Supporting Information

Alkynes as Equivalent of α-Diazo Ketones in Generating α-Oxo Metal Carbenes: A Gold-Catalyzed Expedient Access to Dihydrofuran-3-ones

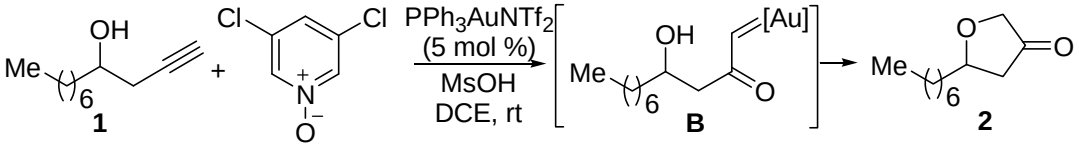
Longwu Ye, Li Cui, Guozhu Zhang and Liming Zhang

Department of Chemistry and Biochemistry,

University of California, Santa Barbara, California, 93106

General. Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous 1, 2-dichloroethane (HPLC grade) was purified by distillation over calcium hydride. Anhydrous tetrahydrofuran in Pure-Pac™ from Aldrich was used directly without further purification. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed over silicycle silica gel (230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian 500 MHz Unity plus spectrometer and a Varian 400 MHz spectrometer using residue solvent peaks as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Micromass QTOF2 Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization.

Reaction Conditions Optimization: Different Ratios of *N*-oxides and Acid



1.0 equiv. 2.0 equiv.

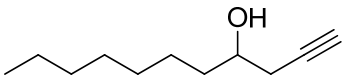
	yield
MsOH(3.0 equiv.)	61%
MsOH(2.0 equiv.)	72%
MsOH(1.5 equiv.)	72%
MsOH(1.2 equiv.)	75%

General procedure for the preparation of propargylic alcohol 1 and 3

In a typical experiment a solution of Grignard reagent, C_3H_3MgBr in Et_2O was prepared by a literature procedure.[1] This solution was cooled to –30 ℃ and a solution of the corresponding aldehyde or ketone in Et_2O was added dropwise. The mixture was slowly warmed to room temperature and stirred for 1 h. It was then poured into ice water. Saturated aqueous NH_4Cl solution was added to dissolve the precipitate and the organic

layer was separated. The aqueous layer was extracted with ether and the extracts were combined with the above organic layer. The combined solution was dried over $MgSO_4$. After evaporation of the solvent the residue was purified by flash silica gel column chromatography.

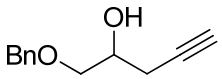
Undec-1-yn-4-ol (1)



1

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with octanal in 97% yield. This compound is known and the spectroscopic data match those reported.[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.80 – 3.72 (m, 1H), 2.47 – 2.28 (m, 1H), 2.06 (t, 1H, *J* = 2.4 Hz), 1.89 (d, 1H, *J* = 4.4 Hz), 1.58 – 1.51 (m, 2H), 1.47 – 1.27 (m, 10H)), 0.88 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 80.9, 70.7, 69.9, 36.2, 31.8, 29.5, 29.2, 27.3, 25.6, 22.6, 14.1.

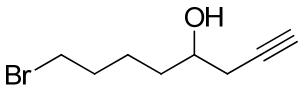
1-Benzyloxypent-4-yn-2-ol (3a)



3a

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with benzyloxy-acetaldehyde in 83% yield. This compound is known and the spectroscopic data match those reported.[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 4.55 (s, 2H), 4.00 – 3.93 (m, 1H), 3.61 – 3.58 (m, 1H), 3.52 – 3.47 (m, 1H), 2.61 (d, 1H, *J* = 4.8 Hz), 2.44 (dd, 2H, *J* = 6.0 Hz, *J* = 3.2 Hz), 2.02 – 2.01 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.7, 128.4, 127.8, 127.7, 80.2, 73.4, 72.7, 70.5, 68.7, 23.4.

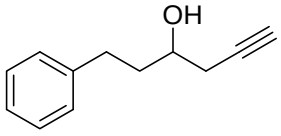
8-Bromooct-1-yn-4-ol (3b)



3b

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 5-bromo-pentanal in 45% yield.[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.77 – 3.74 (m, 1H), 3.40 (t, 2H, *J* = 6.4 Hz), 2.45 – 2.28 (m, 2H), 2.05 (t, 1H, *J* = 2.4 Hz), 1.96 (bs, 1H), 1.92 – 1.82 (m, 2H), 1.64 – 1.47 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 80.6, 71.0, 69.6, 35.2, 33.6, 32.5, 27.4, 24.2; IR (neat): 3386, 3295, 2931, 2864, 1425; MS (ES^+) Calculated for $[C_8H_{13}BrNaO]^+$: 227.0; Found: 227.1.

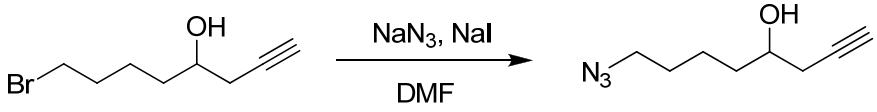
1-Phenylhex-5-yn-3-ol (3c)



3c

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 3-phenyl-propionaldehyde in 77% yield. This compound is known and the spectroscopic data match those reported.[4] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.18 (m, 5H), 3.82 – 3.75 (m, 1H), 2.84 – 2.65 (m, 2H), 2.50 – 2.35 (m, 2H), 2.05 (t, 1H, *J* = 2.4 Hz), 2.01 – 1.93 (m, 1H), 1.88 – 1.83 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.6, 128.4, 128.3, 125.8, 80.6, 70.9, 69.0, 37.7, 31.8, 27.4.

8-Azidooct-1-yn-4-ol (3d)

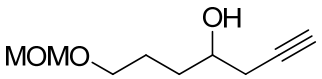


3b **3d**

To a solution of **3b** (612 mg, 3.0 mmol) in DMF (10 mL) was added NaN_3 (0.59 g, 9 mmol) and NaI (0.3 g, 1.5 mmol) at room temperature. The resulting mixture was heated at 80 °C for 12 h and cooled to room temperature before the addition of water (10 mL)

and diethyl ether (20 mL). The aqueous layer was extracted with Et_2O (3 × 30 mL) and the combined organic layers were washed with water (2 × 10 mL), brine (10 mL), dried with $MgSO_4$, and concentrated. The crude product was purified with flash silica gel column chromatography (eluents: hexanes : ethyl acetate = 5:1) to get **3d** (0.256 g, 1.5 mmol) in 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.80 – 3.74 (m, 1H), 3.29 (t, 2H, *J* = 6.4 Hz), 2.47 – 2.30 (m, 2H), 2.07 (t, 1H, *J* = 2.4 Hz), 1.94 (bs, 1H), 1.69 – 1.42 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 80.6, 71.0, 69.5, 51.3, 35.5, 28.7, 27.4, 22.8; IR (neat): 3395(bs), 3299, 2940, 2866, 2098, 1456, 1350, 1264; MS (ES^+) Calculated for $[C_8H_{14}N_3O]^+$: 168.1; Found: 168.1.

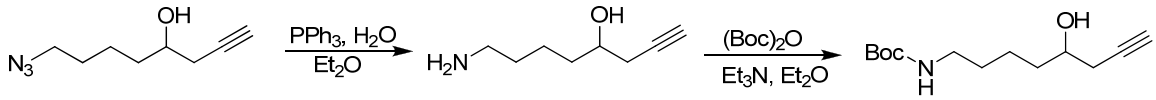
7-Methoxymethoxyhept-1-yn-4-ol (3e)



3e

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 4-methoxymethoxy-butyraldehyde in 55% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.58 (s, 2H), 3.78 – 3.73 (m, 1H), 3.53 (t, 2H, *J* = 6 Hz), 3.32 (s, 3H), 2.61 (bs, 1H), 2.41 – 2.28 (m, 2H), 2.02 (t, 1H, *J* = 2.4 Hz), 1.74 – 1.52 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 96.3, 80.9, 70.6, 69.6, 67.6, 55.2, 33.2, 27.3, 25.9; IR (neat): 3423, 3295, 2937, 1449; Calculated for $[C_9H_{16}NaO_3]^+$: 195.1; Found: 195.1.

tert-Butyl *N*-(5-Hydroxyoct-7-ynyl)carbamate (3f)

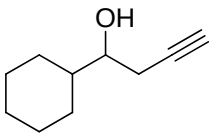


3d **3f**

To a solution of **3d** (70.1 mg, 0.42 mmol) in Et_2O (10 mL) was added PPh_3 (132 mg, 0.5 mmol) and H_2O (0.3 mL) at room temperature. The resulting mixture was stirred for 12 h. The reaction mixture was then added $(Boc)_2O$ (0.685 g, 3.1 mmol) and stirred overnight. The reaction was added water and the aqueous layer was extracted with Et_2O (3 × 20 mL)

and the combined organic layers were washed with brine (10 mL), dried with $MgSO_4$, and concentrated. The crude product was purified with flash silica gel column chromatography (eluents: hexanes : ethyl acetate = 3:1) to give **3f** (65 mg, 0.27 mmol) in 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.54 (bs, 1H), 3.79 – 3.73 (m, 1H), 3.12 (d, 2H, *J* = 5.6 Hz), 2.45 – 2.29 (m, 2H), 2.06 (t, 1H, *J* = 2.4 Hz), 2.02 (bs, 1H), 1.64 – 1.36 (m, 15H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.1, 80.8, 79.1, 70.7, 69.6, 40.2, 35.6, 29.9, 28.4, 27.4, 22.6; IR (neat): 3400(bs), 3311, 2977, 2936, 2865, 2119, 1689, 1526, 1366, 1251; MS (ES^+) Calculated for $[C_{13}H_{23}NNaO_3]^+$: 185.1; Found: 185.1.

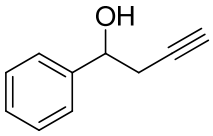
1-Cyclohexylbut-3-yn-1-ol (3g)



3g

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with cyclohexanecarbaldehyde in 80% yield. This compound is known and the spectroscopic data match those reported.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.51 – 3.47 (m, 1H), 2.48 – 2.43 (m, 1H), 2.40 – 2.31 (m, 1H), 2.06 – 2.04 (m, 1H), 1.92 – 1.86 (m, 2H), 1.78 – 1.64 (m, 4H), 1.50 – 1.42 (m, 1H), 1.30 – 0.98 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 81.3, 73.9, 70.6, 42.4, 28.9, 28.1, 26.3, 26.1, 25.9, 24.6.

1-Phenylbut-3-yn-1-ol (3h)

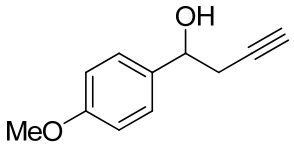


3h

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with benzaldehyde in 85% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.31 (m, 5H), 4.87 (t, 1H, *J* = 6.0

Hz), 2.65 (t, 2H, *J* = 2.8 Hz), 2.47 (bs, 1H), 2.08 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.4, 128.4, 128.0, 125.7, 80.6, 72.3, 70.9, 29.4.

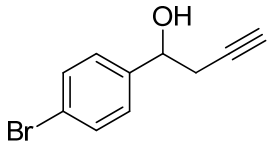
1-(4-Methoxyphenyl)but-3-yn-1-ol (3i)



3i

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 4-methoxy-benzaldehyde in 78% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, 2H, *J* = 8.0 Hz), 6.90 (d, 2H, *J* = 8.0 Hz), 4.84 – 4.80 (m, 1H), 3.80 (s, 3H), 2.63 – 2.61 (m, 2H), 2.41 (d, 1H, *J* = 3.2 Hz),), 2.06 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.3, 134.6, 127.0, 113.8, 80.8, 71.9, 70.8, 55.2, 29.3.

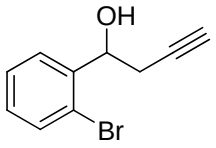
1-(4-Bromophenyl)but-3-yn-1-ol (3j)



3j

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 4-bromo-benzaldehyde in 72% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, 2H, *J* = 8.4 Hz), 7.26 (d, 2H, *J* = 8.4 Hz), 4.83 (t, 1H, *J* = 6.0 Hz), 2.62 – 2.59 (m, 2H), 2.50 (bs, 1H),), 2.08 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.3, 131.5, 127.5, 121.8, 80.1, 71.6, 71.3, 29.4.

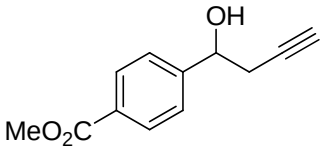
1-(2-Bromophenyl)but-3-yn-1-ol (3k)



3k

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 2-bromo-benzaldehyde in 75% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.59 (m, 1H), 7.54 – 7.50 (m, 1H), 7.38 – 7.34 (m, 1H), 7.18 – 7.14 (m, 1H), 5.25 – 5.21 (m, 1H), 2.84 – 2.79 (m, 1H), 2.58 – 2.50 (m, 2H), 2.11 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.2, 132.6, 129.2, 127.7, 127.3, 121.7, 80.2, 71.2, 70.9, 27.7.

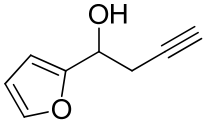
Methyl 4-(1-hydroxybut-3-ynyl)benzoate (3l)



3l

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 4-formyl-benzoic acid methyl ester in 65% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, 2H, *J* = 6.8 Hz), 7.47 (d, 2H, *J* = 6.8 Hz), 4.95 – 4.92 (m, 1H), 3.91 (s, 3H), 2.69 – 2.55 (m, 3H), 2.08 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.8, 147.4, 129.8, 129.7, 125.7, 80.0, 71.8, 71.4, 52.1, 29.4; IR (neat): 3432, 3294, 2952, 1716, 1611, 1435; Calculated for $[C_{12}H_{12}NaO_3]^+$: 227.1; Found: 227.1.

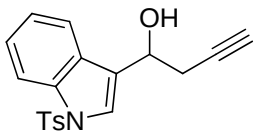
(1-Furan-2-yl)but-3-yn-1-ol (3m)



3m

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with furan-2-carbaldehyde in 71% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (t, 1H, *J* = 1.2 Hz), 6.35 (d, 2H, *J* = 1.2 Hz), 4.88 (dd, 1H, *J* = 12.0 Hz, *J* = 2.0 Hz), 2.78 (dd, 2H, *J* = 6.4 Hz, *J* = 2.8 Hz), 2.45 – 2.43 (m, 1H), 2.07 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.6, 142.3, 110.2, 106.6, 79.8, 71.1, 66.0, 26.0.

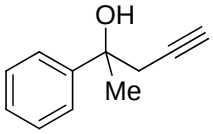
1-[1-(Toluene-4-sulfonyl)-1H-indol-3-yl]but-3-yn-1-ol (3n)



3n

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 1-(toluene-4-sulfonyl)-1H-indole-3-carbaldehyde in 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, 1H, *J* = 8.0 Hz), 7.76 (d, 2H, *J* = 8.0 Hz), 7.62 (d, 2H, *J* = 8.4 Hz), 7.34 – 7.30 (m, 1H), 7.25 – 7.18 (m, 3H), 5.10 (dd, 1H, *J* = 10.4 Hz, *J* = 4.8 Hz), 2.85 – 2.73 (m, 2H), 2.58 – 2.31 (m, 1H), 2.10 (t, 1H, *J* = 2.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0, 135.3, 135.0, 129.8, 128.6, 126.8, 124.9, 123.6, 123.2, 123.1, 120.1, 113.7, 80.2, 71.5, 66.1, 27.6, 21.5; IR (neat): 3532, 3294, 2918, 1596, 1448, 1368, 1174; MS (ES^+) Calculated for $[C_{19}H_{17}NNaO_3S]^+$: 362.1; Found: 362.1.

2-Phenylpent-4-yn-2-ol (3o)

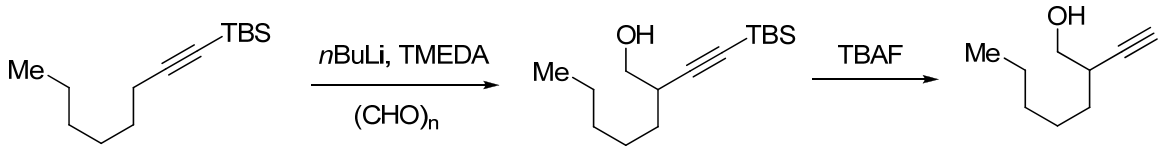


3o

The title alcohol was prepared by reacting prop-2-yne-1-yl magnesium bromide with 1-phenyl-ethanone in 65% yield. This compound is known and the spectroscopic data match those reported.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, 2H, *J* = 7.6 Hz), 7.29 (t,

2H, *J* = 8.0 Hz), 7.20 (t, 1H, *J* = 7.6 Hz), 2.66 (dq, 2H, *J* = 16.0 Hz, , *J* = 2.4 Hz), 2.35 (s, 1H), 1.99 – 1.97 (m, 1H), 1.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.3, 128.3, 128.2(1), 128.1(9), 127.1, 124.7, 80.4, 73.2, 71.7, 34.6, 29.2.

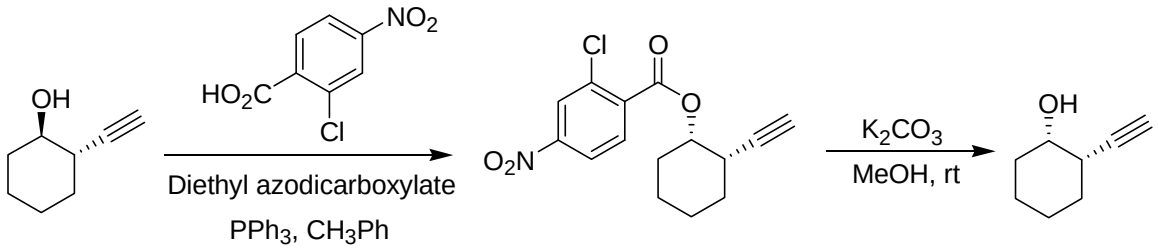
2-Ethynylhexan-1-ol (3p)



3p' **3p**

To a solution of **3p'** (750 mg, 6.8 mmol) and TMEDA (8.2 mmol, 1.2 eq) in anhydrous THF (10 mL) cooled to -78 ℃ was added *n*-BuLi (5.1 mL, 1.6 M in hexanes, 8.2 mmol, 1.2 eq). The resulting mixture was warmed to 0 ^{o}C during a 1 h period and cooled in dry ice-acetone bath again before the addition of an aldehyde (1.1 eq). After stirred at -78 ℃ for 1 h, the reaction mixture was quenched with aq. NH_4Cl, and extracted with Et_2O. The combined organic layers were washed with brine, dried with $MgSO_4$, and concentrated. The residue was dissolved in THF (10 mL), and the resulting solution was treated with TBAF (8.2 mL, 1.0 M in THF, 1.2 eq) at 40 ℃ for 1 h. The reaction mixture was quenched with aq. NH_4Cl, and extracted with Et_2O. The combine organic layer was washed with brine, dried with $MgSO_4$, and concentrated. The alkynyl alcohol product was purified with flash silica gel column chromatography to get **3p** in 25% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.67 – 3.55 (m, 2H), 2.60 – 2.55 (m, 1H), 2.14 (d, 1H, *J* = 2.5 Hz), 1.76 (t, 1H, *J* = 6.5 Hz), 1.53 – 1.25 (m, 8H), 0.89 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 85.1, 70.9, 65.4, 35.2, 31.6, 30.8, 26.8, 22.5, 14.0; IR (neat): 3311, 2929, 2860, 1590, 1467; Calculated for $[C_9H_{16}NaO]^+$: 163.1; Found: 163.1.

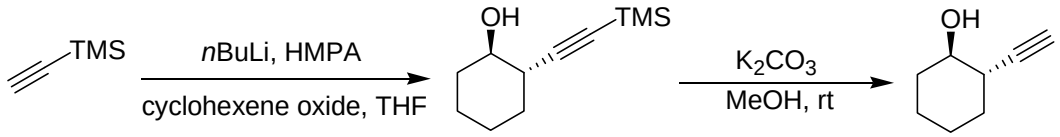
(1*S, 2*S**)- 2-Ethynylcyclohexanol (3q)**



3r **3q'** **3q**

2-Chloro-4-nitro-benzoic acid (202 mg, 1.0 mmol) and Ph_3P (262 mg, 1.0 mmol) were added to a solution of **3r** (62 mg, 0.5 mmol) in toluene (5 mL) at 0 °C under N_2. Diethyl azodicarboxylate (toluene solu., 1.0 mmol) was added dropwise to the resulting solution. The mixture was stirred for 10 h and then treated with saturated aqueous $NaHCO_3$. The resulting solution was extracted with AcOEt. The organic layer was washed with brine, dried over Na_2SO_4, and evaporated in vacuo. The residue was purified by SiO_2 column chromatography using hexane-AcOEt (25/1) as the eluent to give **3q'**(138 mg, 90%). **3q'** (138 mg, 0.45 mmol) was dissolved in MeOH (10 mL), and the resulting solution was treated with K_2CO_3 (249 mg, 1.8 mmol) at rt for 1 h. The reaction mixture was concentrated and purified with flash silica gel column chromatography to get **3q** in 83% yield. This compound is known and the spectroscopic data match those reported.[7] ^1H NMR (400 MHz, $CDCl_3$) δ 3.71 – 3.67 (m, 1H), 2.85 – 2.81 (m, 1H), 2.16 (d, 1H, *J* = 2.4 Hz), 1.89 – 1.82 (m, 2H), 1.74 – 1.64 (m, 3H), 1.62 – 1.50 (m, 2H), 1.42 – 1.36 (m, 1H), 1.35 – 1.26 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.3, 71.7, 69.6, 35.5, 31.2, 28.6, 22.6, 22.0.

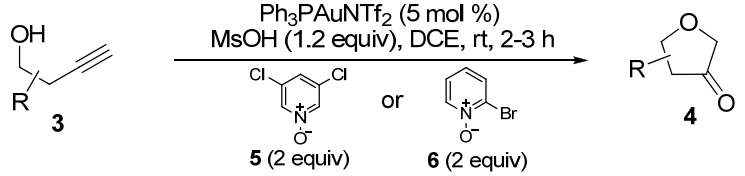
(1*R, 2*S**)-2-Ethynyl-cyclohexanol (3r)**



3r' **3r**

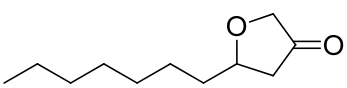
n-BuLi (1.6 M, 11.0 mL, 17.4 mmol) was added dropwise at 0 °C to a solution of triethylsilylacetylene (2.3 mL, 15.8 mmol) in THF (20 mL) and HMPA (7.5 mL). The mixture was stirred for 30 min. then cyclohexene oxide (1.7 mL, 15.8 mmol) added and

stirring continued for 12 h. Aqueous NH_4Cl (10 mL) was added, and the mixture then extracted with Et_2O (3 x 20 mL), the combined extracts washed with brine (20 mL), dried ($MgSO_4$) and then condensed in vacuo. Column chromatography (hexanes: EtOAc, 5:1) gave 2-trimethylsilanylethynyl-cyclohexanol **3r'** (2.38 g, 77%) as a colorless oil. **3r'** (1.49 g, 7.6 mmol) was dissolved in MeOH (15 mL), and the resulting solution was treated with K_2CO_3 (1.60 g, 11.6 mmol) at rt for 6 h. The reaction mixture was concentrated and purified with flash silica gel column chromatography to get **3r** in 85% yield. This compound is known and the spectroscopic data match those reported.[8] ^1H NMR (400 MHz, $CDCl_3$) δ 3.48 – 3.42 (m, 1H), 2.28 (s, 1H), 2.23 – 2.15 (m, 1H), 2.14 (d, 1H, *J* = 2.4 Hz), 2.03 – 1.95 (m, 2H), 1.77 – 1.71 (m, 1H), 1.672 – 1.61 (m, 1H), 1.43 – 1.11 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 85.8, 73.4, 70.2, 38.6, 33.1, 30.8, 24.7, 24.1.



Gold catalysis: general procedure: Pyridine *N*-oxide **5** or **6** (0.60 mmol), MsOH (1.8 mL, 0.20 M in DCE), and $Au(PPh_3)NTf_2$ (11.1 mg, 0.015 mmol) were added in this order to a solution of the propargyl alcohol **1** or **3** (0.30 mmol) in DCE (4.2 mL) at room temperature under N_2. The reaction mixture was stirred at rt and the progress of the reaction was monitored by TLC. The reaction typically took 2 – 3 h. Upon completion, the mixture was concentrated and the residue was purified by chromatography on silica gel (eluent: hexanes/ethyl acetate) to afford the desired products **2** or **4**.

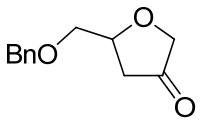
5-Heptyldihydrofuran-3-one (2)



2

Compound **2** was prepared in 73% yield using **5** as oxidant according to the general procedure, and its spectroscopic data match well with those reported.[9] The reaction time was 2.5 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.24 – 4.17 (m, 1H), 4.03 (d, 1H, *J* = 16.8 Hz), 3.81 (d, 1H, *J* = 16.8 Hz), 2.51 (dd, 1H, *J* = 18.0 Hz, *J* = 6.0 Hz), 2.14 (dd, 1H, *J* = 18.0 Hz, *J* = 9.6 Hz), 1.80 – 1.70 (m, 1H), 1.66 – 1.57 (m, 1H), 1.46 – 1.23 (m, 10H), 0.86 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.3, 78.3, 71.3, 43.0, 35.3, 31.7, 29.4, 29.1, 25.4, 22.6, 14.0; IR (neat): 2928, 2857, 1763, 1466, 1384.

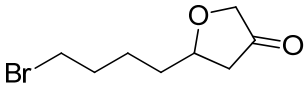
5-Benzyloxymethyldihydrofuran-3-one (4a)



4a

Compound **4a** was prepared in 68% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 5H), 4.59 (dd, 2H, *J* = 18.0 Hz, *J* = 12.0 Hz), 4.54 – 4.49 (m, 1H), 4.12 (d, 1H, *J* = 16.4 Hz), 3.91 (d, 1H, *J* = 16.8 Hz), 3.74 (dd, 1H, *J* = 10.4 Hz, *J* = 3.2 Hz), 3.60 (dd, 1H, *J* = 10.4 Hz, *J* = 4.8 Hz), 2.56 – 2.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.3, 137.7, 128.4, 127.8, 127.6, 76.7, 73.5, 72.0, 71.4, 38. 9; IR (neat): 3087, 3030, 2903, 2864, 1759, 1453, 1361; MS (ES^+) Calculated for $[C_{12}H_{14}NaO_3]^+$: 229.1; Found: 229.1.

5-(4-Bromobutyl)dihydrofuran-3-one (4b)

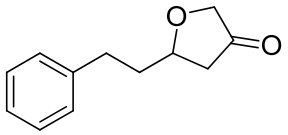


4b

Compound **4b** was prepared in 80% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h.^{1}H NMR (400 MHz, $CDCl_3$) δ 4.27 – 4.20 (m, 1H), 4.06 (d, 1H, *J* = 17.2 Hz), 3.96 (d, 2H, *J* = 12.8 Hz), 3.43 (t, 2H, *J* = 5.6 Hz), 2.56 (dd, 1H, *J* = 17.6 Hz, *J* = 4.8 Hz), 2.17 (dd, 1H, *J* = 17.6 Hz, *J* = 4.8 Hz), 1.96 – 1.89 (m,

2H), 1.79 – 1.56 (m, 3H), 1.57 – 1.49 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.9, 78.0, 71.4, 42.9, 34.4, 33.4, 32.4, 24.1; IR (neat): 2938, 2865, 1759; Calculated for $[C_8H_{13}BrNaO_2]^+$: 243.0; Found: 243.0.

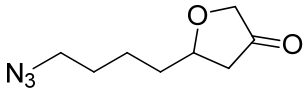
5-Phenethyldihydrofuran-3-one (4c)



4c

Compound **4c** was prepared in 68% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.12 (m, 5H), 4.18 – 4.13 (m, 1H), 4.00 (d, 1H, *J* = 16.8 Hz), 3.78 (d, 1H, *J* = 17.2 Hz), 2.77 – 2.66 (m, 2H), 2.45 (dd, 1H, *J* = 17.6 Hz, *J* = 6.0 Hz), 2.11 (dd, 1H, *J* = 17.6 Hz, *J* = 9.2 Hz), 2.07 – 1.89 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.0, 141.1, 128.5, 128.4, 126.1, 77.4, 71.3, 42.9, 37.0, 31.6; IR (neat): 3085, 3026, 2917, 2862, 1759, 1495, 1386; MS (ES^+) Calculated for $[C_{12}H_{14}NaO_2]^+$: 213.1; Found: 213.1.

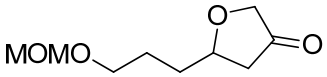
5-(4-Azidobutyl)dihydrofuran-3-one (4d)



4d

Compound **4d** was prepared in 82% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.26 – 4.21 (m, 1H), 4.05 (d, 1H, *J* = 13.6 Hz), 3.84 (d, 1H, *J* = 13.6 Hz), 3.31 (t, 2H, *J* = 5.6 Hz), 2.55 (dd, 1H, *J* = 14.4 Hz, *J* = 4.8 Hz), 2.17 (dd, 1H, *J* = 14.4 Hz, *J* = 7.6 Hz), 1.80 – 1.47 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.9, 78.0, 71.4, 51.3, 42.9, 34.8, 28.7, 22.7; IR (neat): 2937, 2864, 2094, 1759, 1507, 1456; MS (ES^+) Calculated for $[C_8H_{13}N_3NaO_2]^+$: 168.1; Found: 168.1.

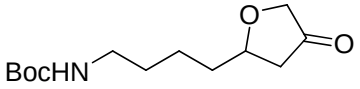
5-(3-Methoxymethoxypropyl)dihydrofuran-3-one (4e)



4e

Compound **4e** was prepared in 62% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.60 (s, 2H), 4.28 – 4.21 (m, 1H), 4.05 (d, 1H, *J* = 16.8 Hz), 3.83 (d, 1H, *J* = 17.2 Hz), 3.58 – 3.51 (m, 2H), 3.34 (s, 3H), 2.53 (dd, 1H, *J* = 18 Hz, *J* = 6 Hz), 2.16 (dd, 1H, *J* = 18 Hz, *J* = 6 Hz), 1.84 – 1.62 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.1, 96.4, 78.0, 71.4, 67.3, 55.2, 43.0, 32.1, 25.7; IR (neat): 2950, 2883, 2253, 1761; Calculated for $[C_9H_{16}NaO_4]^+$: 211.1; Found: 211.1.

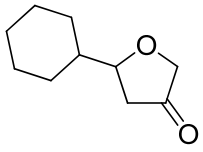
tert-Butyl *N*-[4-(4-Oxotetrahydrofuran-2-yl)butyl]carbamate (4f)



4f

Compound **4f** was prepared in 64% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.54 (bs, 1H), 4.27 – 4.20 (m, 1H), 4.05 (d, 1H, *J* = 17.2 Hz), 3.83 (d, 1H, *J* = 16.8 Hz), 3.14 (dd, 1H, *J* = 12.8 Hz, *J* = 6.8 Hz), 2.53 (dd, 1H, *J* = 18.0 Hz, *J* = 6.0 Hz), 2.16 (dd, 1H, *J* = 18.0 Hz, *J* = 9.2 Hz), 1.80 – 1.37 (m, 15H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.1, 156.0, 79.1, 78.1, 71.3, 42.9, 40.3, 34.9, 30.0, 28.4, 22.7; IR (neat): 3361, 2977, 2934, 2865, 1760, 1698, 1519, 1365; MS (ES^+) Calculated for $[C_{13}H_{23}NNaO_4]^+$: 280.2; Found: 280.1.

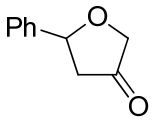
5-Cyclohexyldihydrofuran-3-one (4g)



4g

Compound **4g** was prepared in 72% yield using **5** as oxidant according to the general procedure, and its spectroscopic data match well with those reported.[10] The reaction time was 2 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 (d, 1H, *J* = 16.8 Hz), 3.97 – 3.91 (m, 1H), 3.82 (d, 1H, *J* = 16.8 Hz), 2.46 (dd, 1H, *J* = 18.0 Hz, *J* = 6.0 Hz), 2.23 (dd, 1H, *J* = 17.6 Hz, *J* = 10.0 Hz), 2.00 (d, 1H, *J* = 12.8 Hz), 1.80 – 1.52 (m, 5H), 1.30 – 1.15 (m, 3H), 1.08 – 0.97 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.5, 82.7, 71.5, 42.8, 41.0, 29.0, 28.7, 26.4, 25.8, 25.6; IR (neat): 2926, 2853, 1761, 1449; MS (ES^+) Calculated for $[C_{10}H_{16}NaO_2]^+$: 191.1; Found: 191.1.

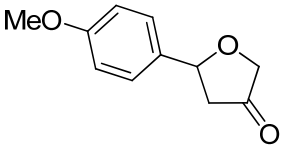
5-Phenyldihydrofuran-3-one (4h)



4h

Compound **4h** was prepared in 63% yield using **6** as oxidant according to the general procedure, and its spectroscopic data match well with those reported.[11] The reaction time was 2 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 5H), 5.29 (dd, 1H, *J* = 9.6 Hz, *J* = 6.8 Hz), 4.25 (d, 1H, *J* = 16.8 Hz), 4.01 (d, 1H, *J* = 17.2 Hz), 2.86 (dd, 1H, *J* = 18.0 Hz, *J* = 6.0 Hz), 2.55 (dd, 1H, *J* = 18.0 Hz, *J* = 9.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.2, 139.9, 128.7, 128.3, 125.8, 79.3, 71.7, 44.7; IR (neat): 3087, 3063, 3033, 2959, 2915, 2879, 2829, 1759, 1495, 1372; MS (ES^+) Calculated for $[C_{10}H_{10}NaO_2]^+$: 185.1; Found: 185.1.

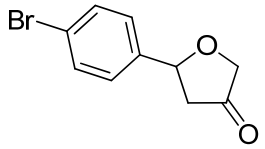
5-(4-Methoxyphenyl)dihydro-furan-3-one (4i)



4i

Compound **4i** was prepared in 75% yield using **6** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, 2H, *J* = 8.0 Hz), 6.92 (d, 2H, *J* = 8.8 Hz), 5.22 (dd, 1H, *J* = 9.6 Hz, *J* = 6.4 Hz), 4.22 (d, 1H, *J* = 17.2 Hz), 3.98 (d, 1H, *J* = 17.2 Hz), 3.82 (s, 3H), 2.81 (dd, 1H, *J* = 17.6 Hz, *J* = 6.0 Hz), 2.55 (dd, 1H, *J* = 18.0 Hz, *J* = 9.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.5, 159.6, 131.8, 127.3, 114.1, 79.1, 71.7, 55.3, 44.6; IR (neat): 3040, 3006, 2960, 2883, 2838, 1759, 1516, 1464, 1304; MS (ES^+) Calculated for $[C_{11}H_{12}NaO_3]^+$: 215.1; Found: 215.1.

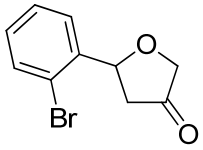
5-(4-Bromophenyl)dihydrofuran-3-one (4j)



4j

Compound **4j** was prepared in 66% yield using **6** as oxidant according to the general procedure. The reaction time was 2 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, 2H, *J* = 8.4 Hz), 7.24 (d, 2H, *J* = 8.8 Hz), 5.20 (dd, 1H, *J* = 9.2 Hz, *J* = 6.4 Hz), 4.20 (d, 1H, *J* = 16.8 Hz), 3.97 (d, 1H, *J* = 17.2 Hz), 2.82 (dd, 1H, *J* = 18.0 Hz, *J* = 6.4 Hz), 2.44 (dd, 1H, *J* = 18.0 Hz, *J* = 9.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.5, 139.0, 131.8, 127.5, 122.1, 78.6, 71.7, 44.6; IR (neat): 3052, 2960, 2880, 2829, 1760, 1490, 1364; Calculated for $[C_{10}H_9BrNaO_2]^+$: 263.0; Found: 263.0.

5-(2-Bromophenyl)dihydrofuran-3-one (4k)

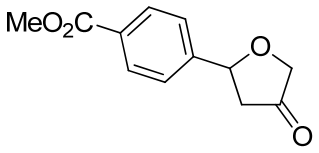


4k

Compound **4k** was prepared in 65% yield using **6** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, 1H, *J* = 8.0 Hz, *J* = 2.0 Hz), 7.56 (dd, 1H, *J* = 8.0 Hz, *J* = 1.2 Hz), 7.40 – 7.36 (m, 1H), 7.22 –

7.17 (m, 1H), 5.52 (dd, 1H, *J* = 9.2 Hz, *J* = 6.4 Hz), 4.32 (d, 1H, *J* = 17.2 Hz), 4.07 (d, 1H, *J* = 17.2 Hz), 3.13 (dd, 1H, *J* = 18.4 Hz, *J* = 6.4 Hz), 2.31 (dd, 1H, *J* = 18.0 Hz, *J* = 9.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.6, 140.0, 132.7, 129.4, 127.9, 126.4, 121.4, 78.5, 71.8, 43.7; IR (neat): 3067, 2915, 2883, 2831, 1761, 1472, 1439, 1363; Calculated for $[C_{10}H_9BrNaO_2]^+$: 263.0; Found: 263.0.

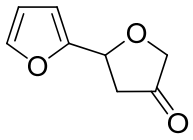
Methyl 4-(4-oxotetrahydrofuran-2-yl)benzoate (4l)



4l

Compound **4l** was prepared in 55% yield using **6** as oxidant according to the general procedure. The reaction time was 2 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, 2H, *J* = 8.4 Hz), 7.42 (d, 2H, *J* = 8.4 Hz), 5.28 (dd, 1H, *J* = 6.4 Hz, *J* = 9.6 Hz), 4.19 (d, 1H, *J* = 17.2 Hz), 3.96 (d, 1H, *J* = 12.8 Hz), 3.86 (s, 3H), 2.83 (dd, 1H, *J* = 18 Hz, *J* = 6.4 Hz), 2.44 (dd, 1H, *J* = 18 Hz, *J* = 9.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.3, 166.6, 145.1, 130.0, 125.6, 78.8, 71.7, 52.2, 44.6; IR (neat): 2954, 2884, 1761, 1719, 1612, 1435; Calculated for $[C_{12}H_{12}NaO_4]^+$: 243.1; Found: 243.1.

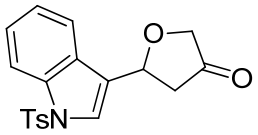
2,3-Dihydro[2,2']bifuranyl-4-one (4m)



4m

Compound **4m** was prepared in 76% yield using **6** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.43 (m, 1H), 6.38 – 6.36 (m, 2H), 5.36 (t, 1H, *J* = 7.2 Hz), 4.12 (d, 1H, *J* = 17.2 Hz), 3.98 (d, 1H, *J* = 17.2 Hz), 2.82 – 2.76 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.5, 152.1, 143.2, 110.4, 108.6, 72.2, 70.4, 40.7; IR (neat): 3120, 2925, 2884, 2834, 1760, 1504, 1435, 1403, 1352; Calculated for $[C_8H_8NaO_3]^+$: 175.0; Found: 175.0.

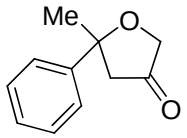
5-[1-(Toluene-4-sulfonyl)-1H-indol-3-yl]dihydrofuran-3-one (4n)



4n

Compound **4n** was prepared in 68% yield using **6** as oxidant according to the general procedure. The reaction time was 2.5 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dd, 1H, *J* = 8.4 Hz, *J* = 0.8 Hz), 7.78 (dd, 2H, *J* = 8.4 Hz, *J* = 2.0 Hz), 7.58 (dd, 2H, *J* = 8.8 Hz, *J* = 1.2 Hz), 7.38 – 7.34 (m, 1H), 7.28 – 7.22 (m, 3H), 5.53 (dd, 1H, *J* = 8.0 Hz, *J* = 6.8 Hz), 4.16 (d, 1H, *J* = 17.2 Hz), 4.02 (d, 1H, *J* = 17.2 Hz), 2.91 (dd, 1H, *J* = 18.0 Hz, *J* = 6.4 Hz), 2.72 (dd, 1H, *J* = 18.4 Hz, *J* = 8.8 Hz), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.6, 145.2, 135.5, 135.1, 130.0, 128.5, 126.9, 125.2, 123.4, 123.2, 121.4, 120.1, 113.8, 73.3, 70.9, 42.6, 21.6; IR (neat): 3112, 3054, 2918, 2849, 1761, 1447, 1369; MS (ES^+) Calculated for $[C_{19}H_{17}NNaO_4S]^+$: 378.1; Found: 378.1.

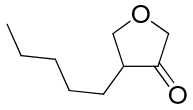
5-Methyl-5-phenyldihydrofuran-3-one (4o)



4o

Compound **4o** was prepared in 58% yield using **5** as oxidant according to the general procedure. The reaction time was 6 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.33 (m, 4H), 7.29 – 7.24 (m, 1H), 4.14 (d, 1H, *J* = 17.2 Hz), 3.95 (d, 1H, *J* = 17.2 Hz), 2.91 (d, 1H, *J* = 17.2 Hz), 2.68 (d, 1H, *J* = 17.6 Hz), 1.66 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.8, 144.4, 128.6, 127.4, 124.7, 83.7, 70.3, 49.9, 30.1; IR (neat): 2980, 2930, 1762, 1447, 1375; MS (ES^+) Calculated for $[C_{11}H_{12}NaO_2]^+$: 199.1; Found: 199.1.

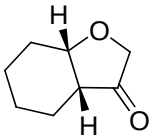
4-Butyldihydrofuran-3-one (4p)



4p

Compound **4p** was prepared in 65% yield using **5** as oxidant according to the general procedure. The reaction time was 3 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.42 (dd, 1H, *J* = 8.4 Hz, *J* = 9.2 Hz), 4.02 (d, 1H, *J* = 16.8 Hz), 3.82 – 3.75 (m, 2H), 2.46 – 2.40 (m, 1H), 1.79 – 1.74 (m, 1H), 1.40– 1.24 (m, 7H), 0.88 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 216.9, 72.5, 71.0, 47.2, 31.6, 27.8, 27.2, 22.4, 13.9; IR (neat): 2931, 2859, 1760, 1462; Calculated for $[C_9H_{16}NaO_2]^+$: 179.1; Found: 179.1.

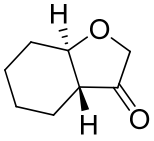
Hexahydrobenzofuran-3-one (4q)



4q

Compound **4q** was prepared in 82% yield using **5** as oxidant according to the general procedure, and its spectroscopic data match well with those reported.[12] The reaction time was 4 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.35 (dd, 1H, *J* = 11.2 Hz, *J* = 6.4 Hz), 4.09 (d, 1H, *J* = 17.2 Hz), 3.93 (d, 1H, *J* = 16.8 Hz), 2.43 (dd, 1H, *J* = 13.6 Hz, *J* = 6.8 Hz), 1.82 – 1.56 (m, 6H), 1.44 – 1.33 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 216.8, 76.7, 69.9, 46.7, 27.7, 23.0, 22.4, 20.9; IR (neat): 2937, 2858, 1756, 1448.

Hexahydro-benzofuran-3-one (4r)

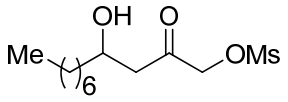


4r

Compound **4r** was prepared in 88% yield at 0 °C using **5** as oxidant according to the general procedure, and its spectroscopic data match well with those reported.[13] The reaction time was 5 h. Due to its propensity for epimerization, the reaction mixture was washed with saturated aqueous $NaHCO_3$ before concentration instead of direct

concentration, and the resulting residue was passed through silica gel column quickly. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.17 (dd, 1H, *J* = 16.0 Hz, *J* = 1.2 Hz), 3.80 (d, 1H, *J* = 17.6 Hz), 3.48 (dt, 1H, *J* = 11.6 Hz, *J* = 4.0 Hz), 2.28 – 2.25 (m, 1H), 2.14 – 2.09 (m, 1H), 1.95 – 1.88 (m, 2H), 1.84 – 1.81 (m, 1H), 1.62 – 1.53 (m, 1H), 1.31 – 1.16 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.5, 82.3, 72.0, 53.7, 32.3, 25.1, 23.8, 23.0; IR (neat): 2939, 2863, 1763, 1447, 1358.

4-Hydroxy-2-oxoundecyl methanesulfonate (7)



7

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (s, 2H), 4.17 – 4.07 (m, 1H), 3.20 (s, 3H), 2.60 – 2.58 (m, 2H), 2.38 (d, 1H, *J* = 4.4 Hz), 1.57 – 1.25 (m, 12H), 0.88 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 203.3, 72.0, 67.7, 45.6, 38.9, 36.9, 31.7, 29.4, 29.2, 25.4, 22.6, 14.1; IR (neat): 2922, 2851, 1762, 1367; Calculated for $[C_{12}H_{24}NaO_5S]^+$: 303.1; Found: 303.1.

Reference:

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Russell, C. A. *J. Org. Chem.* **2004**, *69*, 3886-3898.

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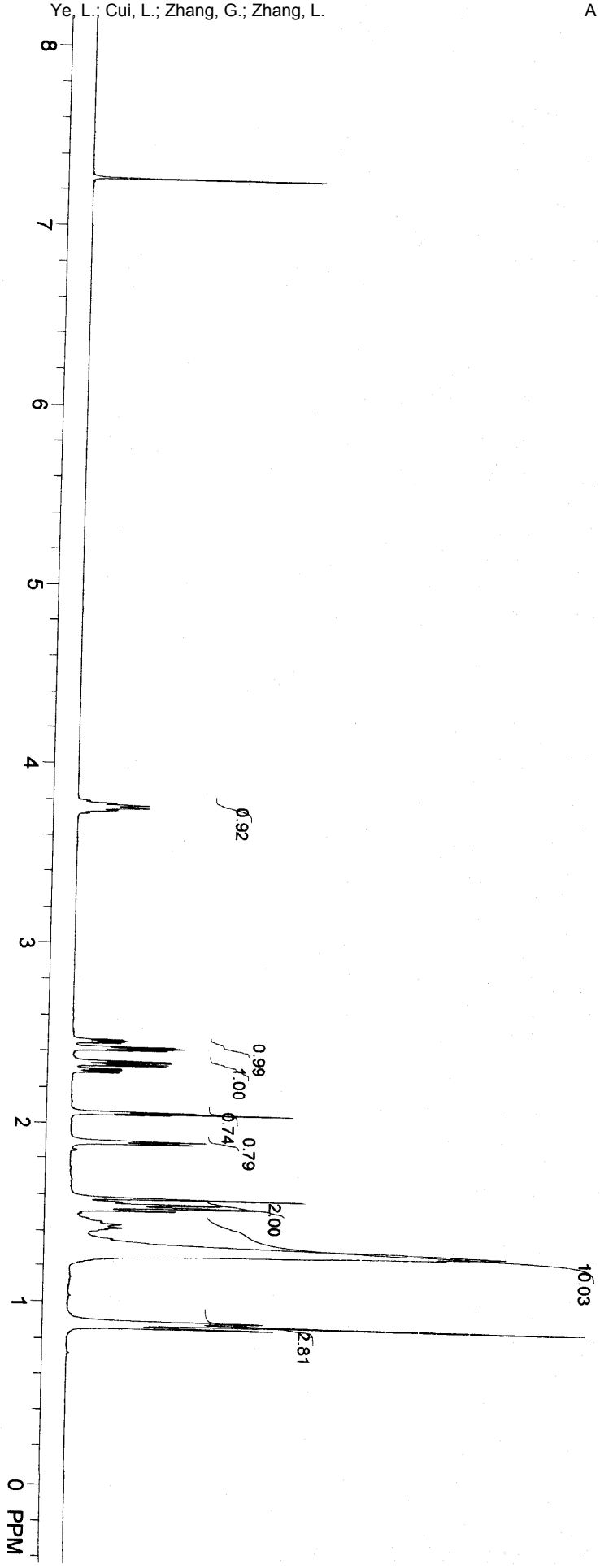

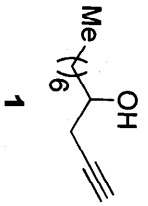

1

ylw-3-71-c

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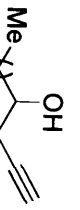
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

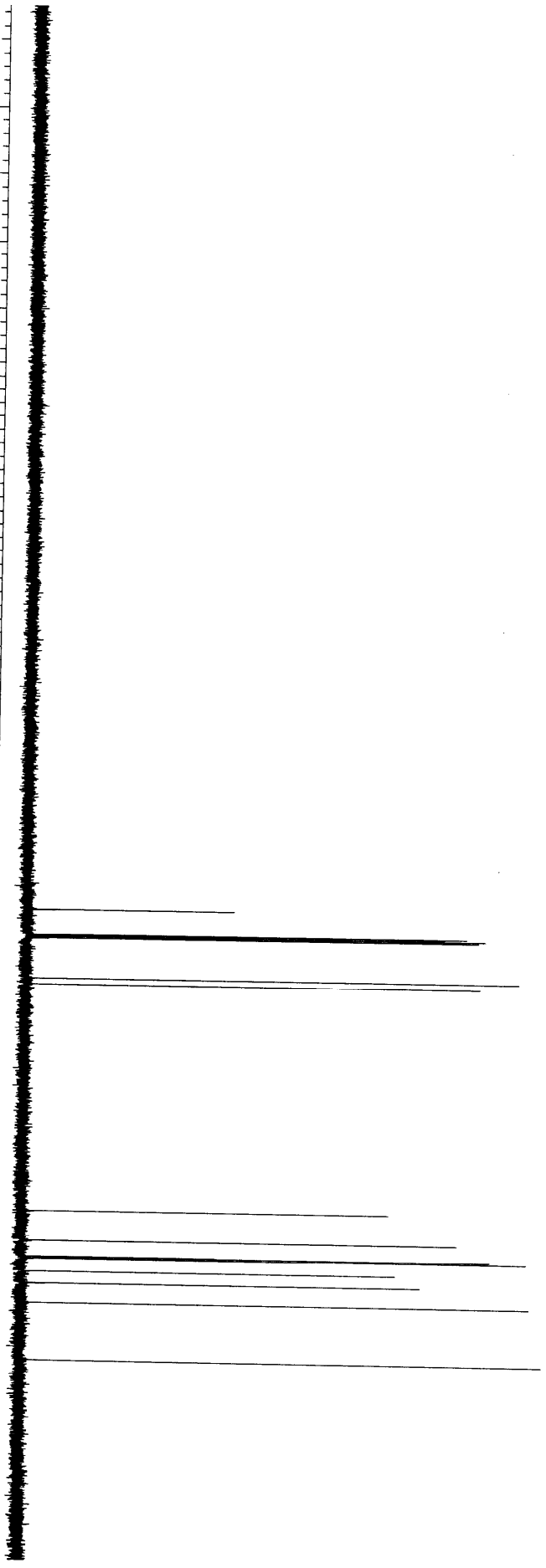
Pulse Sequence: s2pul
Solvent: cdcl3

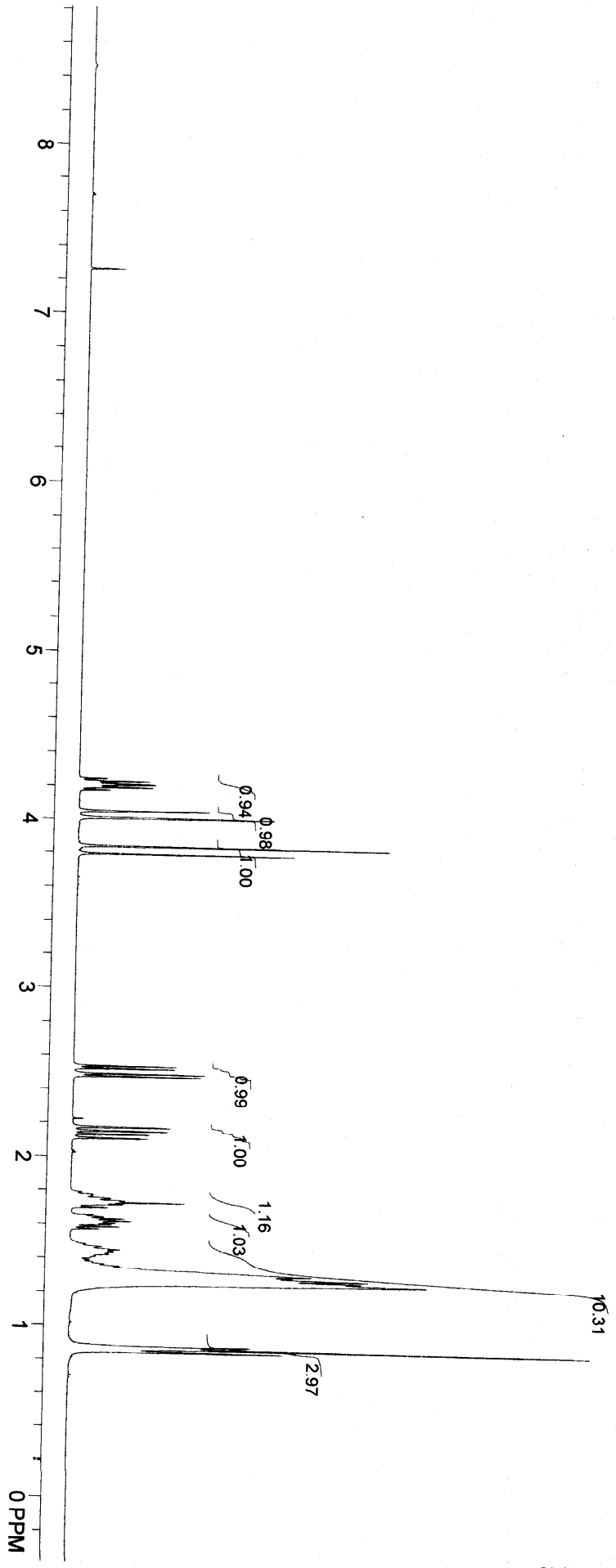
Temp. 22.0 C / 295.1 K
Operator: lye

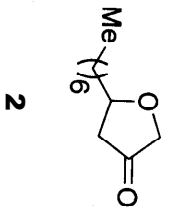
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
width 40000.0 Hz
108 repetitions
OBSERVE C13, 125.6889841 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min



1







2

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ylw-3-118-c

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 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 52 repetitions
OBSERVE  C13, 125.6889866 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
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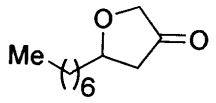
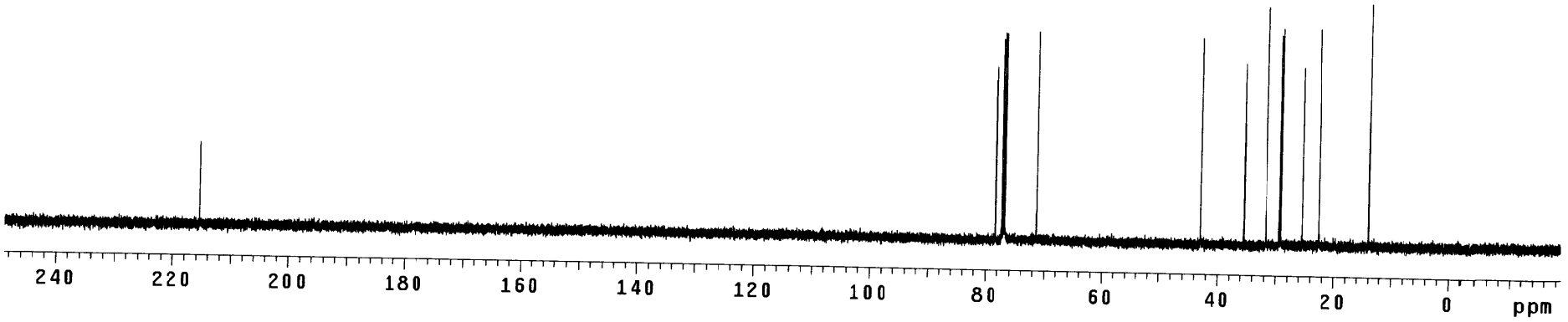
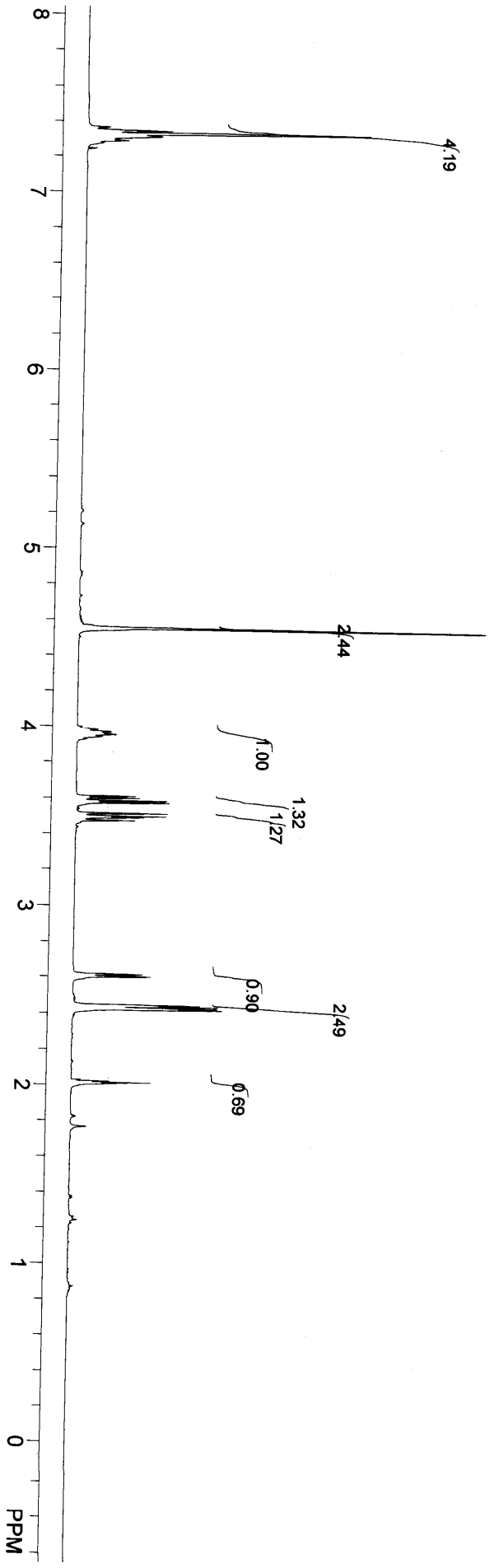
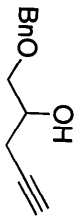


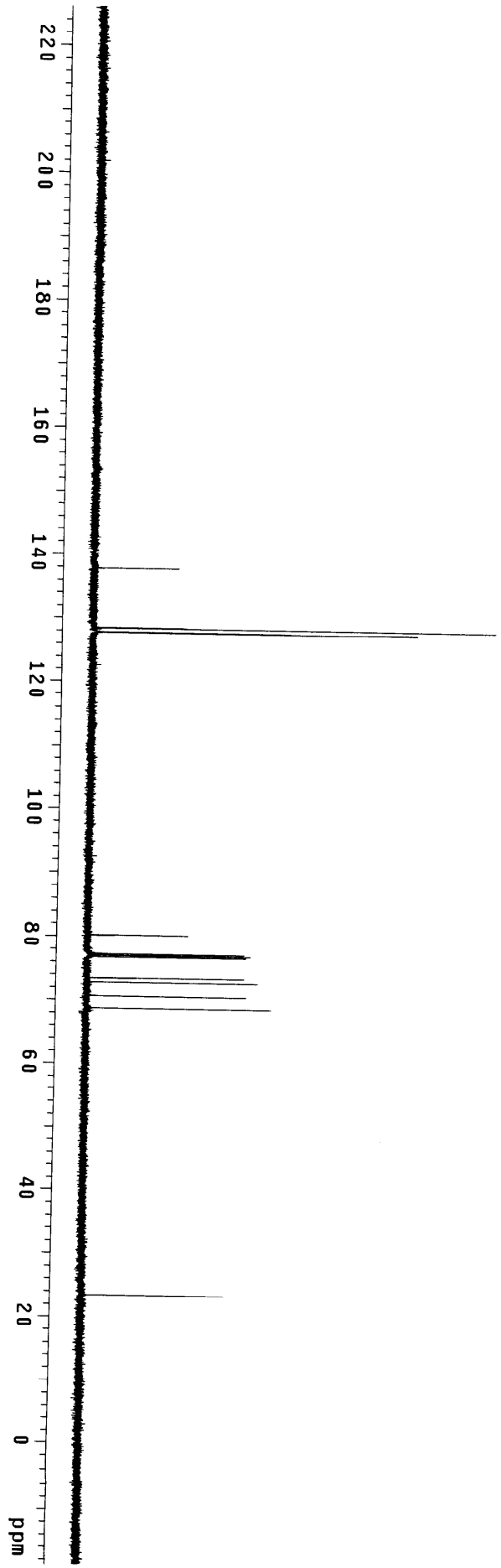
2

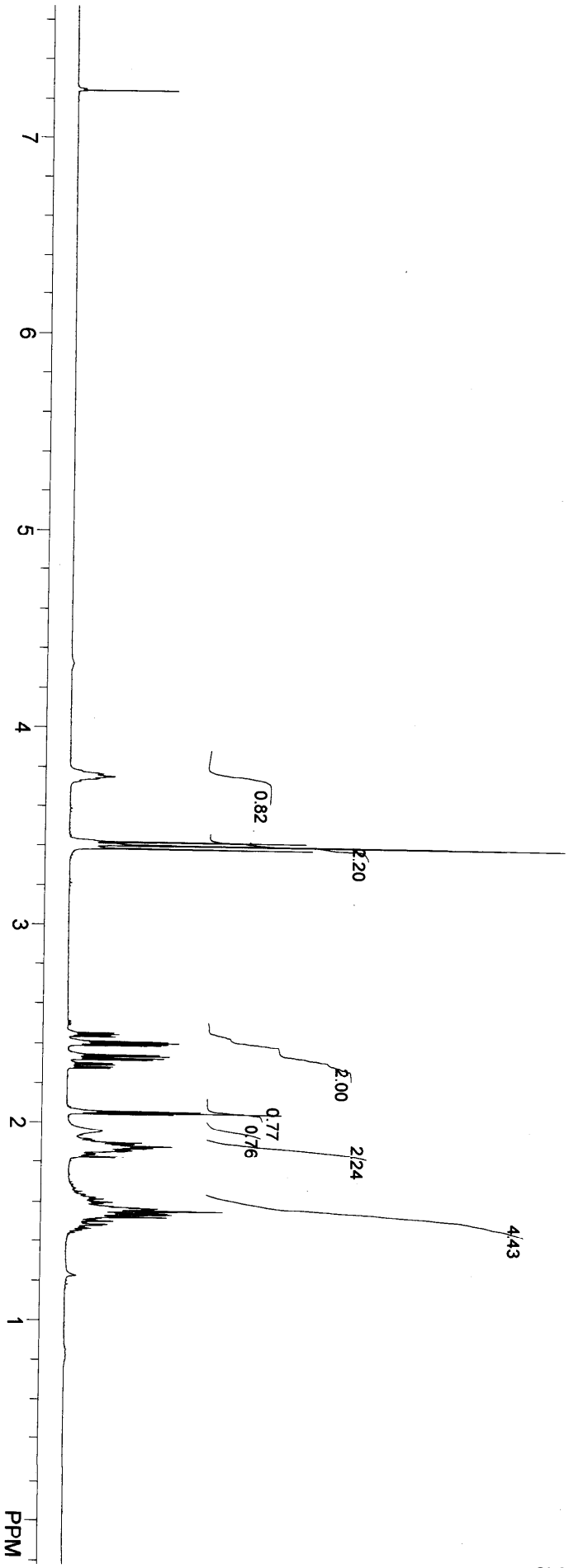


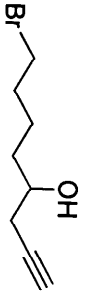




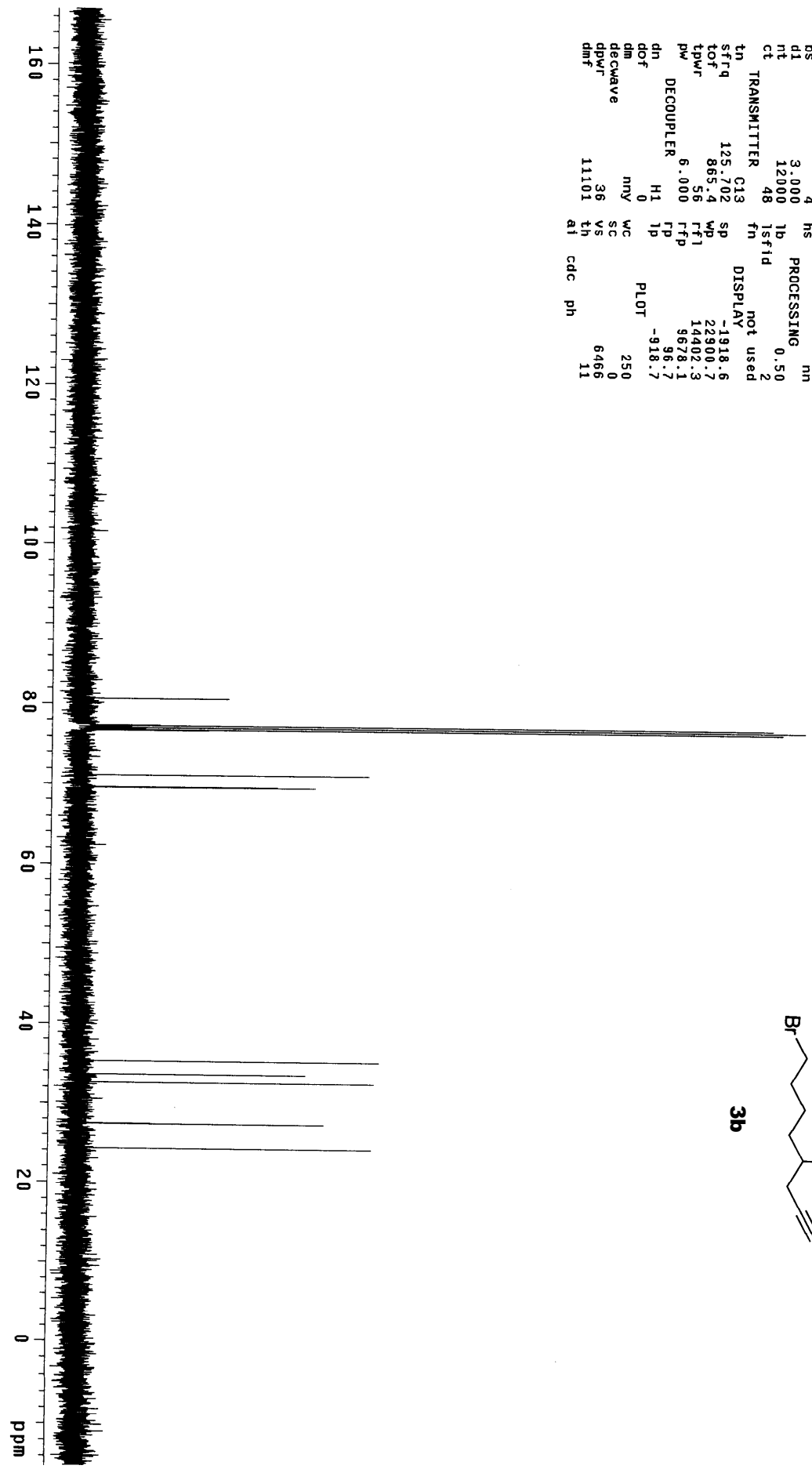
3a


ylw-3-164-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
48 repetitions
OBSERVE C13, 125.6889908 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
BnO
OH
3a
220
200
180
160
140
120
100
80
60
40
20
0
ppm





3b


STANDARD CARBON PARAMETERS
exp1 CARBON
SAMPLE SPECIAL
date Jan 1 2010 temp 22.0
solvent cdcl3 gain 54
file /home/Zhang/gr~ spin 20
uzhang/C13-200121~ hst 0.008
8-1.fid pw90 9.200
ACQUISITION alfa 10.000
sw 34995.6 fl FLAGS n
at 1.301 in n
np 91024 dp y
fb 19000 hs nn
bs 4 PROCESSING
d1 3.000 lb 0.50
nt 12000 lsfid 2
ct 48 fn not used
TRANSMITTER DISPLAY
tn C13 sp -1918.6
sfrq 125.702 wp 22900.7
tof 865.4 rfl 14402.3
tpwr 56 rfp 9678.1
pw 6.000 rp 96.7
DECOUPLER lp -918.7
dn H1 PLOT
dof 0 250
dm nny wc 0
decwave sc 6466
dpwr 36 vs 11
dmf 11101 th
ai cdc ph
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140
120
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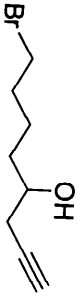
0
ppm


Br
OH

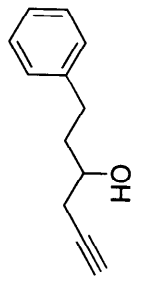
3b

licui-3-108-1-SM-1H

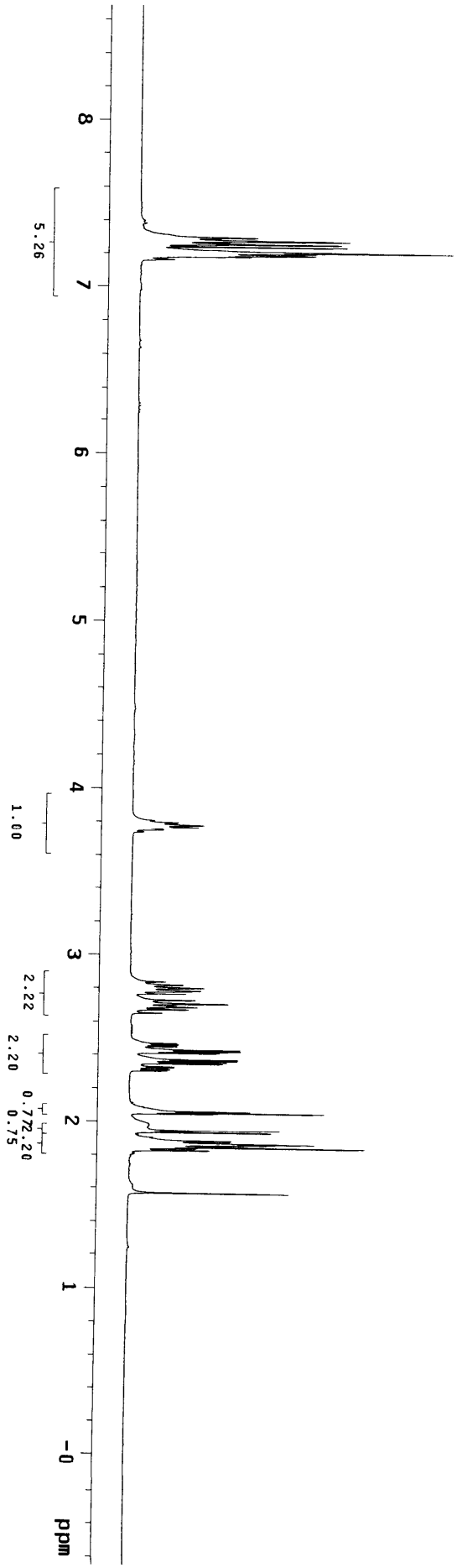
File: home/Zhang/licui/licui-3-108-1-SM-1H.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: lcuj
File: licui-3-108-1-SM-1H
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
8 repetitions
OBSERVE H1, 399.9486787 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 43 sec



3c



```
licui-3-108-1-SM-13C

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: licui

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 28258.6 Hz
 24 repetitions
OBSERVE  C13, 125.6889961 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 131072
Total time 0 min
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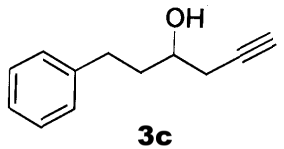
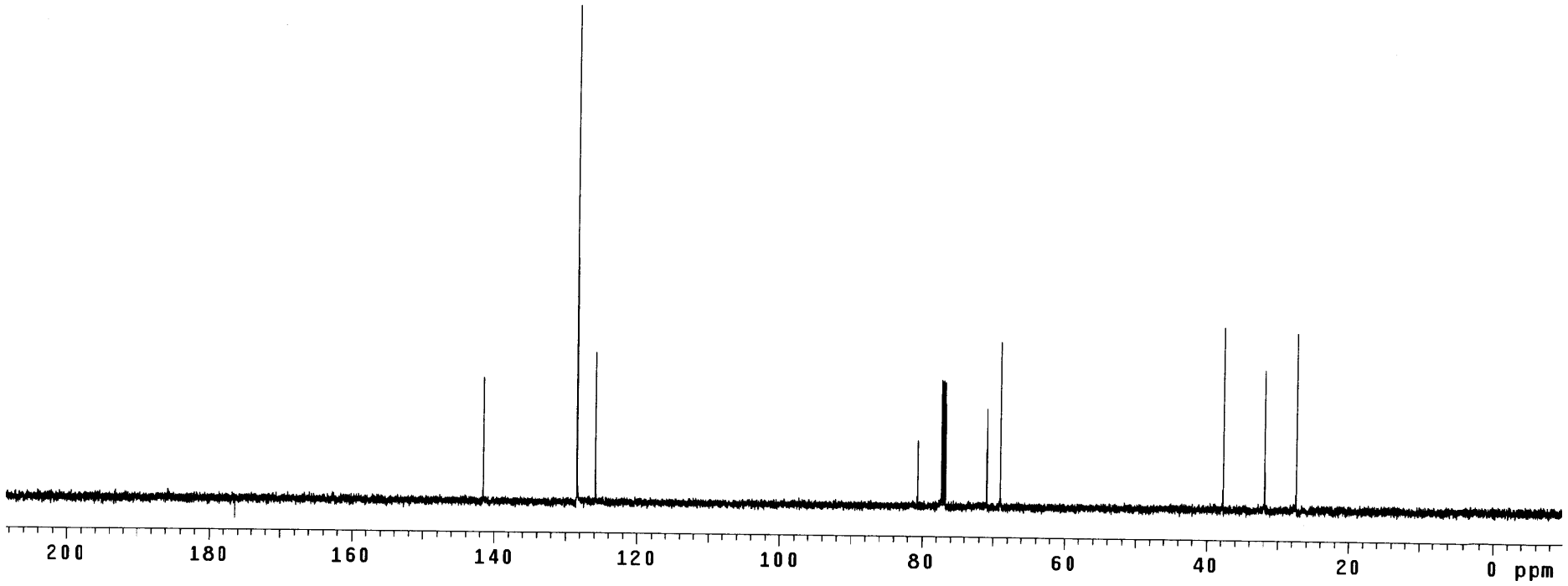
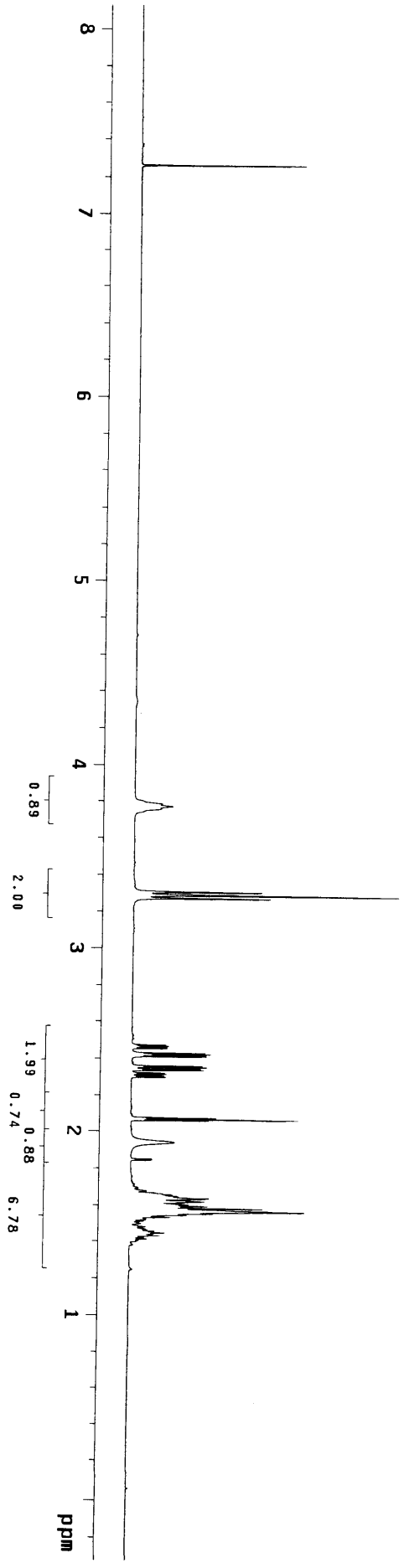


3c





licui-3-109-2-1HH

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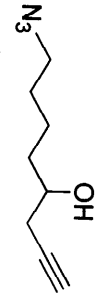
Solvent: cdcl3
Ambient temperature
Operator: licui
File: licui-3-109-2-1HH
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
8 repetitions
OBSERVE H1, 399.9486725 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 43 sec



3d

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licui-3-117-2-13C

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 Archive directory:
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 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 24.5 C / 297.6 K
Operator: lcui

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 28258.6 Hz
 40 repetitions
OBSERVE  C13, 125.6889888 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 131072
Total time 0 min
```
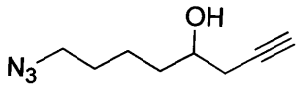
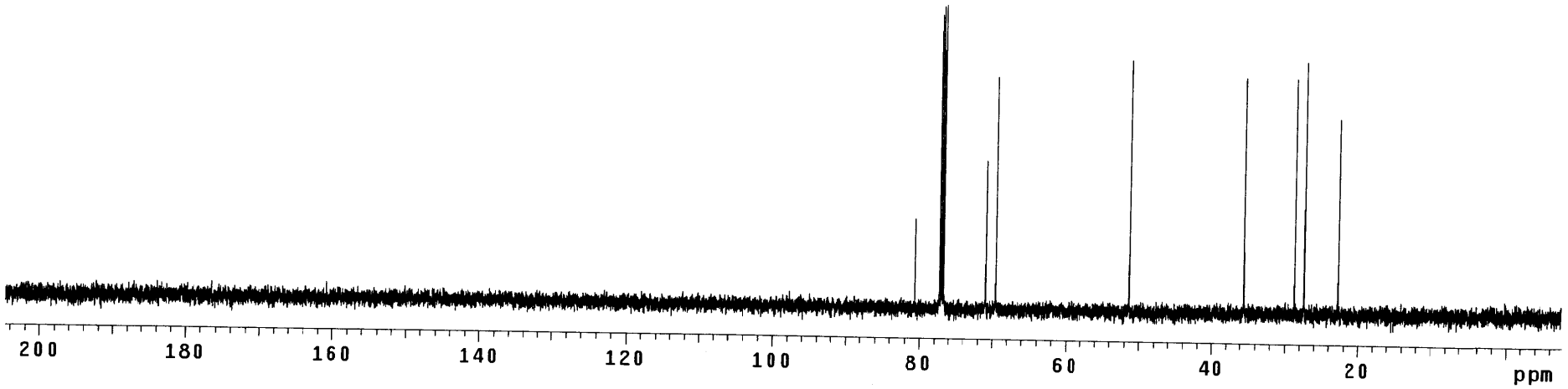
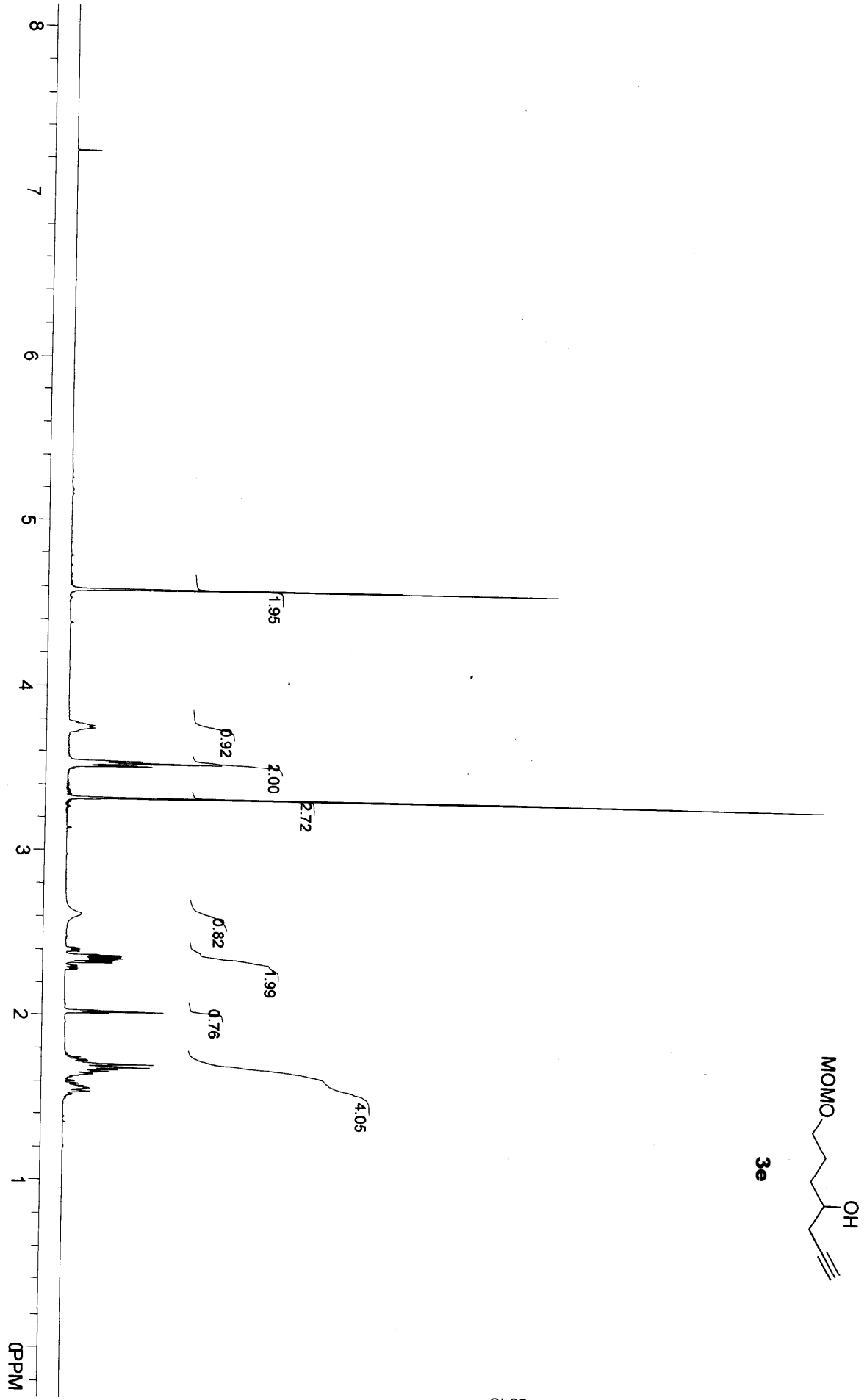



3d

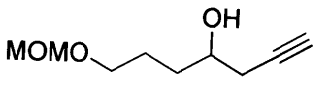



8
7
6
5
4
3
2
1
0
PPM
1.95
0.92
2.00
2.72
0.82
1.99
0.76
4.05
MOMO
OH
3e

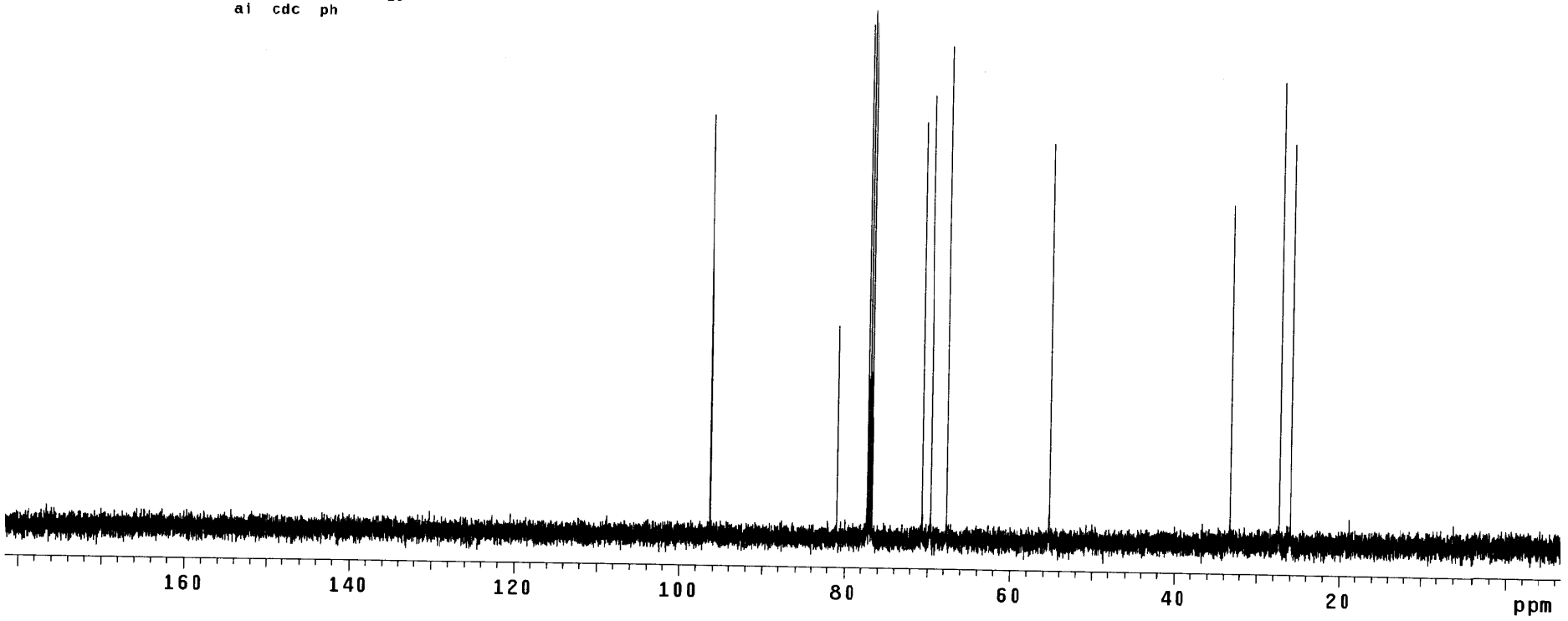
STANDARD CARBON PARAMETERS

exp1 CARBON

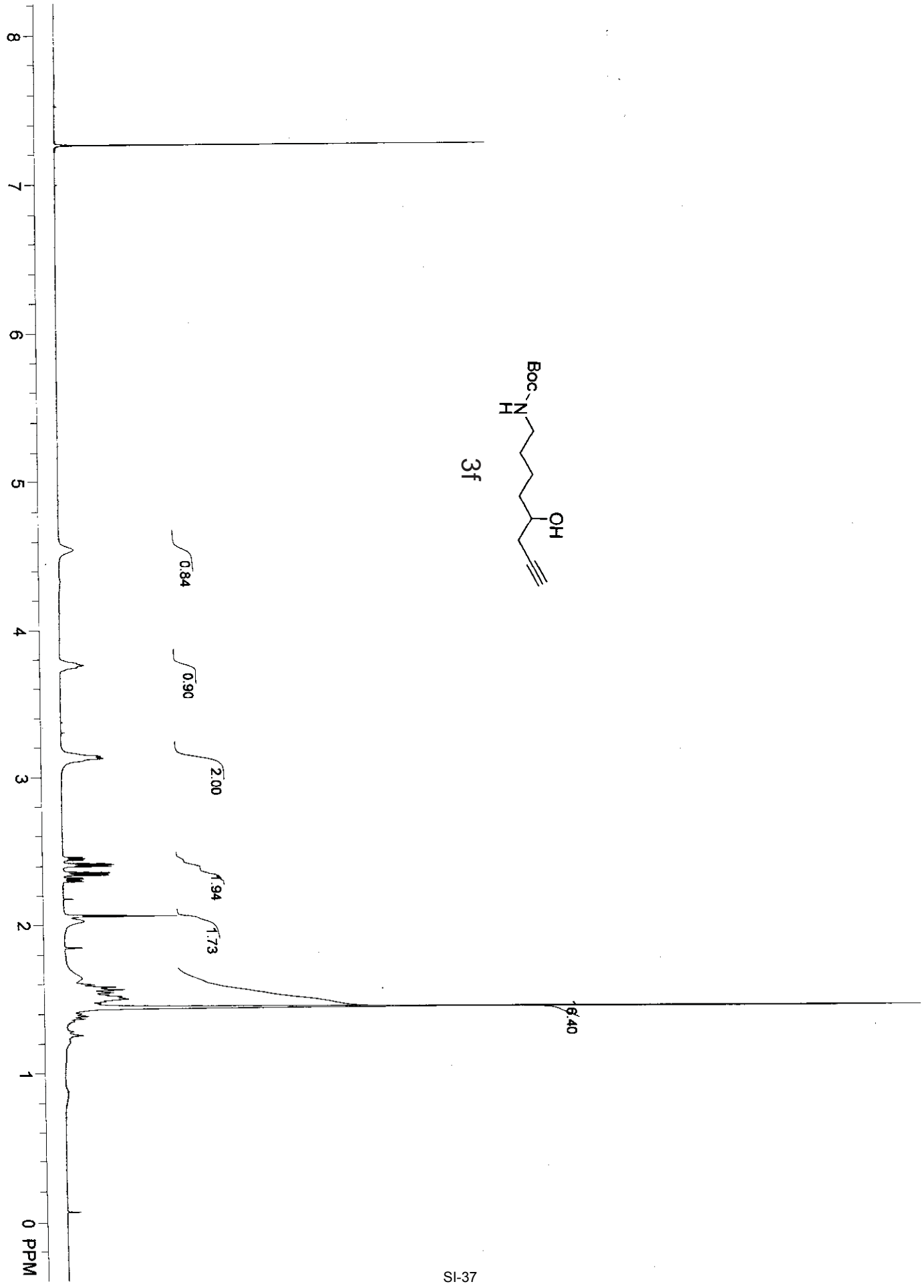
SAMPLE		SPECIAL	
date	Jan 1 2010	temp	22.0
solvent	cdcl3	gain	54
file	/home/Zhang/g~	spin	20
	uzhang/C13-2010010~	hst	0.008
	1-2.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	12000	lb	0.50
ct	52	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-838.7
tof	865.4	wp	23653.4
tpwr	56	rfl	11036.8
pw	6.000	rfp	9678.1
DECOUPLER		rp	95.1
dn	H1	lp	-913.3
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	5794
dmf	11101	th	15
		ai cdc ph	

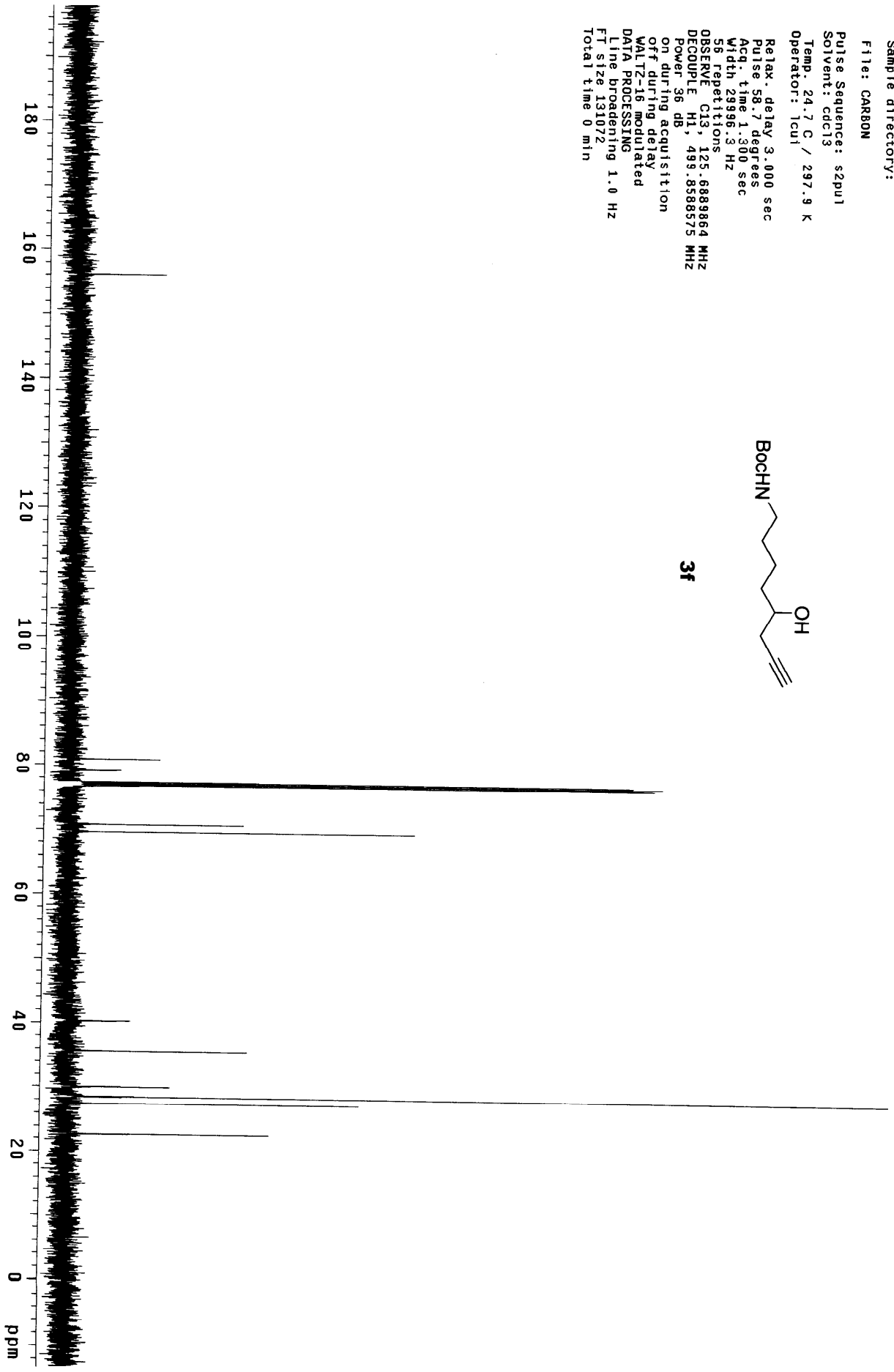


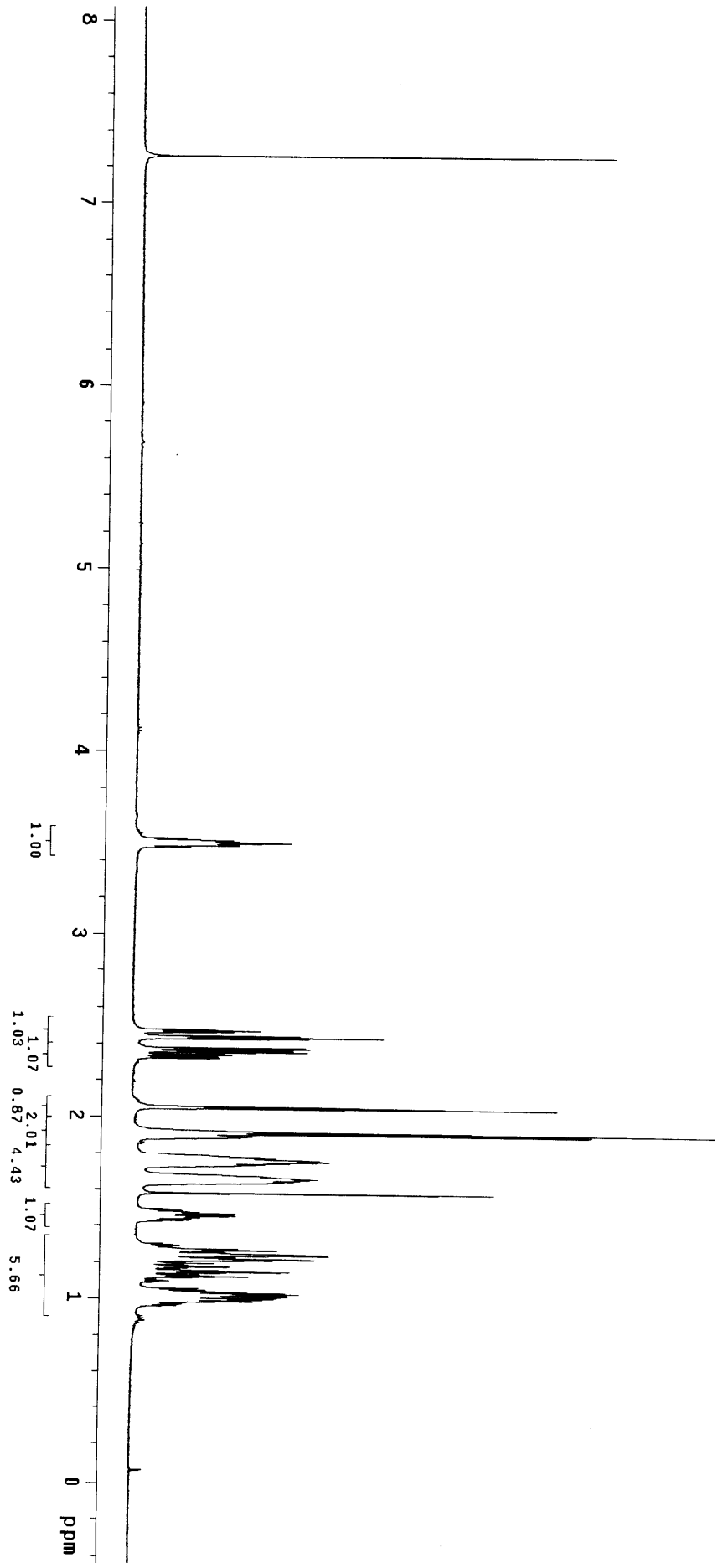
3e



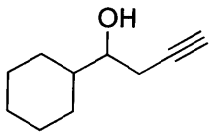
Ye, L.; Cui, L.; Zhang, G.; Zhang, L.


8
7
6
5
4
3
2
1
0 PPM
0.84
0.90
2.00
1.94
1.73
6.40
Boc
N
H
OH
3f

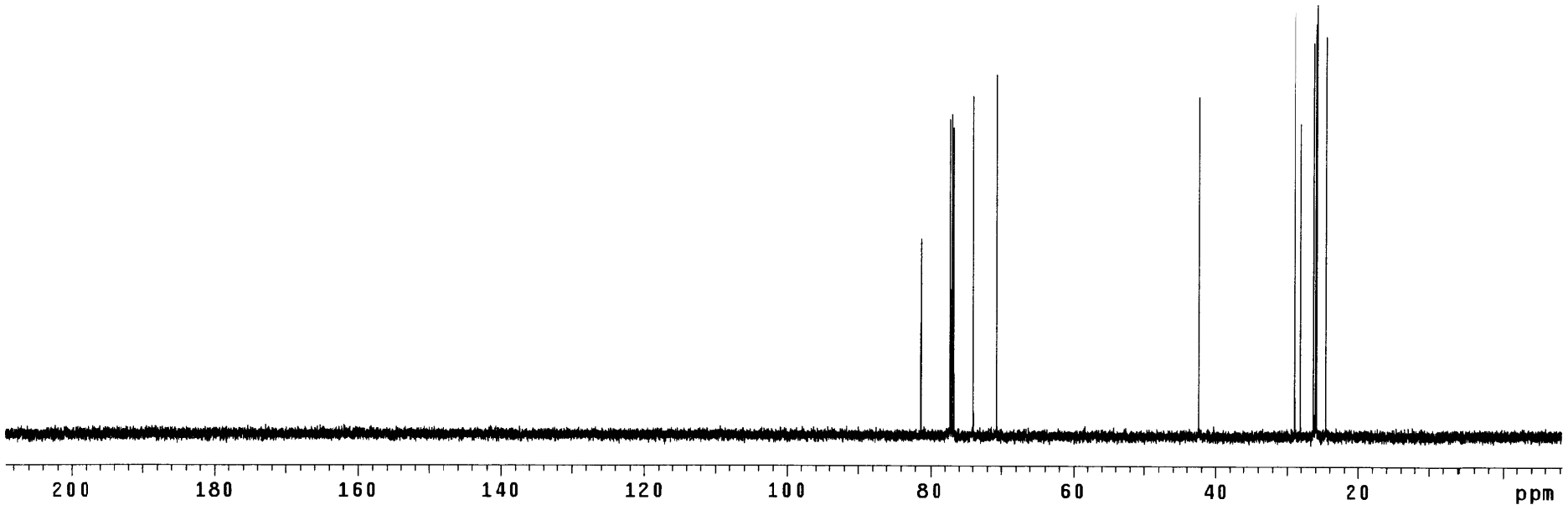

liucl-106-1-13C
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 24.7 C / 297.9 K
Operator: lcui
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 29996.3 Hz
56 repetitions
OBSERVE C13, 125.6889864 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min
BocHN
OH
3f
180
160
140
120
100
80
60
40
20
0
ppm

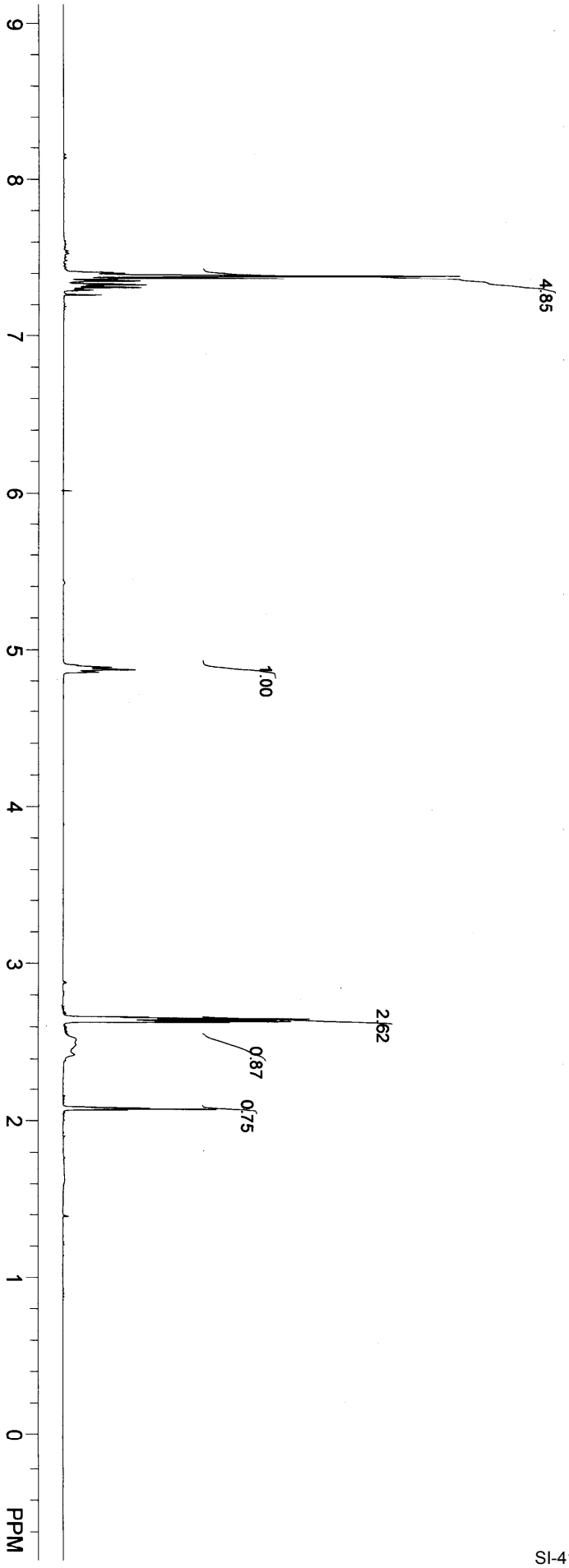

1icui-3-109-2-1H
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: H1
Pulse Sequence: s2pul
Solvent: cdcl3
Operator: lcui
Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
width 5997.0 Hz
12 repetitions
OBSERVE H1, 499.8563629 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
OH
3g
1.00
1.03
1.07
0.87
2.01
4.43
1.07
5.66
8
7
6
5
4
3
2
1
0
ppm

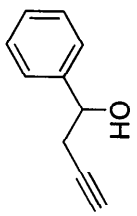
```
ylw-3-169s-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 21.9 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 86 repetitions
OBSERVE  C13, 125.6889872 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```



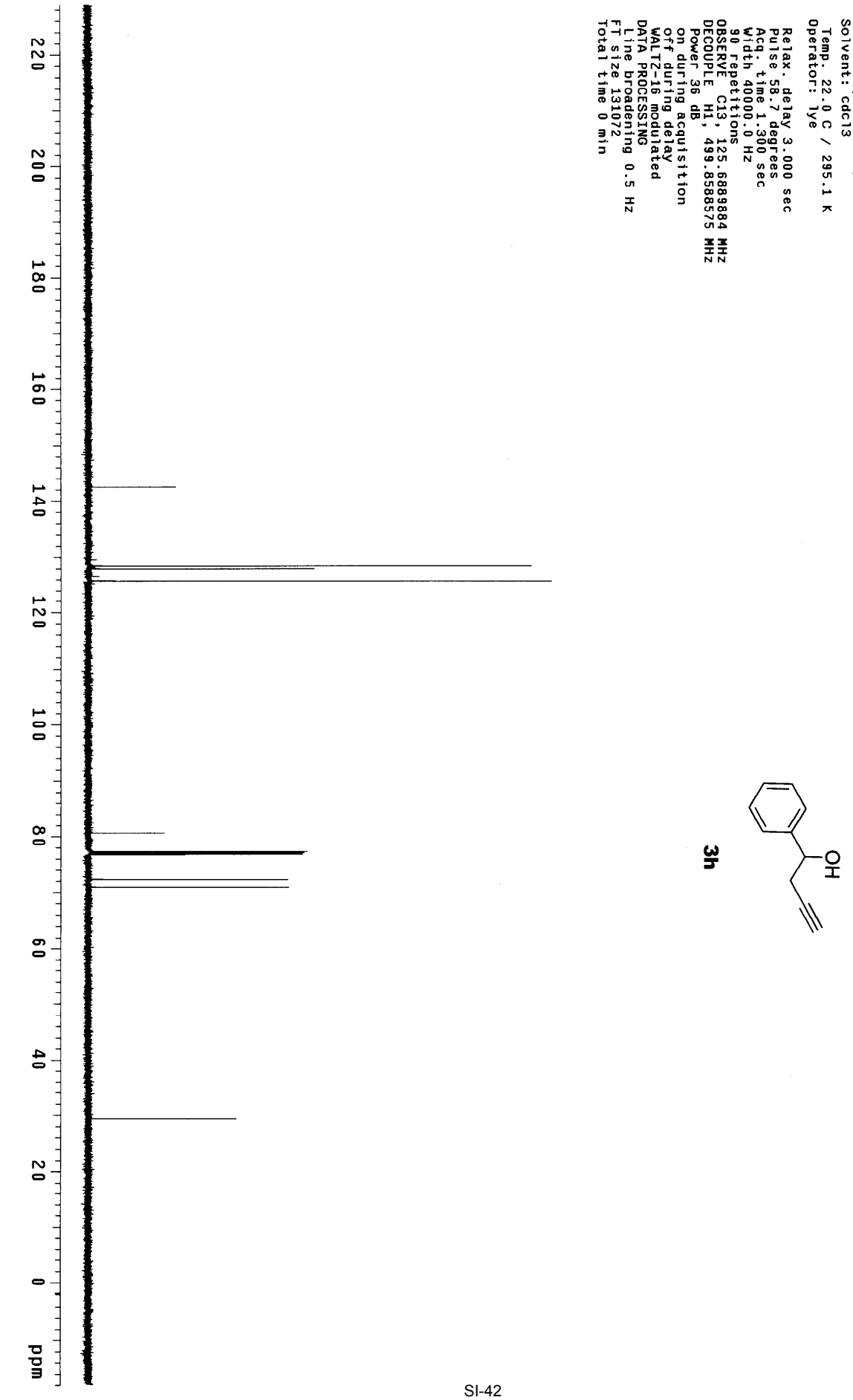
3g

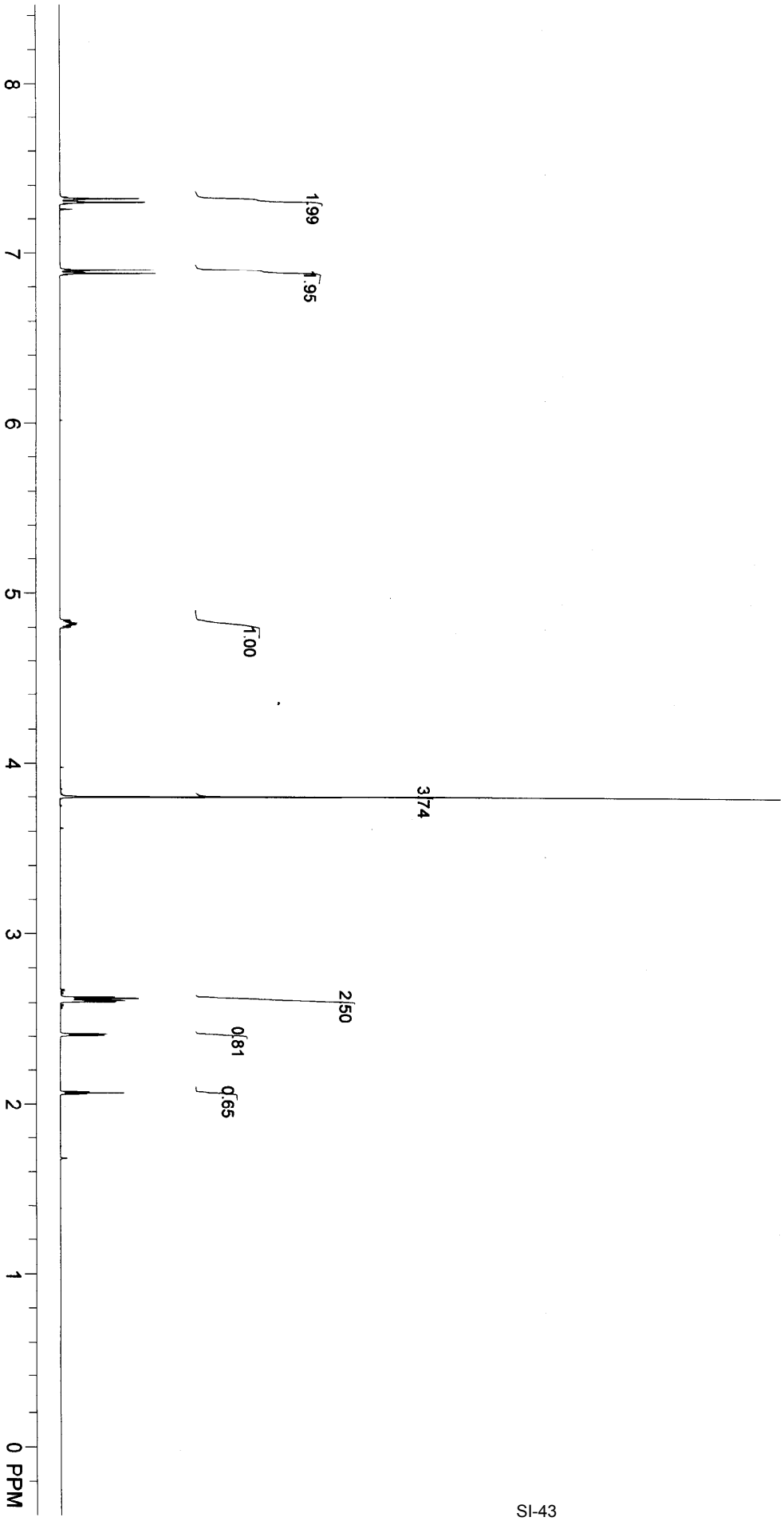


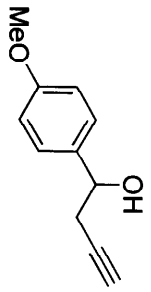




3h


ylw-3-154s-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
90 repetitions
OBSERVE C13, 125.6889884 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
OH
3h
220
200
180
160
140
120
100
80
60
40
20
0
ppm

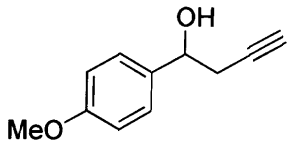




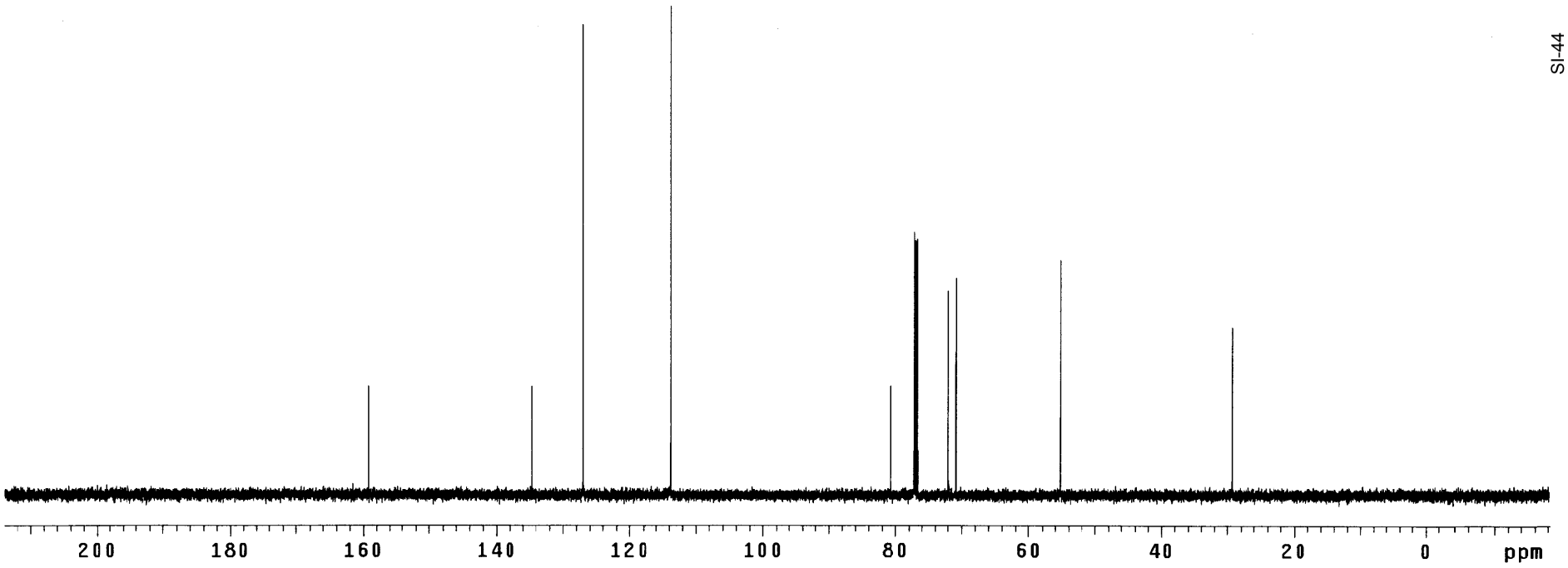
3i

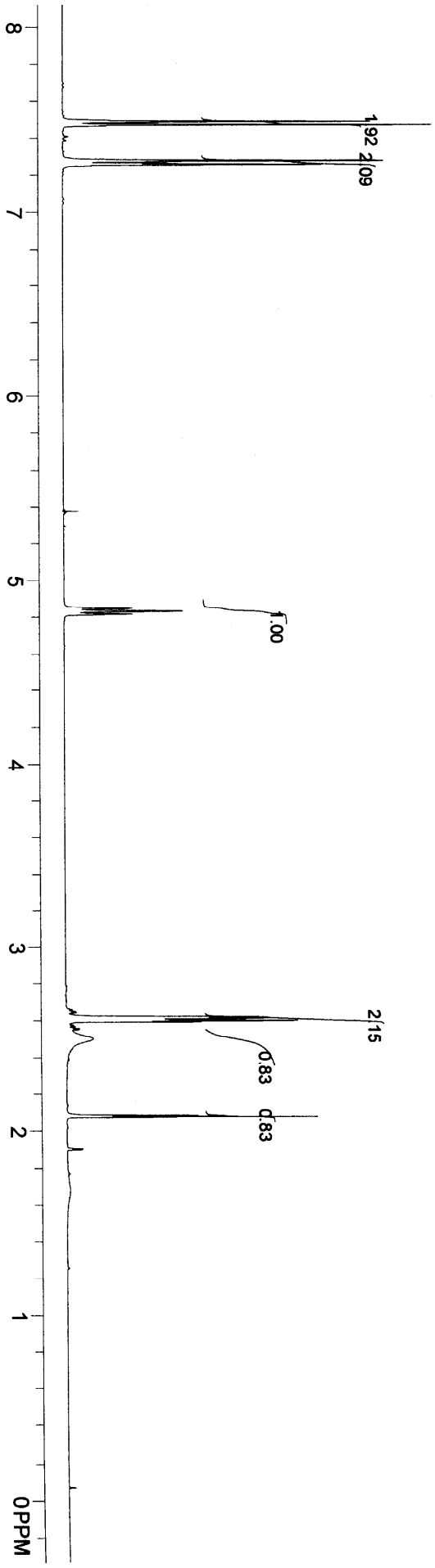
Ye, L.; Cui, L.; Zhang, G.; Zhang, L.

```
ylw-3-155s-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 52 repetitions
OBSERVE  C13, 125.6889884 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```

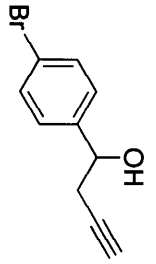


3i

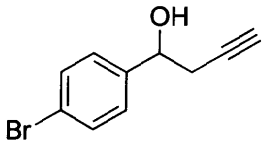




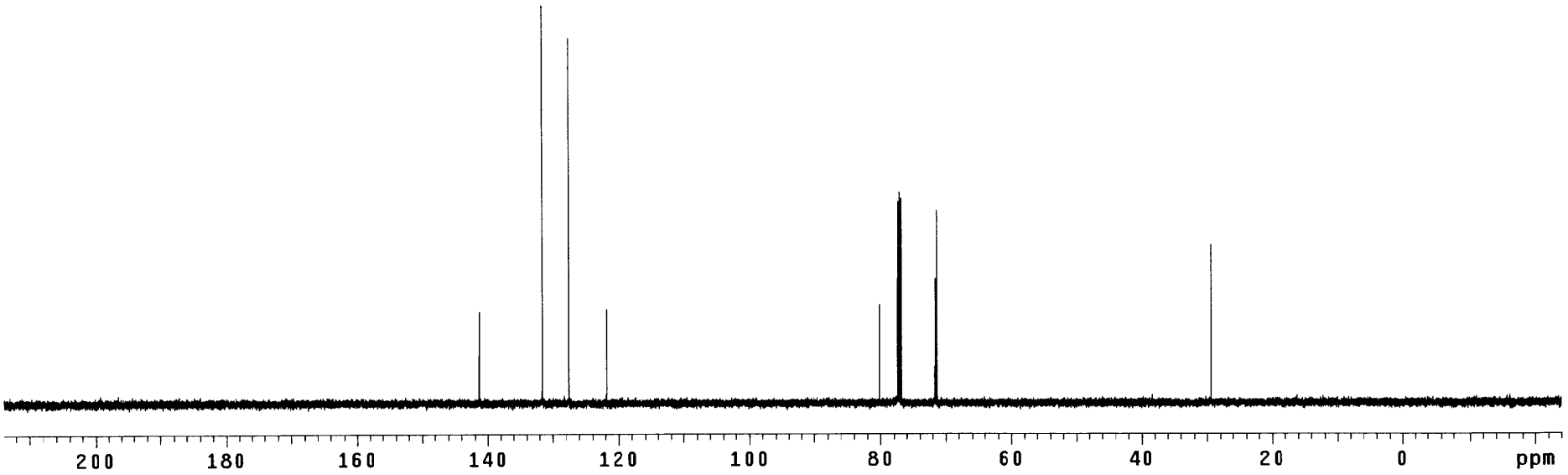
3j

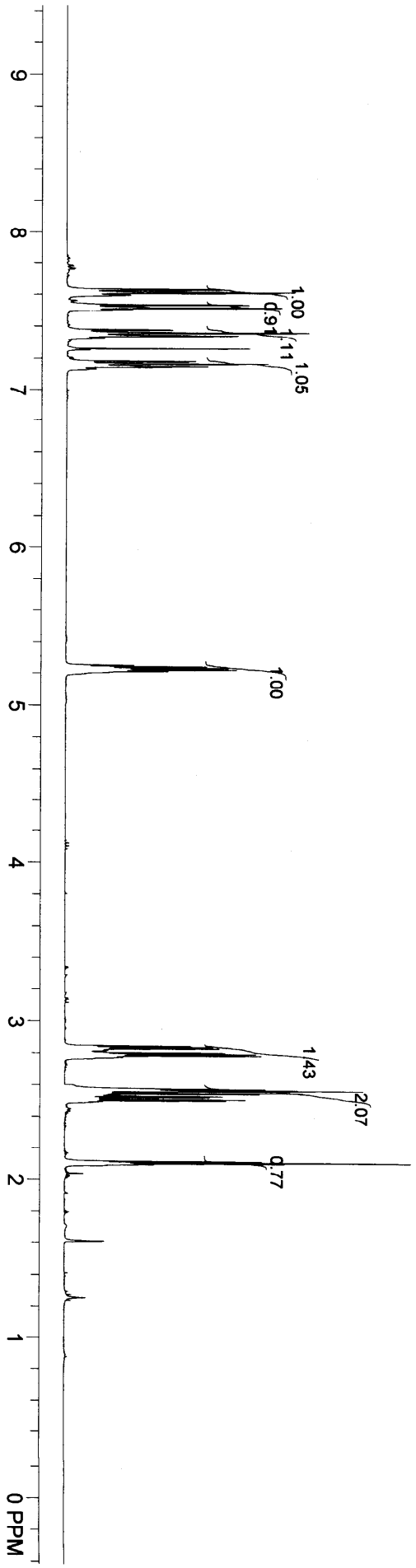


```
ylw-3-153-s

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 50 repetitions
OBSERVE  C13, 125.6889878 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```

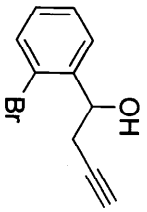


3j

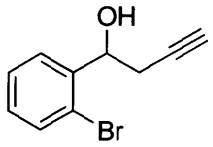




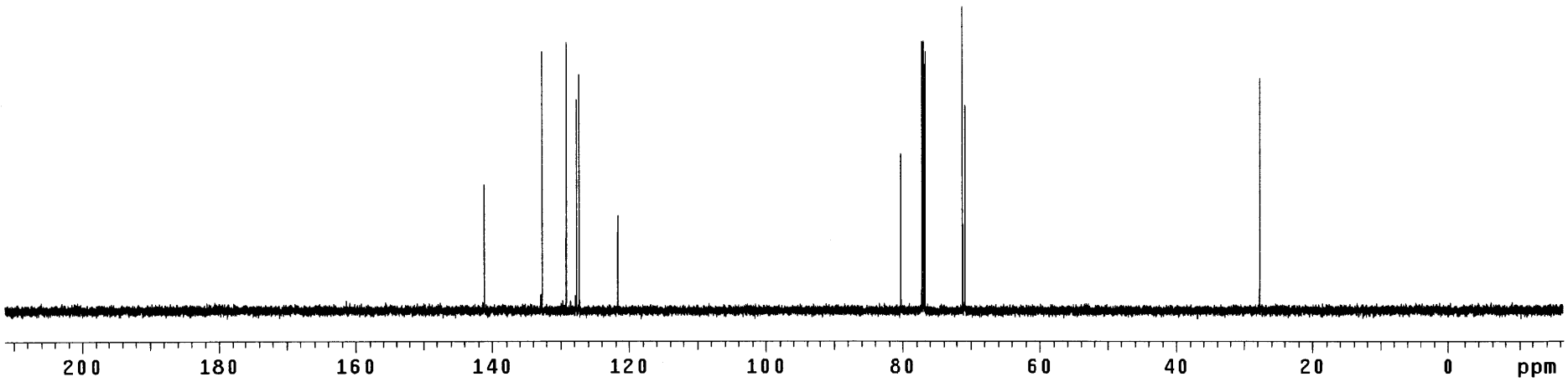
3k

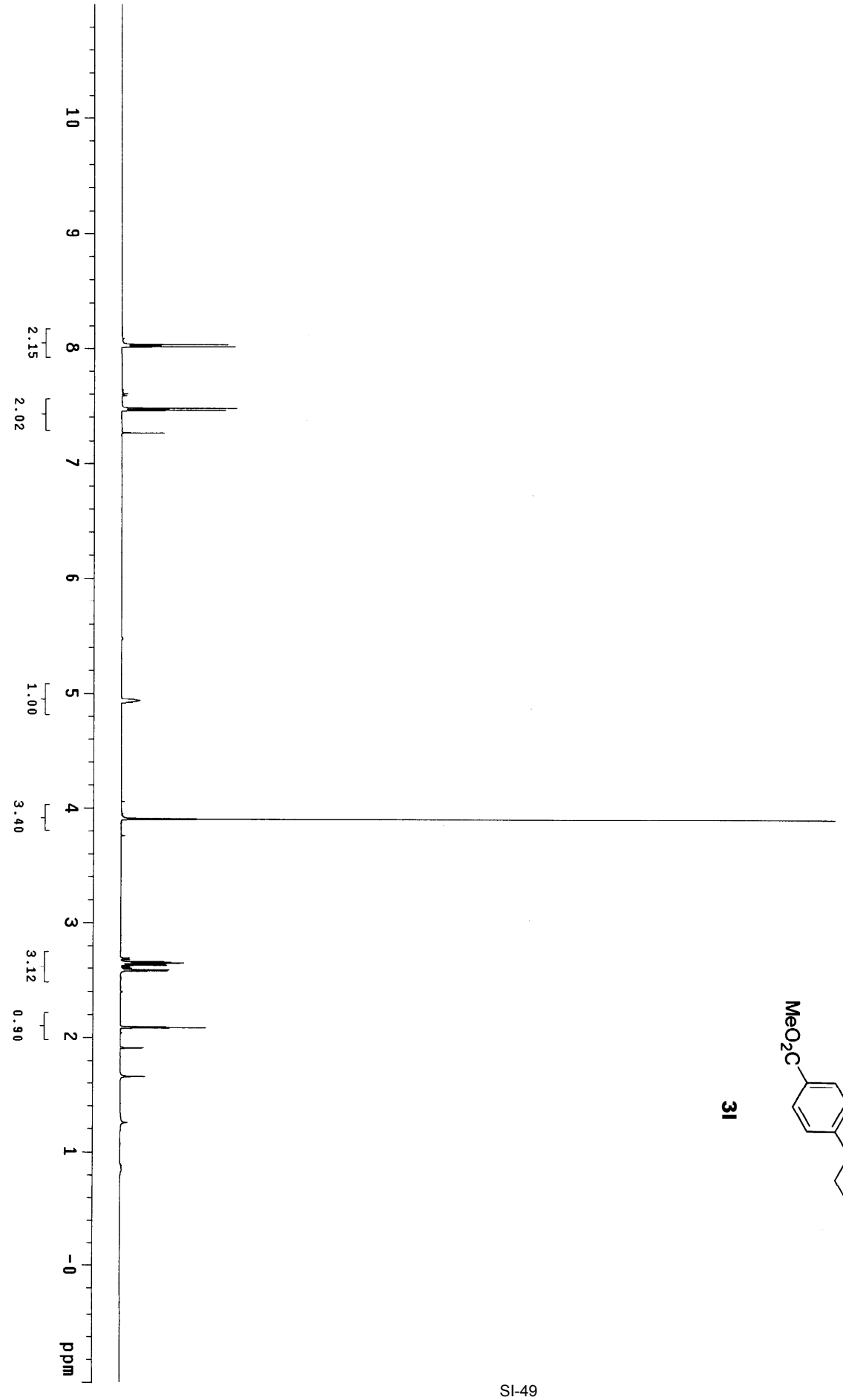


```
ylw-3-165s-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 70 repetitions
OBSERVE  C13, 125.6889872 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```

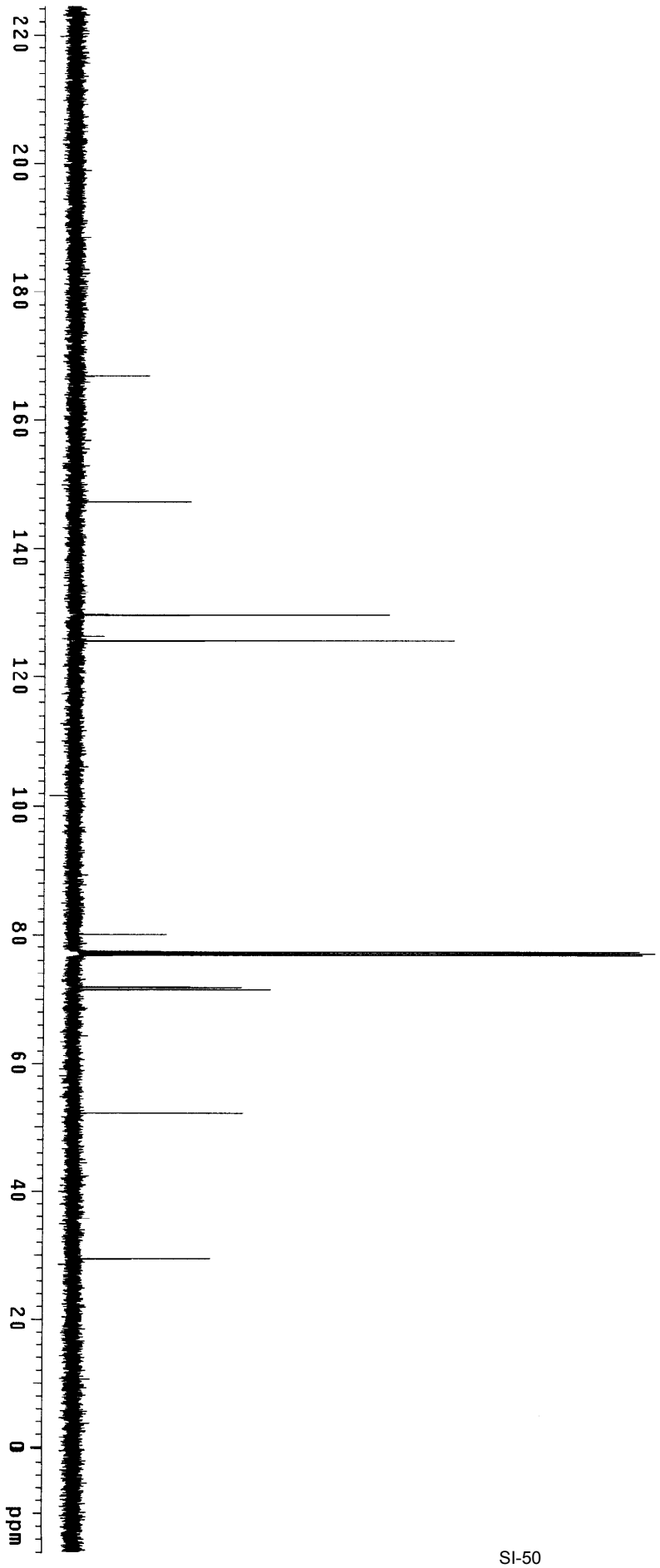


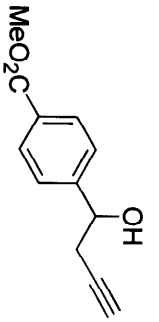
3k



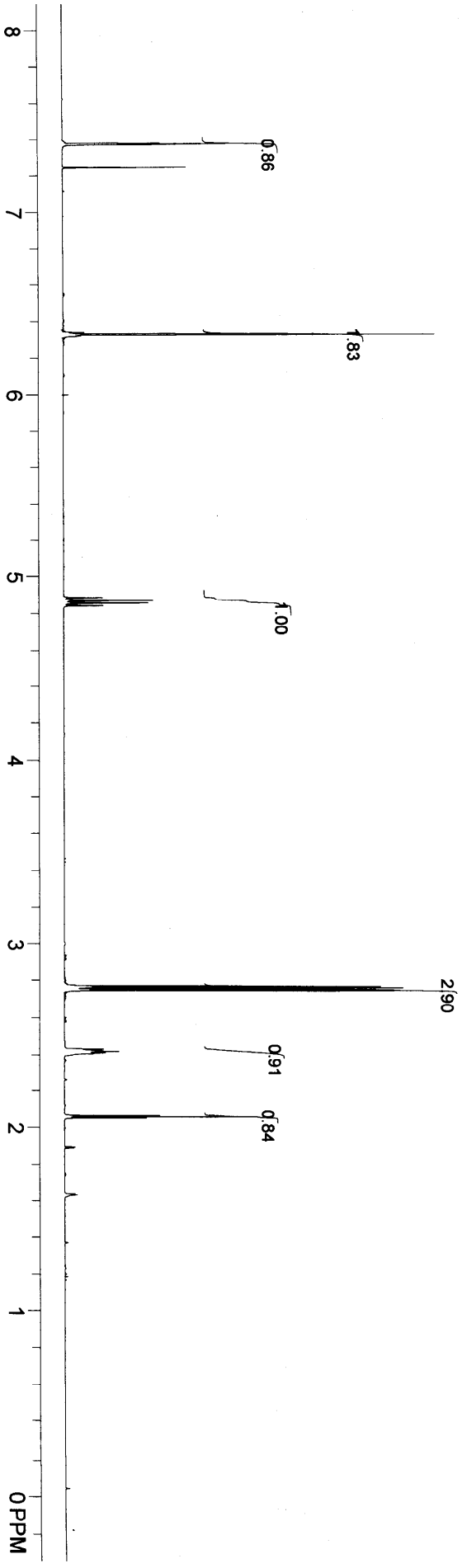


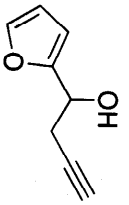
3l





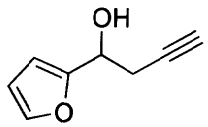
3l



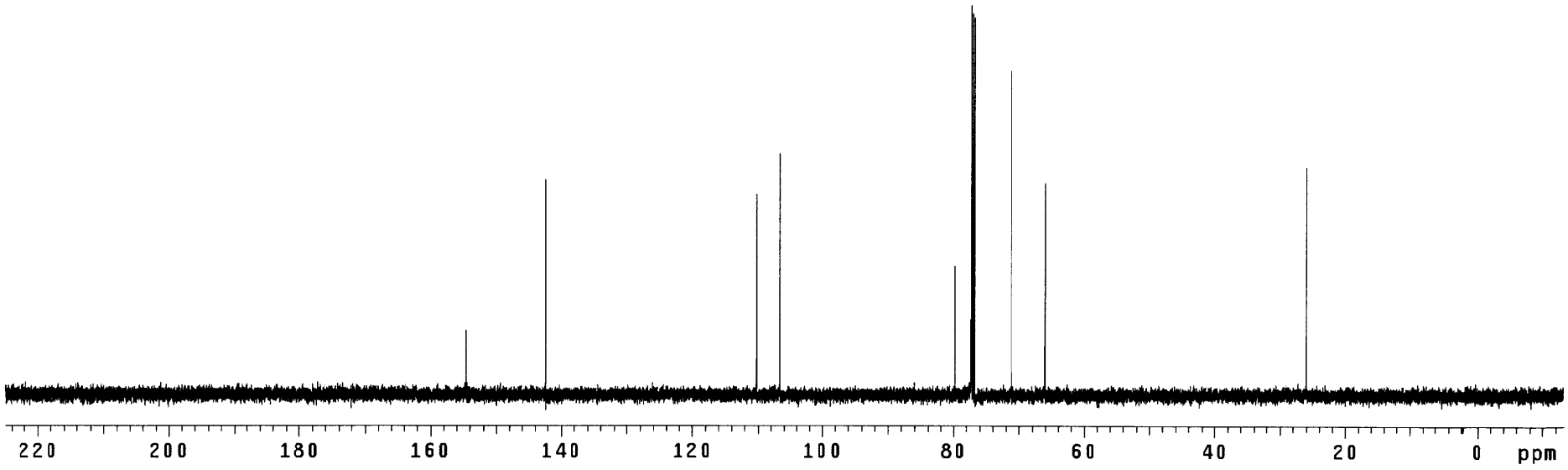


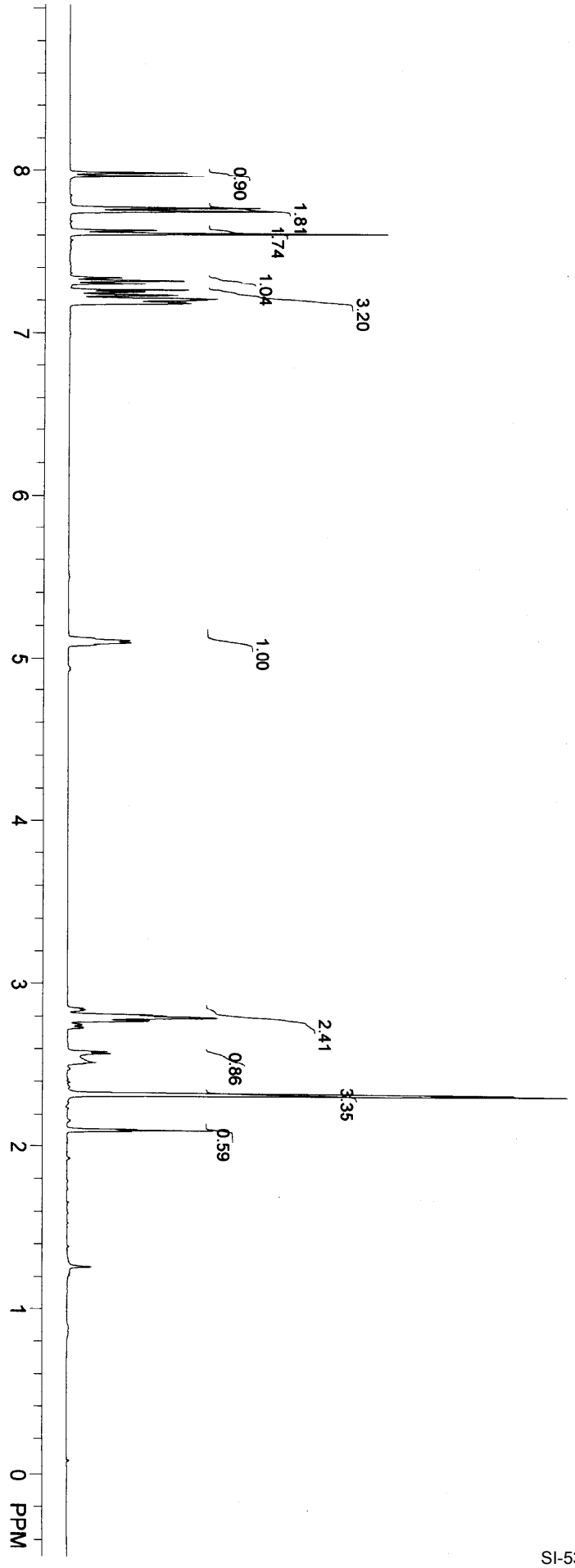
3m

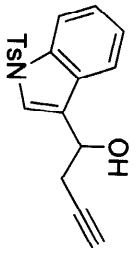
```
ylw-3-166-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 54 repetitions
OBSERVE  C13, 125.6889860 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```



3m







3n

ylw-3-180-cc

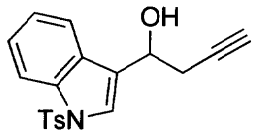
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

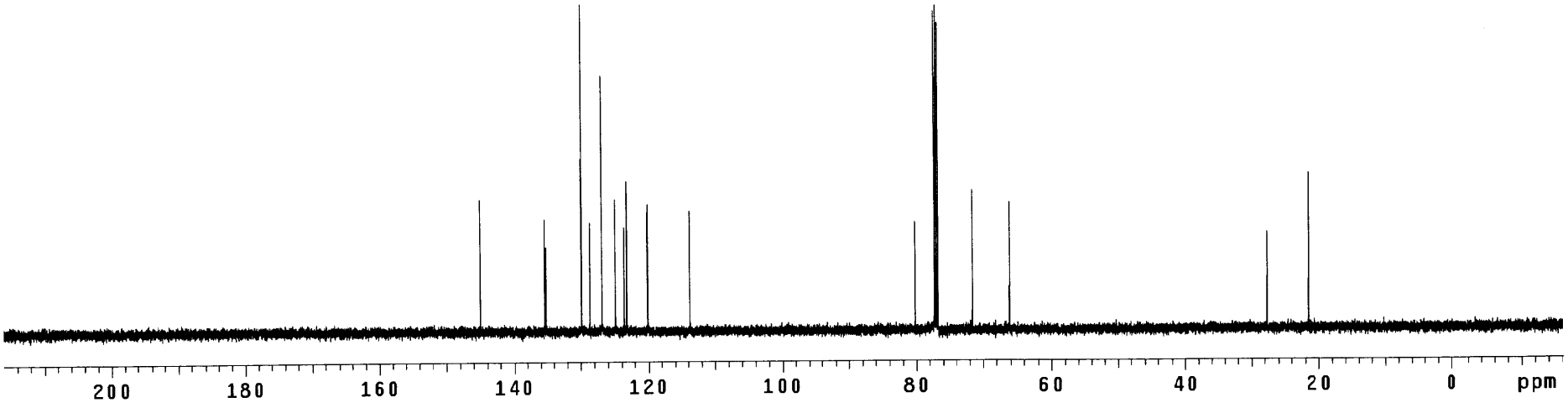
Pulse Sequence: s2pul
Solvent: cdcl3

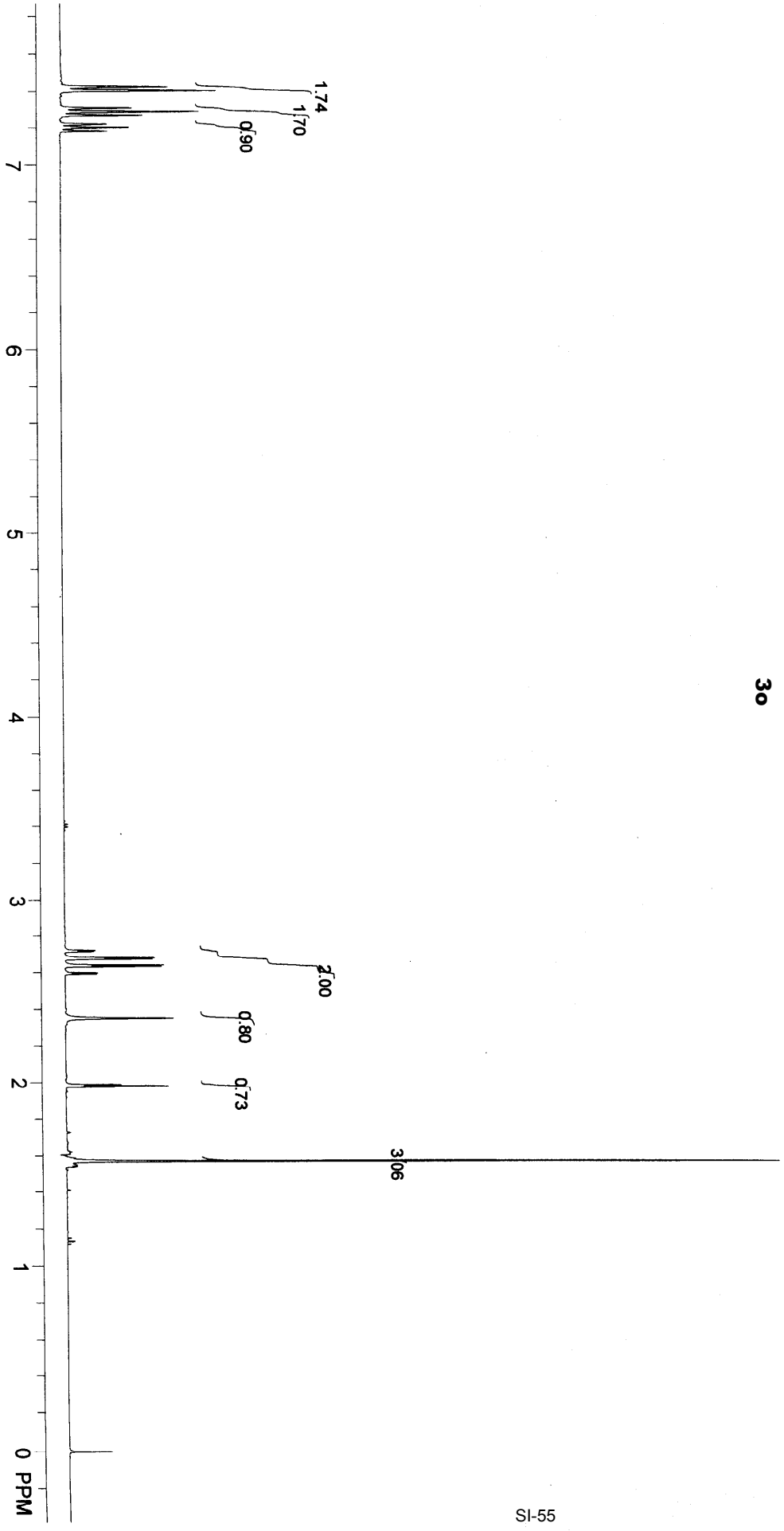
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
100 repetitions
OBSERVE C13, 125.6889902 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min

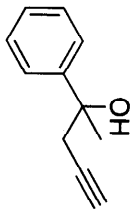


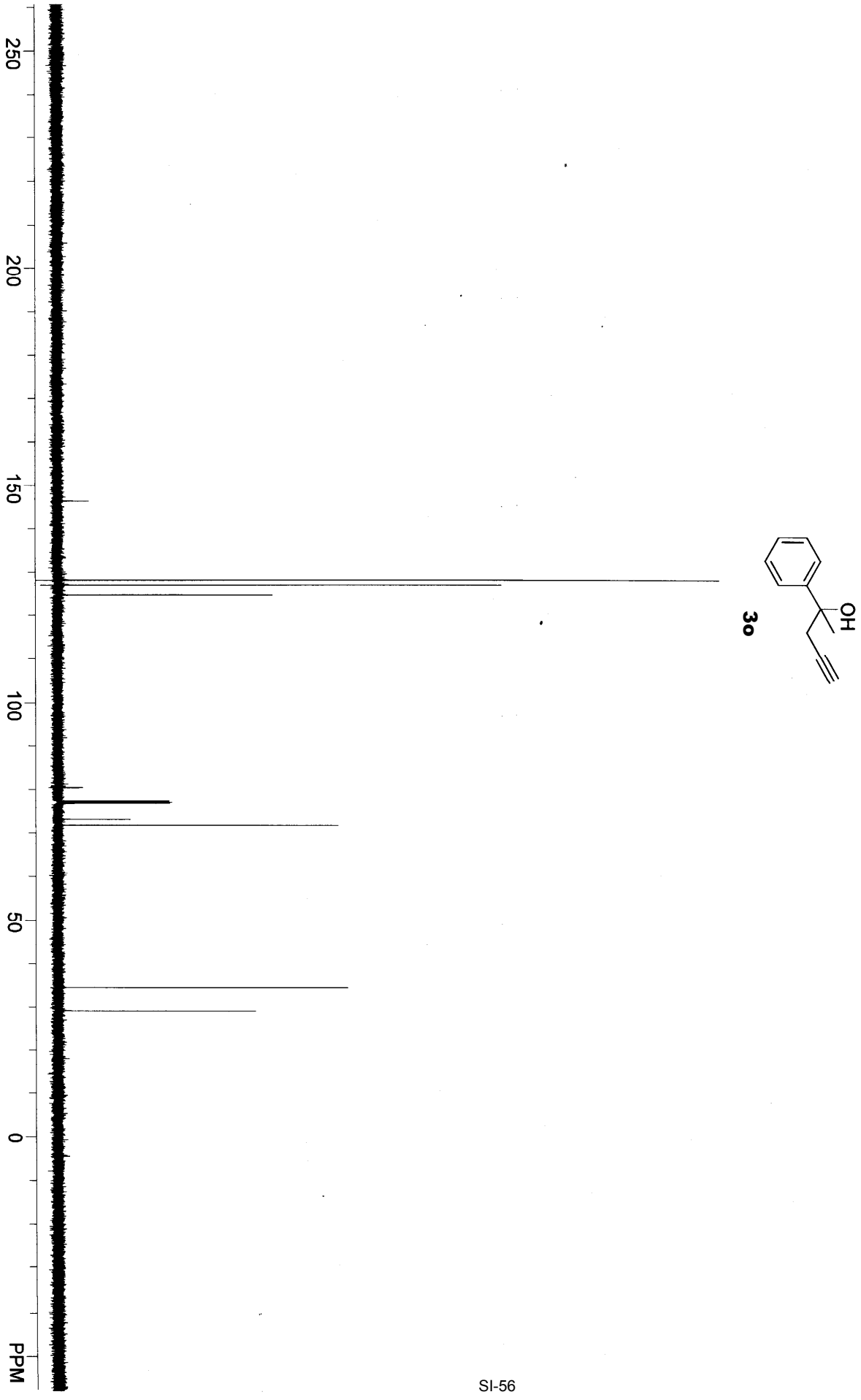
3n

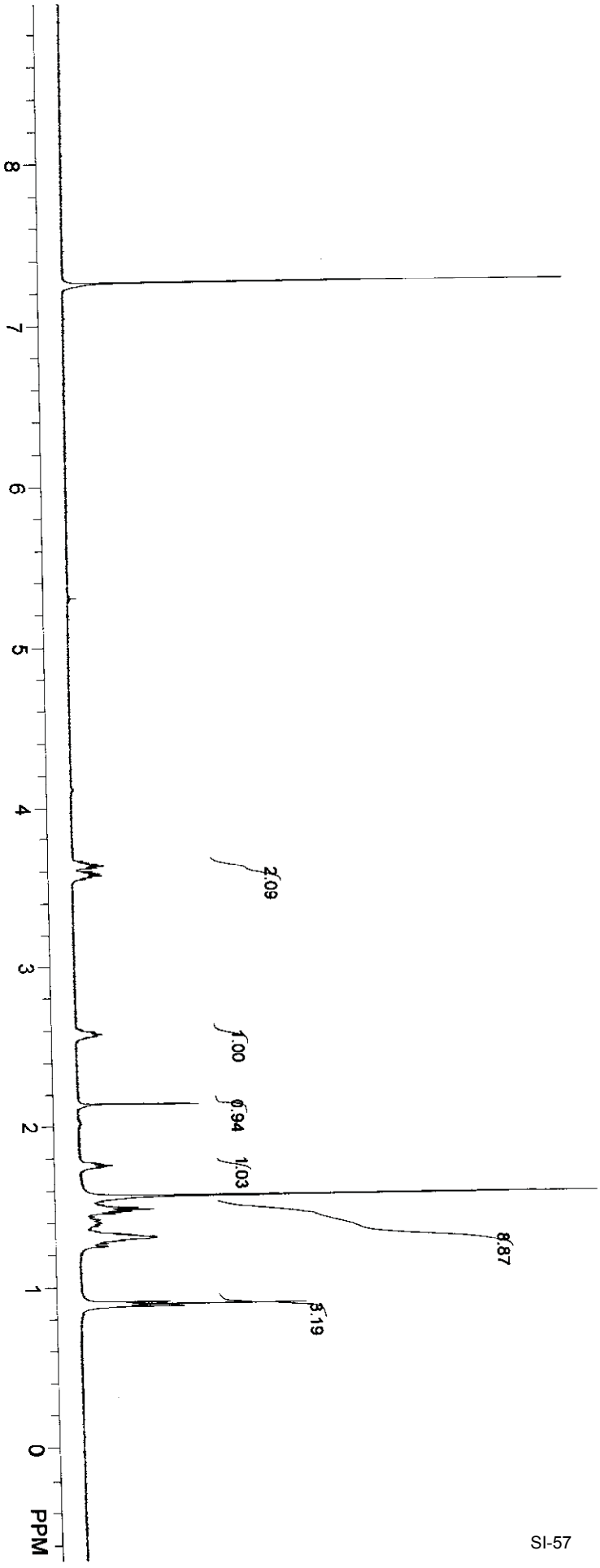




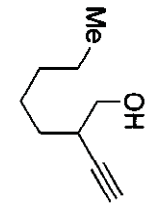
3o

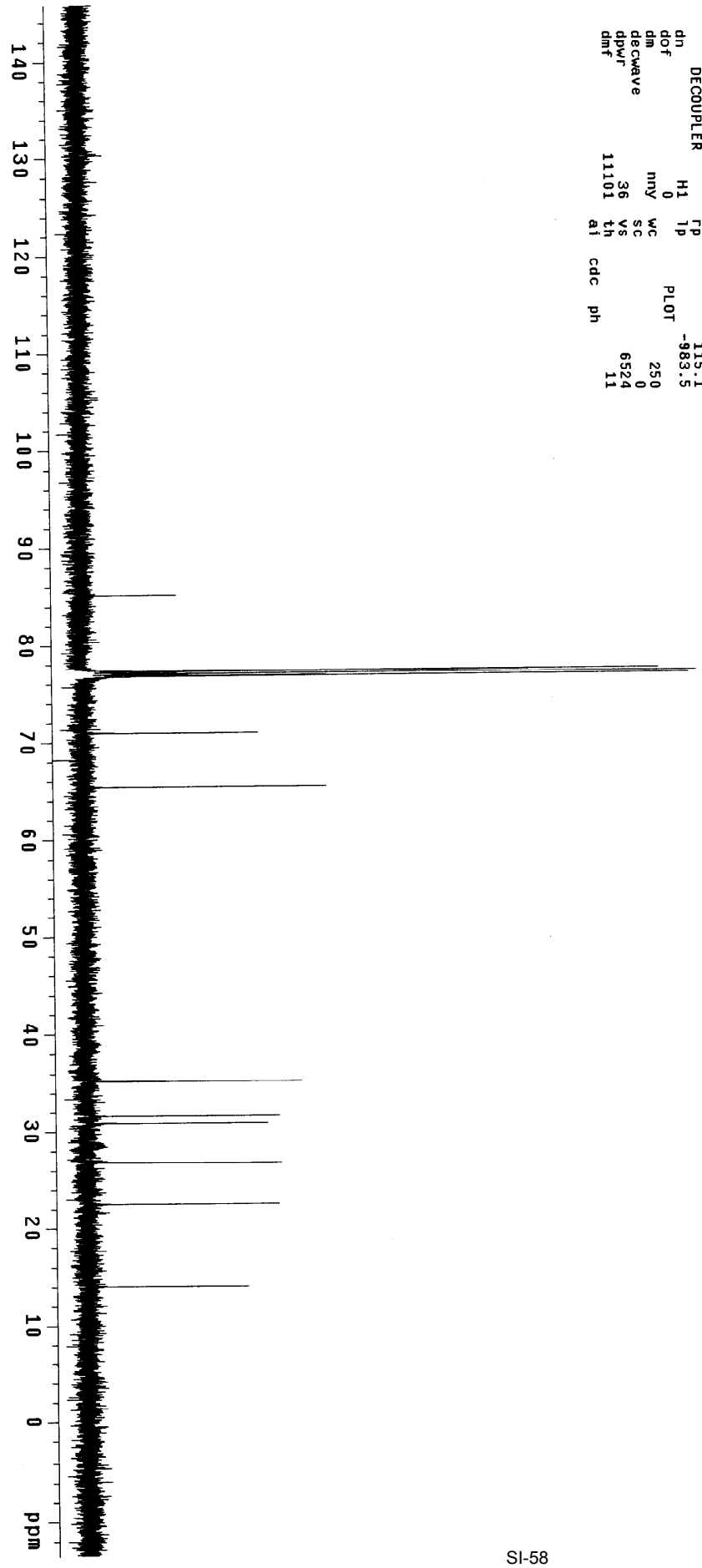


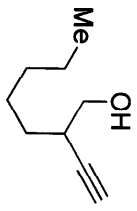

250
200
150
100
50
0
PPM
OH
3o



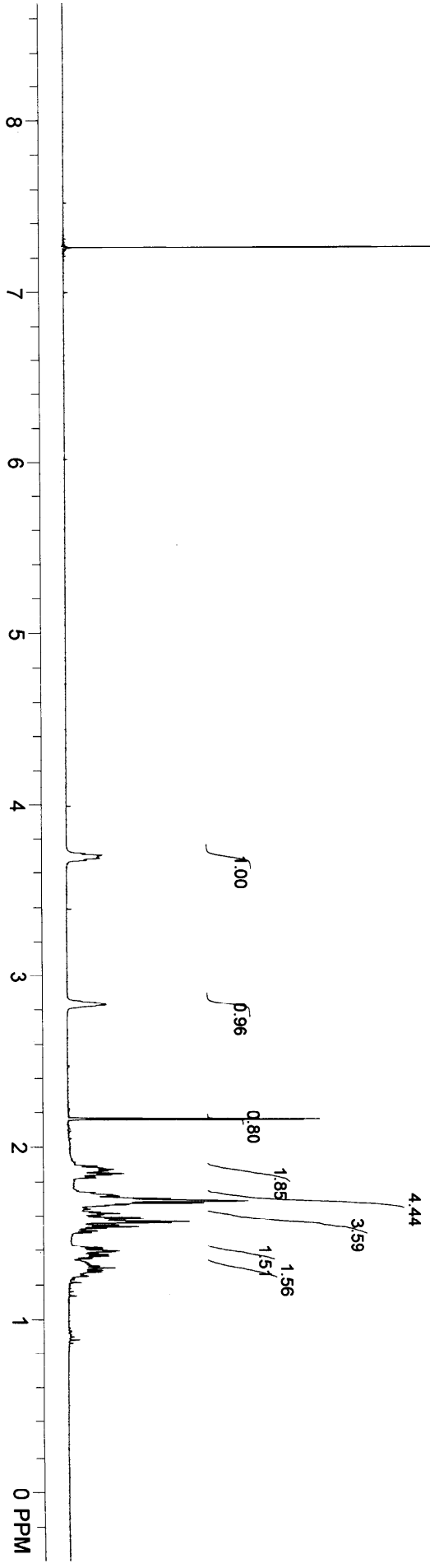
3p

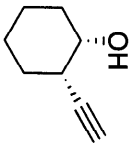



STANDARD CARBON PARAMETERS
exp1 CARBON
SAMPLE
date Jan 2 2010
solvent cdcl3
file /home/Zhang/g~
uzhang/C13-2009122~
3-5.fid
ACQUISITION
sw 40000.0
at 1.300
np 104036
fb 22000
bs 4
d1 3.000
nt 12000
ct 92
TRANSMITTER
tn C13
sfrq 125.702
tof 865.4
tpwr 56
pw 6.000
DECOUPLER
dn H1
dof 0
dm nny
decwave
dpwr 36
dmf 11101
SPECIAL
temp 22.0
gain s4
spin 20
hst 0.008
pw90 9.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
lsfid 2
fn
DISPLAY
not used
sp -1705.6
wp 20025.6
rfl 16903.1
rfp 9678.1
rp 115.1
lp -983.5
PLOT
wc 250
sc 0
vs 6524
th 11
ai cdc ph
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


Me
OH

3p





3q

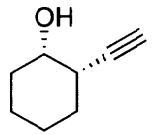
ylw-3-174-c

Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

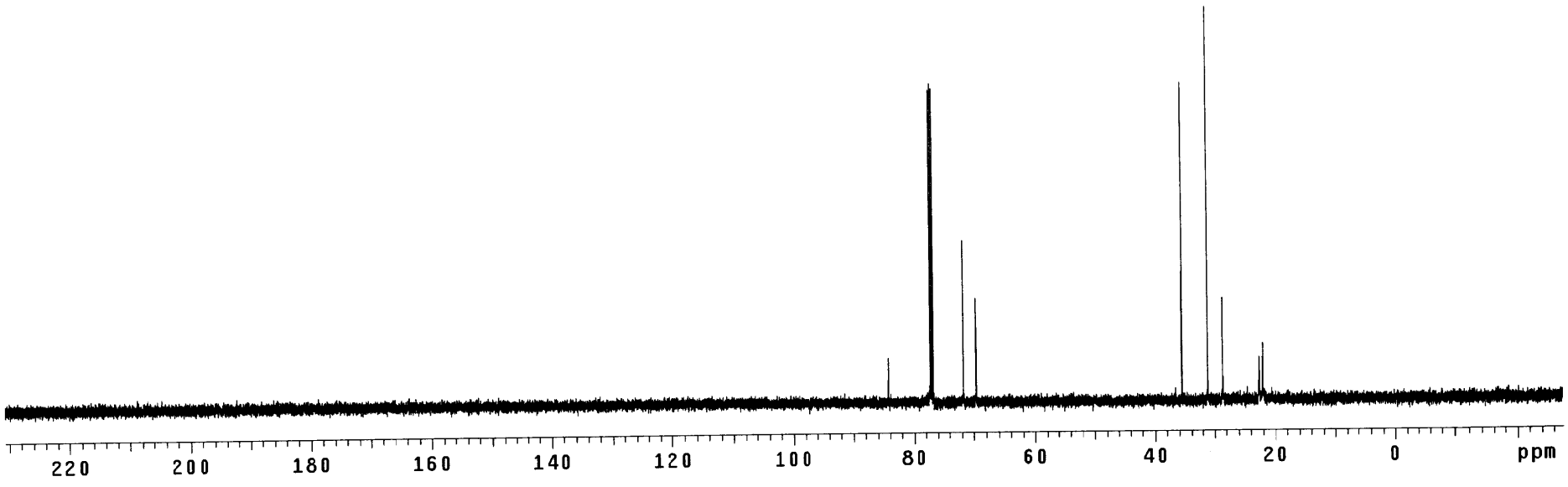
File: CARBON

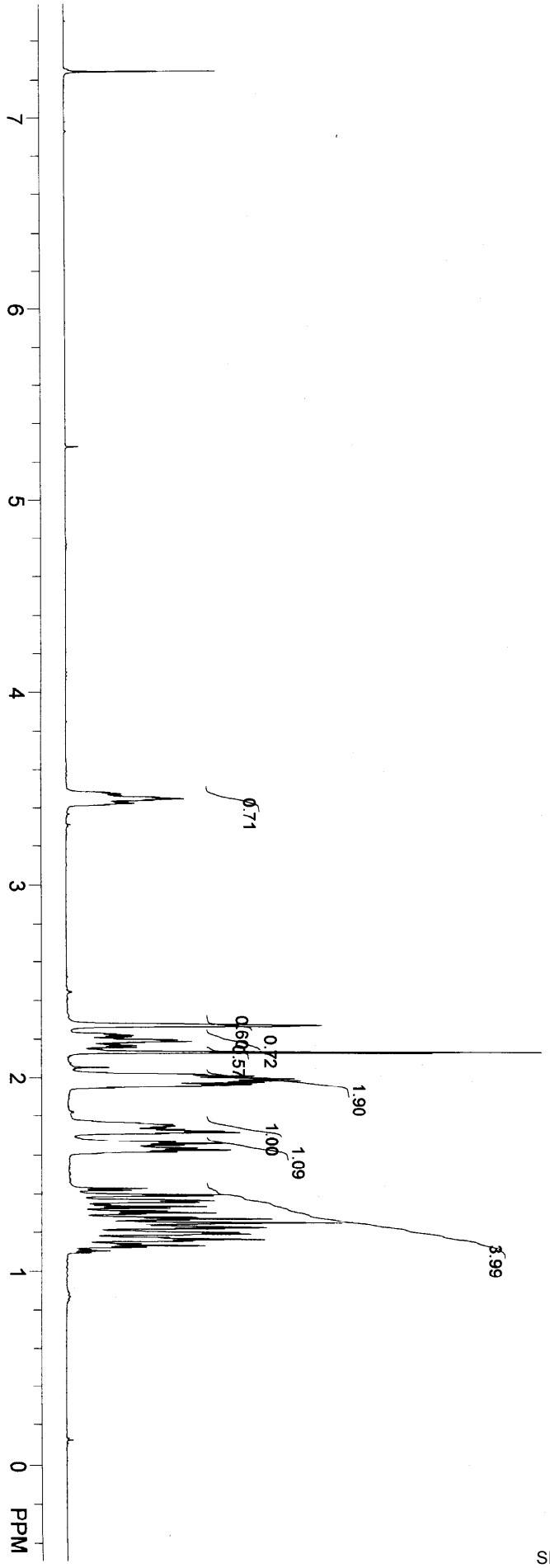
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye

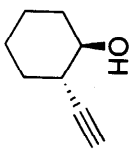
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
106 repetitions
OBSERVE C13, 125.6889872 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min



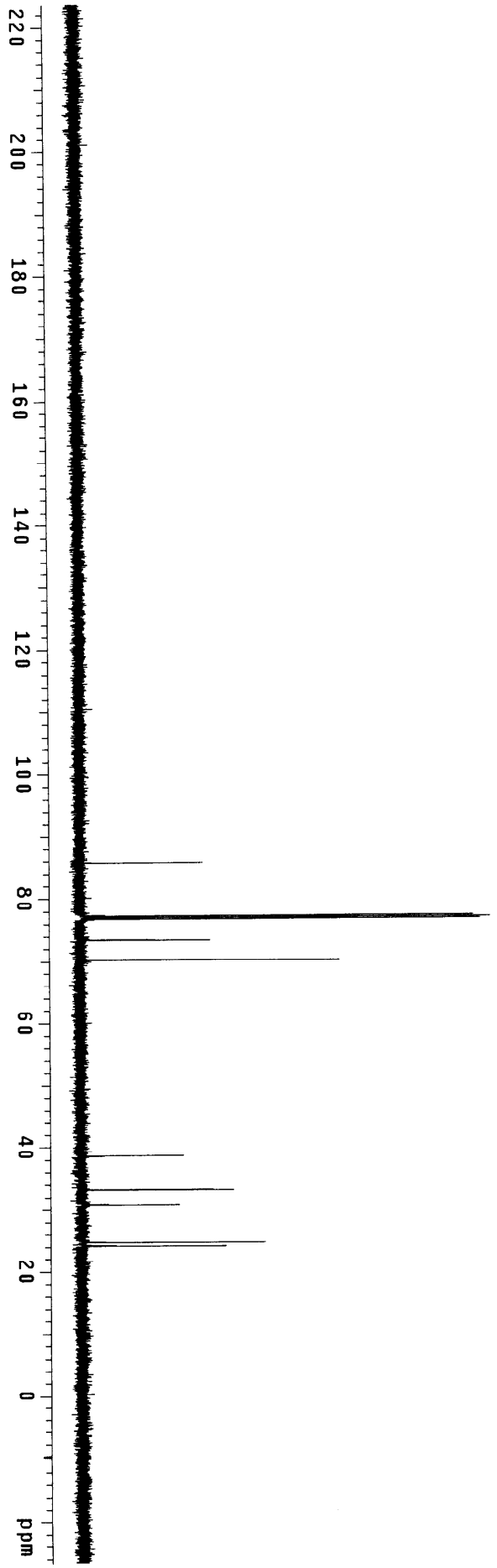
3q

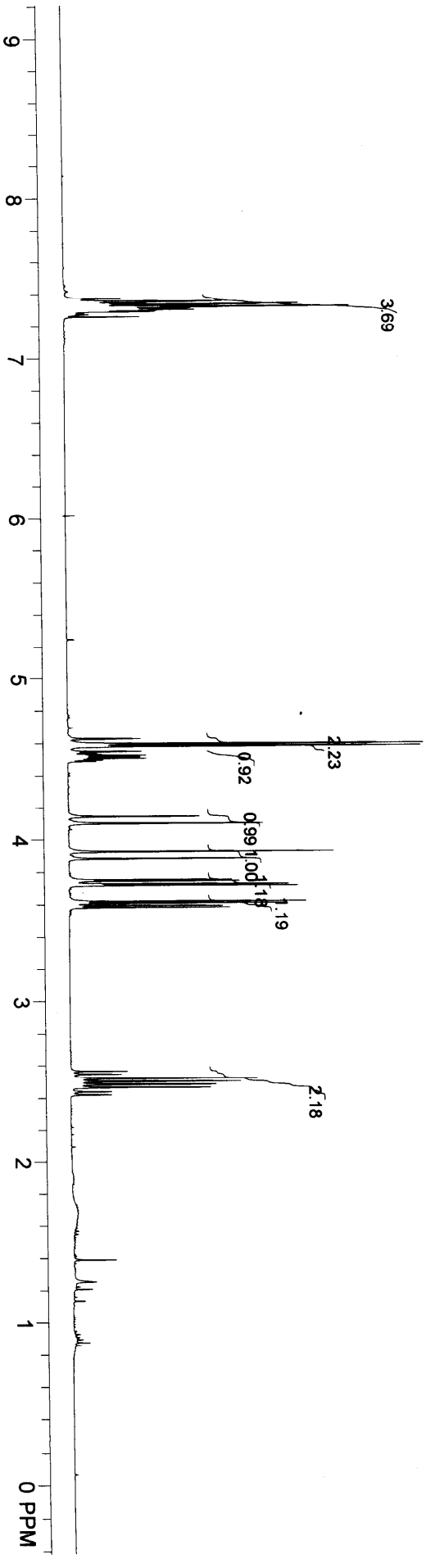


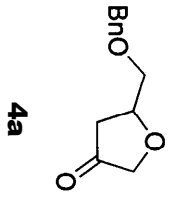




3r

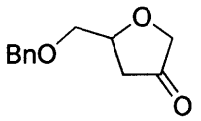

ylw-3-162-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
102 repetitions
OBSERVE C13, 125.6889854 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquistion
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
OH
3r
220
200
180
160
140
120
100
80
60
40
20
0
ppm



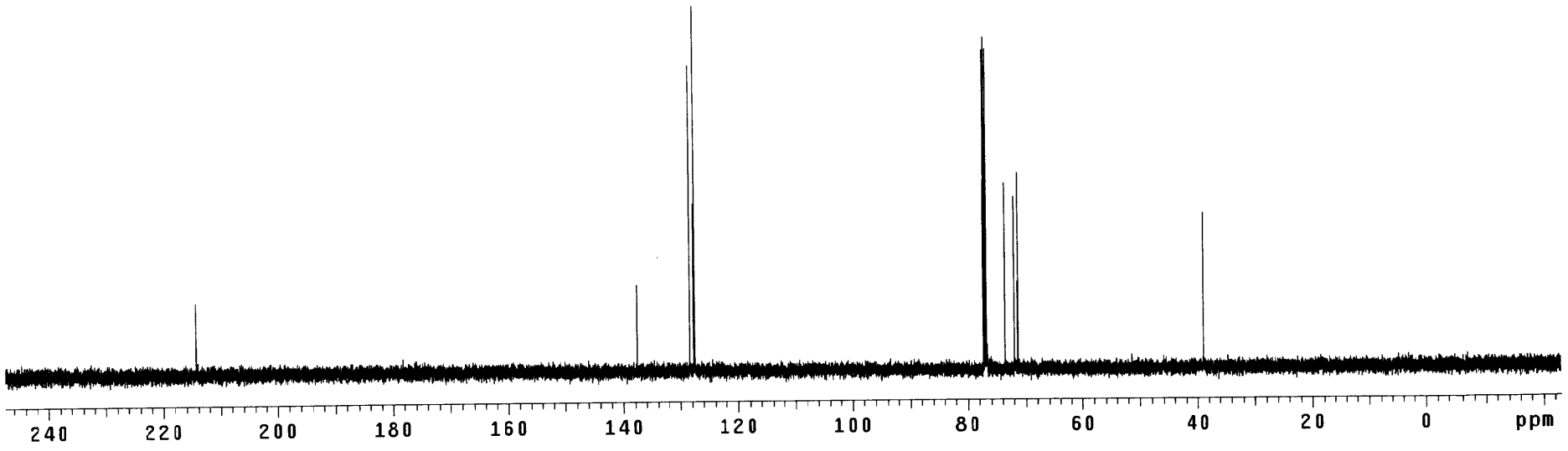


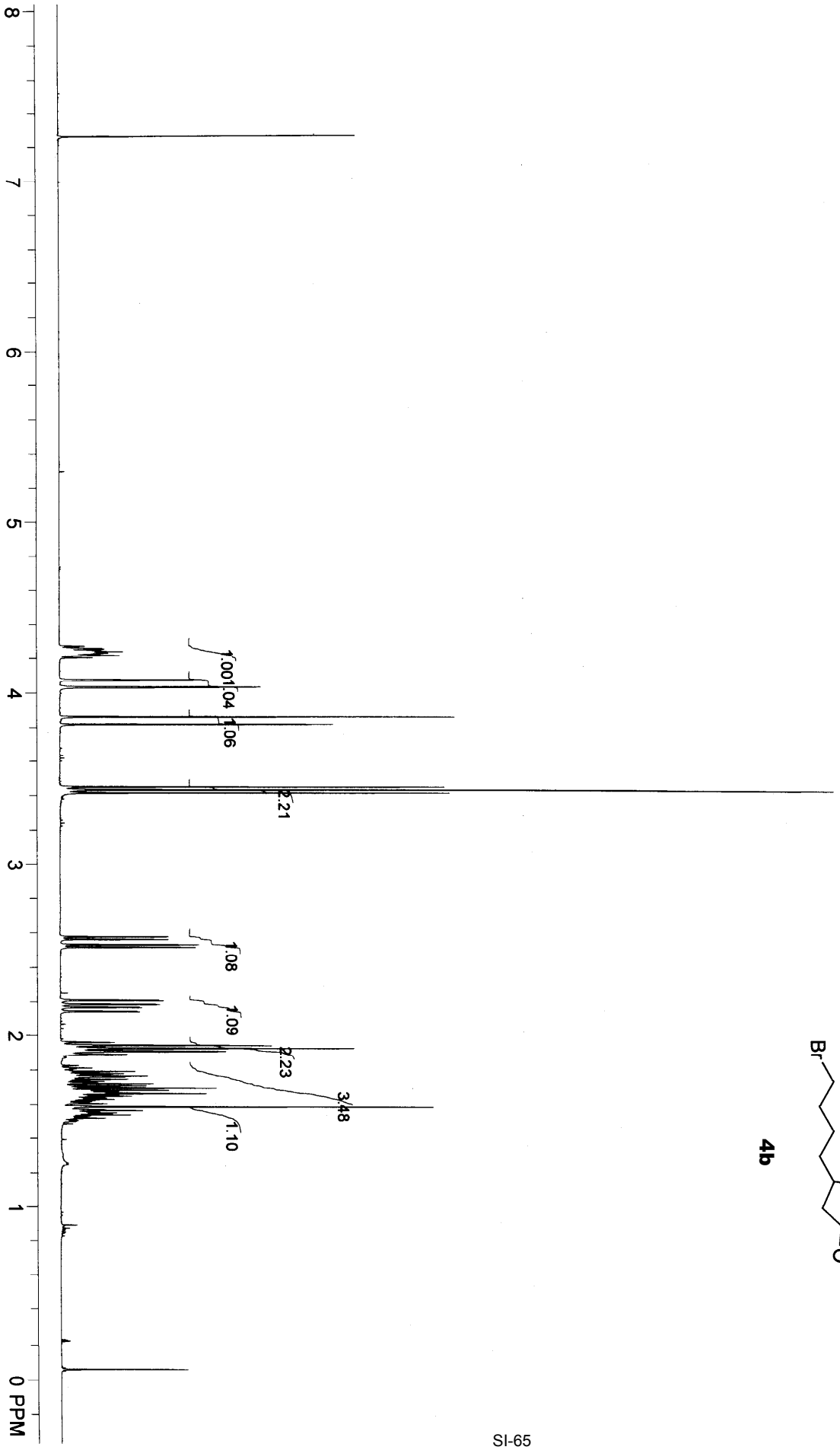
4a

```
ylw-3-171-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 62 repetitions
OBSERVE  C13, 125.6889872 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```



4a



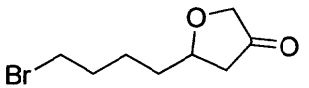


4b

STANDARD CARBON PARAMETERS

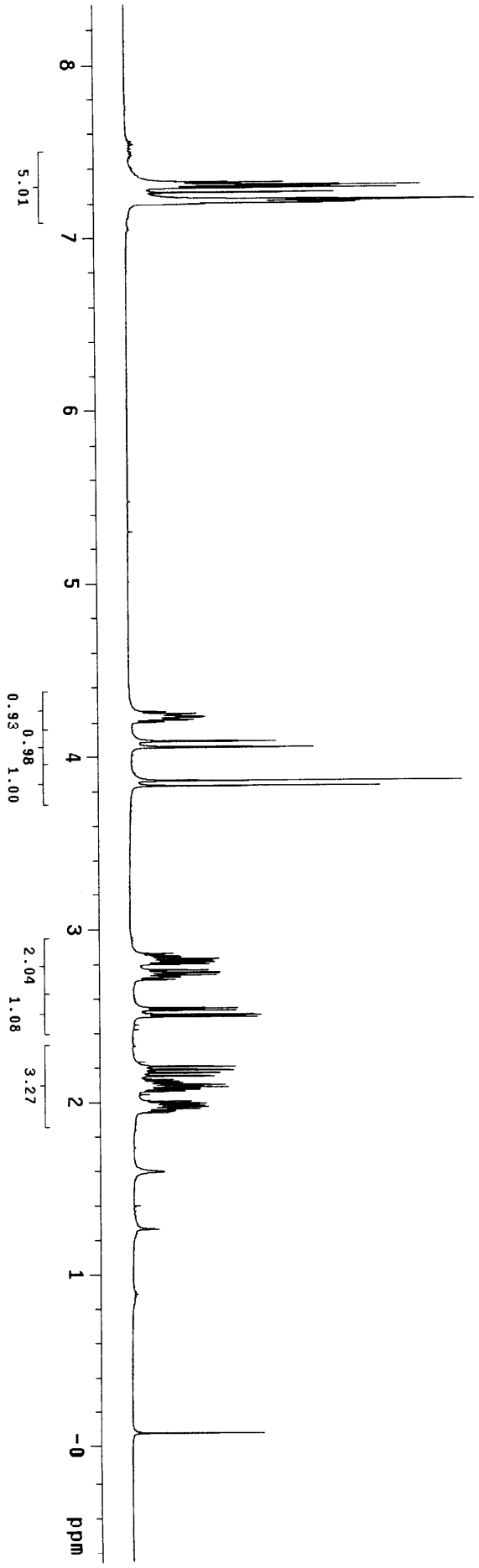
exp1 CARBON

SAMPLE		SPECIAL	
date	Jan 1 2010	temp	22.0
solvent	cdcl3	gain	54
file	/home/Zhang/g~	spin	20
uzhang/C13-2009122~		hst	0.008
	8-2.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	34995.6	FLAGS	
at	1.301	il	n
np	91024	in	n
fb	19000	dp	y
bs	2	hs	nn
d1	3.000	PROCESSING	
nt	12000	lb	0.50
ct	2162	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-4722.1
tof	865.4	wp	34995.1
tpwr	56	rfl	14400.7
pw	6.000	rfp	9678.1
DECOUPLER		rp	105.2
dn	H1	lp	-963.6
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	10368
dmf	11101	th	11
		ai cdc ph	

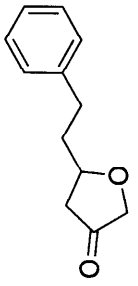


4b

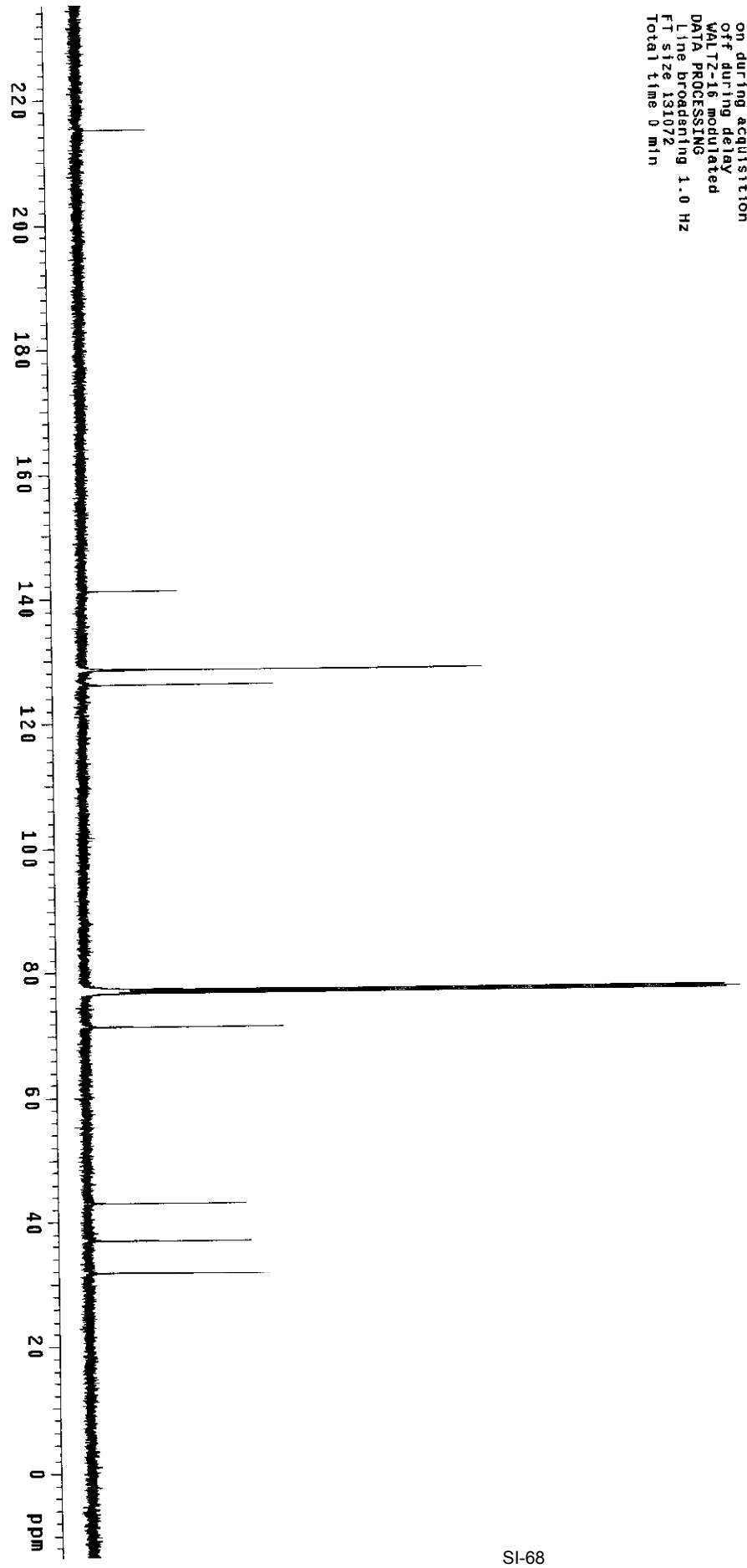
Ye, L.; Cui, L.; Zhang, G.; Zhang, L.

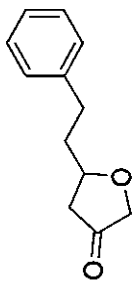



```
11cui-3-108-1-1H

Data Collected on:
     nmr500-inova500
Archive directory:
  /export/home/vnmr1/vnmrsys/data
Sample directory:

File: H1

Pulse Sequence: s2pul
Solvent: cdcl3

Operator: lcui

Relax. delay 2.000 sec
Pulse 58.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
12 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
```

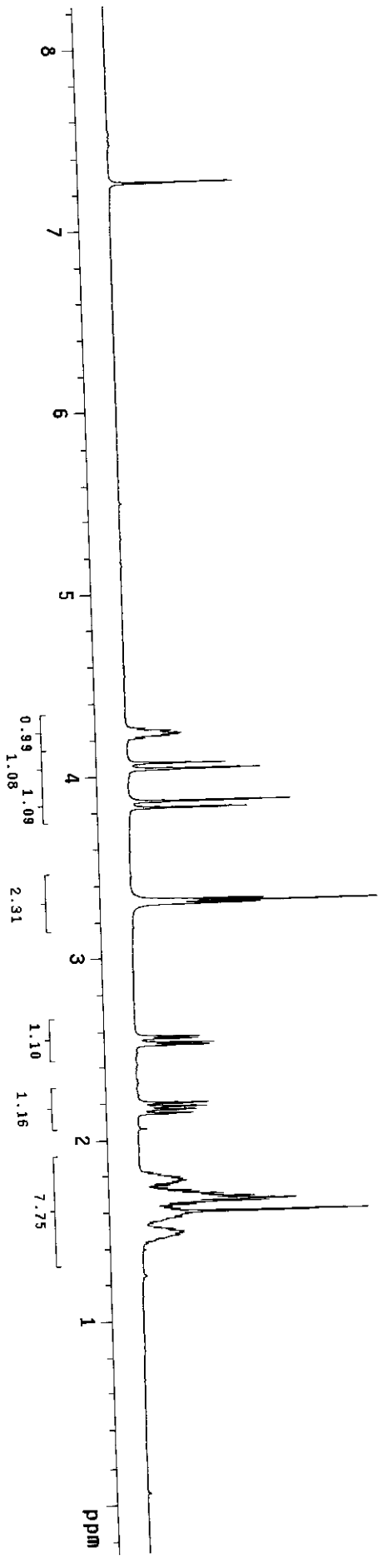


4c

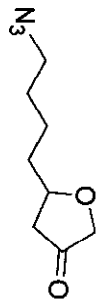

licui-3-108-1-13Ca
Data Collected on:
nmr500-1nova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lcui
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
width 40000.0 Hz
636 repetitions
OBSERVE C13, 125.6889835 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min
220
200
180
160
140
120
100
80
60
40
20
0
ppm



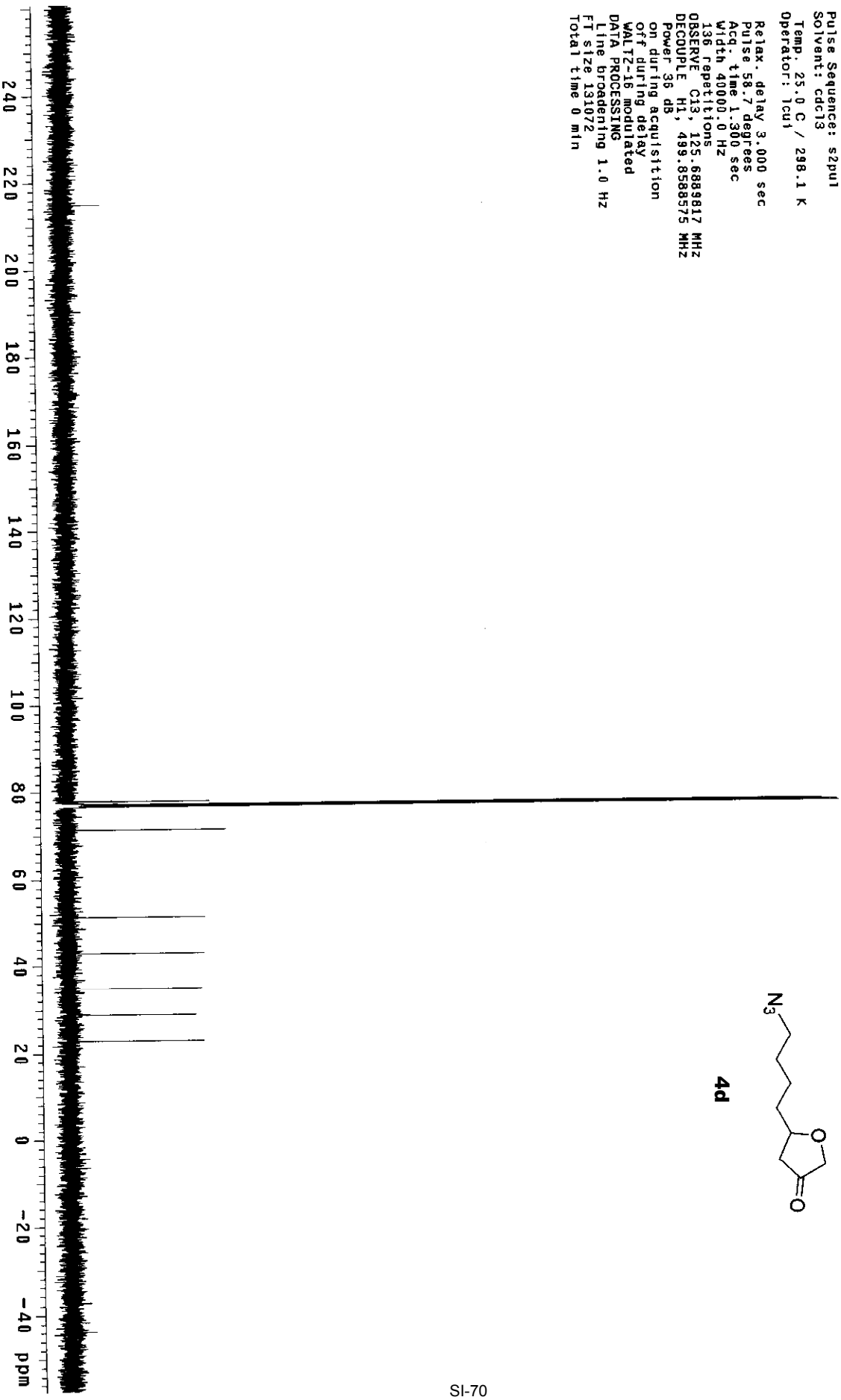
4c

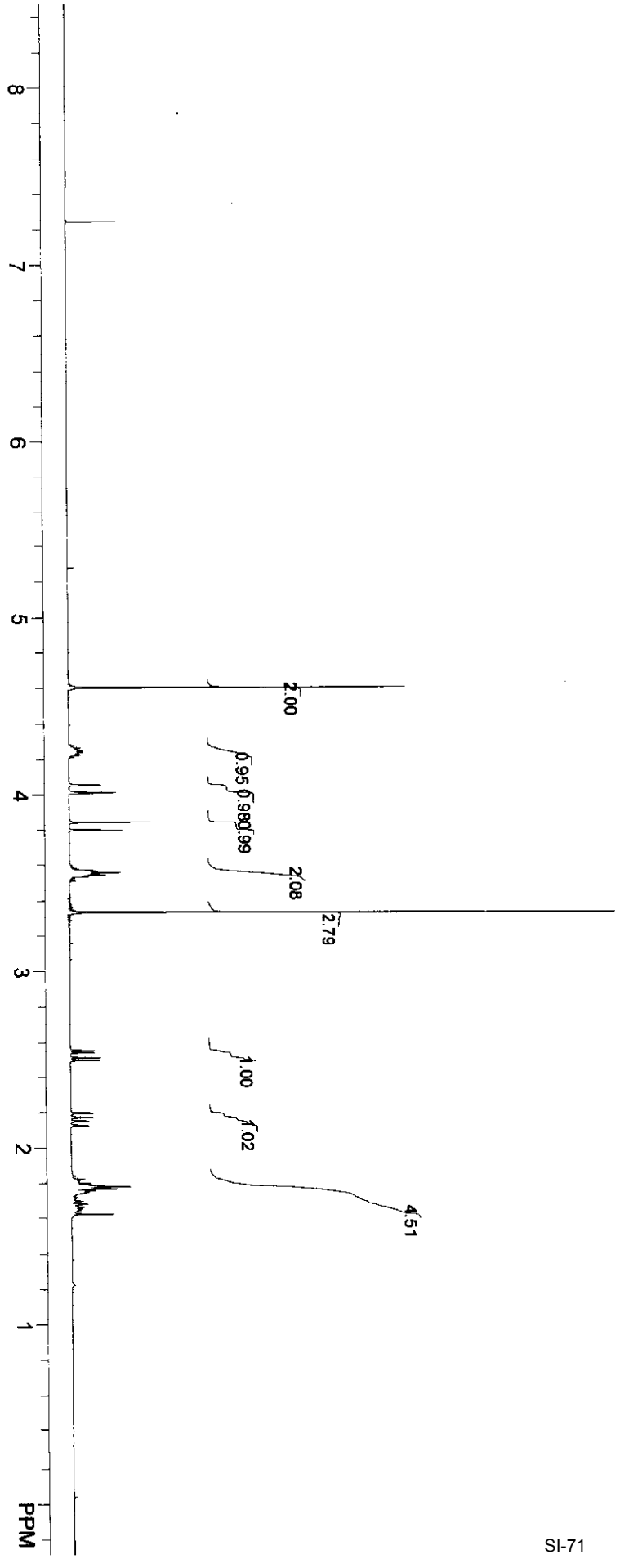



```
1icu1-3-120-2-1H

Data Collected on:
   nmr500-inova500
Archive directory:
   /export/home/vnmr1/vnmrsys/data
Sample directory:

File: H1

Pulse Sequence: s2pul
Solvent: cdcl3
Operator: 1cu1

Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
  16 repetitions
OBSERVE    H1, 499.8563614 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
```

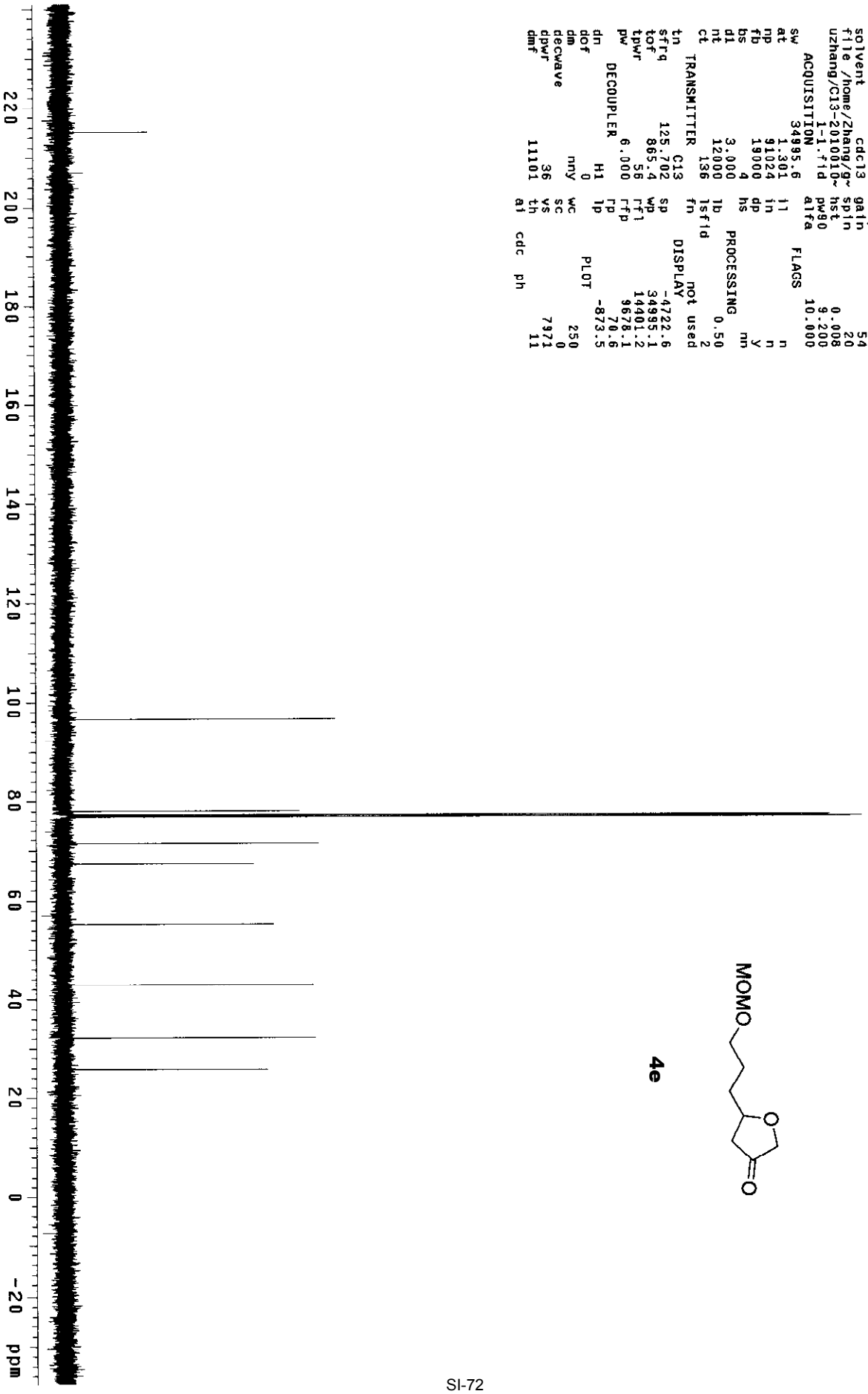


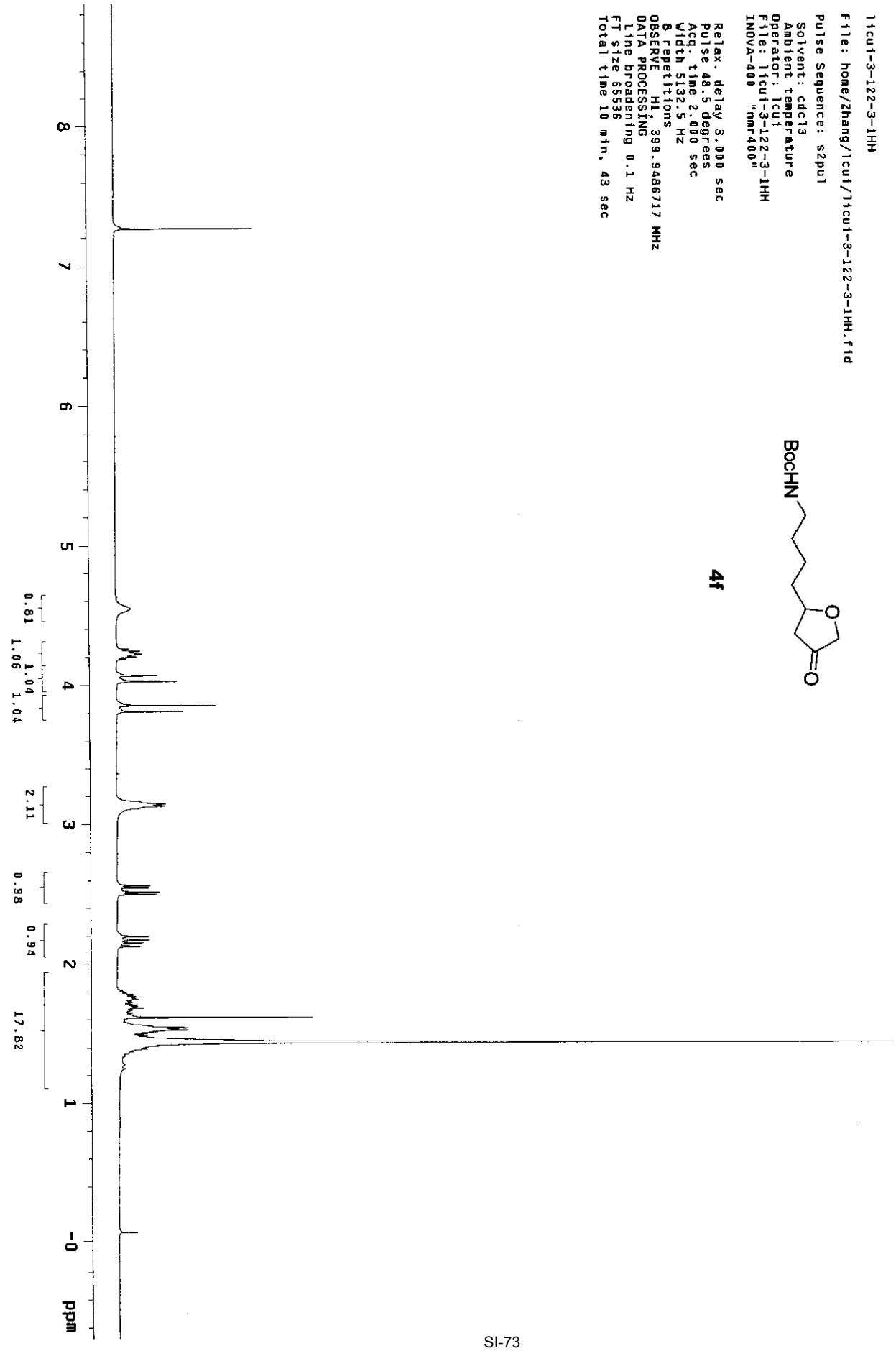
4d

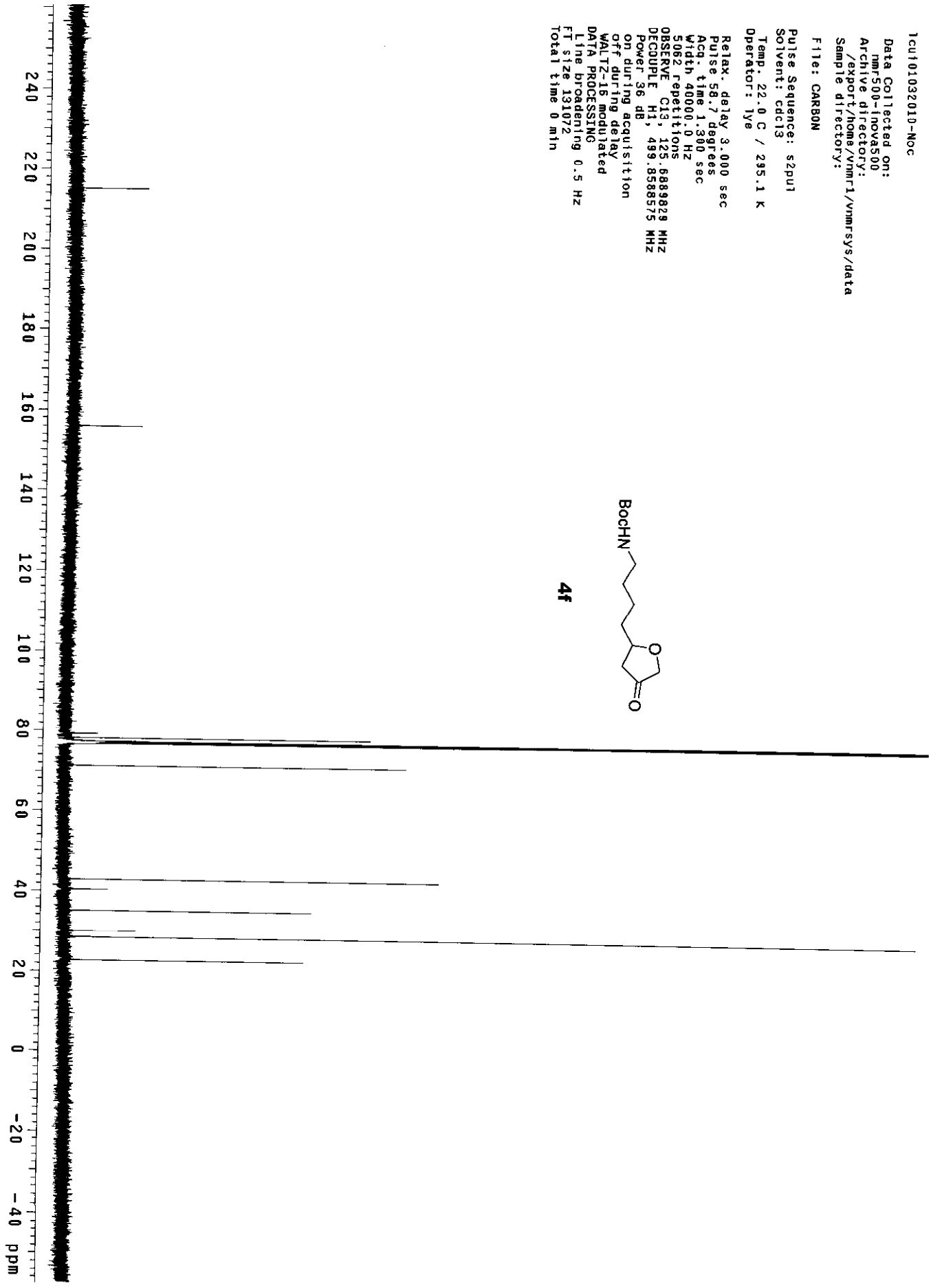

1icu1-3-120-2-13C
Data Collected on:
nmrS00-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: 1cu1
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
136 repetitions
OBSERVE C13, 125.6889817 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40 ppm
N3
O
O
4d

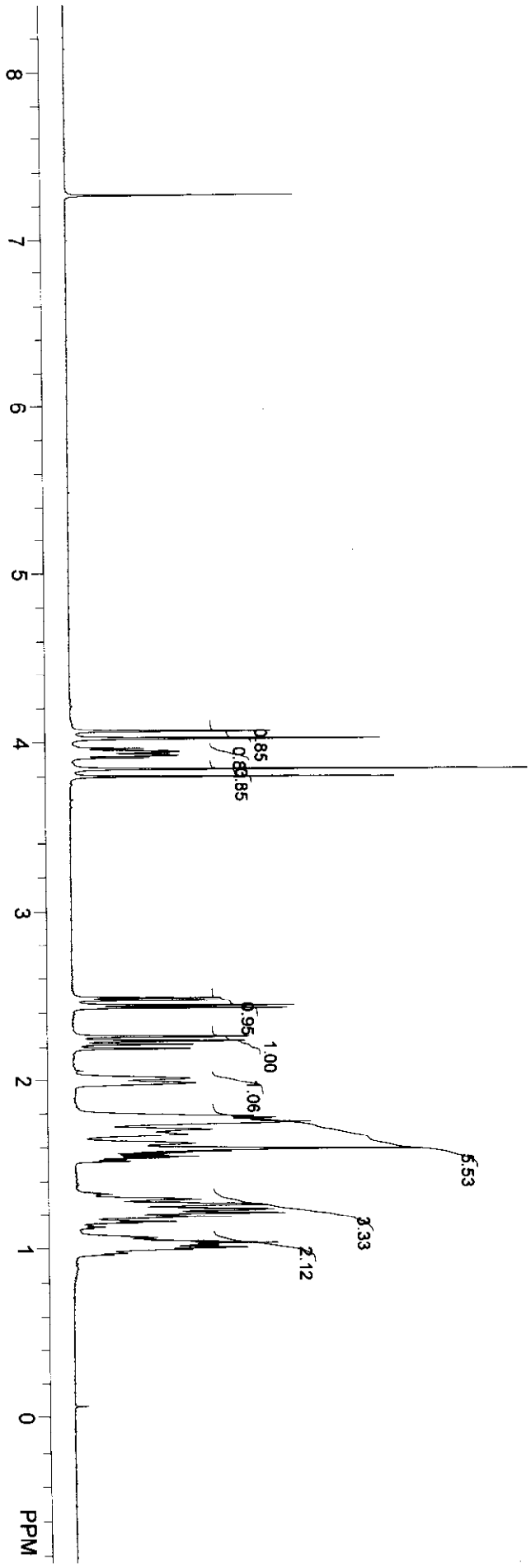


4e

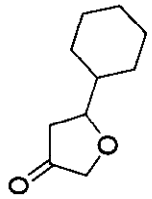

STANDARD CARBON PARAMETERS
exp1 CARBON
SAMPLE
date Jan 1 2010
solvent cdcl3
file /home/Zhang/gr
uzhang/C13-201001
1-1.fid
ACQUISITION
sw 34995.6
at 1.301
np 91024
fb 19000
bs 4
d1 3.000
nt 12000
ct 136
TRANSMITTER
tn C13
sfrq 125.702
tof 865.4
tpwr 56
pw 6.000
DECOUPLER
dn H1
dof 0
dm nny
decpwave
dpwr 36
dmf 11101
SPECIAL
temp 22.0
gain 54
spin 20
hst 0.008
pw90 9.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
lsfid 2
fn not used
DISPLAY
sp -4722.6
wp 34995.1
rfl 14401.2
rfp 9678.1
rp 70.6
lp -873.5
PLOT
wc 250
sc 0
vs 7871
th 11
ai cdc ph
MOMO
O
O
O
4e
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm


11cui-3-122-3-1HH
File: home/Zhang/lcui/11cui-3-122-3-1HH.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: lcui
File: 11cui-3-122-3-1HH
INOVA-400 "mr400"
Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
width 5132.5 Hz
8 repetitions
OBSERVE H1, 399.9486717 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 43 sec
BocHN
O
O
4f
8
7
6
5
4
3
2
1
-0
ppm
0.81
1.06
1.04
1.04
2.11
0.98
0.94
17.82


1cu101032010-Noc
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
5062 repetitions
OBSERVE C13, 125.6889829 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
BocHN
O
O
4f
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
ppm



4g



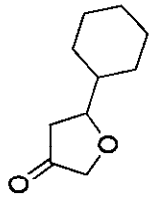
ylw-3-169-c

Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

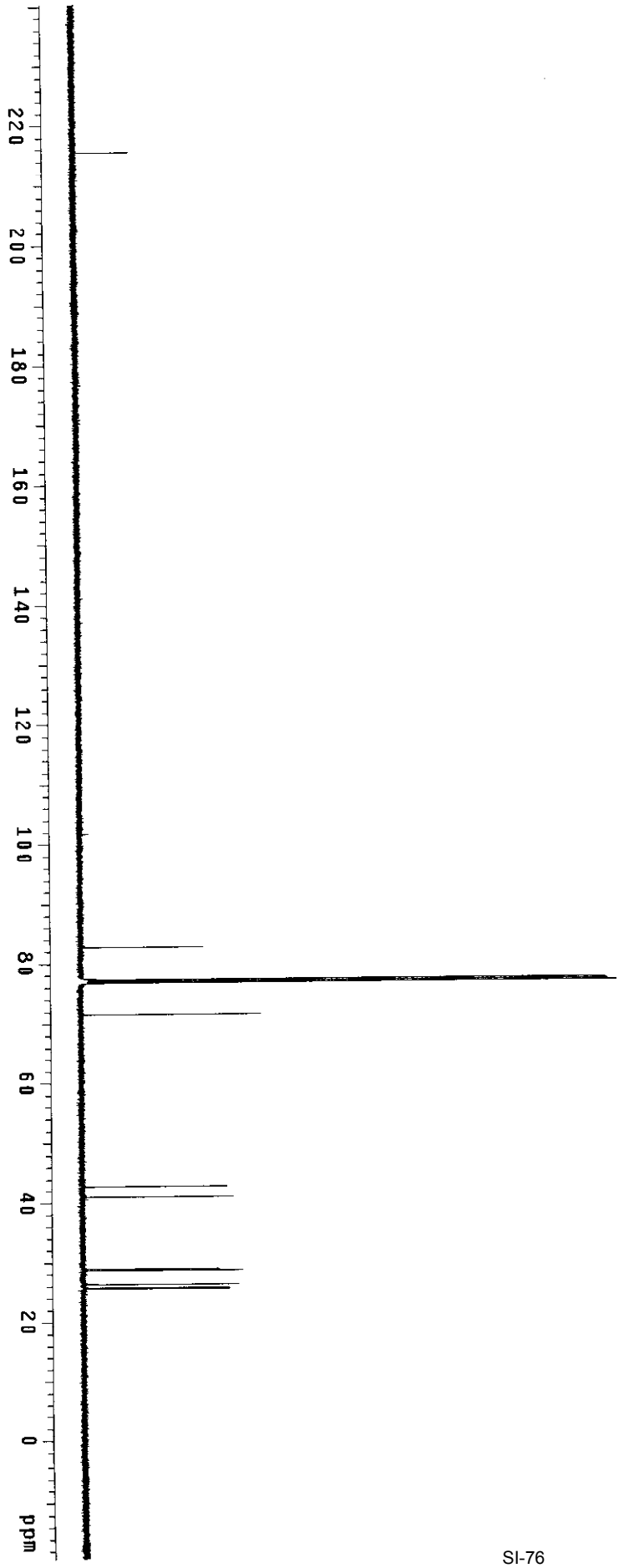
File: CARBON

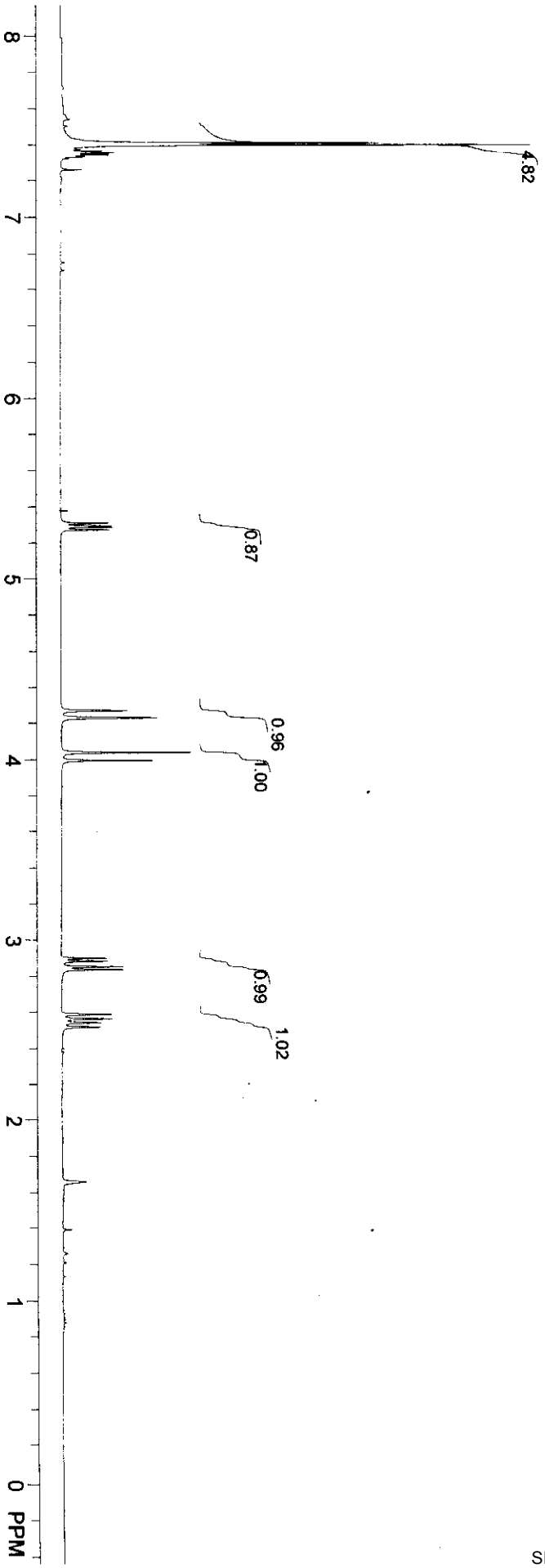
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1000 repetitions
OBSERVE C13, 125.6869829 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min

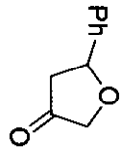


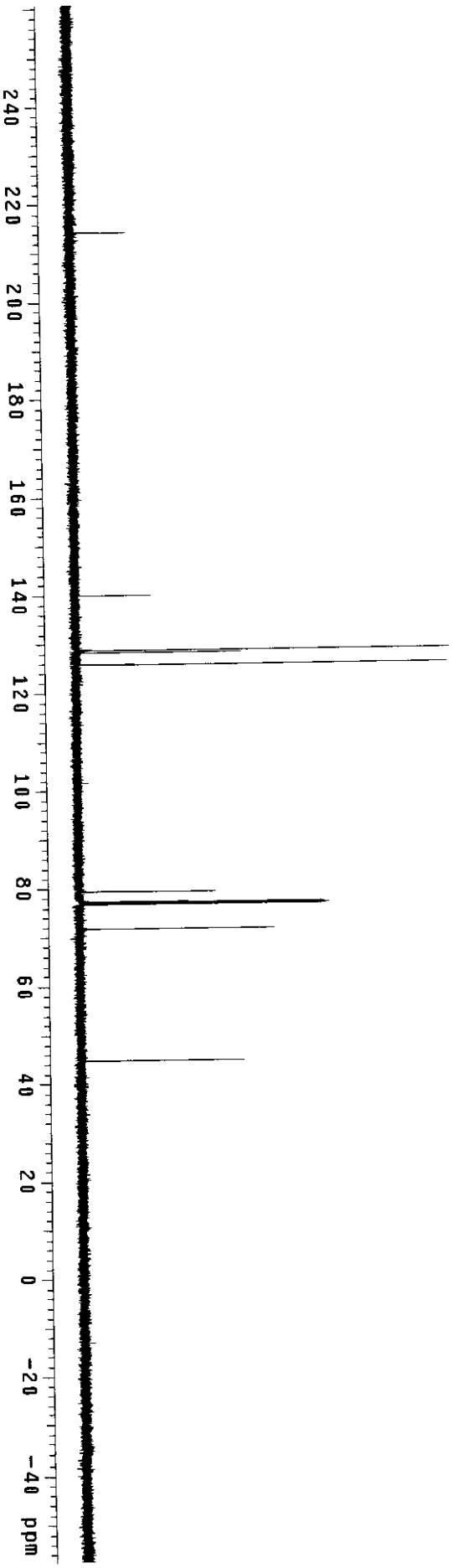
4g

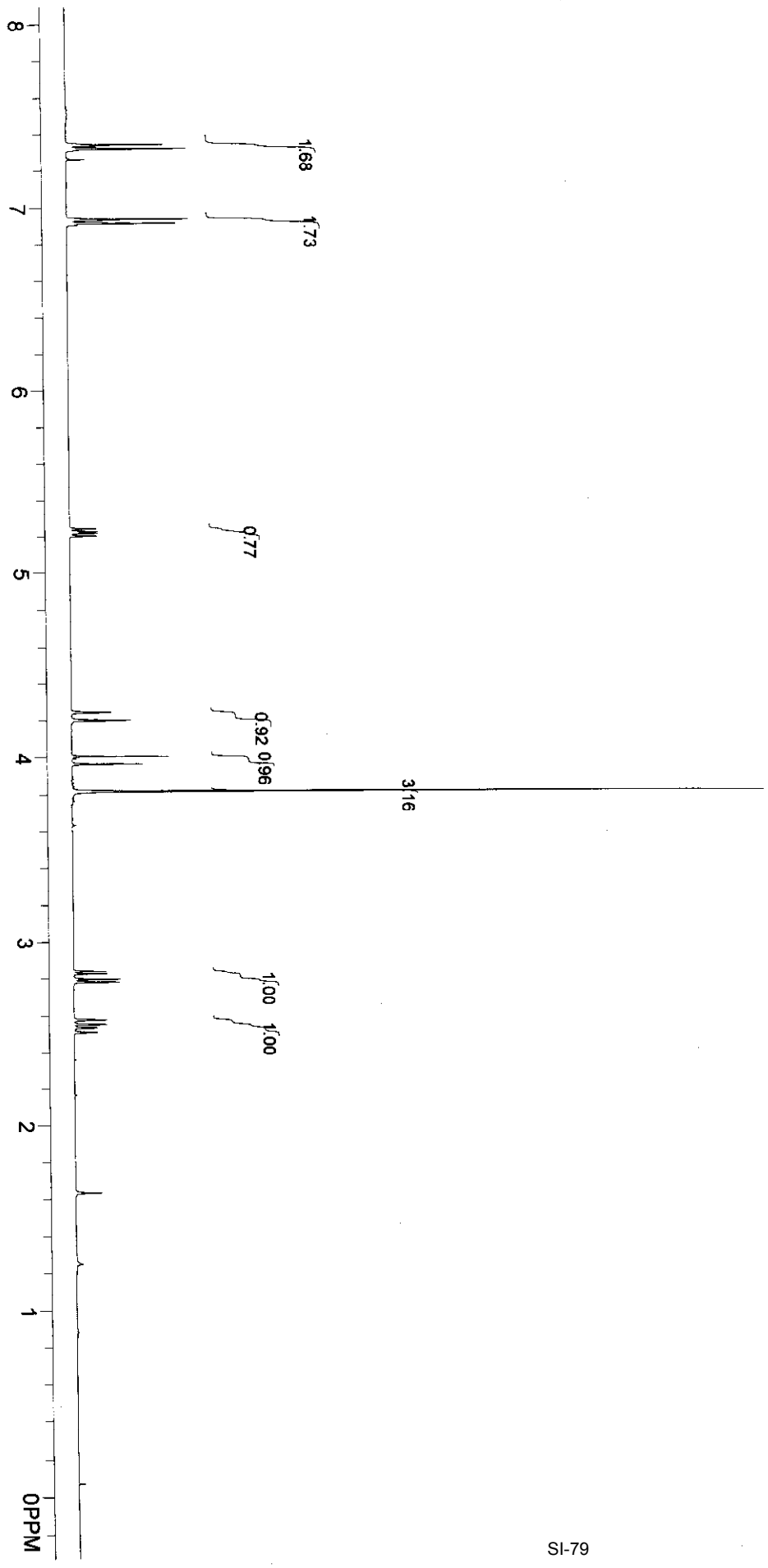


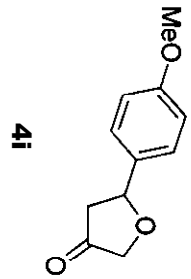


4h

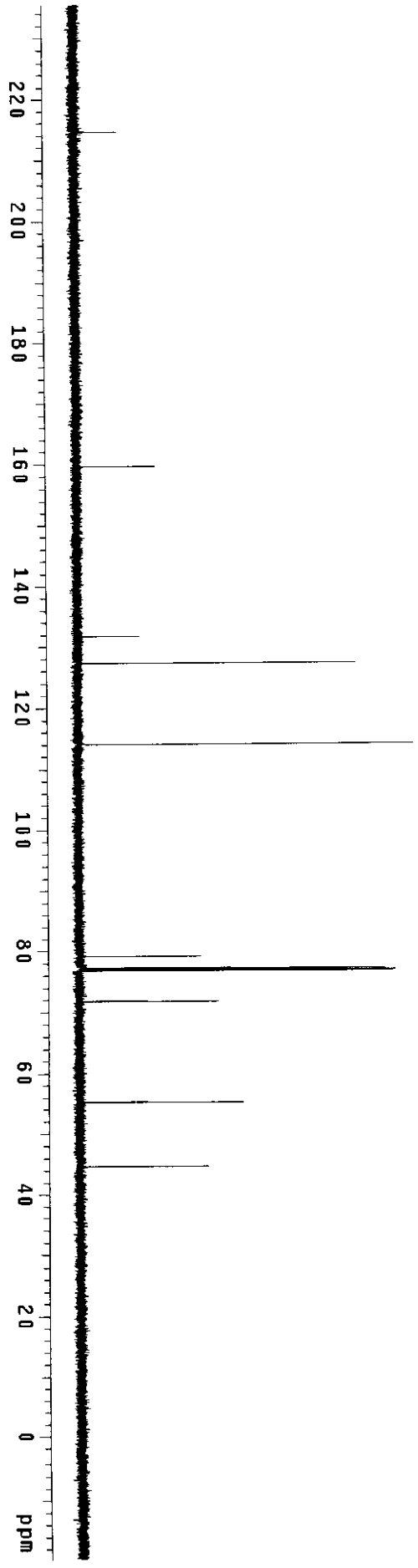



ylw-3-119-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
62 repetitions
OBSERVE C13, 125.6889884 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
Ph
O
O
4h
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
ppm

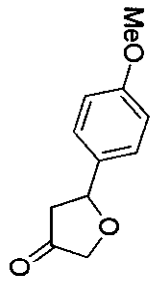




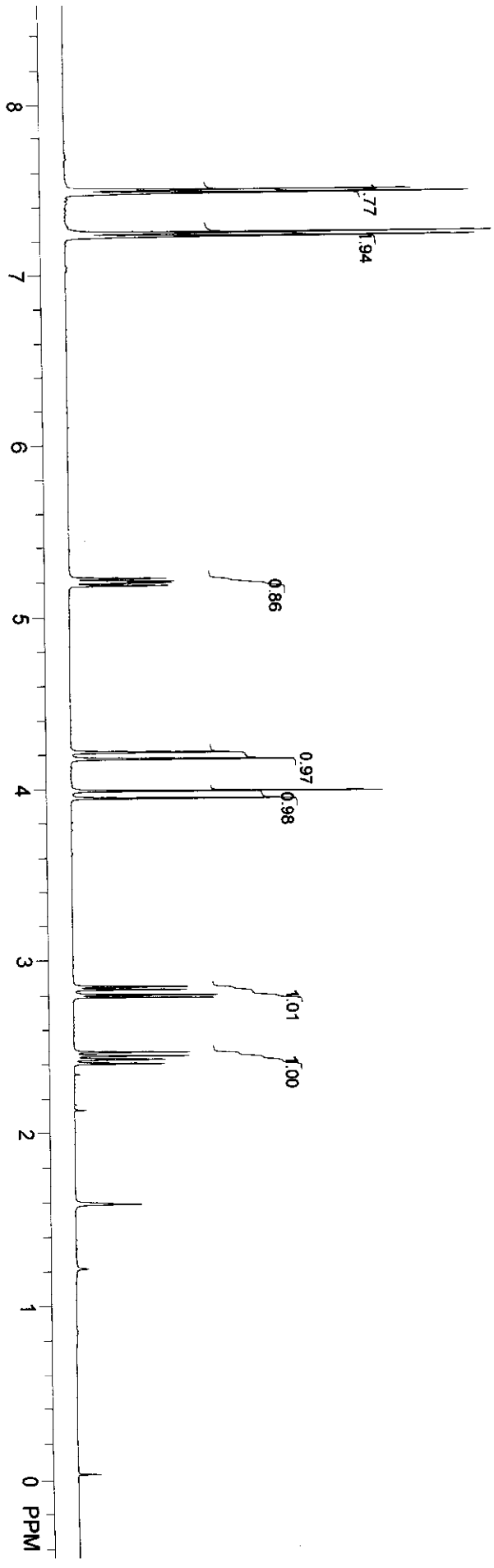
4i



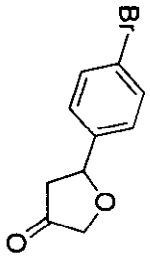

```
ylw-3-155-c

  Data Collected on:
    nmr500-inova500
  Archive directory:
    /export/home/vnmr1/vnmrsys/data
  Sample directory:

  File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 21.9 C / 295.1 K
Operator: lye

  Relax. delay 3.000 sec
  Pulse 58.7 degrees
  Acq. time 1.300 sec
  Width 40000.0 Hz
    88 repetitions
OBSERVE   C13, 125.6889872 MHz
DECOUPLE   H1, 499.8588575 MHz
  Power 36 dB
  on during acquisition
  off during delay
  WALTZ-16 modulated
DATA PROCESSING
  Line broadening 0.5 Hz
  FT size 131072
Total time 0 min
```

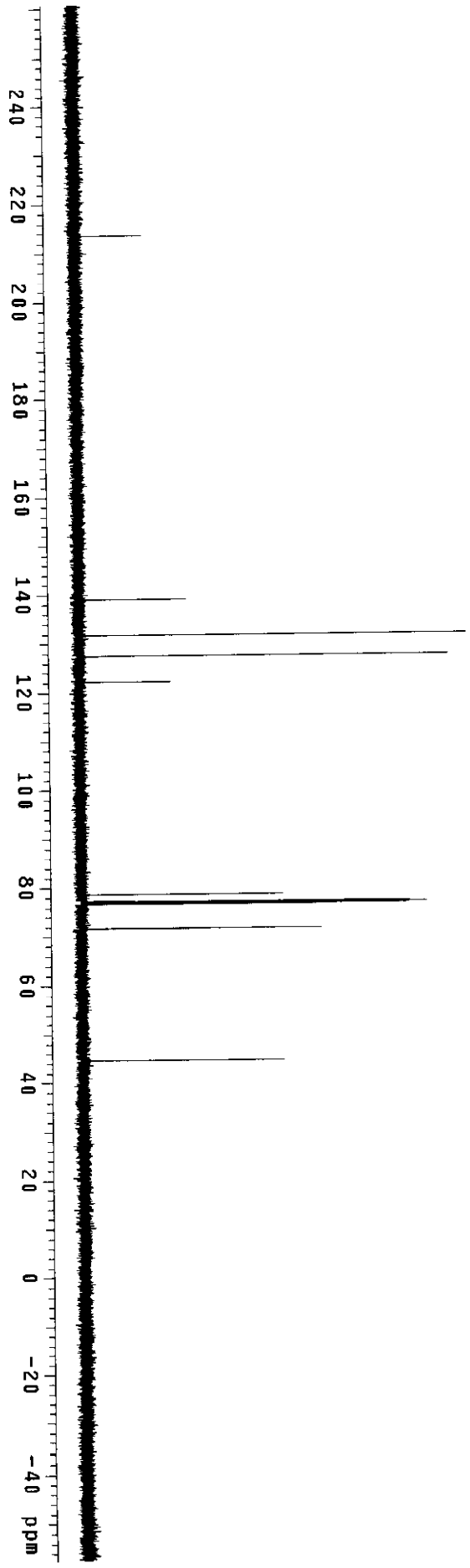


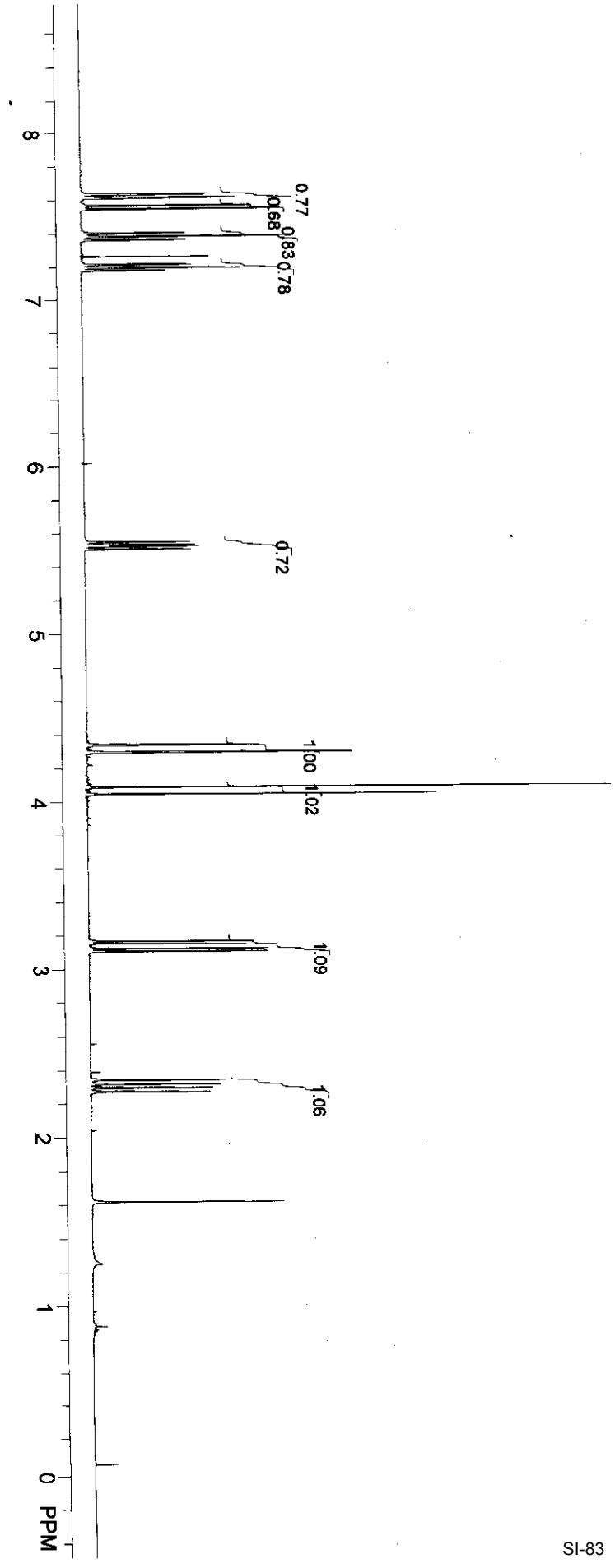
4i



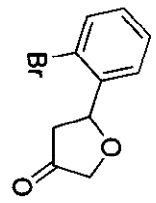
4j

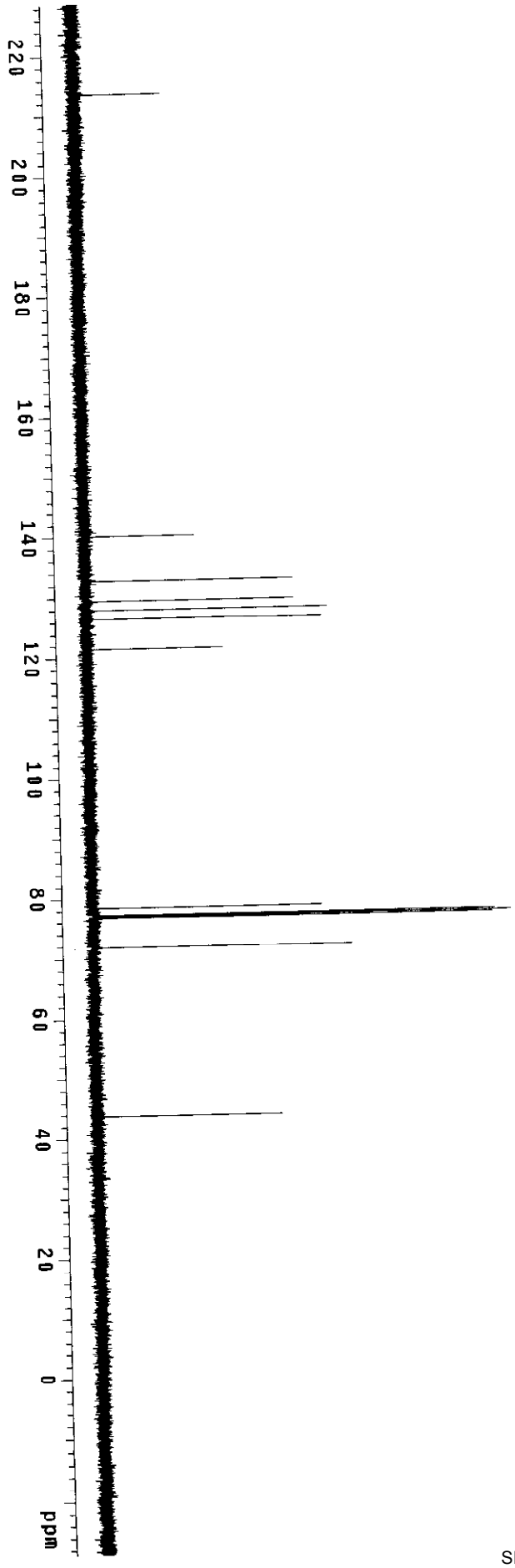


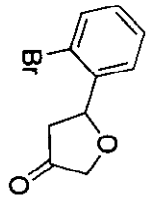

ylw-3-153-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
90 repetitions
OBSERVE C13, 125.6889866 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
Br
O
O
4j
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
ppm



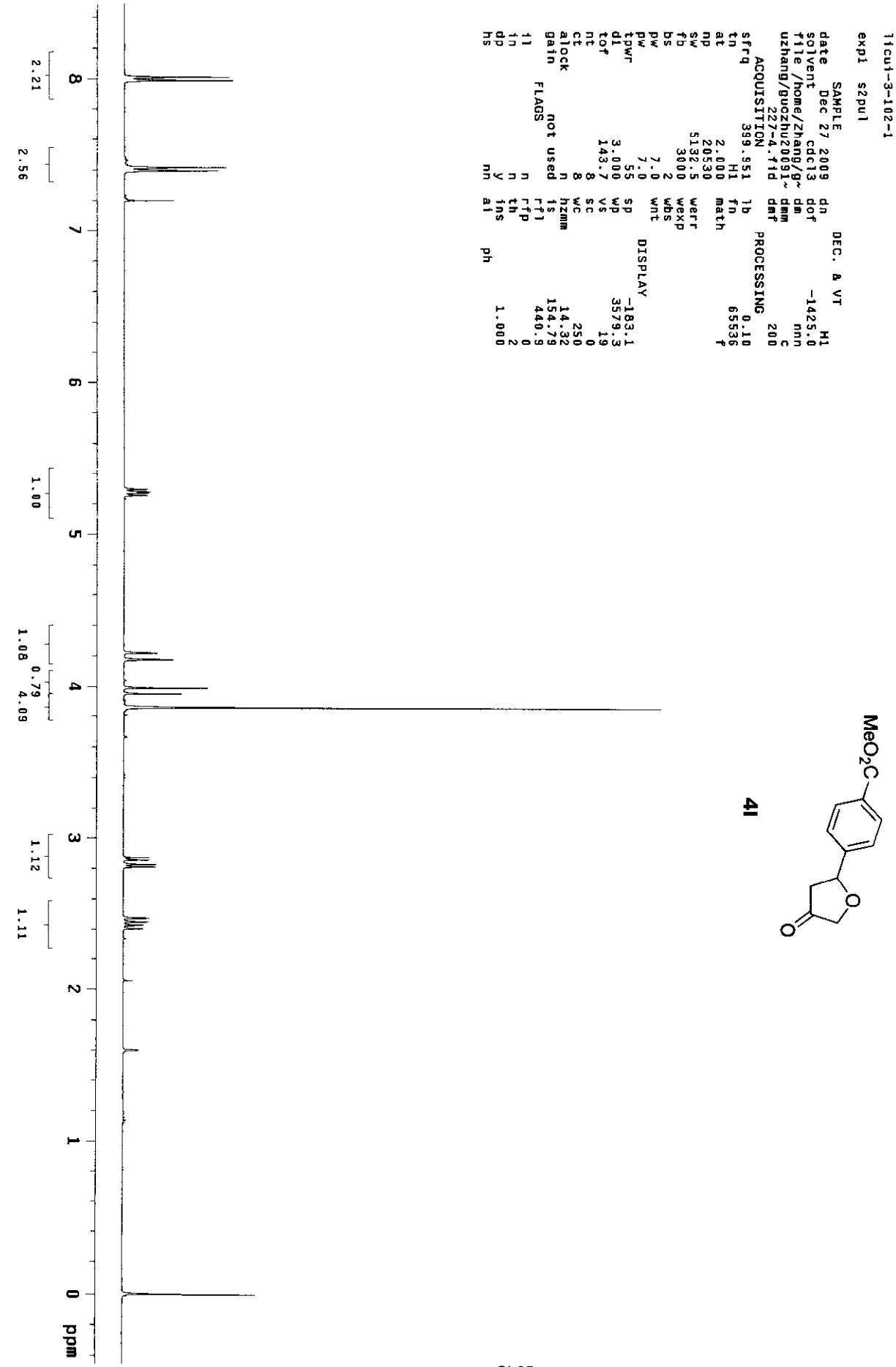
4k




ylw-3-156-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
76 repetitions
OBSERVE C13, 125.6889860 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acqulsition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Br
O
O

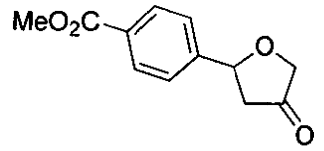
4k


11cui-3-102-1
exp1 s2pul
SAMPLE
date Dec 27 2009
solvent cdcl3
file /home/Zhang/g~
uzhang/guczhu20091~
227-4.fid
ACQUISITION
sfrq 399.951
tn H1
at 2.000
np 20530
sw 5132.5
fb 3000
bs 2
pw 7.0
pw 7.0
tpwr 55
dl 3.000
tof 143.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn H1
dof -1425.0
dm nnn
dmm c
dmf 200
PROCESSING
lb 0.10
fn 65536
math f
wnt
DISPLAY
-183.1
3579.3
19
0
sc
wc 250
hzmm 14.32
is 154.79
rfl 440.9
rfp 0
th 2
ins 1.000
ph
al
MeO2C
O
O
4l
8
7
6
5
4
3
2
1
0
ppm
2.21
2.56
1.00
1.08
0.79
4.09
1.12
1.11

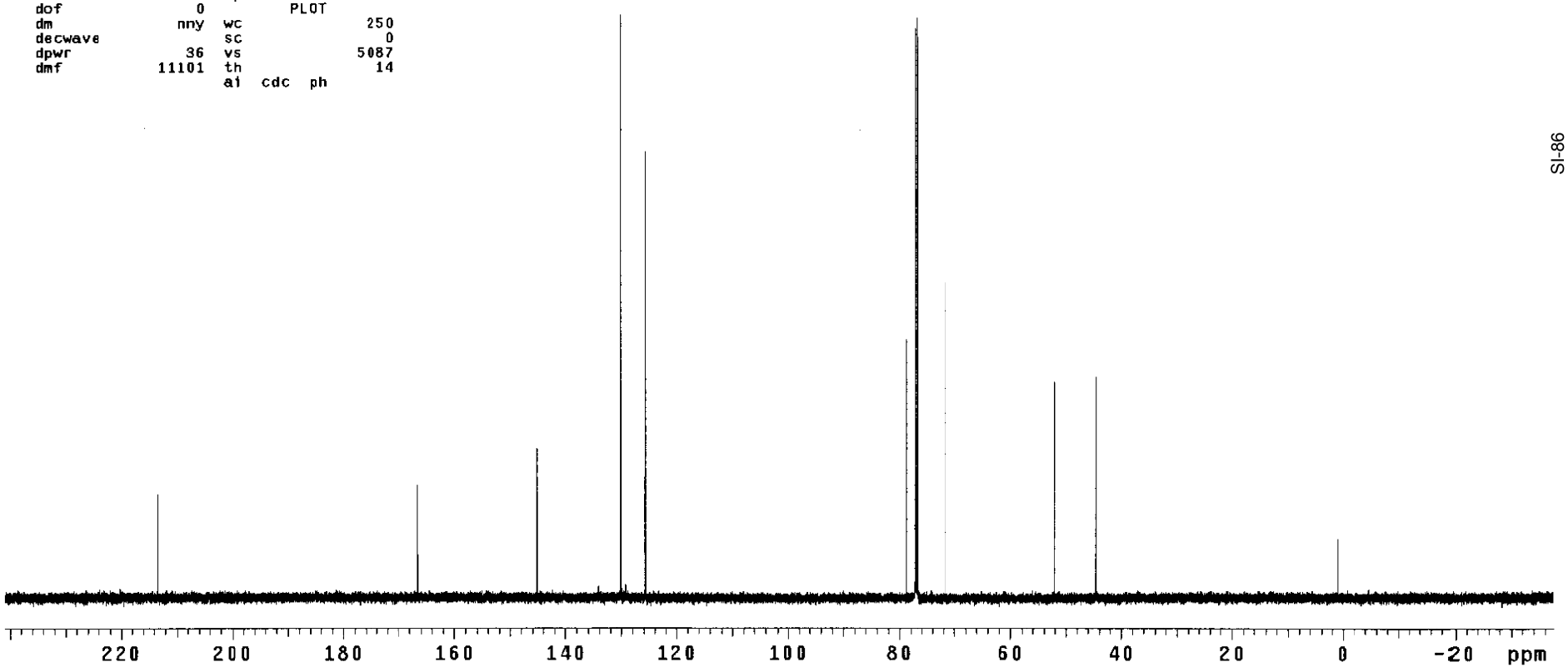
STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jan 1 2010	temp	22.0
solvent	cdcl3	gain	54
file	/home/Zhang/g~	spin	20
uzhang/C13-2009122~		hst	0.008
	7-4.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	34995.6	FLAGS	
at	1.301	il	n
np	91024	in	n
fb	19000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1200	lb	0.50
ct	316	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-4724.2
tof	865.4	wp	34995.1
tpwr	56	rfl	14402.8
pw	6.000	rfp	9678.1
DECOUPLER		rp	102.8
dn	H1	lp	-939.2
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	5087
dmf	11101	th	14
		ai cdc ph	



4l



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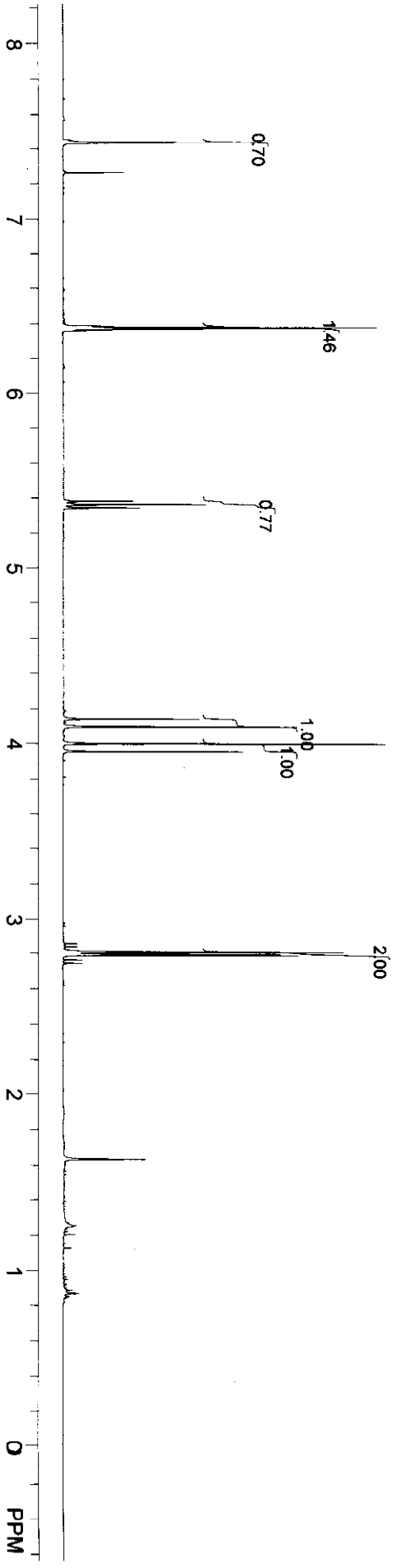
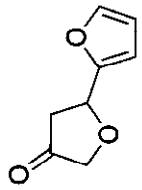
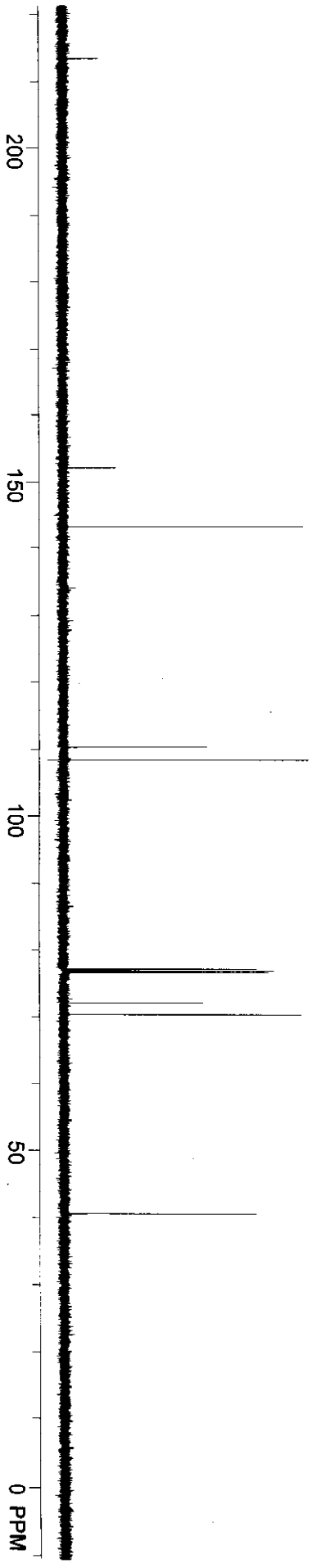
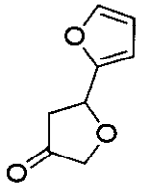


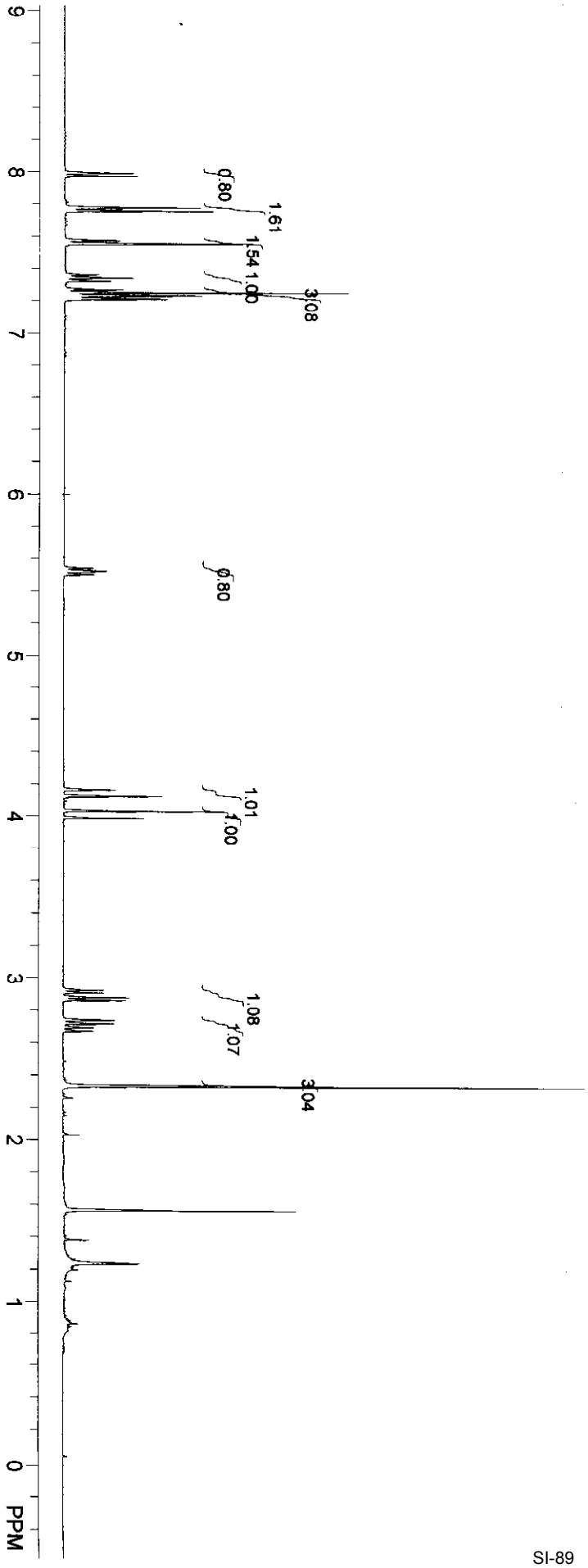
4m

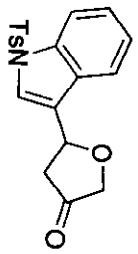




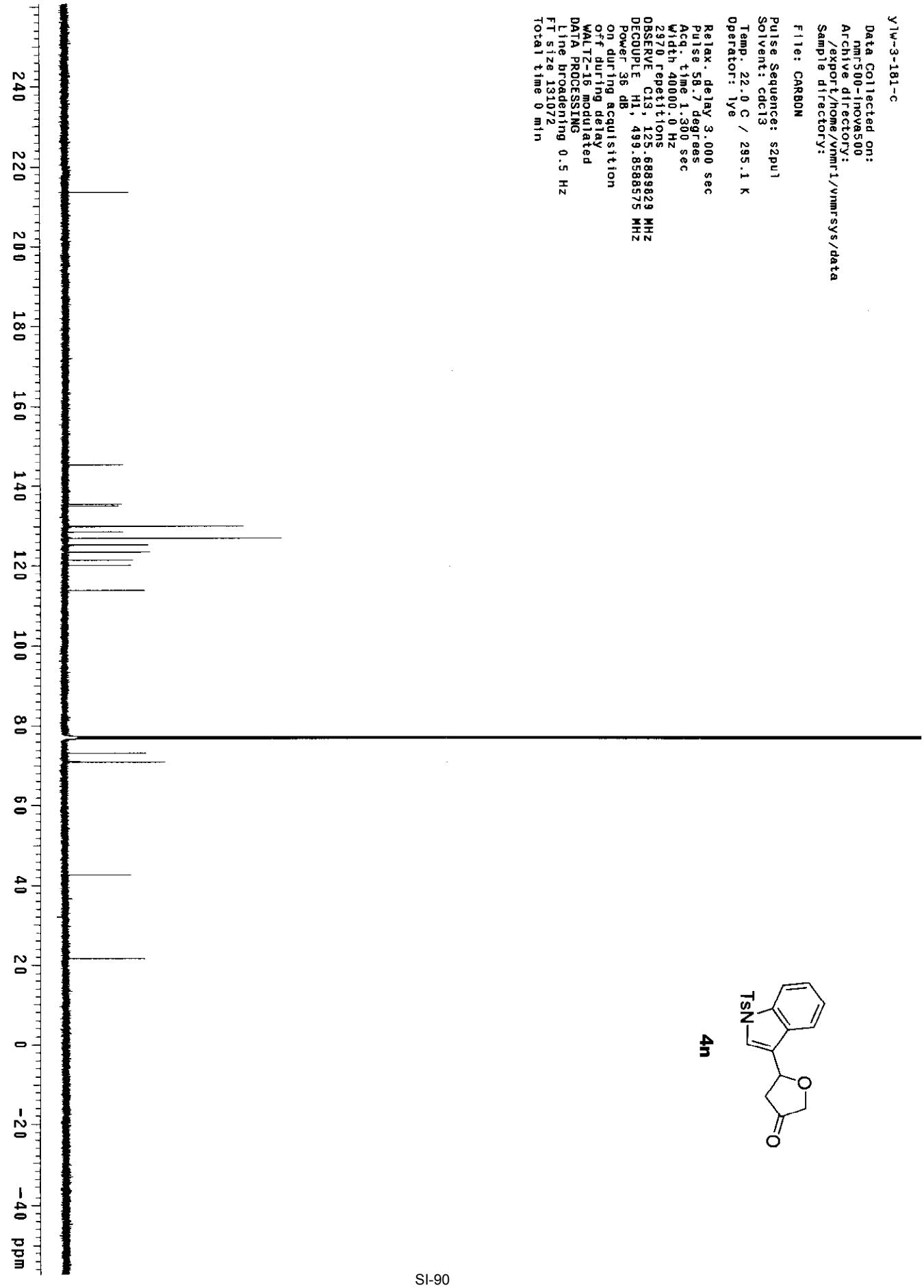
4m

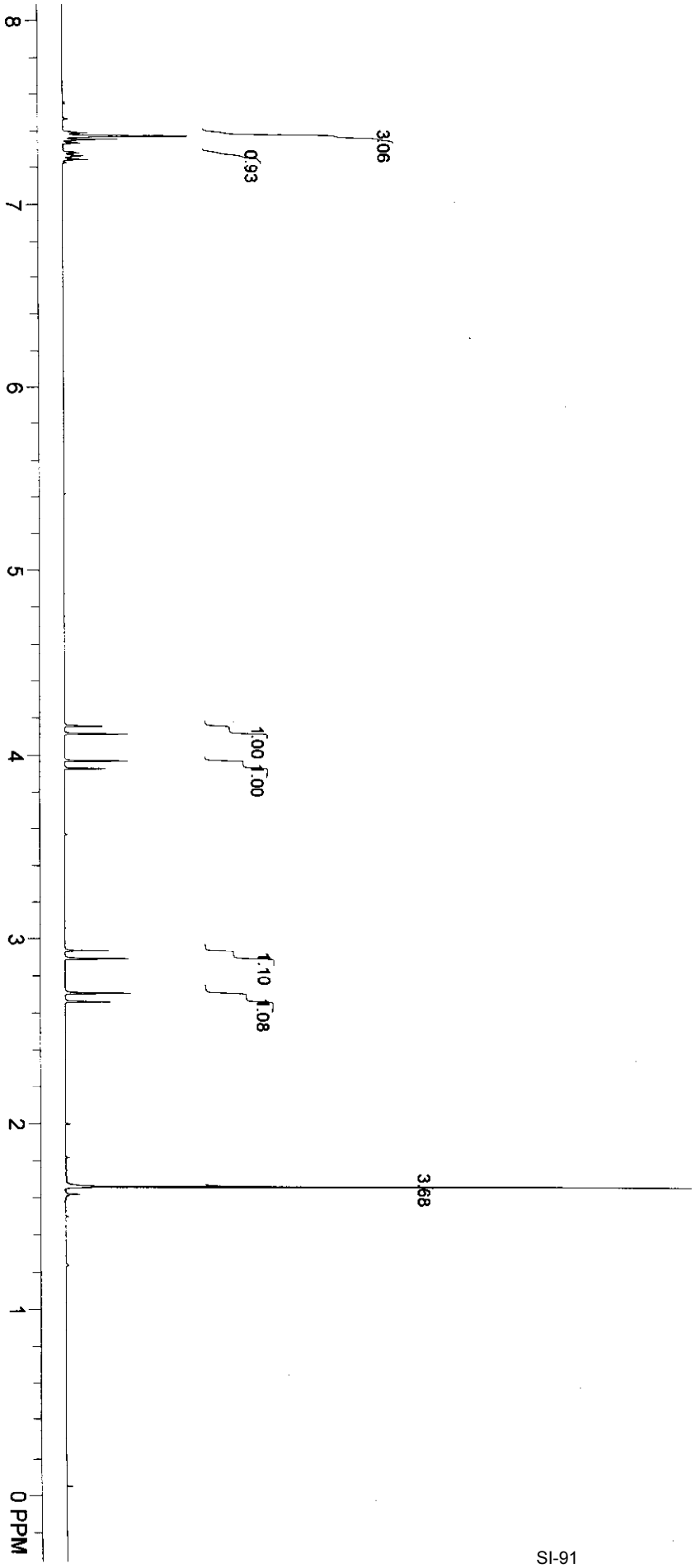



9
8
7
6
5
4
3
2
1
0
PPM
0.80
1.61
1.54
1.00
3.08
0.80
1.01
1.00
1.08
1.07
3.04

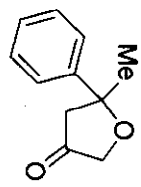

TsN
O
O

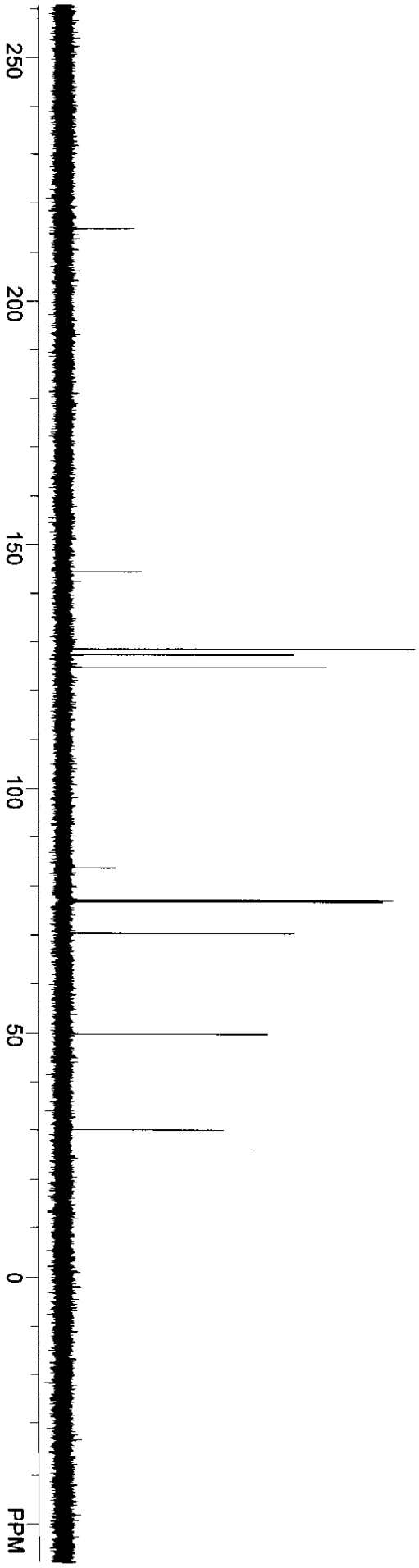
4n

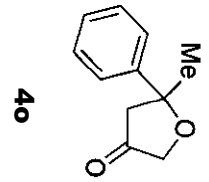

ylw-3-181-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
2970 repetitions
OBSERVE C13, 125.6889029 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
ppm
TsN
O
O
4n



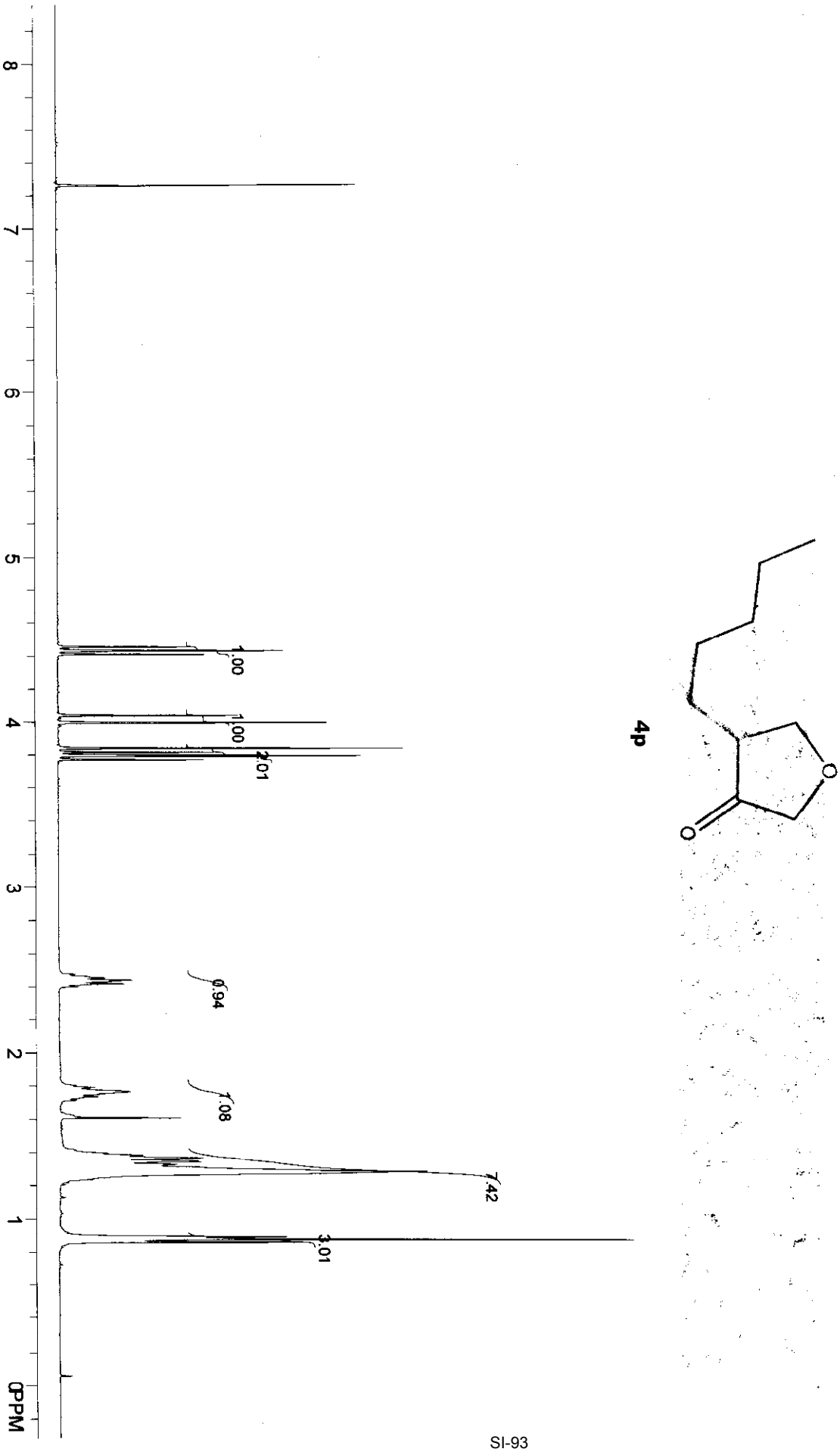
4o



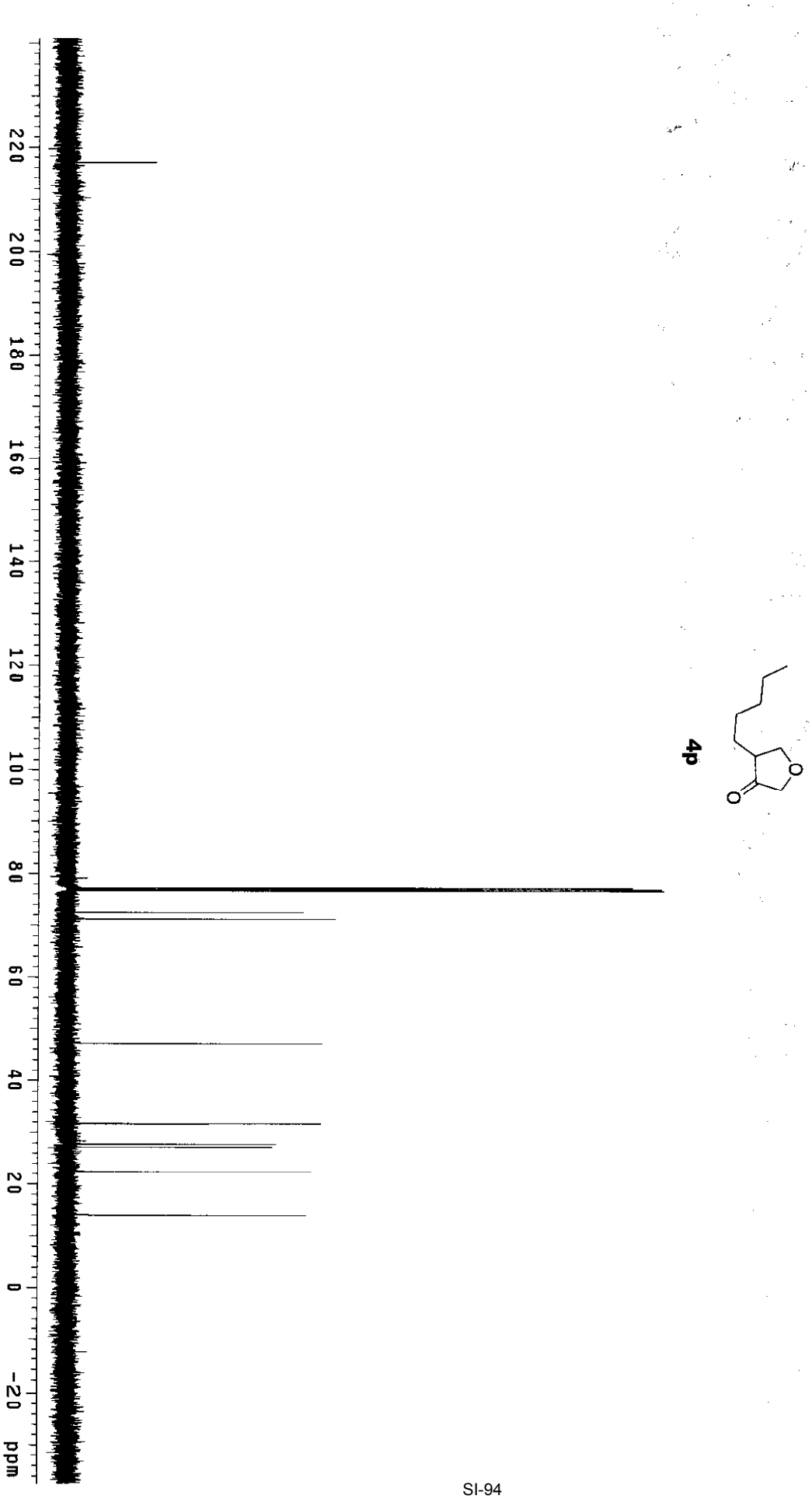




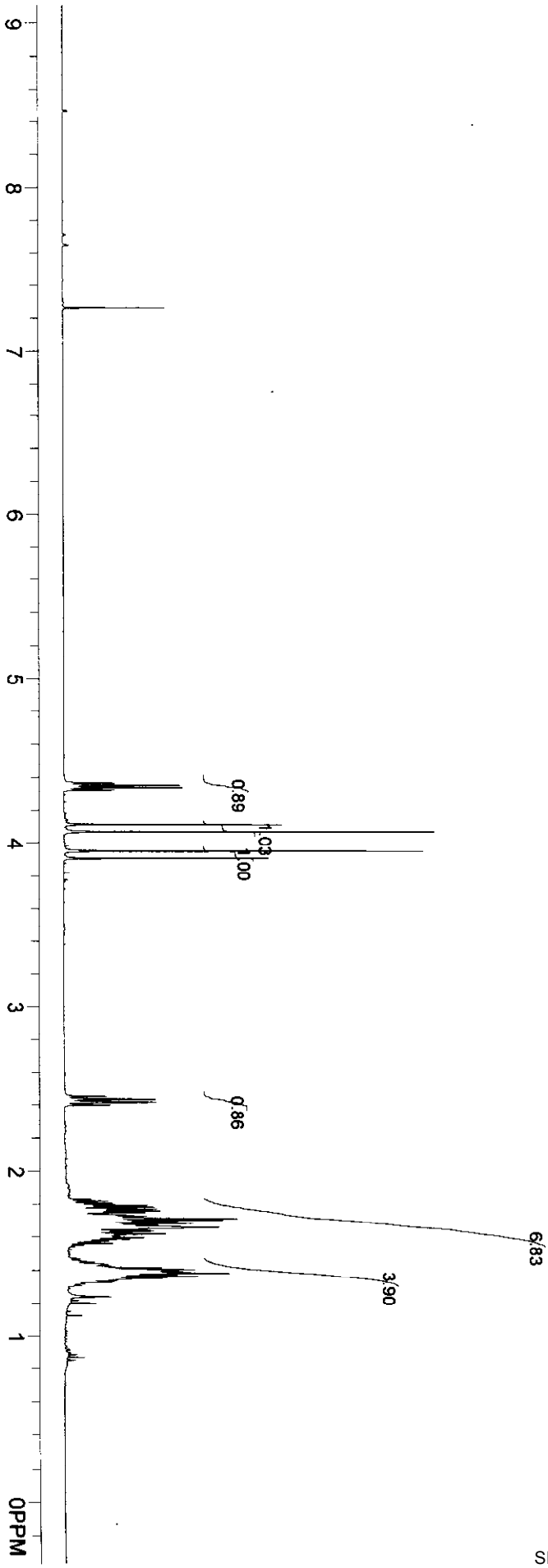
4o



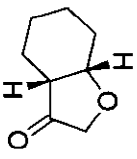
4p



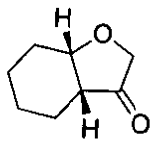
4p



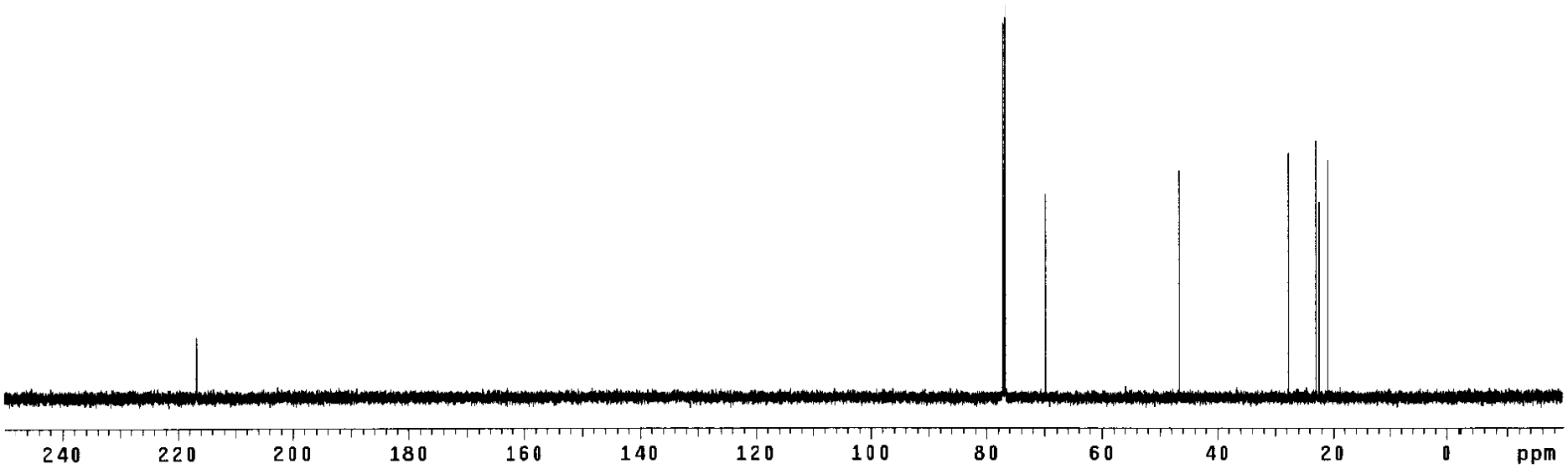
4q

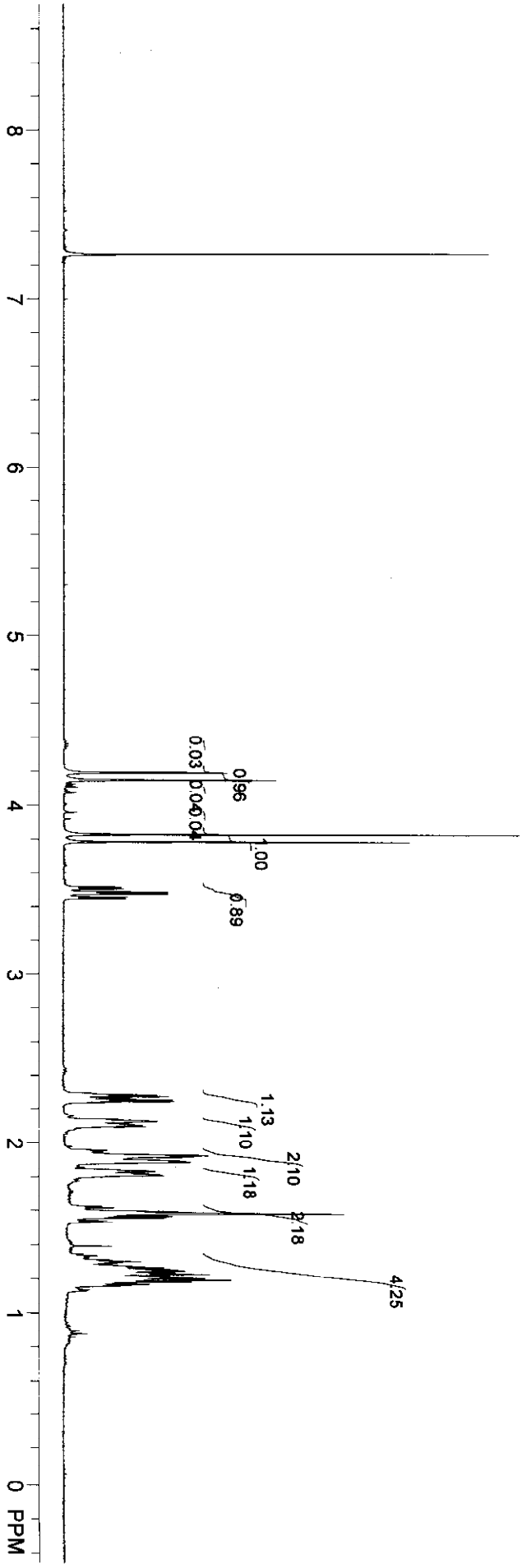


```
ylw-3-175-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 108 repetitions
OBSERVE  C13, 125.6889847 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```

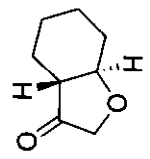


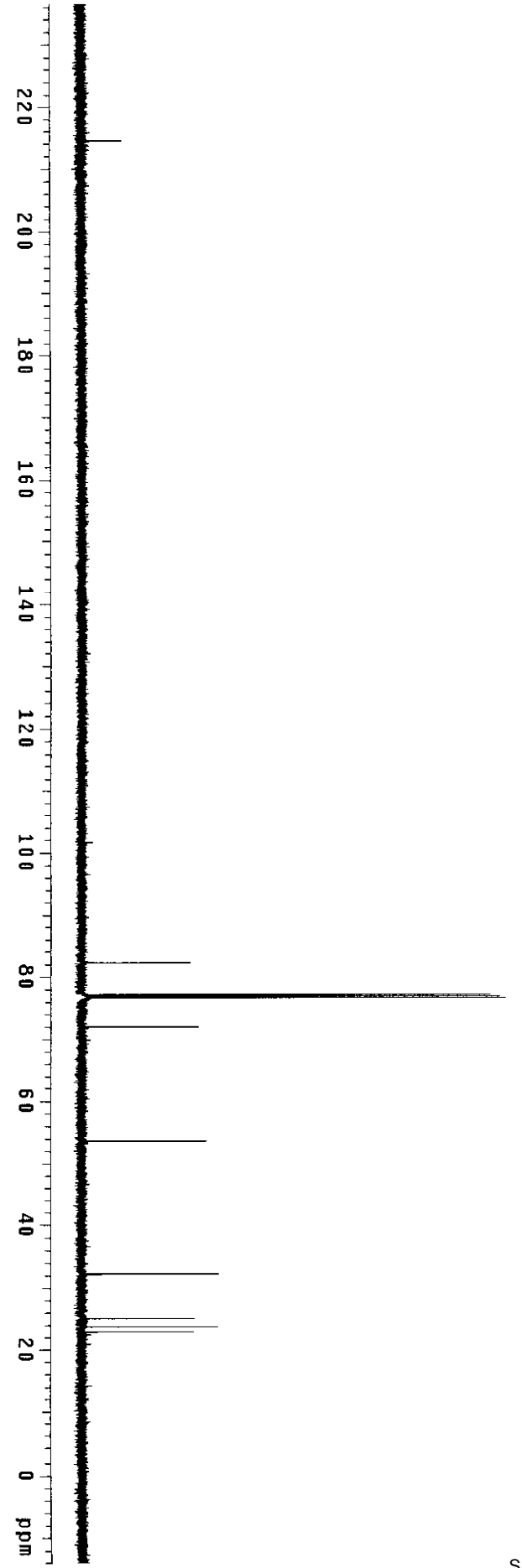
4q

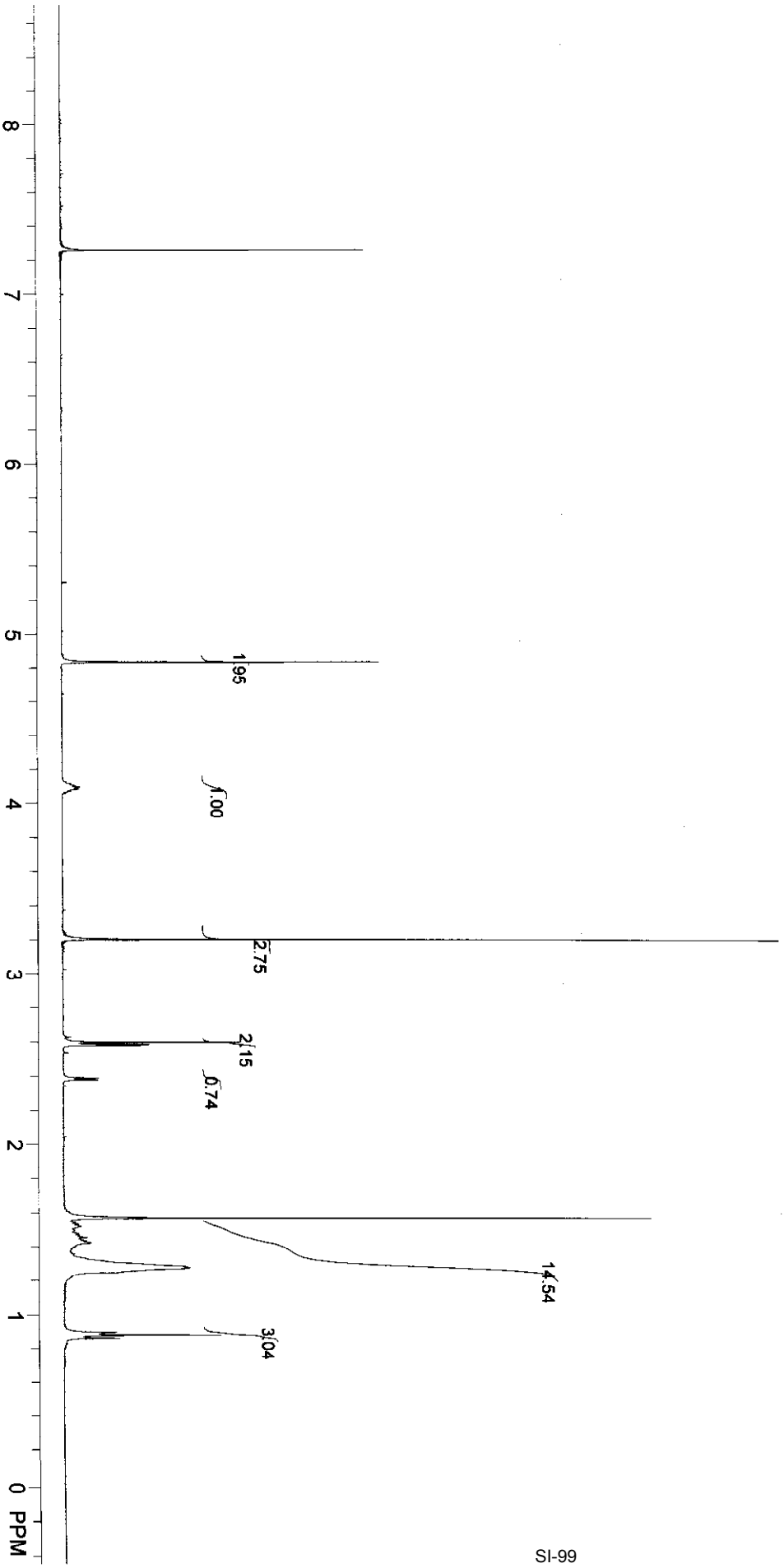


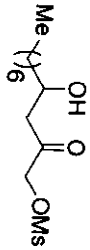

8
7
6
5
4
3
2
1
0 PPM
0.03
0.96
0.04
0.04
1.00
0.89
1.13
1.10
2.10
1.18
2.18
4.25

4r


H
H
O
O


ylw-3-167-5-c
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
176 repetitions
OBSERVE C13, 125.6889829 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min
4r
220
200
180
160
140
120
100
80
60
40
20
0
ppm





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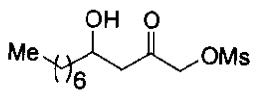
```
ylw-3-182-c

 Data Collected on:
   nmr500-inova500
 Archive directory:
   /export/home/vnmr1/vnmrsys/data
 Sample directory:

 File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: lye

 Relax. delay 3.000 sec
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 1966 repetitions
OBSERVE  C13, 125.6889823 MHz
DECOUPLE  H1, 499.8588575 MHz
 Power 36 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 131072
Total time 0 min
```



7