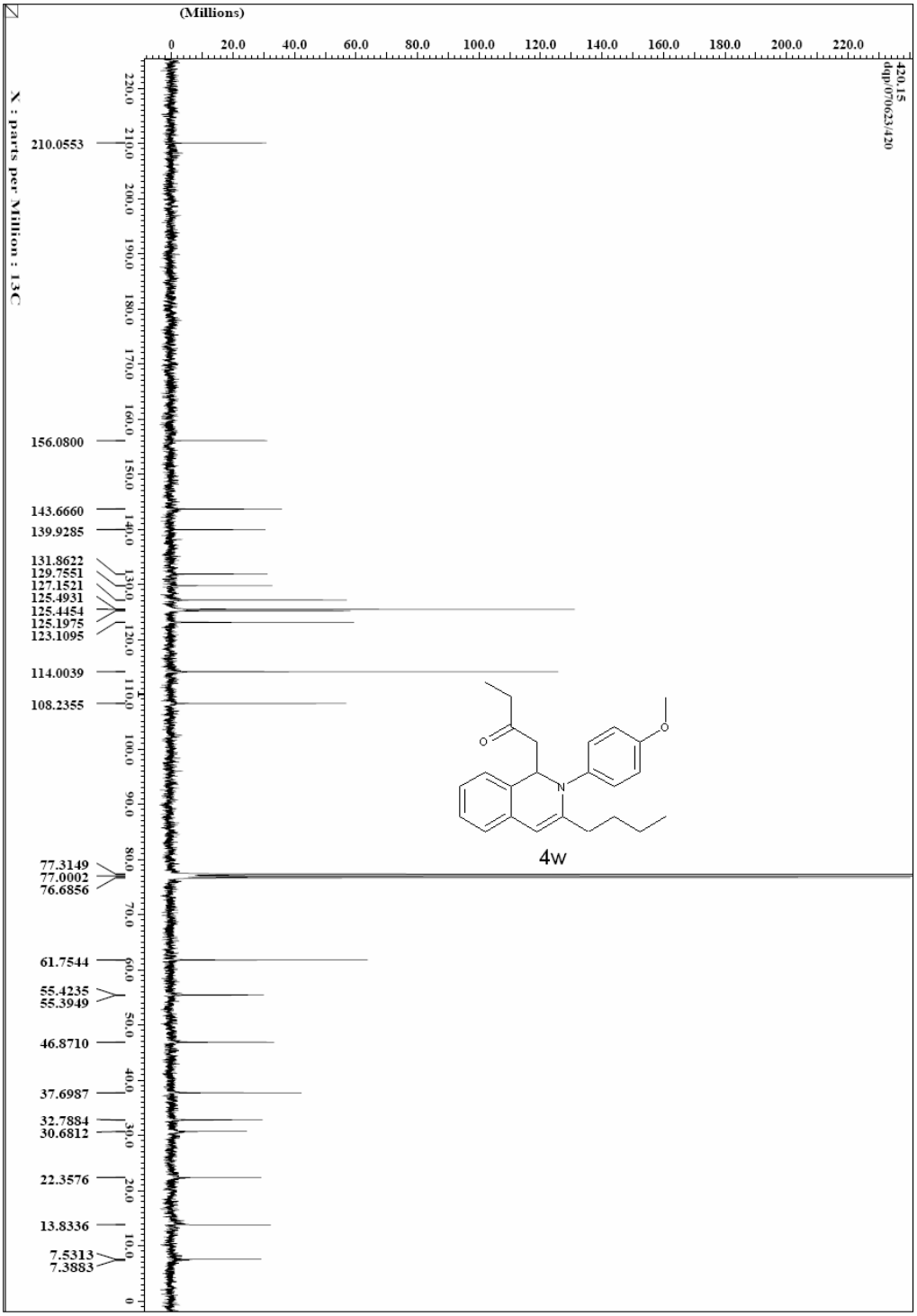
```
---- ACQUISITION PARAMETERS ----
Derived from: 440.4
File Name          = 440.6
Author             = delta
Sample ID          = dqp/070707/440
Content            = dqp/070707/440
Creation Date      =  7-JUL-2007 15:44:53

Revision Date      = 17-JUL-2007 10:52:52
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]
```

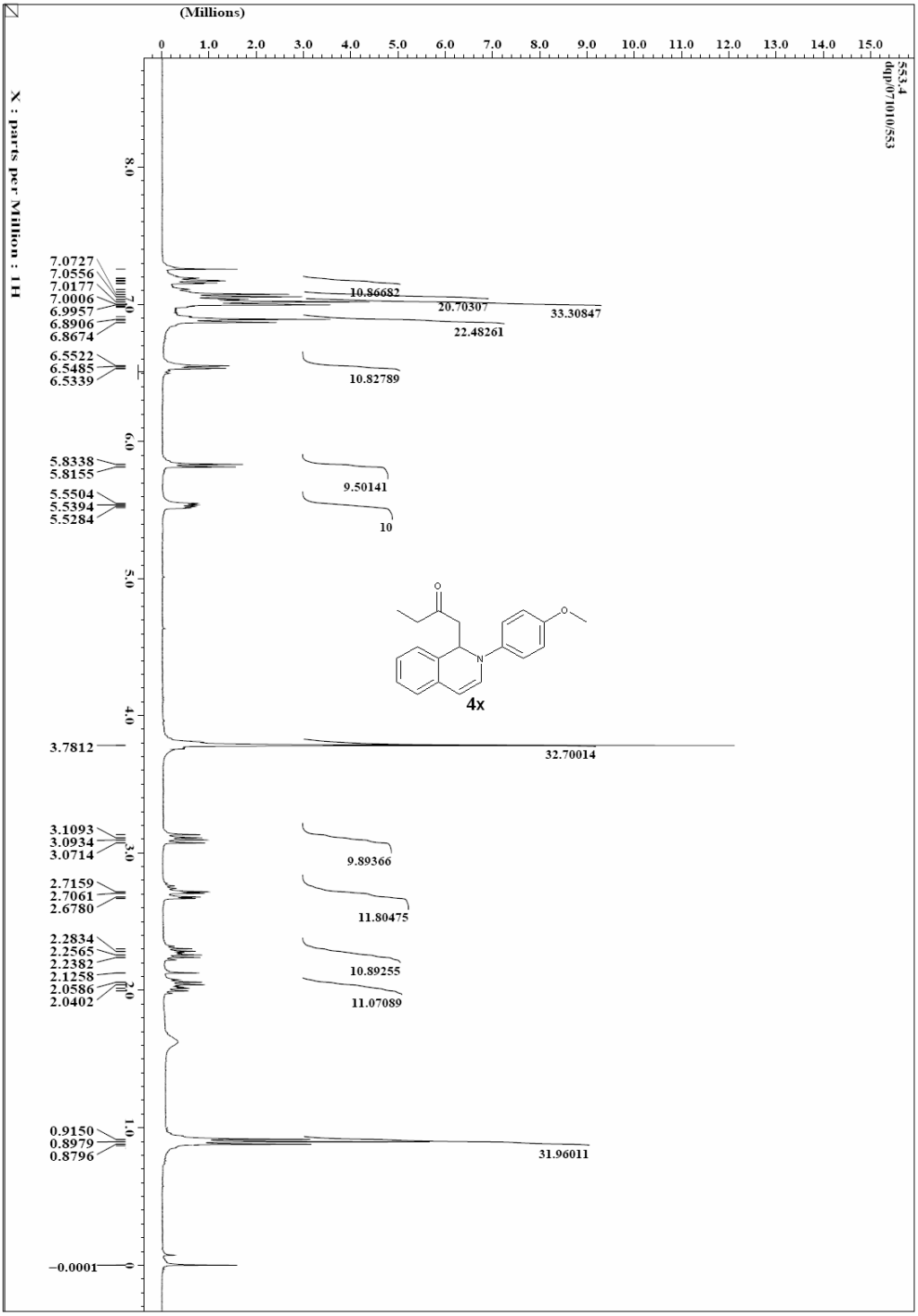

ppm (t1)
200
150
100
50
0
5000
10000
210.58
155.94
145.35
140.01
131.95
129.75
127.19
125.24
125.20
125.13
122.97
113.94
102.46
77.317
77.000
76.682
62.112
55.418
46.423
38.063
14.150
10.087
7.743
7.483
4u

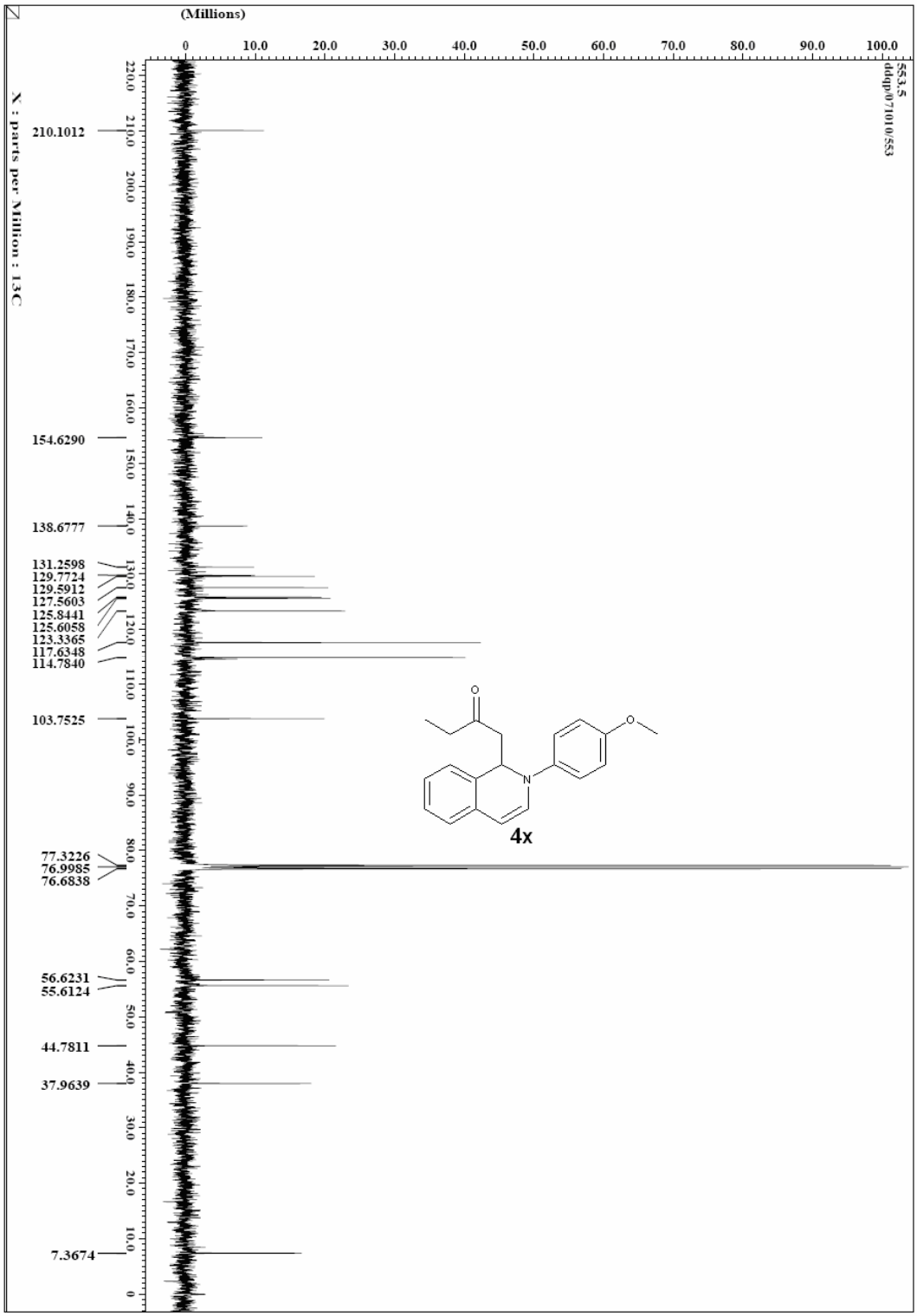

(Millions)
X : parts per Million : 1H
455.4
dup/070719/455
7.1154
7.1032
7.0348
7.0165
6.9419
6.9212
5.9242
5.2119
5.2046
5.1912
3.0677
3.0286
3.0079
2.5143
2.5008
2.3701
2.2980
2.2785
1.3231
1.3035
1.2559
1.0311
1.0127
0.6902
0.6816
0.6719
0.6597
−0.0001
32.18455
23.76575
30.01976
9.60371
10
10.04637
10.19735
11.69177
11.71117
13.09813
32.25177
19.32482
26.70629
O
N
F
4v


(Millions)
N : parts per Million : 13C
455 C,5
ddp/070719/455 C
210.4128
160.2228
157.8106
144.7673
142.8794
127.3285
125.6123
125.0688
124.8495
124.7733
123.2477
115.4294
115.2101
104.4646
77.3196
76.9955
76.6808
61.8260
46.3227
37.9990
14.1340
10.1295
7.9651
7.4884
F
O
N
4v


(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
420.13
ddp/070623/420
X : parts per Million : 1H
7.1509
7.0593
7.0263
7.0092
7.0031
6.8993
6.7807
6.7600
6.0501
5.1424
5.1387
5.1240
3.7532
3.0434
3.0043
2.9835
2.5974
2.5816
2.3348
2.3177
2.2456
2.2395
2.2211
1.4038
1.3842
1.2572
1.2449
1.0153
0.9969
0.8625
0.8442
−0.0000
10.75007
40.05154
10.55721
19.90554
9.93709
10
31.27562
10.0622
10.07272
46.37392
42.31066
31.05714
32.5345
4w


(Millions)
N : parts per Million : 13C
420.15
dup/070623/420
210.0553
156.0800
143.6660
139.9285
131.8622
129.7551
127.1521
125.4931
125.4454
125.1975
123.1095
114.0039
108.2355
77.3149
77.0002
76.6856
61.7544
55.4235
55.3949
46.8710
37.6987
32.7884
30.6812
22.3576
13.8336
7.5313
7.3883
4w


(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
553.4
dqp/071010/553
X : parts per Million : 1H
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.0727
7.0556
7.0177
7.0006
6.9957
6.8906
6.8674
6.5522
6.5485
6.5339
5.8338
5.8155
5.5504
5.5394
5.5284
3.7812
3.1093
3.0934
3.0714
2.7159
2.7061
2.6780
2.2834
2.2565
2.2382
2.1258
2.0586
2.0402
0.9150
0.8979
0.8796
−0.0001
10.86682
20.70307
33.30847
22.48261
10.82789
9.50141
10
32.70014
9.89366
11.80475
10.89255
11.07089
31.96011
4x


(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
553.5
ddqp/071010/553
X : parts per Million : 13C
210.1012
154.6290
138.6777
131.2598
129.7724
129.5912
127.5603
125.8441
125.6058
123.3365
117.6348
114.7840
103.7525
77.3226
76.9985
76.6838
56.6231
55.6124
44.7811
37.9639
7.3674
4x