Supporting Information

Efficient Catalytic Syntheses of α-pyridones and 3(2H)-Isoquinolones through Ruthenium-catalyzed Cycloisomerization of 3-En-5-ynyl and o-alkynylphenyl Nitrones

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Contents:

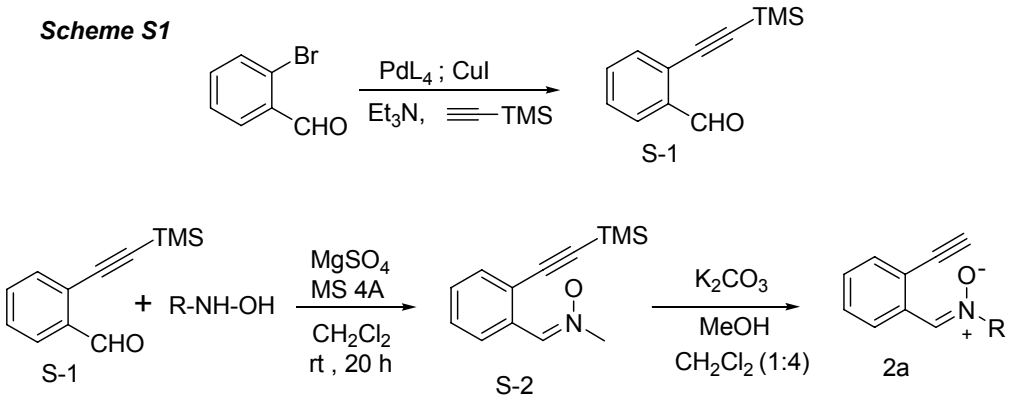
(1) Representative Synthetic procedures.

(a) General procedures

Unless otherwise noted, all experiments were carried out under argon atmosphere in oven-dried glassware using stander syringe, cannula and septa apparatus. Tetrahydrofuran, diethyl ether, Toluene and hexane were dried with sodium and distilled before use. DMF and CH_2Cl_2 were dried over CaH_2, and distilled before use. All the ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ solution. Coupling constants (*J* values) are given in Hertz (Hz) and chemical shifts are expressed in parts per million (ppm). Substrate was prepared from the corresponding readily available bromobenzaldehyde after several steps.

(b) Synthesis of substrate:

Scheme S1



(c) Synthesis of 2-trimethylsilanylethynyl benzaldehyde (S-1):

To a solution of CuI (0.103 g, 10 mol %) in Et_3N (30 ml), was added 2-bromo-benzaldehyde (1.0 g, 5.43 mmol) and degassed with nitrogen for 15 minutes at 23 □. To this resulting solution was added $Pd(PPh_3)_4$ (0.315 g, 5 mol %), and the mixture was stirred for 15 min before being treated with ethynyltrimethylsilane (0.586 g, 5.98 mmol) drop wise. The resulting solution

was stirred at room temperature for 10 h, and then filtered through a celite pad, concentrated and eluted through a silica column to give the desired trimethylsilanylethynyl benzaldehyde (**S**-1) (0.92 g, 84%).

(d) Synthesis of 2-trimethylsilanylethynyl-nirtone (S-2):

Compound **S-1** 1g (5.2 m.mol) was dissolved in dry CH_2Cl to this solution methyl hydroxylamine hydrochloride 1.24g (7.8 m.mol) and anhydrous $MgSo_4$ 1.32g (10.4 m.mol) powdered molecularsives 1g, was added under nitrogen atmosphere, and stirred for 20h at room temperature. After completion of starting material reaction mixture was filtered and concentrated and purified by column chromatography, which yielded compound-(**S-2)** 86% (1.3g).

(e) Synthesis of 2-arylethynyl-nitrone (2a):

Compound-(**S-2)** 0.9g (3.0m.mol) was dissolved in CH_2Cl_2 and MeOH (4:1 ratio mixture) and to this solution K_2CO_3 0.83g (6.0 m.mol) was added and stirred at room temperature for 6 h. Completion of starting material reaction mixture was filtered and concentrated and purified by column chromatography, which yielded compound-(**2a)** 80 %(500mg).

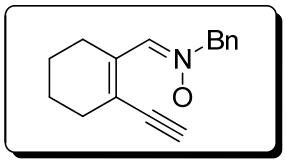
(f) Typical procedure for the synthesis of compound (3a):

A catalytic tube contains $TpRuPPh_3$ $(CH_3CN)_2SbF_6$ (72 mg 0.084 mmol) was applied to vacuum for 15 minutes, and evacuated with argon gas. Then compound (**2a**) (150mg 0.84 m.mol) was dissolved in dry toluene this solution was added to above catalytic tube by using syringe, and degassed by purging argon gas into reaction mixture, then this reaction mixture was heated to 90^oC and reaction monitored by TLC. After completion of starting material heating was stopped, and cooled to room temperature, filter through small celite bed. Solvent was removed under vacuum and purified by column chromatography over flurosil,

which yielded compound-(**3a)** 75% (120mg).

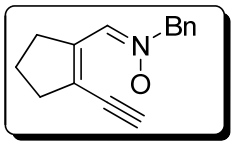
(2) Spectral data for key compounds:

Spectral data for compound 1a:



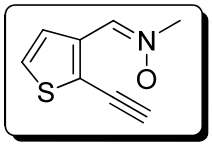
IR (neat, cm^{-1}): 3250 (s), 2250 (m), 1680 (s), 1540(s), 1464 (s), 1250(m), 1093 (s), 820(s), 776 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.60 (s, 1 H), 7.35-7.41 (m, 5 H), 4.88 (s, 2H), 3.31 (s, 3H), 2.84 (m, 2H), 2.24 (m, 2H), 1.57 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 135.5, 133.3, 129.2, 128.83, 128.8, 84.8, 82.6, 71.1, 31.0, 26.7, 21.7, 21.3; HRMS calcd for $C_{16}H_{17}NO$: 239.1310, found 239.1314.

Spectral data for compound 1b:



IR (neat, cm^{-1}): 3280 (s), 2989 (m), 2050(m), 1950(w), 1650(m), 1440 (s), 1204 (s), 1093 (s), 830(s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.46 (s, 1 H), 7.33-7.42 (m, 5 H), 4.89 (s, 2H), 3.48 (s, 1H), 3.15-3.10 (m, 2H), 2.49-2.45 (m, 2H), 1.92-1.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.3, 133.2, 131.6, 129.1, 128.8, 128.0, 87.1, 70.5, 36.2, 33.8, 23.0 ; HRMS calcd for $C_{15}H_{15}NO$: 225.1154, found 225.1150.

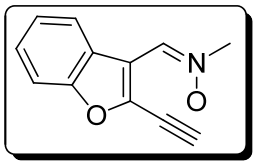
Spectral data for compound 1c :



IR (neat, cm^{-1}): 3280 (s), 2950 (m), 1643 (s), 1540(b), 1460(s), 1320(m), 1204 (s),

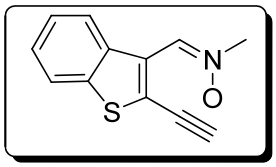
1093 (s), 706 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (s, 1H), 7.31 (d, *J* = 5.2 Hz, 1H,), 7.13 (d, *J* = 5.2 Hz, 1H,), 3.87 (s, 3H) 3.34 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.5, 130.0, 129.4, 127.8, 121.5, 83.2, 77.3, 52.0 ; HRMS calcd C_8H_7NOS for: 165.0248, found 165.0252.

Spectral data for compound 1d :



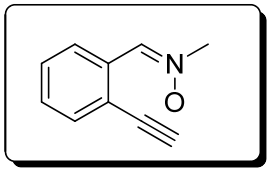
IR (neat, cm^{-1}): 3320 (s), 3000(s), 2830 (m), 1673 (s),1540(m), 1310(s), 1240 (s), 1093 (s), 760 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64. (d, *J* = 6.4 Hz, 1H), 7.63 (s, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.39 (m, 1H), 7.30(m, 1H), 3.93 (s, 3H), 3.66 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.2, 150.5, 127.4, 127.2, 124.0, 123.9, 120.4, 112.3, 87.7, 73.7, 54.5 ; HRMS calcd for $C_{12}H_9NO_2$: 199.0633, found 199.0637.

Spectral data for compound 1e :



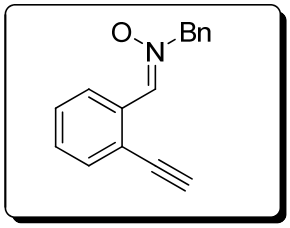
IR (neat, cm^{-1}): 3238 (s), 2989 (m), 2920(s), 2750(s), 1654 (s), 1270(s), 1204 (s), 1093 (s), 705 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (s, 1H), 7.91 (m, 1H,), 7.84(m, 1H), 7.43(m, 2H), 3.93 (s, 3H), 3.71 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.2, 137.9, 136.5, 130.8, 126.4, 125.1, 122.6, 122.5, 117.3, 86.3, 76.5, 52.6 ; HRMS calcd for $C_{12}H_9NOS$: 215.0405, found215.0409.

Spectral data for compound 2a:



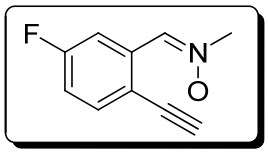
IR (neat, cm^{-1}): 3180 (s), 2940(s), 2890 (m), 1653 (s), 1426(m), 1280(s), 1204 (s), 1093 (s), 740 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.25 (d, *J* = 8 Hz, 1 H), 7.97 (s, 1 H), 7.53(d, *J* = 0.8 Hz, 1H), 7.44-7.30 (m, 2H), 3.91 (s, 3H), 3.40(s, 1H),; ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.8, 131.6, 129.6, 129.2, 127.3, 120.8, 83.3, 80.9, 54.9 ; HRMS calcd for $C_{10}H_9NO$, 169.0684, found169.0687.

Spectral data for compound 2b:



IR (neat, cm^{-1}): 3280 (s), 2960(s), 2850 (m), 1630 (s), 1460(m), 1280(s),, 746 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.29 (d, *J* = 3.2 Hz, 1 H), 8.03 (S, 1 H), 7.29-7.51(m, 8H), 5.06 (s, 1 H); 3.32 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.0, 132.6, 132.0, 131.6, 129.6, 129.2, 128.9, 127.5, 121.2, 83.2, 80.8, 71.6; HRMS calcd for $C_{16}H_{13}NO$: 235.0997, found 235.0994.

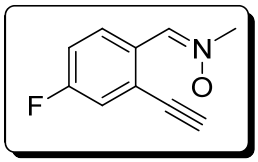
Spectral data for compound 2c:



IR (neat, cm^{-1}): 3230 (s), 2900(s), 2840 (m), 1680 (s), 1436(m), 1305(s), 1243 (s), 756 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.05 (d, *J* = 2.4 Hz, 1H,), 7.92 (s, 1H), 7.47-7.43 (m, 1H), 7.01-6.96(m, 1H), 3.87 (s, 3H), 3.38 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 161.1, 134.4 (d, *J* = 42), 133.5 132.0, 116.8 (d, *J* = 35),

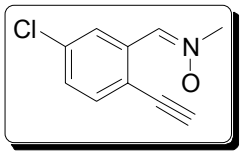
114.2 (d), 83.1, 80.0, 55.1; HRMS calcd for $C_{10}H_8FNO$:177.0590, found177.0593.

Spectral data for compound 2d:



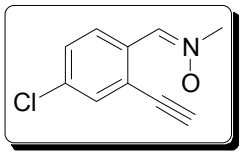
IR (neat, cm^{-1}): 3238 (s), 2980 (m), 1645 (s), 1295(s), 1245 (s), 1190 (s), 786 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.36 (d, *J* = 8.6 Hz, 1H), 7.89 (s, 1H), 7.21-7.18 (m, 1H), 7.12-7.07 (m, 1H), 3.87 (s, 3H), 3.45 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 161.1, 134.4 (d ,*J* = 32 Hz), 133.5, 132.0, 116.8 (d, *J* = 88 Hz), 114.2 (d, *J*=35 Hz), 83.1, 80.0, 55.1; HRMS calcd for$C_{10}H_8FNO$:177.0590, found177.0593.

Spectral data for compound 2e:



IR (neat, cm^{-1}): 3300 (s), 3100 (s), 2940 (m), 1658 (s), 1304 (s), 1253 (s), 850 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.35(d, *J* = 2.8 Hz, 1H), 7.93(s, 1H), 7.44(d, *J* = 8.4 Hz, 1H), 7.30(dd, *J* = 6.4 Hz, 1H), 3.91 (s, 3H), 3.44(s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.6, 133.6, 133.0, 131.8, 129.8, 127.1, 119.1, 84.2,80.1, 55.2; HRMS calcd for $C_{10}H_8ClNO$: 193.0294, found193.0291.

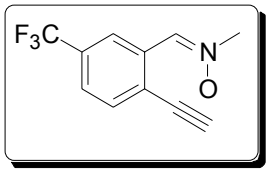
Spectral data for compound 2f:



IR (neat, cm^{-1}): 3280 (s), 3150 (s), 2800 (m), 1650 (s), 1314 (s), 1260, 840 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.27 (d, *J* = 8.8 Hz, 1H), 7.91(s, 1H), 7.39(s, 1H), 7.37(dd, *J* = 2.4 Hz, 1H,), 3.90 (s, 3H), 3.44(s,1H) ; ^{13}C NMR (100 MHz, $CDCl_3$):

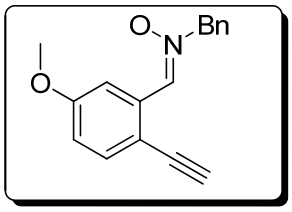
δ 135.1, 132.6, 132.0, 130.2, 129.5, 128.5, 122.4, 84.4,79.6, 55.0; HRMS calcd for $C_{10}H_8ClNO$: 193.0294, found193.0297.

Spectral data for compound 2g:



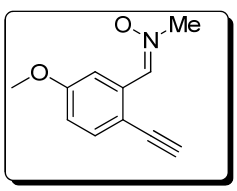
IR (neat, cm^{-1}): 3180 (s), 3050(s), 2900 (m), 1640 (s), 1330(s), 1290 (m), 1053 (s), 750 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.65 (s, 1H), 8.0 (s, 1H), 7.63 (d, *J* = 8 Hz, 1H) 7.56(m, 1H), 3.94 (s, 3H), 3.53 (s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.1, 132.3, 131.6, 126.0, 125.9, 124.0, 123.9, 85.6, 79.6, 55.1 ; HRMS calcd for $C_{11}H_8F_3NO_2$: 227.0558, found 227.0561.

Spectral data for compound 2h :



IR (neat, cm^{-1}): 3250(s), 2960(s), 2850 (m), 1640 (s), 1530(m), 1460(m), 1425(s), 1300(s),1050(s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.23 (d, *J* = 8.8 Hz, 1 H), 7.96 (s, 1H), 7.43-7.32 (m, 5 H), 7.01(s, 1H), 6.89 (d, *J* = 8 Hz, 1H) 5.03 (s, 2 H), 3.79 (s, 1 H), 3.24 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.1, 133.2, 131.9, 129.6, 129.3, 128.9, 124.8, 122.9, 118.0, 114.8, 83.1, 80.6, 71.2, 55.4 ; HRMS calcd for $C_{17}H_{15}NO_2$: 265.1103, found265.1107.

Spectral data for compound 2i :



IR (neat, cm^{-1}): 3180(s), 2960(s), 2850 (m), 1640 (s), 1530(m), 1460(m), 1425(s), 1300(s), 1050(s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.30 (d, J = 8.8 Hz, 1 H), 7.87 (S, 1 H), 7.04 (d, J = 2.8 Hz, 1 H), 6.94 (dd, J = 2.8 Hz, 1 H); 3.87 (s, 3H), 3.82 (s, 3H), 3.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7, 132.3, 130.1, 129.0, 124.6, 122.4, 117.7, 114.6, 83.1, 80.4, 55.1, 54.2 ; HRMS calcd for $C_{11}H_{11}NO_2$:189.0790, found189.0793.

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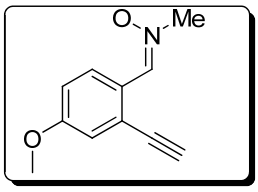
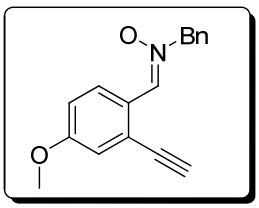
Spectral data for compound 2j :



IR (neat, cm^{-1}): 3300 (s), 2990 (m), 2850(s), 1663 (s),1305(m), 1204 (s), 1093 (s), 750 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 9.30 (d, J = 8.8 Hz, 1 H), 7.87 (S, 1 H), 7.04 (d, J = 2.8 Hz, 1 H), 6.94 (dd, J = 2.8 Hz, 1 H); 3.87 (s, 3H), 3.82 (s, 3H), 3.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7, 132.3, 130.1, 129.0, 124.6, 122.4, 117.7, 114.6, 83.1, 80.4, 55.1, 54.2; HRMS calcd for $C_{11}H_{11}NO_2$:189.0790, found189.0793.

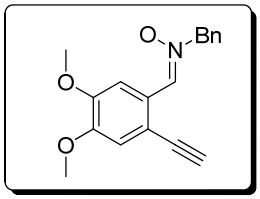
Spectral data for compound 2k :



IR (neat, cm^{-1}): 3260(s), 2960(s), 2850 (m), 1640 (s), 1530(m), 1460(m), 1425(s), 1305(s), 1050(s); ^1H NMR (400 MHz, $CDCl_3$): δ 9.32 (d, J = 8.8 Hz, 1 H), 7.90 (s, 1 H), 7.45-7.37 (m, 5 H), 7.02 (s,1H), 6.89 (d, J = 7.6 Hz, 1H), 5.03 (s, 2 H); 3.79

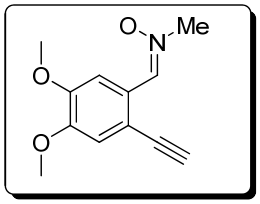
(s, 3H), 3.29 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.1, 133.2, 131.9, 129.6, 129.3, 128.9, 124.8, 122.9, 118.0, 114.8, 83.1, 80.6, 71.2, 55.4 ; HRMS calcd for $C_{17}H_{15}NO_2$: 265.1103, found265.1107.

Spectral data for compound 2l :



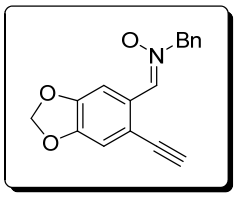
IR (neat, cm^{-1}): 3150(s), 3040 (s), 2989 (m), 2850 (m), 2150 (m), 1640 (s), 1530(m), 1460(m), 1425(s), 1300(s), 1050(s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.07 (s, 1 H), 7.97 (s, 1 H), 7.48-7.35 (m, 5 H), 6.97 (s, 1 H), 5.04 (s, 2H), 3.90 (s, 3 H), 3.87 (s, 3 H), 3.28 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ160.1, 133.2, 131.9, 129.6, 129.3, 128.9, 124.8, 122.9, 118.0, 114.8, 83.1, 81.0, 55.9, 55.8 ; HRMS calcd for $C_{18}H_{17}NO_3$: 295.1208, found295.1211.

Spectral data for compound 2m :



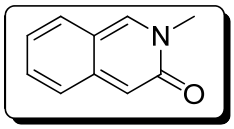
IR (neat, cm^{-1}): 3300 (s), 2950 (m), 2850(s), 2150(s), 1653 (s), 1450(m), 1307(m), 1210 (s), 1093 (s), 750 (m) ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.10 (s, 1 H), 7.89 (s, 1 H), 7.0 (s, 1 H), 3.94 (s, 3 H), 3.89 (s, 3 H), 3.88 (s, 3H), 3.35 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.6, 149.2, 133.1, 126.0, 114.7, 114.3, 109.9, 82.1, 81.0, 55.9, 54.6 ; HRMS calcd for $C_{12}H_{13}NO_3$: 219.0895, found219.0897.

Spectral data for compound 2n :



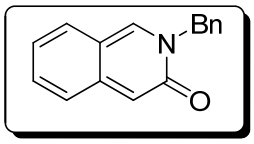
IR (neat, cm^{-1}): 3250 (s), 3050 (m), 2850(s), 2405(m), 2100(s), 1670 (s), 1450(m), 645(m) ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.89 (s, 1 H), 7.95 (s, 1 H), 7.24-7.46 (m, 5 H), 6.92 (s, 1 H), 5.98 (s, 2 H), 5.02 (s, 2 H), 3.24 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 148.2, 133.1, 132.2, 129.3, 128.9, 127.4, 116.1, 112.8, 107.8, 101.9, 82.3, 80.8, 71.4 ; HRMS calcd for$C_{17}H_{13}NO_3$: 279.0895, found 279.0897.

Spectral data for compound 3a :



solid; m.p: 180-184℃; IR (neat, cm^{-1}): 3050 (s), 2940(s), 2860 (m), 2150(s), 1670(s), 1623 (s), 1540(m), 1430(s), 1204 (s), 1053 (s), 840 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 7.34 (d, J = 8.4 Hz, 1H), 7.18-7.22 (m, 2H), 6.85 (t, J = 7.2 Hz, 1H), 6.70 (s, 1H), 3.79 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.9, 142.8, 140.5, 131.3, 124.6, 122.1, 117.3, 109.7, 39.2 ; HRMS calcd for $C_{10}H_9NO$:159.0684, found159.0683.

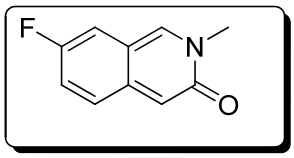
Spectral data for compound 3b :



solid; m.p: 190-195℃; IR (neat, cm^{-1}): 3100 (s), 2840(s), 2450 (m), 2150(s), 1670(s), 1623 (s), 1545(m), 1430(s), 1224 (s), 1053 (s), 780 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (s, 1 H), 7.15-7.3 (m, 8H), 6.83 (s, 1 H), 6.58 (s, 1 H), 5.25 (s, 2H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.1, 143.6, 136.9, 135.3, 125.2,

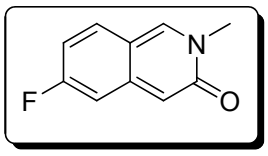
122.9, 118.2, 110.5, 53.8 ; HRMS calcd for $C_{16}H_{13}NO$: 235.0997, found 235.0993.

Spectral data for compound 3c :



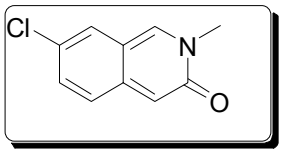
solid; m.p: 78-82 ℃; IR (neat, cm^{-1}): 3050 (s), 2960(s), 2850(s), 2430(m), 2150(s), 1680(s), 1623 (s), 1545(m), 1430(s), 1245 (s), 769(s); ^1H NMR (400 MHz, $CDCl_3$): δ 8.07 (s,1H), 7.29-7.26 (m, 1H), 7.07 (m, 1H), 6.96(m, 1H), 6.76(s, 1H), 3.80 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7, 158.7, 156.2, 140.4, 139.1(d, *J* = 35.2 Hz), 127.7(d *J* = 32 Hz), 124.1, 123.8, 111.0, 108.0(d, j=88 Hz), 39.3;HRMS calcd for $C_{10}H_8FNO$:177.0590, found177.0587.

Spectral data for compound 3d :



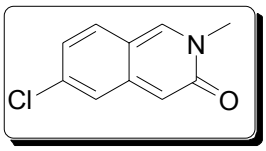
solid; IR (neat, cm^{-1}): 3050 (s), 2980(s), 2840(s), 2430(m), 2150(s), 1670(s), 1623 (s), 1540(m), 1420(s), 1245 (s), 780(s); ^1H NMR (400 MHz, $CDCl_3$): δ 8.14 (s,1H), 7.40 (dd, *J* = 5.6, 1H), 6.83 (dd, *J* = 2, 1H), 6.72(m, 1H), 6.65(s, 1H), 3.80 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.3, 162.7, 160.6, 144.0, 141.0, 130.1(d, *J* = 40 Hz), 114.9, 114.6, 109.0, (d, *J* =32 Hz), 106.2,106.0, 39.2; HRMS calcd for $C_{10}H_8FNO$:177.0590, found177.0592.

Spectral data for compound 3e:



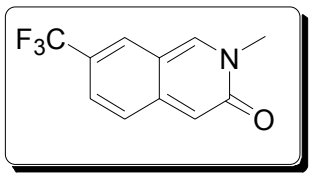
solid; IR (neat, cm^{-1}): 3100 (s), 2840(s), 2450 (m), 2150(s), 1670(s), 1623 (s), 1545(m), 1430(s), 1224 (s), 1053 (s), 840 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.30 (s,1H), 7.18 (d, J = 8.8 Hz, 1H), 7.07(d, J = 8.8 Hz, 1H), 6.69 (s, 1H), 3.78 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.8, 140.9 , 139.6, 132.5, 127.3, 126.6 , 124.8 , 117.1, 110.6, 39.3 ; HRMS calcd for $C_{10}H_8ClNO$: 193.0294, found 193.0291.

Spectral data for compound 3f:



solid; IR (neat, cm^{-1}): 3100 (s), 2840(s), 2420 (m), 2150(s), 1670(s), 1630 (s), 1545(m), 1425(s), 1230 (s), 1060 (s), 840 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 7.31 (d, J = 9.2 Hz, 1H), 7.25(s, 1H), 6.82(d, J = 2 Hz, 1H), 6.60 (s, 1H),3.78 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.2, 143.2, 141.2 , 138.2 129.3, 124.0 , 123.1 (d), 115.6, 109.3, 39.6 ; HRMS calcd for $C_{10}H_8ClNO$: 193.0294, found 193.0298.

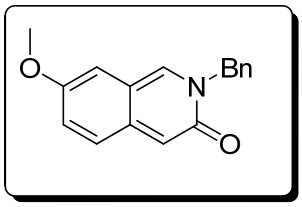
Spectral data for compound 3g:



solid; m.p: 128-130℃ ; IR (neat, cm^{-1}): 3180 (s), 2950 (m), 2430(s), 1710(s), 1623 (s), 1450(s), 1320(s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 8.28 (s, 1H), 7.68(s, 1H), 7.30 (d, J = 9.2Hz, 1H), 7.26(s, 1H), 6.82(dd, J = 1.6 Hz, 1H), 6.62 (s, 1H), 3.80 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.2, 142.8, 142.6, 126.3(d J = 10.2 Hz) , 126.1-126.2(m) 126.0, 124.0 , 115.1, 110.4, 39.4 ; HRMS calcd for

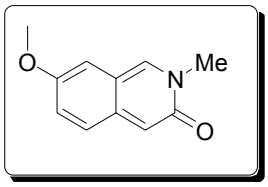
$C_{10}H_8CF_3NO$: 227.0558, found 227.0562.

Spectral data for compound 3h:



solid; m.p: 140-145℃ ; IR (neat, cm^{-1}): 3100 (s), 2840(s), 2450 (m), 2150(s), 1670(s), 1625 (s), 1545(m), 1430(s), 1040 (s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.88 (s, 1 H), 7.25-7.34 (m, 5 H), 7.17 (d, , J = 9.2 Hz, 1H), 6.93 (d, J = 2.4 Hz, 1H), 6.72 (s, 1 H), 6.46 (s, 1 H), 5.26 (s, 2H), 3.74 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 154.6, 140.0, 136.2, 136.0, 128.9, 128.8, 128.6, 128.5, 128.3, 128.2, 127.7, 126.5, 117,9, 111.5, 101.0, 55.1, 52.9 ; HRMS calcd for $C_{17}H_{15}NO_2$: 265.1103, found265.1105.

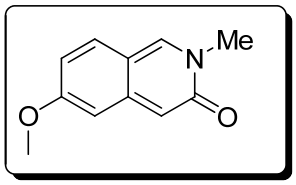
Spectral data for compound 3i :



solid; m.p: 130-134 ℃; IR (neat, cm^{-1}): 3050 (s), 2940(s), 2840(s), 2450 (m), 2150(s), 1720(s), 1625 (s), 1545(m), 1040 (s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.897 (s, 1 H), 7.26 (s, 1 H), 7.23 (s,1H), 6.57-6.54 (m, 2H), 6.42 (d, J=2 Hz , 1 H), 3.82 (s, 3H), 3.73(s, 3 H), ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.6, 161.0, 144.8, 139.6, 129.0, 118.2, 114.2, 107.5, 99.2, 55.3, 38.8; HRMS calcd for $C_{11}H_{11}NO_2$:189.0790, found189.0792.

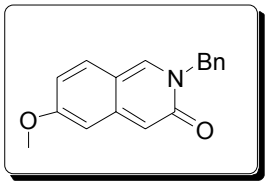
.

Spectral data for compound 3j :



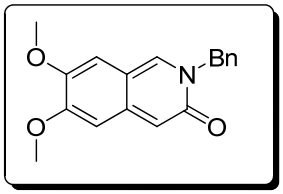
solid; m.p: 133-136 ℃; IR (neat, cm^{-1}): 3080 (s), 2950(s), 2840(s), 2450 (m), 2150(s), 1720(s), 1625 (s), 1545(m), 1040 (s), 780 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.98 (s, 1 H), 7.25 (d, J = 9.2 Hz, 1H), 6.56 (m, 2 H), 6.41 (d, J = 2 Hz, 1H), 3.83 (s, 3H), 3.74 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.7, 161.0, 144.8, 139.9, 129.0, 118.2, 114.2, 107.5, 99.2, 55.3, 38.8 ; HRMS calcd for $C_{11}H_{11}NO_2$:189.0790, found189.0793.

Spectral data for compound 3k :



solid; m.p: 155-159 ℃; IR (neat, cm^{-1}): 3100(s), 2840(s), 2450 (m), 2150(s), 1670(s), 1625 (s), 1545(m), 1420(s), 1250 (s), 780 (m; ^1H NMR (400 MHz, $CDCl_3$): δ 7.91 (s, 1 H), 7.34-7.26 (m, 5 H), 7.17 (d, J = 9.2 Hz, 1H), 6.93 (d, J = 2.4 Hz, 1H), 6.72 (s, 1 H), 6.46 (s, 1 H), 5.26 (s, 2H), 3.74 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 154.6, 140.0, 136.2, 136.0, 128.9, 128.8, 128.6, 128.5, 128.3, 128.2, 127.7, 126.5, 117,9, 111.5, 101.0, 55.1, 52.9 ; HRMS calcd for $C_{17}H_{15}NO_2$: 265.1103, found265.1101.

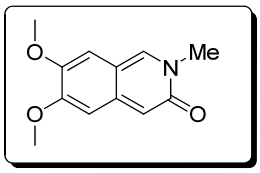
Spectral data for compound 3l :



solid; m.p: 143-146 ℃; IR (neat, cm^{-1}): 3150 (s), 2850(s), 2140(s), 1720(s),

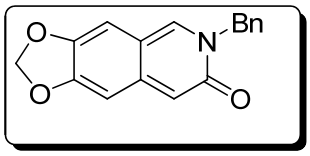
1625 (s), 1545(m), 1280(s), 1040 (s), 780 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (s, 1 H), 7.28-7.33 (m, 5 H), 6.60 (d, J = 4.26 Hz, 1H), 6.45 (d, J = 12.3 Hz, 2 H), 5.32 (s, 2H), 3.89 (s, 3 H), 3.76 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4, 155.1, 147.9, 141.25, 136.3, 135.6, 128.9, 128.5, 128.3, 114.3, 108.8, 102.8, 100.9, 56.1, 55.7, 52.5 ; HRMS calcd for $C_{16}H_{20}O_2$: 244.1463, found 244.1467.

Spectral data for compound 3m :



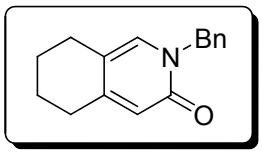
solid; m.p: 167-171 ℃; IR (neat, cm^{-1}): 3038 (s), 2989 (m), 1603 (s), 1204 (s), 1093 (s), 776 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1 H), 6.51 (d, J = 3.2 Hz, 2 H,), 6.40 (s, 1H), 3.90 (s, 3 H), 3.82 (s, 3 H), 3.70 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.6, 154.8, 147.8, 147.2, 141.2, 136.7, 113.9, 108.2, 102.6, 100.9, 56.0, 55.7, 38.6 ; HRMS calcd for $C_{12}H_{13}NO_3$: 219.0895, found219.0893.

Spectral data for compound 3n :



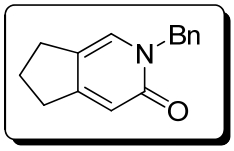
solid; m.p: 210-215 ℃; IR (neat, cm^{-1}): 3050 (s), 2840(s), 2450 (m), 2150(s), 1680(s), 1625 (s), 1535(m), 1425(m), 1050 (s), 680 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (s, 1 H), 7.27-7.31 (m, 5 H), 6.60 (s, 1 H), 5.89 (s, 2 H), 5.29 (s, 2 H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2, 152.6, 145.6, 152.5, 136.2, 135.9, 128.9, 128.5, 128.2, 128.1, 114.8, 110.3, 101.2, 100.2, 98.6, 52.4 ; HRMS calcd for $C_{17}H_{13}NO_3$:279.0895, found 279.0897.

Spectral data for compound 5a :



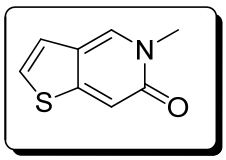
solid; m.p: 120-124 ℃; IR (neat, cm^{-1}): 3138 (s), 2859 (m), 1683 (s), 1425(s), 1375(m), 1254 (s), 1093 (s), 760 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.18-7.27 (m, 5 H), 6.92 (s, 1H), 6.29 (s, 1H), 5.04 (s, 2H), 2.54 (d, *J* = 6 Hz, 2 H), 2.38 (d, *J* = 5.6 Hz, 2 H), 1.60-1.63 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.7, 151.7, 136.7, 134.1, 128.5, 127.8, 127.5, 117.9, 116.4, 51.1, 28.6,25.1, 22.5, 21.9 ; HRMS calcd for $C_{16}H_{17}NO$: 229.1310, found 229.1313.

Spectral data for compound 5b :



solid; m.p: 98-102 ℃; IR (neat, cm^{-1}): 3058 (s), 2809 (m), 1603 (s), 1540(s), 1450(b), 1224 (s), 1093 (s), 830 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.27-7.25 (m, 3 H), 7.21 (d, *J* = 1.6 Hz, 1H,), 7.16-7.14 (m, 2H), 7.03 (s, 1H), 5.08 (s, 2H), 2.86 (m, 2H), 2.78 (t, *J* = 7.6 Hz, 2H), 1.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0, 150.9, 142.6, 133.5, 131.3, 129.1, 127.8, 118.6, 63.5, 31.3, 30.2, 23.7 ; HRMS calcd for $C_{15}H_{15}NO$: 225.1154, found 225.1152.

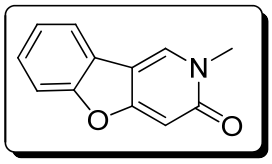
Spectral data for compound 5c :



solid; m.p: 92-94 ℃; IR (neat, cm^{-1}): 3080 (s), 2949 (m), 2450(s), 1683 (s), 1304 (s), 1093 (s), 706 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.82 (s, 1H), 7.44 (d, *J* = 5.6

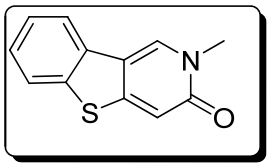
Hz, 1H), 6.82 (d, J = 5.2 Hz, 1H), 6.75 (s, 1H), 3.64 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.5, 152.4, 136.7, 132.2, 121.7, 119.0, 109.4, 38.8 ; HRMS calcd for C_7H_8NOS: 165.0248, found 165.0246.

Spectral data for compound 5d :



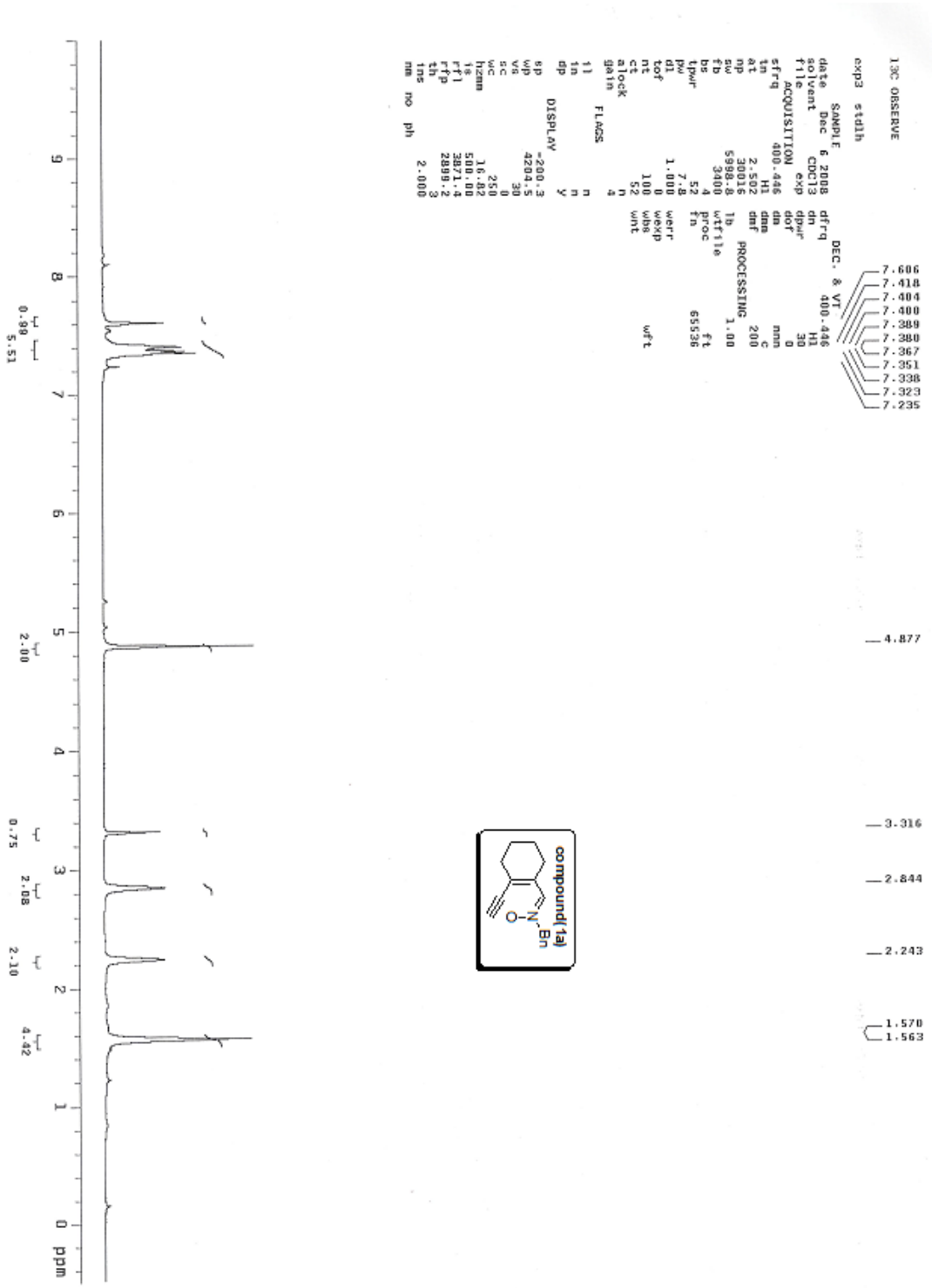
solid; m.p: 210-214 ℃; IR (neat, cm^{-1}): 3100(s), 2940(s), 2850(m), 1673(s), 1540(m), 1240 (s), 1093 (s), 760 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 7.6 Hz, 1H), 7.58 (s, 1H), 7.54 (m, 1H), 7.36 (d, J = 8.4 Hz, 1H), 7.27 (m, 1H), 6.93 (s, 1H), 3.66 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.7, 159.7, 141.6, 140.2, 131.6, 123.2, 121.6, 119.7, 112.0, 107.4, 38.8; HRMS calcd for $C_{12}H_9NO_2$: 199.0633, found 199.0635.

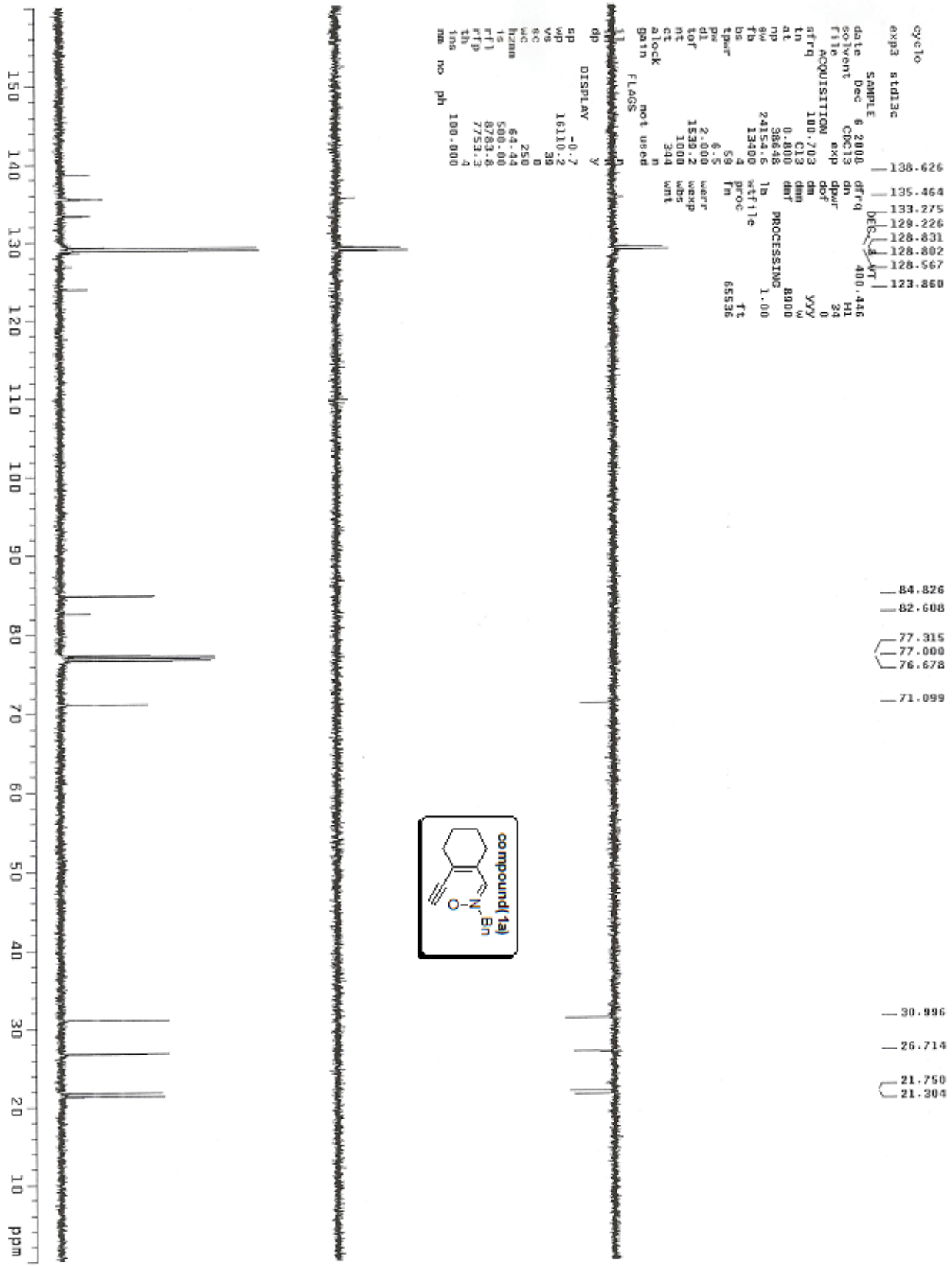
Spectral data for compound 5e :



solid; m.p: 235-240℃ ; IR (neat, cm^{-1}): 3100(s), 2930 (m), 2780(s), 1668(s), 1540(m),1450(m), 1320(s), 1240 (s), 1053 (s), 705 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (m, 1H), 7.70 (s, 1H), 7.62 (d, J = 8 Hz, 1H), 7.46 (m, 1H), 7.37 (m, 1H), 7.15 (s, 1H), 3.68 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9, 149.3, 143.2, 132.4, 131.3, 130.5, 125.0, 123.8, 117.7, 109.3, 39.7; HRMS calcd for $C_{12}H_9NOS$: 215.0405, found 215.0403.

(3) NMR spectra- for key compounds:

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date Dec 6 2008
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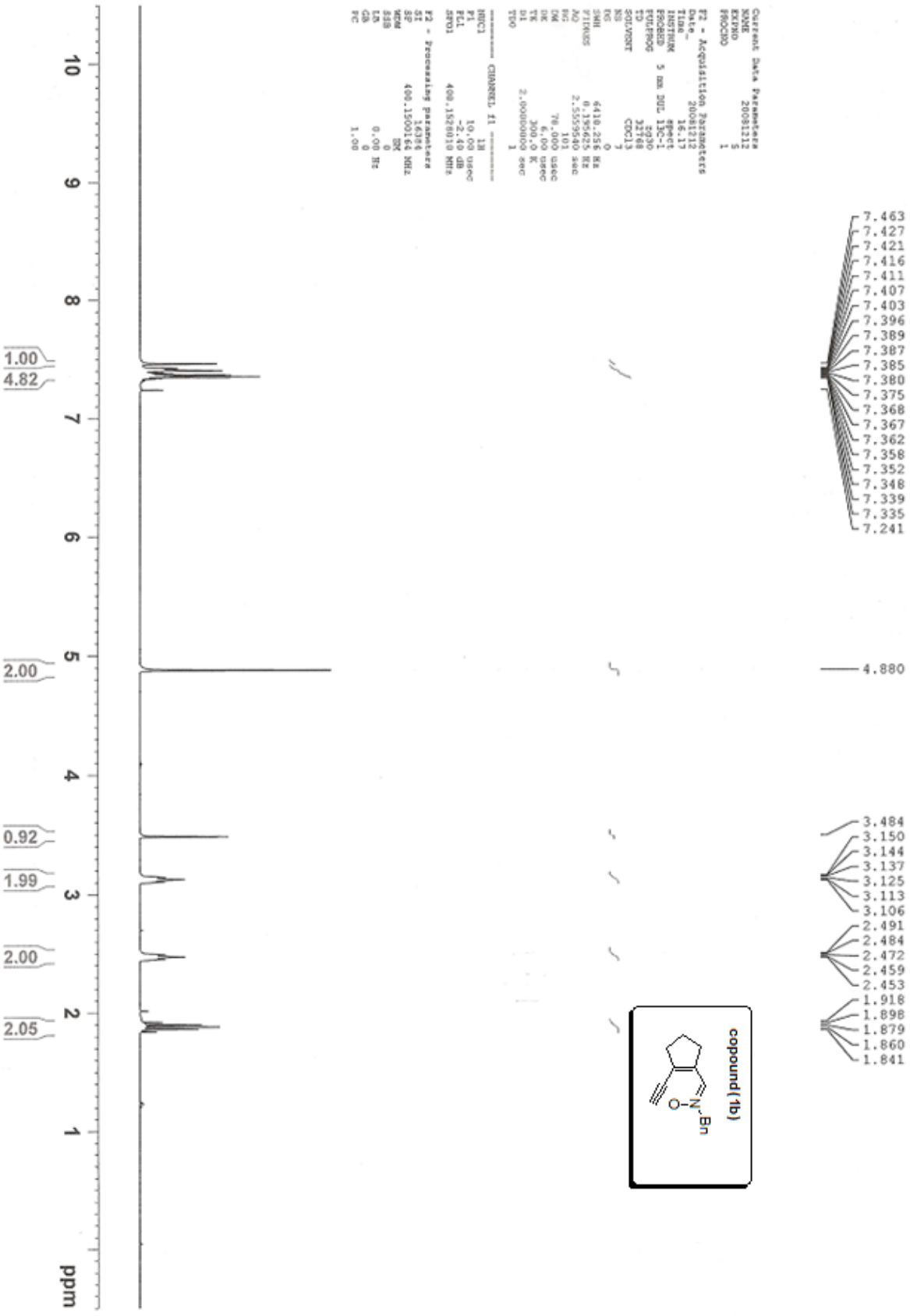
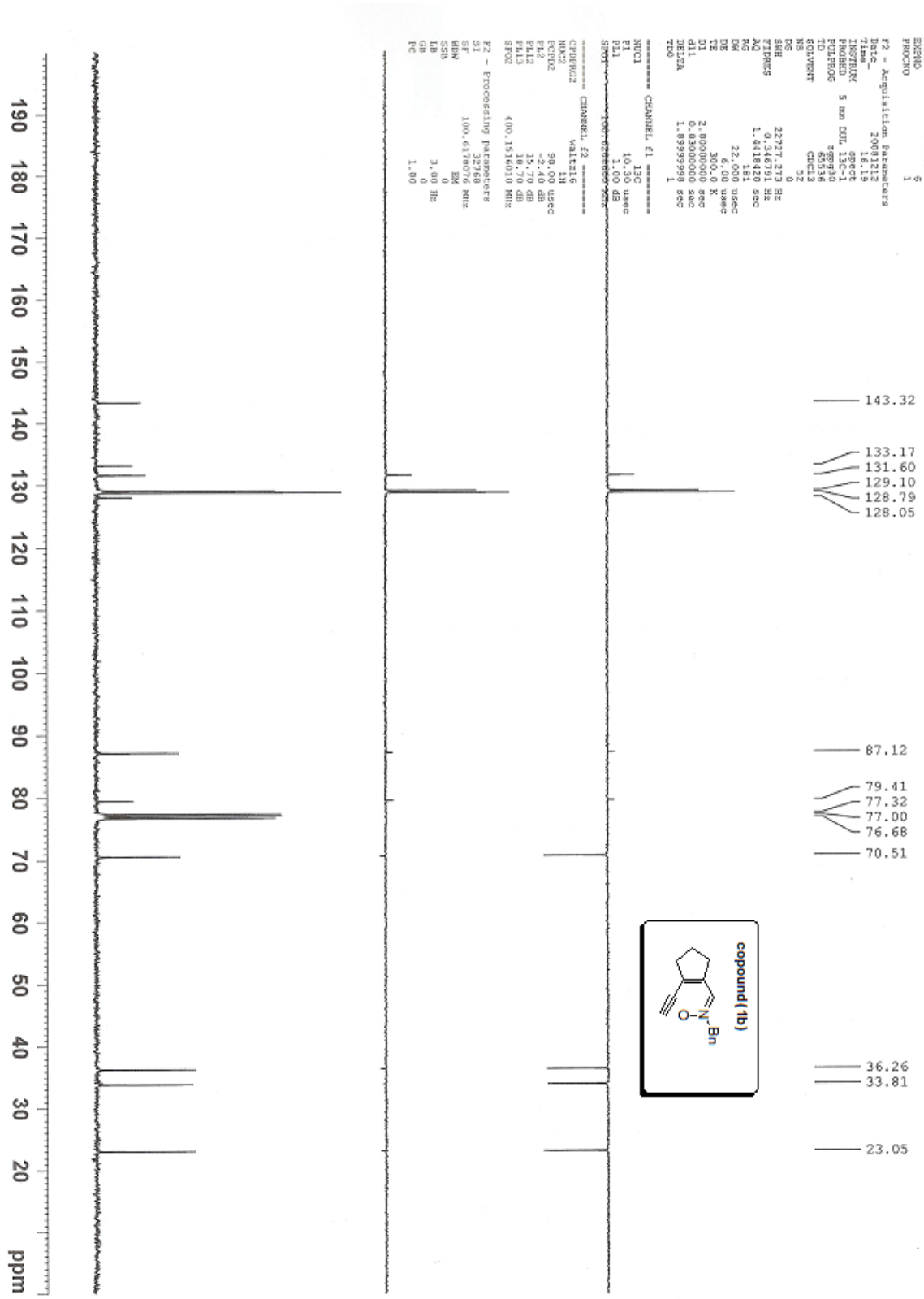
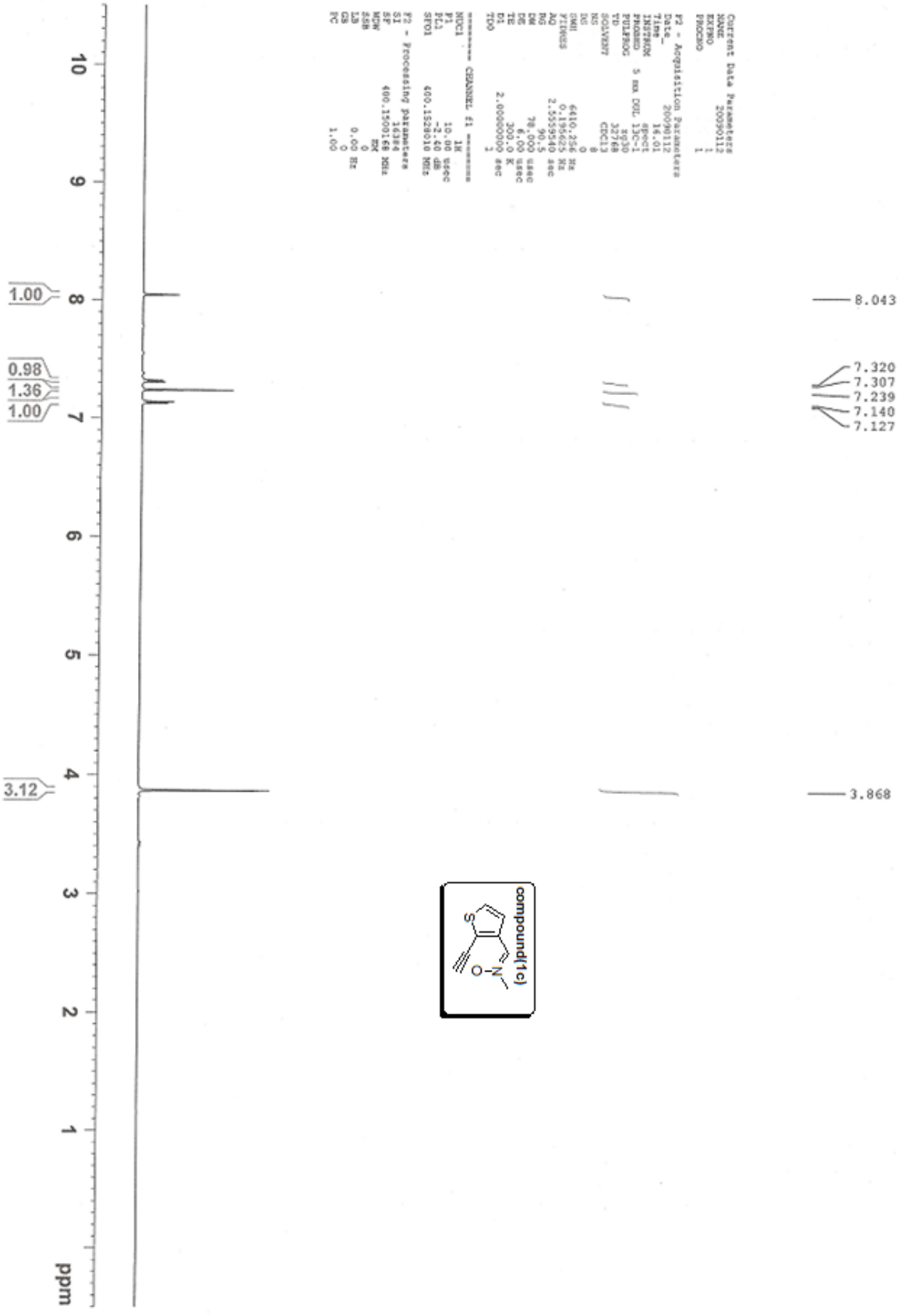
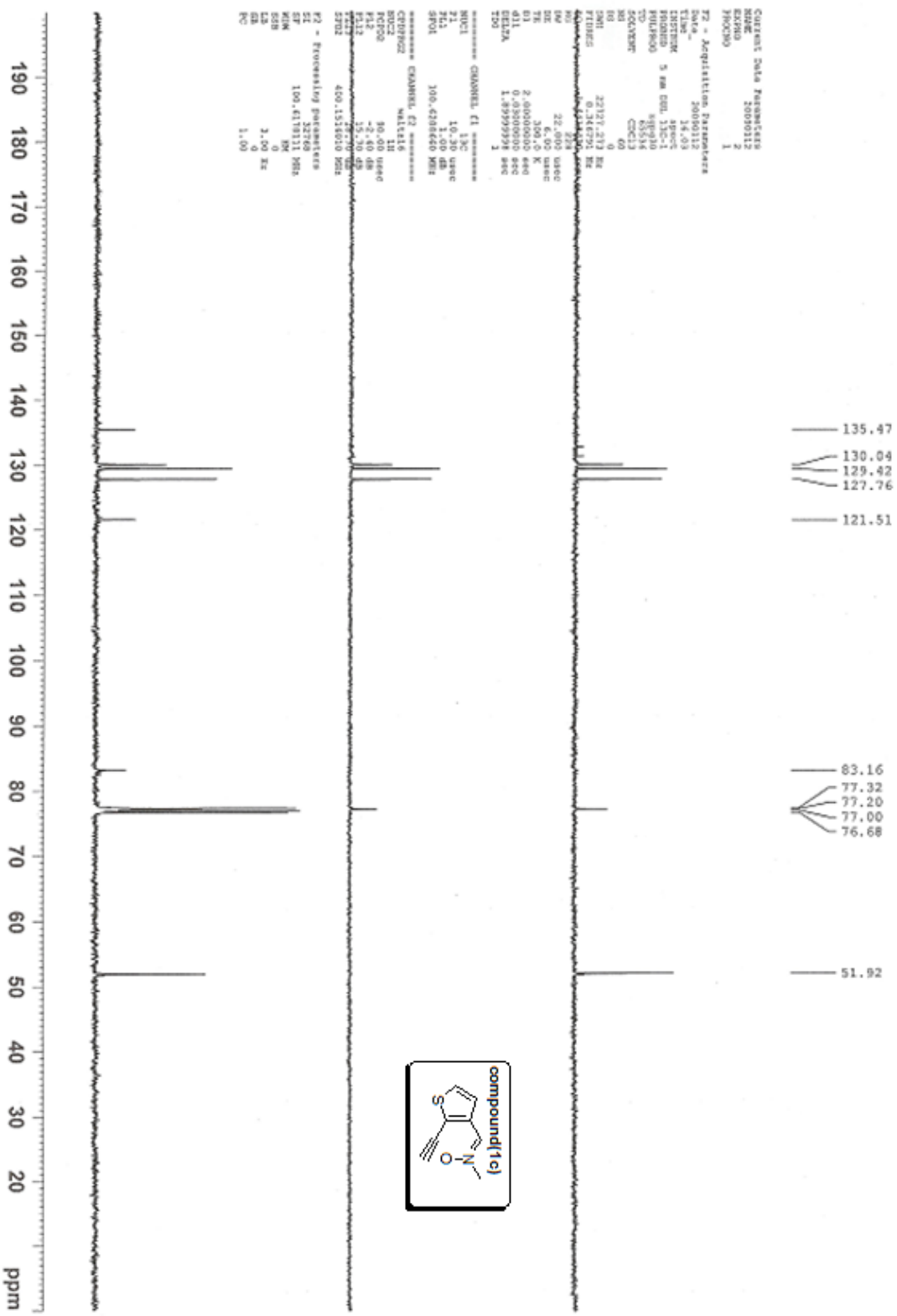
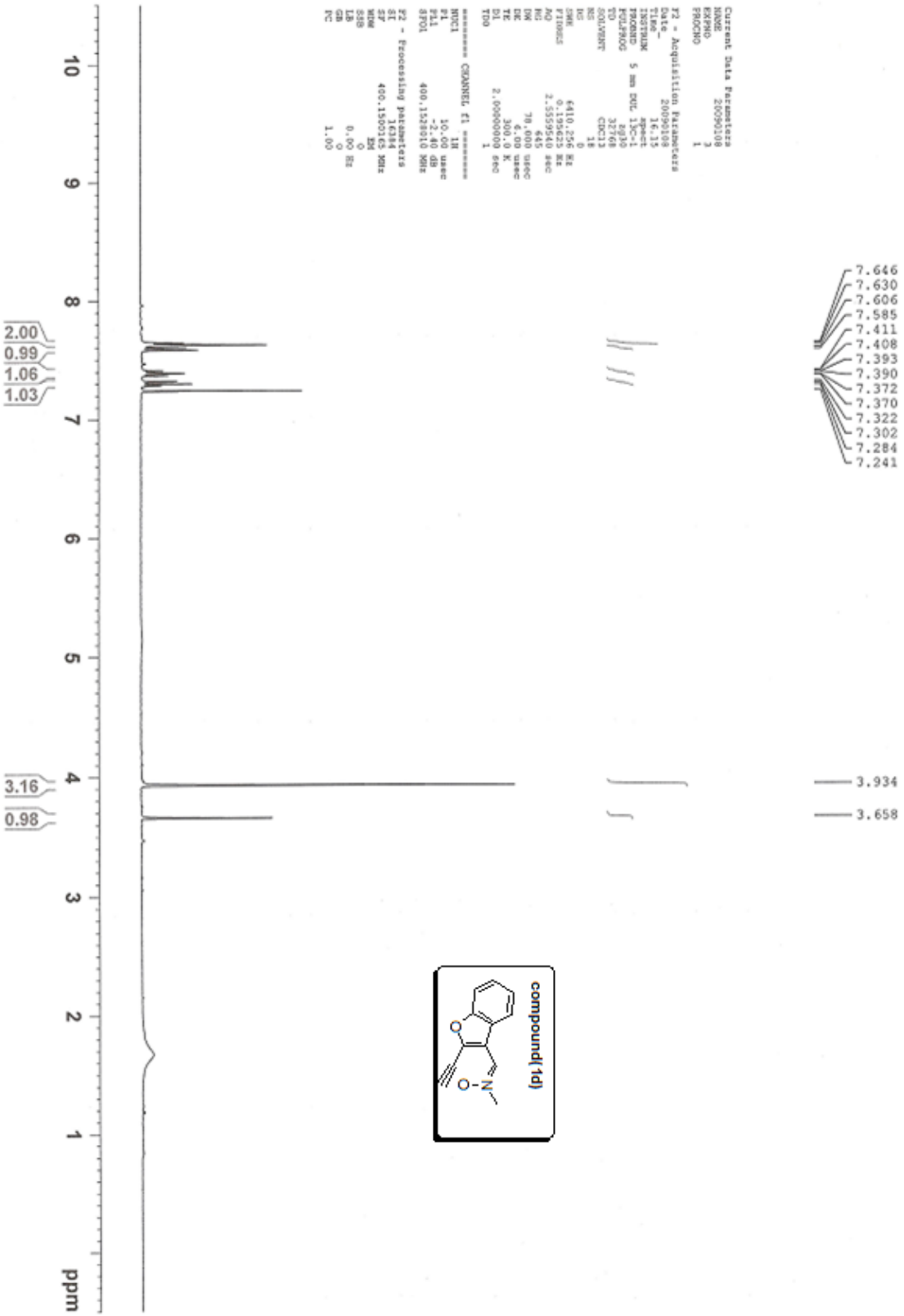
sfrq 100.703 dm
tn C13 dmm
at 0.800 dmf
np 38648
sw 24154.6 lb
fb 13400 wtfile
bs 4 proc
tpwr 59 fn
pw 6.5
d1 2.000 werr
tof 1539.2 wexp
nt 1000 wbs
ct 344 wnt
alock n
gain
FLAGS not used
DISPLAY
sp -0.7
wp 16110.2
vs 39
sc 0
wc 250
hzmm 64.44
is 500.00
rfl 8783.8
rfp 7753.3
th 4
ins
nm no ph 100.000
dfrq 400.446
dn H1
dpwr 34
dcf 0
yyy
w 8000
PROCESSING
lb 1.00
ft
65536
DEPT
138.626
135.464
133.275
129.226
128.831
128.802
128.567
123.860
84.826
82.608
77.315
77.000
76.678
71.099
30.996
26.714
21.750
21.304
compound(1a)
O
N
Bn
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

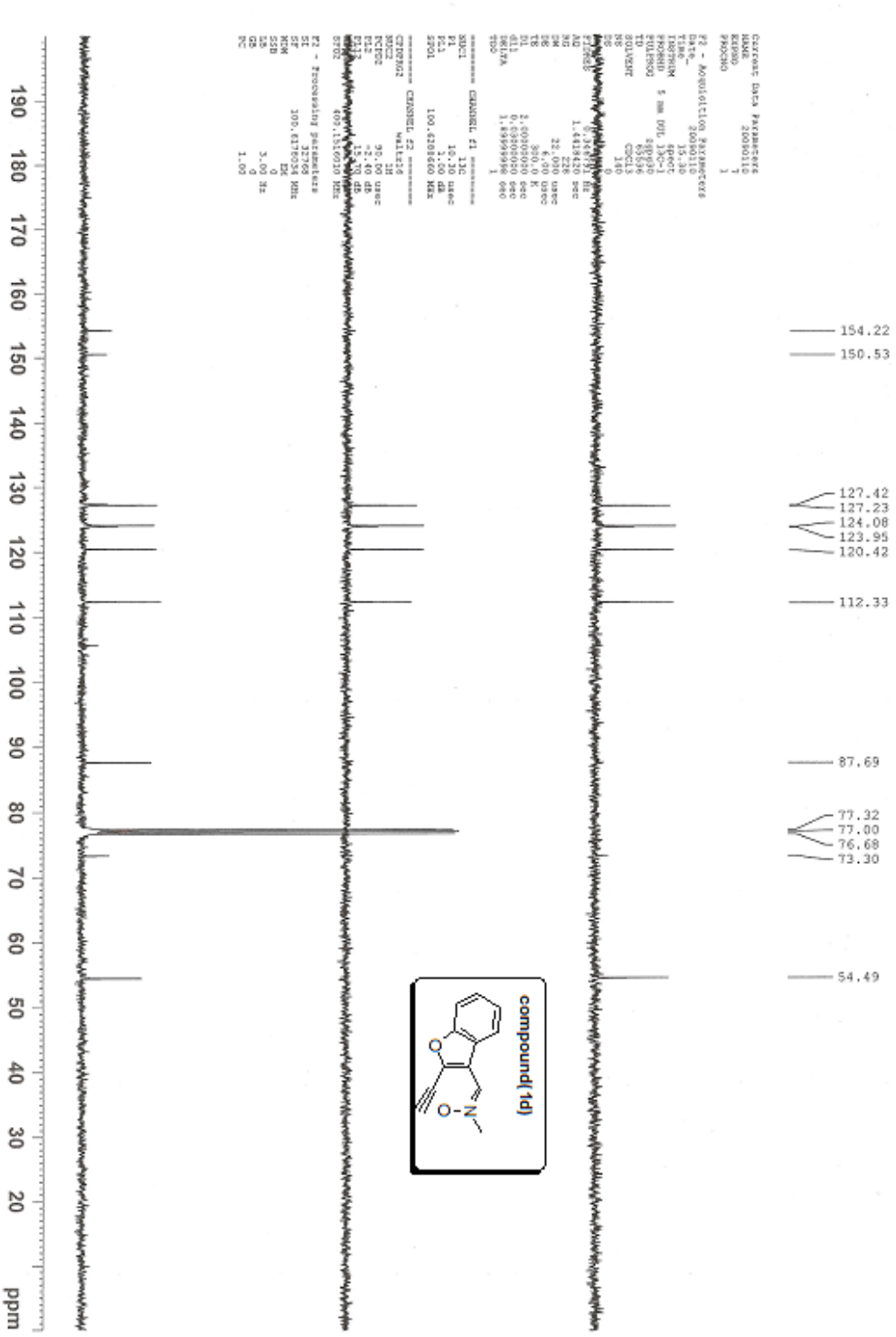

7.463
7.427
7.421
7.416
7.411
7.407
7.403
7.396
7.389
7.387
7.385
7.380
7.375
7.368
7.367
7.362
7.358
7.352
7.348
7.339
7.335
7.241
4.880
3.484
3.150
3.144
3.137
3.125
3.113
3.106
2.491
2.484
2.472
2.459
2.453
1.918
1.898
1.879
1.860
1.841
1.00
4.82
2.00
0.92
1.99
2.00
2.05
ppm
copound(1b)
Bn

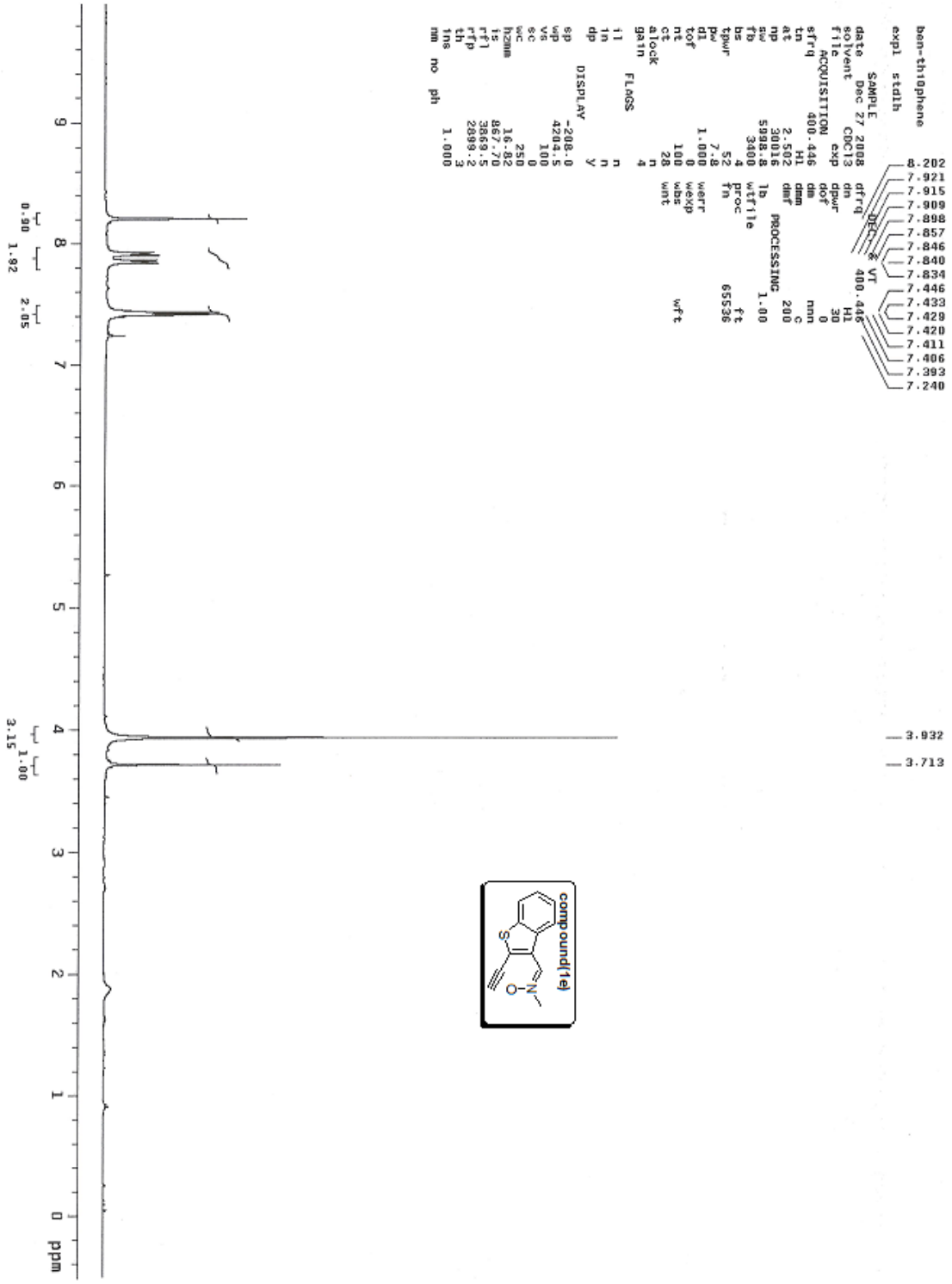

143.32
133.17
131.60
129.10
128.79
128.05
87.12
79.41
77.32
77.00
76.68
70.51
36.26
33.81
23.05
copound(1b)
O
N
Bn
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

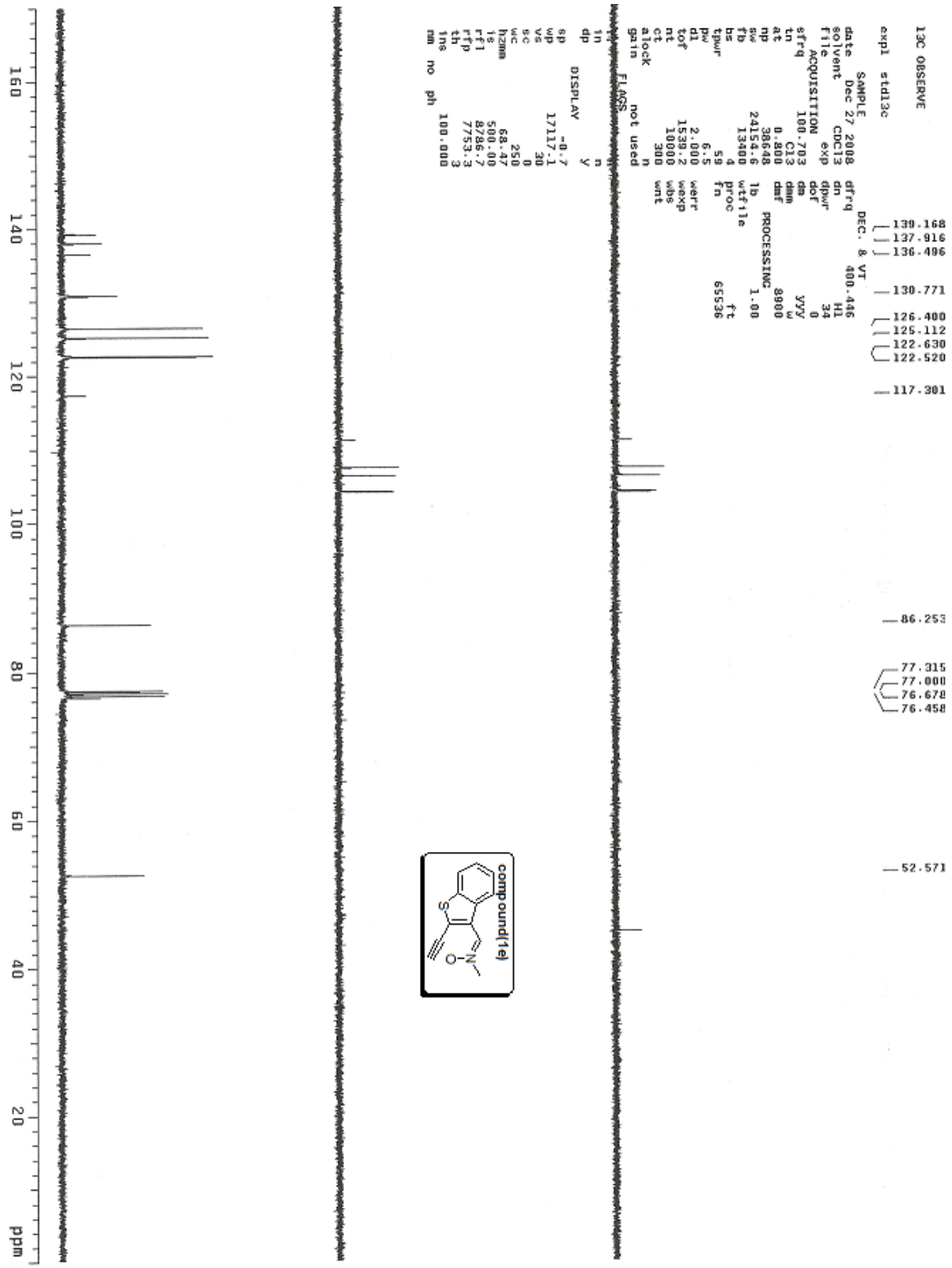

1.00
0.98
1.36
1.00
3.12
8.043
7.320
7.307
7.239
7.140
7.127
3.868
ppm
compound(1c)

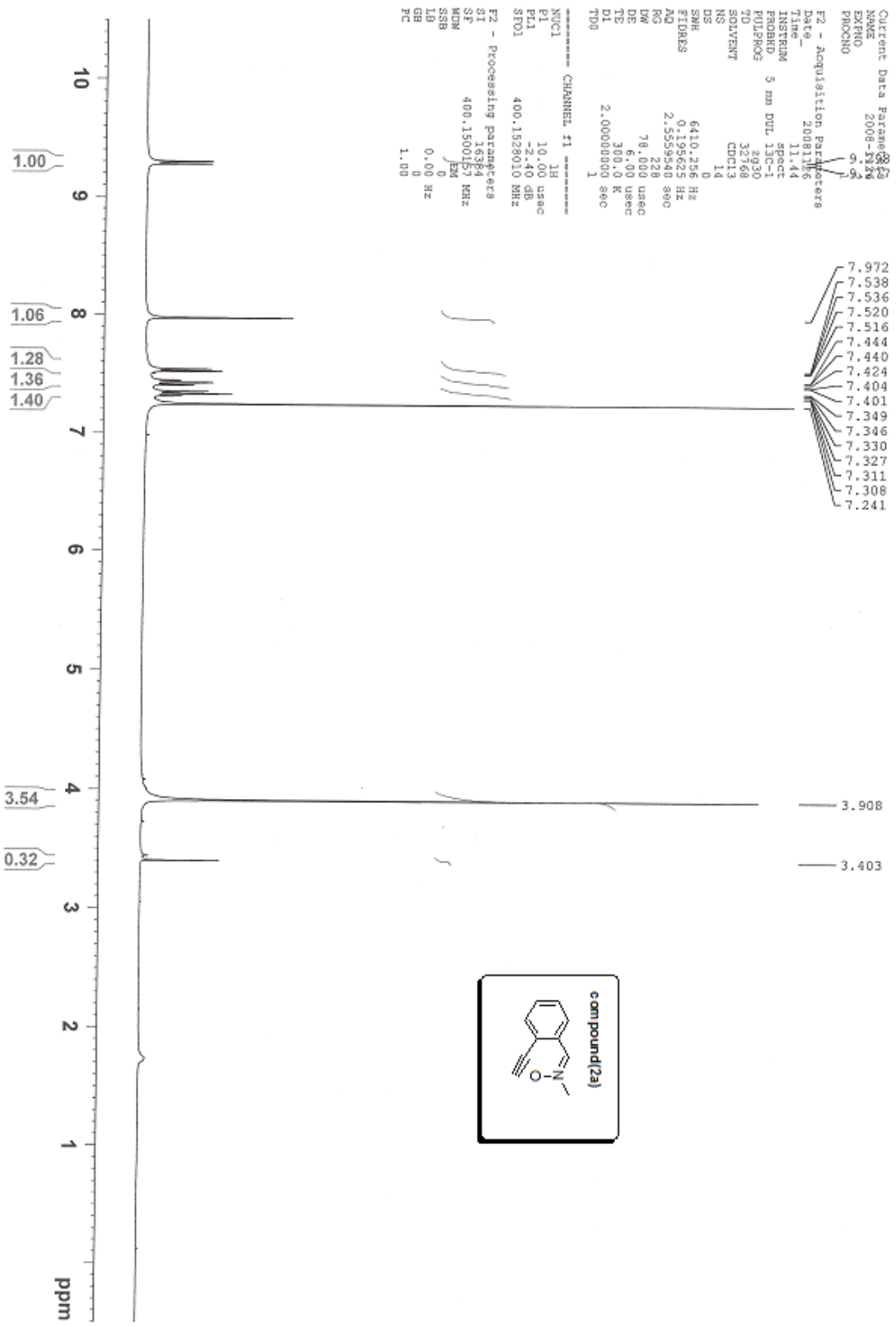

135.47
130.04
129.42
127.76
121.51
83.16
77.32
77.20
77.00
76.68
51.92
compound(1c)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

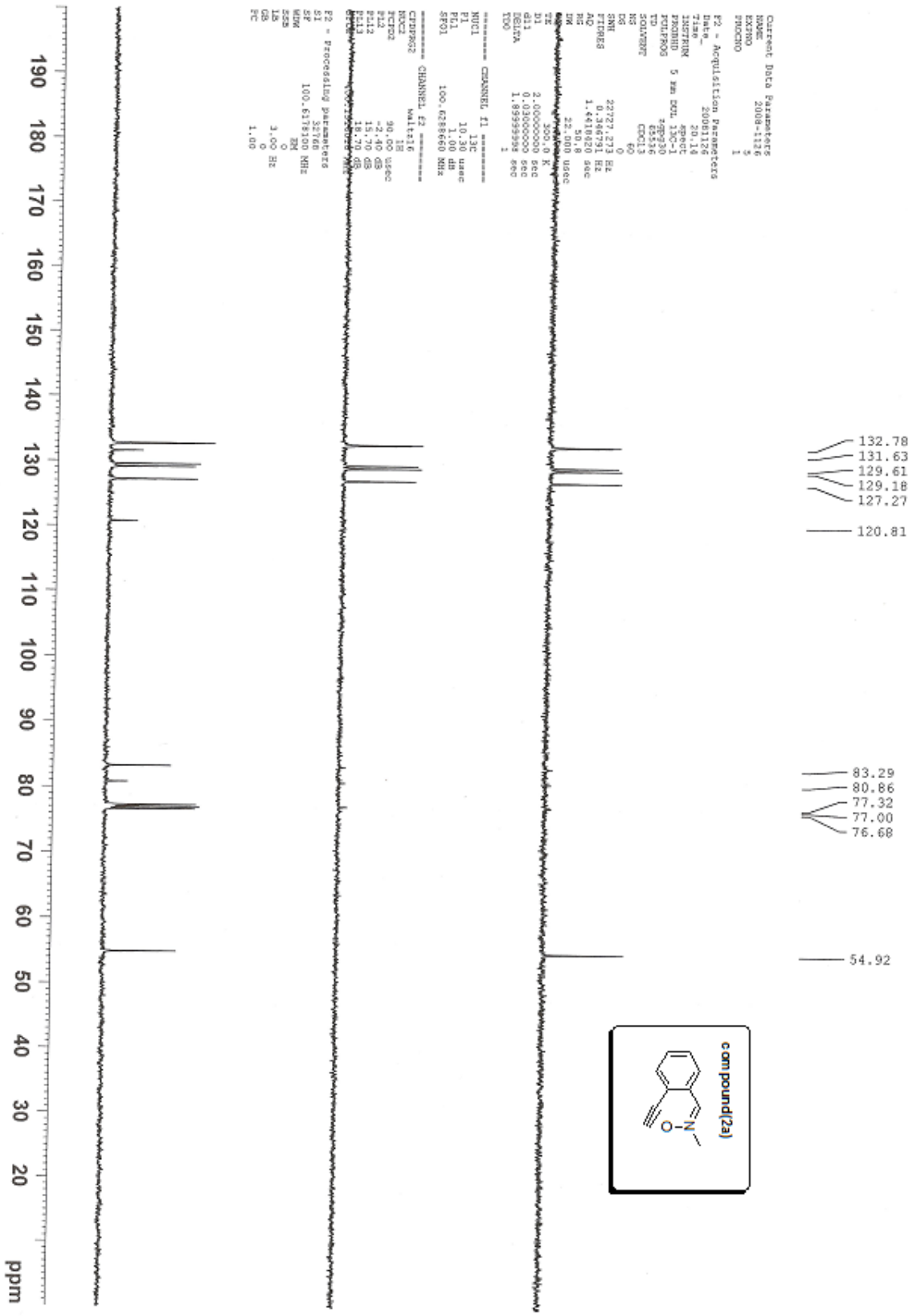

7.646
7.630
7.606
7.585
7.411
7.408
7.393
7.390
7.372
7.370
7.322
7.302
7.284
7.241
3.934
3.658
2.00
0.99
1.06
1.03
3.16
0.98
ppm
compound (1d)

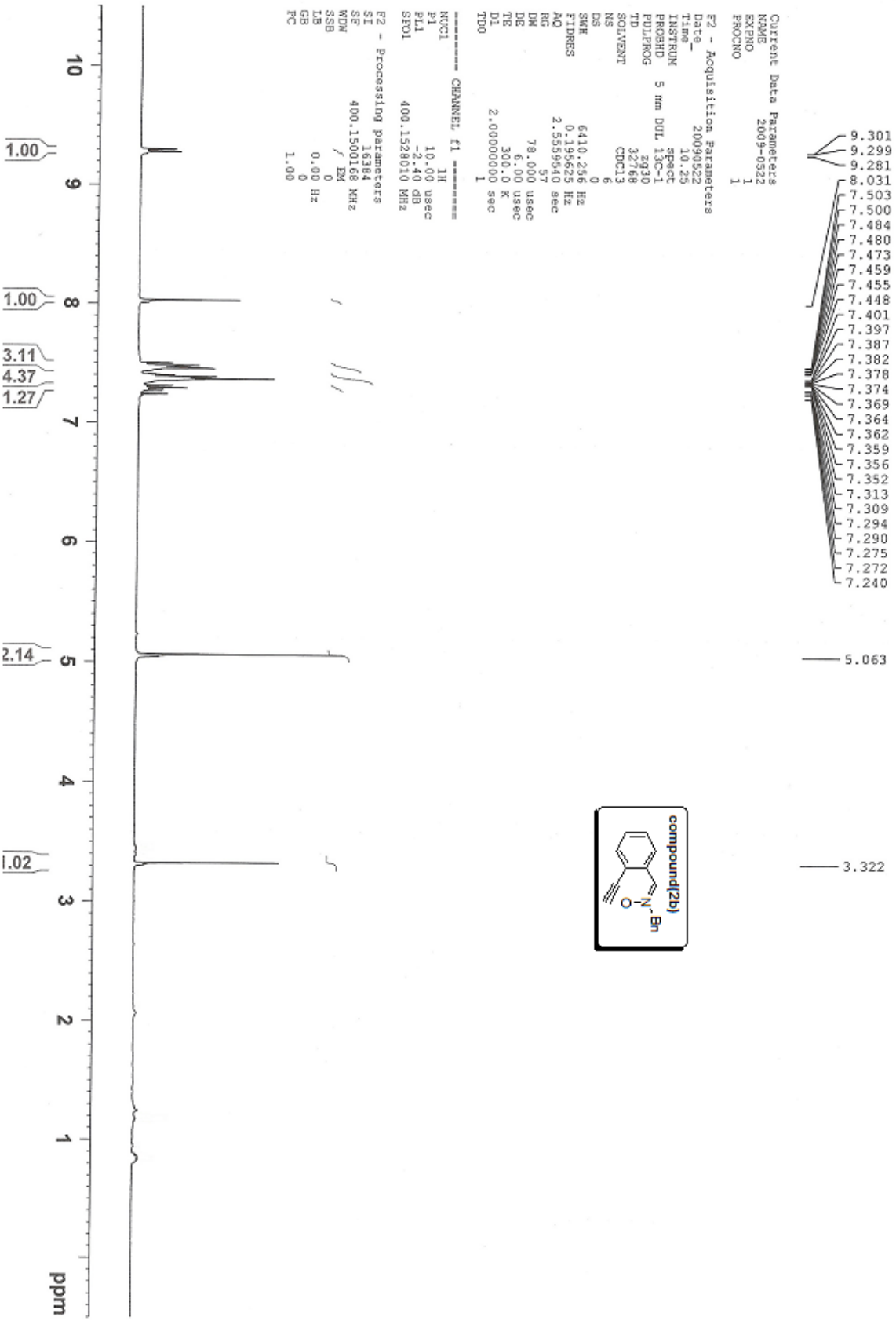

154.22
150.53
127.42
127.23
124.08
123.95
120.42
112.33
87.69
77.32
77.00
76.68
73.30
54.49
compound (1d)
ppm

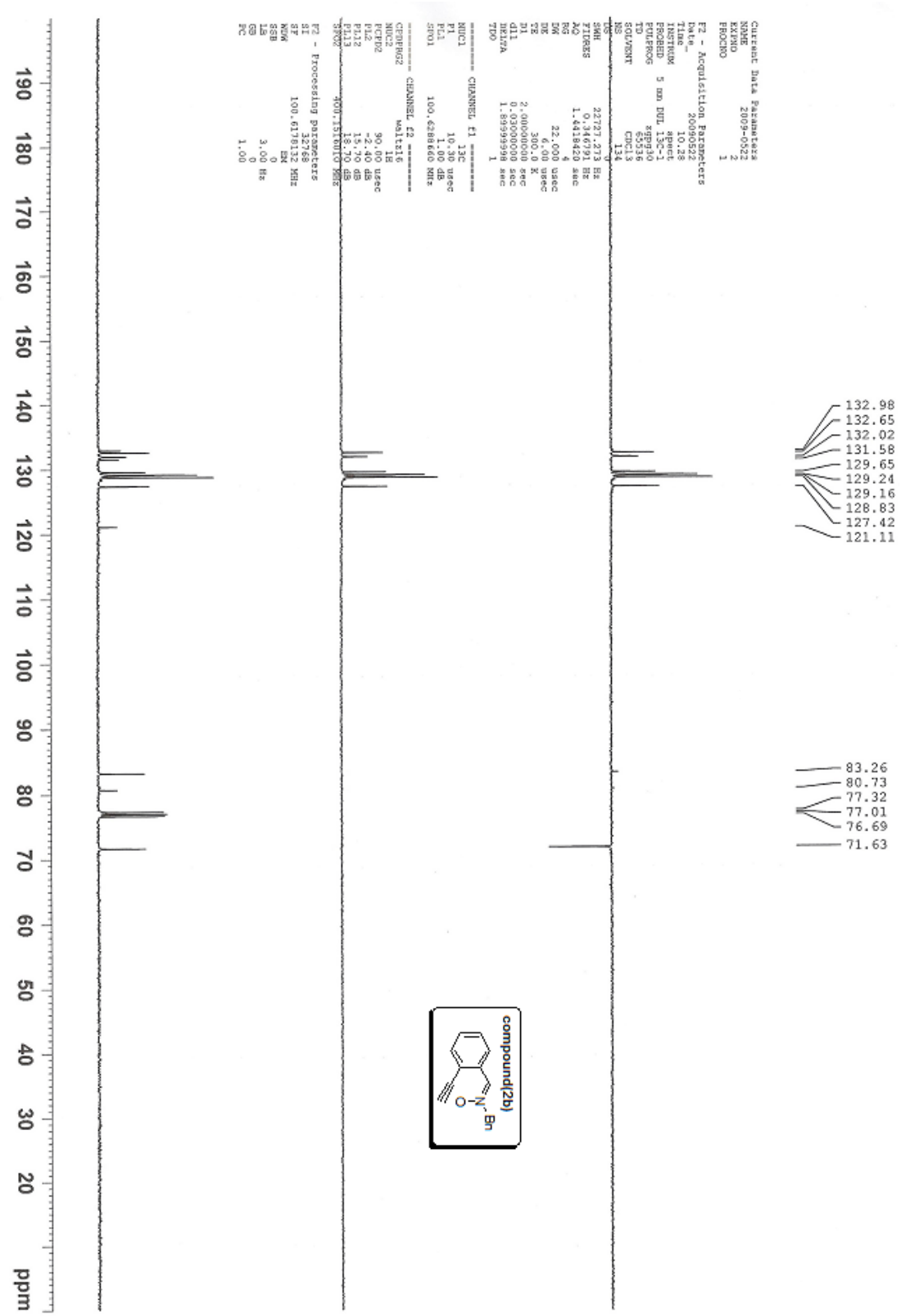

ben-thiophene
expl stdih
SAMPLE
date Dec 27 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446 dm
tn H1 dmm
at 2.502 dmf
np 30016 PROCESSING
sw 5998.8 lb 1.00
fb 3400 wtfile
bs 4 proc ft
tpwr 52 fn 65536
pw 7.8
d1 1.000 werr
0 wexp
tof 0 wbs
nt 100 wnt wft
ct 28
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -208.0
wp 4204.5
vs 100
sc 0
wc 250
hzcm 16.82
is 867.70
rfl 3869.5
rfp 2899.2
th 3
ins 1.000
nm no ph
DEC
VT 400.446 H1
8.202
7.921
7.915
7.909
7.898
7.857
7.846
7.840
7.834
7.446
7.433
7.429
7.420
7.411
7.406
7.393
7.240
3.932
3.713
compound(1e)
0.90
1.92
2.05
3.15
1.00
ppm

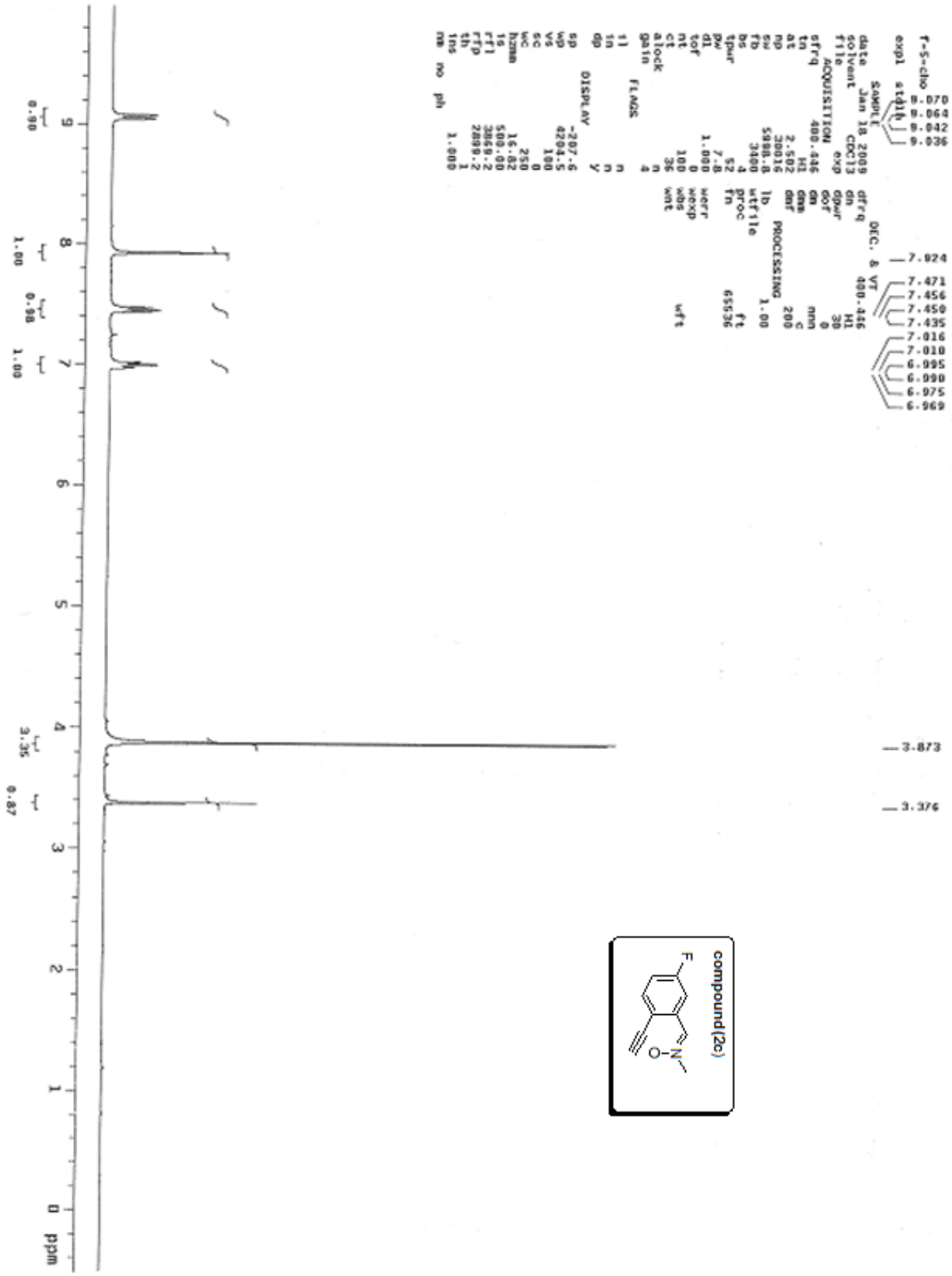

13C OBSERVE
expl std13c
SAMPLE
date Dec 27 2008
solvent CDCl3
file
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 300
alock n
gain not used
FLAGS
in n
dp y
DISPLAY
sp -8.7
wp 17117.1
vs 30
sc 0
wc 250
hzmm 68.47
is 500.00
rfl 8786.7
rfp 7753.3
th 3
ins 1
nm no ph 100.000
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
139.168
137.916
136.496
130.771
126.400
125.112
122.630
122.520
117.301
86.253
77.315
77.000
76.678
76.458
52.571
compound(1e)
160
140
120
100
80
60
40
20
ppm

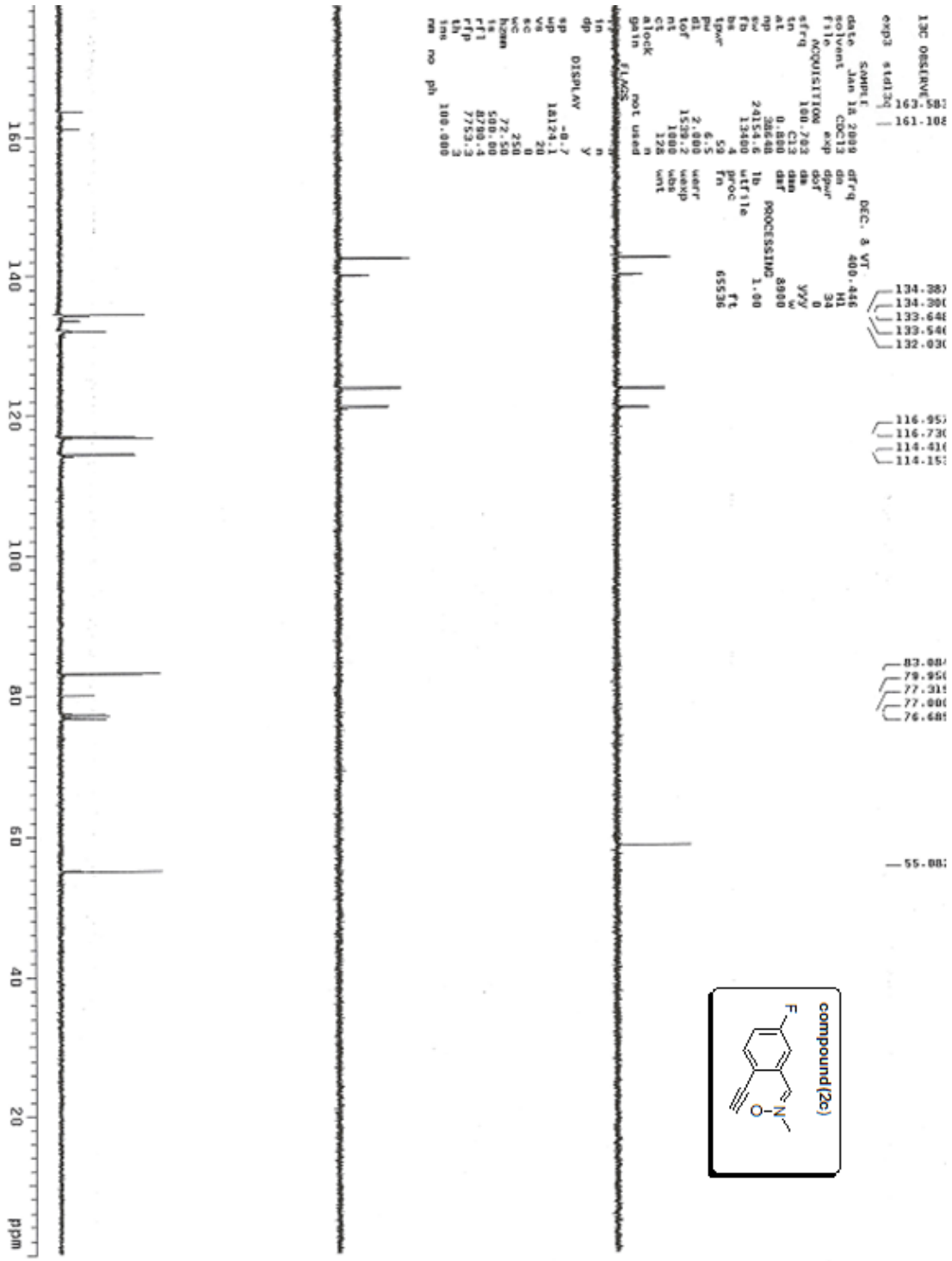

1.00
1.06
1.28
1.36
1.40
3.54
0.32
7.972
7.538
7.536
7.520
7.516
7.444
7.440
7.424
7.404
7.401
7.349
7.346
7.330
7.327
7.311
7.308
7.241
3.908
3.403
ppm
compound(2a)

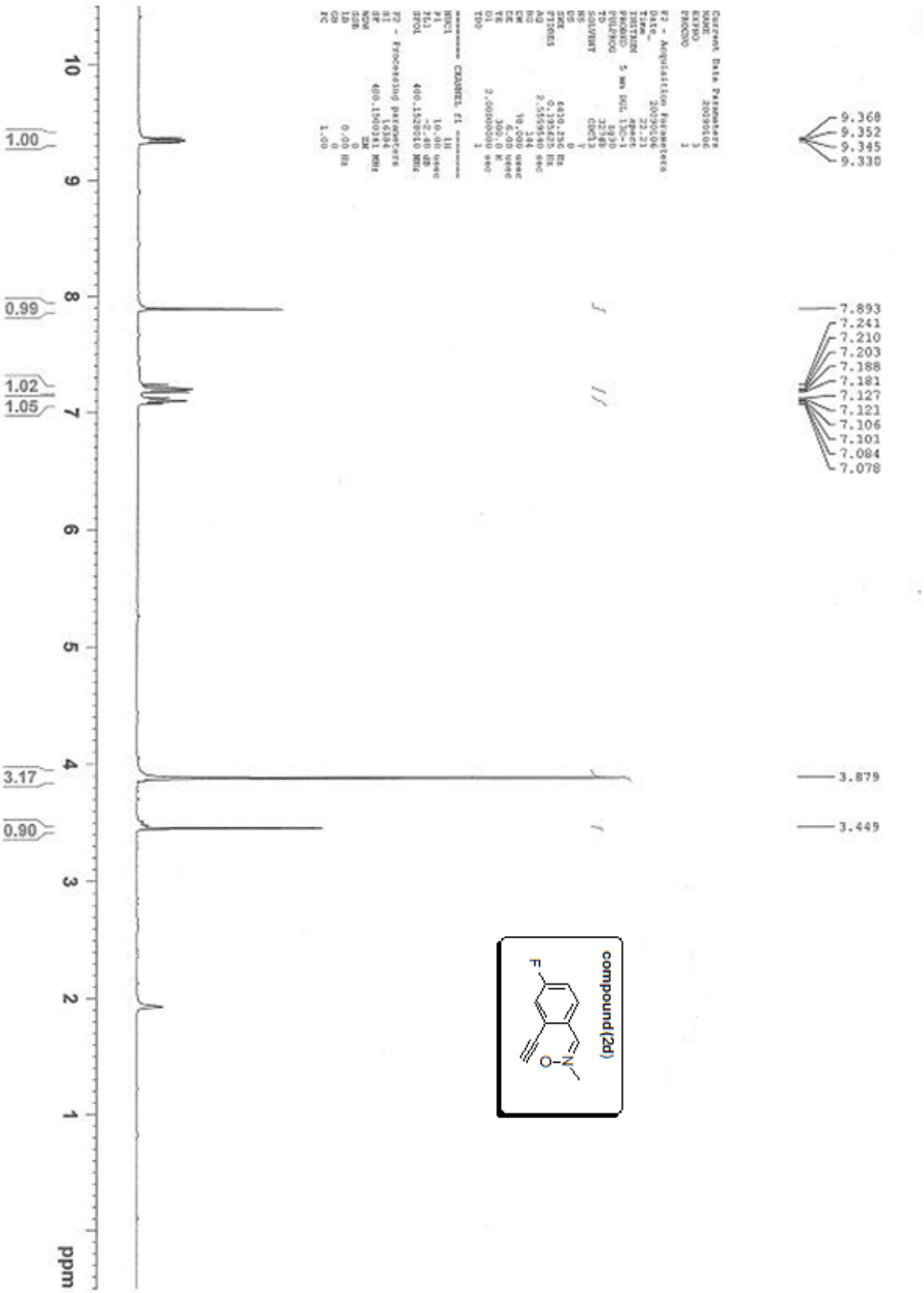

132.78
131.63
129.61
129.18
127.27
120.81
83.29
80.86
77.32
77.00
76.68
54.92
compound(2a)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

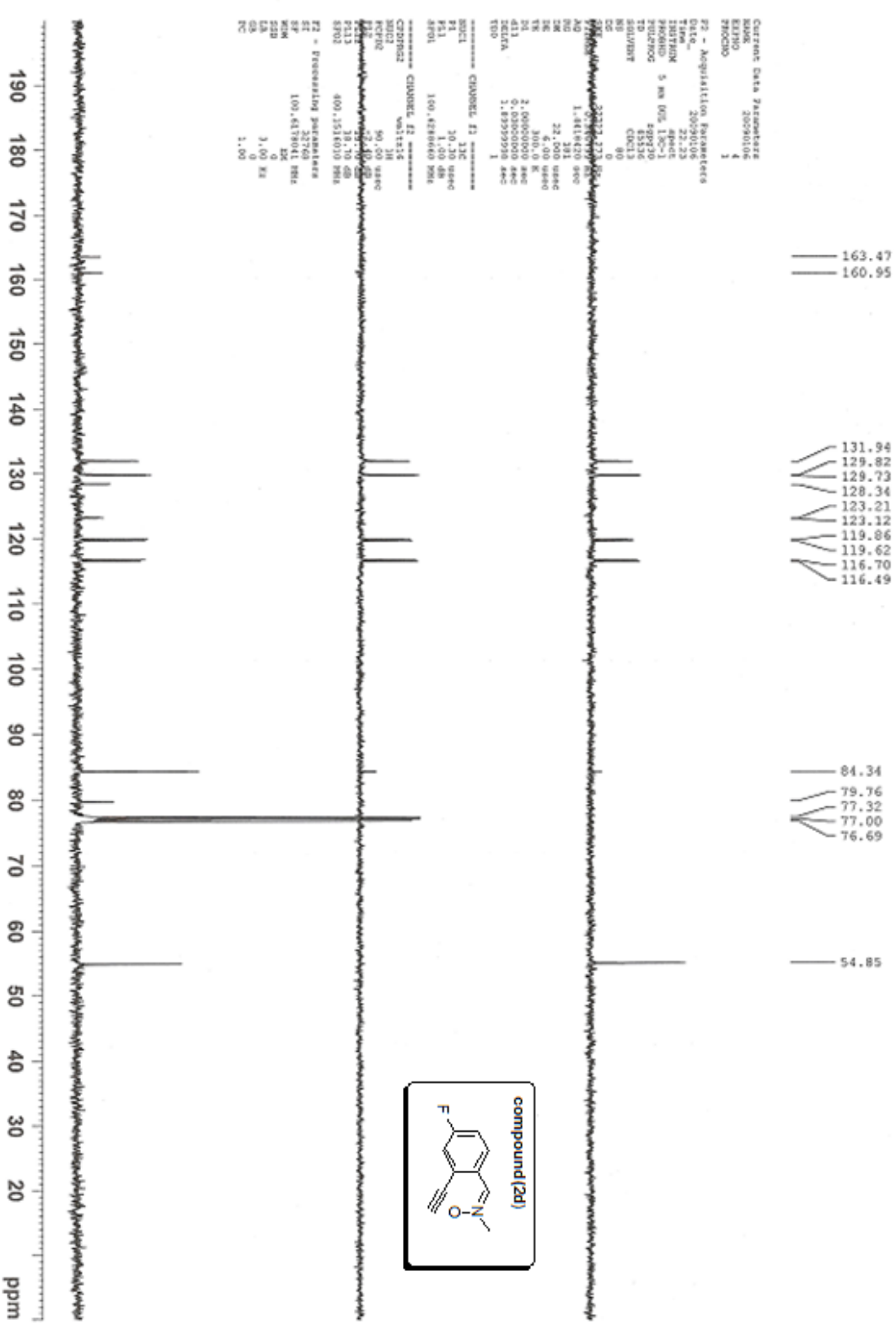

Current Data Parameters
NAME 2009-0522
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090522
Time 10.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 57
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9.301
9.299
9.281
8.031
7.503
7.500
7.484
7.480
7.473
7.459
7.455
7.448
7.401
7.397
7.387
7.382
7.378
7.374
7.369
7.364
7.362
7.359
7.356
7.352
7.313
7.309
7.294
7.290
7.275
7.272
7.240
5.063
3.322
compound(2b)
Bn
1.00
1.00
3.11
4.37
1.27
2.14
1.02
ppm

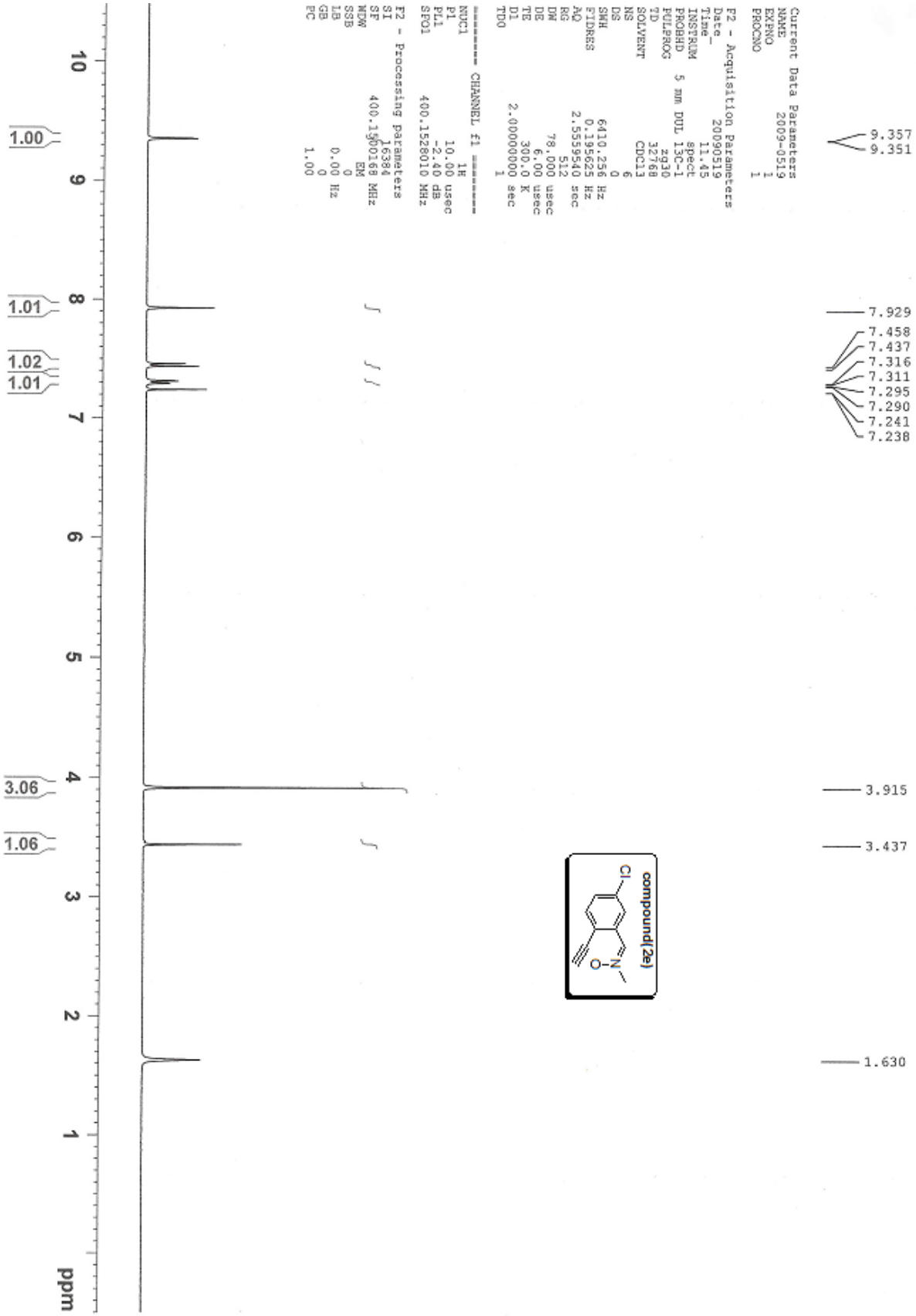

132.98
132.65
132.02
131.58
129.65
129.24
129.16
128.83
127.42
121.11
83.26
80.73
77.32
77.01
76.69
71.63
compound(2b)
Bn
O
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

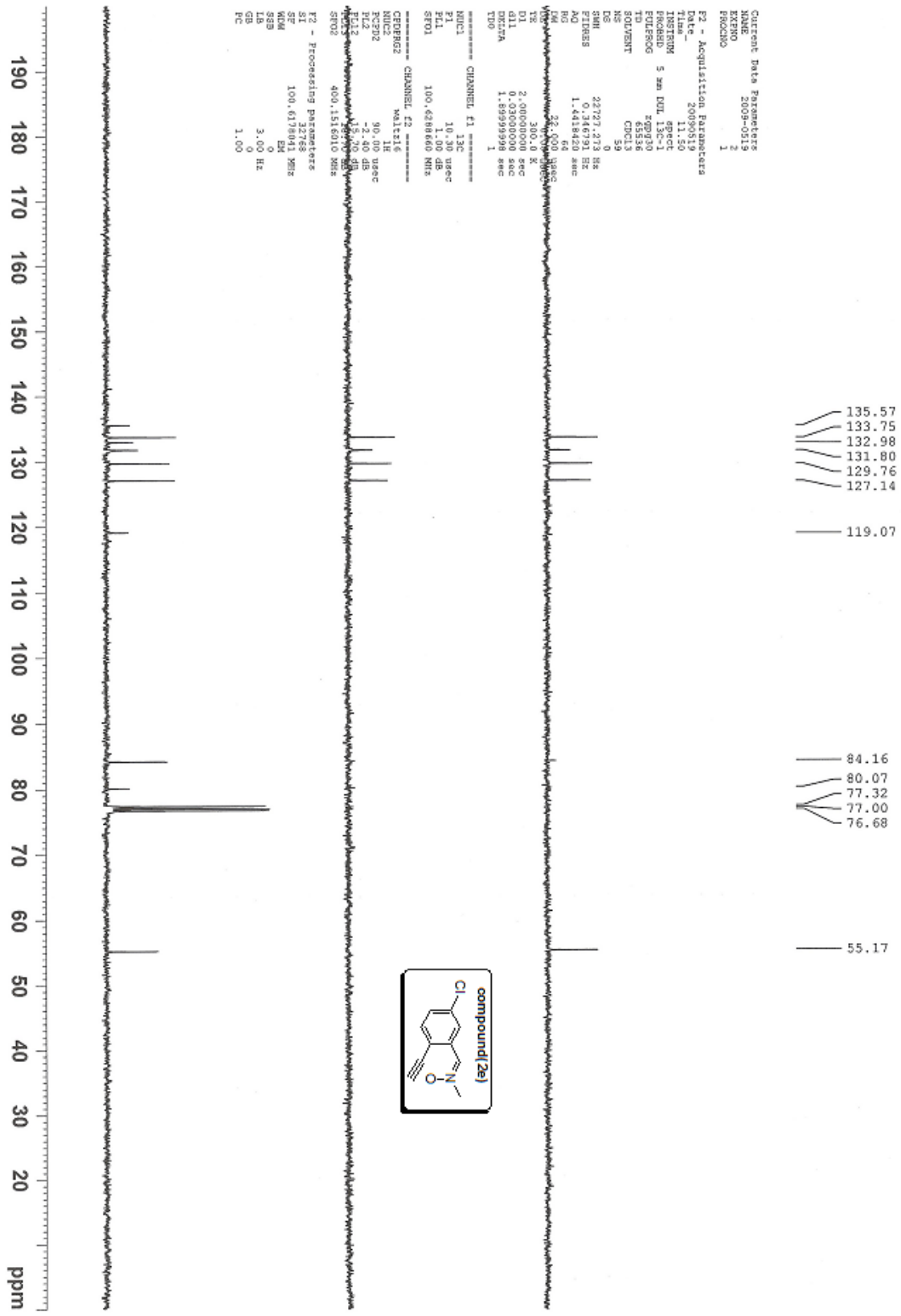

f-5-cho
expl
9.070
9.064
9.042
9.036
7.924
7.471
7.456
7.450
7.435
7.016
7.010
6.995
6.990
6.975
6.969
3.873
3.376
compound(2c)
F
O
N
ppm

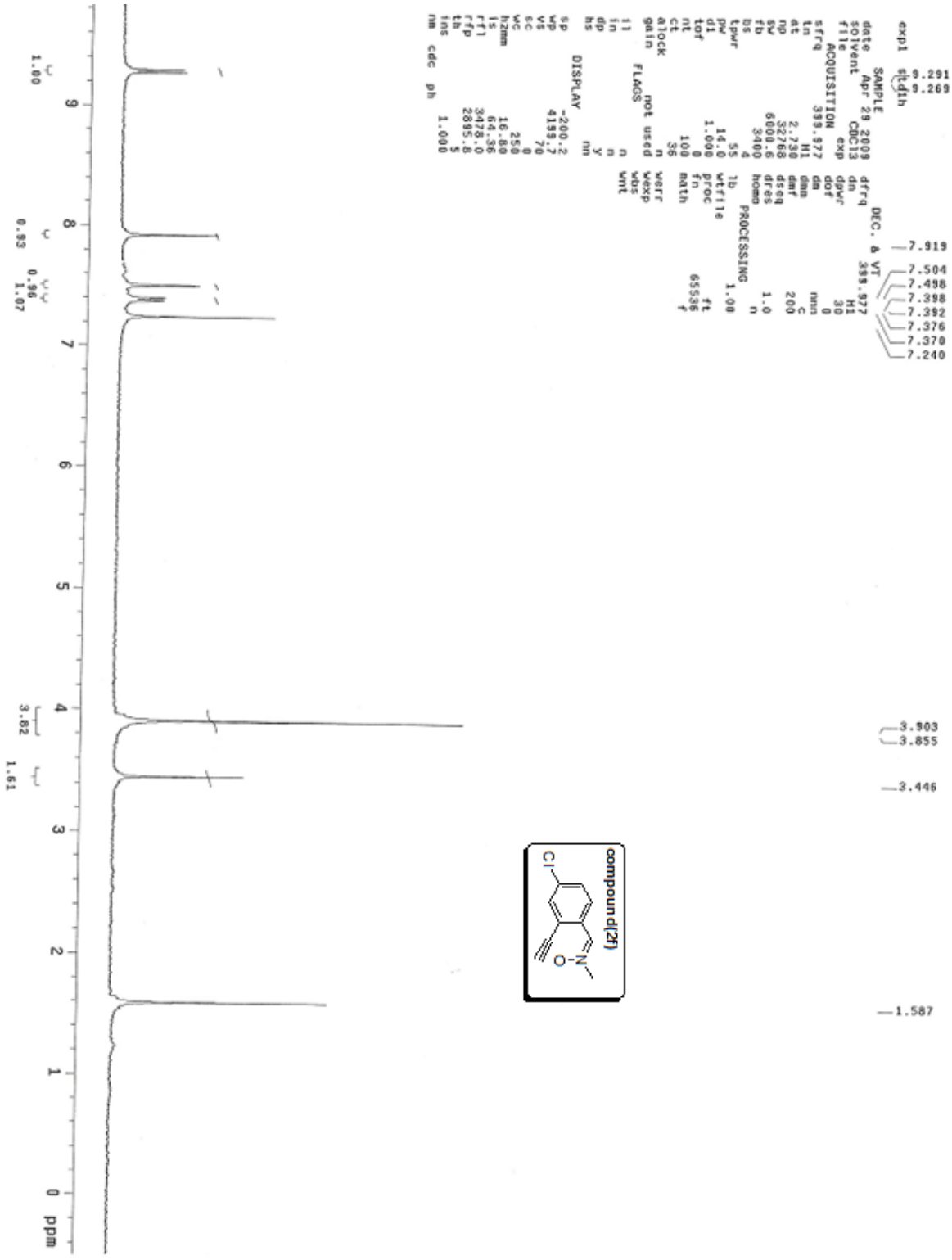

compound(2c)
F
O
N
ppm

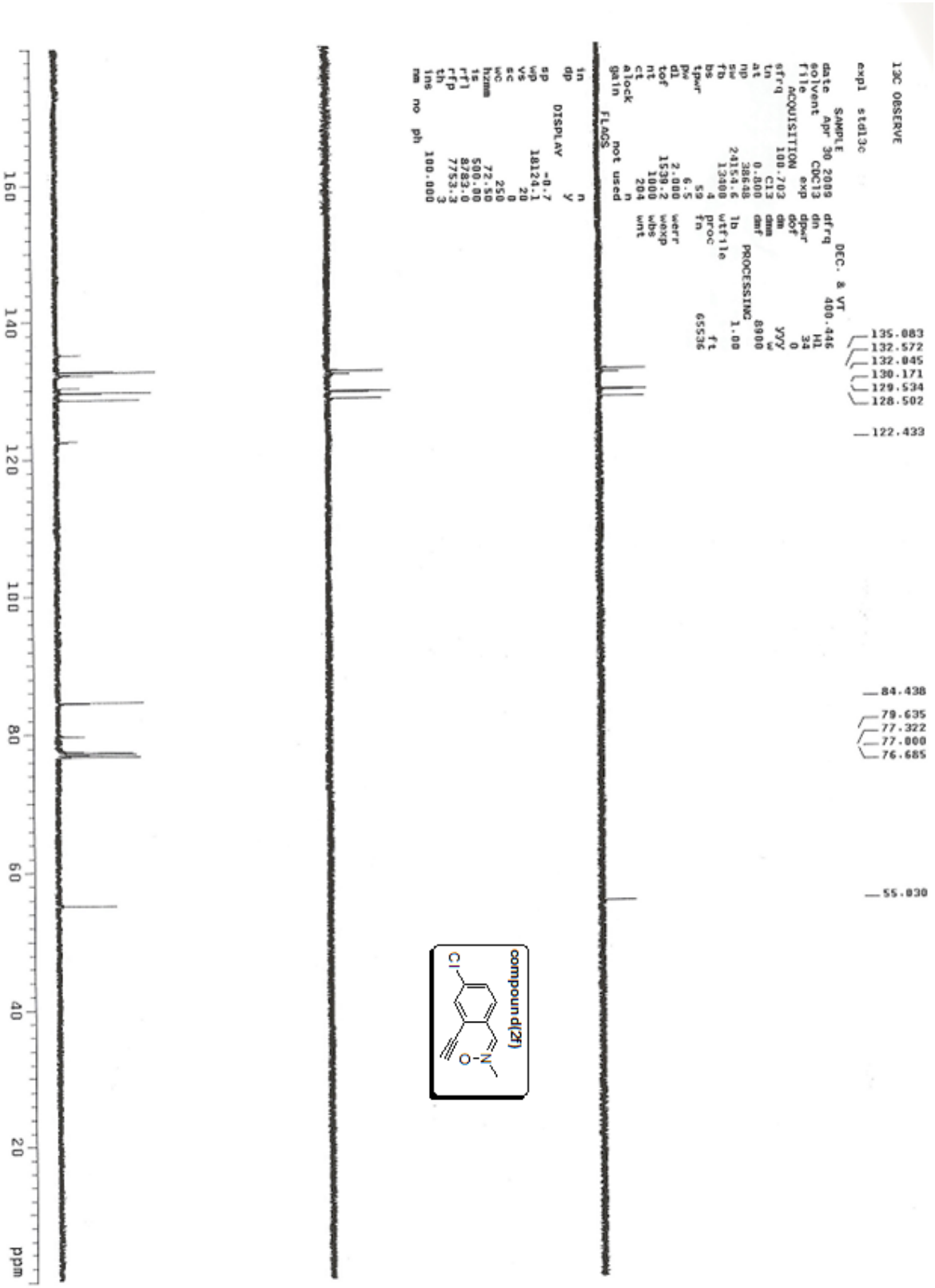

9.368
9.352
9.345
9.330
7.893
7.241
7.210
7.203
7.188
7.181
7.127
7.121
7.106
7.101
7.084
7.078
3.879
3.449
1.00
0.99
1.02
1.05
3.17
0.90
compound(2d)
F
O
N
10
9
8
7
6
5
4
3
2
1
ppm

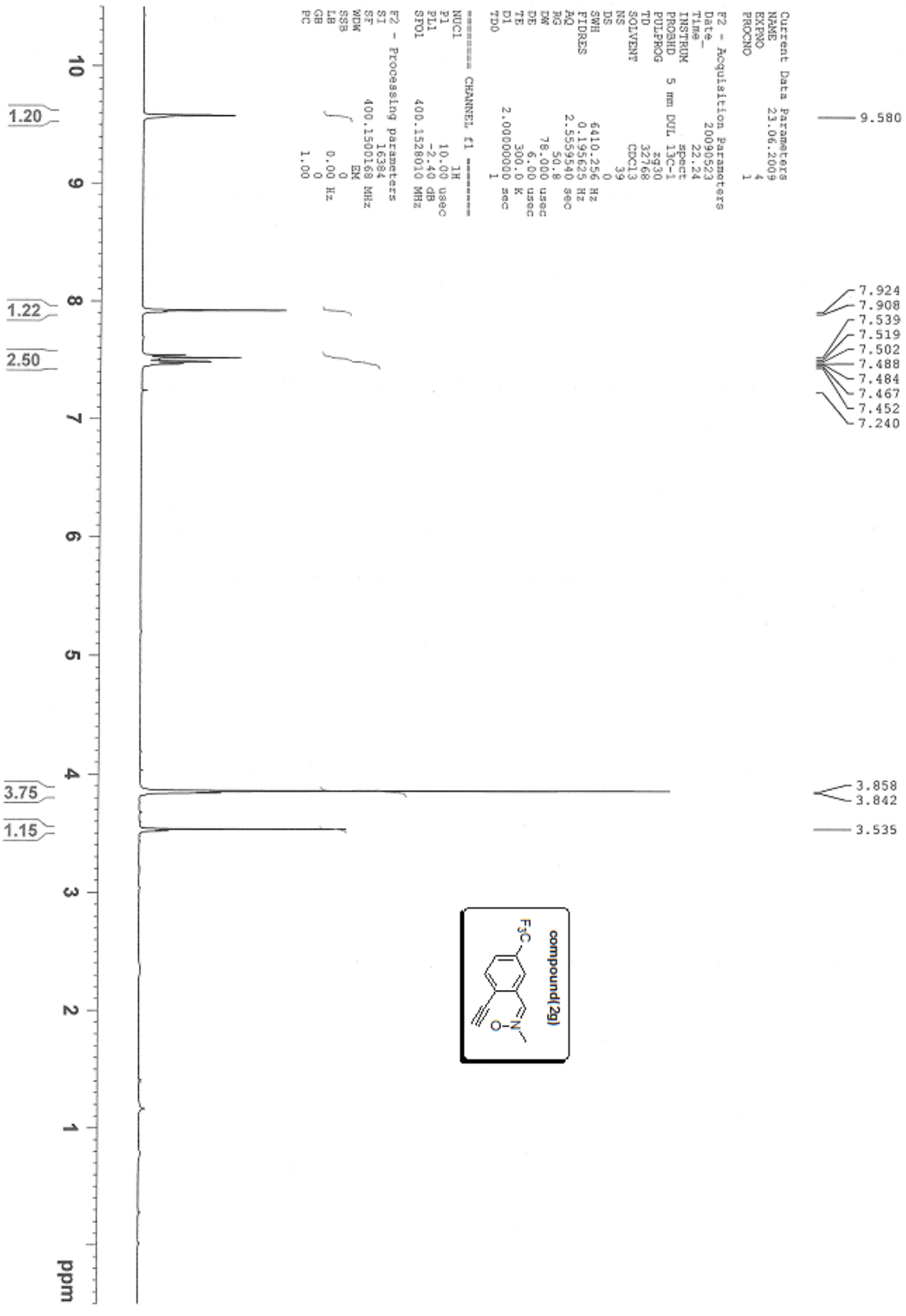

163.47
160.95
131.94
129.82
129.73
128.34
123.21
123.12
119.86
119.62
116.70
116.49
84.34
79.76
77.32
77.00
76.69
54.85
compound(2d)
F
O
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

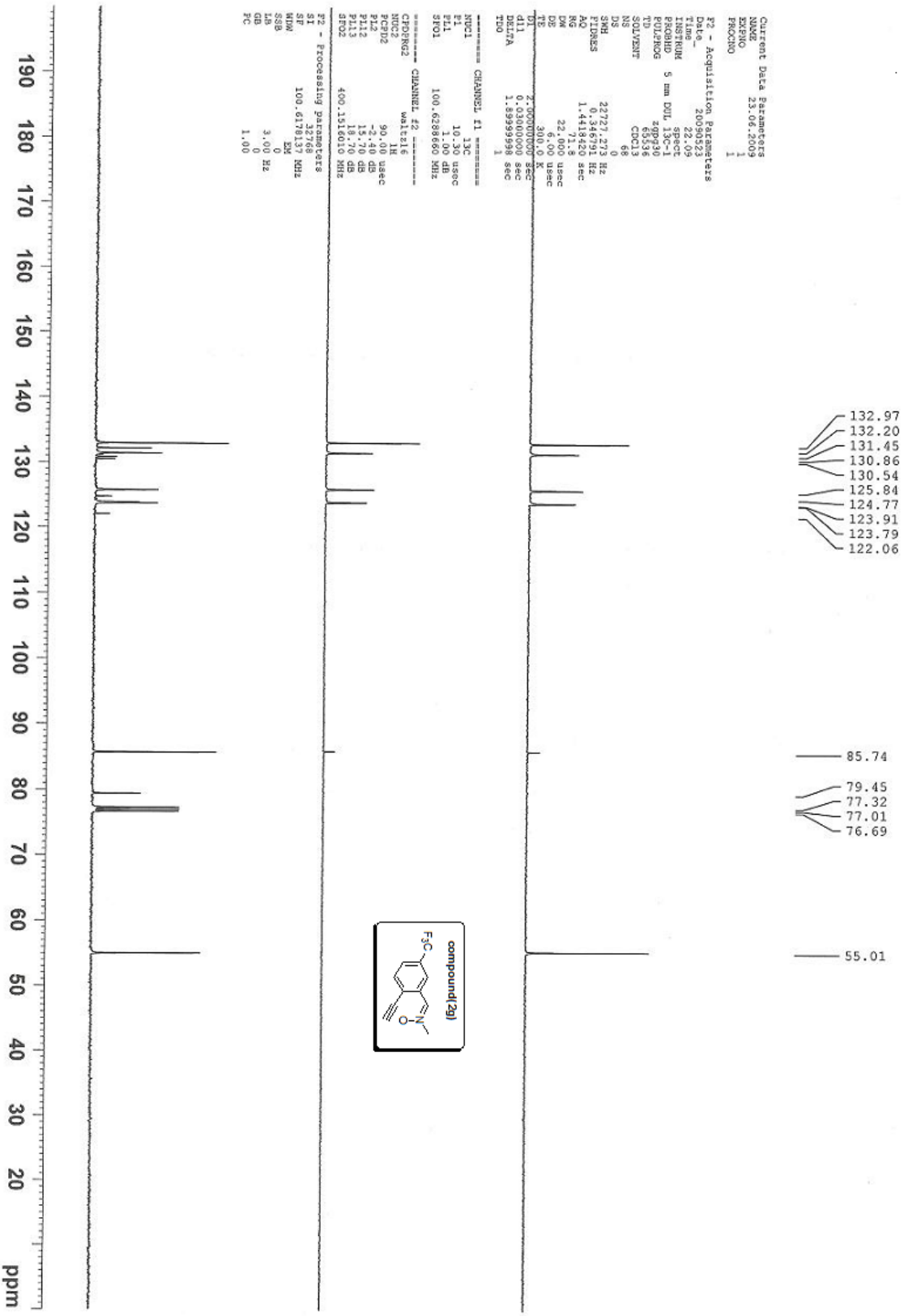

Current Data Parameters
NAME
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090519
Time 11.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 512
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9.357
9.351
7.929
7.458
7.437
7.316
7.311
7.295
7.290
7.241
7.238
3.915
3.437
1.630
1.00
1.01
1.02
1.01
3.06
1.06
compound(2e)
Cl
O
N
ppm

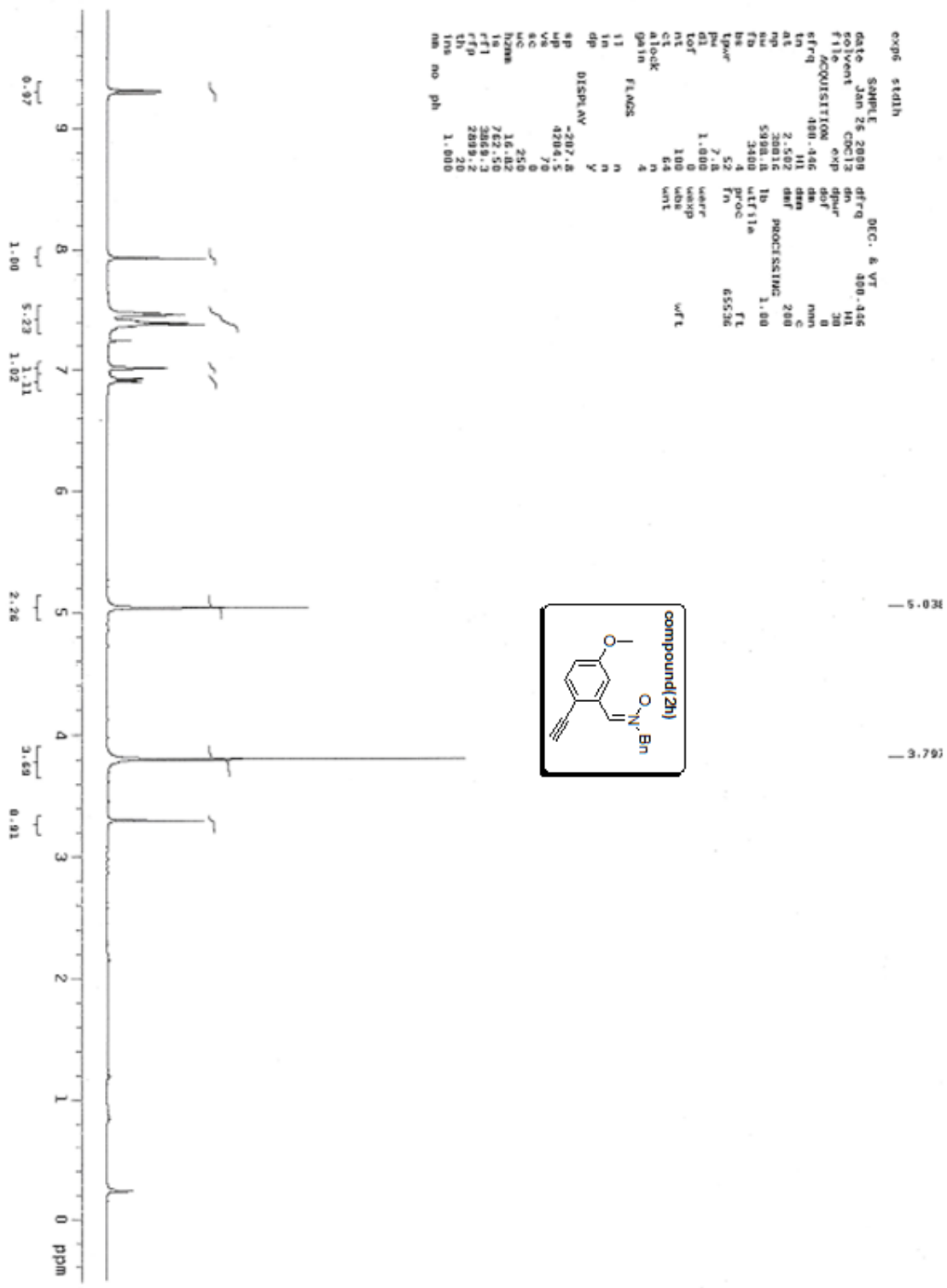

135.57
133.75
132.98
131.80
129.76
127.14
119.07
84.16
80.07
77.32
77.00
76.68
55.17
compound(2e)
Cl
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

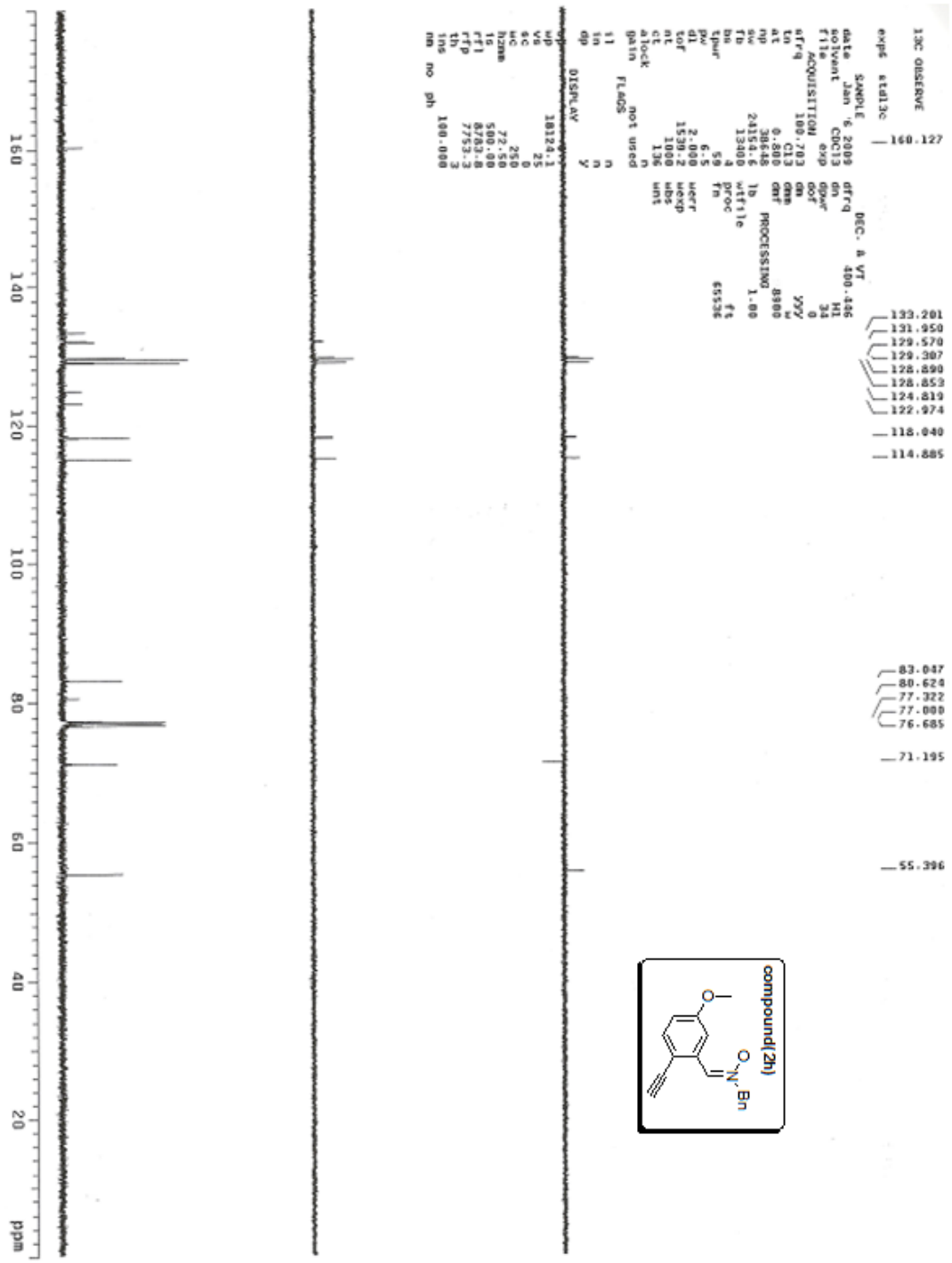

compound(2f)
Cl
O
N
9.291
9.269
7.919
7.504
7.498
7.398
7.392
7.376
7.370
7.240
3.903
3.855
3.446
1.587
1.00
0.93
0.96
1.07
3.82
1.61
ppm

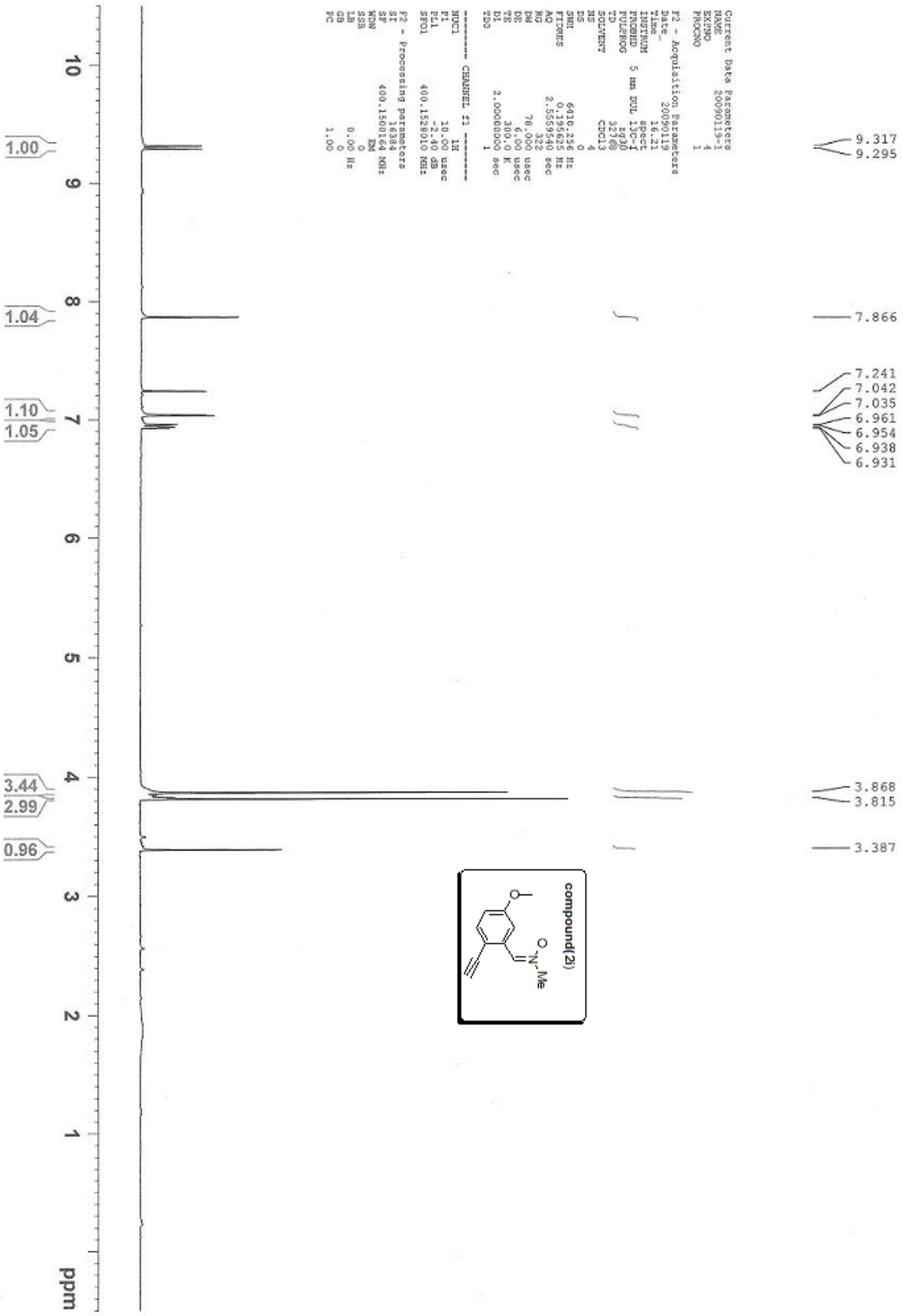

135.083
132.572
132.045
130.171
129.534
128.502
122.433
84.438
79.635
77.322
77.000
76.685
55.030
compound(2f)
Cl
O
N
160
140
120
100
80
60
40
20
ppm

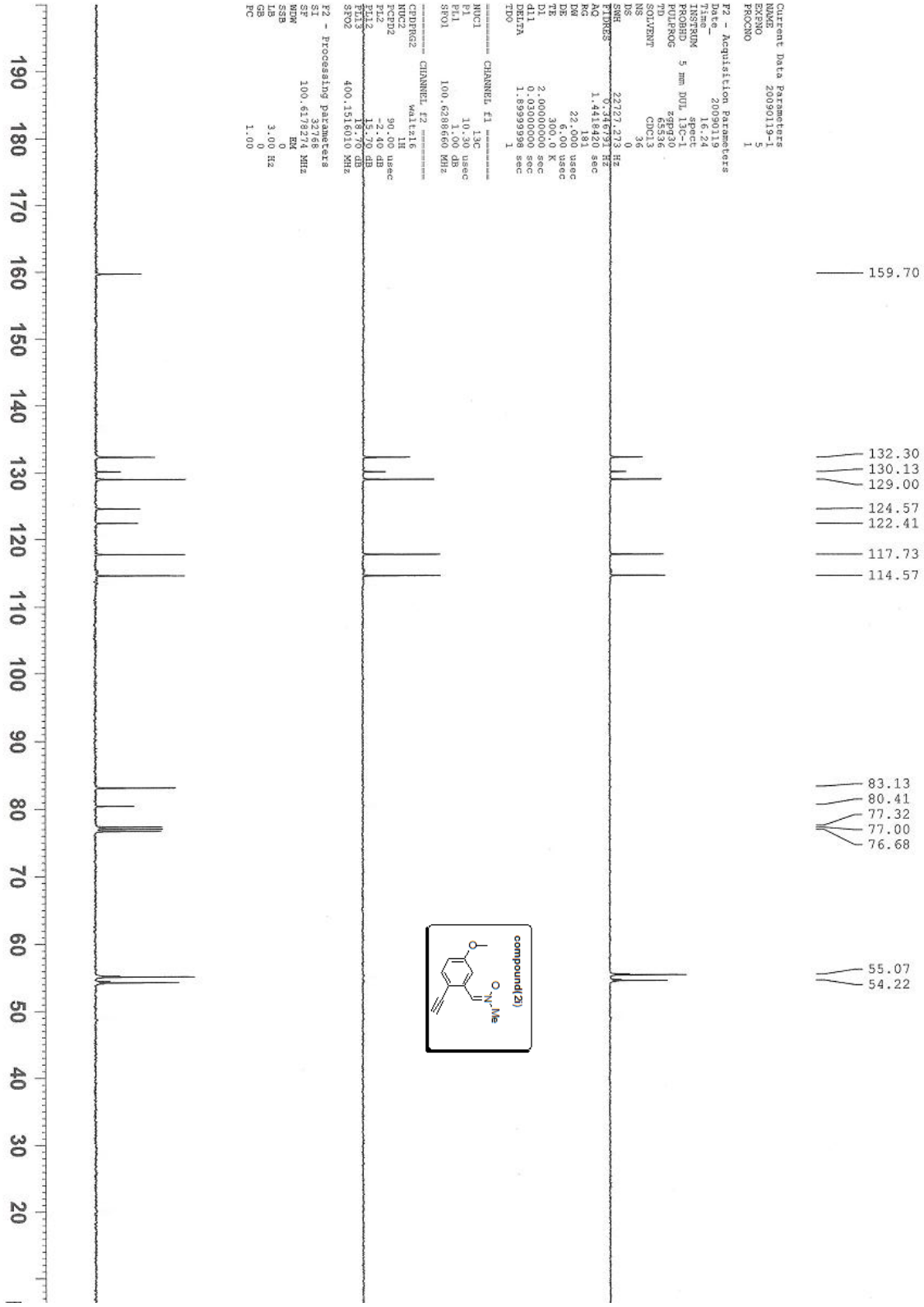

Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD 5 mm DUL 13C-1
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TD0
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1.00
compound(2g)
9.580
7.924
7.908
7.539
7.519
7.502
7.488
7.484
7.467
7.452
7.240
3.858
3.842
3.535
1.20
1.22
2.50
3.75
1.15
ppm

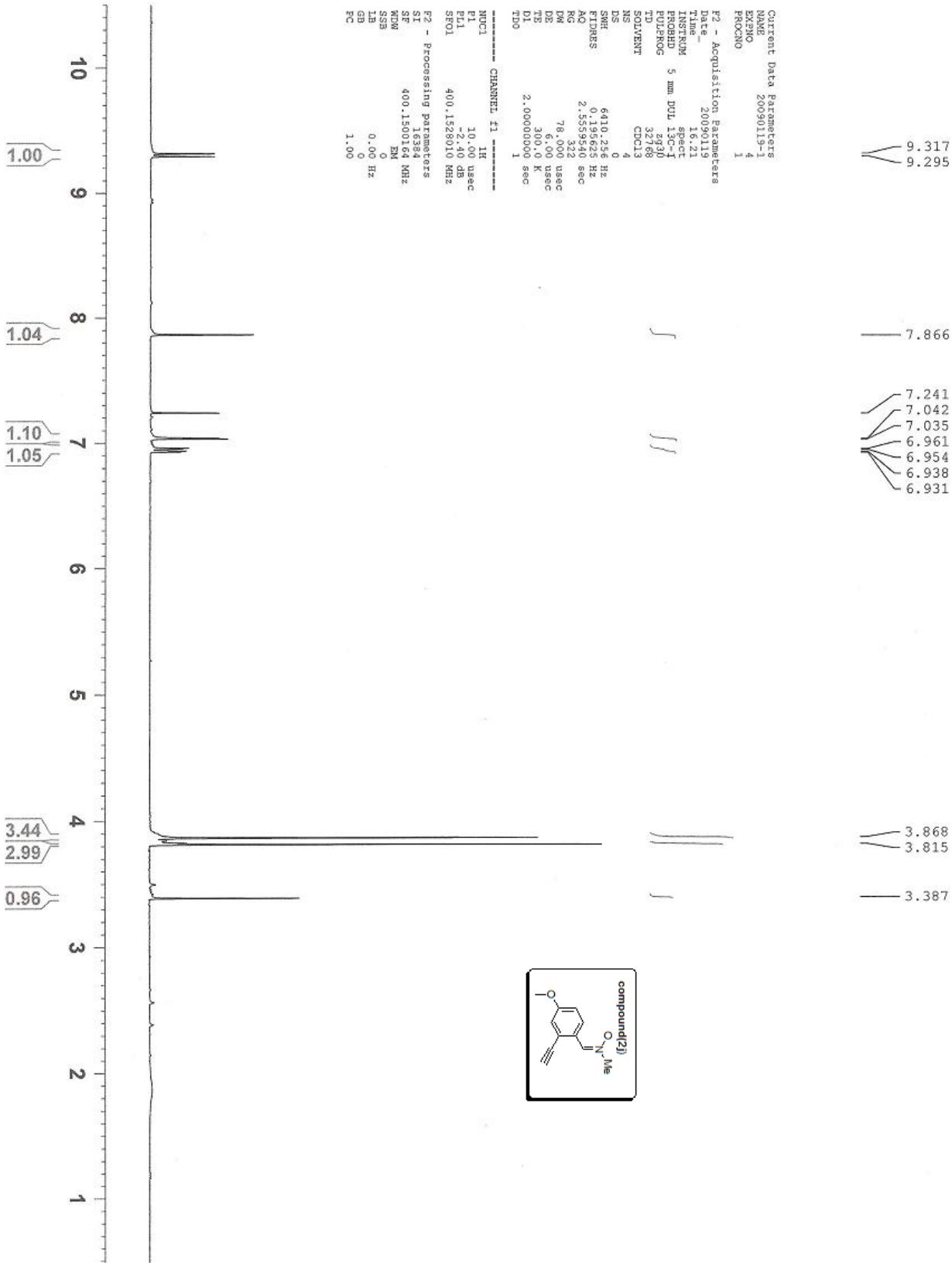

Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
d11
DELTA
TD0
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
5 mm DUL 13C-1
zgpg30
65536
CDCl3
68
0
22727.273 Hz
0.346791 Hz
1.4418420 sec
71.8
22.000 usec
6.00 usec
300.0 K
2.00000000 sec
0.03000000 sec
1.89999998 sec
1
13C
10.30 usec
1.00 dB
100.6288660 MHz
waltz16
90.00 usec
12.40 dB
15.70 dB
18.70 dB
400.1516010 MHz
32768
100.6178137 MHz
EM
0
3.00 Hz
0
1.00
compound(2g)
F3C
O
N
132.97
132.20
131.45
130.86
130.54
125.84
124.77
123.91
123.79
122.06
85.74
79.45
77.32
77.01
76.69
55.01
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

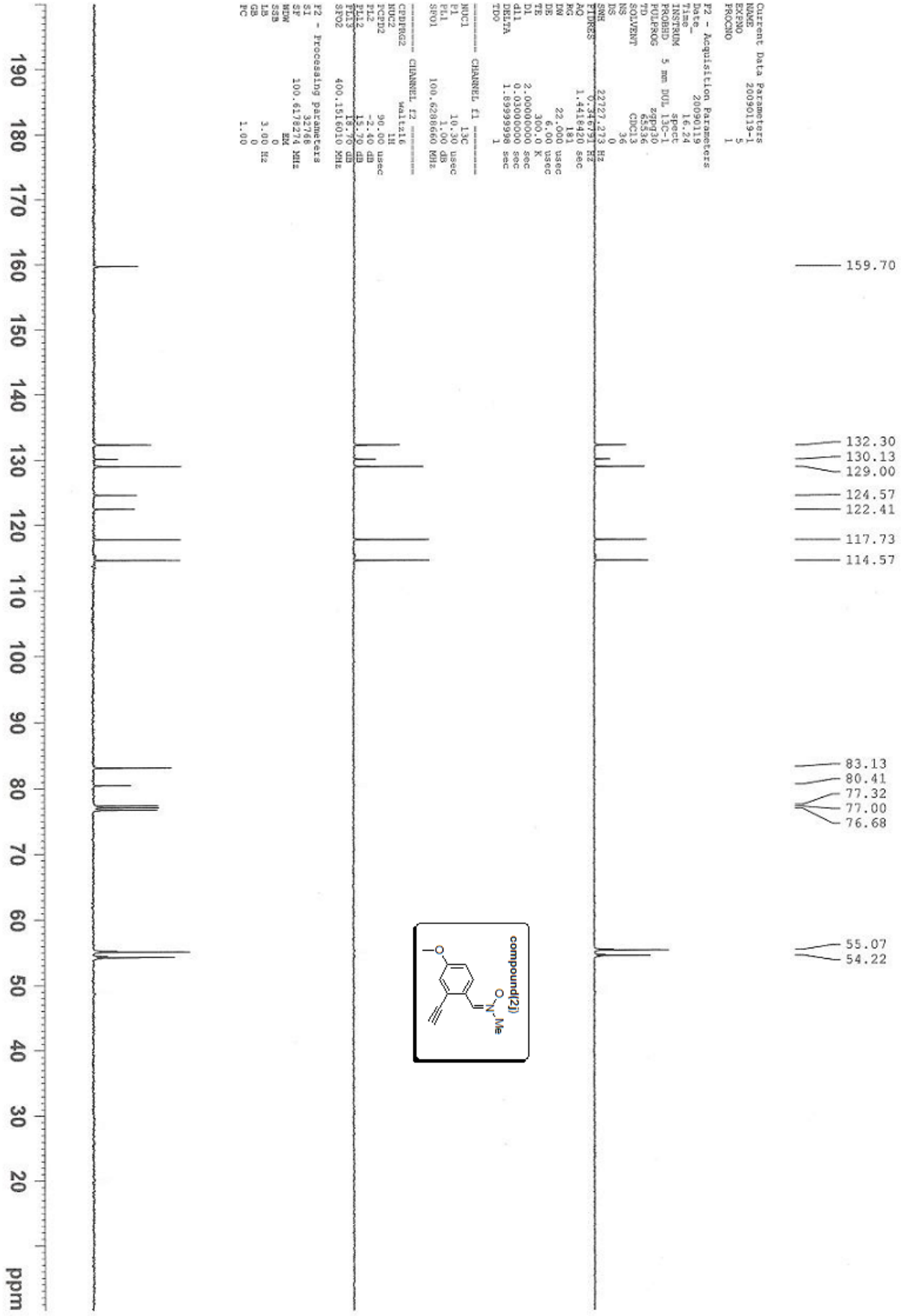

exp6 std1h
SAMPLE
date Jan 25 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 400.486
tn H1
at 2.502
np 30816
nm 5908.8
fb 3400
bs 4
tpwr 52
pw 7.8
pl1 1.000
sfrq 400.486
dfrq DEC. & VT
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
wft
FLAGS
il n
in n
dp y
DISPLAY
sp -207.8
wp 4208.5
vs 70
sc 0
wc 250
hzmm 16.82
is 762.50
rfl 3869.3
rfp 2899.2
th 20
ins 1.000
nm no ph
compound(2h)
Bn
5.038
3.793
0.97
1.00
5.23
1.11
1.02
2.26
3.69
0.91
ppm

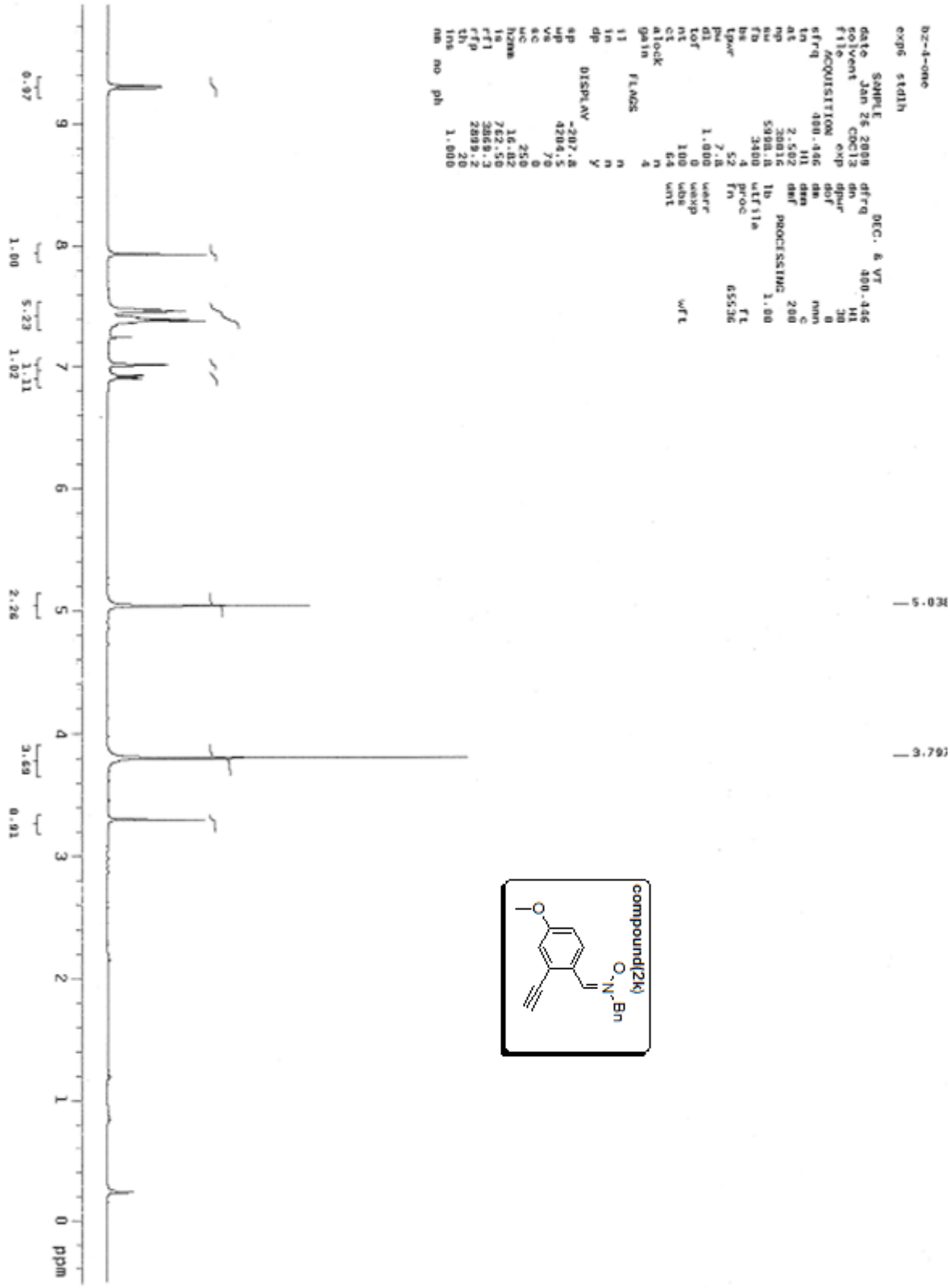

compound(2h)
O
O
N
Bn

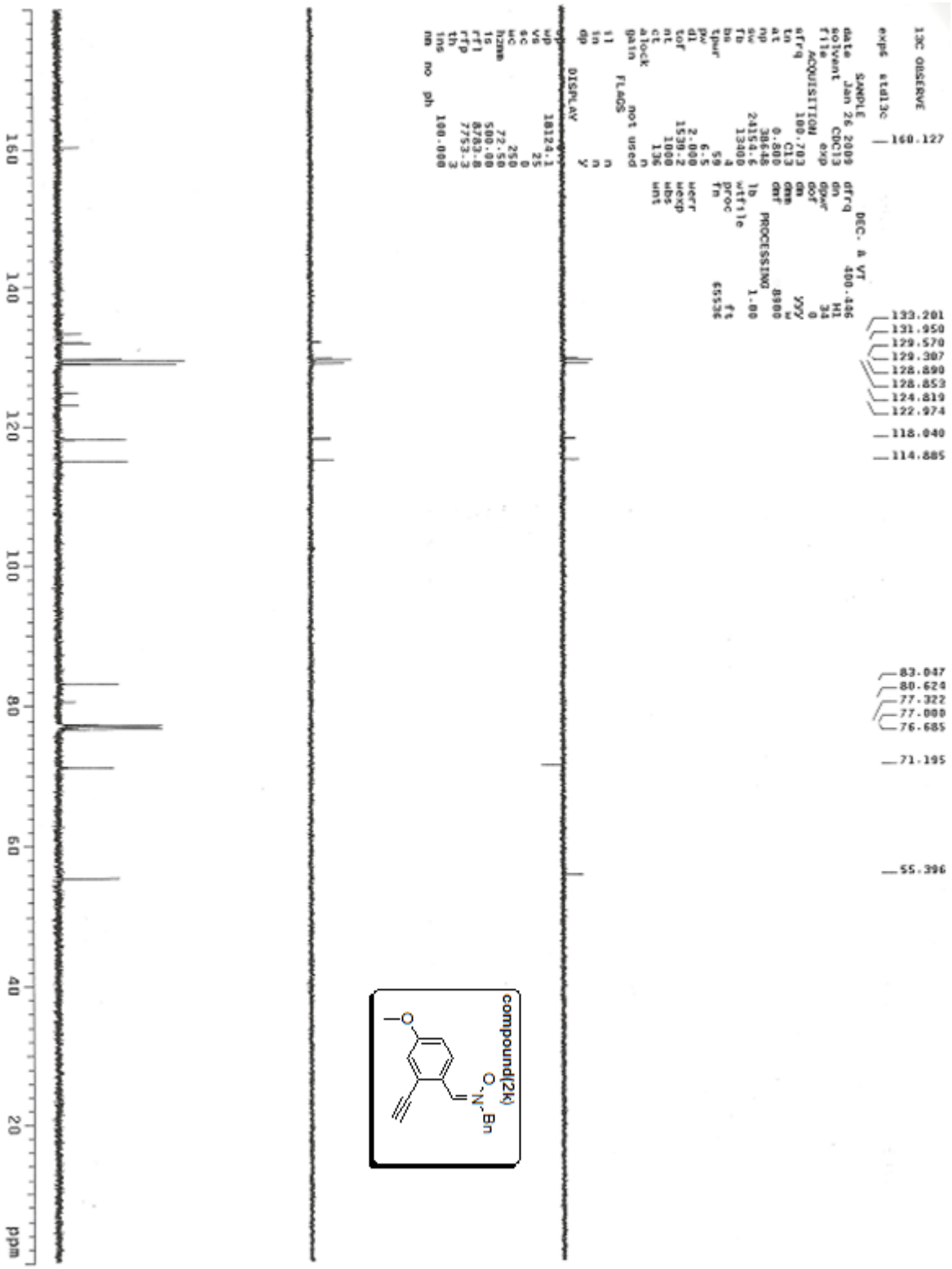

1.00
1.04
1.10
1.05
3.44
2.99
0.96
10
9
8
7
6
5
4
3
2
1
ppm
9.317
9.295
7.866
7.241
7.042
7.035
6.961
6.954
6.938
6.931
3.868
3.815
3.387
compound(2i)
O
N
Me

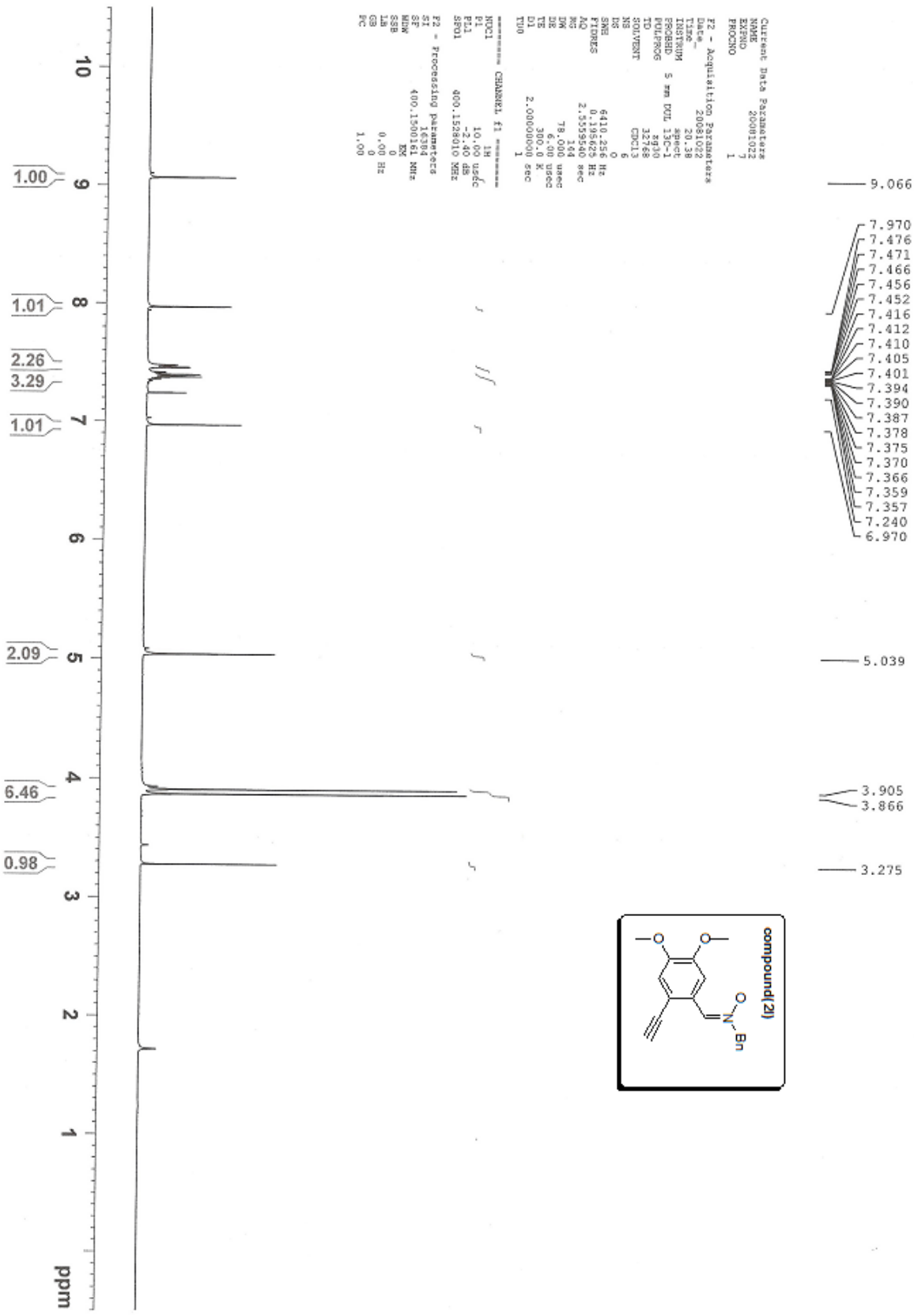

159.70
132.30
130.13
129.00
124.57
122.41
117.73
114.57
83.13
80.41
77.32
77.00
76.68
55.07
54.22
compound(2i)
O
N
Me
O

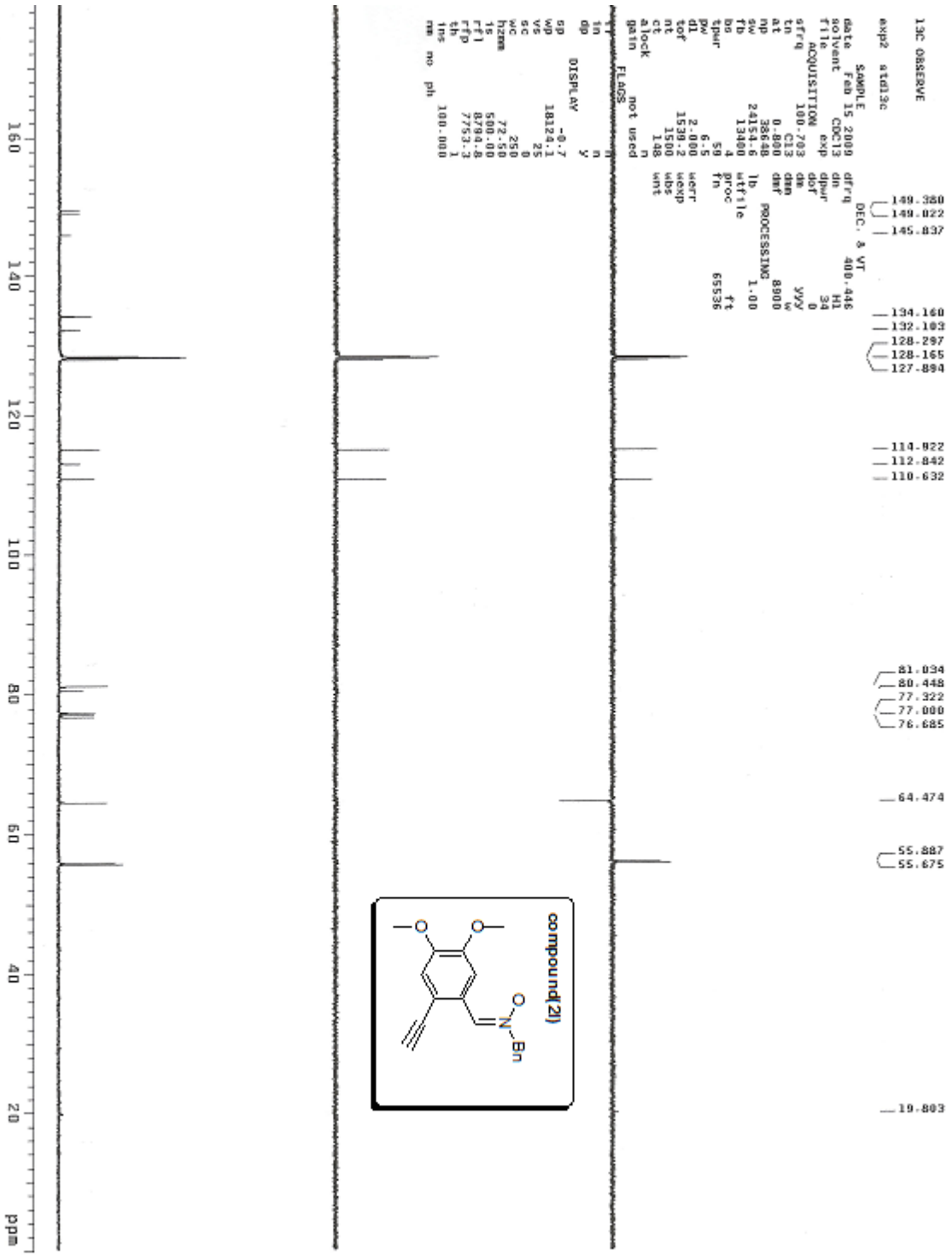

Current Data Parameters
NAME 20090119-1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090119
Time_ 16.21
INSTRUM spect
PROBHD 5 mm DUL 13C-I
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 322
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.00
1.04
1.10
1.05
3.44
2.99
0.96
9.317
9.295
7.866
7.241
7.042
7.035
6.961
6.954
6.938
6.931
3.868
3.815
3.387
compound(2j)
O
O
N
Me

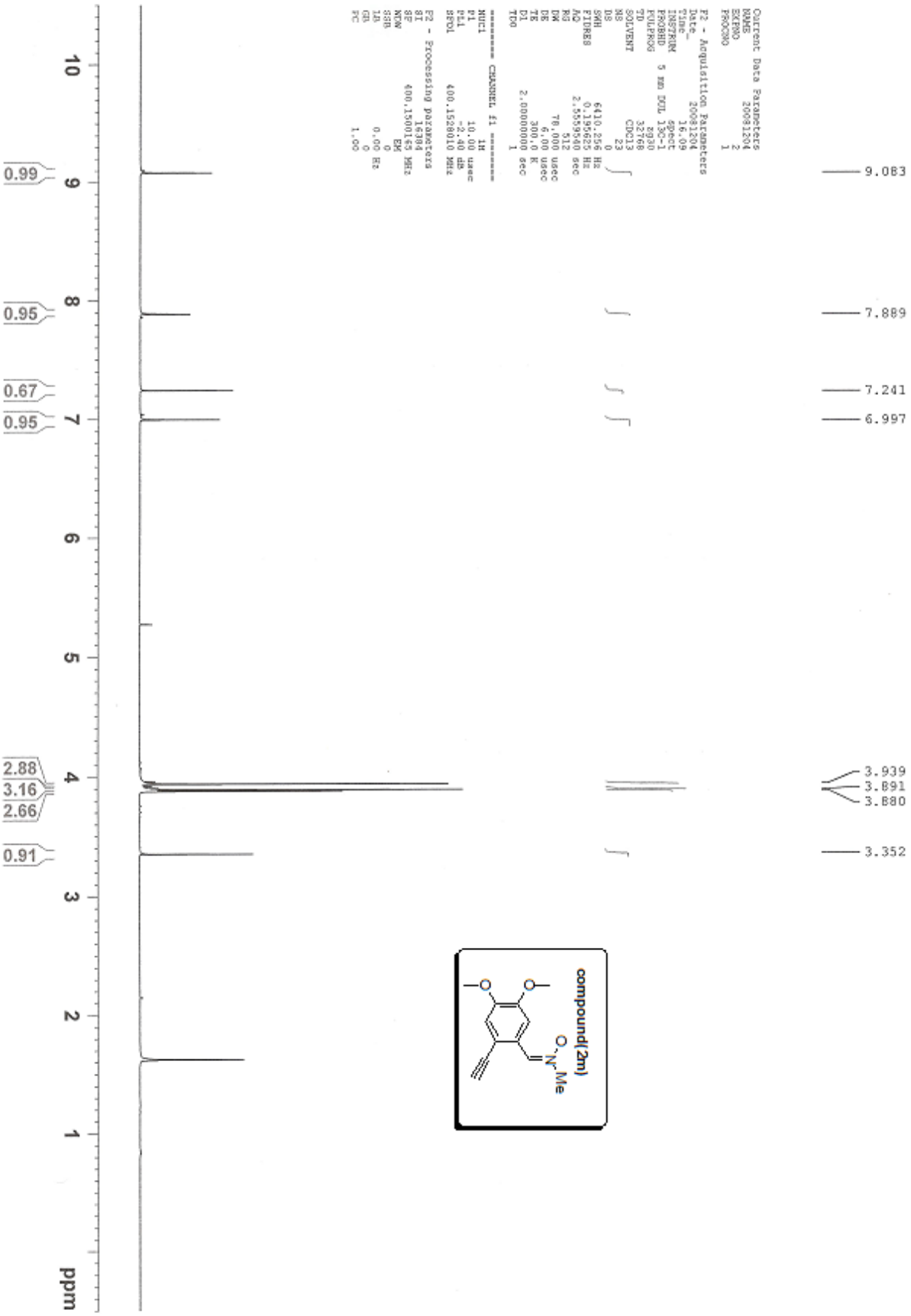

159.70
132.30
130.13
129.00
124.57
122.41
117.73
114.57
83.13
80.41
77.32
77.00
76.68
55.07
54.22
compound(2j)
Me
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

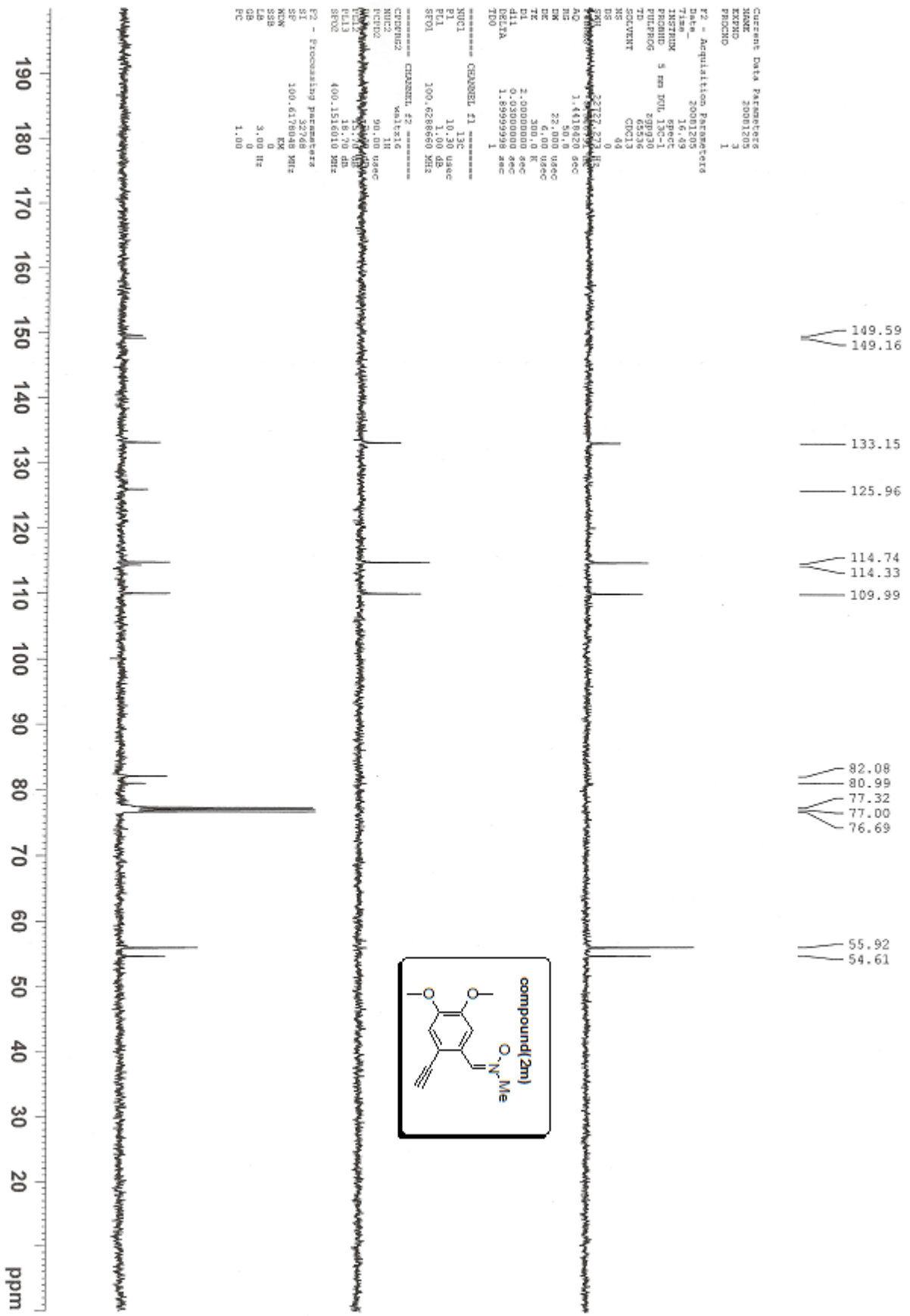

bz-4-one
exp6 std1h
SAMPLE
date Jan 25 2008
solvent cdcl3
file
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 28816
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
pl1 1.000
tof 0
nt 100
ct 64
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -207.8
wp 4204.5
vs 70
sc 0
wc 250
hzmm 16.82
is 282.50
rfl 3869.3
rfp 2899.2
th 20
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
nnn
dm nnn
dmm c
dmf 208
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
wft
wft1
wexp
wbs
wnt
5.038
3.793
compound(2k)
O
N
Bn
0.97
1.00
5.23
1.11
1.02
2.26
3.69
0.91
9
8
7
6
5
4
3
2
1
0
ppm

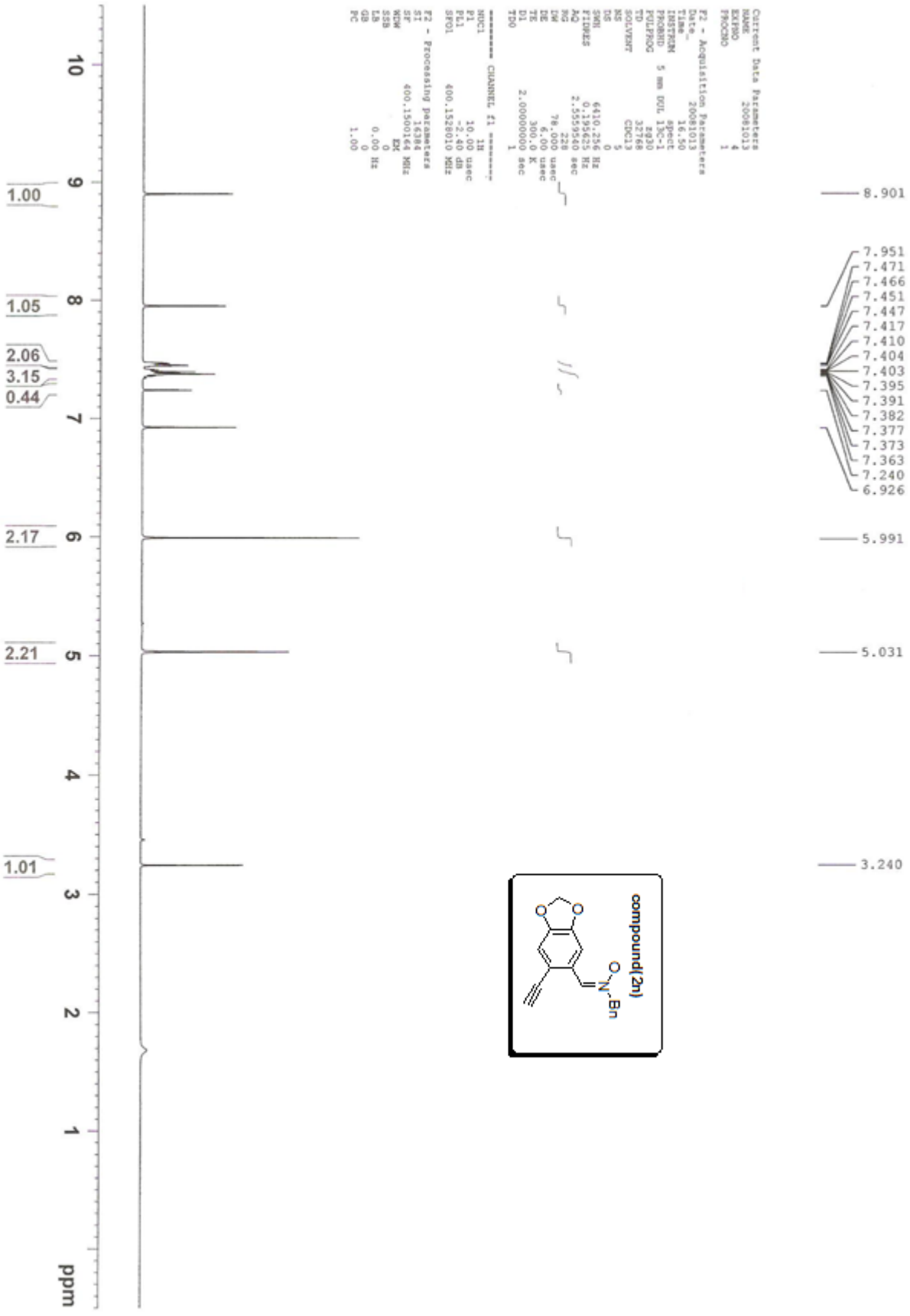

compound(2k)
O
N
Bn
160
140
120
100
80
60
40
20
ppm

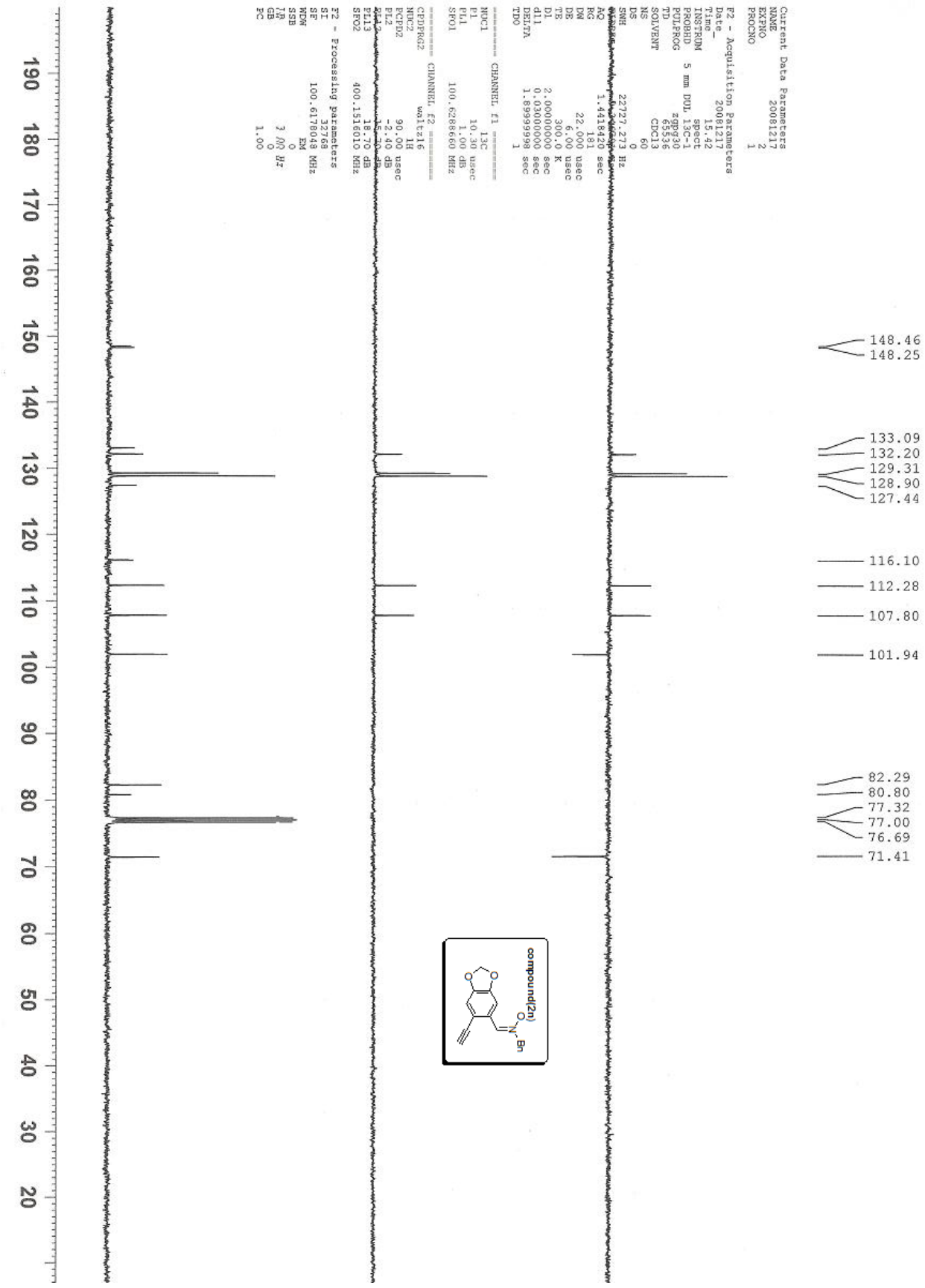

1.00
1.01
2.26
3.29
1.01
2.09
6.46
0.98
ppm
9.066
7.970
7.476
7.471
7.466
7.456
7.452
7.416
7.412
7.410
7.405
7.401
7.394
7.390
7.387
7.378
7.375
7.370
7.366
7.359
7.357
7.240
6.970
5.039
3.905
3.866
3.275
compound(2l)
O
N
Bn

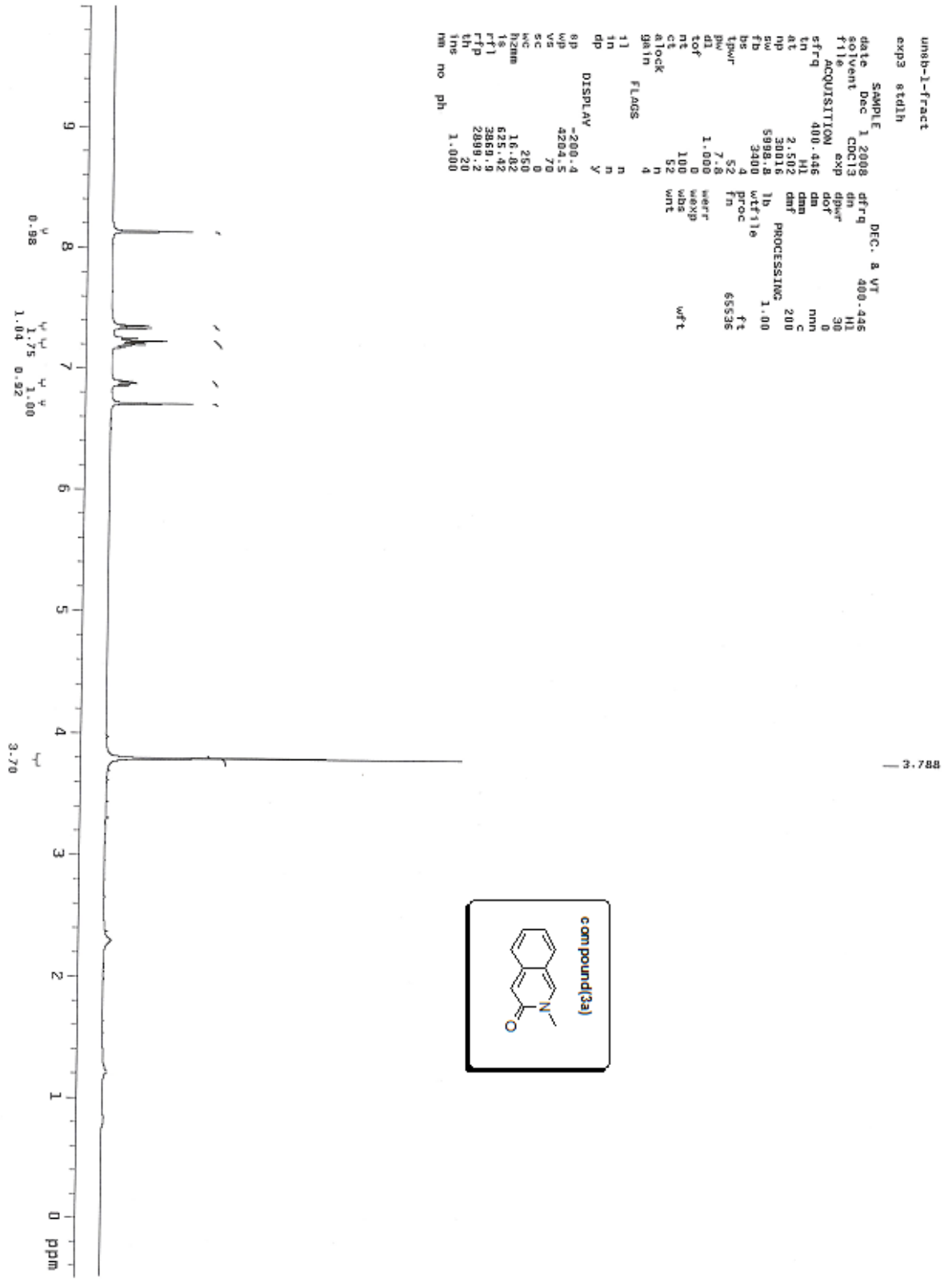

compound(2l)
O
N
Bn
149.380
149.022
145.837
134.160
132.103
128.297
128.165
127.894
114.922
112.842
110.632
81.034
80.448
77.322
77.000
76.685
64.474
55.887
55.675
19.803
ppm

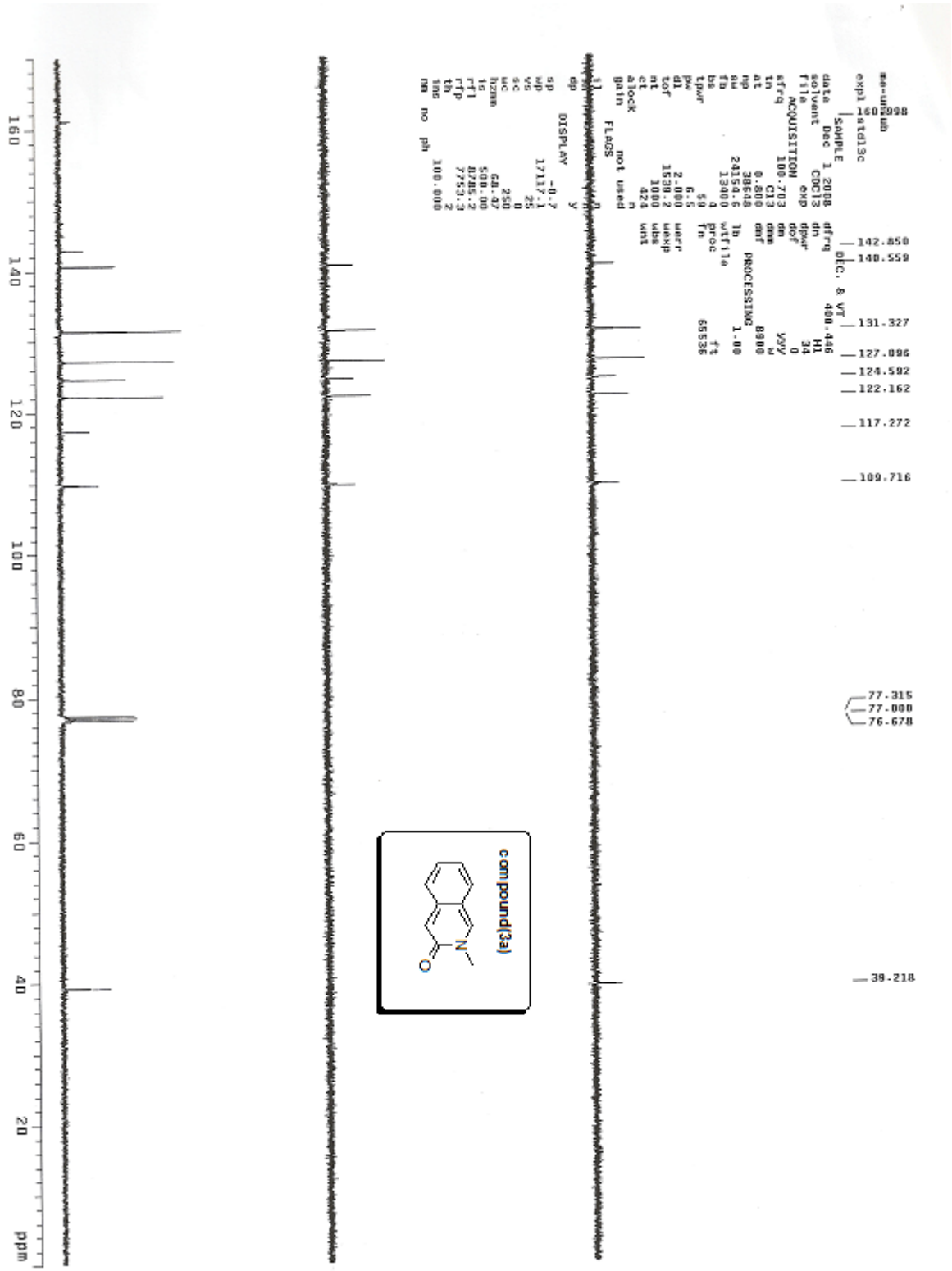

0.99
0.95
0.67
0.95
2.88
3.16
2.66
0.91
9.083
7.889
7.241
6.997
3.939
3.891
3.880
3.352
ppm
compound(2m)
O
N
Me

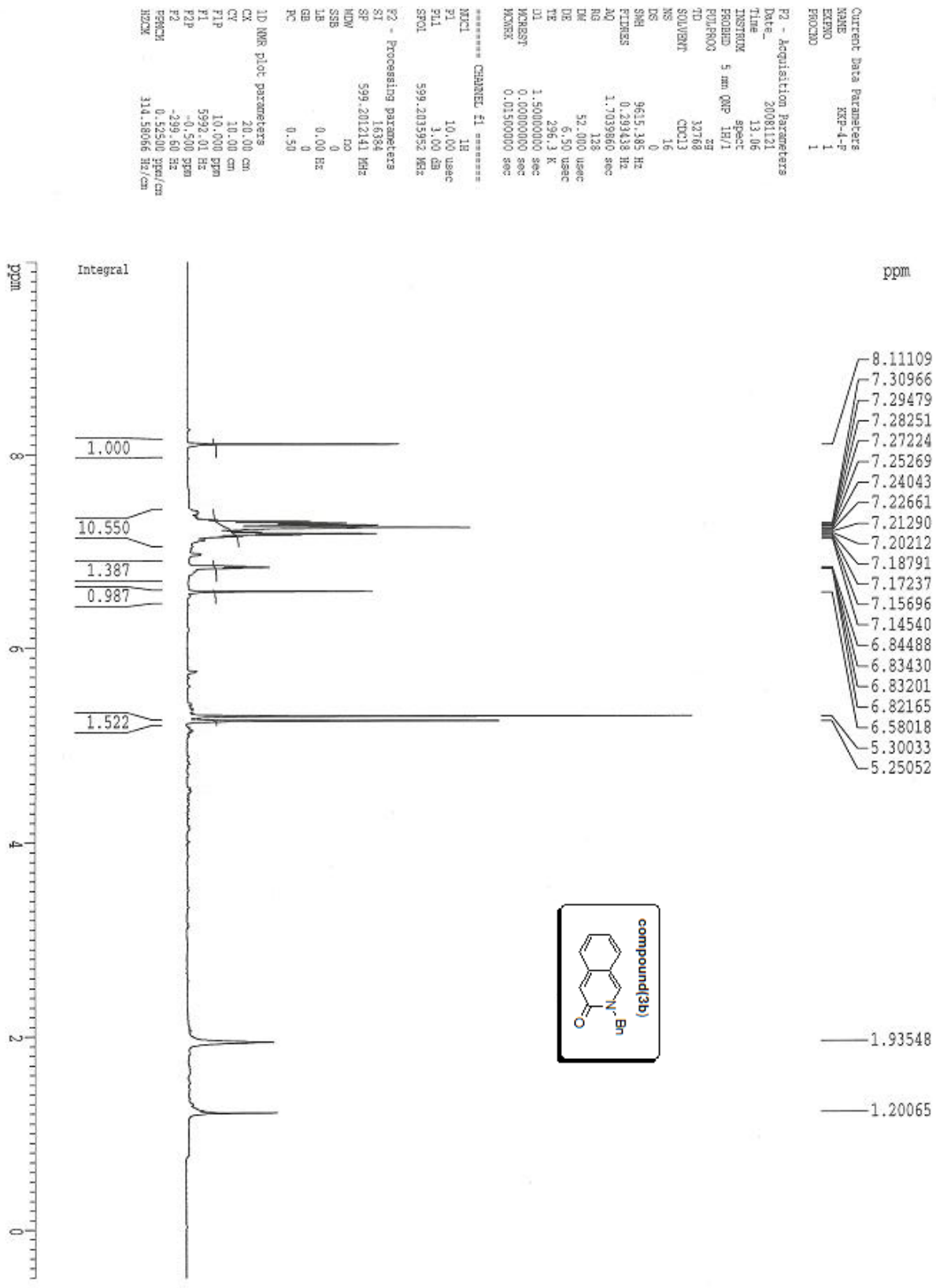

149.59
149.16
133.15
125.96
114.74
114.33
109.99
82.08
80.99
77.32
77.00
76.69
55.92
54.61
compound(2m)
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

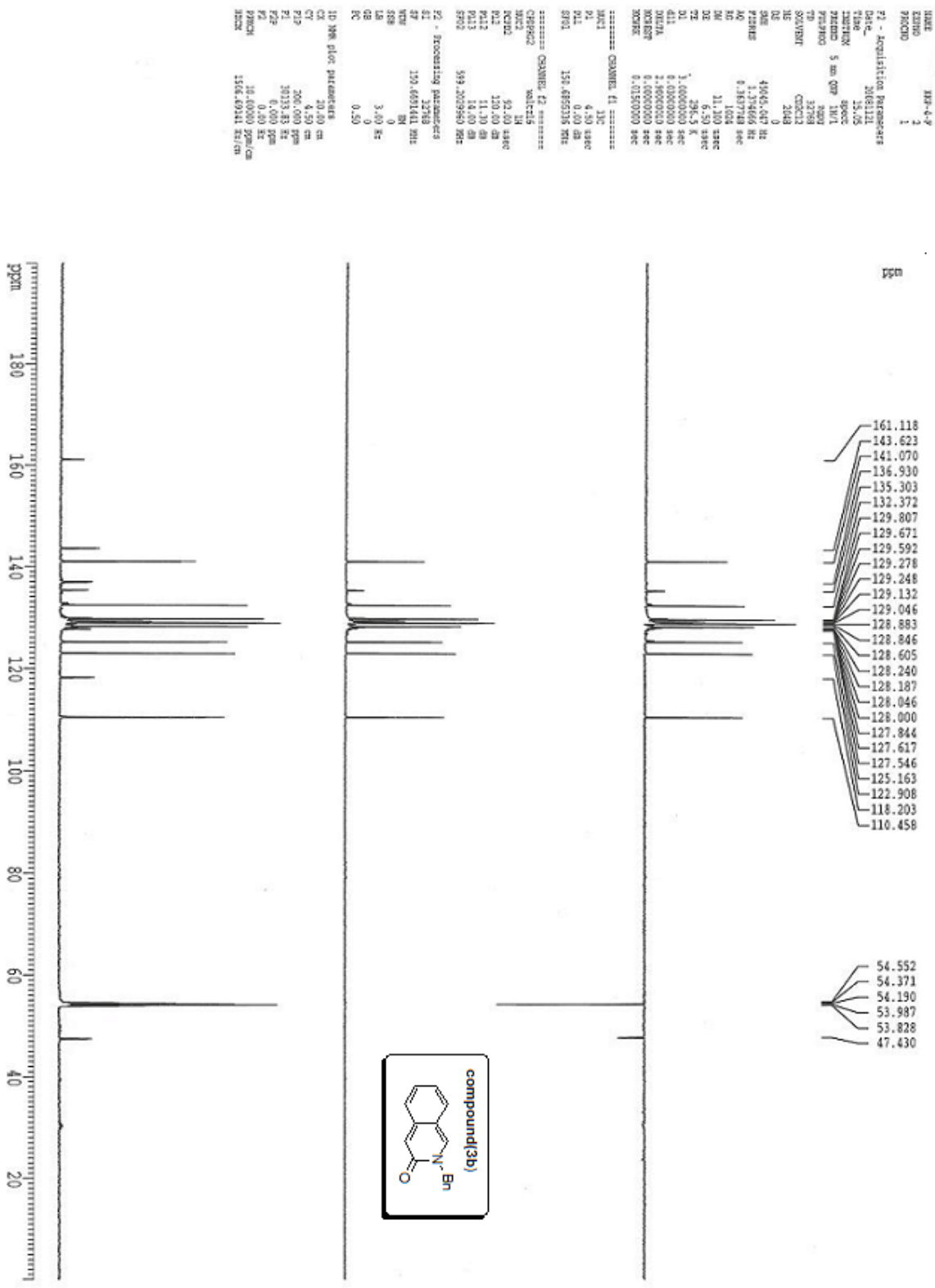

1.00
1.05
2.06
3.15
0.44
2.17
2.21
1.01
8.901
7.951
7.471
7.466
7.451
7.447
7.417
7.410
7.404
7.403
7.395
7.391
7.382
7.377
7.373
7.363
7.240
6.926
5.991
5.031
3.240
ppm
compound(2n)
O
N
Bn

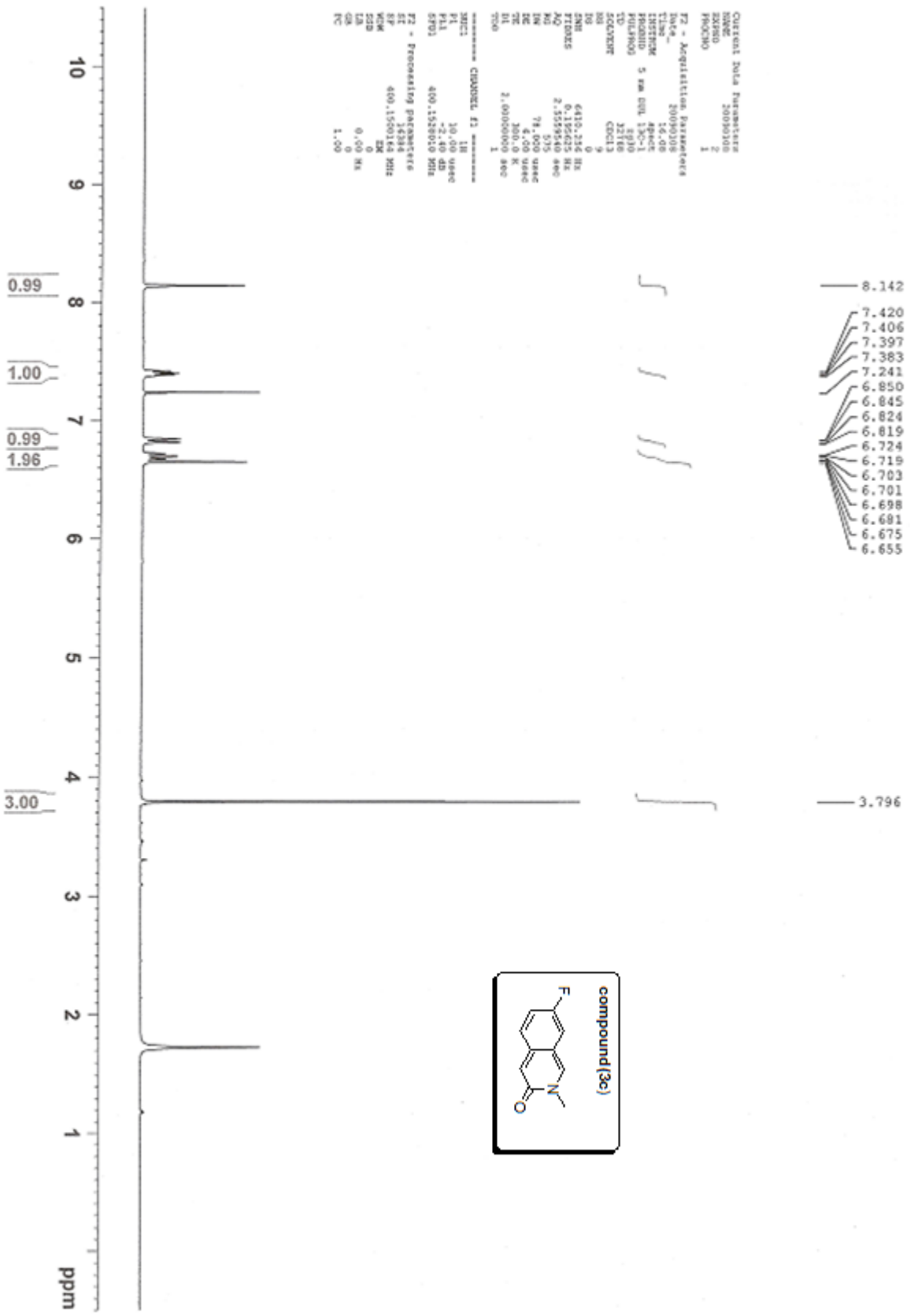

148.46
148.25
133.09
132.20
129.31
128.90
127.44
116.10
112.28
107.80
101.94
82.29
80.80
77.32
77.00
76.69
71.41
compound(2n)
O
O
N
O
Bn
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

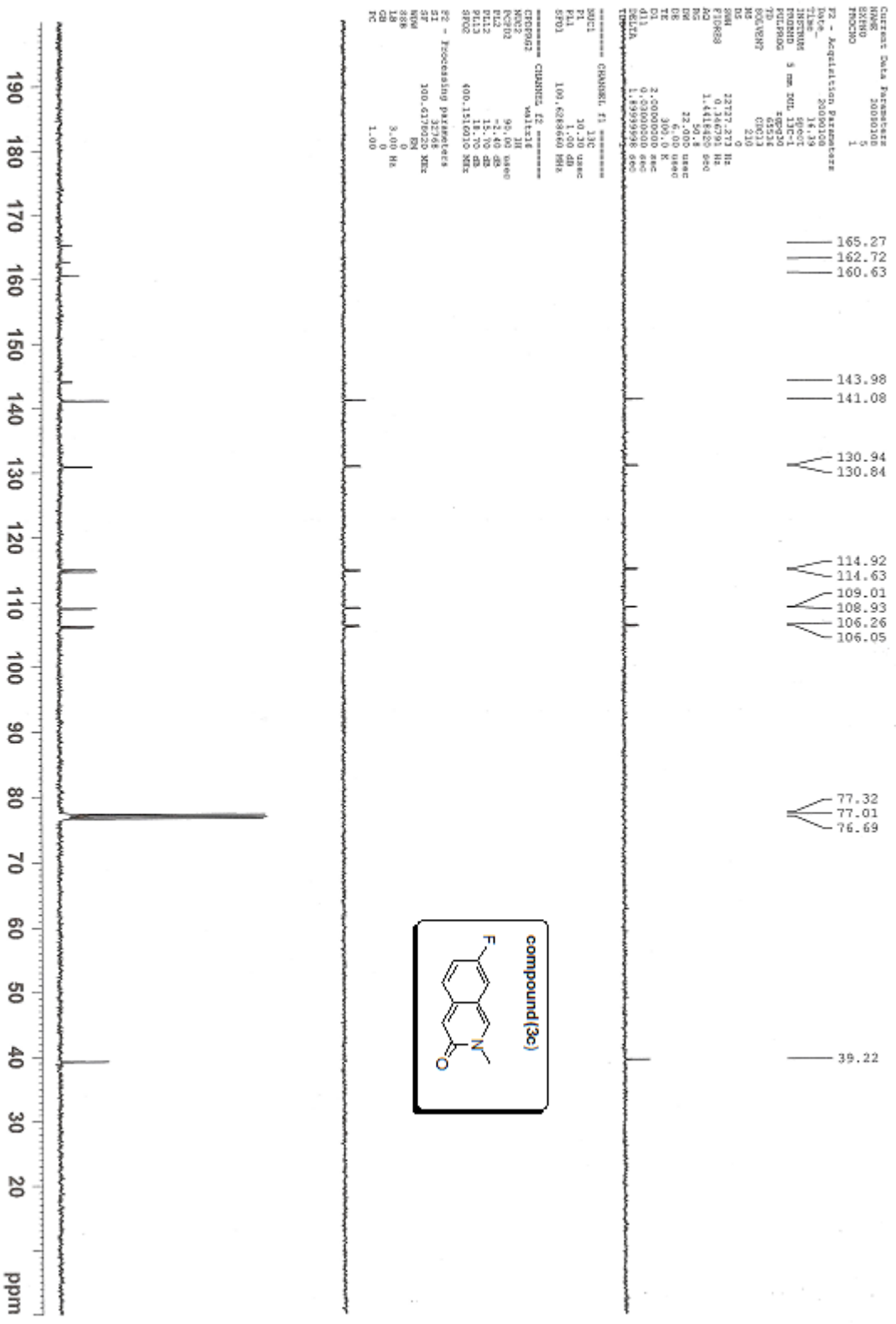

unsb-1-fract
exp3 std1h
SAMPLE
date Dec 1 2008
solvent CDCl3
file
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
pl 1.000
tof 0
nt 100
ct 52
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.4
wp 4204.5
vs 70
sc 0
wc 250
hzmm 16.82
is 625.42
rfl 3869.9
rfp 2899.2
th 20
ins 1.000
nm no ph
dfrq DEC. & VT
dn 400.446
dpwr H1
dof 30
nnn 0
c 200
dmf PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
3.788
compound(3a)
0.98
1.75
1.00
1.04
0.92
3.70
ppm

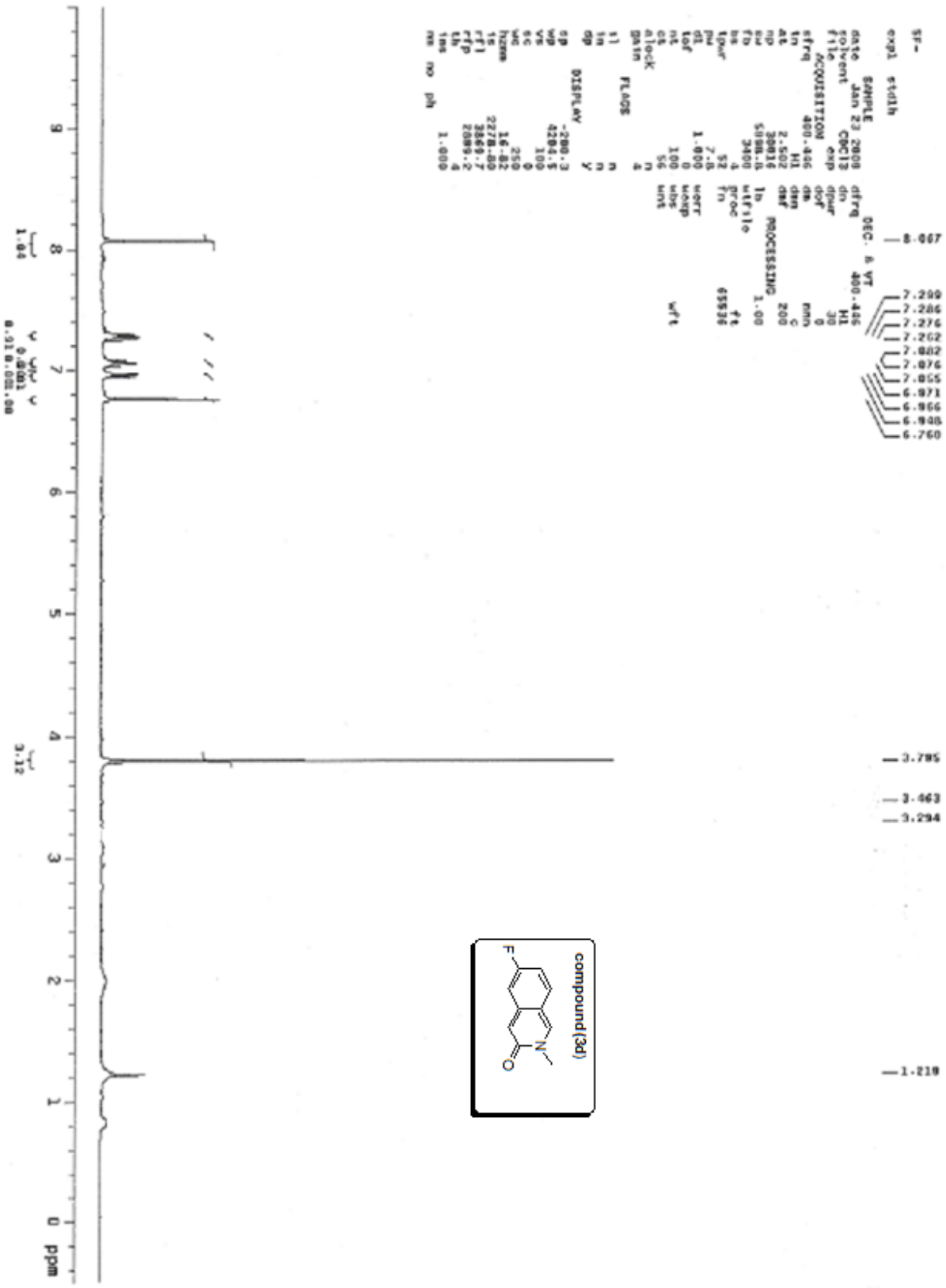

compound(3a)
142.850
140.559
131.327
127.096
124.592
122.162
117.272
109.716
77.315
77.000
76.678
39.218
ppm

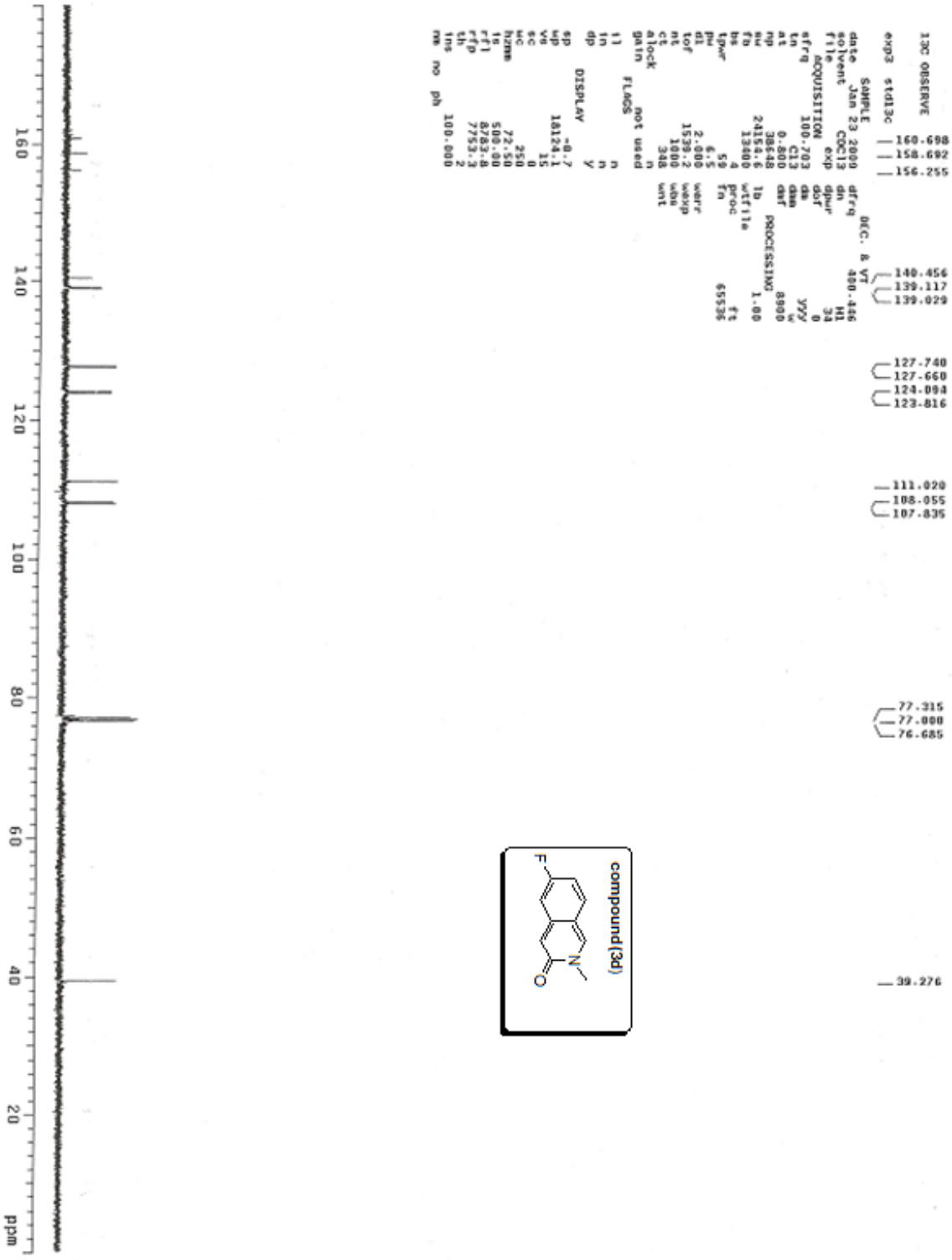

Current Data Parameters
NAME KKP-4-F
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081121
Time 13.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 128
DW 52.000 usec
DE 6.50 usec
TE 296.1 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 599.2035952 MHz
F2 - Processing parameters
SI 16384
SF 599.2012141 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 5992.01 Hz
F2P -0.500 ppm
F2 -299.60 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.58066 Hz/cm
Integral
1.000
10.550
1.387
0.987
1.522
ppm
8.11109
7.30966
7.29479
7.28251
7.27224
7.25269
7.24043
7.22661
7.21290
7.20212
7.18791
7.17237
7.15696
7.14540
6.84488
6.83430
6.83201
6.82165
6.58018
5.30033
5.25052
1.93548
1.20065
compound(3b)
N
Bn
O

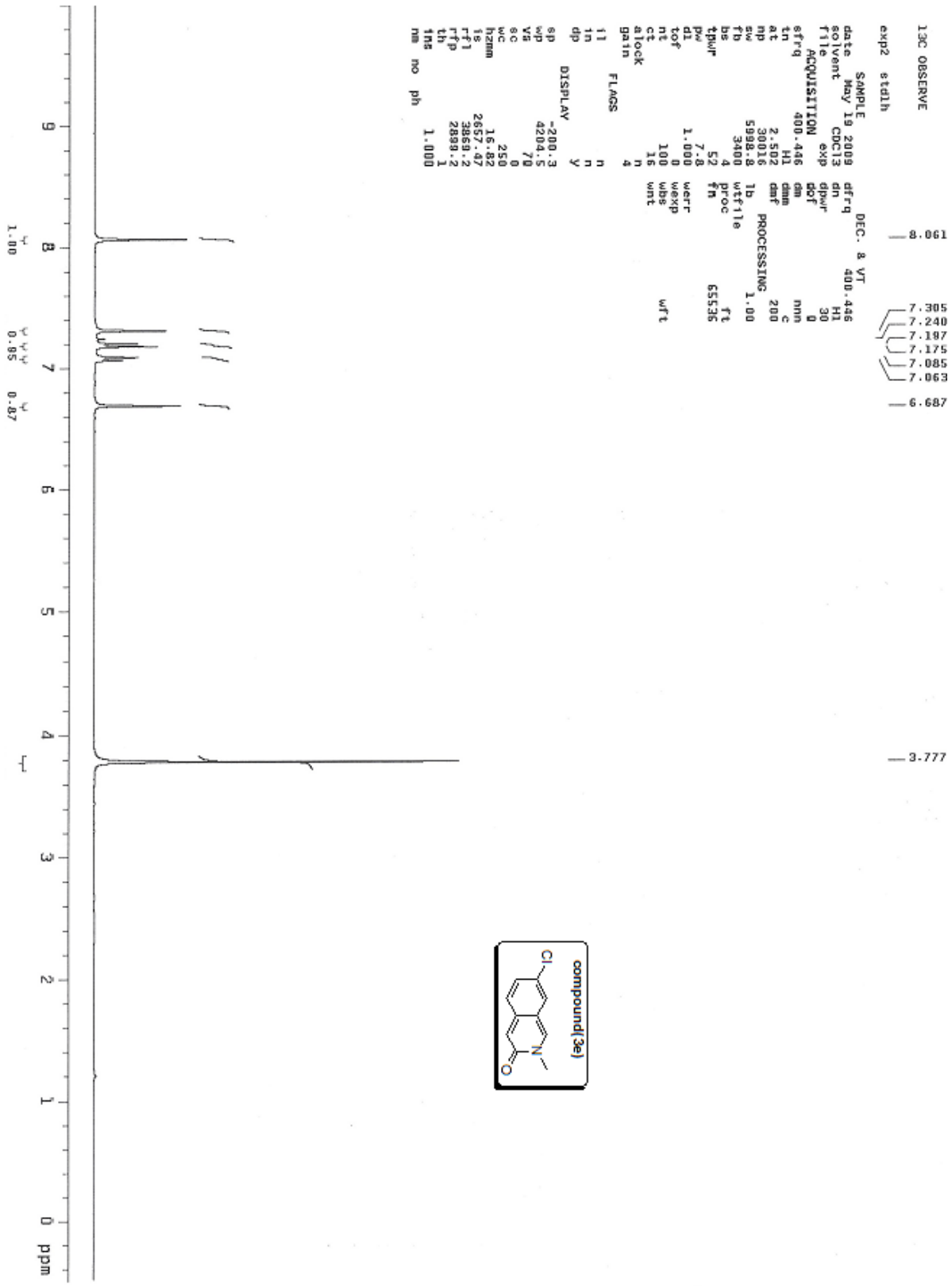

ppm
161.118
143.623
141.070
136.930
135.303
132.372
129.807
129.671
129.592
129.278
129.248
129.132
129.046
128.883
128.846
128.605
128.240
128.187
128.046
128.000
127.844
127.617
127.546
125.163
122.908
118.203
110.458
54.552
54.371
54.190
53.987
53.828
47.430
compound(3b)
N
Bn
O
ppm
180
160
140
120
100
80
60
40
20

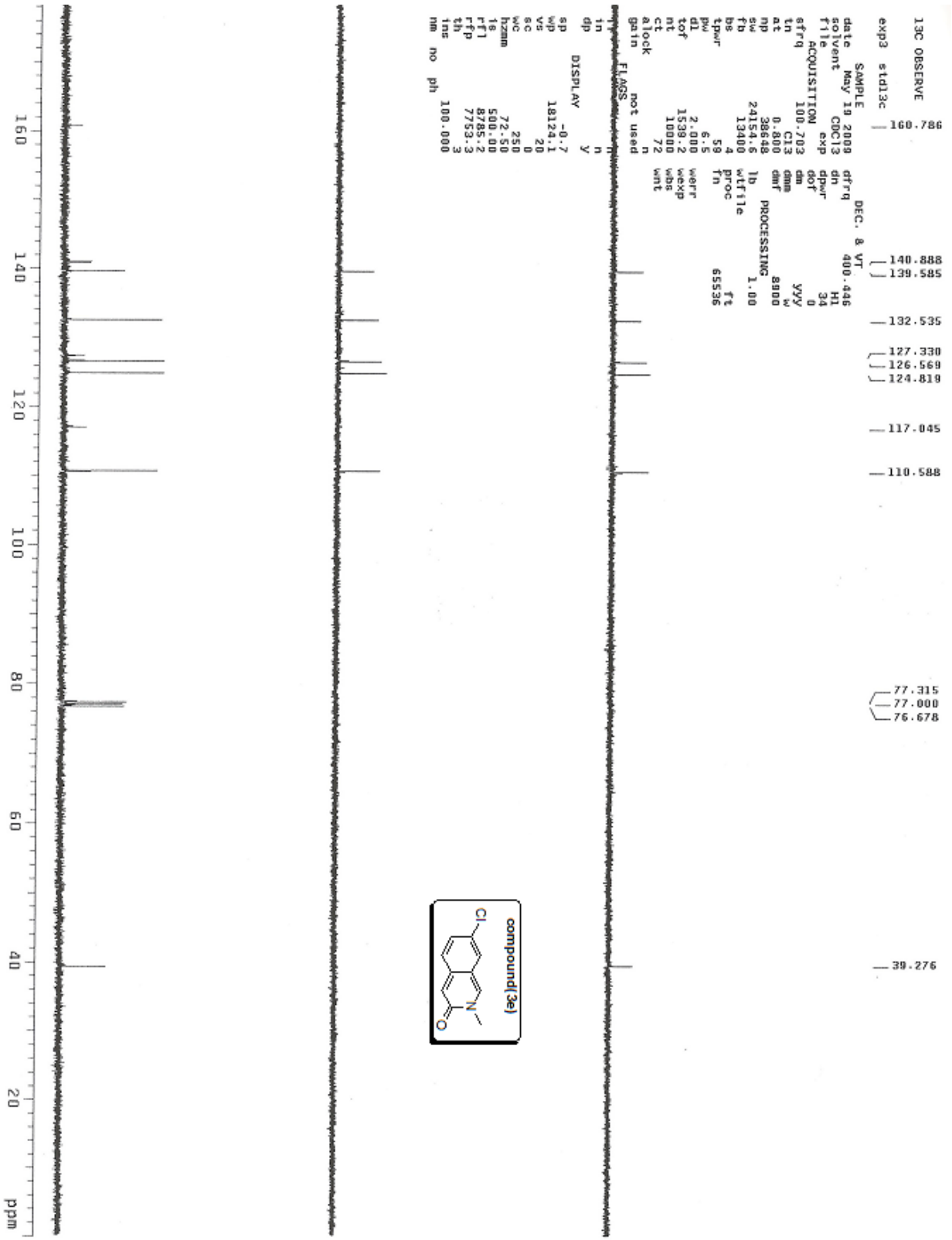

0.99
1.00
0.99
1.96
3.00
8.142
7.420
7.406
7.397
7.383
7.241
6.850
6.845
6.824
6.819
6.724
6.719
6.703
6.701
6.698
6.681
6.675
6.655
3.796
compound(3c)
F
N
O
ppm

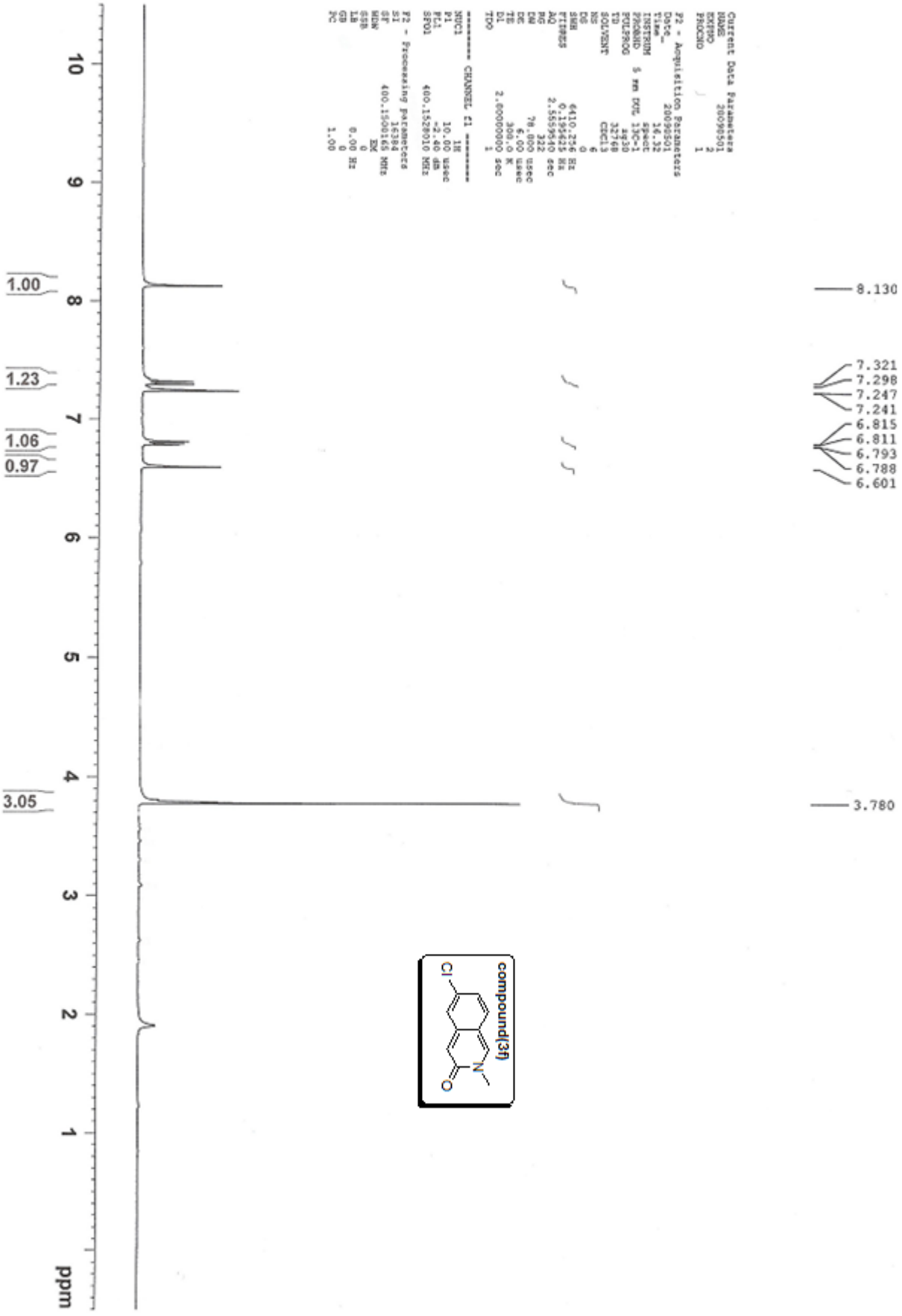

165.27
162.72
160.63
143.98
141.08
130.94
130.84
114.92
114.63
109.01
108.93
106.26
106.05
77.32
77.01
76.69
39.22
compound(3c)
F
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

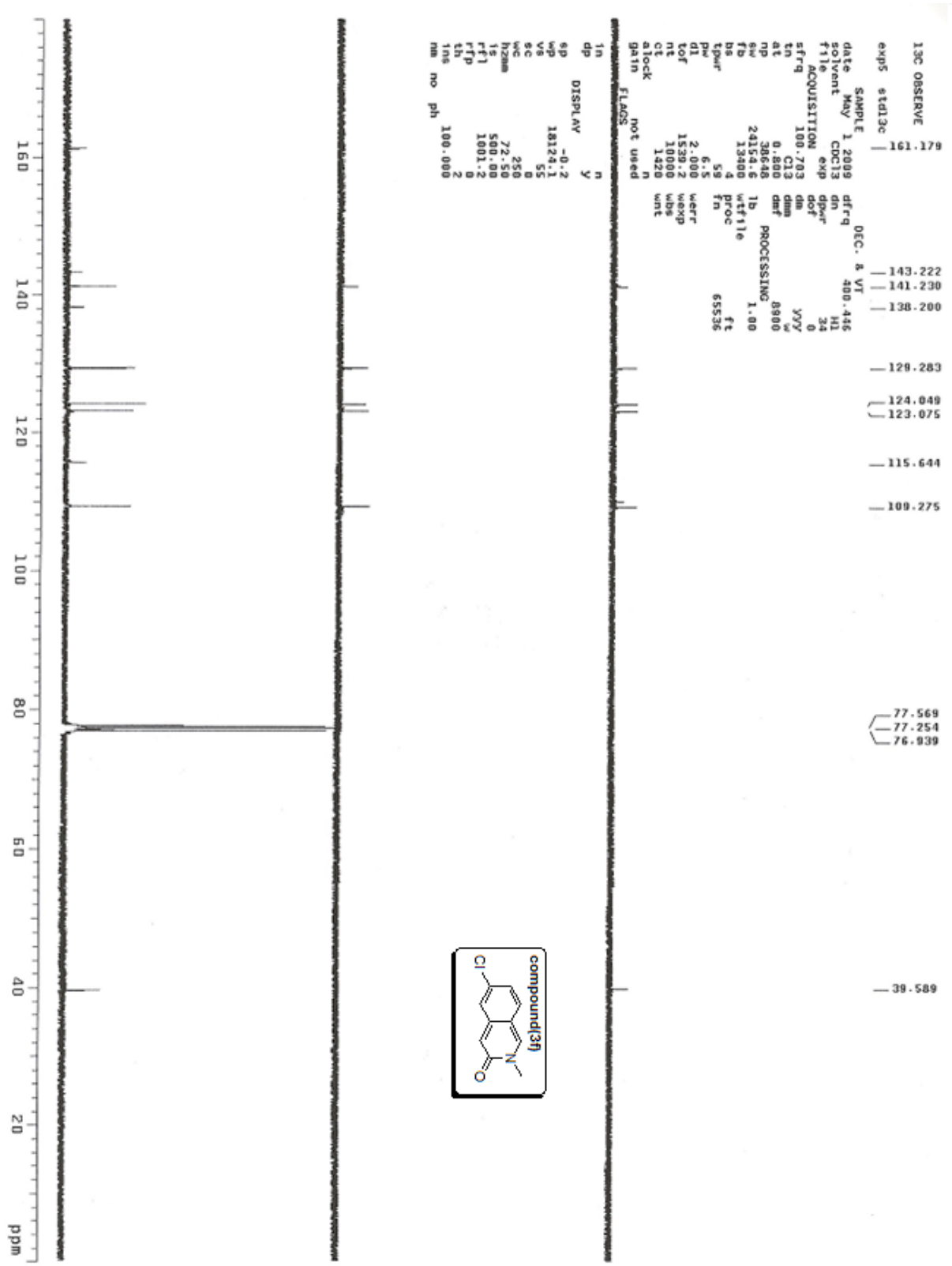

compound(3d)
8.067
7.299
7.286
7.276
7.262
7.082
7.076
7.055
6.971
6.966
6.948
6.760
3.785
3.463
3.294
1.219
ppm

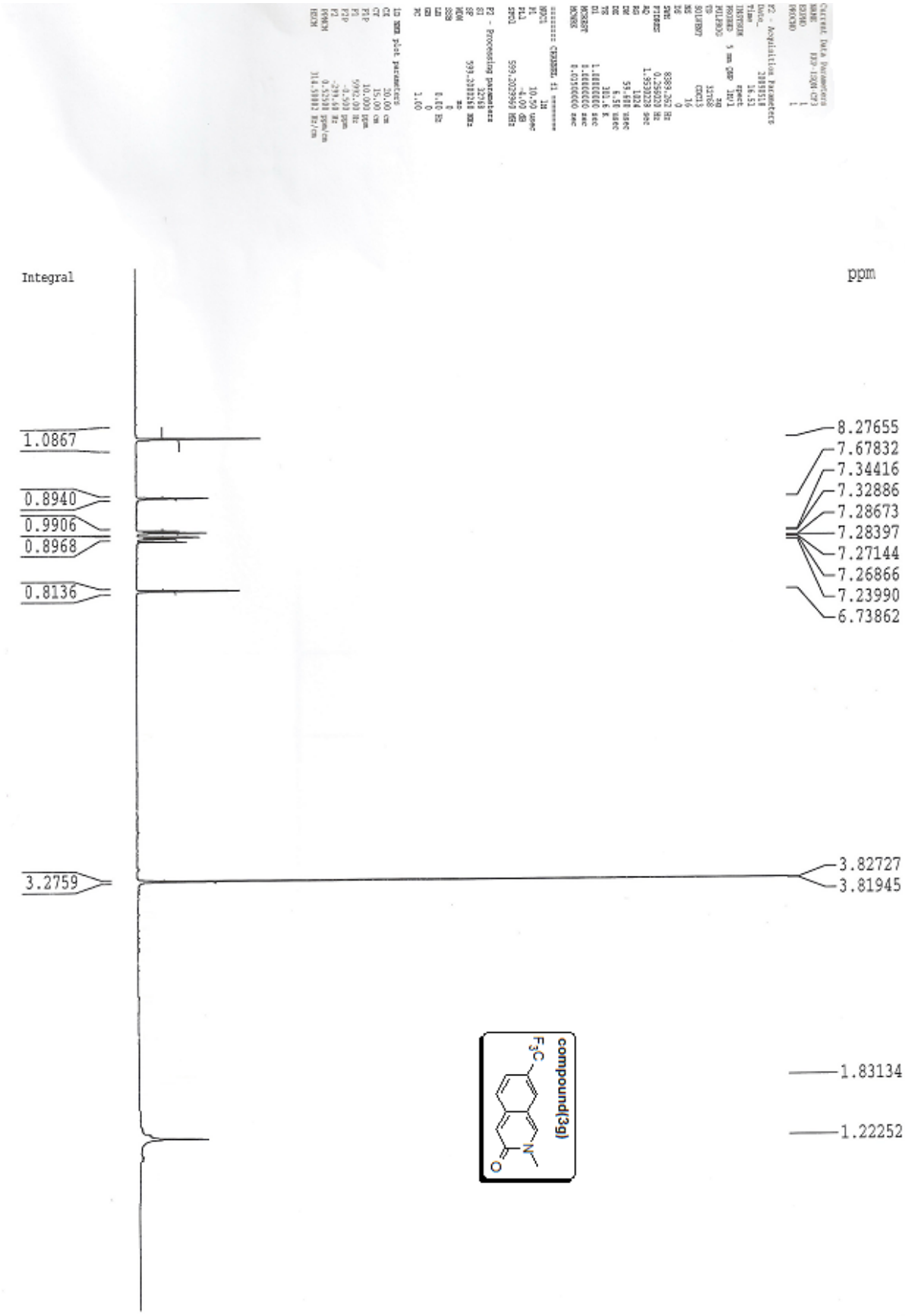

160.698
158.692
156.255
140.456
139.117
139.029
127.740
127.660
124.094
123.816
111.020
108.055
107.835
77.315
77.000
76.685
39.276
compound(3d)
F
O
N
160
140
120
100
80
60
40
20
ppm

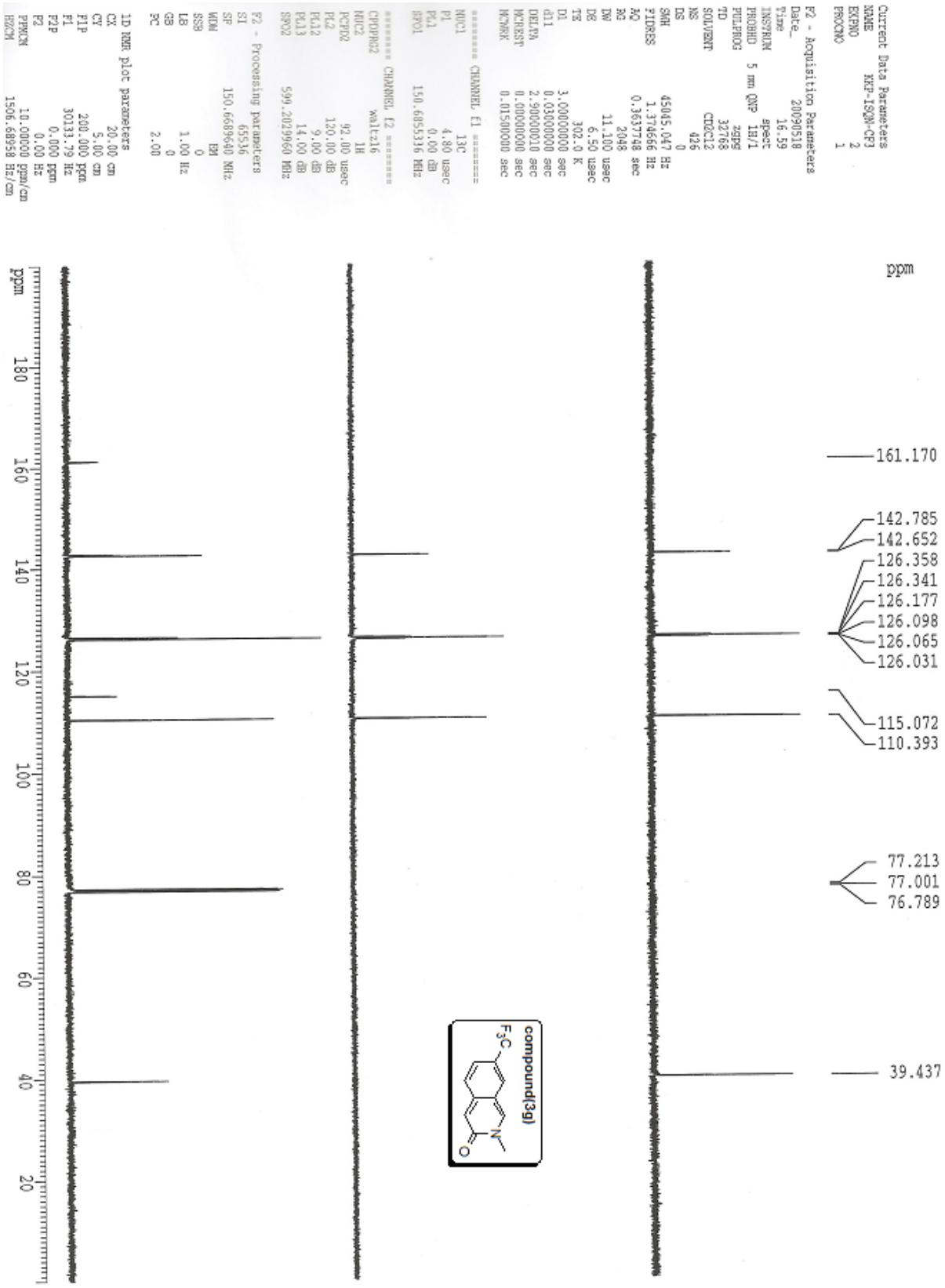

13C OBSERVE
exp2 std1h
SAMPLE
date May 19 2009
solvent CDCl3
file
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
d1 1.000
tof 0
nt 100
ct 16
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 70
sc 0
wc 250
hzmm 16.82
is 2657.47
rfl 3869.2
rfp 2889.1
th 1
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
wft
wnt
8.061
7.305
7.240
7.197
7.175
7.085
7.063
6.687
3.777
compound(3e)
Cl
N
O
ppm

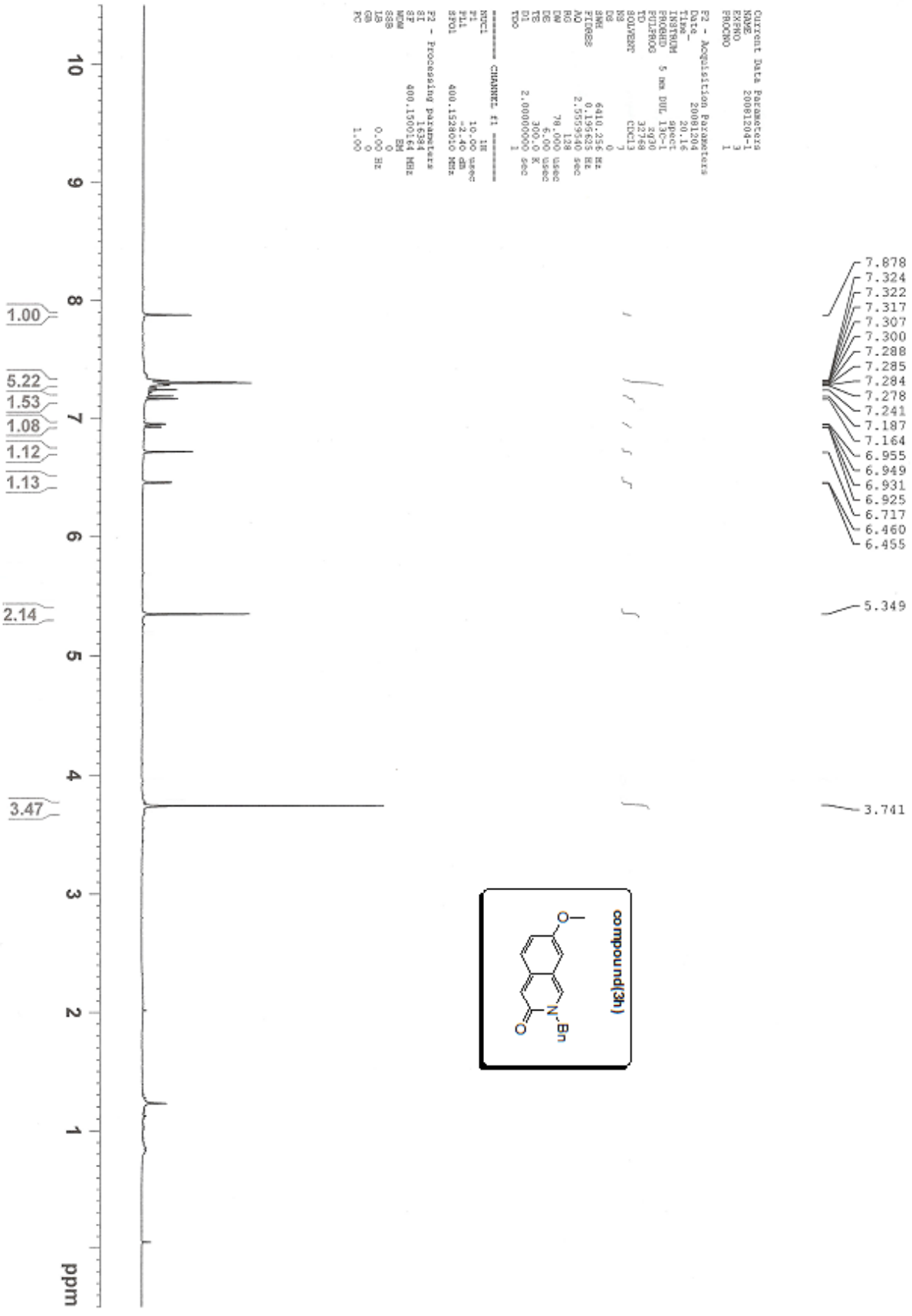

13C OBSERVE
exp3 std13c
SAMPLE
date May 19 2009
solvent CDCl3
file
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 72
ct
alock n
gain not used
FLAGS
in n
dp y
DISPLAY
sp -0.7
wp 18124.1
vs 20
sc 0
wc 250
hzmm 72.50
is 500.00
rfl 8785.2
rfp 7753.3
th 3
ins
nm no ph 100.000
DEC. & VT
dfrq
dn
dpwr
dof
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
160.786
140.888
139.585
132.535
127.330
126.569
124.819
117.045
110.588
77.315
77.000
76.678
39.276
compound[3e]
Cl
N
O
160
140
120
100
80
60
40
20
ppm

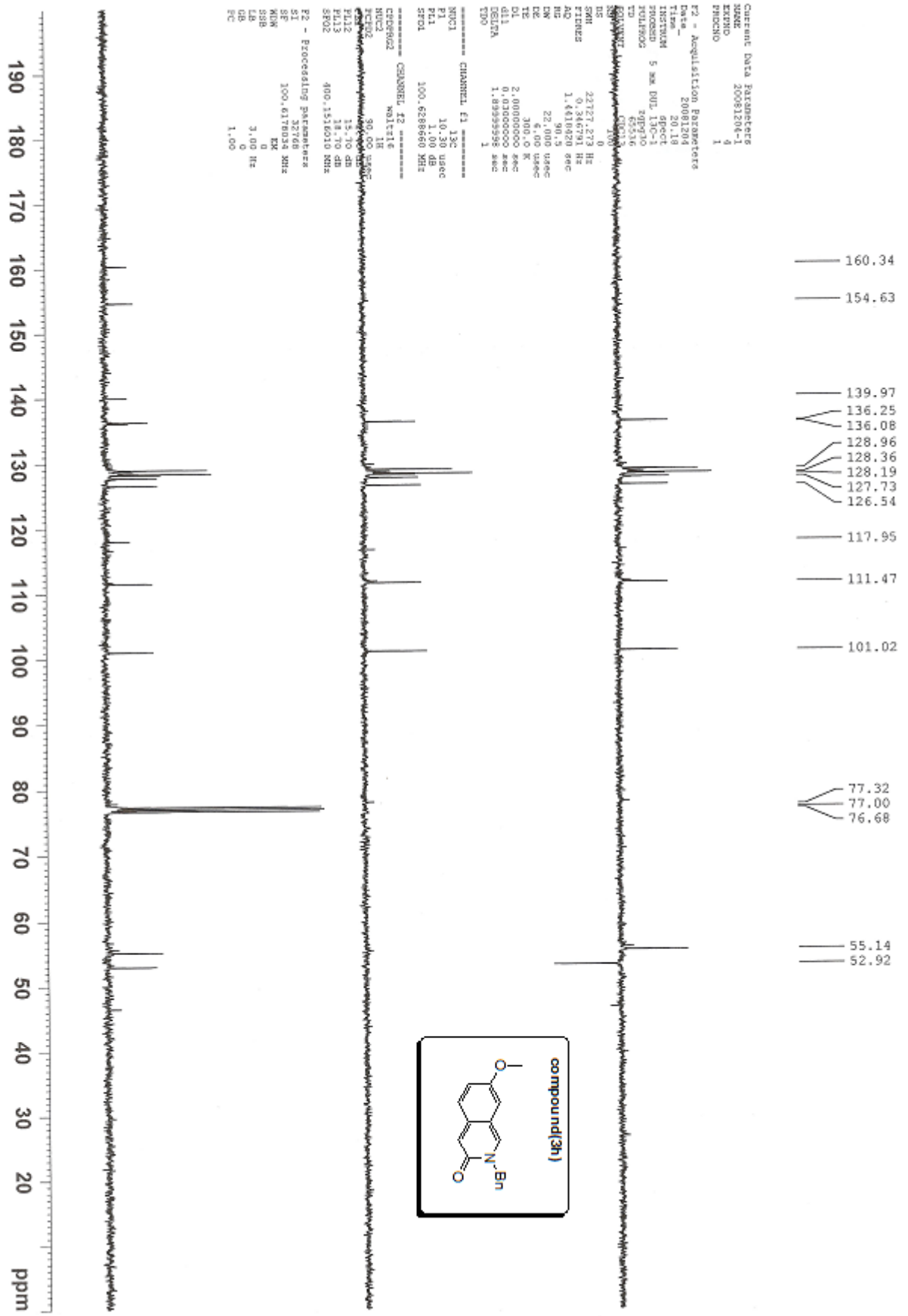

1.00
1.23
1.06
0.97
3.05
8.130
7.321
7.298
7.247
7.241
6.815
6.811
6.793
6.788
6.601
3.780
compound(3fl)
Cl
N
O
ppm

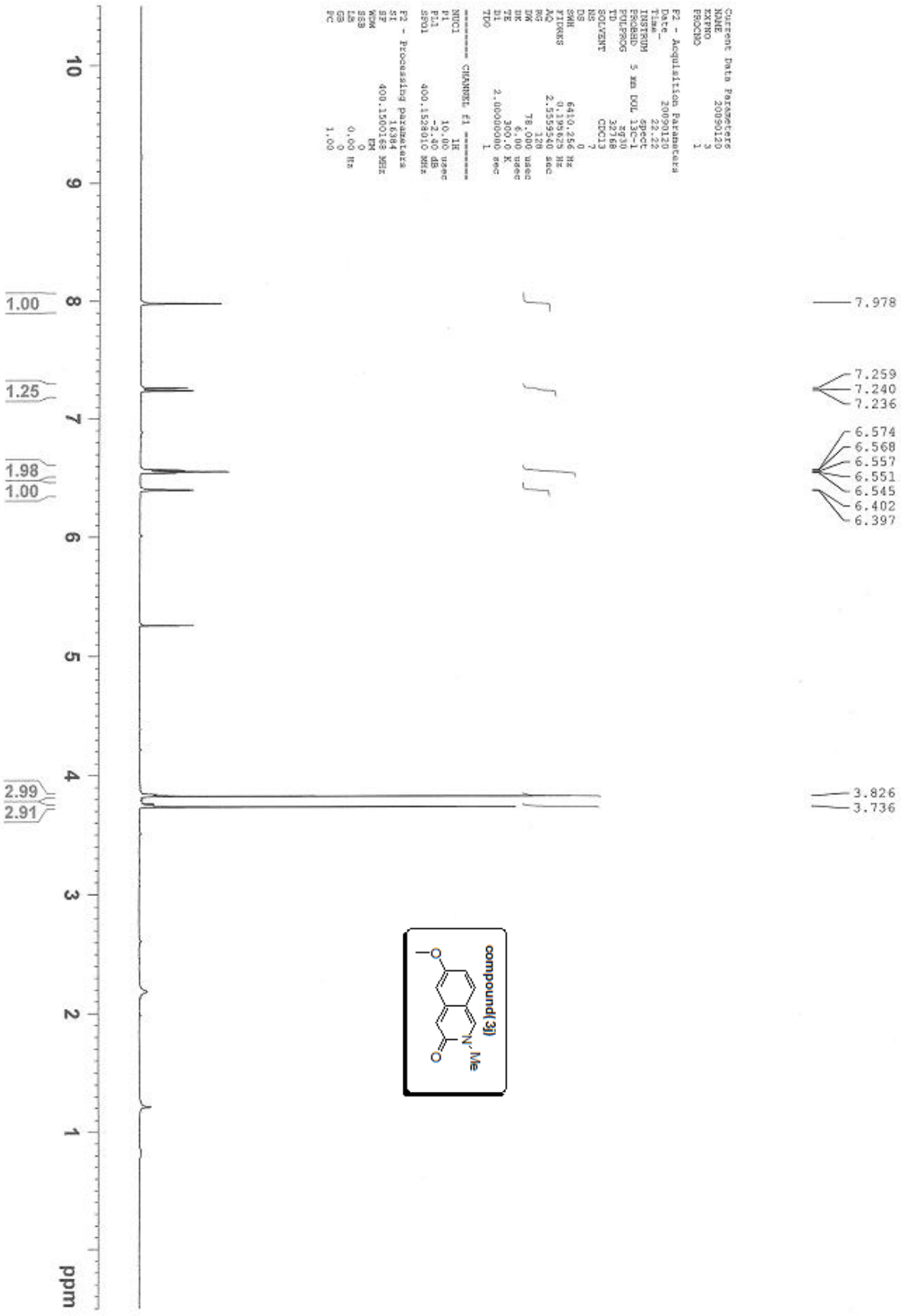

13C OBSERVE
exp5 std13c
SAMPLE
date May 1 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
dl 2.000
tof 1539.2
nt 10000
ct 1420
alock n
gain n
FLAGS
not used
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
wexp
wbs
wnt
in n
dp y
DISPLAY
sp -0.2
wp 18124.1
vs 55
sc 0
wc 250
hzmm 72.50
is 500.00
rfl 1001.2
rfp 0
th 2
ins
nm no ph 100.000
161.179
143.222
141.230
138.200
129.283
124.049
123.075
115.644
109.275
77.569
77.254
76.939
39.589
compound(3fl)
Cl
N
O
160
140
120
100
80
60
40
20
ppm

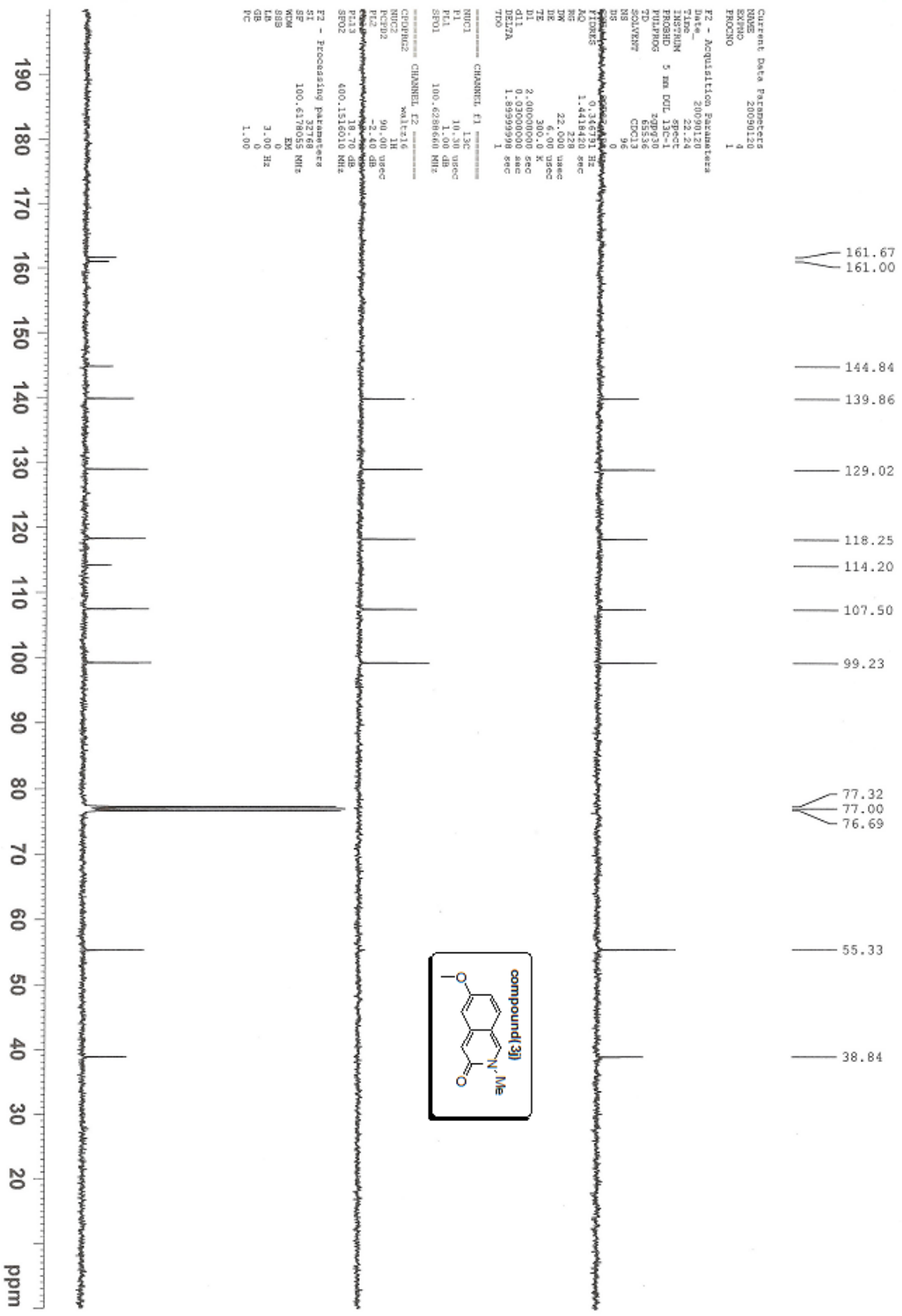

Integral
ppm
1.0867
0.8940
0.9906
0.8968
0.8136
3.2759
8.27655
7.67832
7.34416
7.32886
7.28673
7.28397
7.27144
7.26866
7.23990
6.73862
3.82727
3.81945
1.83134
1.22252
compound(3g)

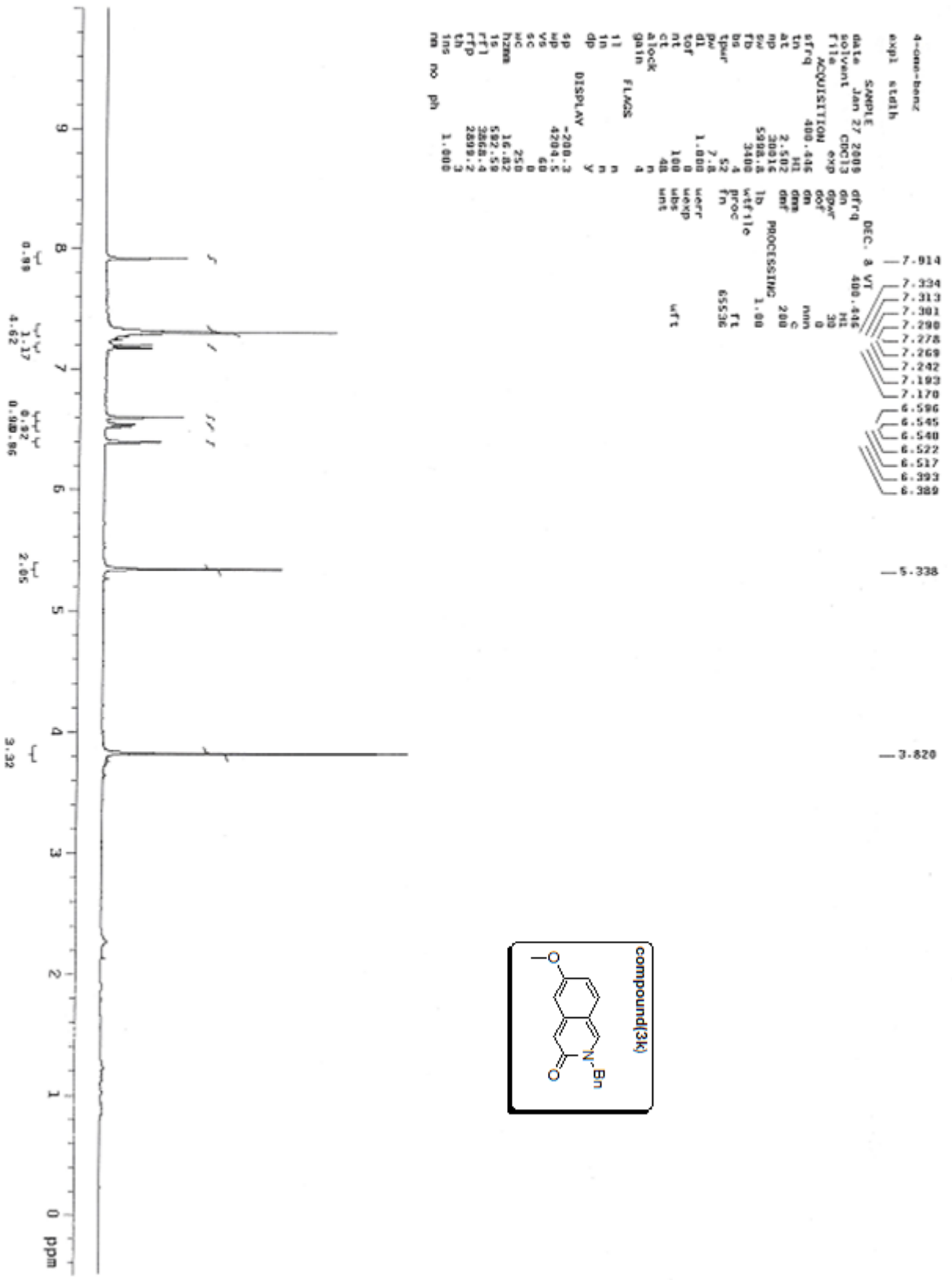

Current Data Parameters
NAME KKP-ISQN-CP3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 16.59
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 426
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 302.0 K
D1 3.00000000 sec
d11 0.03000000 sec
DELTA 2.90000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.6855336 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 599.2029960 MHz
F2 - Processing parameters
SI 65536
SF 150.6689640 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.000 ppm
F1 30133.79 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1506.68958 Hz/cm
ppm
180
160
140
120
100
80
60
40
20
161.170
142.785
142.652
126.358
126.341
126.177
126.098
126.065
126.031
115.072
110.393
77.213
77.001
76.789
39.437
compound(3g)
F3C
N
O

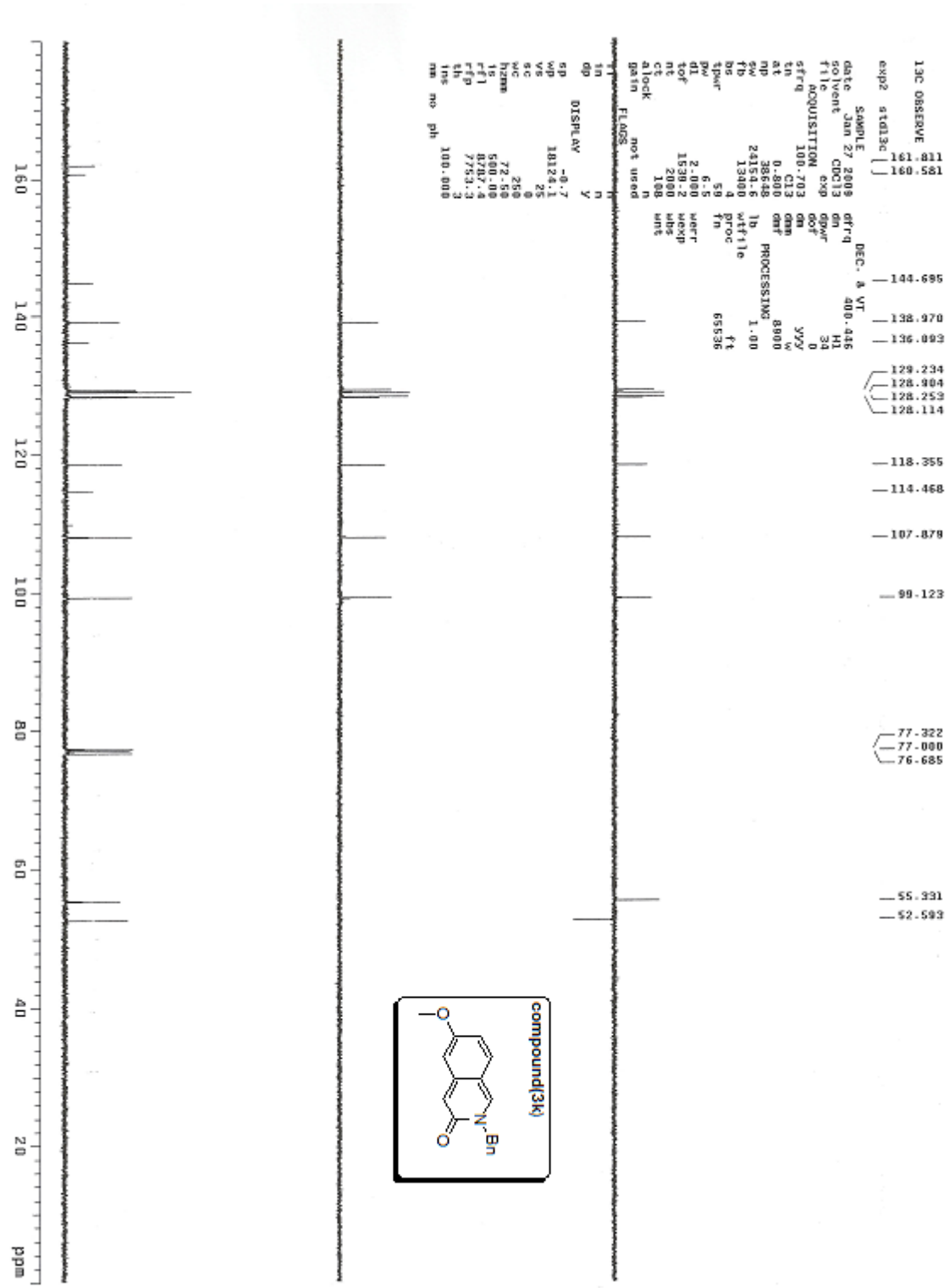

1.00
5.22
1.53
1.08
1.12
1.13
2.14
3.47
ppm
7.878
7.324
7.322
7.317
7.307
7.300
7.288
7.285
7.284
7.278
7.241
7.187
7.164
6.955
6.949
6.931
6.925
6.717
6.460
6.455
5.349
3.741
compound(3h)
O
N
Bn
O

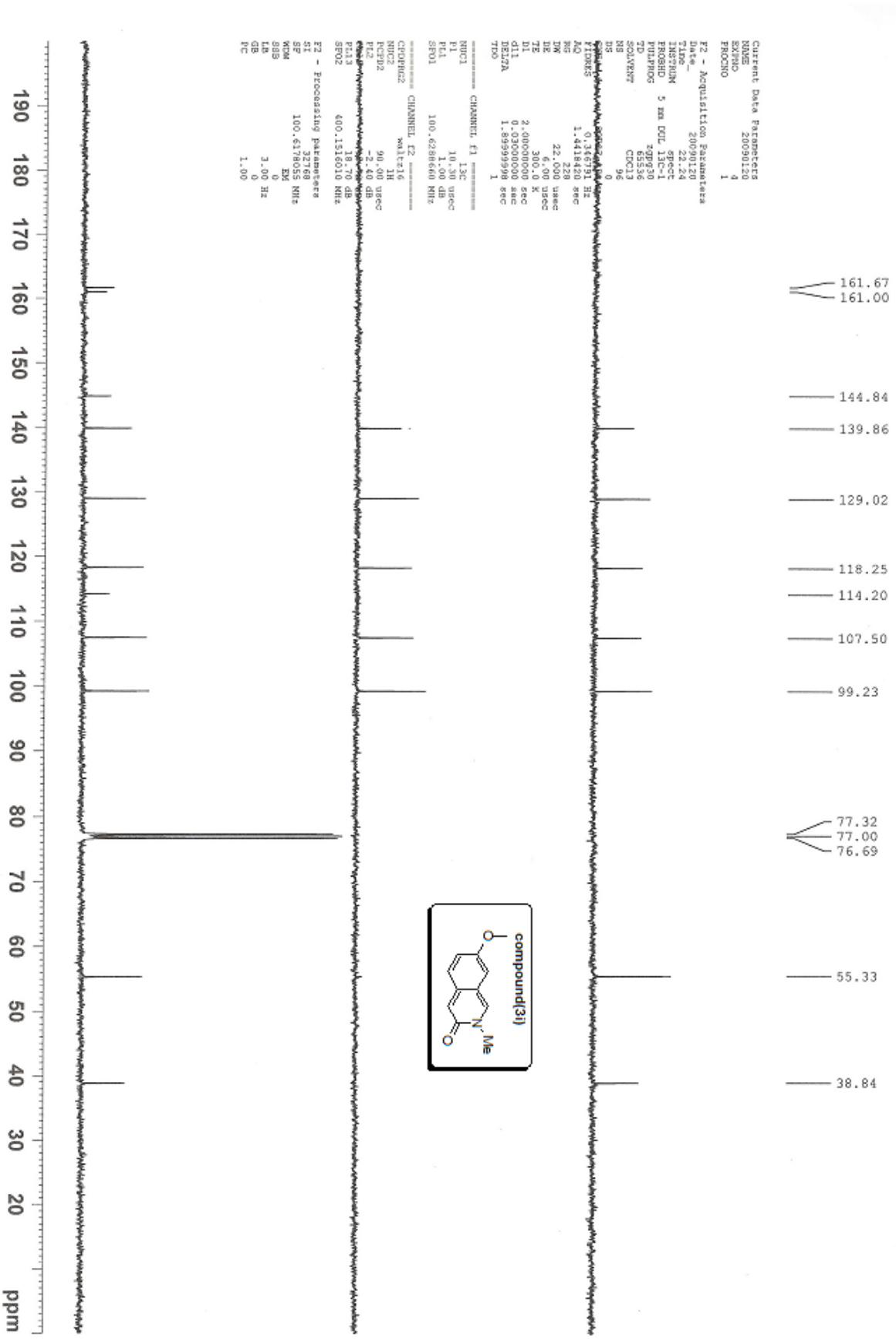

160.34
154.63
139.97
136.25
136.08
128.96
128.36
128.19
127.73
126.54
117.95
111.47
101.02
77.32
77.00
76.68
55.14
52.92
compound(3h)
O
N
Bn
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

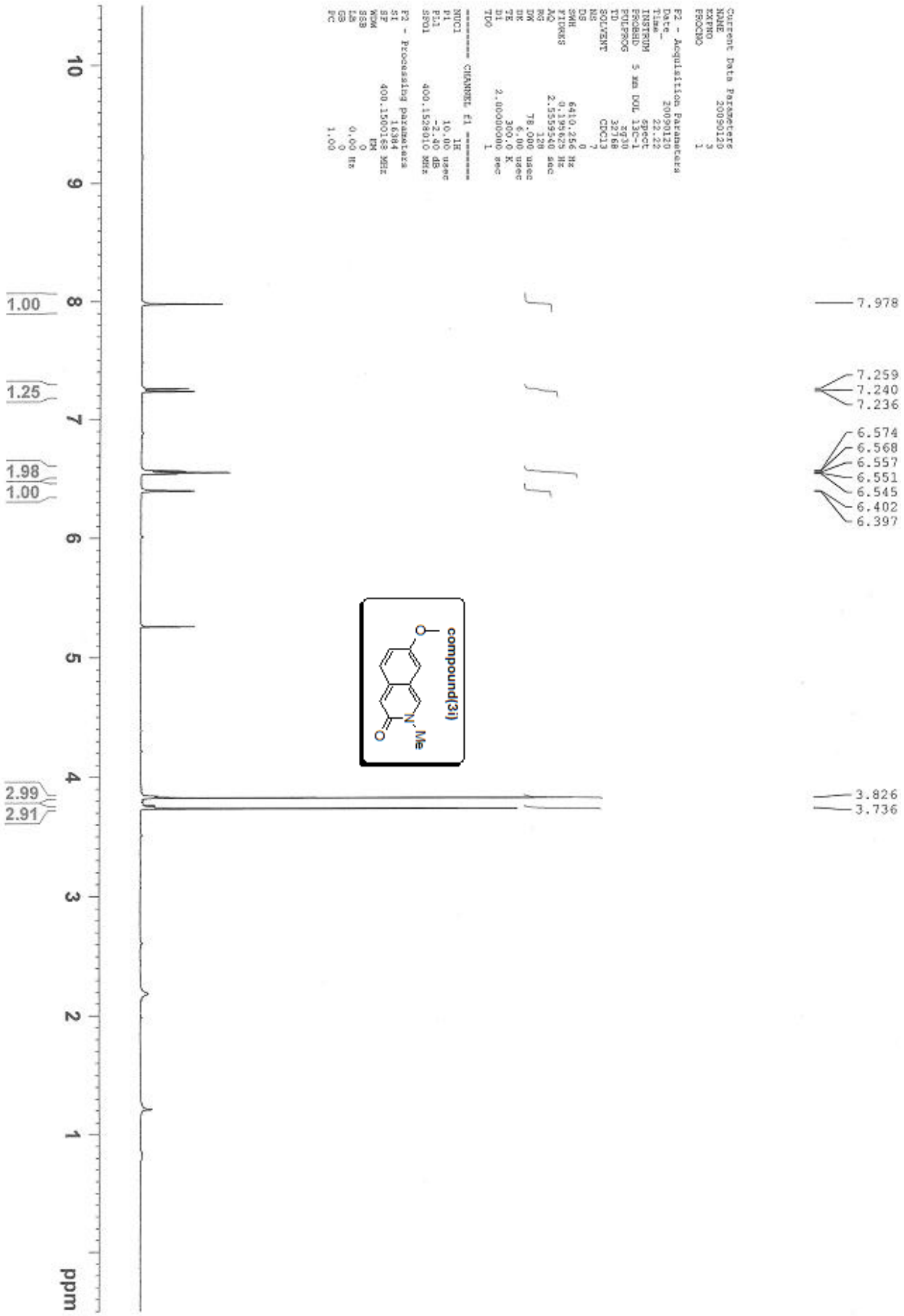

compound(3j)
N
Me
O
O
1.00
1.25
1.98
1.00
2.99
2.91
7.978
7.259
7.240
7.236
6.574
6.568
6.557
6.551
6.545
6.402
6.397
3.826
3.736
ppm

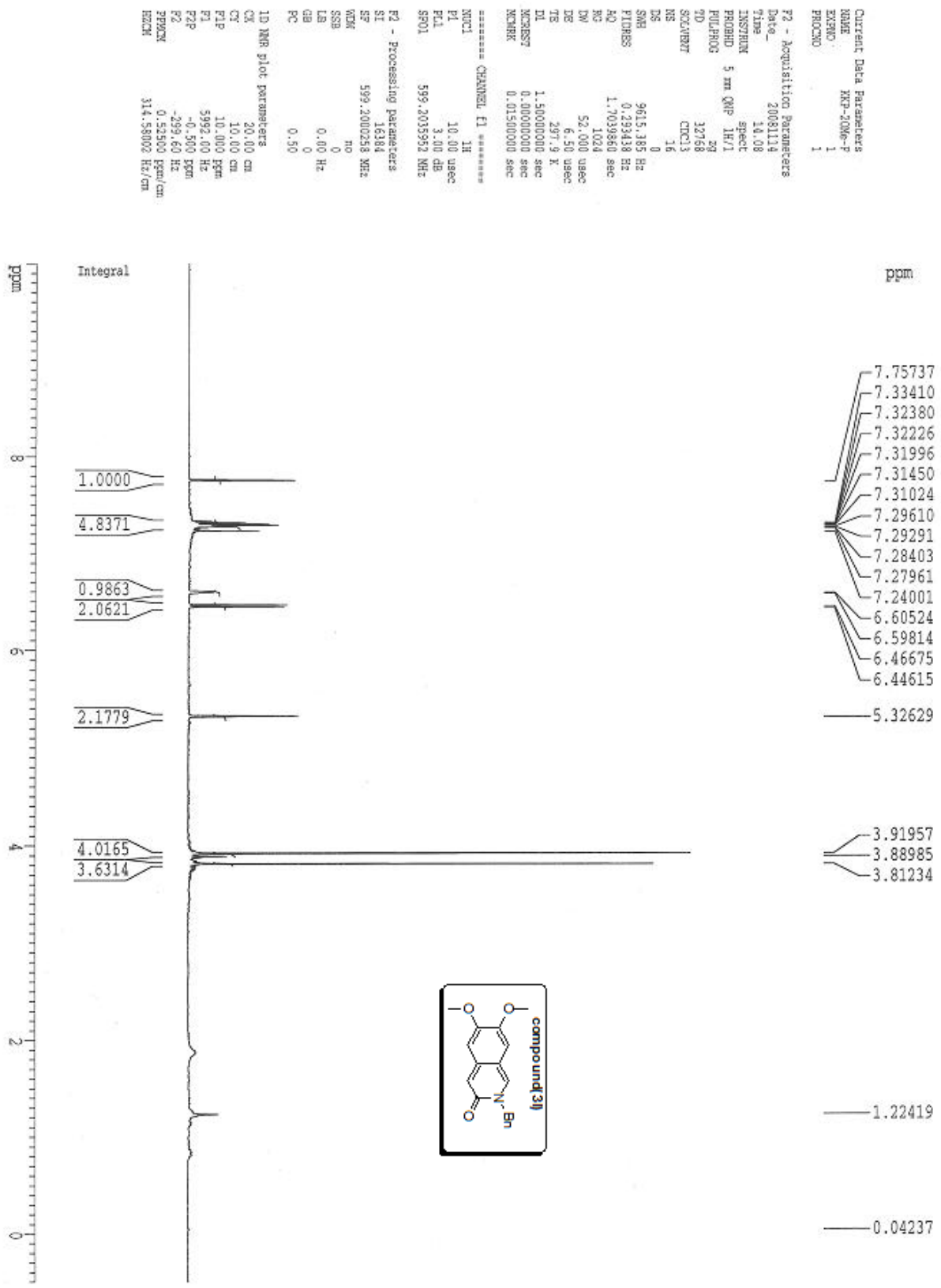

161.67
161.00
144.84
139.86
129.02
118.25
114.20
107.50
99.23
77.32
77.00
76.69
55.33
38.84
compound(3j)
N
Me
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

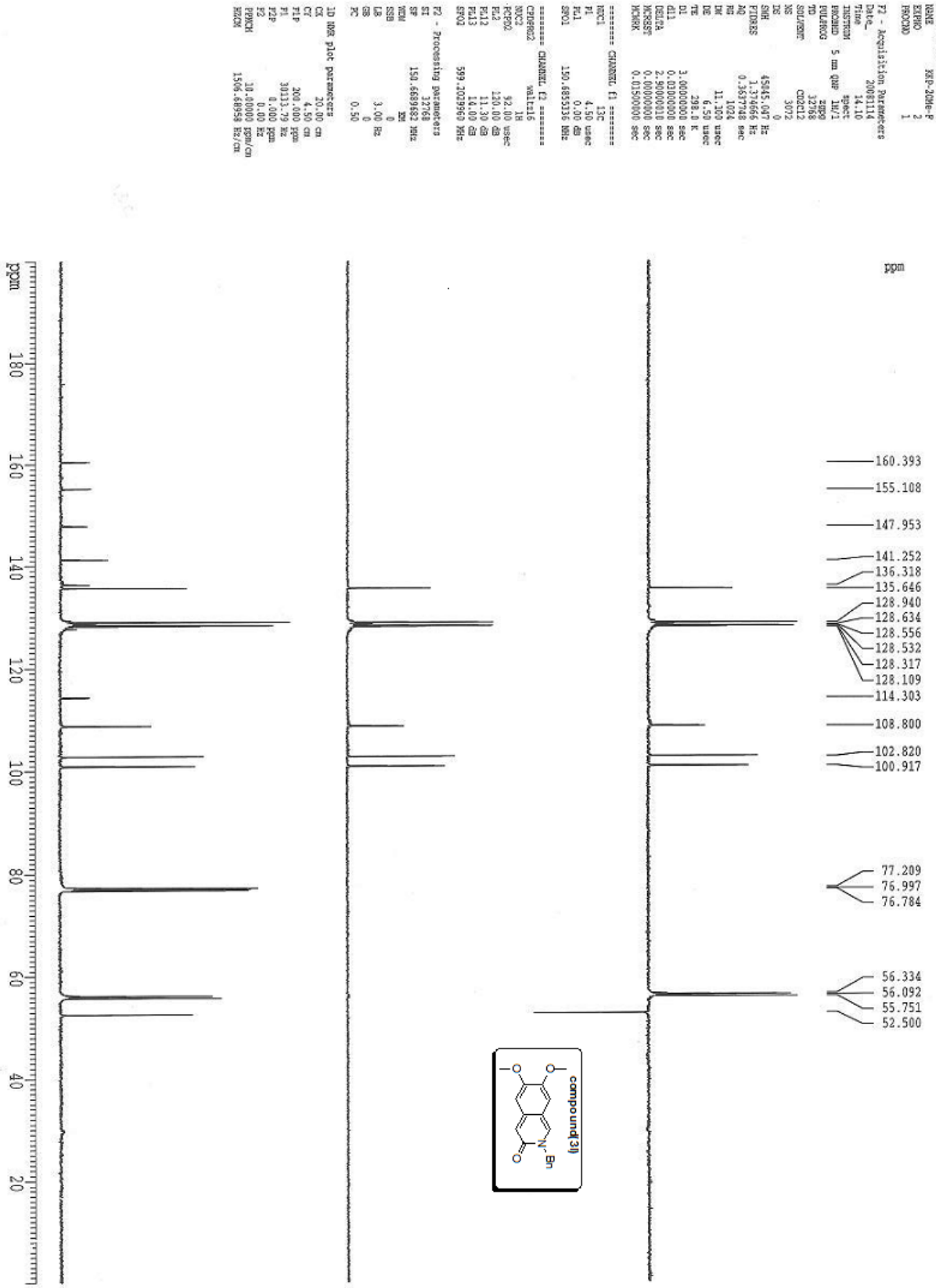

4-ome-benz
expl stdih
SAMPLE
date Jan 27 2009
solvent CDCl3
file
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
fw 5988.8
fb 3408
bs 4
tpwr 52
pw 7.8
d1 1.000
tof 0
nt 100
ct 48
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -208.3
wp 4204.5
vs 69
sc 0
wc 250
hzmm 16.82
is 592.59
rfl 3868.4
rfp 2889.2
th 3
ins 1.000
nm no ph
dfrq DEC. 8 VT
400.446
H1
30
0
nnn
c 200
PROCESSING 1.00
lb
wtfile
proc ft
fn 65536
wft
wexp
wbs
wnt
7.914
7.334
7.313
7.301
7.290
7.278
7.269
7.242
7.193
7.170
6.596
6.545
6.540
6.522
6.517
6.393
6.389
5.338
3.820
compound(3k)
O
N
Bn
O
ppm

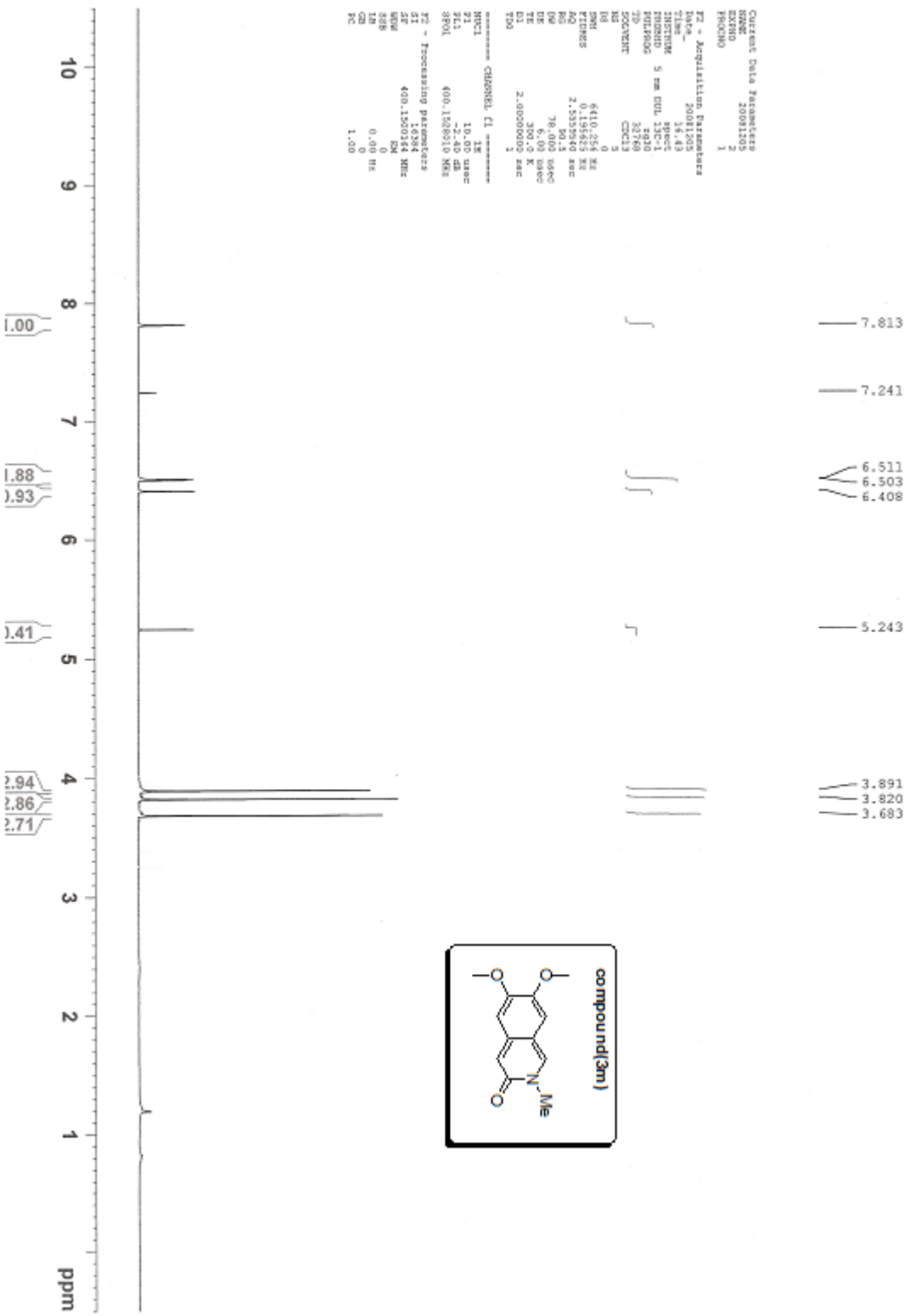

compound(3k)
O
N
Bn
O
160
140
120
100
80
60
40
20
ppm

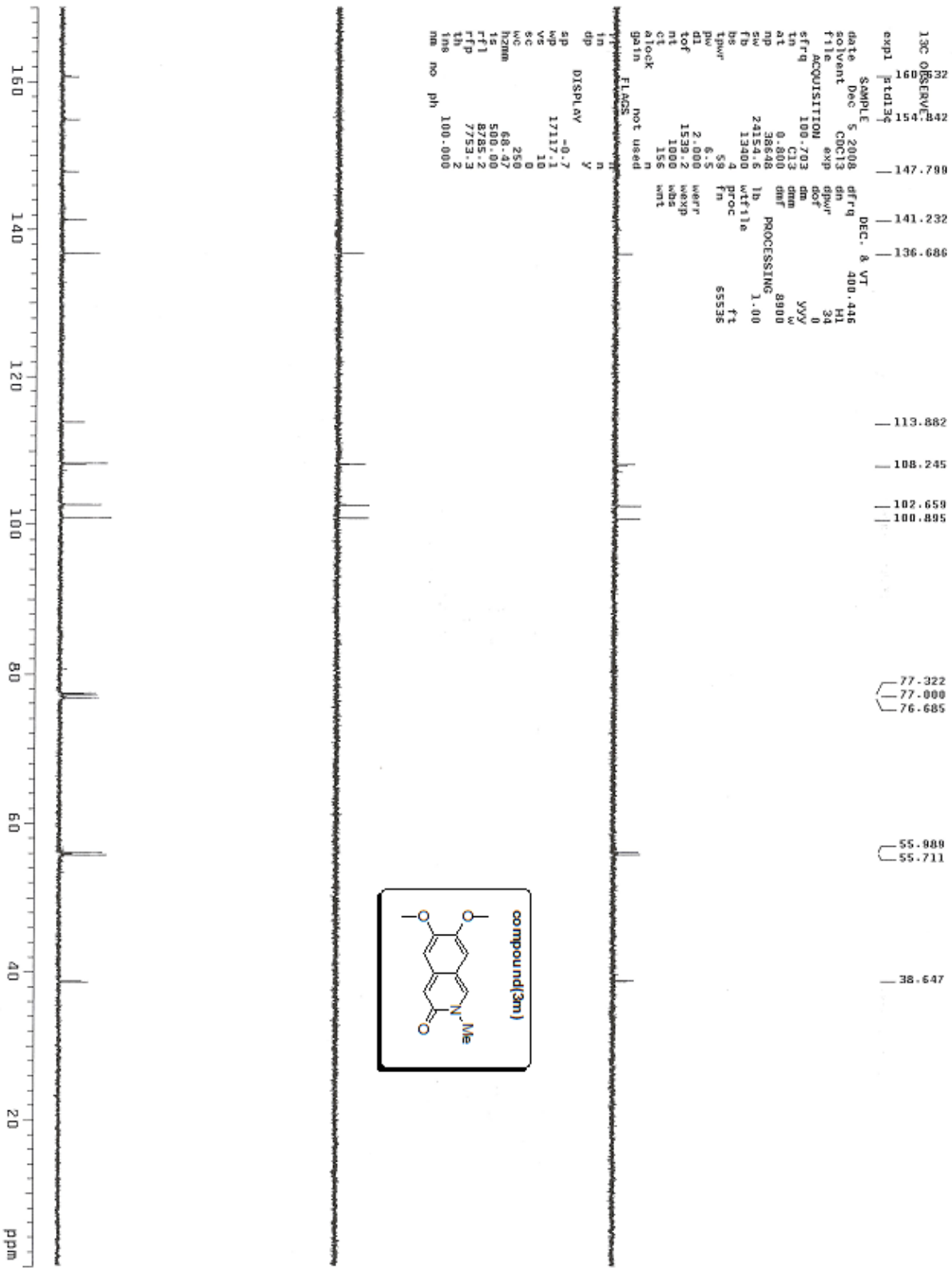

161.67
161.00
144.84
139.86
129.02
118.25
114.20
107.50
99.23
77.32
77.00
76.69
55.33
38.84
compound(3i)
O
N
Me
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

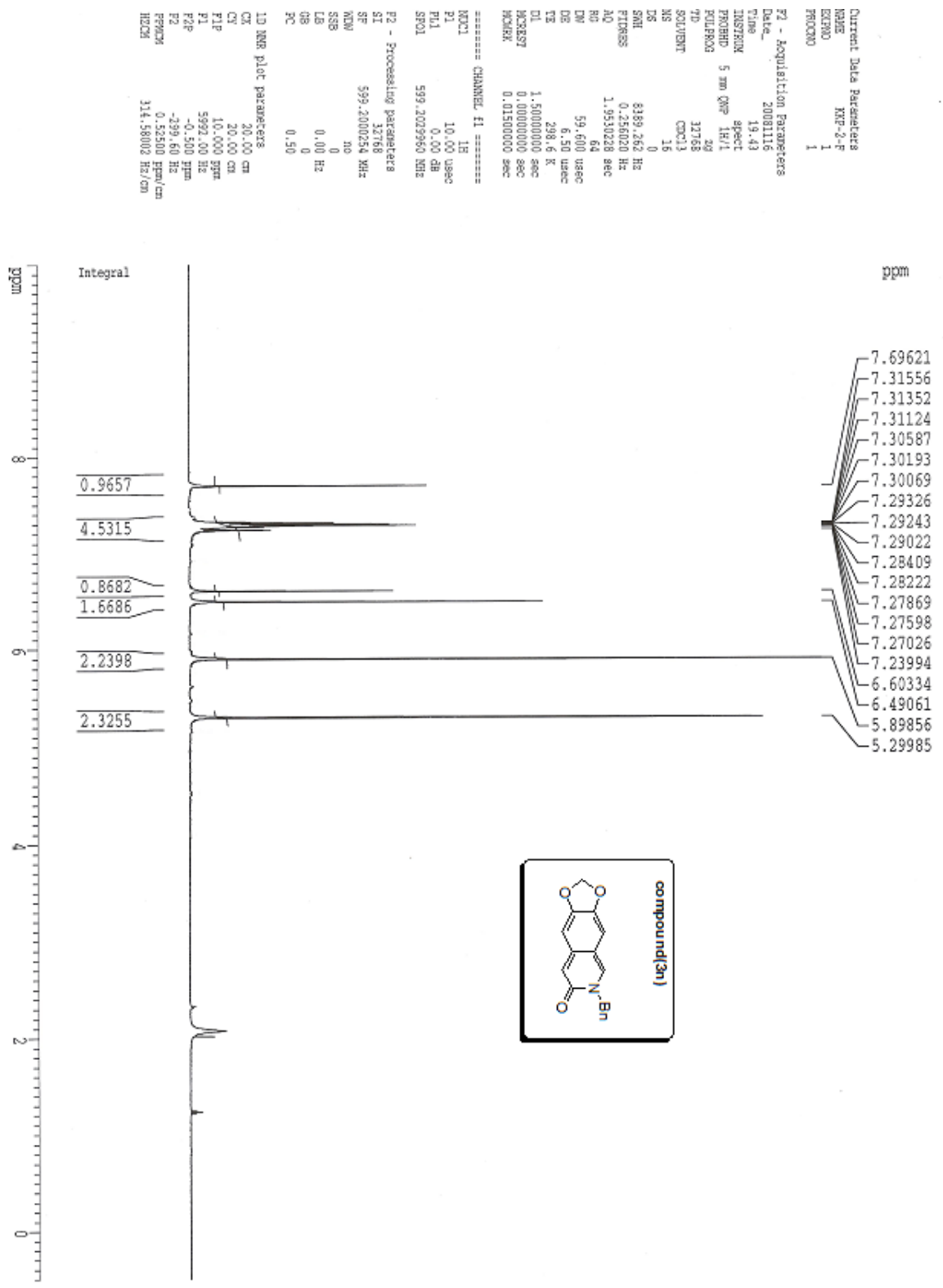

compound(3i)
O
N
Me
O
7.978
7.259
7.240
7.236
6.574
6.568
6.557
6.551
6.545
6.402
6.397
3.826
3.736
1.00
1.25
1.98
1.00
2.99
2.91
ppm

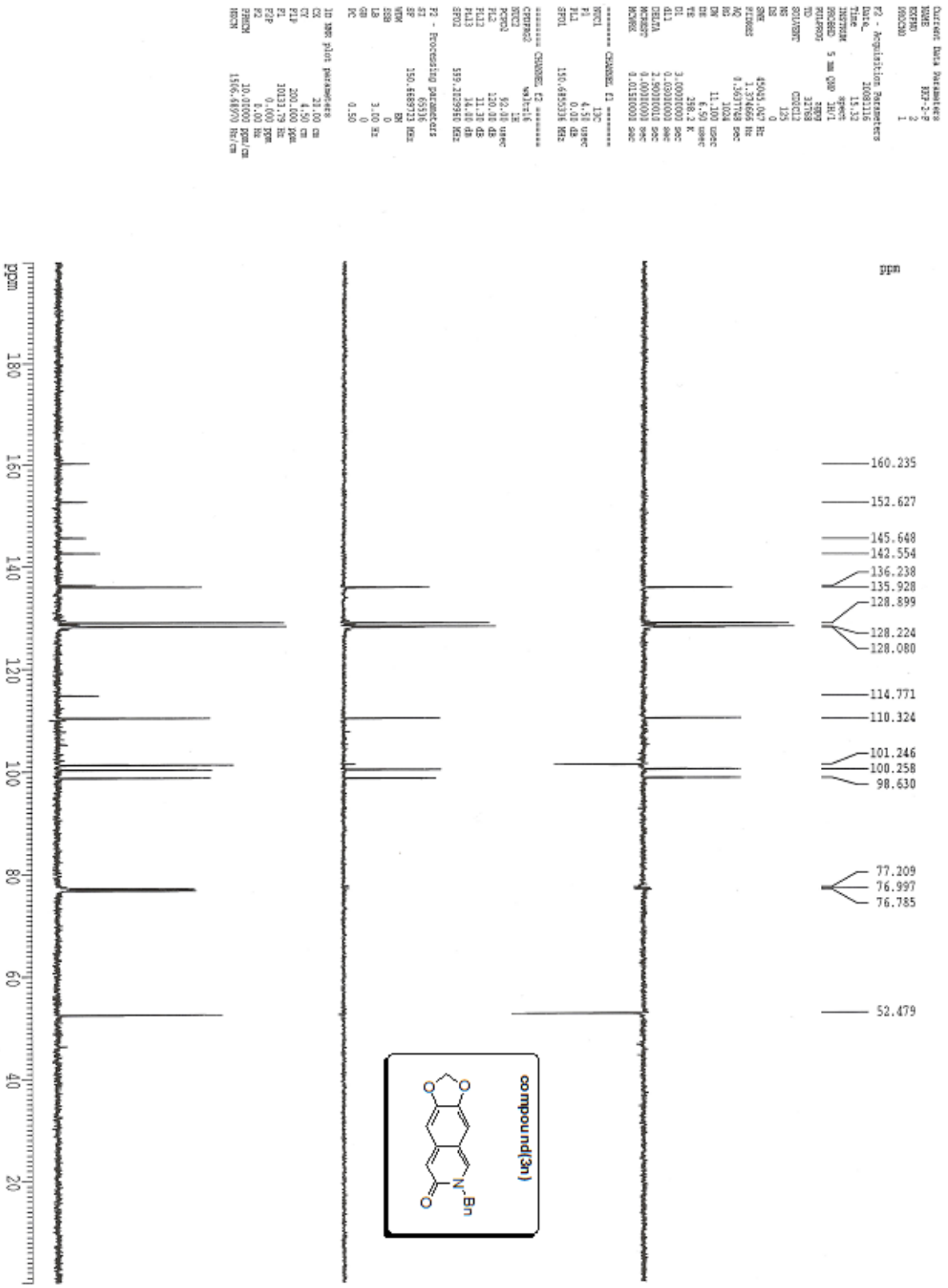

Current Data Parameters
NAME XKP-2OMe-P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081114
Time 14.08
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 1024
DW 52.000 usec
DE 6.50 usec
TE 297.9 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 599.2035952 MHz
F2 - Processing parameters
SI 16384
SF 599.2000258 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 5992.00 Hz
F2P -0.500 ppm
F2 -299.60 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.58002 Hz/cm
Integral
ppm
1.0000
4.8371
0.9863
2.0621
2.1779
4.0165
3.6314
7.75737
7.33410
7.32380
7.32226
7.31996
7.31450
7.31024
7.29610
7.29291
7.28403
7.27961
7.24001
6.60524
6.59814
6.46675
6.44615
5.32629
3.91957
3.88985
3.81234
1.22419
0.04237
compound(3l)
O
O
N
Bn
O

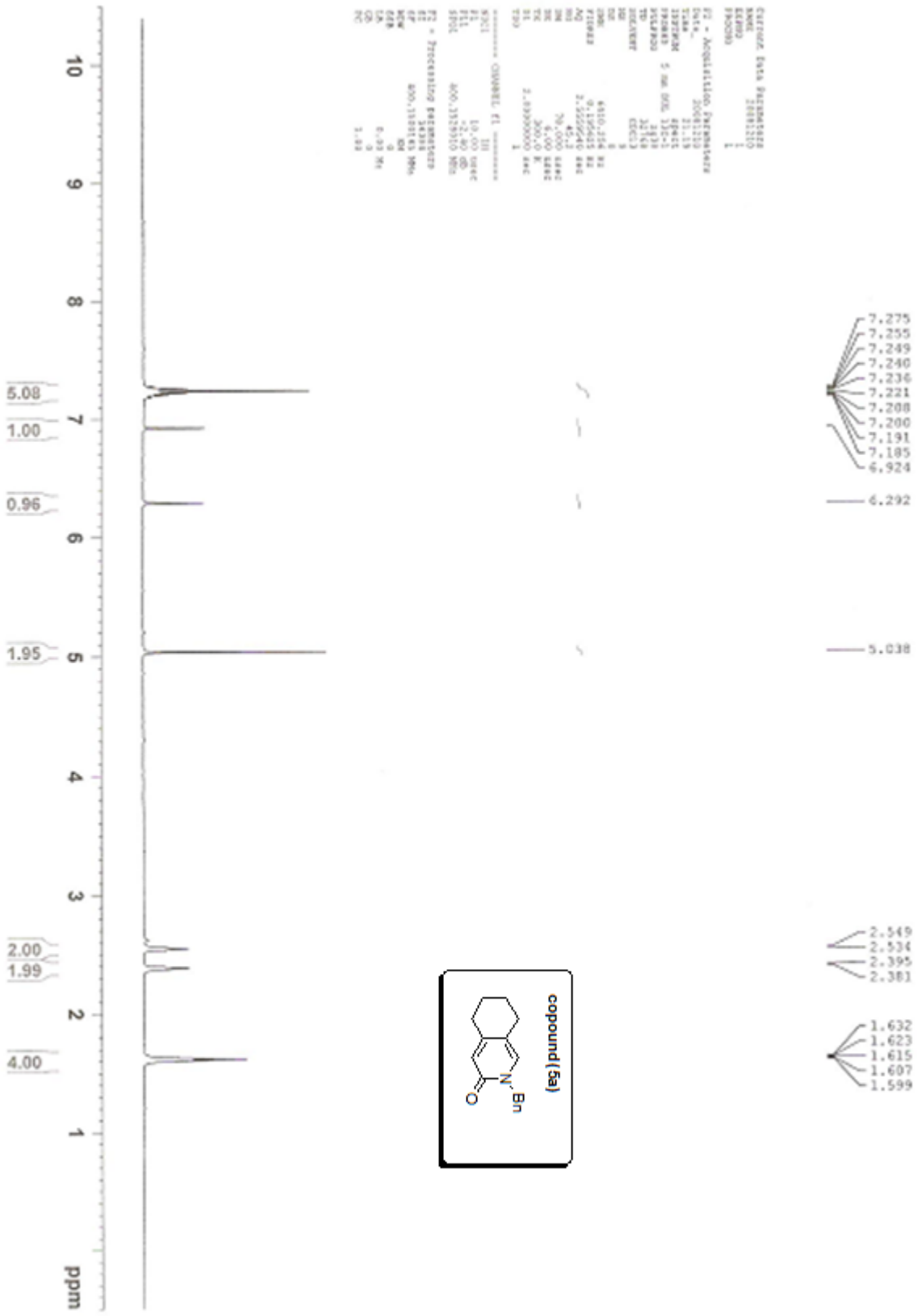

Current Data Parameters
NAME XEP-20Me-P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081114
Time 14.10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 3072
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 1024
DW 11.100 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
d11 0.03000000 sec
DELTA 2.90000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.50 usec
PL1 0.00 dB
SFO1 150.6853336 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 11.30 dB
PL13 14.00 dB
SFO2 599.2023960 MHz
F2 - Processing parameters
SI 32768
SF 150.6689682 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
CY 4.50 cm
F1P 200.000 ppm
F2P 30133.79 Hz
F1 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1506.68958 Hz/cm
ppm
160.393
155.108
147.953
141.252
136.318
135.646
128.940
128.634
128.556
128.532
128.317
128.109
114.303
108.800
102.820
100.917
77.209
76.997
76.784
56.334
56.092
55.751
52.500
compound 3j
N
Bn
O
O
O

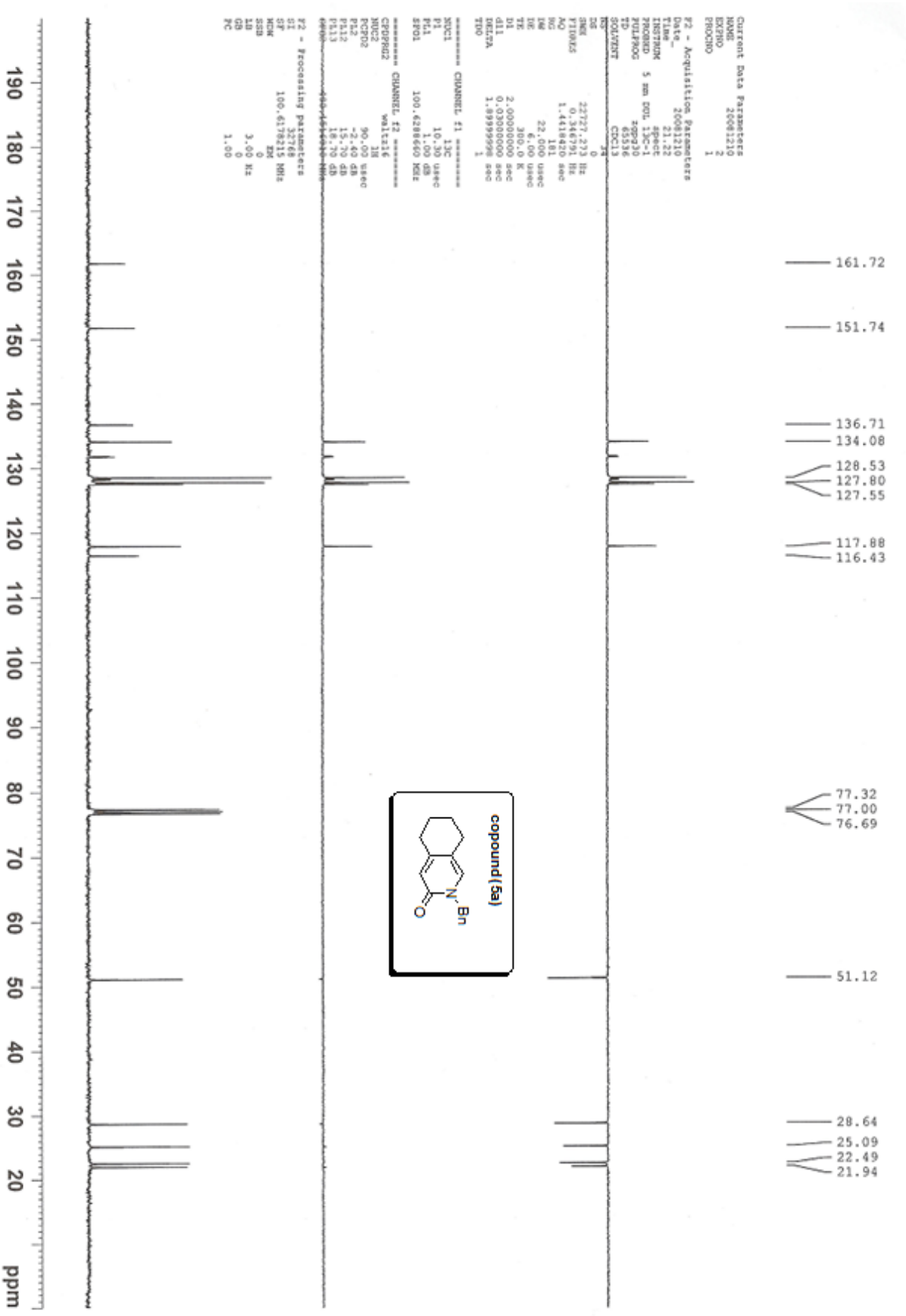

7.813
7.241
6.511
6.503
6.408
5.243
3.891
3.820
3.683
1.00
1.88
0.93
0.41
2.94
2.86
2.71
compound(3m)
Me
O
O
N
O
ppm

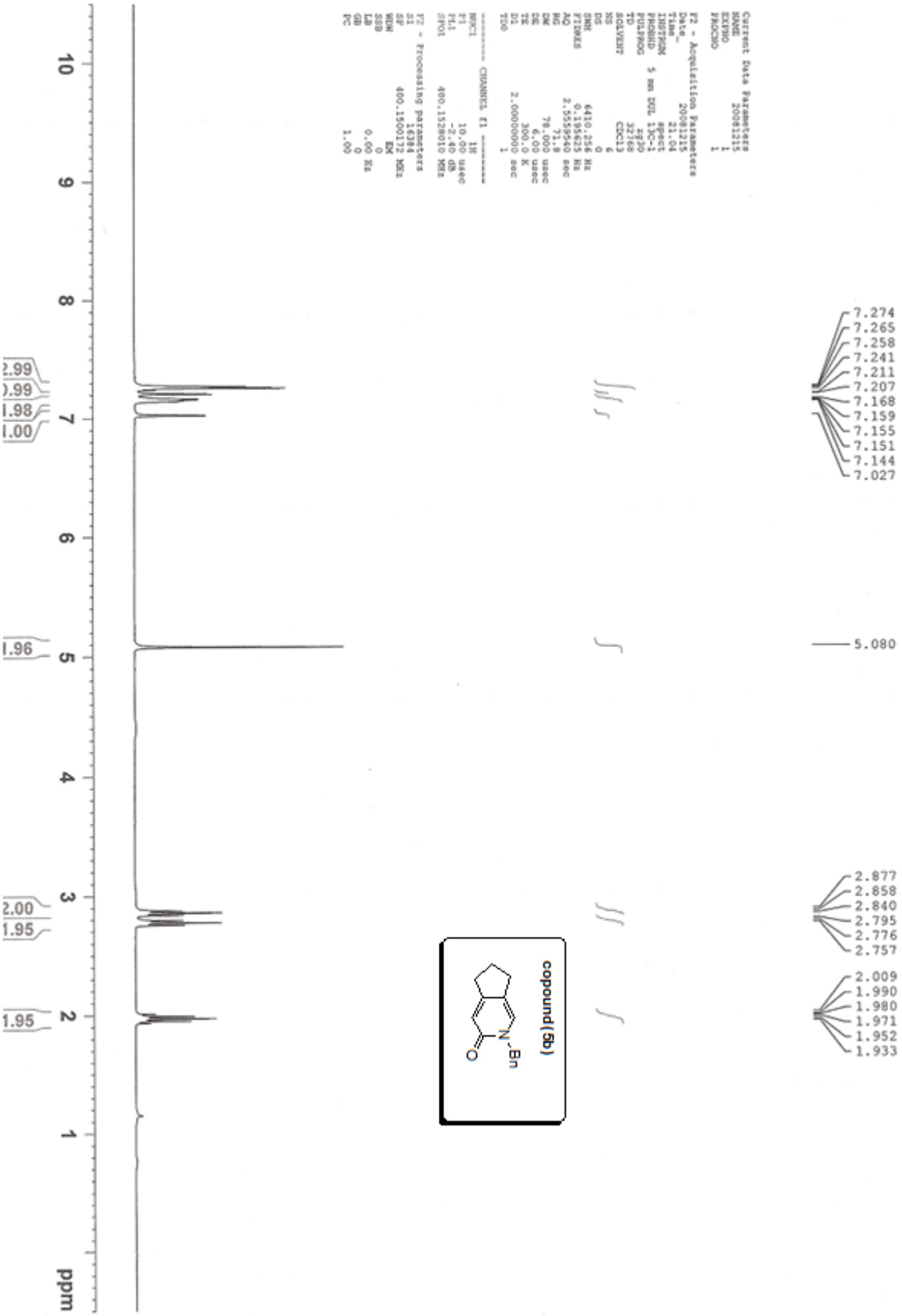

160.832
154.842
147.700
141.232
136.686
113.882
108.245
102.659
100.895
77.322
77.000
76.685
55.989
55.711
38.647
compound(3m)
N
Me
O
O
O
160
140
120
100
80
60
40
20
ppm

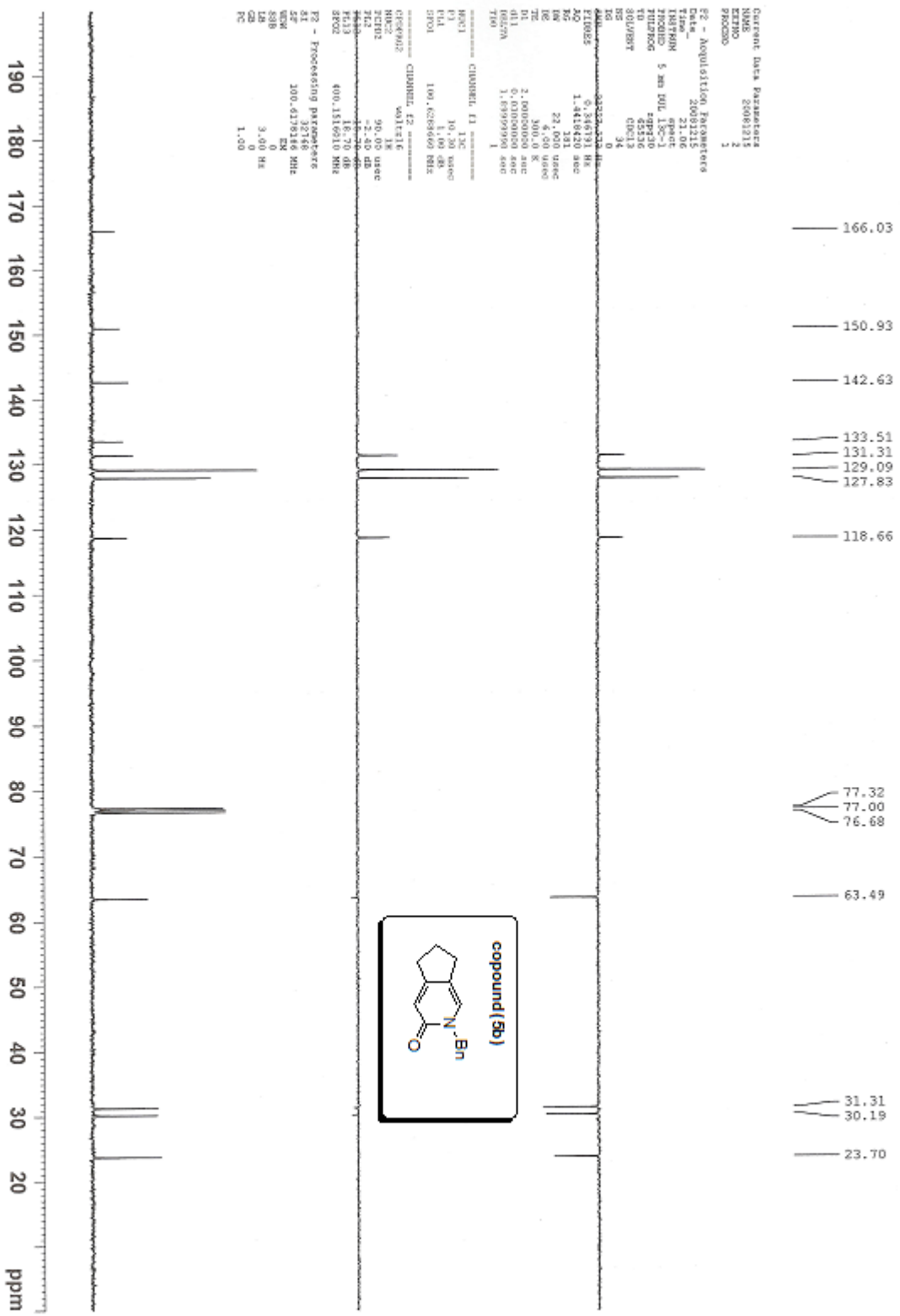

Current Data Parameters
NAME KXP-2-F
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081116
Time 19.43
INSTRUM spect
PROBHD 5 mm CNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 59.600 usec
DE 6.50 usec
TE 298.6 K
D1 1.50000000 sec
MCREST 0.10000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 599.2029960 MHz
F2 - Processing parameters
SI 32768
SF 599.2000254 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 10.000 ppm
F1 5992.00 Hz
F2P -0.500 ppm
F2 -299.60 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.58002 Hz/cm
Integral
ppm
0.9657
4.5315
0.8682
1.6686
2.2398
2.3255
7.69621
7.31556
7.31352
7.31124
7.30587
7.30193
7.30069
7.29326
7.29243
7.29022
7.28409
7.28222
7.27869
7.27598
7.27026
7.23994
6.60334
6.49061
5.89856
5.29985
compound(3n)
O
O
N
Bn
O

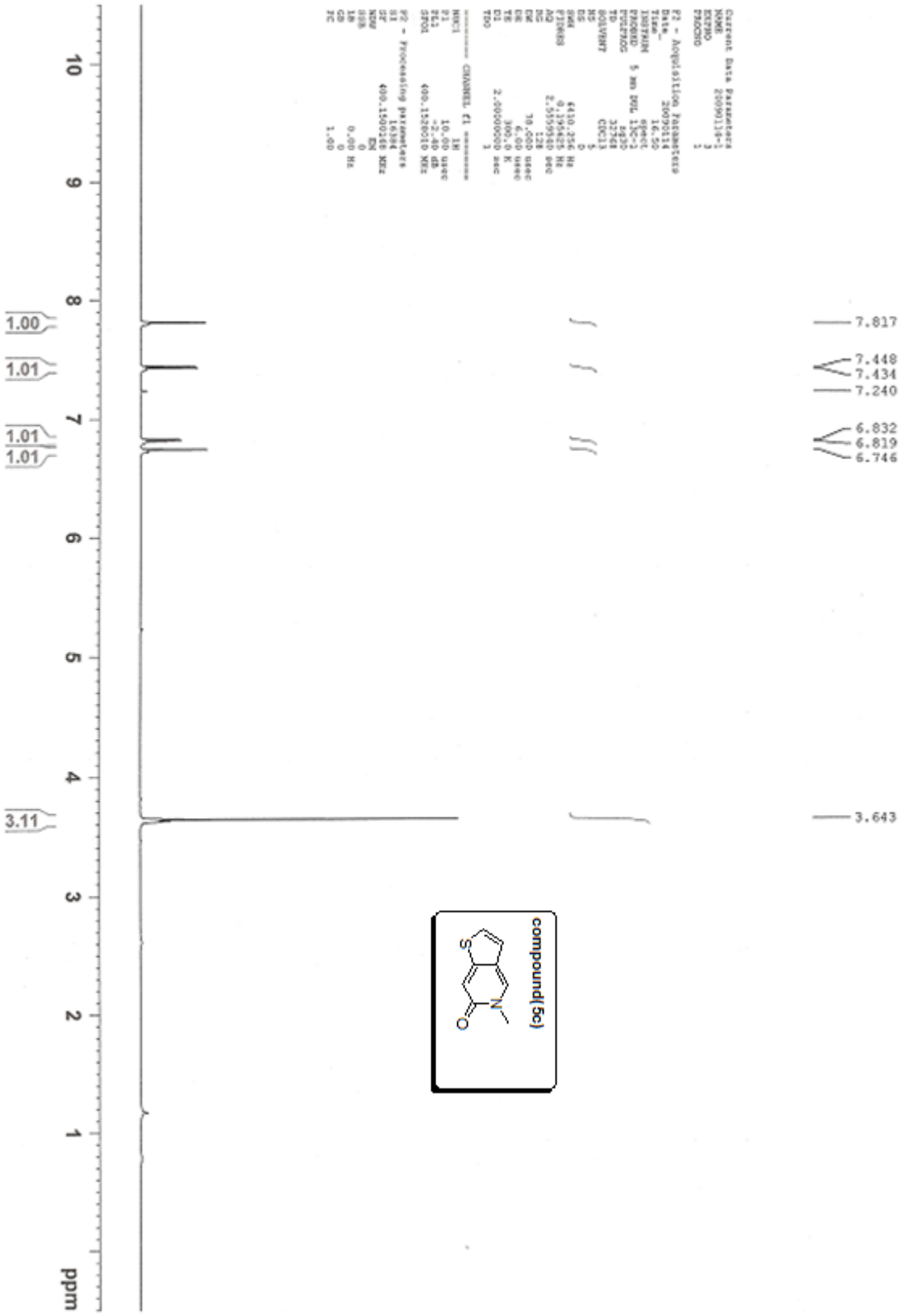

160.235
152.627
145.648
142.554
136.238
135.928
128.899
128.224
128.080
114.771
110.324
101.246
100.258
98.630
77.209
76.997
76.785
52.479
ppm
compound(3n)
O
O
N
Bn
O

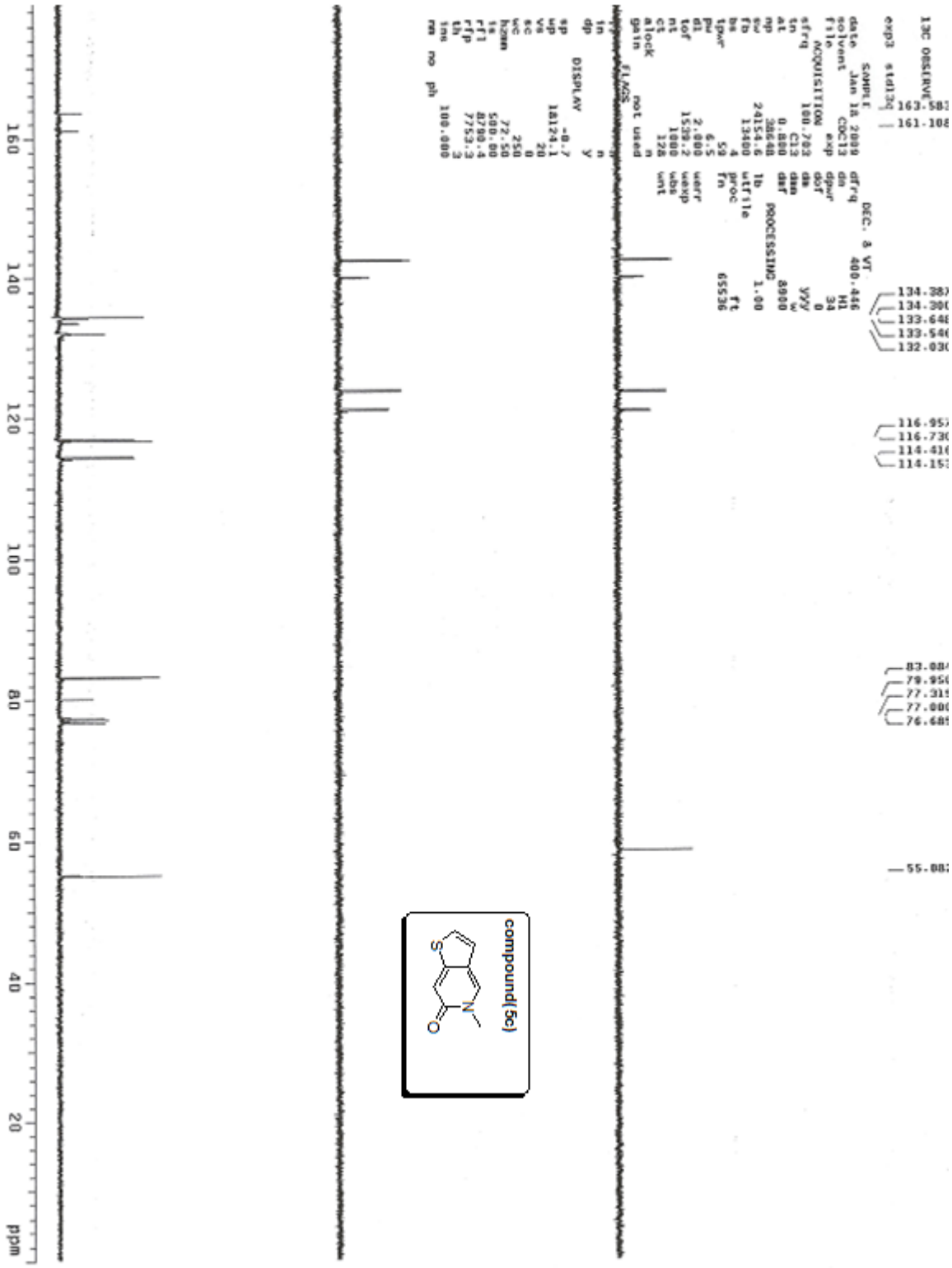

7.275
7.255
7.249
7.240
7.236
7.221
7.208
7.200
7.191
7.185
6.924
6.292
5.038
2.549
2.534
2.395
2.381
1.632
1.623
1.615
1.607
1.599
5.08
1.00
0.96
1.95
2.00
1.99
4.00
ppm
copound(5a)
N
Bn
O

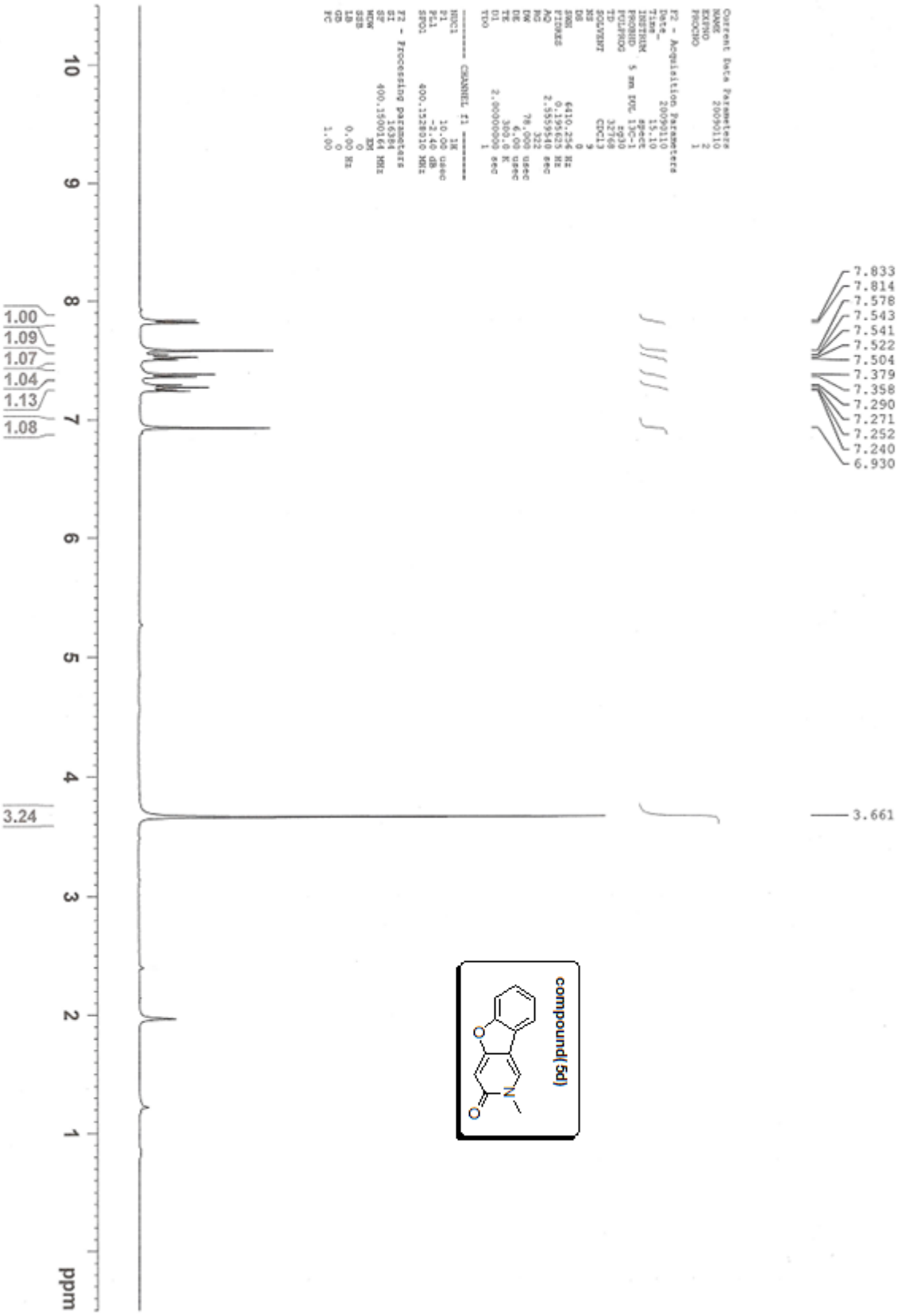

161.72
151.74
136.71
134.08
128.53
127.80
127.55
117.88
116.43
77.32
77.00
76.69
51.12
28.64
25.09
22.49
21.94
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
copound(5a)
N
Bn
O

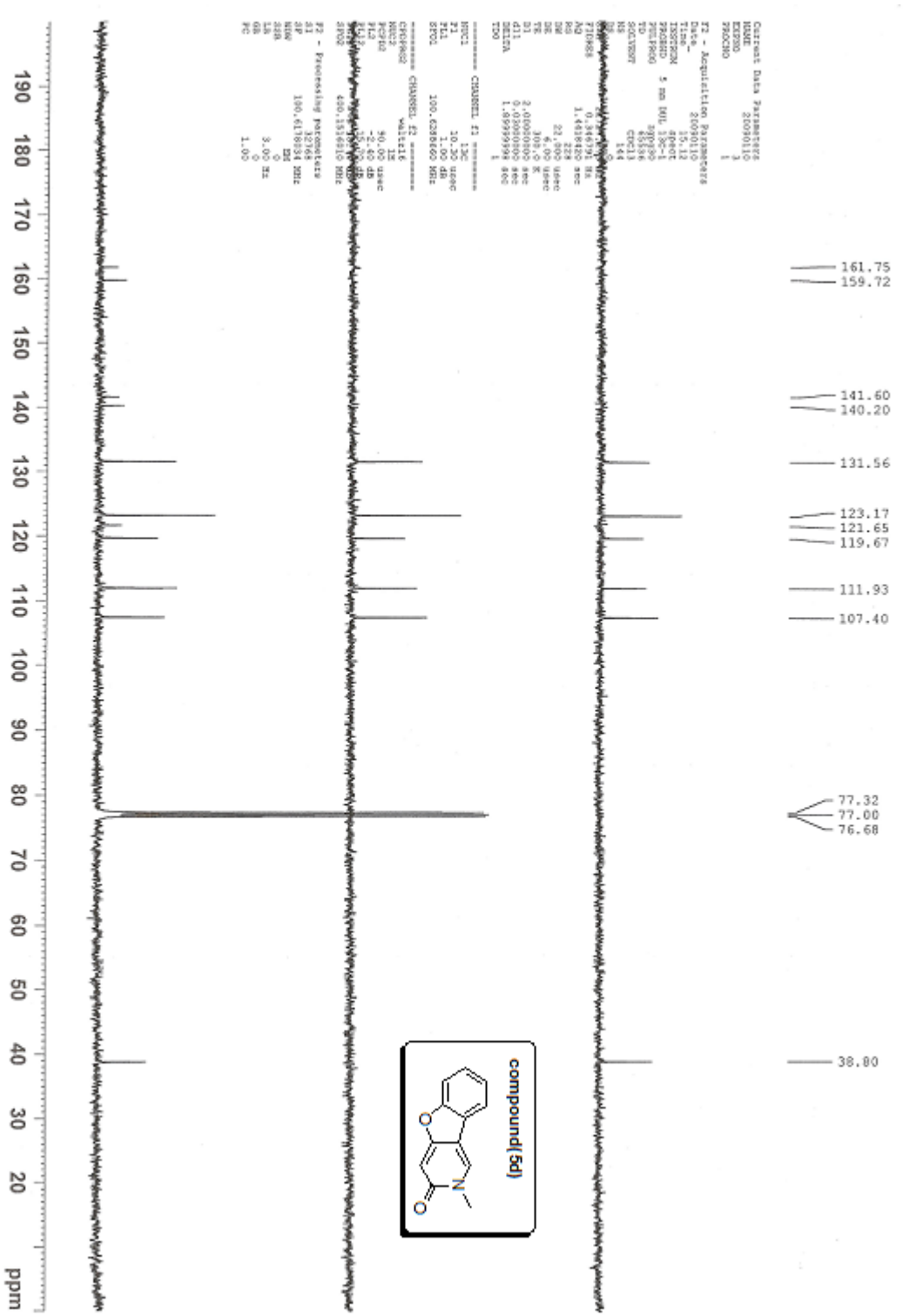

Current Data Parameters
NAME 20081215
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081215
Time 21.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559560 sec
RG 71.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
---------- CHANNEL f1 ----------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.99
2.99
1.98
1.00
1.96
2.00
1.95
1.95
10
9
8
7
6
5
4
3
2
1
ppm
7.274
7.265
7.258
7.241
7.211
7.207
7.168
7.159
7.155
7.151
7.144
7.027
5.080
2.877
2.858
2.840
2.795
2.776
2.757
2.009
1.990
1.980
1.971
1.952
1.933
copound(5b)
N
Bn
O

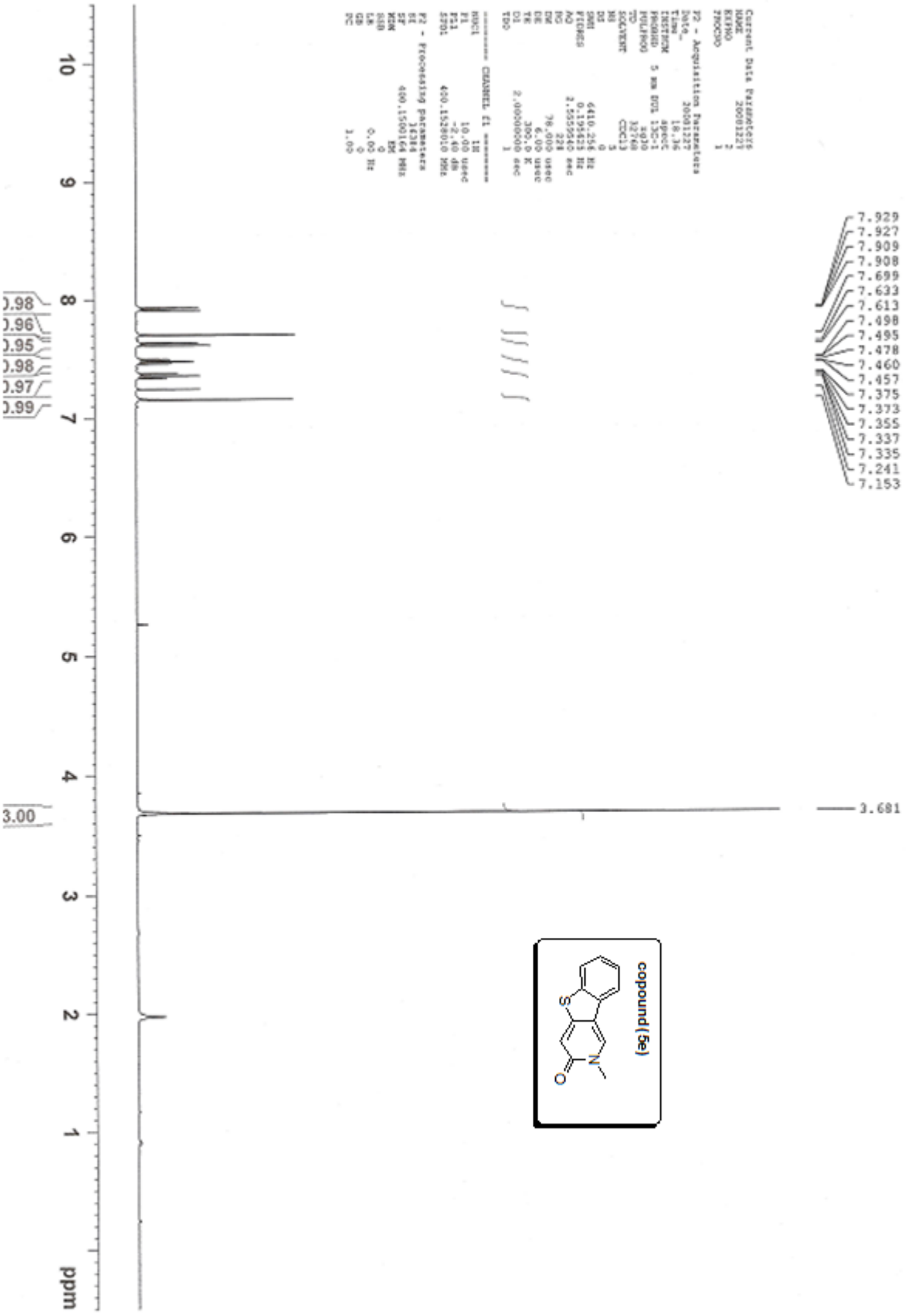

166.03
150.93
142.63
133.51
131.31
129.09
127.83
118.66
77.32
77.00
76.68
63.49
31.31
30.19
23.70
ppm
copound (5b)
N
Bn
O

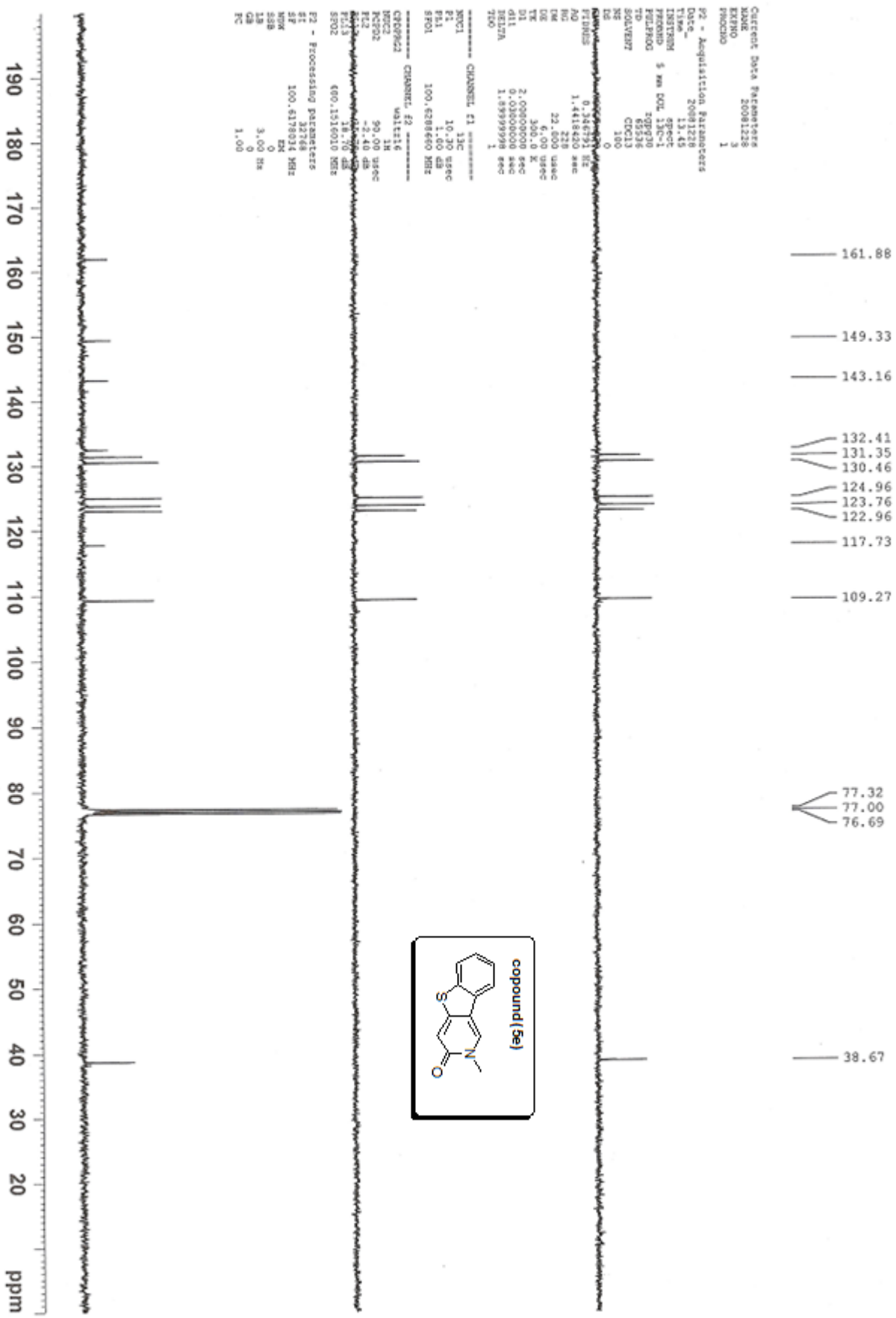

1.00
1.01
1.01
1.01
3.11
7.817
7.448
7.434
7.240
6.832
6.819
6.746
3.643
ppm
compound(5c)

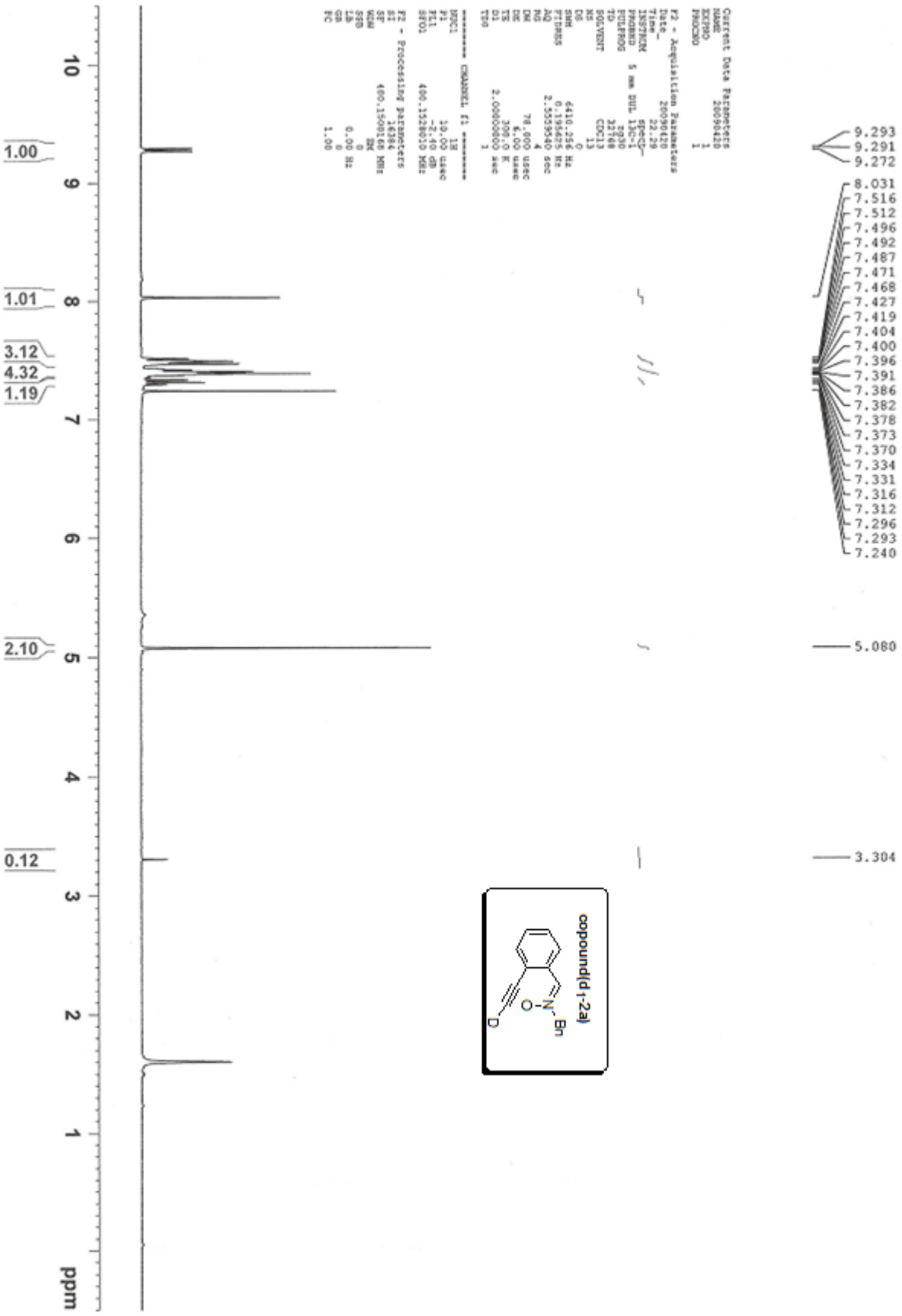

compound(5c)

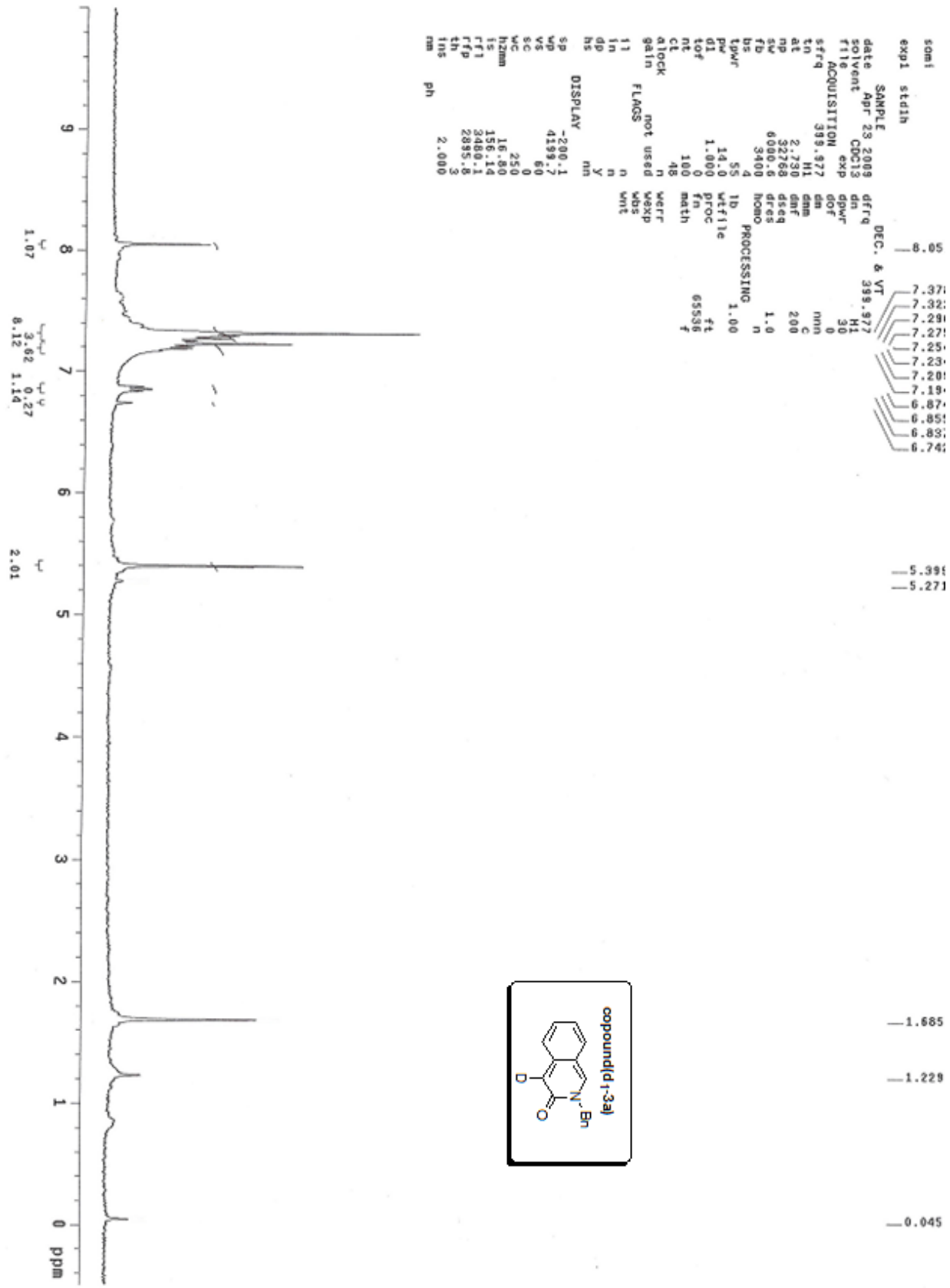

compound(5d)
7.833
7.814
7.578
7.543
7.541
7.522
7.504
7.379
7.358
7.290
7.271
7.252
7.240
6.930
3.661
1.00
1.09
1.07
1.04
1.13
1.08
3.24
ppm


161.75
159.72
141.60
140.20
131.56
123.17
121.65
119.67
111.93
107.40
77.32
77.00
76.68
38.80
compound(5d)
ppm

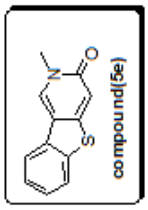

7.929
7.927
7.909
7.908
7.699
7.633
7.613
7.498
7.495
7.478
7.460
7.457
7.375
7.373
7.355
7.337
7.335
7.241
7.153
3.681
3.00
compound (3c)
ppm


161.88
149.33
143.16
132.41
131.35
130.46
124.96
123.76
122.96
117.73
109.27
77.32
77.00
76.69
38.67
compound (3e)
O
S
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


1.00
1.01
3.12
4.32
1.19
2.10
0.12
ppm
9.293
9.291
9.272
8.031
7.516
7.512
7.496
7.492
7.487
7.471
7.468
7.427
7.419
7.404
7.400
7.396
7.391
7.386
7.382
7.378
7.373
7.370
7.334
7.331
7.316
7.312
7.296
7.293
7.240
5.080
3.304
copound(d-1-2a)
Bn
D


8.05
7.37
7.32
7.29
7.27
7.25
7.23
7.20
7.19
6.87
6.85
6.83
6.74
5.39
5.27
1.685
1.229
0.045
1.07
3.62
8.12
0.27
1.14
2.01
9
8
7
6
5
4
3
2
1
0
ppm
copound(d+-3a)
D
N
Bn
O

(4) X-ray data for compound(5e) :



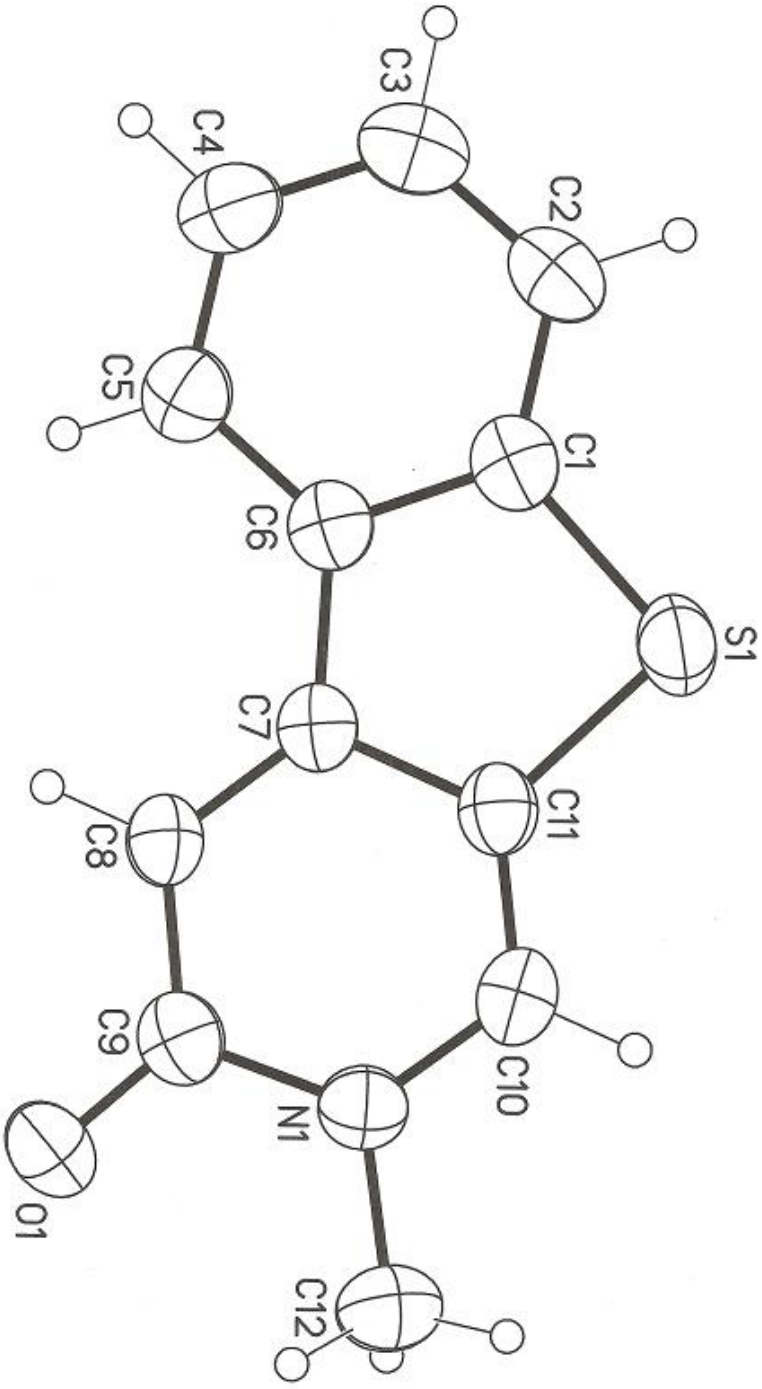


Table 1. Crystal data and structure refinement for 090119_0m.

Identification code	090119_0m	
Empirical formula	C12 H9 N O S	
Formula weight	215.26	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*P*2(1)/*n*	
Unit cell dimensions	a = 11.749(4) Å	砀= 90°.
	b = 5.6906(18) Å	砀= 106.489(10)°.
	c = 15.174(5) Å	砀 = 90°.
Volume	972.8(6) Å^3	
Z	4	
Density (calculated)	1.470 Mg/m^3	
Absorption coefficient	0.299 mm^{-1}	
F(000)	448	
Crystal size	0.30 x 0.15 x 0.03 mm^3	
Theta range for data collection	1.95 to 25.12°.	
Index ranges	-14≤h≤13, -6≤k≤4, -18≤l≤16	
Reflections collected	4566	
Independent reflections	1716 [R(int) = 0.0343]	
Completeness to theta = 25.12°	98.5 %	
Absorption correction	Empirical	
Max. and min. transmission	0.7452 and 0.5579	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1716 / 0 / 137	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0431, wR2 = 0.1158	
R indices (all data)	R1 = 0.0554, wR2 = 0.1255	
Largest diff. peak and hole	0.408 and -0.302 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 090119_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9933(2)	4709(4)	2004(2)	42(1)
C(2)	10493(2)	4711(5)	1318(2)	51(1)
C(3)	11286(2)	6482(5)	1310(2)	55(1)
C(4)	11517(2)	8238(5)	1973(2)	53(1)
C(5)	10960(2)	8247(5)	2655(2)	45(1)
C(6)	10154(2)	6463(4)	2677(2)	39(1)
C(7)	9472(2)	6116(4)	3329(1)	36(1)
C(8)	9416(2)	7452(4)	4062(2)	40(1)
C(9)	8681(2)	6801(4)	4627(2)	42(1)
C(10)	8130(2)	3300(4)	3674(2)	42(1)
C(11)	8780(2)	4022(4)	3118(2)	38(1)
C(12)	7363(2)	3836(5)	4983(2)	57(1)
N(1)	8098(2)	4636(3)	4413(1)	40(1)
O(1)	8533(2)	7980(4)	5271(1)	58(1)
S(1)	8912(1)	2598(1)	2136(1)	49(1)

Table 3. Bond lengths [Å] and angles [°] for 090119_0m.

C(1)-C(2)	1.380(3)
C(1)-C(6)	1.399(3)
C(1)-S(1)	1.750(2)
C(2)-C(3)	1.375(4)
C(2)-H(2)	0.9300
C(3)-C(4)	1.389(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.372(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.396(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.451(3)
C(7)-C(8)	1.365(3)
C(7)-C(11)	1.427(3)
C(8)-C(9)	1.427(3)
C(8)-H(8)	0.9300
C(9)-O(1)	1.237(3)
C(9)-N(1)	1.402(3)
C(10)-C(11)	1.353(3)
C(10)-N(1)	1.365(3)
C(10)-H(10)	0.9300
C(11)-S(1)	1.742(2)
C(12)-N(1)	1.457(3)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(2)-C(1)-C(6)	121.4(2)
C(2)-C(1)-S(1)	125.2(2)
C(6)-C(1)-S(1)	113.36(17)
C(3)-C(2)-C(1)	118.5(2)
C(3)-C(2)-H(2)	120.8
C(1)-C(2)-H(2)	120.8
C(2)-C(3)-C(4)	121.0(2)
C(2)-C(3)-H(3)	119.5

C(4)-C(3)-H(3)	119.5
C(5)-C(4)-C(3)	120.8(3)
C(5)-C(4)-H(4)	119.6
C(3)-C(4)-H(4)	119.6
C(4)-C(5)-C(6)	119.2(2)
C(4)-C(5)-H(5)	120.4
C(6)-C(5)-H(5)	120.4
C(5)-C(6)-C(1)	119.2(2)
C(5)-C(6)-C(7)	128.7(2)
C(1)-C(6)-C(7)	112.2(2)
C(8)-C(7)-C(11)	119.4(2)
C(8)-C(7)-C(6)	130.1(2)
C(11)-C(7)-C(6)	110.52(19)
C(7)-C(8)-C(9)	121.5(2)
C(7)-C(8)-H(8)	119.2
C(9)-C(8)-H(8)	119.2
O(1)-C(9)-N(1)	119.3(2)
O(1)-C(9)-C(8)	125.3(2)
N(1)-C(9)-C(8)	115.4(2)
C(11)-C(10)-N(1)	119.7(2)
C(11)-C(10)-H(10)	120.1
N(1)-C(10)-H(10)	120.1
C(10)-C(11)-C(7)	120.1(2)
C(10)-C(11)-S(1)	126.52(19)
C(7)-C(11)-S(1)	113.38(16)
N(1)-C(12)-H(12A)	109.5
N(1)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
N(1)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(10)-N(1)-C(9)	123.62(19)
C(10)-N(1)-C(12)	118.2(2)
C(9)-N(1)-C(12)	118.1(2)
C(11)-S(1)-C(1)	90.52(11)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 090119_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	38(1)	44(1)	42(1)	-2(1)	6(1)	5(1)
C(2)	55(1)	60(2)	40(1)	-7(1)	14(1)	5(1)
C(3)	57(2)	64(2)	46(2)	6(1)	20(1)	7(1)
C(4)	50(1)	58(2)	53(2)	6(1)	18(1)	-5(1)
C(5)	45(1)	45(2)	43(1)	2(1)	9(1)	-1(1)
C(6)	34(1)	41(1)	38(1)	0(1)	5(1)	3(1)
C(7)	32(1)	35(1)	36(1)	0(1)	4(1)	3(1)
C(8)	38(1)	38(1)	40(1)	-5(1)	6(1)	-3(1)
C(9)	40(1)	44(1)	39(1)	-1(1)	6(1)	3(1)
C(10)	39(1)	36(1)	48(1)	-1(1)	7(1)	-1(1)
C(11)	35(1)	37(1)	40(1)	-3(1)	6(1)	1(1)
C(12)	64(2)	61(2)	53(2)	5(1)	26(1)	-8(1)
N(1)	41(1)	41(1)	38(1)	4(1)	11(1)	2(1)
O(1)	64(1)	63(1)	51(1)	-19(1)	25(1)	-9(1)
S(1)	49(1)	48(1)	51(1)	-16(1)	13(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for 090119_0m.

	x	y	z	U(eq)
H(2)	10337	3541	871	62
H(3)	11673	6505	853	66
H(4)	12055	9422	1954	64
H(5)	11118	9428	3097	54
H(8)	9866	8818	4197	47
H(10)	7708	1898	3553	50
H(12A)	6597	4568	4779	86
H(12B)	7735	4252	5612	86
H(12C)	7273	2161	4932	86