Supplementary Material (ESI) for Chemical Communications

Supporting Information

Gold-Catalyzed Efficient Synthesis of Azepan-4-ones via A Two-Step [5+2] Annulation

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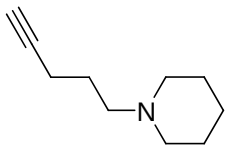
General. Ethyl acetate (ACS grade), hexanes (ACS grade), diethyl ether (ACS grade), NH_4OH (29.4% in H_2O, ACS reagent) were purchased from Fisher Scientific and used without further purification. Anhydrous dichloromethane (HPLC grade) was purified by distillation over calcium hydride. Tetrahydrofuran was distilled over sodium/benzophenone. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using Silicycle precoated 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 Waters micromass ZQ detector using electrospray method.

General procedure A: preparation of *N*-alkynylpiperidine

Pent-4-yn-1-yl tosylate (2 equiv) was added to a mixture of a secondary amine, NaI (0.5 equiv), and K_2CO_3 (3 equiv) in CH_3CN (0.5 M). The reaction was heated to reflux for 12 h and then cooled to room temperature. The reaction mixture was diluted with CH_2Cl_2 (10 mL/mmol), and the solid was filtered off. The filtrate was concentrated under *vacuum*, and the residue was dissolved in CH_2Cl_2. The resulting solution was washed with 5 % of aqueous NaOH, brine, dried with anhydrous $MgSO_4$, and concentrated under *vacuum*. The residue was purified through silica gel flash chromatography.

***N*-Pent-4-ynylpiperidine (1)**

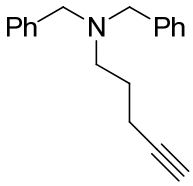


1

Compound **1** was prepared in 89 % yield according to the general procedure A (eluents: ethyl acetate: methanol : NH_4OH = 10: 1: 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.36 – 2.39 (m, 6H), 2.21 (td, 2H, *J* = 7.2, 2.8 Hz), 1.94 (t, 1H, *J* = 2.4 Hz), 1.67 – 1.75 (m, 2H),

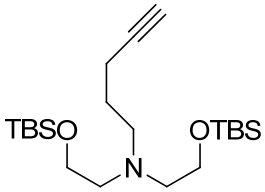
1.54 – 1.61 (m, 4H), 1.41 – 1.44 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 84.1, 68.2, 58.1, 54.5, 25.9, 25.8, 24.3, 16.4; IR (neat): 3312, 2936, 2763, 2739, 2119, 1443, 1352; MS (ES^+) Calculated for $[C_{10}H_{18}N]^+$: 152.1; Found: 152.1.

***N*, *N*-Dibenzylpent-4-ynylamine**



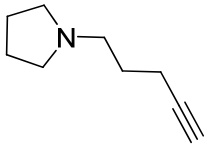
The above compound was prepared in 99 % yield according to the general procedure A (eluents: hexanes: ethyl acetate: Et_3N = 10 : 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.36 (m, 10H), 3.55 (s, 4H), 2.50 (t, 2H, *J* = 6.8 Hz), 2.12 (td, 2H, *J* = 7.2, 2.4 Hz), 1.85 (t, 1H, *J* = 2.8 Hz), 1.71 – 1.74 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.6, 128.6, 128.1, 126.8, 84.5, 68.1, 58.3, 52.3, 30.9, 26.3, 16.2; IR (neat): 3298, 3085, 3062, 3027, 2949, 2797, 2117, 1494, 1452, 1366; MS (ES^+) Calculated for $[C_{19}H_{22}N]^+$: 264.1; Found: 264.1.

***N*, *N*-Bis-[2-(tert-butyldimethylsilanyloxy)ethyl]pent-4-ynylamine**



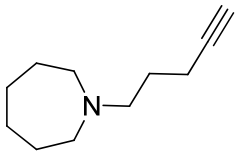
The above compound was prepared in 98 % yield according to the general procedure A (eluents: hexanes: ethyl acetate: Et_3N = 20 : 1 : 0.21). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.64 (t, 4H, *J* = 7.0 Hz), 2.59 – 2.65 (m, 6H), 2.21 (td, 2H, *J* = 2.5, 3.9 Hz), 1.93 (t, 1H, *J* = 3.5 Hz), 1.62 – 1.68 (m, 2H), 0.89 (s, 18H), 0.02 (s, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.5, 68.2, 62.0, 57.2, 54.3, 26.7, 25.9, 18.3, 16.0, -5.3; IR (neat): 3310, 2952, 2931, 2857, 2116, 1468,; MS (ES^+) Calculated for $[C_{21}H_{46}NO_2Si_2]^+$: 400.3; Found: 400.3.

***N*-Pent-4-ynylpyrrolidine**



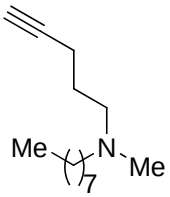
The above compound was prepared in 88 % yield according to the general procedure A (eluents: ethyl acetate : methanol : Et_3N = 1 : 1 : 0.02). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.42 – 2.48 (m, 6H), 2.18 (td, 2H, *J* = 7.2, 2.8 Hz), 1.89 (t, 1H, *J* = 2.4 Hz), 1.66 – 1.71 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.2, 68.4, 55.4, 54.2, 27.8, 23.4, 16.6; IR (neat): 3309, 2958,2116, 1466; MS (ES^+) Calculated for $[C_9H_{16}N]^+$: 138.1; Found: 137.7.

_N_-Pent-4-ynylazepane



The above compound was prepared in 83 % yield according to the general procedure A (eluents: hexanes: ethyl acetate: Et_3N = 10: 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.62 (t, 4H, *J* = 4.8 Hz), 2.55 (t, 2H, *J* = 7.2 Hz), 2.22 (td, 2H, *J* = 7.2, 2.8 Hz), 1.94 (t, 1H, *J* = 2.8 Hz), 1.59 –1.70 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.5, 68.1, 56.9, 55.4, 28.1, 27.0, 26.6, 16.3; IR (neat): 3310, 2926, 2857, 2815, 2778, 2116, 1458; MS (ES^+) Calculated for $[C_{11}H_{20}N]^+$: 166.2; Found: 166.2.

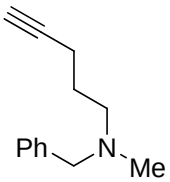
_N_-Methyl-_N_-octylpent-4-ynylamine



The above compound was prepared in 78 % yield according to the general procedure A (eluents: ethyl acetate : methanol: NH_4OH = 10 : 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.40 (t, 2H, *J* = 7.2 Hz), 2.28 (t, 2H, *J* = 3.6 Hz), 2.20 – 2.24 (m, 5H), 1.94 (t, 1H, *J* = 2.4 Hz), 1.62 – 1.72 (m, 2H), 1.40 – 1.49 (m, 2H), 1.20 – 1.34 (m, 10H), 0.87 (t, 3H, *J* = 6.4 Hz), ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.2, 68.1, 57.8, 56.3, 42.2, 31.7, 29.5, 29.2, 27.4,

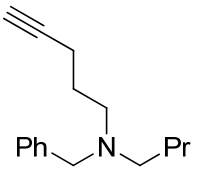
27.2, 26.2, 22.5, 16.2, 14.0; IR (neat):3310, 2926, 2857, 2794, 2121, 1463; MS (ES^+) Calculated for $[C_{14}H_{28}N]^+$: 210.2; Found: 210.2.

_N_-Benzyl-_N_-methylpent-4-ynylamine



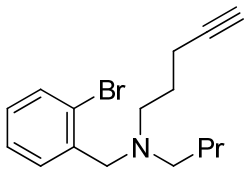
The above compound was prepared in 85 % yield according to the general procedure A (eluents: hexanes : ethyl acetate : Et_3N = 5 : 1 : 0.06). ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 – 7.34 (m, 5H), 3.49 (s, 2H), 2.47 (t, 2H, *J* = 6.8 Hz), 2.25 (td, 2H, *J* = 7.2, 2.8 Hz), 2.19 (s, 3H), 1.93 (t, 1H, *J* = 2.8 Hz), 1.70 – 1.78 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.2, 128.9, 128.1, 126.8, 84.4, 68.2, 62.3, 56.1, 42.1, 26.4, 16.2; IR (neat): 3302, 2949, 2838, 2790, 2117, 1494, 1453, 1365; MS (ES^+) Calculated for $[C_{13}H_{18}N]^+$: 188.1; Found: 188.1.

_N_-Benzyl-_N_-butylpent-4-ynylamine



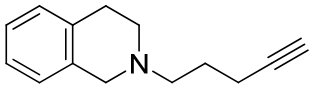
The above compound was prepared in 93 % yield according to the general procedure A (eluents: hexanes : ethyl acetate : Et_3N = 10 : 1 : 0.11). ^1H NMR (500 MHz, $CDCl_3$) δ 7.23- 7.35 (m, 5H), 3.56 (s, 2H), 2.52 (t, 2H, *J* = 6.5 Hz), 2.43 (t, 2H, *J* = 5.5 Hz), 2.23 (td, 2H, *J* = 7.0, 2.5 Hz), 1.92 (t, 1H, *J* = 2.5 Hz), 1.67 – 1.73 (m, 2H), 1.44 – 1.50 (m, 2H), 1.29 – 1.34 (m, 2H), 0.90 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.1, 128.7, 128.0, 126.6, 84.5, 68.1, 58.7, 53.5, 52.5, 29.2, 26.3, 20.5, 16.2, 14.0; IR (neat):3309, 3027, 2955, 2933, 2799, 2118, 1495, 1453; MS (ES^+) Calculated for $[C_{16}H_{24}N]^+$: 230.2; Found: 230.2.

_N_-(2-Bromobenzyl)-_N_-butylpent-4-ynylamine



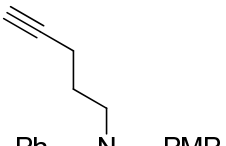
The above compound was prepared in 91 % yield according to the general procedure A (eluents: hexanes: ethyl acetate: Et_3N = 10 : 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.54 (m, 2H), 7.27 (t, 1H, *J* = 6.0 Hz), 7.09 (t, 1H, *J* = 5.6 Hz), 3.63 (s, 2H), 2.56 (t, 2H, *J* = 5.6 Hz), 2.45 (t, 2H, *J* = 5.6 Hz), 2.22 (td, 2H, *J* = 5.6, 2.0 Hz), 1.90 (t, 1H, *J* = 2.4), 1.63 – 1.71 (m, 2H), 1.43 –1.49 (m, 2H), 1.26 – 1.34 (m, 2H), 0.87 (t, 3H, *J* = 6.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.3, 132.5, 130.5, 127.9, 127.1, 124.1, 84.5, 68.2, 58.3, 53.9, 52.8, 29.3, 26.4, 20.6, 16.3, 14.0; IR (neat):3305, 2952, 2931, 2857, 2799, 2121, 1463; MS (ES^+) Calculated for $[C_{16}H_{23}BrN]^+$: 308.1; Found: 308.1.

2-(Pent-4-ynyl)-1,2,3,4-tetrahydroisoquinoline



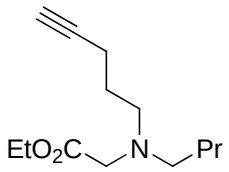
The above compound was prepared in 80 % yield according to the general procedure A (eluents: hexanes : ethyl acetate : Et_3N = 20 : 1 : 0.21). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.00 – 7.13 (m, 4H), 3.63 (s, 2H), 2.90 (t, 2H, *J* = 5.5 Hz), 2.74 (t, 2H, *J* = 6.0 Hz), 2.61 (t, 2H, *J* = 7.5 Hz), 2.29 (td, 2H, *J* = 2.5, 7.0 Hz), 1.96 (t, 1H, *J* = 2.5 Hz), 1.81 – 1.86 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.8, 134.3, 128.6, 126.5, 126.0, 125.5, 84.2, 68.4, 57.0, 56.2, 50.9, 29.1, 26.1, 16.3; IR (neat): 3296, 3021, 2921, 2804, 2768, 2116, 1498, 1466, 1376; MS (ES^+) Calculated for $[C_{14}H_{18}N]^+$: 200.1; Found: 200.2.

***N*-Benzyl-*N*-(4-methoxybenzyl)pent-4-ynylamine**



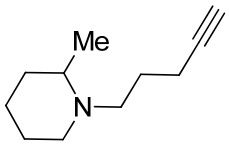
The above compound was prepared in 99 % yield according to the general procedure A (eluents: hexanes: ethyl acetate: Et_3N = 10 : 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22- 7.35 (m, 7H), 6.84 (d, 2H, *J* = 8.4 Hz), 3.79 (s, 3H), 3.53 (s, 2H), 3.49 (s, 2H), 2.48 (t, 2H, *J* = 7.2 Hz), 2.18 (td, 2H, *J* = 7.6, 2.8 Hz), 1.86 (t, 1H, *J* = 2.4 Hz), 1.68 – 1.75 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.4, 139.6, 131.3, 129.7, 128.6, 128.0, 126.6, 113.4, 84.3, 68.2, 58.0, 57.5, 55.0, 52.0, 26.2, 16.1; IR (neat): 3294, 2931, 2831, 2794, 2115, 1610, 1515, 1463; MS (ES^+) Calculated for $[C_{20}H_{23}NO+H]^+$: 294.2; Found: 294.2.

Ethyl butylpent-4-ynylaminoacetate



The above compound was prepared in 90 % yield according to the general procedure A (eluents: ethyl acetate : methanol : NH_4OH = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.13 (dd, 2H, *J* = 14.0, 7.5 Hz), 3.29 (s, 2H), 2.66 (t, 2H, *J* = 6.5 Hz), 2.54 (t, 2H, *J* = 8.0 Hz), 2.21 (td, 2H, *J* = 2.5, 7.0 Hz), 1.92 (t, 1H, *J* = 2.5 Hz), 1.62 – 1.67 (m, 2H), 1.39 – 1.43 (m, 2H), 1.23 – 1.31 (m, 5H), 0.88 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 84.3, 68.2, 60.1, 55.1, 54.0, 53.1, 29.7, 26.6, 20.4, 16.0, 14.2, 13.9; IR (neat): 3308, 2956, 2933, 2861, 2117, 1738, 1457; MS (ES^+) Calculated for $[C_{13}H_{24}NO_2]^+$: 226.2; Found: 226.2.

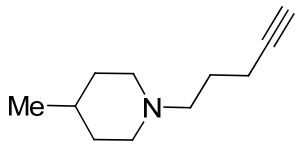
2-Methyl-1-(pent-4-ynyl)piperidine



The above compound was prepared in 87 % yield according to the general procedure A (eluents: hexanes : ethyl acetate= 10: 1, then hexanes : ethyl acetate : Et_3N = 4 : 1 : 0.05). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.72 – 2.87 (m, 2H), 2.40 – 2.47 (m, 1H), 2.13 – 2.30 (m, 4H), 1.94 (t, 1H, *J* = 2.8 Hz), 1.51 – 1.72 (m, 6H), 1.26 – 1.31 (m, 2H), 1.07 (d, 3H, *J* = 6.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.3, 68.3, 55.8, 52.8, 52.2, 34.6, 26.1, 24.5,

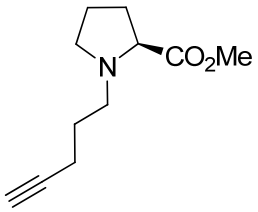
24.0, 19.1, 16.5; IR (neat): 3309, 2937, 2858, 2118, 1469, 1378; MS (ES^+) Calculated for $[C_{11}H_{20}N]^+$: 166.2; Found: 165.9

4-Methyl-1-(pent-4-ynyl)piperidine



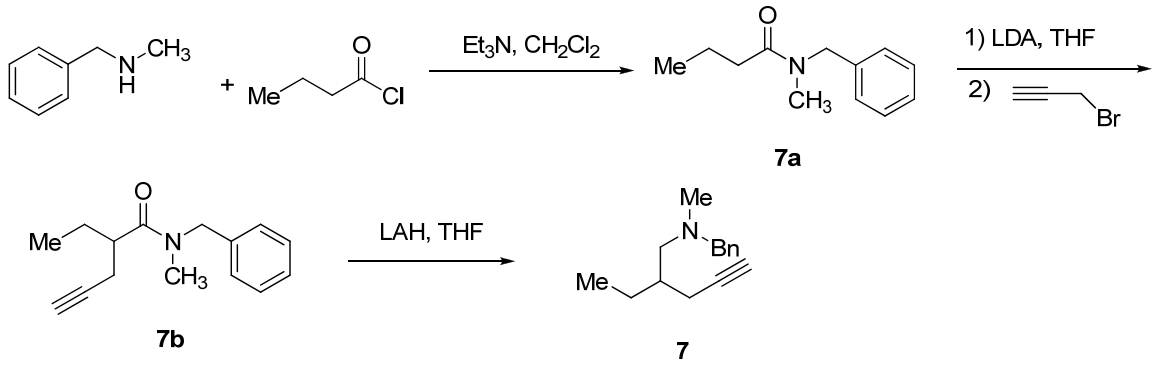
The above compound was prepared in 84 % yield according to the general procedure A (eluents: hexanes : ethyl acetate : Et_3N = 3 : 1 : 0.04). ^1H NMR (500 MHz, $CDCl_3$) δ 2.86 (d, 2H, *J* = 11.5 Hz), 2.39 (m, 2H), 2.21 (td, 2H, *J* = 7.0, 2.5 Hz), 1.88 – 1.94 (m, 3H), 1.69 – 1.75 (m, 2H), 1.59 – 1.62 (m, 2H), 1.32 – 1.36 (m, 1H), 1.19 – 1.27 (m, 2H), 0.91 (d, 3H, *J* = 11.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 84.3, 68.3, 57.9, 54.0, 34.3, 30.8, 26.0, 21.9, 16.6, 16.5; IR (neat):3310, 2947, 2926, 2116, 1452; MS (ES^+) Calculated for $[C_{11}H_{20}N]^+$: 166.2; Found: 165.9.

Methyl 1-(pent-4-ynyl)pyrrolidine-2-carboxylate



The above compound was prepared in 90 % yield according to the general procedure A using methyl prolinate hydrochloride as the starting material (eluents: ethyl acetate : methanol : NH_4OH = 9 : 1 : 0.1). ^1H NMR (500 MHz, $CDCl_3$) δ 3.68 (s, 3H), 3.11 – 3.15 (m, 2H), 2.69 – 2.75 (m, 1H), 2.44 – 2.49 (m, 1H), 2.29 – 2.34 (m, 1H), 2.18 – 2.23 (m, 2H), 2.05 – 2.09 (m, 1H), 1.87 – 1.91 (m, 3H), 1.75 – 1.80 (m, 1H), 1.65 – 1.71 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.7, 84.0, 68.3, 66.0, 53.7, 53.4, 51.7, 29.2, 27.5, 23.2, 16.3; IR (neat): 3304, 3053, 2953, 2811, 2117, 1742, 1435; MS (ES^+) Calculated for $[C_{11}H_{18}NO_2]^+$: 196.1; Found: 196.1.

Preparation of *N*-benzyl-*N*-(2-ethylpent-4-ynyl)methylamine (7)



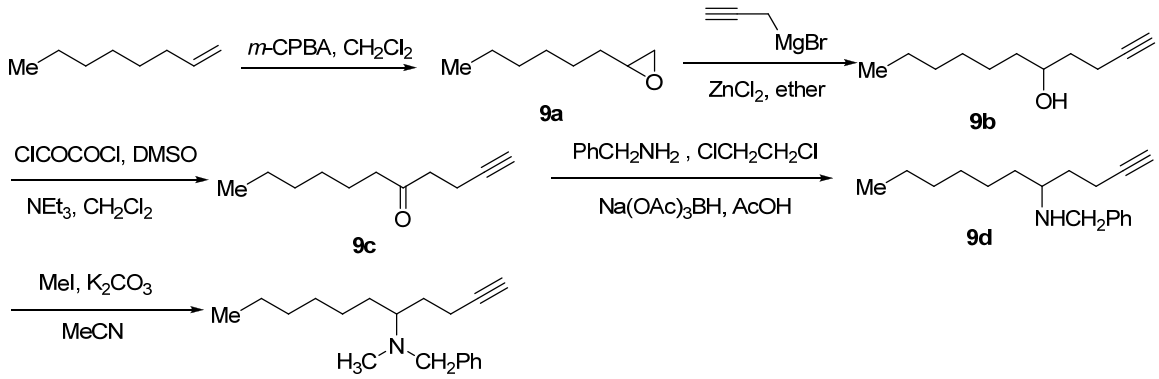
Benzylmethylamine (3.87 mL, 30 mmol) and NEt_3 (4.9 mL, 35 mmol) in CH_2Cl_2 (40 mL) was added dropwise to a solution of butyryl chloride (3.6 mL, 35 mmol) in CH_2Cl_2 (20 mL) at 0 °C. The mixture was stirred at room temperature for 5 h. The reaction mixture was treated with aqueous NH_4Cl (1N, 20 mL) and was extracted with CH_2Cl_2 (3 x 30 mL). The combined organic phase was dried and concentrated. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate = 5 : 1) to give compound **7a** (5.73 g, 28.5 mmol) in 95% yield.

At –78 °C, a solution of amide **7a** (2.0 g, 10.5 mmol) in THF (10 mL) was added dropwise to a THF solution of LDA (50 mL, 0.25 M in THF). The resulting mixture was stirred at –78 °C for 2 h before the addition of propargyl bromide (1.12 mL, 12.6 mmol). The reaction mixture was allowed to warm to room temperature overnight. The mixture was quenched by 1 N NH_4Cl (20 mL) and extracted with Et_2O (3 × 30 mL). The combined organic phases were washed with brine, dried with anhydrous $MgSO_4$ and filtered. The filtrate was concentrated, and the resulting residue was purified through silica gel flash column (eluents: hexanes: ethyl acetate = 10:1) to afford amide 7**b** (1.88 g, 8.19 mmol) in 78 % yield.

To a solution of compound **7b** (1.306 g, 5.6 mmol) in THF (75 mL) was added LAH (0.64 g, 16.8 mmol) at 0 °C. The resulting mixture was refluxed overnight and then diluted with ether (50 mL). Upon cooling to 0 °C, the reaction mixture was treated dropwise and sequentially with 0.64 mL water, 0.64 mL 15 % aqueous sodium hydroxide and 1.92 mL water and then stirred at room temperature for 15 min. After removal of the white solids via filtration, the filtrate was concentrated, and the resulting residue was

purified through silica gel flash column (eluents: hexanes: ethyl acetate : Et_3N = 10 : 1 : 0.11) to afford tertiary amine **7** (0.71 g, 3.30 mmol) in 59 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.35 (m, 5H), 3.54 (d, 1H, *J* = 13.0 Hz), 3.46 (d, 1H, *J* = 13.0 Hz), 2.25 – 2.40 (m, 4H), 2.18 (s, 3H), 1.91 (td, 1H, *J* = 3.0 Hz), 1.73 (m, 1H), 1.48 (m, 2H), 0.94 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.5, 128.9, 128.1, 126.8, 83.0, 69.0, 62.9, 60.9, 42.5, 36.9, 24.2, 20.5, 11.1; IR (neat): 3307, 2963, 2841, 2794, 2114, 1454, 1380; MS (ES^+) Calculated for $[C_{15}H_{22}N]^+$: 216.2; Found: 216.2.

Preparation of *N*-benzyl-*N*-(1-but-3-ynylheptyl)methylamine (9)



m-CPBA (4.93 g, 22 mmol) was added to a solution of 1-octene (2.24 g, 20 mmol) in H_2O (20 mL) at 0 °C. The reaction was vigorously stirred at 0 °C for 4 h and at room temperature for 2 h. The reaction mixture was extracted twice with diethyl ether (2 x 100 mL). The combined organic phases were washed sequentially with a cooled solution of 10% NaOH (20 mL) and saturated brine (20 mL) and dried with Na_2SO_4. Subsequent filtration and concentration afford practically pure **9a** (1.72 g, 13.4 mmol) in 67% yield.

To a solution of **9a** (1.0 g, 5.8 mmol) and $ZnCl_2$ (79 mg, 0.58 mmol) in diethyl ether (6 mL) at -78 °C was added propargylmagnesium bromide (20 mL, 1 M solution in diethyl ether, 20 mmol). The reaction was slowly warmed to room temperature and further stirred for 6 h. The reaction was then quenched with aq. NH_4Cl (20 mL), and the resulting mixture was extracted with diethyl ether (50 mL × 2). The combined organic phases were dried (Na_2SO_4) and filterted. The filtrate was concentrated to afford alcohol **9b** (1.1g), which was used in the next step without further purification.

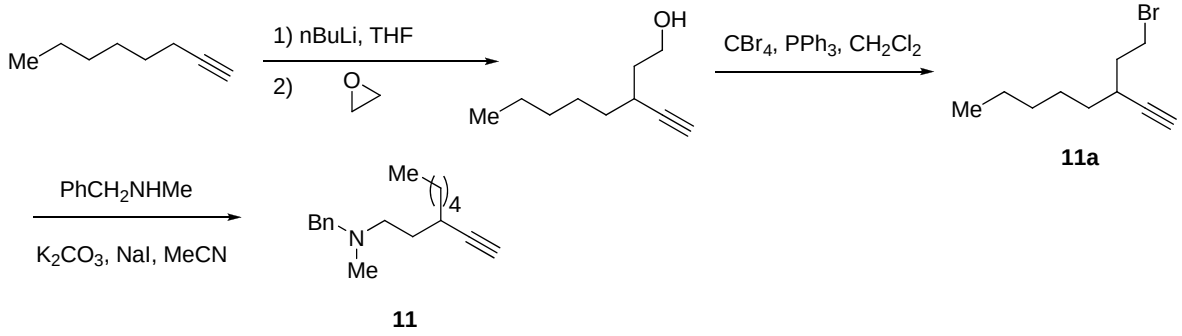
A solution of dry DMSO (1.0 g, 12.7 mmol) in CH_2Cl_2 (5 mL) was added dropwise to a stirred solution of oxalyl chloride (0.36 g, 6.4 mmol) in CH_2Cl_2 (15 mL) under N_2 at -78 °C. The solution was stirred for 0.5 h before the slow addition of a solution of **9b** (0.974 g, 5.8 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred for 1 h at -78 °C and then treated drop-wise with NEt_3 (4 mL, 29 mmol). The reaction mixture was warmed to room temperature over 0.5 h before addition of water (30 mL). The mixture was extracted with CH_2Cl_2 (3 × 50 mL), and the combined organic phases were washed with brine, dried ($MgSO_4$) and filtered. The filtrate was concentrated under *vacuum*. The resulting residue was purified through silica gel flash column (eluents: hexanes: ethyl acetate = 5:1) to afford ketone **9c** (0.65 g, 3.89 mmol) in 67 % yield.

Benzylamine (0.197 g, 1.84 mmol) and ketone **9c** (0.278 g, 1.67 mmol) were mixed in 1,2-dichloroethane (10 mL) and then treated with sodium triacetoxyborohydride (0.7 g, 3.32 mmol) and AcOH (0.5 mL) at 0 °C. The mixture was stirred at room temperature for 12 h. The reaction mixture was quenched by the addition of 1 N aqueous NaOH (10 mL)and then extracted with CH_2Cl_2 (3 x 20 mL). The organic phases were combined and washed with brine and dried ($MgSO_4$). Upon filtration, the filtrate was evaporated to give amine **9d** (0.357 g, 1.39 mmol, 83% yield), which was used in the next step without further purification.

MeI (0.088 mL, 1.42 mmol) was added to a mixture of a **9d** (0.303 g, 1.18 mmol), K_2CO_3 (0.85 g, 4.72 mmol) in CH_3CN (30 mL). The reaction was heated to reflux for 4 h and then the reaction was cooled to room temperature. The reaction mixture was diluted with CH_2Cl_2 (20 mL), and the solids were filtered off. The filtrate was washed with 5 % of aqueous NaOH (15 mL), brine, and dried with anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under *vacuum*. The residue was purified through silica gel flash column (eluents: hexanes: ethyl acetate : Et_3N = 5:1:0.06) to afford tertiary amine **9** (0.288 g, 1.06mmol) in 90 % yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 – 7.36 (m, 5H), 3.62 (d, 1H, *J* = 13.5 Hz), 3.56 (d, 1H, *J* = 13.5 Hz), 2.62 – 2.65 (m, 1H), 2.28 – 2.40 (m, 2H), 2.15 (s, 3H), 1.96 (t, 1H, *J* = 2.5 Hz), 1.59 – 1.74 (m, 3H), 1.24 – 1.38 (m, 9H),

0.94 (t, 3H, J = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.5, 128.5, 128.1, 126.6, 85.0, 68.0, 61.6, 58.1, 35.9, 31.8, 29.7, 29.5, 28.6, 27.2, 22.6, 16.1, 14.1; IR (neat): 3310, 2931, 2857, 2789, 2121, 1457; MS (ES^+) Calculated for $[C_{19}H_{30}N]^+$: 272.2; Found: 272.2.

Preparation of *N*-benzyl-*N*-(3-ethynyloctyl)methylamine (11)



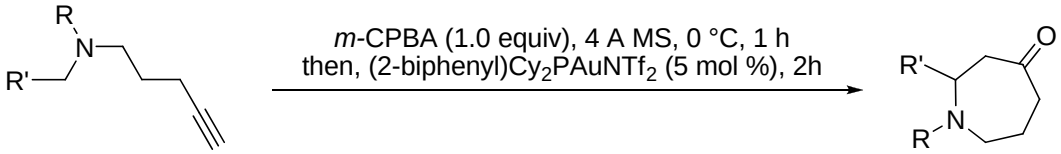
11

3-Pentyl-4-pentyn-1-ol was synthesized according to the literature procedure (Miura, K.; Wang, D.; Matsumoto, Y.; Hosomi, A. *Org. Lett.* **2005**, *7*, 503-505). 3-Pentyl-4-pentyn-1-ol (0.820 g, 5.32 mmol), CBr_4 (3.182 g, 9.58 mmol), and 2,6-lutidine (3.03 mL, 26.1 mmol) were mixed at 0 ºC in CH_2Cl_2 (30 mL) and then treated drop-wise with a solution of PPh_3 (2.788 g, 10.64 mmol) in CH_2Cl_2 (10 ml). The reaction mixture was stirred for 5 h prior to concentration, addition of ether, and removal of solid by filtration through Celite. The solution was washed with 1N HCl, dried with anhydrous $MgSO_4$, and concentrated. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate : Et_3N = 5 : 1 : 0.06) to give bromide **11a** which used directly in the next step.

Compound **11a** was added to a mixture of benzylmethyl amine (0.752 mL, 5.83 mmol), NaI (0.40 g, 2.67 mmol), and K_2CO_3 (3.8 g, 21.1 mmol) in CH_3CN (50 mL). The reaction was heated to reflux for 12 h and then cooled to room temperature. The reaction mixture was diluted with CH_2Cl_2 (50 mL), and the solids were filtered off. The filtrate was concentrated under *vacuum*. The residue was dissolved in CH_2Cl_2, and washed with 5 % aqueous NaOH. The organic layer was dried with anhydrous $MgSO_4$ and concentrated under *vacuum*. The residue was purified through silica gel flash chromatography (eluents:

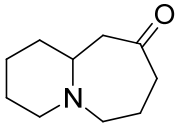
hexanes : ethyl acetate : Et_3N = 3 : 1 : 0.04) to yield tertiary amine **11** in 79% yield in two steps. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.33 (m, 5H), 3.47 – 3.55 (m, 2H), 2.55 (t, 2H, *J* = 7.2 Hz), 2.43 – 2.49 (m, 1H), 2.20 (s, 3H), 2.02 (t, 1H, *J* = 2.4 Hz), 1.28 – 1.71 (m, 10H), 0.91 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.3, 129.2, 128.3, 127.0, 88.0, 69.3, 62.6, 55.4, 42.4, 35.0, 32.9, 31.8, 29.5, 27.0, 22.7, 14.2; IR (neat):3305, 2931, 2857, 2789, 2110, 1455; MS (ES^+) Calculated for $[C_{18}H_{28}N]^+$: 258.2; Found: 258.2.

General procedure B: Preparation of Azepan-4-ones



m-CPBA (1.0 equiv) was added into a mixture of a pent-4-yn-1-ylamine and 4 Å MS (5 x weight of *m*-CPBA) in CH_2Cl_2 (0.05 M) under N_2 at 0 ℃. The *N*-oxide formation was monitored by TLC. Upon completion (~1 h), (2-biphenyl)$Cy_2PAuNTf_2$ (5 mol %) was added and the reaction mixture was stirred at 0 ℃ until all the *N*-oxide was consumed (~2 h). The reaction mixture was diluted with CH_2Cl_2, and the molecular sieves were filtered off. The filtrate was washed with 5 % aqueous Na_2CO_3, dried with Na_2SO_4, and concentrated under *vacuum*. The residue was purified through silica gel flash chromatography.

Octahydropyrido[1,2-*a*]azepin-9-one (2)

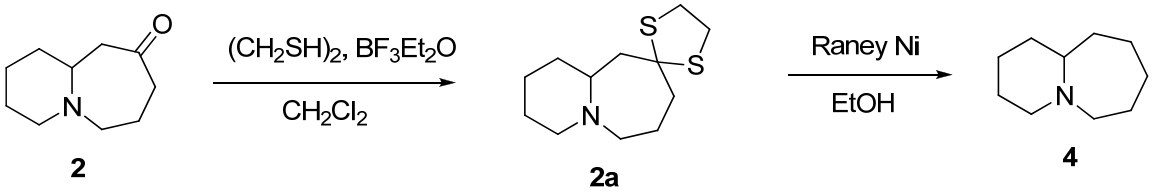


2

Compound **2** was prepared in 79 % yield according to the general procedure B. The reaction times are 1 h for the formation of N-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: ethyl acetate: methanol : NH_4OH = 2: 1: 0.03). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.03 (dd, 1H, *J* = 7.6, 13.6 Hz), 2.86 – 2.94 (m, 2H), 2.55 (dt, 1H, *J* = 5.0, 16.8 Hz), 2.02- 2.42 (m, 6H), 1.89 – 1.96 (m,

1H), 1.55 – 1.73 (m, 4H), 1.35 – 1.45 (m, 1H), 1.24 – 1.32 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.8, 60.9, 59.5, 56.8, 51.5, 42.3, 34.8, 25.6, 24.0, 23.0; IR (neat): 2934, 2807, 2770, 1707, 1444, 1348; MS (ES^+) Calculated for $[C_{10}H_{18}NO]^+$: 168.1; Found: 167.9.

Decahydropyrido[1,2-a]azepine (4)

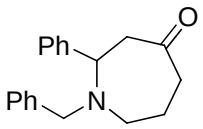


2 **2a** **4**

To a solution of **2** (55.1 mg, 0.33 mmol) in dry CH_2Cl_2 (6 mL) was added 1,2-ethanedithiol (0.27 mL, 10 equiv) and boron trifluoride etherate (0.103 mL, 2.5 equiv). The mixture was stirred for 2 h and then quenched with 1 N NaOH (5 mL). The aqueous phase was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were washed with 1 N NaOH (5 mL), dried Na_2SO_4 and concentrated. The crude product was purified by silica column chromatography (eluents: ethyl acetate: methanol : NH_4OH = 1: 1: 0.02) to give **2a** (62.4 mg, 0.26 mmol) in 78% yield.

To a solution of **2a** (62.4 mg, 0.26 mmol) in absolute methanol (5 mL) was added Raney nickel (1.8 g) under H_2 atmosphere and heated to reflux for 2 h. The reaction mixture was filtered through Celite and washed with diethyl ether. The filtrate was concentrated and purified by silica column chromatography (eluents: ethyl acetate: methanol : Et_3N = 1: 3: 0.04) to give **4** (41.1 mg, 0.17 mmol) in 65% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.84 (d, 1H, *J* = 11.0 Hz), 2.72 – 2.74 (m, 1H), 2.47 (t, 1H, *J* = 12.5 Hz), 2.22 – 2.27 (m, 1H), 1.96 – 2.40 (m, 1H), 1.67 – 1.76 (m, 4H), 1.49- 1.66 (m, 8H), 1.30 – 1.42 (m, 1H), 1.21 – 1.28 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 65.6, 57.5, 35.4, 34.4, 27.1, 26.8, 26.1, 24.9, 24.5; IR (neat): 2930, 2854, 2762, 1467, 1442, 1367; MS (ES^+) Calculated for $[C_{10}H_{20}N]^+$: 154.2; Found: 154.2.

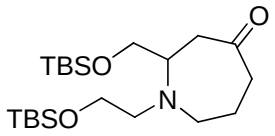
***N*-Benzyl-2-phenylazepan-4-one (6a)**



6a

Compound **6a** was prepared in 87 % yield according to the general procedure B. The reaction times are 1 h for the formation of N-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : NEt_3 = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, 2H, *J* = 9.0 Hz), 7.37 (t, 2H, *J* = 7.0 Hz), 7.20 – 7.30 (m, 6H), 4.00 (dd, 1H, *J* = 4.0, 9.5 Hz), 3.61 (d, 1H, *J* = 13.5 Hz), 3.34 (d, 1H, *J* = 14.0 Hz), 3.09 – 3.36 (m, 2H), 2.77 (dd, 1H, *J* = 4.5, 14.0 Hz), 2.51 – 2.60 (m, 3H), 1.97 – 2.01 (m, 1H), 1.85 – 1.90 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 211.9, 142.4, 139.5, 128.6, 128.4, 128.3, 128.2, 127.4, 126.9, 64.2, 57.1, 50.9, 49.0, 42.4, 22.2 ; IR (neat): 3084, 3061, 3027, 2935, 2810, 1705, 1494, 1451; MS (ES^+) Calculated for $[C_{19}H_{21}NNaO]^+$: 302.2; Found: 302.2.

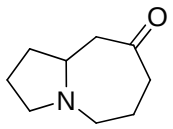
***N*-[2-(tert-Butyldimethylsilanyloxy)ethyl]-2-(tert-butyldimethylsilanyloxymethyl)azepan-4-one (6b)**



6b

Compounds **6b** were prepared in 51 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 8 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.70 (dd, 1H, *J* = 5.2, 9.6 Hz), 3.57 (t, 2H, *J* = 6.4 Hz), 3.47 – 3.51 (m, 1H), 3.17 – 3.26 (m, 2H), 2.84 – 2.91 (m, 1H), 2.57 – 2.72 (m, 5H), 2.41 – 2.47 (m, 1H), 1.84 – 1.93 (m, 1H), 1.59 (m, 1H), 0.88 (s, 18H), 0.04 (s, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.0, 64.9, 62.8, 60.0, 52.9, 51.5, 44.7, 42.9, 25.9, 25.8, 21.8, 18.3, 18.2, –5.3, –5.4, –5.5; IR (neat): 2953, 2929, 2857, 1705, 1646, 1471; MS (ES^+) Calculated for $[C_{21}H_{46}NO_3Si_2]^+$: 416.3; Found: 416.3.

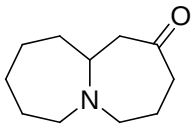
Hexahydropyrrolo[1,2-a]azepin-8-one (6c)



6c

Compounds **6c** were prepared in 80 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: ethyl acetate : methanol : NH_4OH = 5 : 1 : 0.06). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.28 – 3.33 (m, 1H), 3.17 – 3.22 (m, 1H), 2.26 – 2.71 (m, 7H), 1.83 – 2.06 (m, 4H), 1.68 – 1.75 (m, 1H), 1.55 – 1.61 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.2, 60.4, 57.3, 56.6, 50.0, 43.2, 32.8, 24.3, 21.8; IR (neat): 2930, 2853, 1708, 1556, 1376, 1352; MS (ES^+) Calculated for $[C_9H_{16}NO]^+$: 154.1; Found: 153.8.

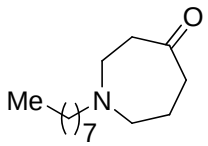
Octahydroazepino[1,2-a]azepin-2-one (6d)



6d

Compounds **6d** were prepared in 89 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate: Et_3N = 1 : 1 : 0.02). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.09 – 3.15 (m, 1H), 2.97 – 3.03 (m, 1H), 2.71 – 2.92 (m, 3H), 2.48 – 2.62 (m, 3H), 2.36 – 2.43 (m, 1H), 1.70 – 1.84 (m, 4H), 1.55 – 1.62 (m, 1H), 1.24 – 1.48 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.7, 59.9, 56.2, 53.0, 51.7, 42.1, 34.6, 28.5, 28.3, 26.8, 24.9 ; IR (neat): 2923, 2850, 1703, 1446, 1404; MS (ES^+) Calculated for $[C_{11}H_{20}NO]^+$: 182.2; Found: 182.2.

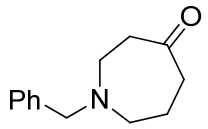
***N*-Octylazepan-4-one (6e)**



6e

Compounds **6e** were prepared in 69 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 2 : 1 : 0.03). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.71 – 2.75 (m, 4H), 2.60 – 2.63 (m, 2H), 2.50 – 2.53 (m, 2H), 2.46 (t, 2H, *J* = 7.2 Hz), 1.79 – 1.84 (m, 2H), 1.43 – 1.47 (m, 2H), 1.24 – 1.30 (m, 10H), 0.87 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.6, 58.1, 57.9, 50.6, 44.2, 42.9, 31.8, 29.5, 29.2, 27.4, 27.3, 23.9, 22.6, 14.1; IR (neat): 2929, 2856, 1702, 1467; MS (ES^+) Calculated for $[C_{14}H_{28}NO]^+$: 226.2; Found: 226.2.

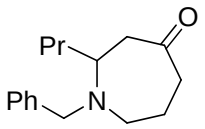
***N*-Benzylazepan-4-one (6f)**



6f

Compounds **6f** were prepared in 85 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 5 : 1 : 0.06). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.33 (m, 5H), 3.65 (s, 2H), 2.71 – 2.76 (m, 4H), 2.59 – 2.62 (m, 2H), 2.52 – 2.55 (m, 2H), 1.82 – 1.88 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.5, 138.9, 128.7, 128.3, 127.1, 62.7, 57.9, 50.4, 44.3, 42.9, 24.1; IR (neat): 2939, 2813, 1702, 1453, 1351; MS (ES^+) Calculated for $[C_{13}H_{18}NO]^+$: 204.1; Found: 204.1.

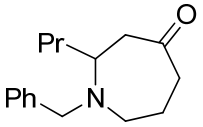
***N*-Benzyl-2-propylazepan-4-one (6g)**



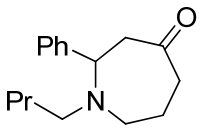
6g

Compounds **6g and 6g'** were prepared in 73 % combined yield (ratio: **6g** : **6g'** = 2 : 1) according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 6 h for Au catalysis. PEt_3AuNTf_2 was used as Au catalyst. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate : NEt_3 = 10 : 1 : 0.11) to afford some **6g** pure along with a mixture of **6g** and **6g'**. **6g**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.33 (m, 5H), 3.78 (d, 1H, *J* = 13.6 Hz), 3.58 (d, 1H, *J* = 13.6 Hz), 3.15 – 3.18 (m, 1H), 2.96 (dt, 1H, *J* = 4.0, 14.0 Hz), 2.71 – 2.78 (m, 2H), 2.63 – 2.55 (m, 2H), 2.37 – 2.44 (m, 1H), 1.79 – 1.88 (m, 1H), 1.59 – 1.74 (m, 1H), 1.35 – 1.43 (m, 4H), 0.91 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.1, 139.8, 128.4, 128.3, 126.9, 57.6, 53.9, 50.1, 47.6, 42.6, 33.7, 20.9, 19.9, 14.0; IR (neat): 2955, 2931, 1698, 1455; MS (ES^+) Calculated for $[C_{16}H_{24}NO]^+$: 246.2; Found: 246.2.

***N*-Benzyl-2-propylazepan-4-one (6g)** and ***N*-butyl-2-phenylazepan-4-one (6g')**



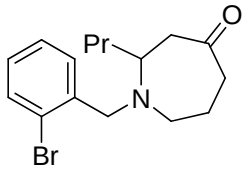
6g



6g'

A mixture of **6g and 6g'**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18- 7.31 (m, 8.3 H), 3.89 (dd, 0.86 H, *J* = 10.4, 4.4 Hz), 3.73 (d, 1H, *J* = 13.6 Hz), 3.53 (d, 1H, *J* = 13.6 Hz), 3.19 (ddd, 1.03 H, *J* = 3.6, 10.4, 14.4 Hz), 3.10- 3.13 (m, 1.03 H), 3.01 (dd, 0.99 H, *J* = 10.0, 14.4 Hz), 2.92 (m, 1.06 H), 2.59 –2.73 (m, 4.13 H), 2.44 – 2.56 (m, 4.24 H), 2.31– 2.39 (m, 2.28 H), 2.21– 2.27 (m, 1H), 1.75 – 1.98 (m, 3.38 H), 1.67 – 1.74 (m, 1.23 H), 1.55 – 1.59 (m,1.03 H), 1.26 – 1.38 (m, 5.25 H), 1.09 – 1.41 (m, 2.21 H), 0.87 (t, 2.93 H, *J* = 7.2 Hz), 0.73 (t, 2.49 H, *J* = 7.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.1, 212.2, 142.8, 139.8, 128.4, 128.4.128.3, 127.2, 127.0, 126.9, 63.7, 57.6, 53.9, 53.4, 51.9, 51.0, 50.1, 49.1, 47.6, 42.7, 42.6, 33.7, 29.5, 22.5, 20.9, 20.2, 19.9, 14.0, 13.9; IR (neat): 2955, 2930, 1698, 1455; MS (ES^+) Calculated for $[C_{16}H_{24}NO]^+$: 246.2; Found: 246.2.

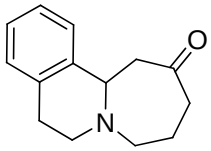
_N_-(2-Bromobenzyl)-2-propylazepan-4-one (6h)



6h

Compounds **6h** were prepared in 71 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 8 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate: NEt_3 = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (dd, 1H, *J* = 7.6, 1.2 Hz), 7.45 (dd, 1H, *J* = 2.5, 9.5 Hz), 7.28 (td, 1H, *J* = 7.2, 1.2 Hz), 7.09 (td, 1H, *J* = 1.4, 8.4 Hz), 3.78 (s, 2H), 3.07 – 3.12 (m, 1H), 2.96 – 3.02 (m, 1H), 2.76 – 2.82 (m, 2H), 2.56 – 2.58 (m, 2H), 2.37 – 2.44 (m, 1H), 1.79 – 1.90 (m, 2H), 1.59 – 1.64 (m, 1H), 1.32 – 1.45 (m, 3H), 0.89 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.8, 138.6, 132.6, 130.3, 128.2, 127.2, 124.1, 57.7, 54.4, 50.1, 47.4, 42.5, 33.4, 21.4, 19.8, 14.0; IR (neat): 2954, 2860, 2804, 1699, 1566, 1457, 1437, 1363; MS (ES^+) Calculated for $[C_{16}H_{23}BrNO]^+$: 324.1; Found: 324.1.

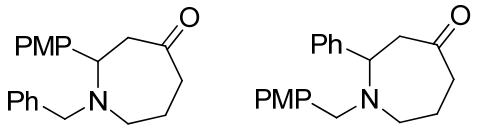
1,4,5,7,8,12b-Hexahydro-3H-azepino[2,1-a]isoquinolin-2-one (6i)



6i

Compounds **6i** were prepared in 70 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 8 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate: Et_3N = 1 : 1 : 0.02). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07- 7.18 (m, 4H), 4.00 (dd, 1H, *J* = 2.8, 9.6 Hz), 3.18 – 3.23 (m, 1H), 3.07 – 3.11 (m, 1H), 2.93 – 3.00 (m, 2H), 2.58 – 2.86 (m, 6H), 1.84 – 2.04 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 211.8, 138.0, 134.8, 128.5, 126.9, 126.1, 59.8, 59.0, 53.5, 50.5, 42.2, 29.8, 23.0 ; IR (neat): 2935, 2817, 1698, 1493, 1452, 1363; MS (ES^+) Calculated for $[C_{14}H_{18}NO]^+$: 216.1; Found: 216.1.

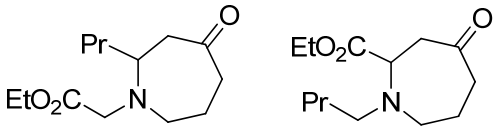
***N*-Benzyl-2-(4-methoxyphenyl)azepan-4-one (6j)** and ***N*-(4-methoxybenzyl)-2-phenylazepan-4-one (6j')**



6j **6j'**

Compounds **6j and 6j'** were prepared in 63 % combined yield (ratio = 1.3 : 1) according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes: ethyl acetate : Et_3N = 10 : 1 : 0.11). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, 1.14 H, *J* = 6.8 Hz), 7.36 (t, 1.89 H, *J* = 7.2 Hz), 7.19 – 7.34 (m, 2.90 H), 7.14 (d, 1.09 H, *J* = 8.8 Hz), 6.89 (d, 0.70 H, *J* = 8.8 Hz), 6.82 (d, 1.06 H, *J* = 9.2 Hz), 3.95 – 4.00 (m, 0.95 H), 3.80 (s, 1.22 H), 3.78 (s, 1.64 H), 3.52 – 3.61 (m, 1.0 H), 3.27 – 3.35 (m, 0.97 H), 3.06 – 3.19 (m, 1.93 H), 2.76 (dd, 0.96 H, *J* = 4.4, 13.6 Hz), 2.49 – 2.59 (m, 2.95 H), 1.96 – 2.02 (m, 1.05 H), 1.84 – 1.90 (m, 1.08 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.2, 212.1, 158.8, 158.6, 142.4, 139.6, 134.3, 131.4, 129.5, 128.6, 128.4, 128.3, 127.4, 127.3, 126.8, 113.9, 113.6, 64.0, 63.4, 56.8, 56.4, 55.2, 55.2, 50.7, 50.5, 49.0, 48.9, 42.4, 42.3, 22.2; IR (neat): 2930, 1701, 1698, 1646, 1455; MS (ES^+) Calculated for $[C_{20}H_{24}NO_2]^+$: 310.2; Found: 310.2.

Ethyl (4-oxo-2-propylazepan-1-yl)acetate (6k) and **ethyl 1-butyl-4-oxoazepane-2-carboxylate (6k')**



6k **6k'**

Compounds **6k and 6k'** were prepared in 93 % combined yield (ratio: **6j** : **6j'** = 5 : 1) according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. PEt_3AuNTf_2 was used as Au catalyst. These two products

were separated through silica gel flash chromatography (eluents: hexanes: ethyl acetate : Et_3N = 5 : 1 : 0.06).

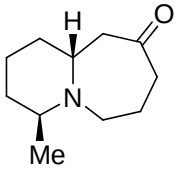
Compound **6k**:

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.15 (dd, 2H, *J* = 6.8, 14.0 Hz), 7.38 (dd, 2H, *J* = 16.8, 32.8 Hz), 3.08 – 3.18 (m, 2H), 2.91 – 2.98 (m, 1H), 2.59 – 2.72 (m, 2H), 2.39 – 2.53 (m, 2H), 1.72 – 1.82 (m, 2H), 1.46 – 1.52 (m, 1H), 1.32 – 1.39 (m, 3H), 1.26 (t, 3H, *J* = 8.5 Hz), 0.90 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.5, 171.7, 60.6, 58.0, 52.6, 51.6, 47.8, 42.8, 34.8, 21.6, 19.7, 14.2, 14.0; IR (neat): 2934, 1744, 1698, 1456; MS (ES^+) Calculated for $[C_{13}H_{24}NO_3]^+$: 242.2; Found: 242.2.

Compound **6k'**:

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.13 – 4.24 (m, 2H), 3.74 (t, 1H, *J* = 8.0 Hz), 3.03 –3.09 (m, 2H), 2.79 – 2.86 (m, 1H), 2.71 – 2.74 (m, 1H), 2.56 – 2.66 (m, 2H), 2.42 – 2.51 (m, 2H), 1.71- 1.98 (m, 2H), 1.38- 1.45 (m, 2H), 1.23- 1.36 (m, 5H), 0.88 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 210.7, 171.8, 60.7, 60.4, 54.8, 50.7, 44.3, 42.3, 30.4, 23.2, 20.2, 14.4, 13.9; IR (neat): 2957, 1725, 1540, 1456, 1363; MS (ES^+) Calculated for $[C_{13}H_{24}NO_3]^+$: 242.2; Found: 242.2.

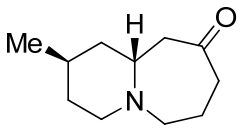
(4*S,10a*R**)-4-Methyloctahydropyrido[1,2-a]azepin-9-one (6l)**



6l

Compounds **6l** were prepared in 74 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 4 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: ethyl acetate : methanol : NH_4OH = 5 : 1 : 0.06). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.16 (q, 1H, *J* = 6.8 Hz), 2.97-3.06 (m, 3H), 2.39 – 2.62 (m, 3H), 2.26 – 2.32 (m, 1H), 1.91 – 2.02 (m, 1H), 1.34 – 1.77 (m, 7H), 1.13 (d, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.0, 54.2, 54.1, 52.4, 48.7, 42.9, 33.8, 33.1, 21.2, 19.0, 14.6 ; IR (neat): 2931, 1704, 1446, 1372, 1347; MS (ES^+) Calculated for $[C_{11}H_{20}NO]^+$: 182.2; Found: 182.0.

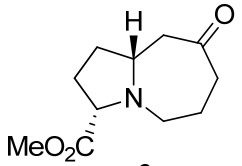
(2*R,10a*R**)-2-Methyloctahydropyrido[1,2-a]azepin-9-one (6m)**



6m

Compounds **6m** were prepared in 76 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 4 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: ethyl acetate : methanol : NH_4OH = 5 : 1 : 0.06). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.08 – 3.15 (m, 2H), 2.84 – 2.90 (m, 1H), 2.79 – 2.83 (m, 1H), 2.41 – 2.65 (m, 4H), 2.18 (d, 1H, *J* = 12.0 Hz), 2.01 – 2.06 (m, 1H), 1.67 – 1.79 (m, 3H), 1.56 – 1.61 (m, 1H), 1.56 (m, 1H), 1.42 – 1.47 (m, 1H), 1.29 – 1.34 (m, 1H), 0.94 (d, 3H, *J* = 6.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.0, 58.8, 54.9, 48.2, 48.0, 42.7, 41.0, 32.9, 25.1, 20.7, 19.8; IR (neat): 2925, 1701, 1457, 1354; MS (ES^+) Calculated for $[C_{11}H_{20}NO]^+$: 182.2; Found: 181.9.

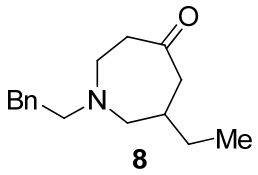
Methyl (3*S, 9*aR**)-8-oxooctahydropyrrolo[1,2-a]azepine-3-carboxylate (6n)**



6n

Compounds **6n** were prepared in 71 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. PEt_3AuNTf_2 was used as Au catalyst. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate: Et_3N = 5 : 1 : 0.06). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.86 (dd, 1H, *J* = 8.0, 1.6 Hz), 3.68 (s, 3H), 3.08 – 3.44 (m, 1H), 3.05 (dt, 1H, *J* = 12.4, 4.0 Hz), 2.73 – 2.80 (m, 1H), 2.47 – 2.62 (m, 4H), 2.27 – 2.34 (m, 1H), 2.08 – 2.18 (m, 1H), 1.78 – 1.86 (m, 3H), 1.51 – 1.57 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.6, 174.3, 67.5, 57.1, 51.9, 51.3, 51.2, 43.0, 31.6, 27.3, 25.3; IR (neat): 2949, 1731, 1702, 1435, 1327, 1193, 1160; MS (ES^+) Calculated for $[C_{11}H_{17}NO_3+H]^+$: 212.1; Found: 212.1.

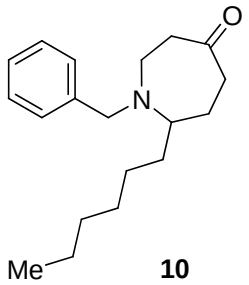
6-Ethyl-1-phenethylazepan-4-one (8)



8

Compounds **8** were prepared in 81 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis (PEt_3AuNTf_2 as catalyst). The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 – 7.32 (m, 5H), 3.68 (d, 1H, *J* =13.5 Hz), 3.63 (d, 1H, *J* = 13.5 Hz), 2.82 – 2.87 (m, 2H), 2.64 – 2.73 (m, 2H), 2.52 (dd, 1H, *J* = 14.5, 3.0 Hz), 2.40 – 2.46 (m, 3H), 1.84 – 1.87 (m, 1H), 1.26 – 1.35 (m, 2H), 0.87 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.7, 139.0, 128.6, 128.3, 127.0, 63.4, 62.9, 50.7, 48.5, 44.2, 36.9, 27.1, 11.5; IR (neat): 2953, 2930, 2818, 1702, 1455, 1027; MS (ES^+) Calculated for $[C_{15}H_{22}NO]^+$: 232.2; Found: 232.2.

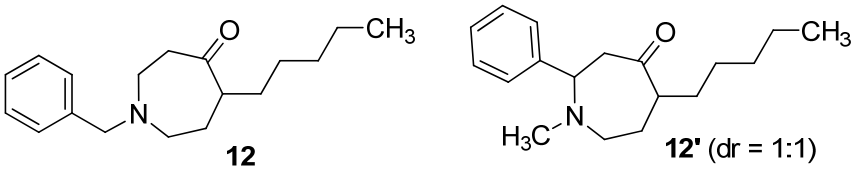
_N_-Benzyl-7-hexylazepan-4-one (10)



10

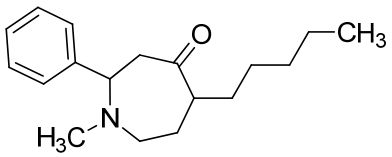
Compounds **10** were prepared in 73 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 3 h for Au catalysis. The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 10 : 1 : 0.11). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22 – 7.34 (m, 5H), 3.77 (d, 1H, *J* =14.0 Hz), 3.66 (d, 1H, *J* =14.0 Hz), 3.02 – 3.06 (m, 1H), 2.86 – 2.90 (m, 1H), 2.73 – 2.79 (m, 1H), 2.46 – 2.60 (m, 4H), 1.94 – 2.00 (m, 1H), 1.63 – 1.73 (m, 2H), 1.30- 1.43 (m, 9H), 0.89 (t, 3H, *J* = 6.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.4, 139.5, 128.4, 128.3, 126.9, 61.6, 55.1, 43.4, 41.8, 40.1, 31.8, 30.2, 29.4, 26.7, 25.5, 22.6, 14.0; IR (neat): 2956, 2930, 2857, 1697, 1455, 1377; MS (ES^+) Calculated for $[C_{19}H_{30}NO]^+$: 288.2; Found: 288.2.

1-Benzyl-5-pentylazepan-4-one (12) and **1-methyl-5-pentyl-2-phenylazepan-4-one (12')**



Compounds **12** and **12'** were prepared in a combined 71 % yield according to the general procedure B. The reaction time is 1 h for the formation of *N*-oxide and 2 h for Au catalysis. The ratio of **12** and **12'** is 1.7/1, and no diastereoselectivity for compound **12'** was observed (dr = 1:1)**.** The residue was purified through silica gel flash chromatography (eluents: hexanes : ethyl acetate : Et_3N = 10 : 1 : 0.11). Compound **12**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.34 (m, 5H), 3.64 (d, 1H, *J* =11.2 Hz), 3.62 (d, 1H, *J* =11.2 Hz), 2.38 – 2.95 (m, 7H), 1.63 – 1.82 (m, 3H), 1.26 – 1.34 (m, 7H), 0.89 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 214.3, 138.9, 128.6, 128.3, 127.0, 68.2, 62.5, 56.2, 51.3, 50.7, 43.4, 31.9, 31.5, 30.6, 26.8, 22.5, 14.0; IR (neat): 2931, 1698, 1465, 1397; MS (ES^+) Calculated for $[C_{18}H_{27}NO+H]^+$: 274.2; Found: 274.2.

1-Methyl-5-pentyl-2-phenylazepan-4-one (12')



12' (one diastereomer)

One of the diastereomers of compound **12'** was isolated pure. While its relative stereochemistry was not determined, its spectra data are shown as following : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.33 (m, 5H), 3.38 (dd, 1H, *J* = 3.6, 10.4 Hz), 3.05 (m, 2H), 2.41– 2.58 (m, 3H), 2.24 – 2.32 (m, 1H), 2.03 – 2.15 (m, 3H), 1.79 – 1.92 (m, 2H), 1.20 – 1.39 (m, 7H), 0.89 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.2, 143.6, 128.5, 127.2, 127.1, 68.2, 67.8, 53.4, 50.8, 50.4, 44.5, 31.9, 29.5, 27.9, 27.0, 22.5, 14.0; IR (neat): 2931, 1698, 1465, 1397; MS (ES^+) Calculated for $[C_{18}H_{27}NO+H]^+$: 274.2; Found: 274.2.

X-Ray crystal data and structure for azepan-4-one 6a

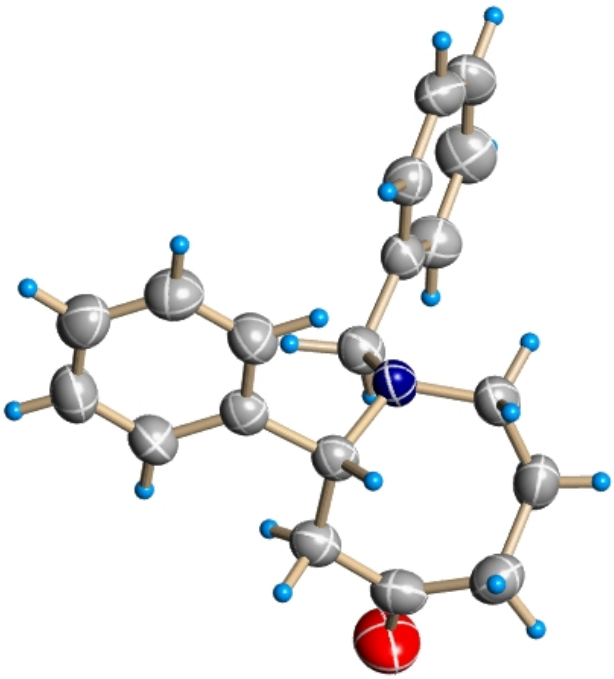


Table 1. Crystal data and structure refinement for 6a.

Identification code	6a
Empirical formula	C38 H42 N2 O2
Formula weight	558.74
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 5.7366(19) A alpha = 109.154(5) deg. b = 10.508(3) A beta = 91.003(5) deg. c = 13.624(4) A gamma = 102.607(5) deg.
Volume	753.5(4) A^3
Z, Calculated density	1, 1.231 Mg/m^3
Absorption coefficient	0.075 mm^-1
F(000)	300
Crystal size	0.3 x 0.1 x 0.1 mm
Theta range for data collection	1.59 to 26.41 deg.
Limiting indices	-7<=h<=7, -13<=k<=12, -17<=l<=17
Reflections collected / unique	6116 / 2958 [R(int) = 0.0596]
Completeness to theta = 26.41	95.5 %
Absorption correction	Empirical
Max. and min. transmission	0.554 and 0.320
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2958 / 0 / 274
Goodness-of-fit on F^2	0.914
Final R indices [I>2sigma(I)]	R1 = 0.0498, wR2 = 0.1030
R indices (all data)	R1 = 0.0865, wR2 = 0.1170
Largest diff. peak and hole	0.153 and -0.276 e.A^-3

♀

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 6a.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

x	y	z	U(eq)

C(1)	1735(3)	8214(2)	1570(1)	41(1)
C(2)	238(3)	9488(2)	3183(1)	38(1)
C(3)	2737(3)	10349(2)	3683(2)	44(1)
C(4)	3837(4)	11545(2)	3333(1)	50(1)
C(5)	2284(4)	12150(2)	2801(2)	54(1)
C(6)	1545(4)	11209(2)	1667(2)	53(1)
C(7)	-490(4)	9954(2)	1552(2)	47(1)
C(8)	-630(3)	8269(2)	3568(1)	39(1)
C(9)	737(4)	7906(2)	4237(1)	46(1)
C(10)	-182(4)	6776(2)	4542(2)	55(1)
C(11)	-2470(4)	6000(2)	4209(2)	55(1)
C(12)	-3869(4)	6358(2)	3553(2)	57(1)
C(13)	-2949(3)	7469(2)	3233(2)	47(1)
C(14)	1033(3)	7323(2)	438(1)	38(1)
C(15)	2708(4)	7291(2)	-280(2)	51(1)
C(16)	2123(4)	6409(2)	-1306(2)	60(1)
C(17)	-119(4)	5564(2)	-1621(2)	55(1)
C(18)	-1819(4)	5603(2)	-913(2)	50(1)
C(19)	-1233(3)	6477(2)	108(1)	43(1)
N	-51(2)	8960(1)	2032(1)	39(1)
O	5992(3)	11995(2)	3470(1)	81(1)

♀

Table 3. Bond lengths [A] and angles [deg] for 6a.

C(1)-N	1.453(2)
C(1)-C(14)	1.510(2)
C(1)-H(1A)	1.003(17)
C(1)-H(1B)	1.010(18)
C(2)-N	1.474(2)
C(2)-C(3)	1.527(2)
C(2)-C(8)	1.528(2)
C(2)-H(2)	1.019(16)
C(3)-C(4)	1.503(3)
C(3)-H(3A)	0.987(19)
C(3)-H(3B)	0.98(2)
C(4)-O	1.211(2)
C(4)-C(5)	1.501(3)
C(5)-C(6)	1.528(3)
C(5)-H(5A)	0.976(19)
C(5)-H(5B)	0.980(19)
C(6)-C(7)	1.521(3)
C(6)-H(6A)	0.91(2)
C(6)-H(6B)	1.02(2)
C(7)-N	1.463(2)
C(7)-H(7A)	0.981(18)
C(7)-H(7B)	1.024(19)
C(8)-C(9)	1.387(2)
C(8)-C(13)	1.387(2)
C(9)-C(10)	1.386(3)
C(9)-H(9)	0.960(18)
C(10)-C(11)	1.365(3)
C(10)-H(10)	0.92(2)
C(11)-C(12)	1.383(3)
C(11)-H(11)	1.001(18)
C(12)-C(13)	1.380(3)
C(12)-H(12)	0.95(2)
C(13)-H(13)	1.020(19)
C(14)-C(19)	1.377(2)
C(14)-C(15)	1.381(2)
C(15)-C(16)	1.386(3)
C(15)-H(15)	0.983(19)

C(16)-C(17)	1.363(3)
C(16)-H(16)	0.99(2)
C(17)-C(18)	1.380(3)
C(17)-H(17)	1.00(2)
C(18)-C(19)	1.380(3)
C(18)-H(18)	0.981(17)
C(19)-H(19)	0.999(18)

N-C(1)-C(14)	113.26(14)
N-C(1)-H(1A)	114.6(10)
C(14)-C(1)-H(1A)	107.3(10)
N-C(1)-H(1B)	106.4(10)
C(14)-C(1)-H(1B)	109.2(9)
H(1A)-C(1)-H(1B)	105.8(13)
N-C(2)-C(3)	115.49(14)
N-C(2)-C(8)	108.43(13)
C(3)-C(2)-C(8)	112.55(15)
N-C(2)-H(2)	104.2(8)
C(3)-C(2)-H(2)	108.2(8)
C(8)-C(2)-H(2)	107.4(8)
C(4)-C(3)-C(2)	117.66(16)
C(4)-C(3)-H(3A)	105.7(10)
C(2)-C(3)-H(3A)	108.9(11)
C(4)-C(3)-H(3B)	107.8(10)
C(2)-C(3)-H(3B)	112.5(10)
H(3A)-C(3)-H(3B)	103.1(15)
O-C(4)-C(5)	120.47(18)
O-C(4)-C(3)	119.22(19)
C(5)-C(4)-C(3)	120.29(18)
C(4)-C(5)-C(6)	110.27(17)
C(4)-C(5)-H(5A)	106.4(11)
C(6)-C(5)-H(5A)	109.8(11)
C(4)-C(5)-H(5B)	110.8(10)
C(6)-C(5)-H(5B)	109.5(10)
H(5A)-C(5)-H(5B)	110.0(15)
C(7)-C(6)-C(5)	113.16(17)
C(7)-C(6)-H(6A)	105.8(12)
C(5)-C(6)-H(6A)	109.0(12)
C(7)-C(6)-H(6B)	111.0(11)
C(5)-C(6)-H(6B)	110.6(11)
H(6A)-C(6)-H(6B)	107.0(16)
N-C(7)-C(6)	117.60(17)
N-C(7)-H(7A)	107.6(10)
C(6)-C(7)-H(7A)	108.2(10)
N-C(7)-H(7B)	104.5(9)
C(6)-C(7)-H(7B)	110.5(9)
H(7A)-C(7)-H(7B)	108.1(14)
C(9)-C(8)-C(13)	117.39(17)
C(9)-C(8)-C(2)	124.54(16)
C(13)-C(8)-C(2)	118.06(16)
C(10)-C(9)-C(8)	120.93(19)
C(10)-C(9)-H(9)	119.3(11)
C(8)-C(9)-H(9)	119.7(11)
C(11)-C(10)-C(9)	121.0(2)
C(11)-C(10)-H(10)	119.2(11)
C(9)-C(10)-H(10)	119.7(11)
C(10)-C(11)-C(12)	118.8(2)
C(10)-C(11)-H(11)	122.5(10)
C(12)-C(11)-H(11)	118.6(10)
C(13)-C(12)-C(11)	120.4(2)
C(13)-C(12)-H(12)	121.6(12)
C(11)-C(12)-H(12)	118.1(12)
C(12)-C(13)-C(8)	121.47(19)

L1.TXT

C(12)-C(13)-H(13)	119.3(10)
C(8)-C(13)-H(13)	119.2(10)
C(19)-C(14)-C(15)	118.51(16)
C(19)-C(14)-C(1)	121.24(16)
C(15)-C(14)-C(1)	120.17(16)
C(14)-C(15)-C(16)	120.56(19)
C(14)-C(15)-H(15)	117.5(11)
C(16)-C(15)-H(15)	122.0(11)
C(17)-C(16)-C(15)	120.4(2)
C(17)-C(16)-H(16)	121.9(11)
C(15)-C(16)-H(16)	117.7(11)
C(16)-C(17)-C(18)	119.55(19)
C(16)-C(17)-H(17)	121.0(11)
C(18)-C(17)-H(17)	119.5(11)
C(19)-C(18)-C(17)	120.02(19)
C(19)-C(18)-H(18)	119.4(10)
C(17)-C(18)-H(18)	120.6(10)
C(14)-C(19)-C(18)	120.94(18)
C(14)-C(19)-H(19)	118.5(10)
C(18)-C(19)-H(19)	120.5(10)
C(1)-N-C(7)	114.60(14)
C(1)-N-C(2)	113.59(13)
C(7)-N-C(2)	114.83(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 6a.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	43(1)	45(1)	38(1)	12(1)	3(1)	16(1)
C(2)	41(1)	40(1)	33(1)	9(1)	4(1)	14(1)
C(3)	46(1)	45(1)	38(1)	8(1)	1(1)	12(1)
C(4)	52(1)	47(1)	41(1)	6(1)	3(1)	5(1)
C(5)	61(1)	41(1)	56(1)	15(1)	6(1)	9(1)
C(6)	74(2)	46(1)	47(1)	21(1)	7(1)	20(1)
C(7)	58(1)	46(1)	39(1)	12(1)	-1(1)	20(1)
C(8)	43(1)	41(1)	31(1)	8(1)	6(1)	15(1)
C(9)	46(1)	52(1)	41(1)	15(1)	0(1)	12(1)
C(10)	66(1)	62(1)	45(1)	24(1)	5(1)	23(1)
C(11)	65(1)	50(1)	52(1)	21(1)	12(1)	11(1)
C(12)	51(1)	54(1)	59(1)	17(1)	5(1)	5(1)
C(13)	45(1)	52(1)	46(1)	16(1)	0(1)	14(1)
C(14)	44(1)	36(1)	36(1)	11(1)	4(1)	16(1)
C(15)	48(1)	55(1)	48(1)	12(1)	8(1)	14(1)
C(16)	63(1)	69(1)	48(1)	14(1)	20(1)	25(1)
C(17)	75(2)	49(1)	38(1)	6(1)	2(1)	24(1)
C(18)	55(1)	38(1)	52(1)	10(1)	-4(1)	10(1)
C(19)	47(1)	41(1)	43(1)	14(1)	7(1)	13(1)
N	45(1)	40(1)	33(1)	10(1)	3(1)	17(1)
O	52(1)	93(1)	94(1)	42(1)	-5(1)	-7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic
displacement parameters (A^2 x 10^3) for 6a.

	x	y	z	U(eq)
H(2)	-940(30)	10111(16)	3384(11)	37(4)
H(9)	2370(30)	8420(18)	4469(13)	54(5)
H(10)	790(30)	6517(19)	4954(15)	57(6)
H(11)	-3180(30)	5186(19)	4424(13)	55(5)
H(12)	-5480(40)	5830(20)	3343(15)	71(6)
H(13)	-4010(30)	7724(18)	2755(14)	59(5)
H(15)	4310(40)	7919(19)	-36(15)	66(6)
H(16)	3380(30)	6430(19)	-1796(15)	67(6)
H(17)	-560(40)	4930(20)	-2361(16)	74(6)
H(18)	-3430(30)	4992(18)	-1125(13)	53(5)
H(19)	-2460(30)	6516(18)	624(14)	54(5)
H(1A)	3380(30)	8816(18)	1612(13)	49(5)
H(3A)	2680(30)	10753(19)	4443(16)	62(6)
H(5A)	3260(30)	13050(20)	2823(14)	60(6)
H(6A)	980(30)	11690(20)	1307(15)	63(6)
H(7A)	-950(30)	9447(18)	805(15)	52(5)
H(1B)	1910(30)	7603(18)	1991(13)	50(5)
H(3B)	3910(30)	9777(19)	3626(13)	52(5)
H(5B)	850(30)	12273(18)	3163(14)	55(5)
H(6B)	3000(40)	10910(20)	1316(15)	73(6)
H(7B)	-1960(30)	10260(18)	1879(13)	54(5)

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Table 6. Torsion angles [deg] for 6a.

N-C(2)-C(3)-C(4)	52.2(2)
C(8)-C(2)-C(3)-C(4)	177.50(15)
C(2)-C(3)-C(4)-O	-159.13(18)
C(2)-C(3)-C(4)-C(5)	19.5(3)
O-C(4)-C(5)-C(6)	101.9(2)
C(3)-C(4)-C(5)-C(6)	-76.7(2)
C(4)-C(5)-C(6)-C(7)	78.6(2)
C(5)-C(6)-C(7)-N	-62.5(2)
N-C(2)-C(8)-C(9)	124.50(17)
C(3)-C(2)-C(8)-C(9)	-4.5(2)
N-C(2)-C(8)-C(13)	-55.78(19)
C(3)-C(2)-C(8)-C(13)	175.20(16)
C(13)-C(8)-C(9)-C(10)	0.8(3)
C(2)-C(8)-C(9)-C(10)	-179.46(16)
C(8)-C(9)-C(10)-C(11)	-1.1(3)
C(9)-C(10)-C(11)-C(12)	0.2(3)
C(10)-C(11)-C(12)-C(13)	0.9(3)
C(11)-C(12)-C(13)-C(8)	-1.1(3)
C(9)-C(8)-C(13)-C(12)	0.2(3)
C(2)-C(8)-C(13)-C(12)	-179.51(16)
N-C(1)-C(14)-C(19)	-46.1(2)
N-C(1)-C(14)-C(15)	137.29(17)
C(19)-C(14)-C(15)-C(16)	-1.0(3)
C(1)-C(14)-C(15)-C(16)	175.77(17)
C(14)-C(15)-C(16)-C(17)	0.3(3)
C(15)-C(16)-C(17)-C(18)	0.7(3)
C(16)-C(17)-C(18)-C(19)	-1.0(3)
C(15)-C(14)-C(19)-C(18)	0.7(3)
C(1)-C(14)-C(19)-C(18)	-175.98(16)
C(17)-C(18)-C(19)-C(14)	0.2(3)
C(14)-C(1)-N-C(7)	-62.48(19)
C(14)-C(1)-N-C(2)	162.78(14)

C(6)-C(7)-N-C(1)	-66.2(2)
C(6)-C(7)-N-C(2)	67.9(2)
C(3)-C(2)-N-C(1)	52.4(2)
C(8)-C(2)-N-C(1)	-75.00(17)
C(3)-C(2)-N-C(7)	-82.3(2)
C(8)-C(2)-N-C(7)	150.37(15)

Symmetry transformations used to generate equivalent atoms:

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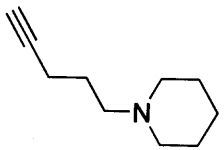
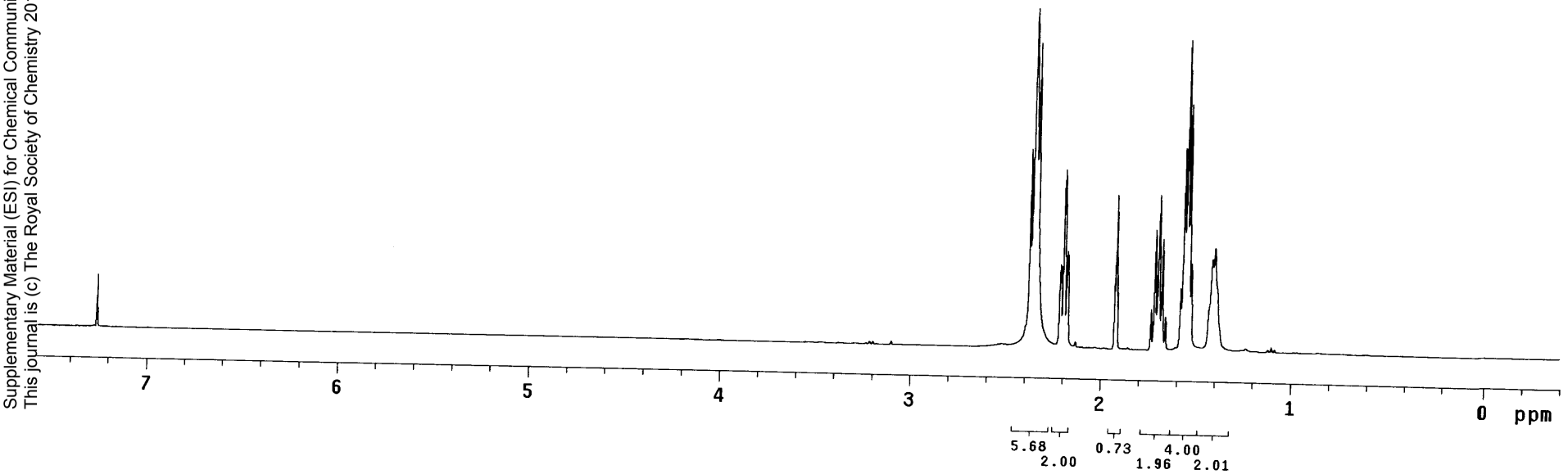
Z*SPEC IDG400-5FE
UCSB

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
20 repetitions
OBSERVE H1, 399.9486720 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec



1



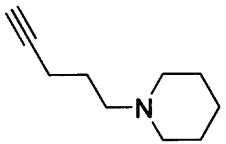
licui-6m3cSM

Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.17_27
File : exp
Sample id : tmpstudy

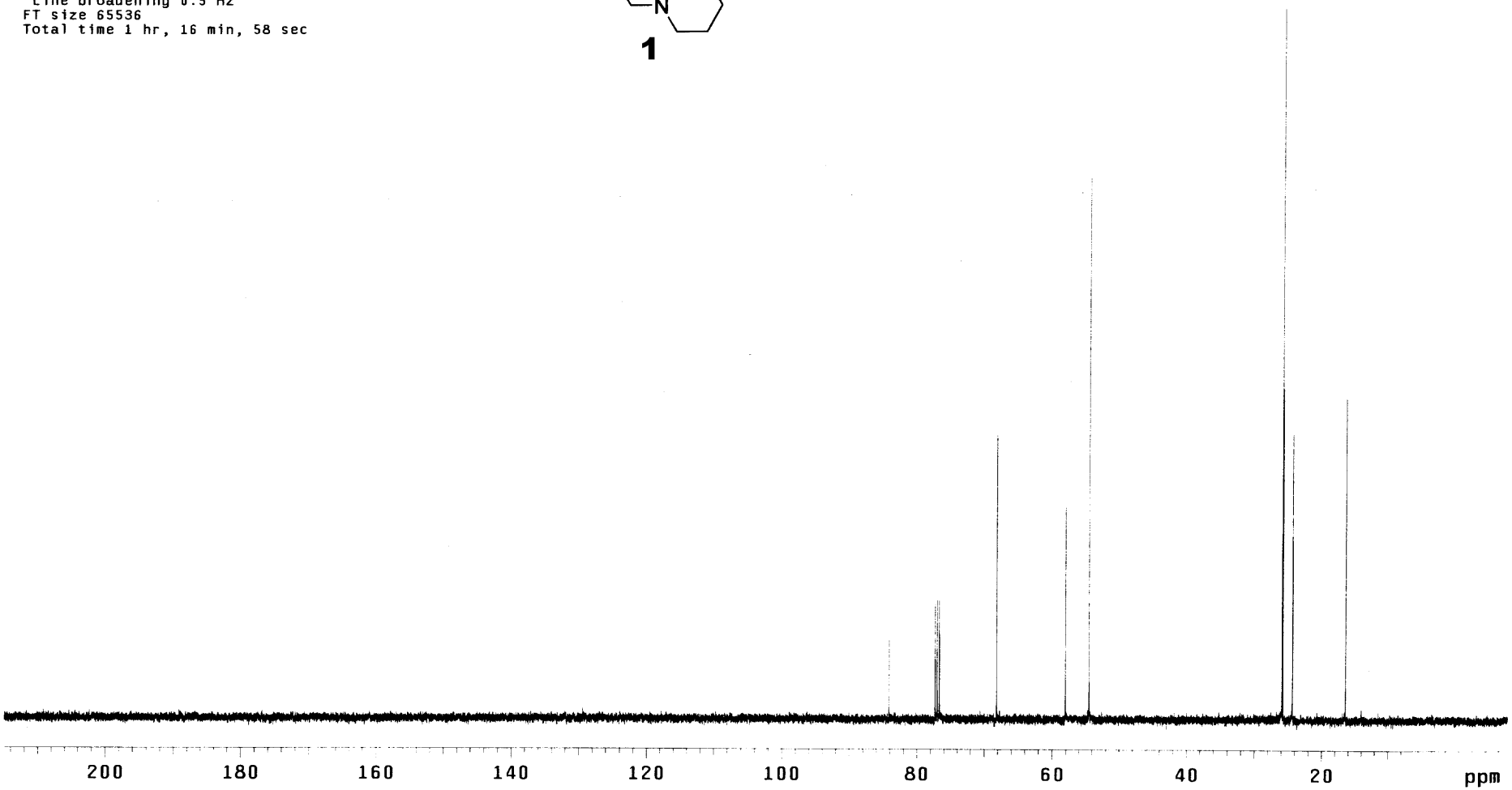
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-400 "mr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
16 repetitions
OBSERVE C13, 100.5943068 MHz
DECOUPLE H1, 400.0585543 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 58 sec



1



H1_CDCL3

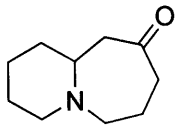
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: H1

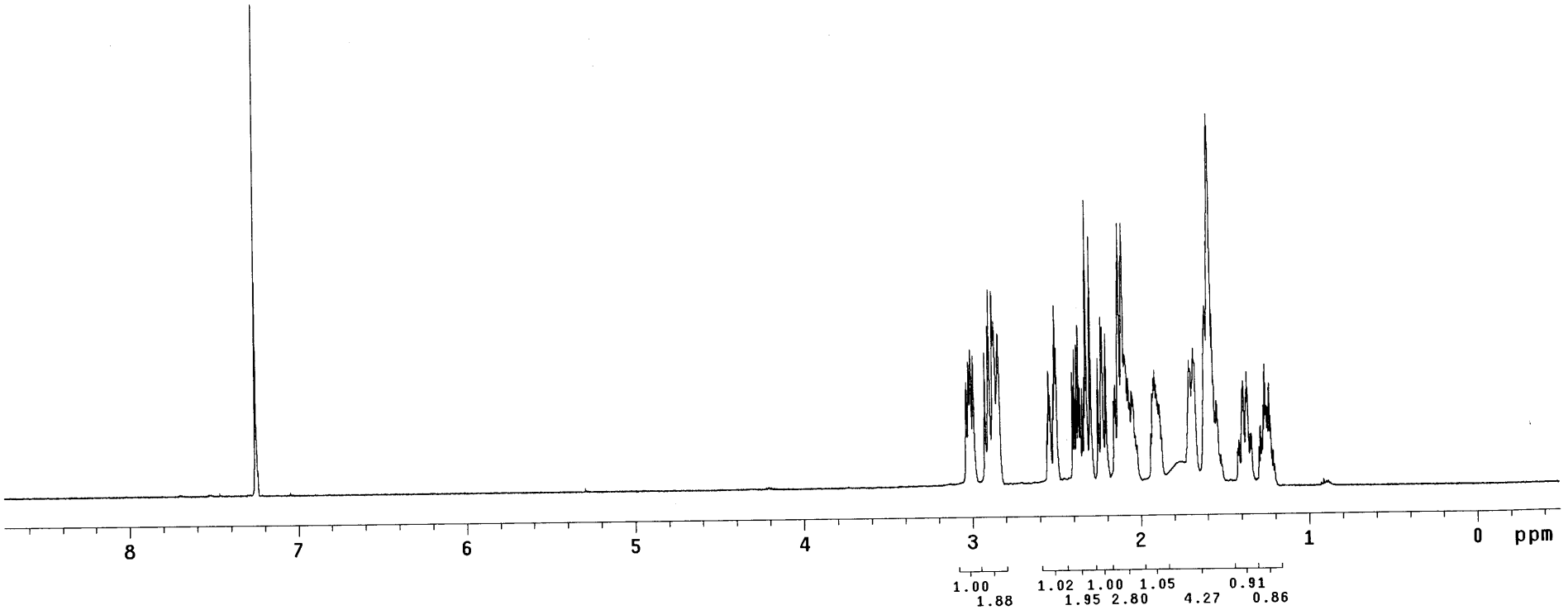
Pulse Sequence: s2pul
Solvent: cdcl3

Operator: lcui

Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
24 repetitions
OBSERVE H1, 499.8563625 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min



3

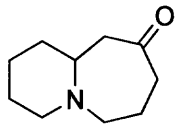


licui-59-2

Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.14_03
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
17752 repetitions
OBSERVE C13, 125.6732818 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec



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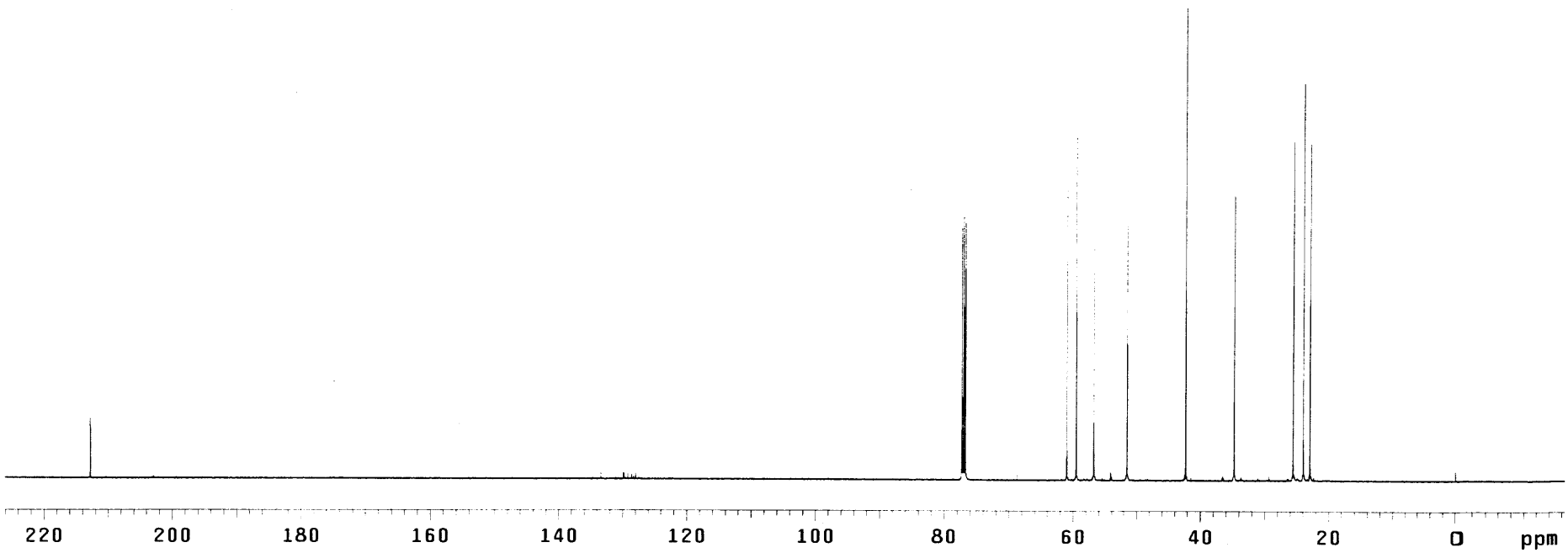


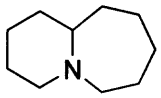
licui-61-2

Archive directory:
Sample directory:

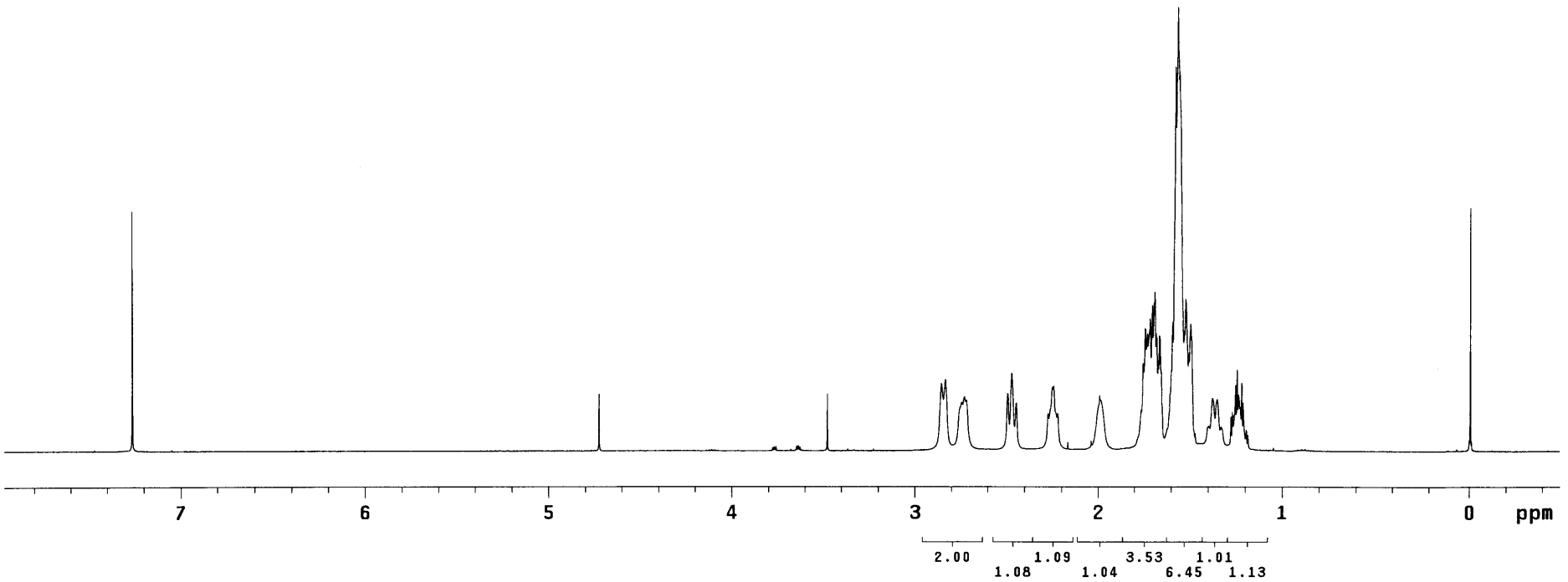
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: licui-61-2-1H
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
28 repetitions
OBSERVE H1, 499.7939123 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min, 37 sec



4



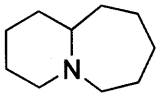
licui-61-2

Archive directory:
Sample directory:

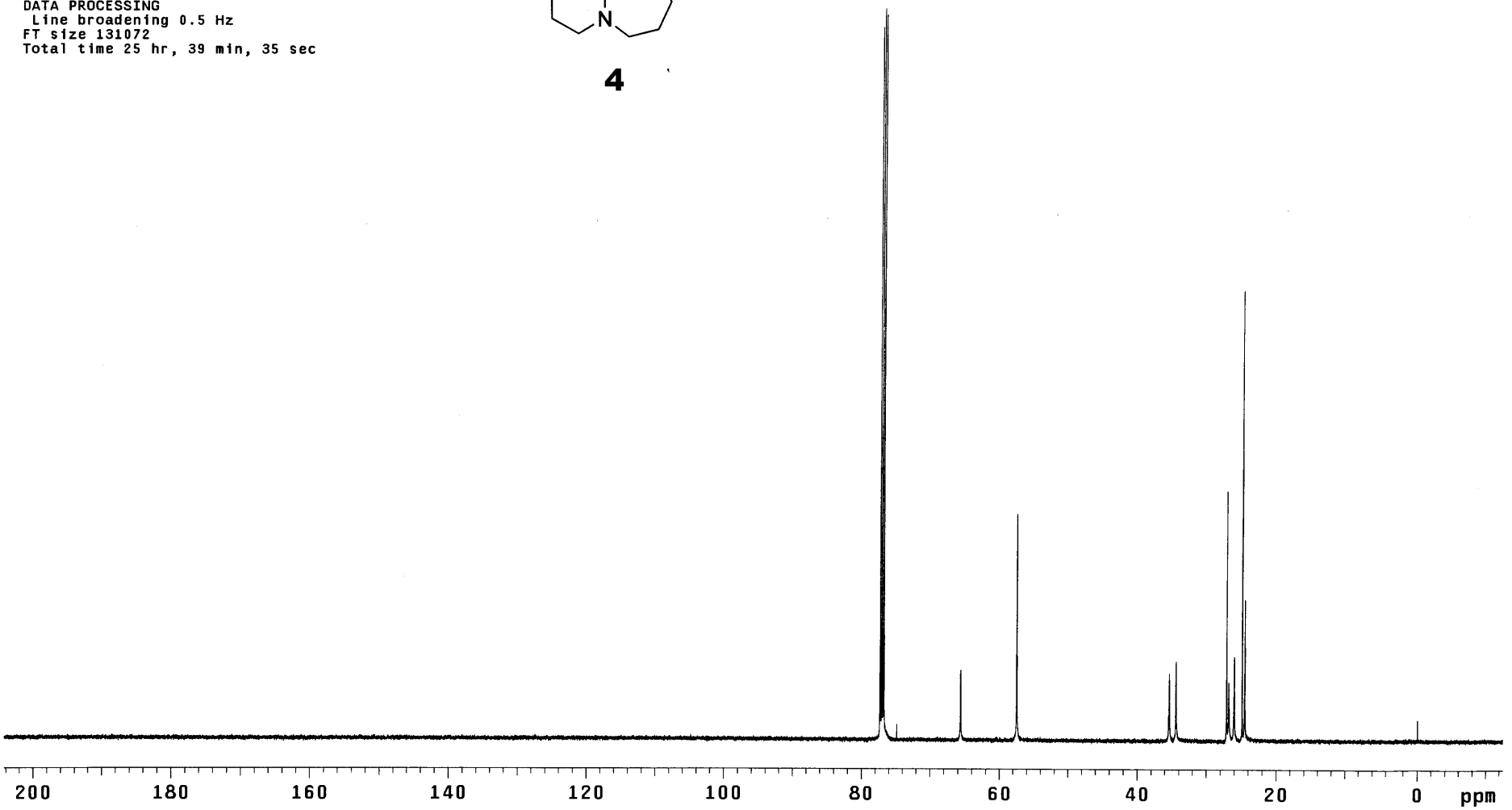
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: licui-61-2-13C
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
25200 repetitions
OBSERVE C13, 125.6732790 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 25 hr, 39 min, 35 sec



4

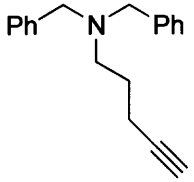


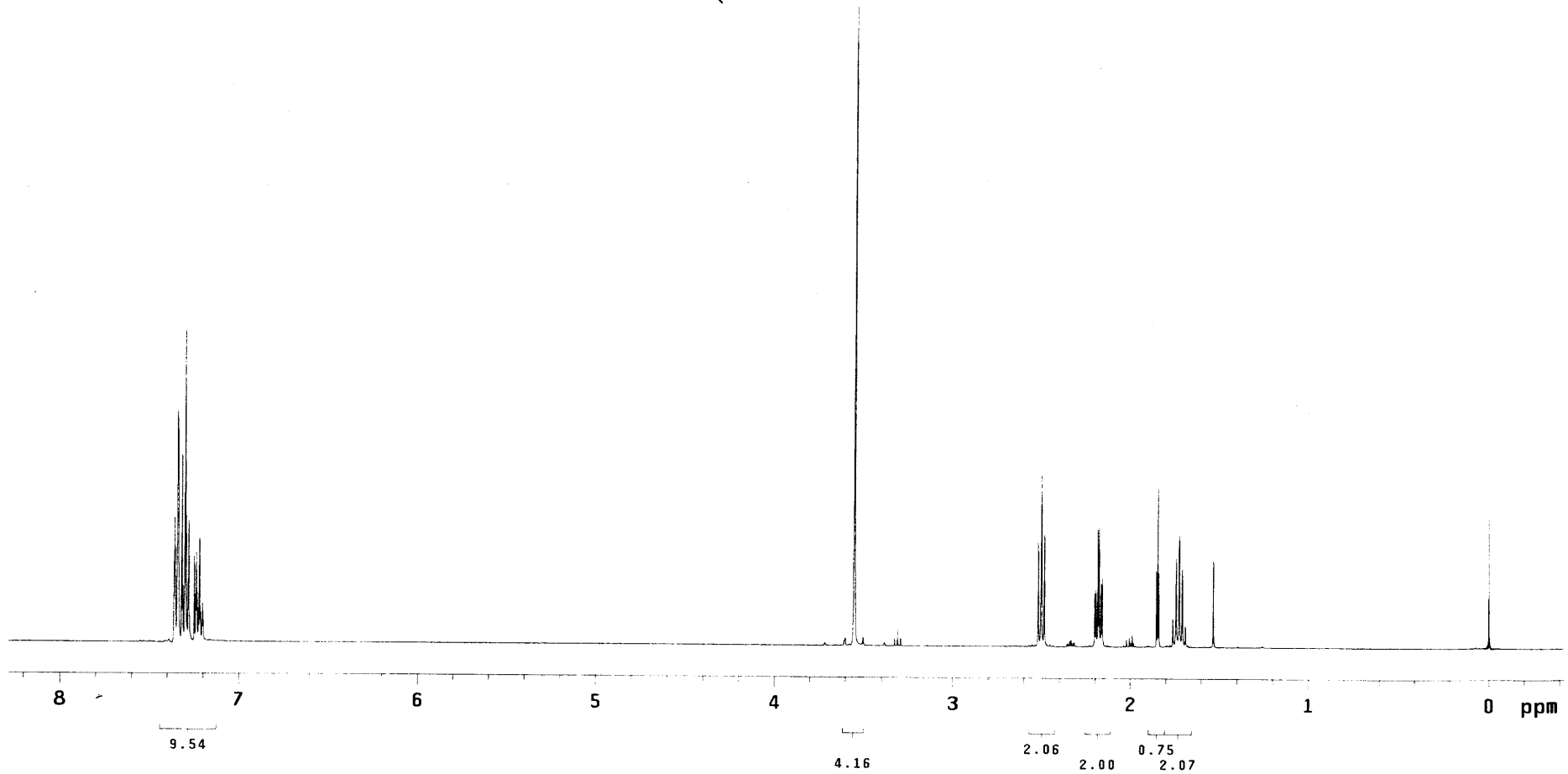
licui-62-1-1H

Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.19_24
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-400 "mr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
12 repetitions
OBSERVE H1, 400.0565536 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 6 min, 31 sec





liuci-106-1-13C

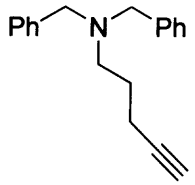
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

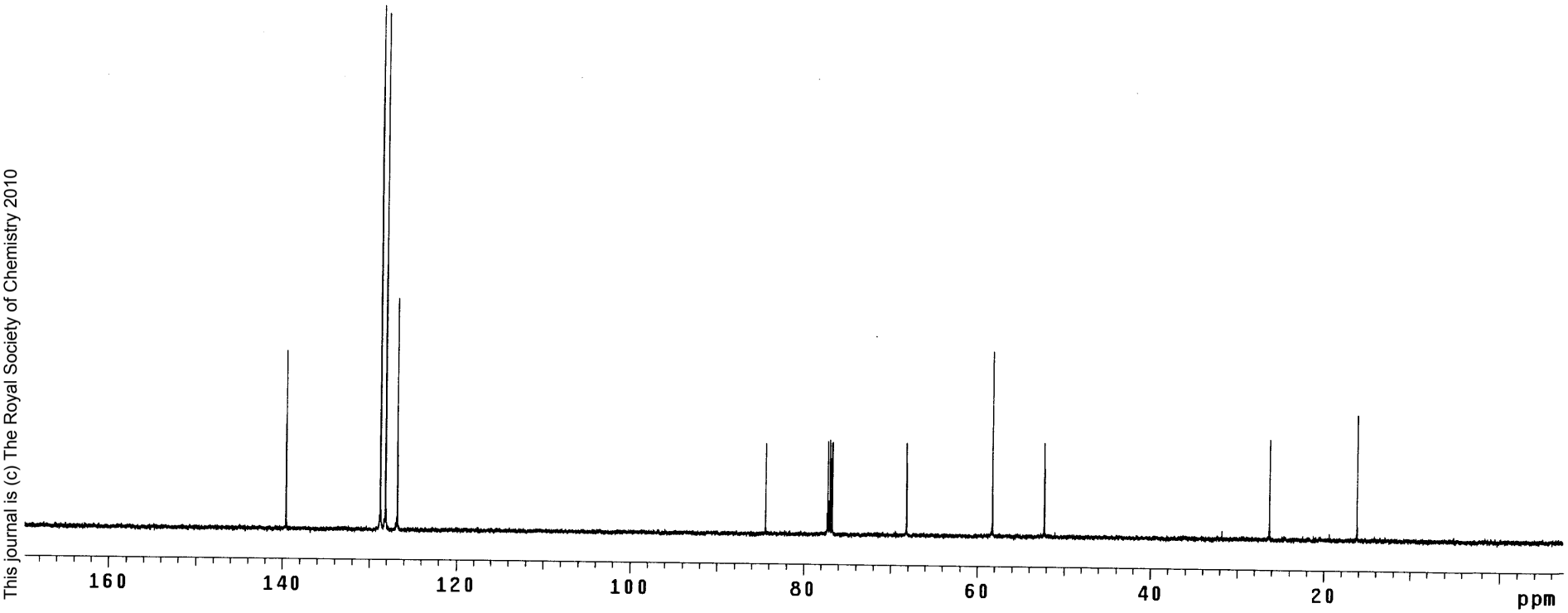
File: CARBON

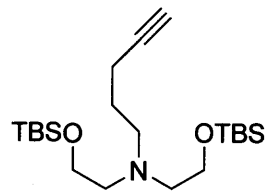
Pulse Sequence: s2pul
Solvent: cdcl3

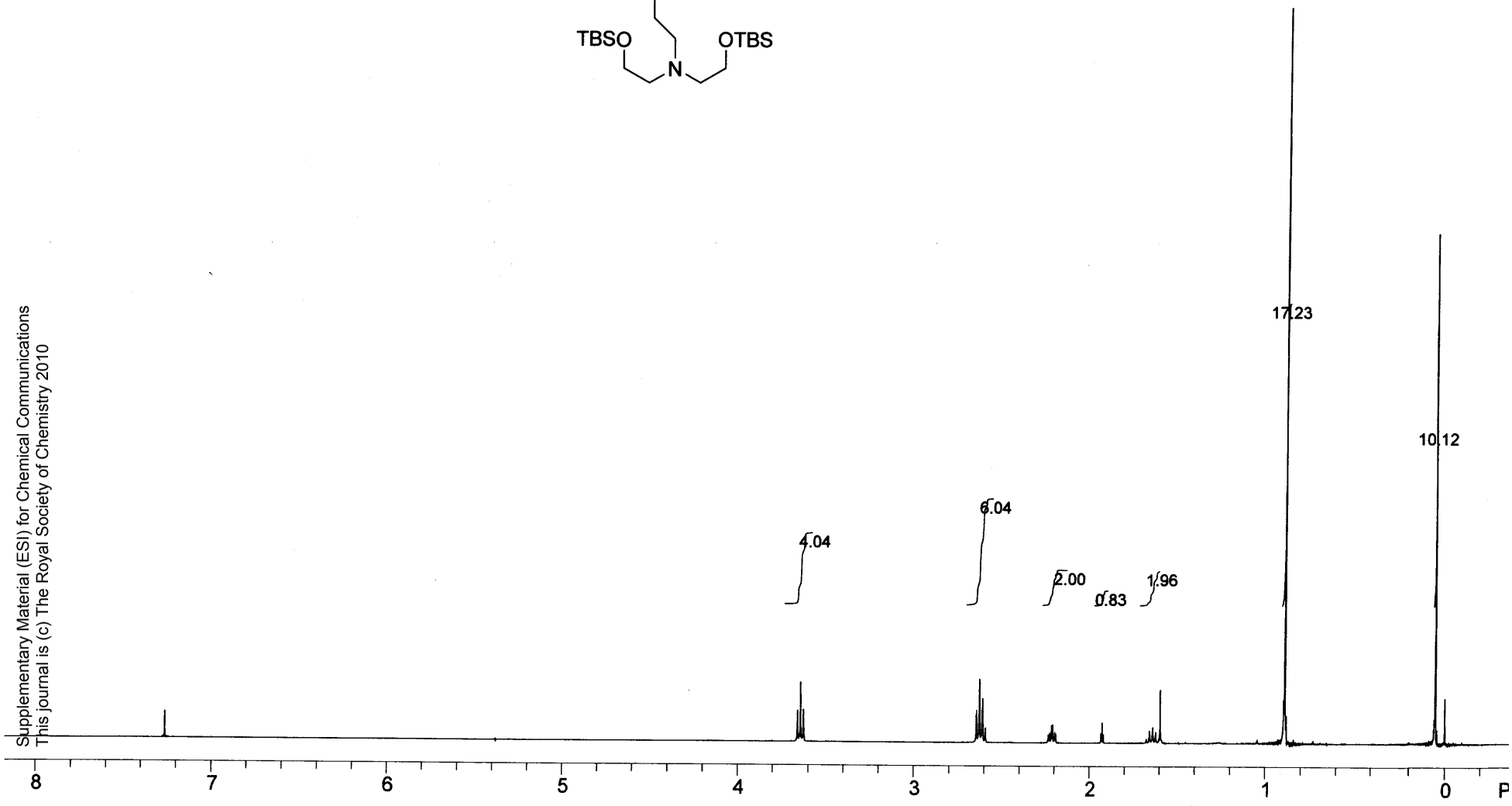
Temp. 25.0 C / 298.1 K
Operator: lcui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
80 repetitions
OBSERVE C13, 125.6889979 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









STANDARD CARBON PARAMETERS

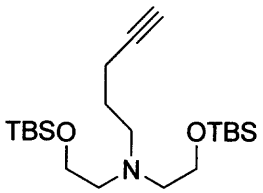
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

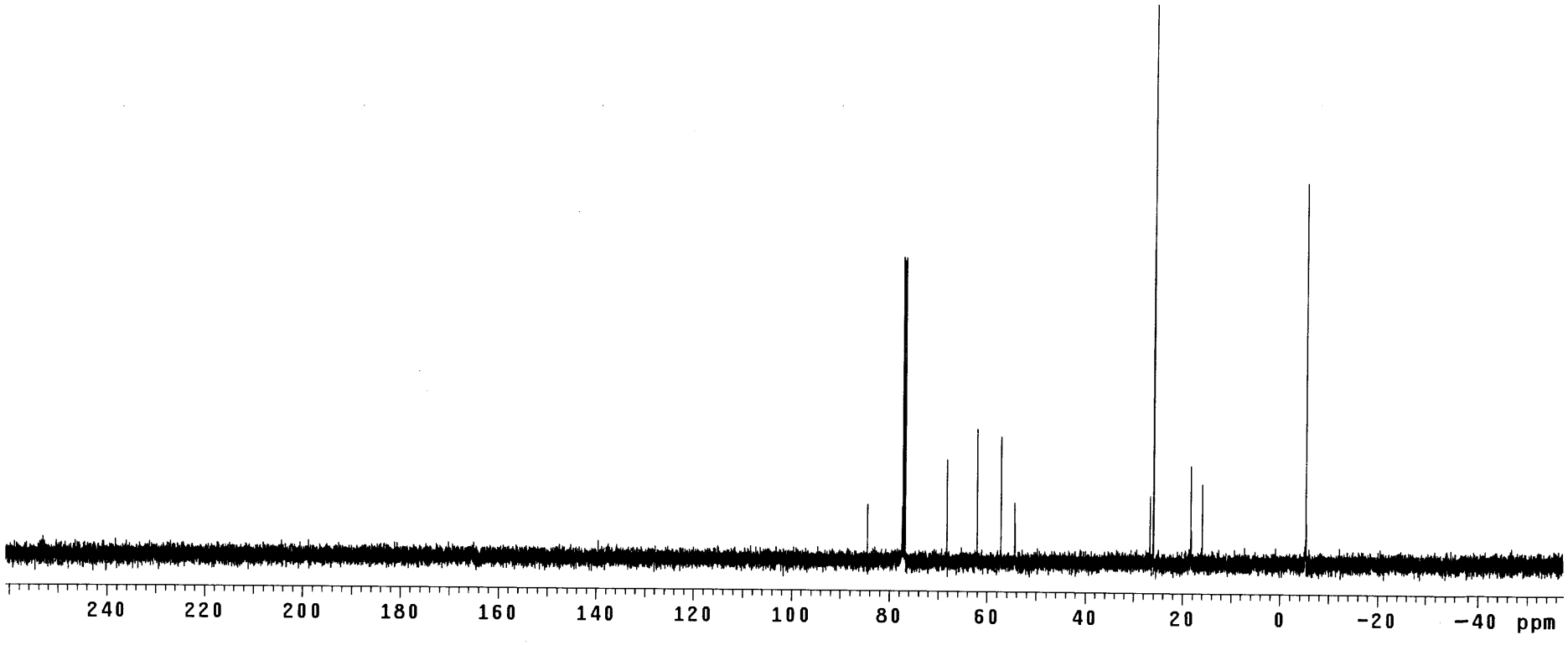
File: CARBON

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
136 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





H1_CDCL3

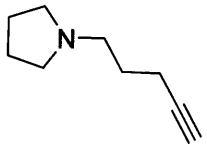
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

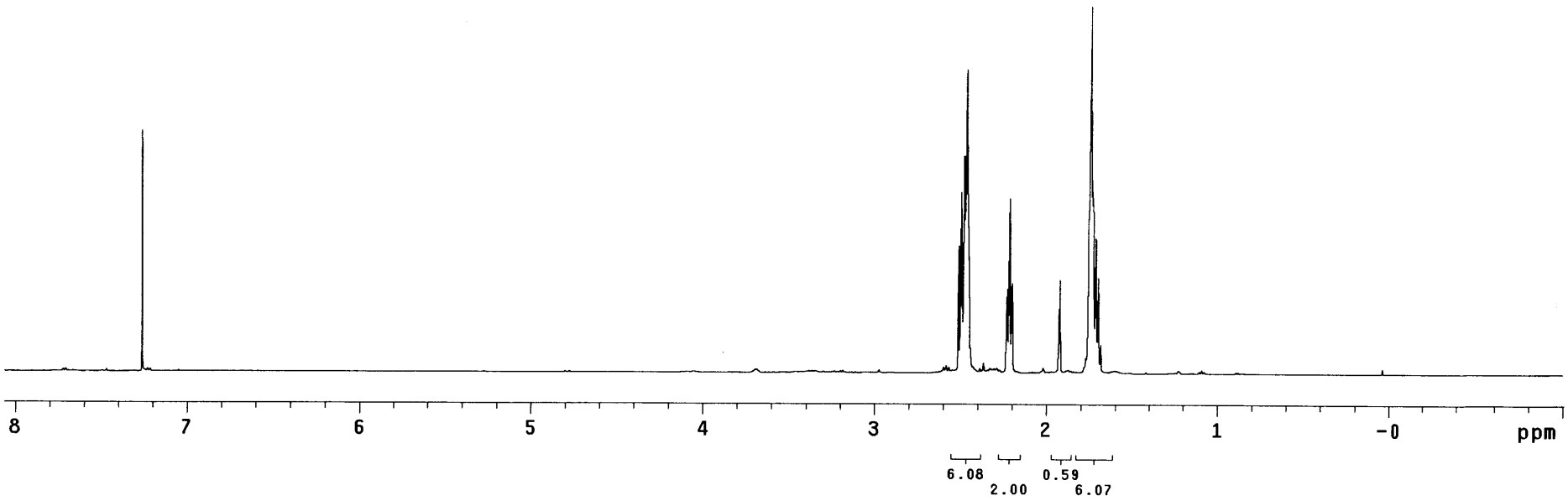
File: H1

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, 499.8563618 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min



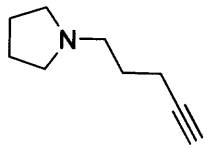


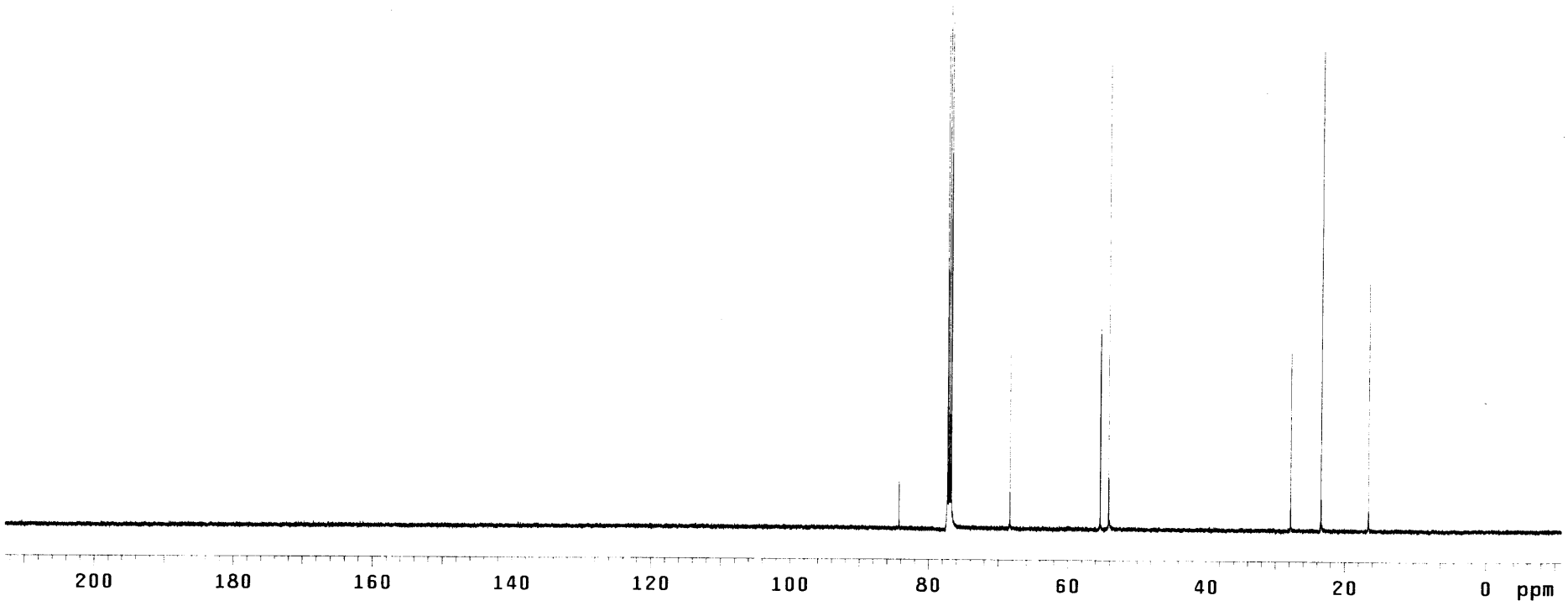
licui-54-1

Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.10
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
7264 repetitions
OBSERVE C13, 125.6732785 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec

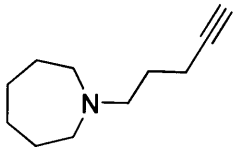


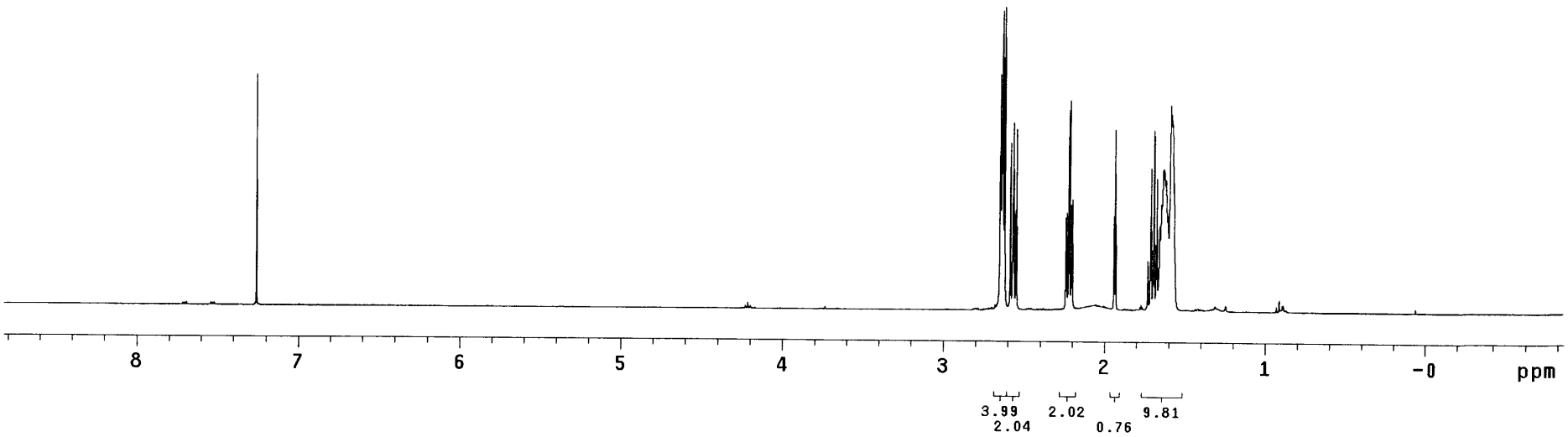


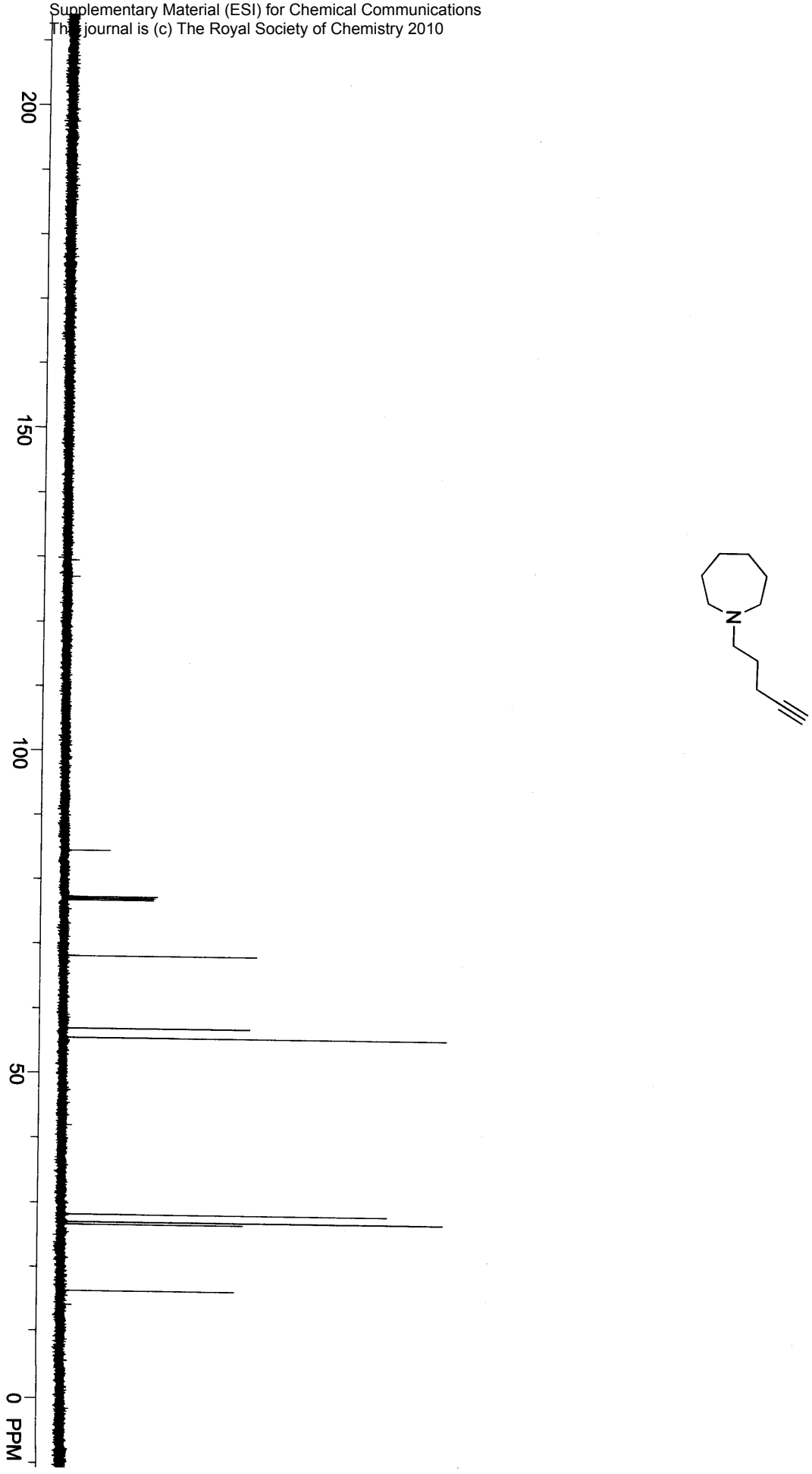
Z*SPEC IDG400-5FE
UCSB

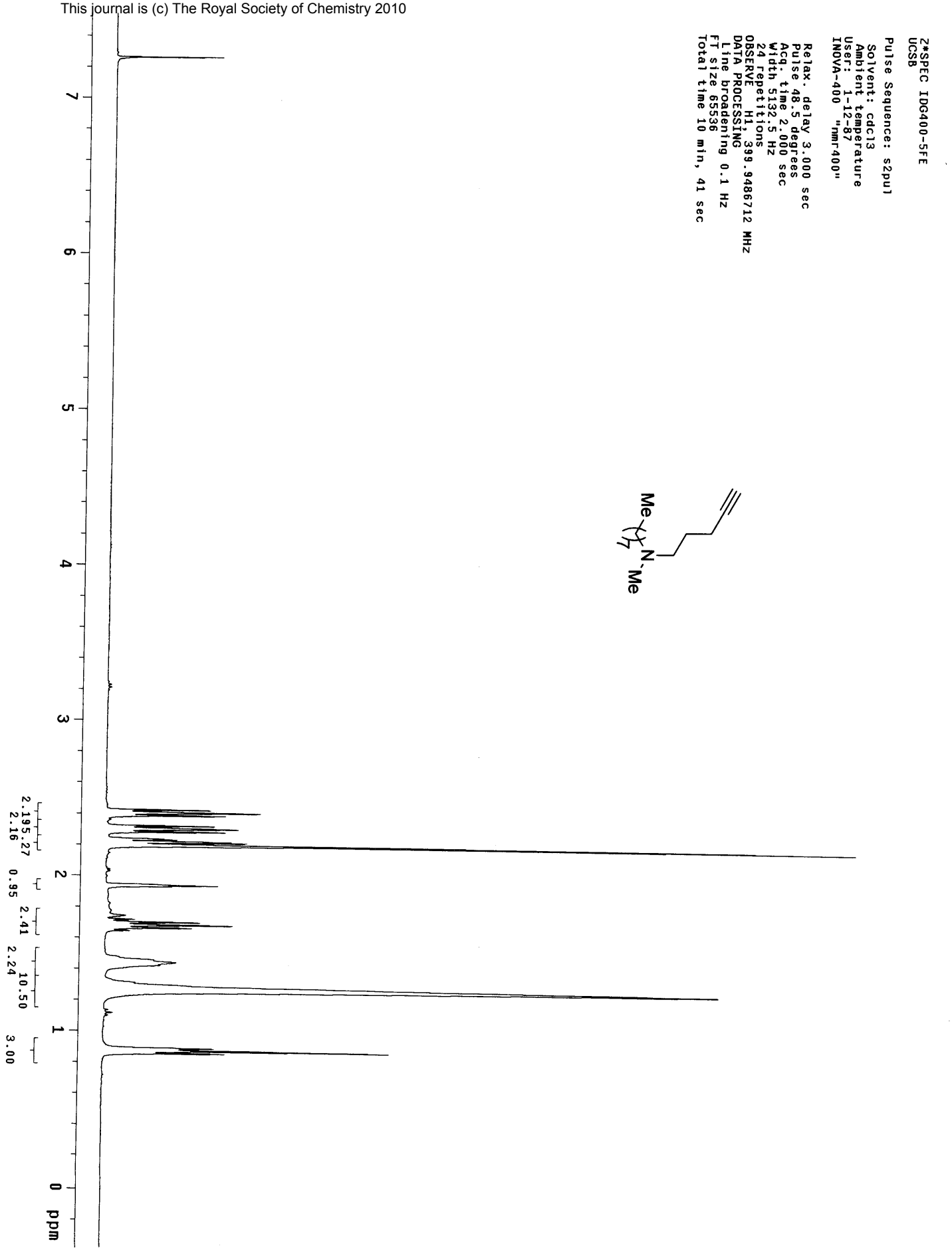
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
20 repetitions
OBSERVE H1, 399.9486720 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec








Z*SPEC IDG400-5FE
UCSB
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"
Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
24 repetitions
OBSERVE H1, 399.9486712 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec
Me
N
Me
7
2.19 5.27
2.16
0.95
2.41
10.50
2.24
3.00
ppm

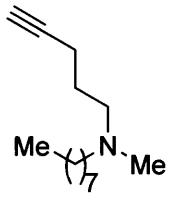
licui-104-1-13C

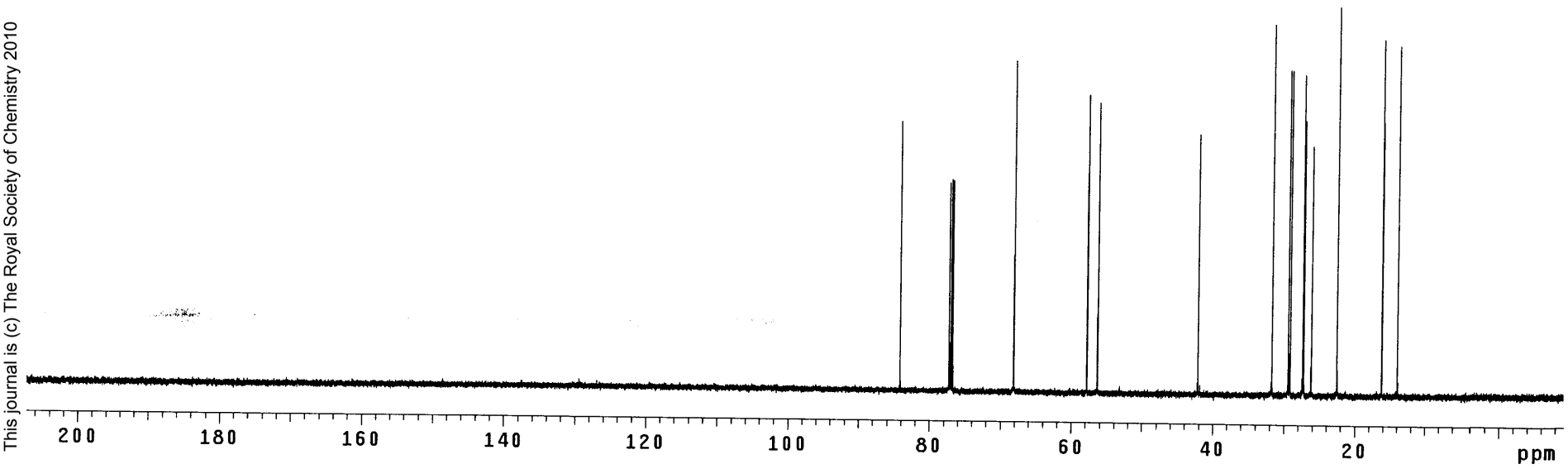
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

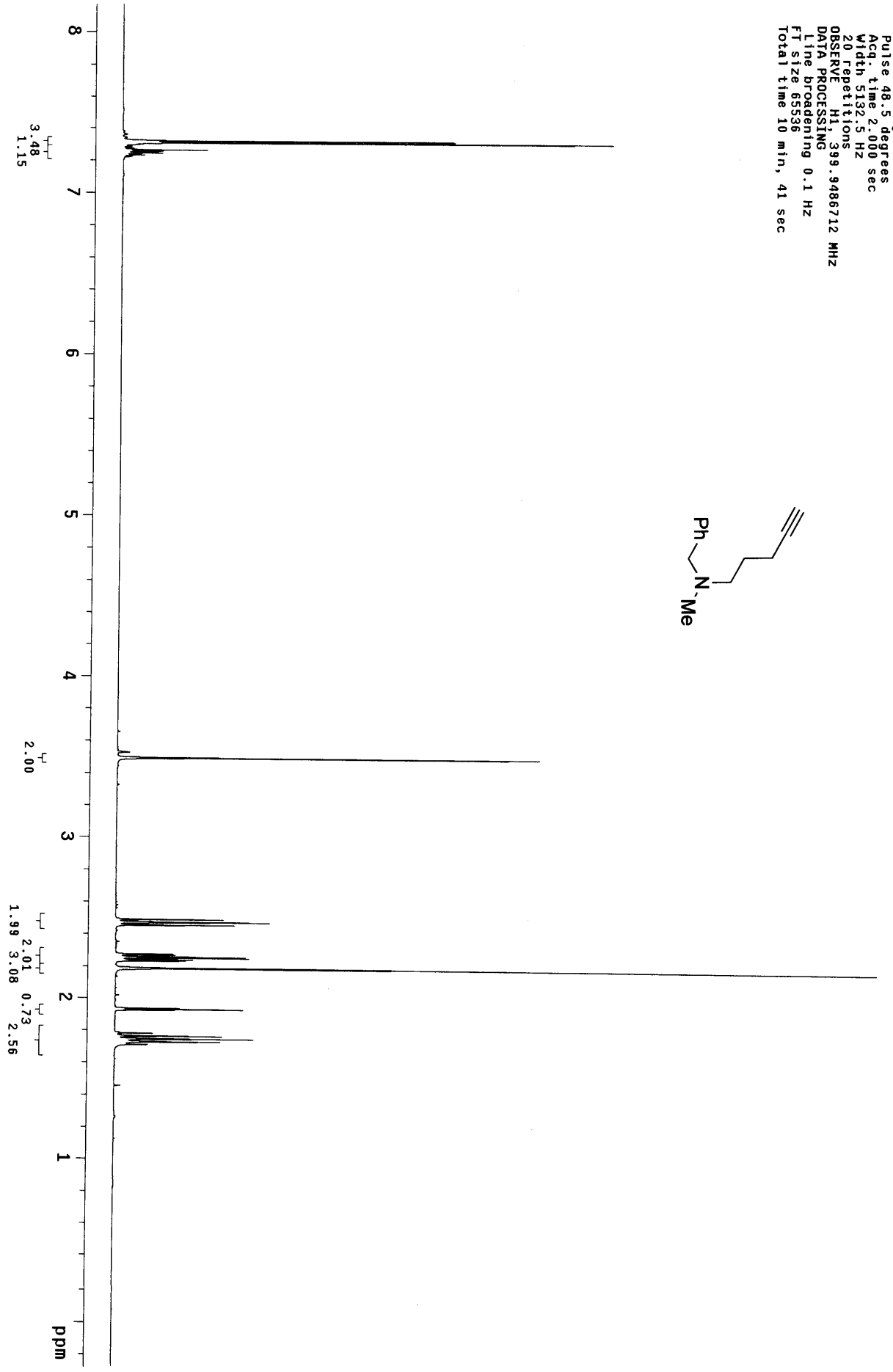
File: CARBON

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

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
112 repetitions
OBSERVE C13, 125.6889897 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






licui-109-1-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"
Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
20 repetitions
OBSERVE H1, 399.9486712 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec
Ph
N
Me
8
7
6
5
4
3
2
1
ppm
3.48
1.15
2.00
1.99
2.01
3.08
0.73
2.56

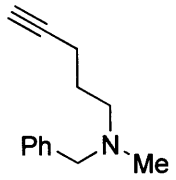
licui-109-1-13C

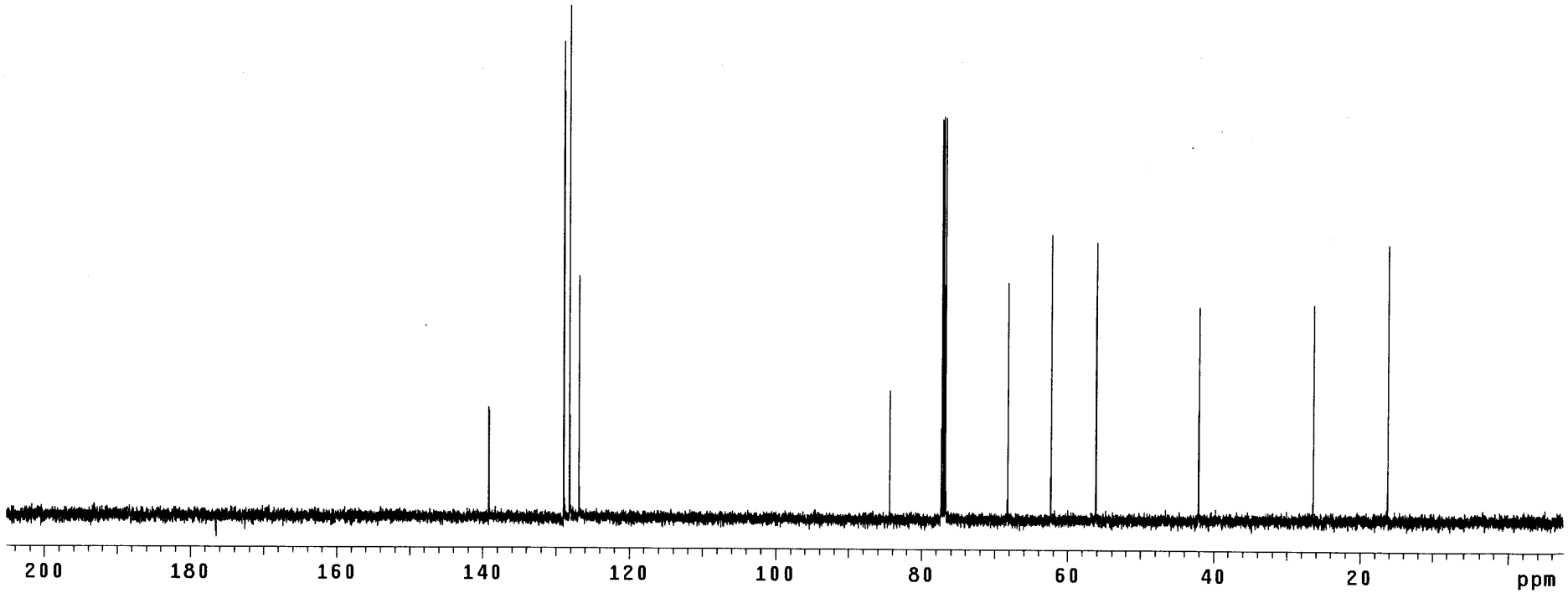
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

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
71 repetitions
OBSERVE C13, 125.6889879 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





liuci-114-1-SM

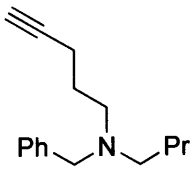
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

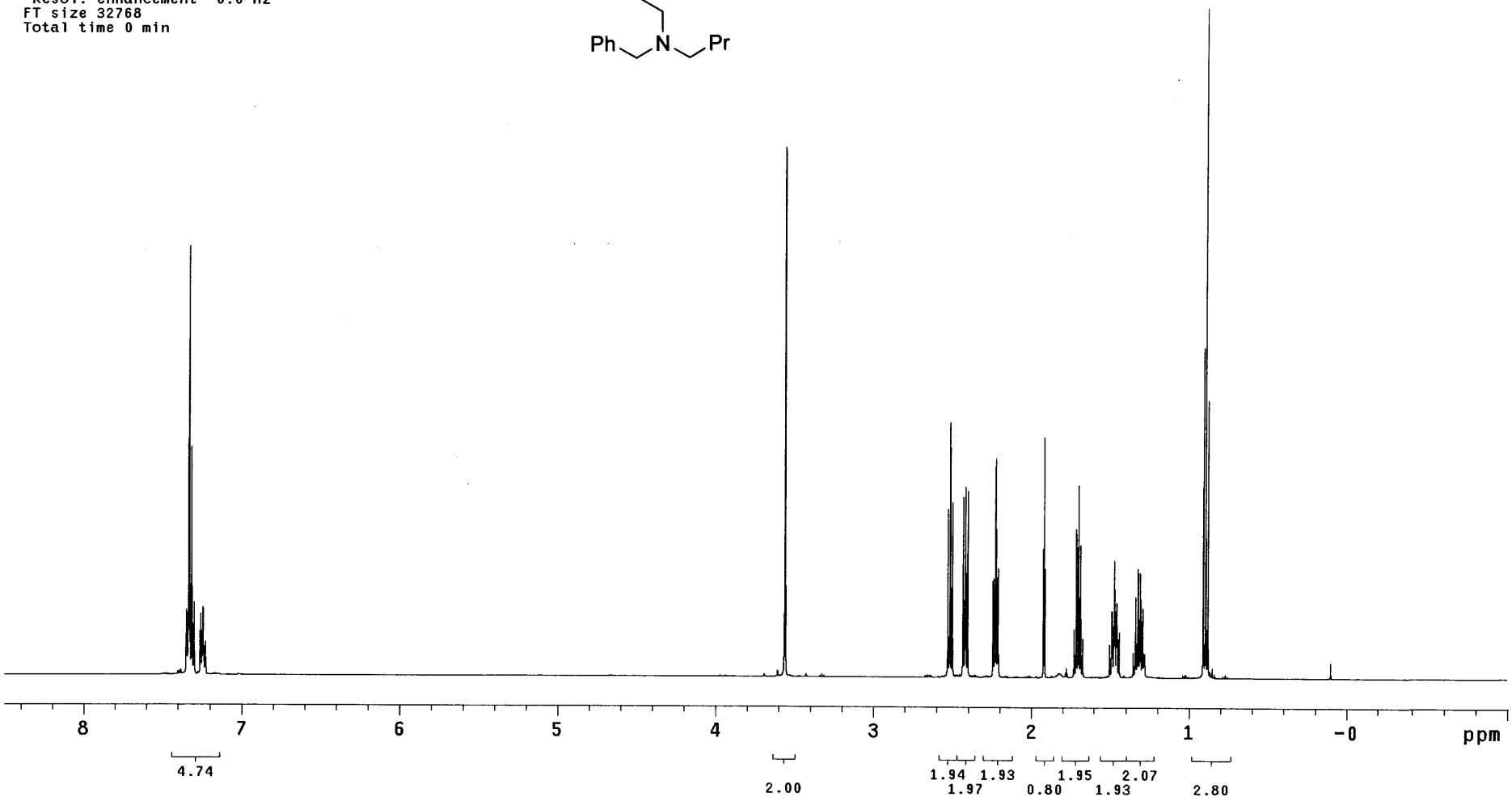
File: H1

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
4 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min





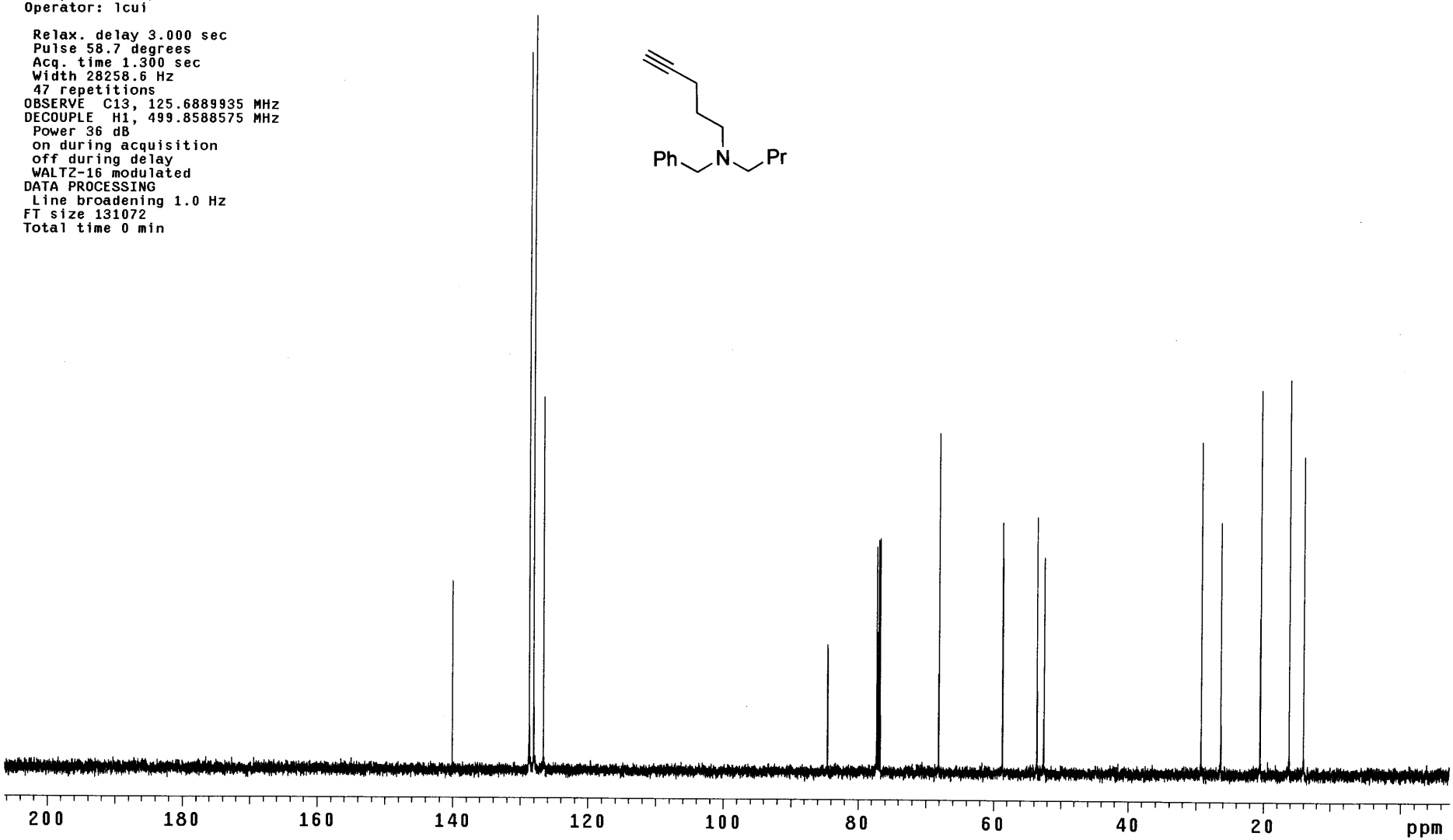
liuci-114-1-SM-13C

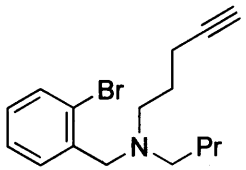
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

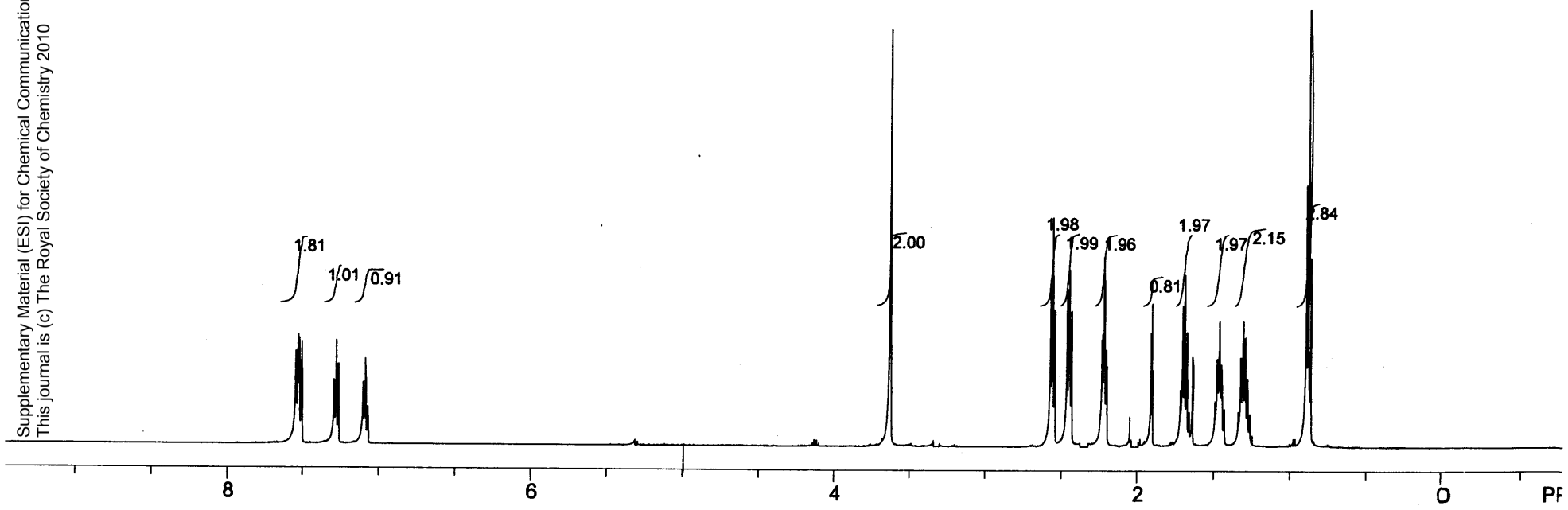
File: CARBON

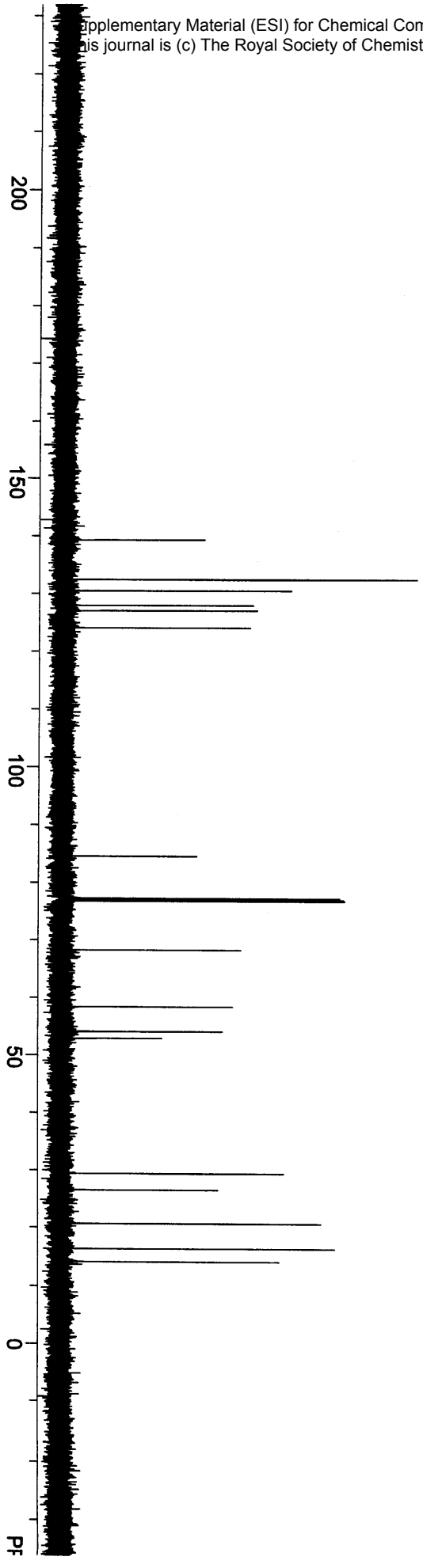
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lcui

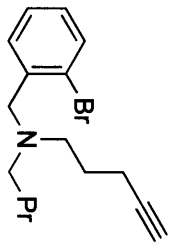
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
47 repetitions
OBSERVE C13, 125.6889935 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min

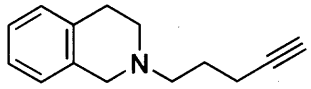


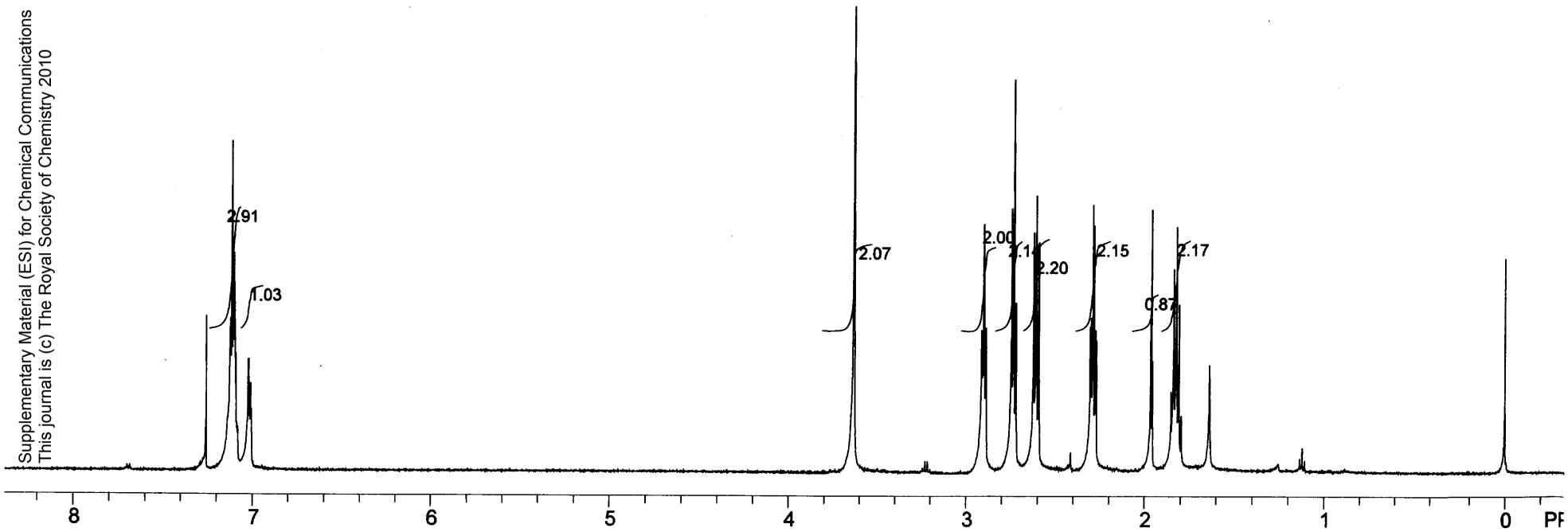












STANDARD CARBON PARAMETERS

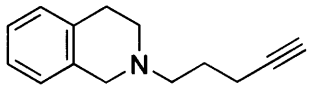
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

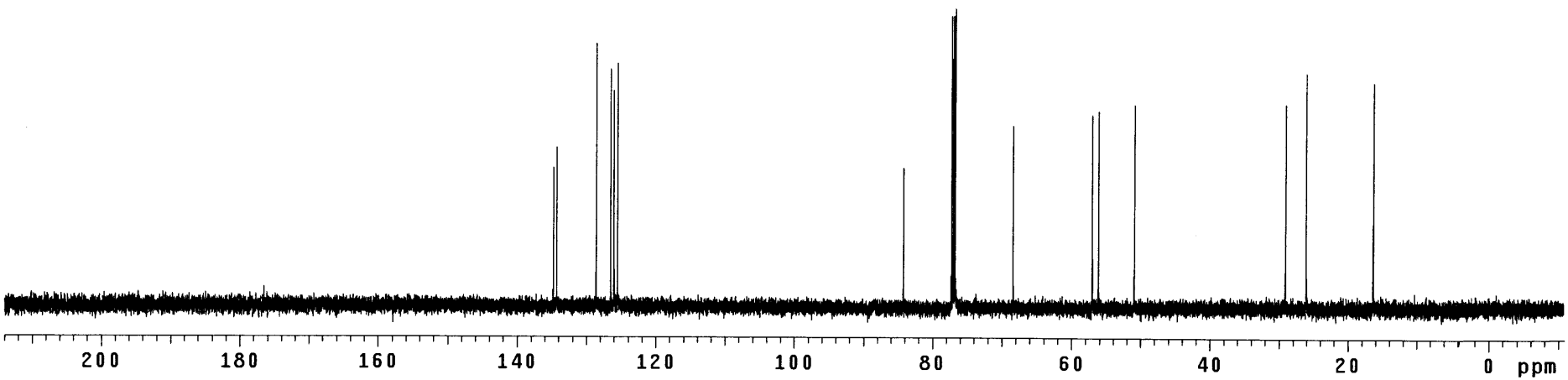
File: CARBON

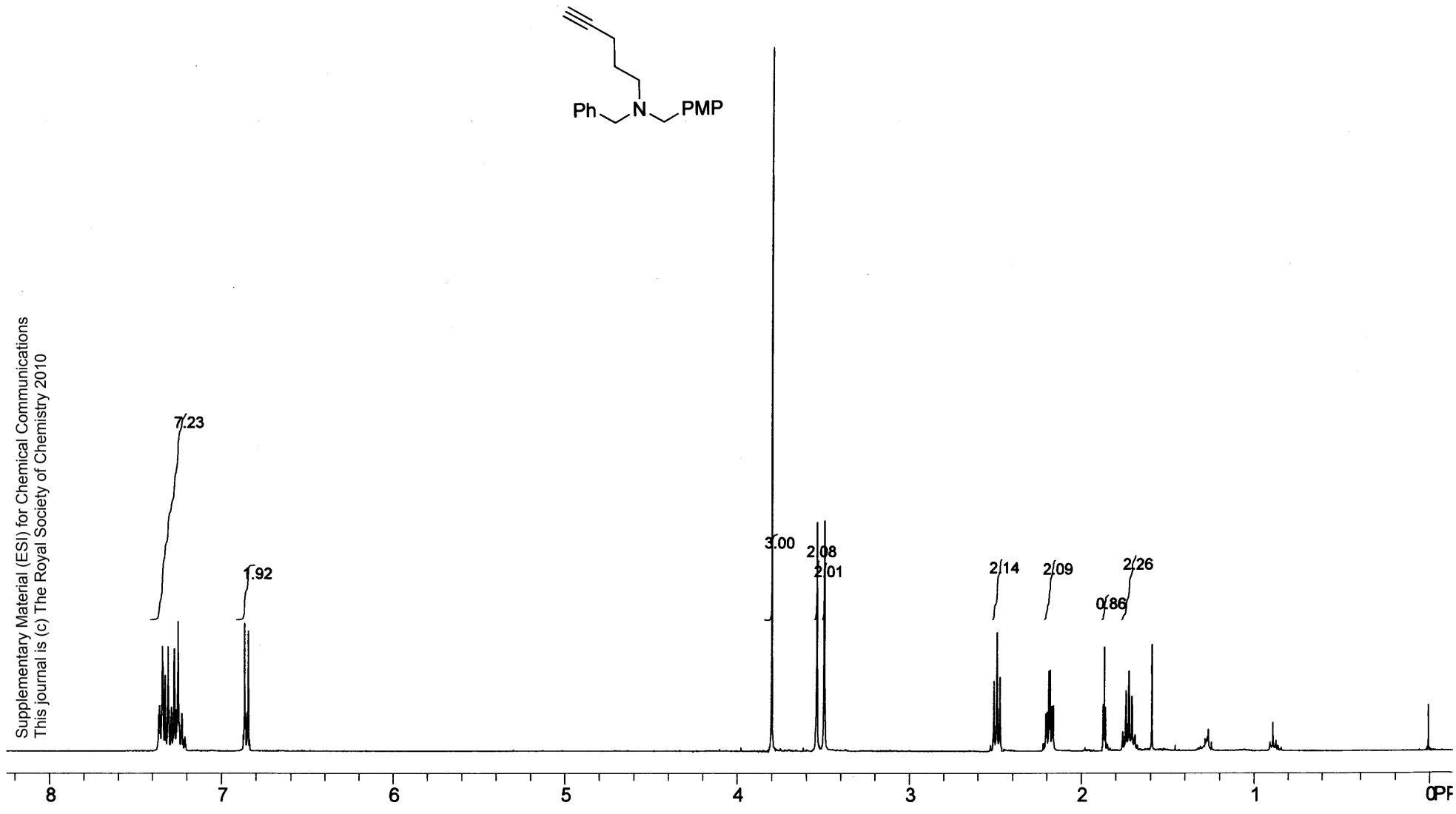
Pulse Sequence: s2pul
Solvent: cdcl3

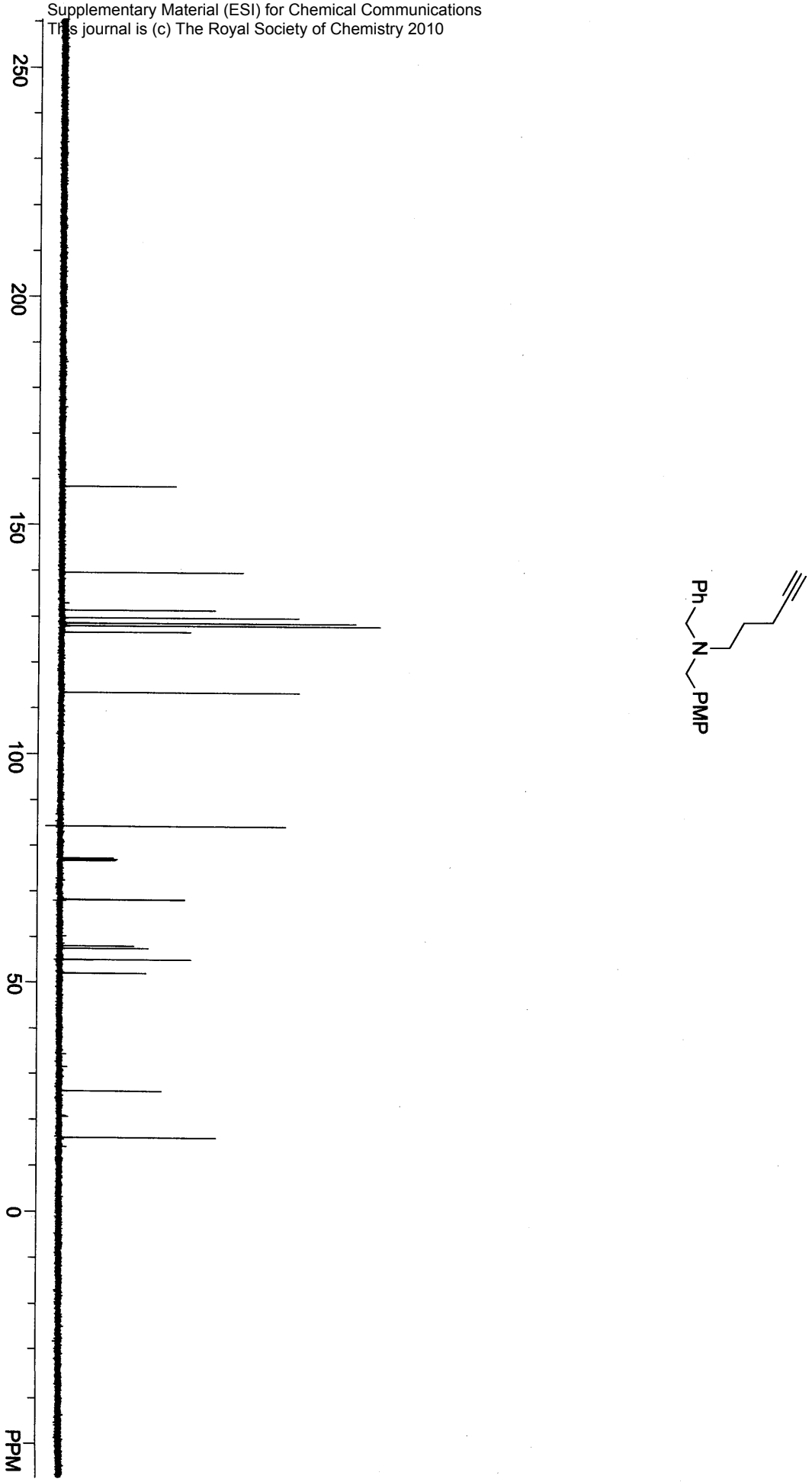
Temp. 21.9 C / 295.1 K
Operator: lye

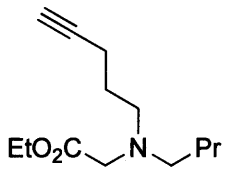
Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
86 repetitions
OBSERVE C13, 125.6889897 MHz
DECOUPLE H1, 499.8588575 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 0 min

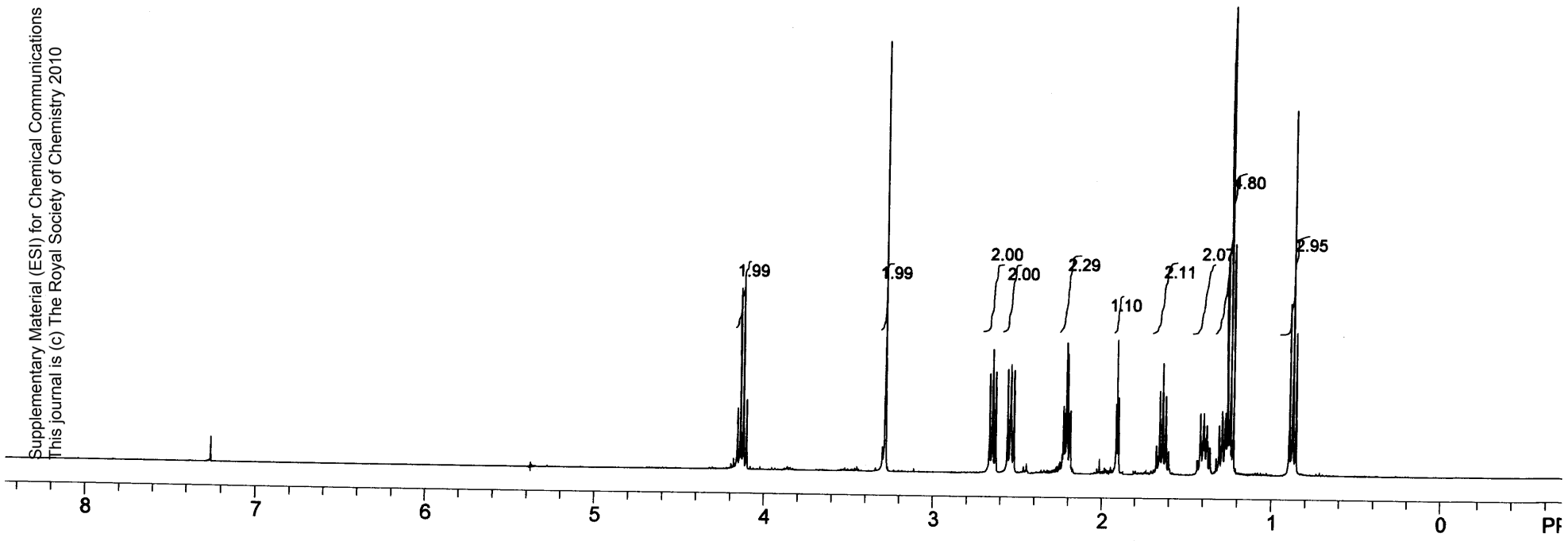







250
200
150
100
50
0
PPM
Ph
N
PMP





STANDARD CARBON PARAMETERS

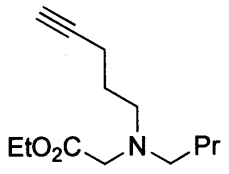
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

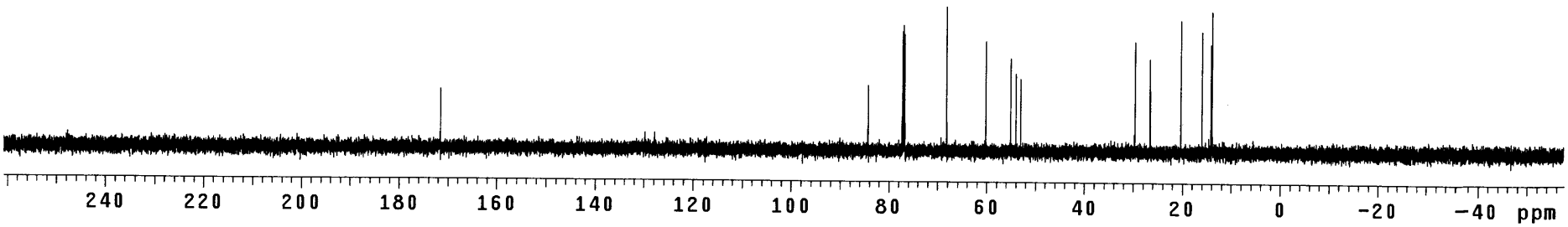
File: CARBON

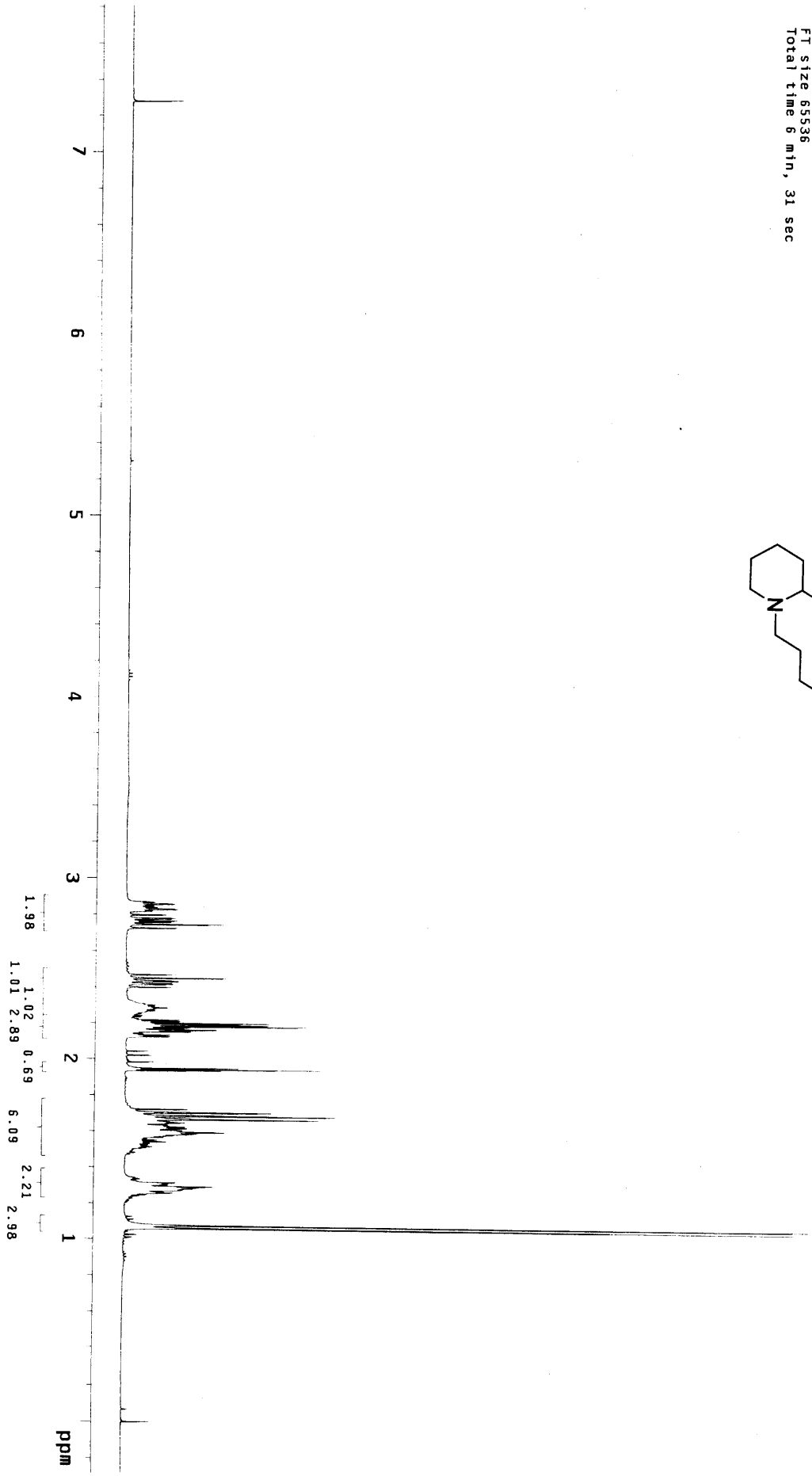
Pulse Sequence: s2pul
Solvent: cdcl3

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
14 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






11cui-70-2-1H
Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.28_17
File : exp
Sample id : tmpstudy
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-400 "mr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
28 repetitions
OBSERVE H1, 400.0565418 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 6 min, 31 sec
Me
N
7
6
5
4
3
2
1
ppm
1.98
1.02
2.89
0.69
1.01
6.09
2.21
2.98

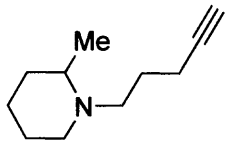
licui-72-2-13C

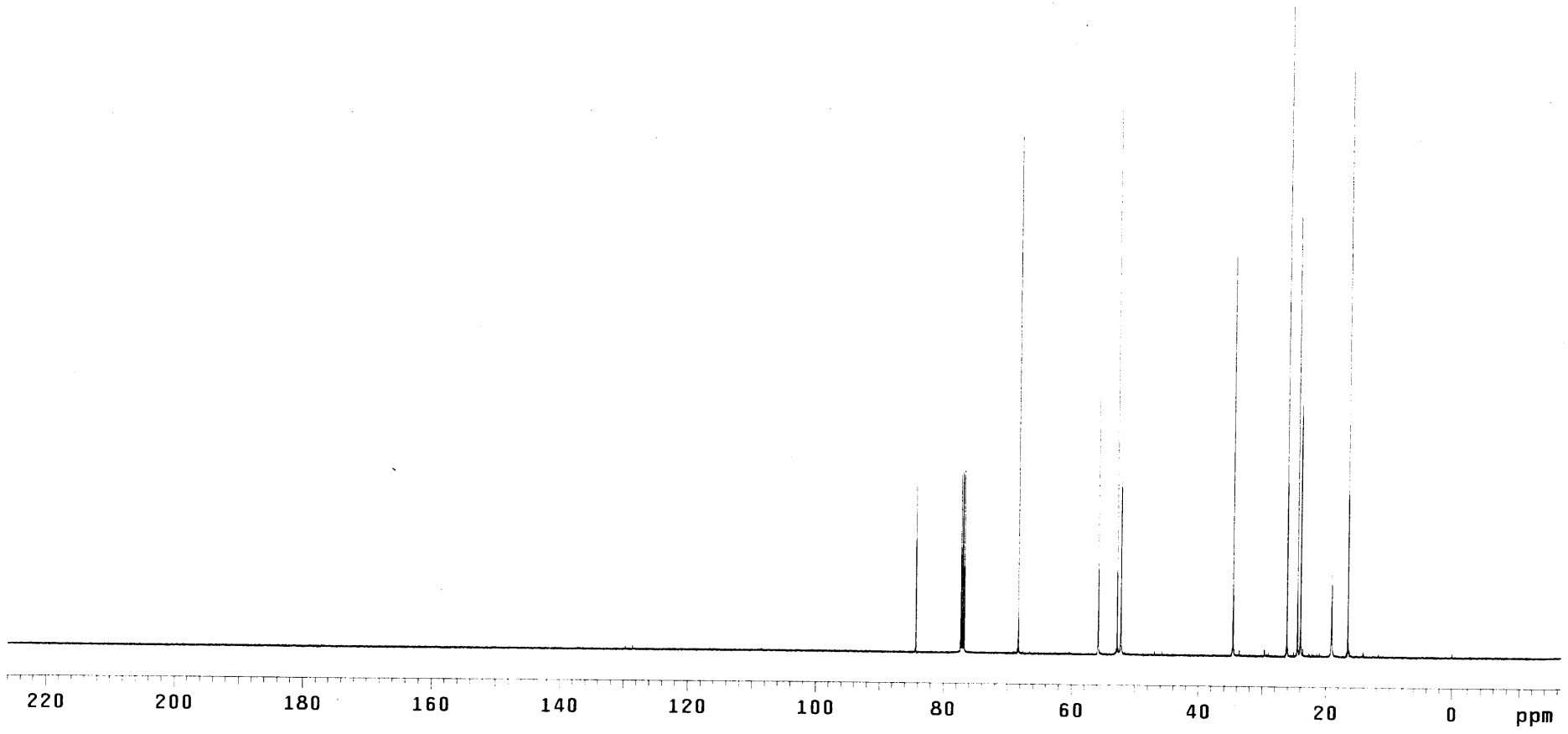
Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.29_02
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
5300 repetitions
OBSERVE C13, 125.6732813 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec

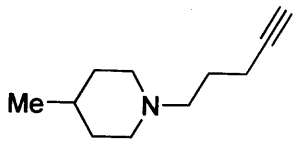


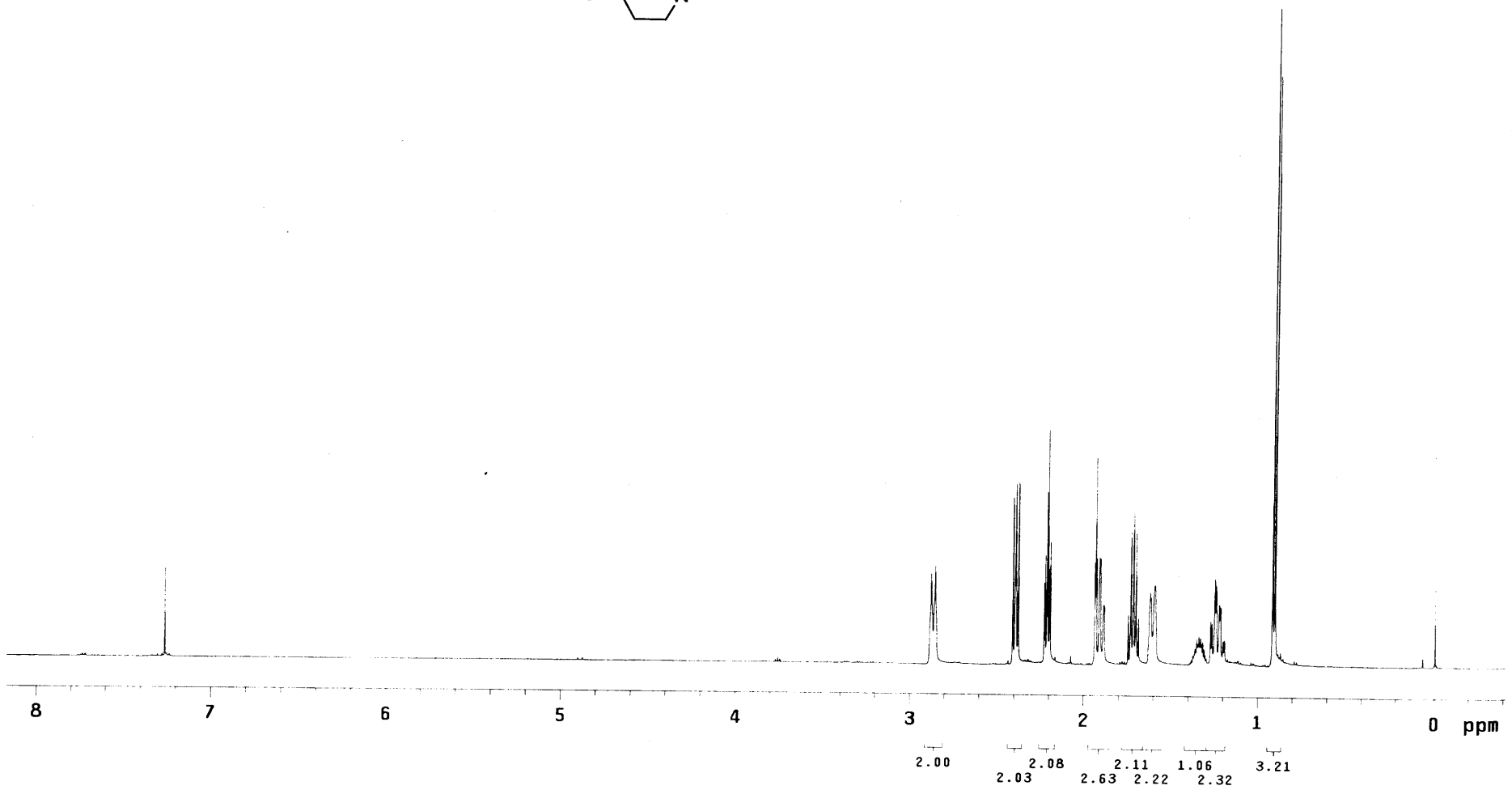


Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.04_06
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
28 repetitions
OBSERVE H1, 499.7939123 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min, 37 sec



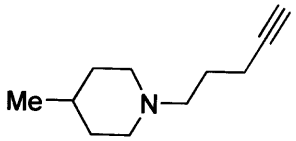


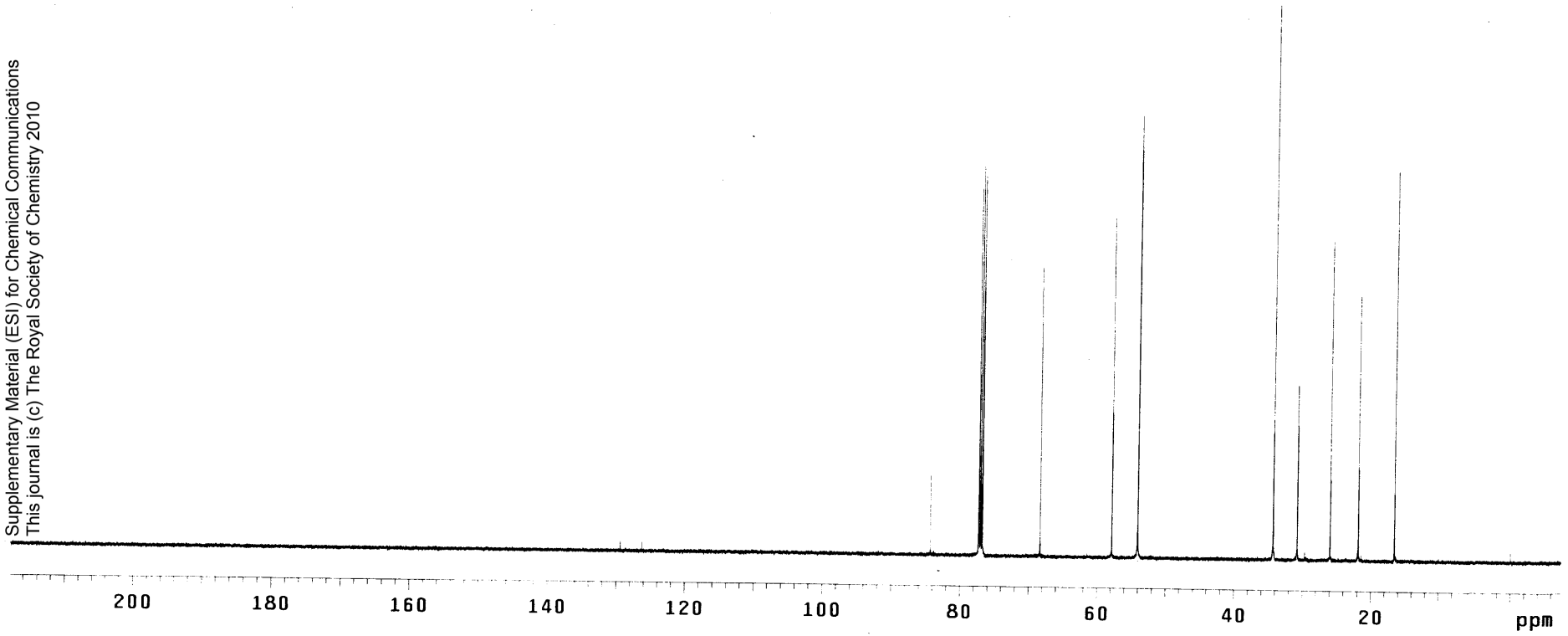
Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.05_01
File : /mnt/argenta/nmr500/data/Zhang/licui/licui-72-1-C13.fid
Sample id : tmpstudy

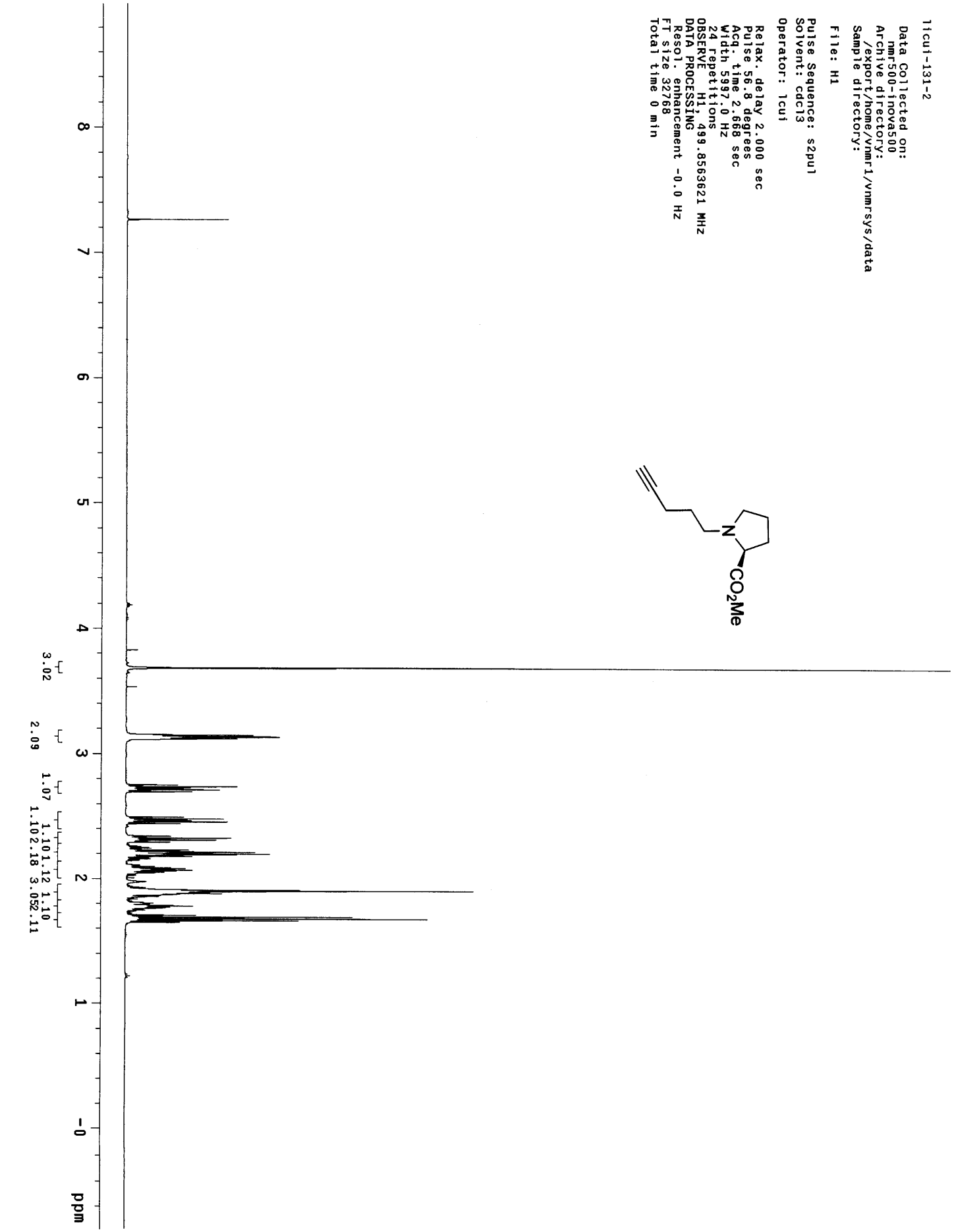
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: licui-72-1-C13
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
4420 repetitions
OBSERVE C13, 125.6732795 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec






1icui-131-2
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: H1
Pulse Sequence: s2pul
Solvent: cdcl3
Operator: 1cui
Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
24 repetitions
OBSERVE H1, 499.8563621 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
N
CO2Me
8
7
6
5
4
3
2
1
-0
ppm
3.02
2.09
1.07
1.10
1.12
1.10
1.10
2.18
3.05
2.11

licui-131-1-13C

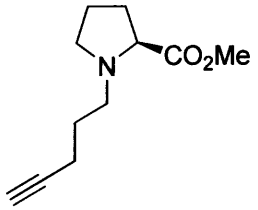
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

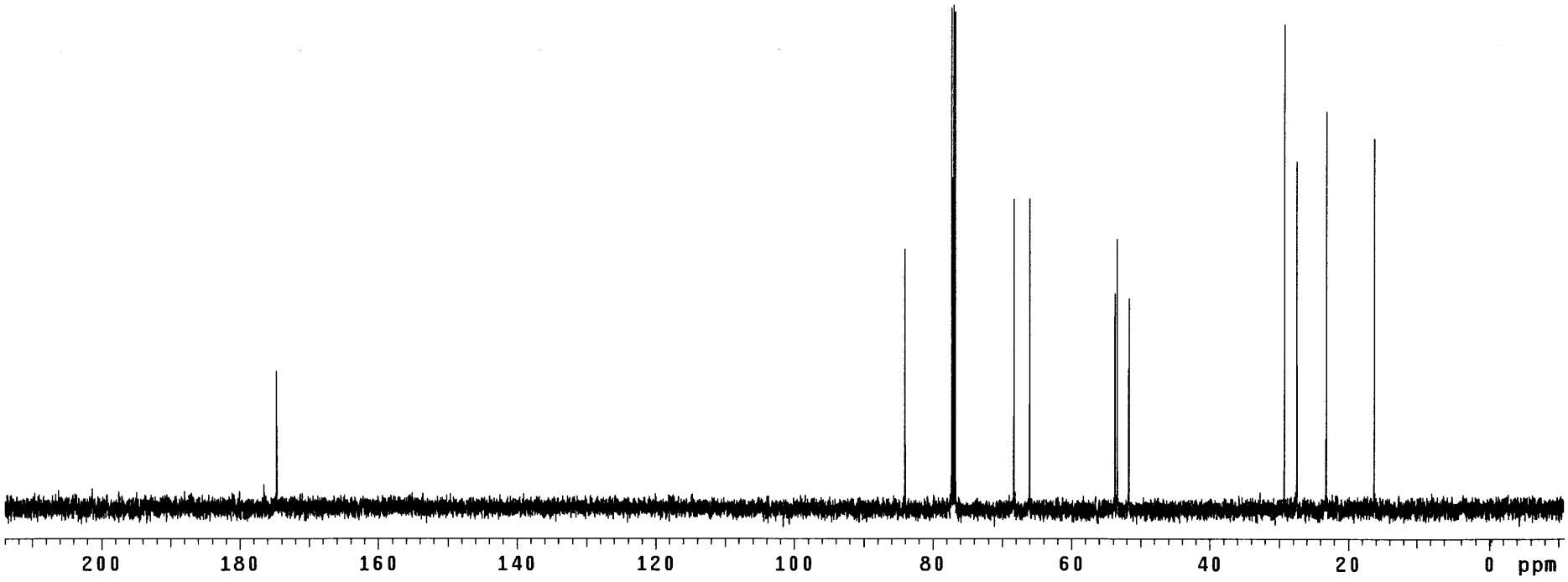
File: CARBON

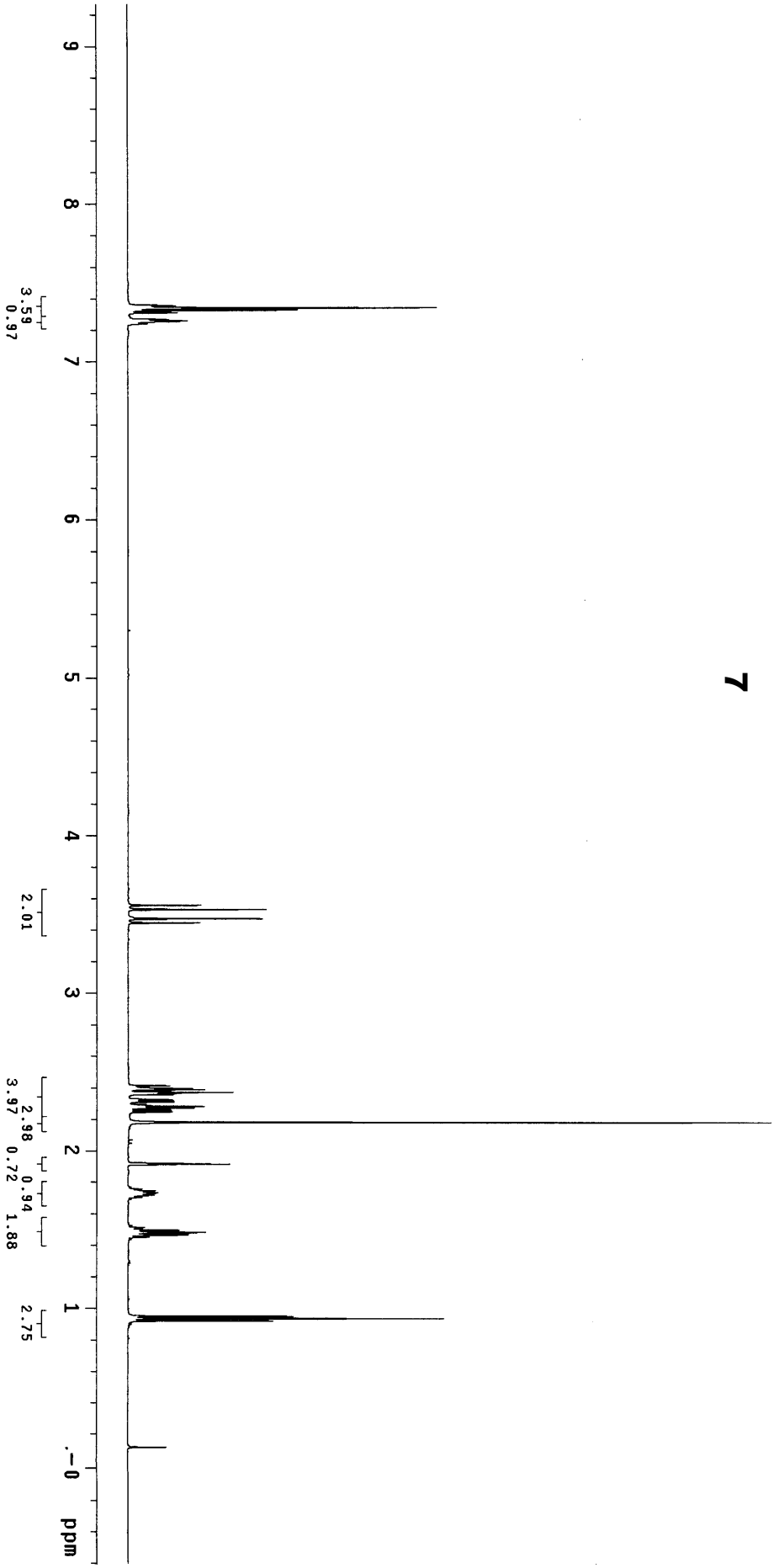
Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 24.7 C / 297.9 K
Operator: licui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
48 repetitions
OBSERVE C13, 125.6889901 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






licui-3-9-1H
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
16 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
Me
N
Me
Bn
7
3.59
0.97
2.01
2.98
3.97
0.72
0.94
1.88
2.75
9
8
7
6
5
4
3
2
1
0
ppm

licui-3-9-1-13C

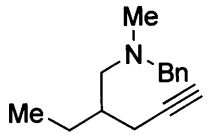
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

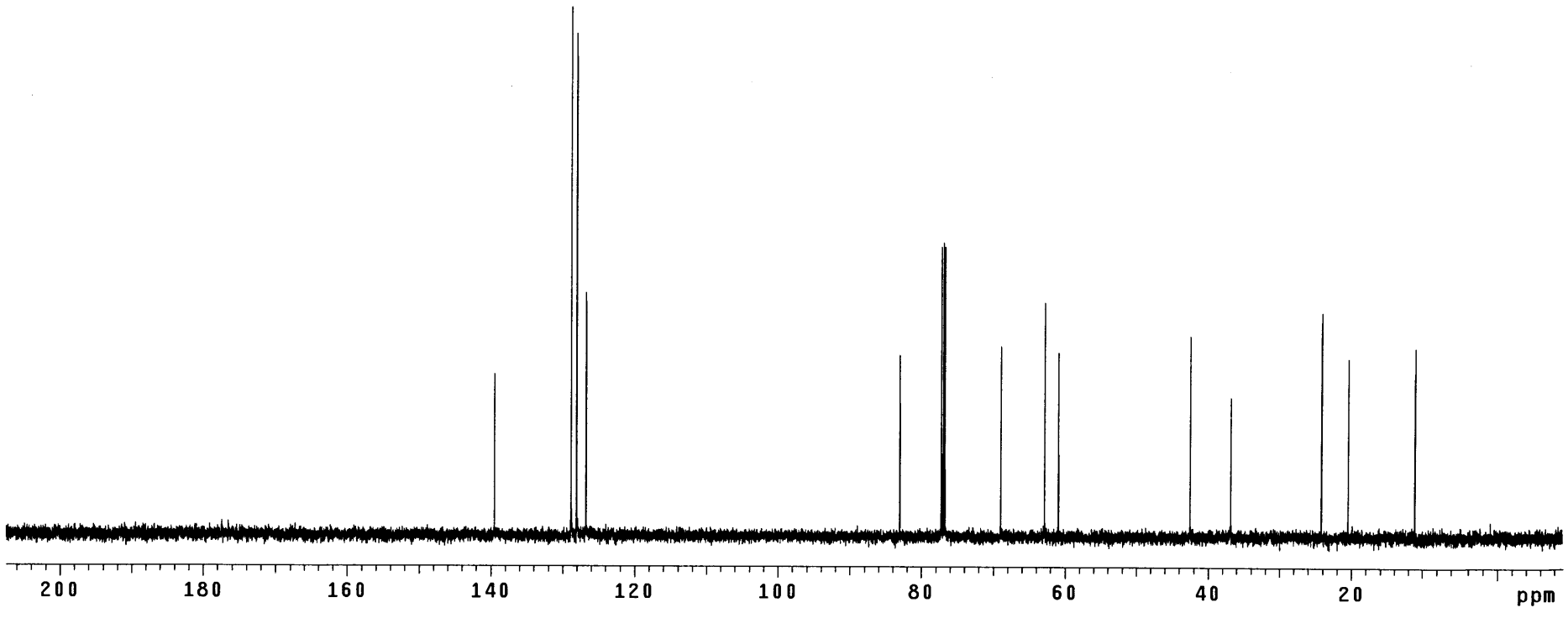
Pulse Sequence: s2pul
Solvent: cdcl3

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
20 repetitions
OBSERVE C13, 125.6889901 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



7



licui-11-2-1H

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

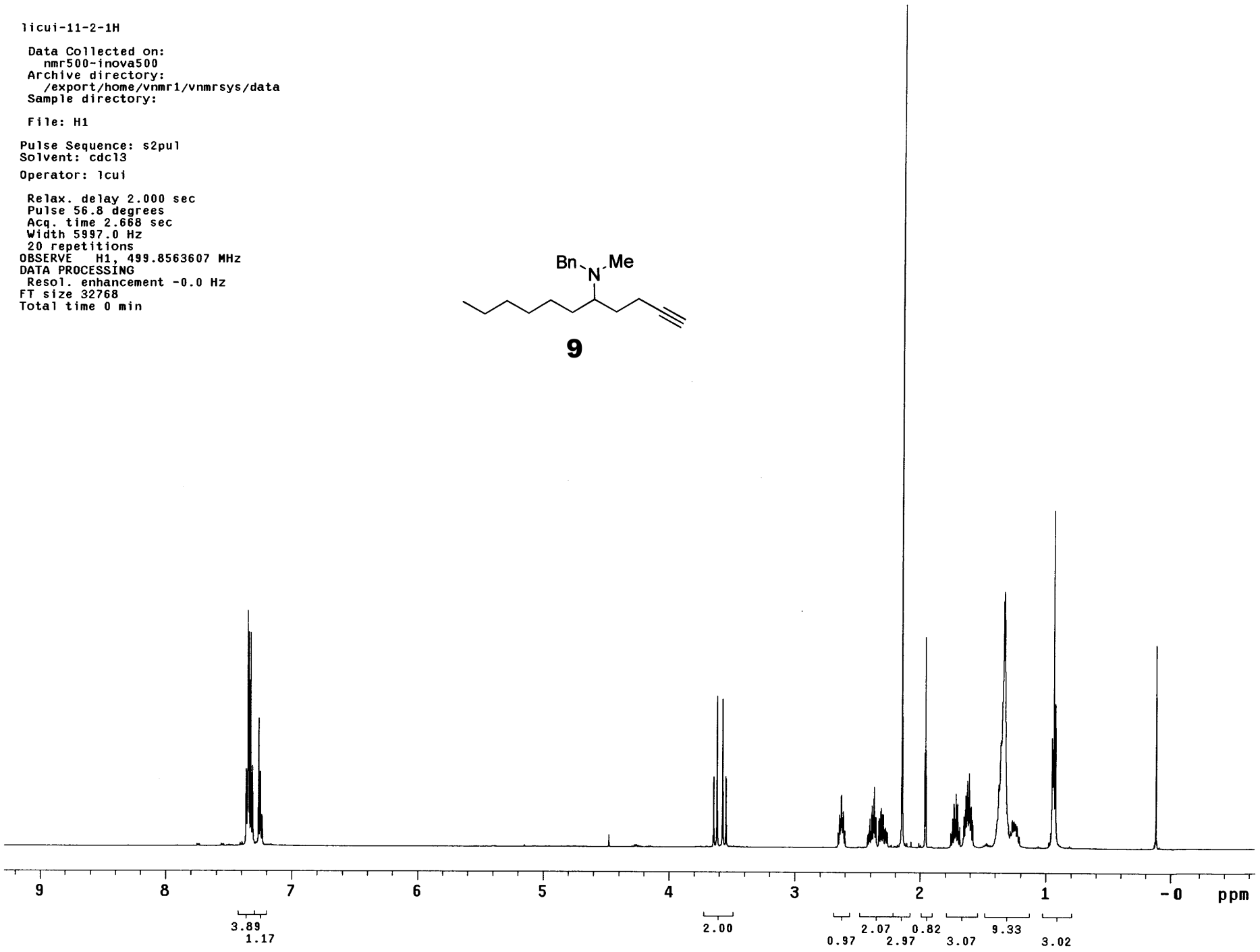
File: H1

Pulse Sequence: s2pul
Solvent: cdcl3

Operator: licui

Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
20 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min

9



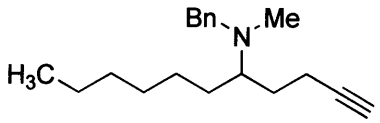
liuci-106-1-13C

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

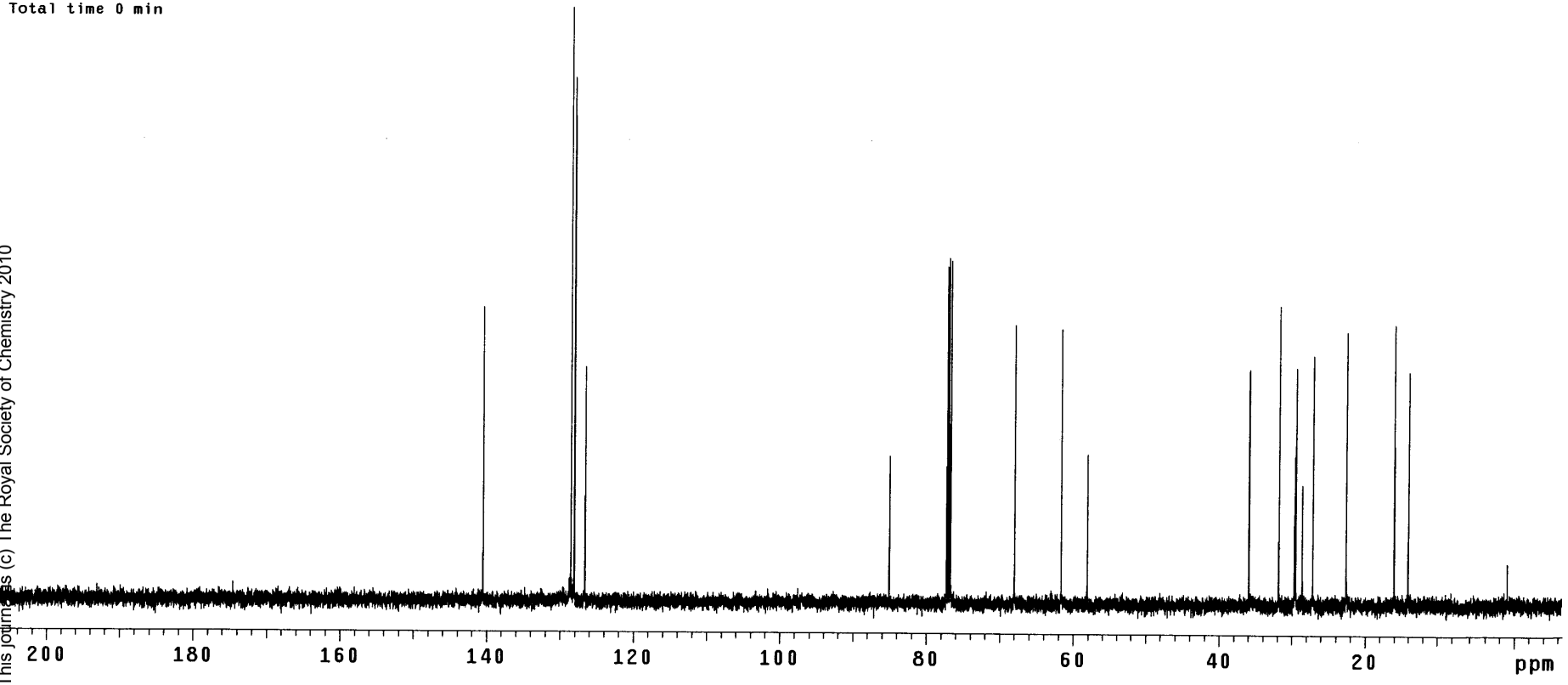
File: CARBON

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

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
24 repetitions
OBSERVE C13, 125.6889918 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



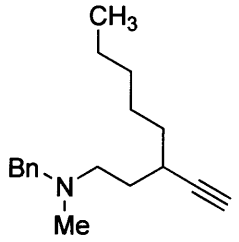
9



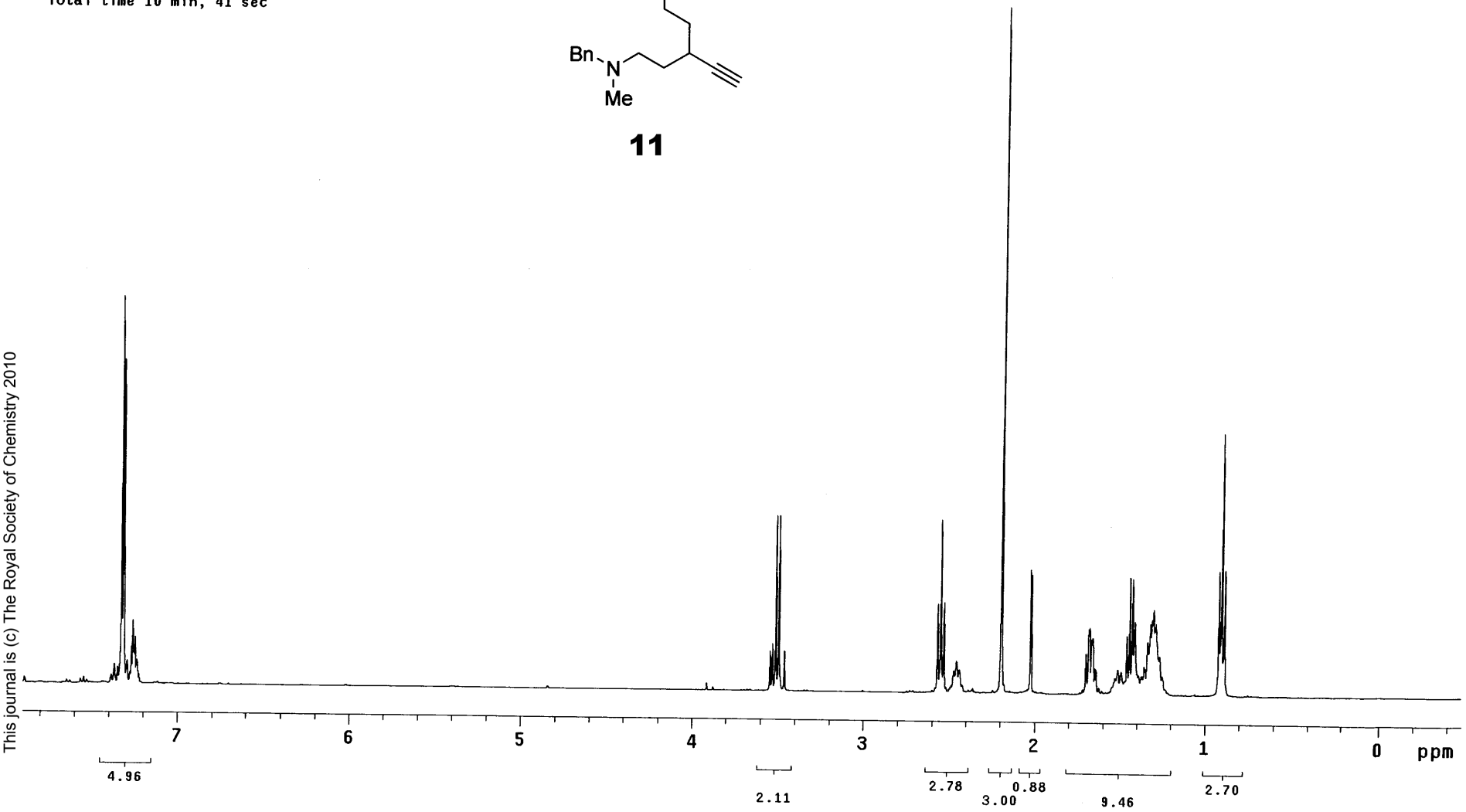
Z*SPEC IDG400-5FE
UCSB

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
20 repetitions
OBSERVE H1, 399.9486712 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec



11



liuci-106-1-13C

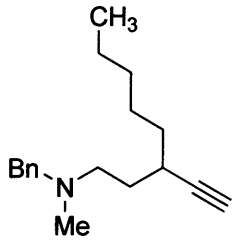
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

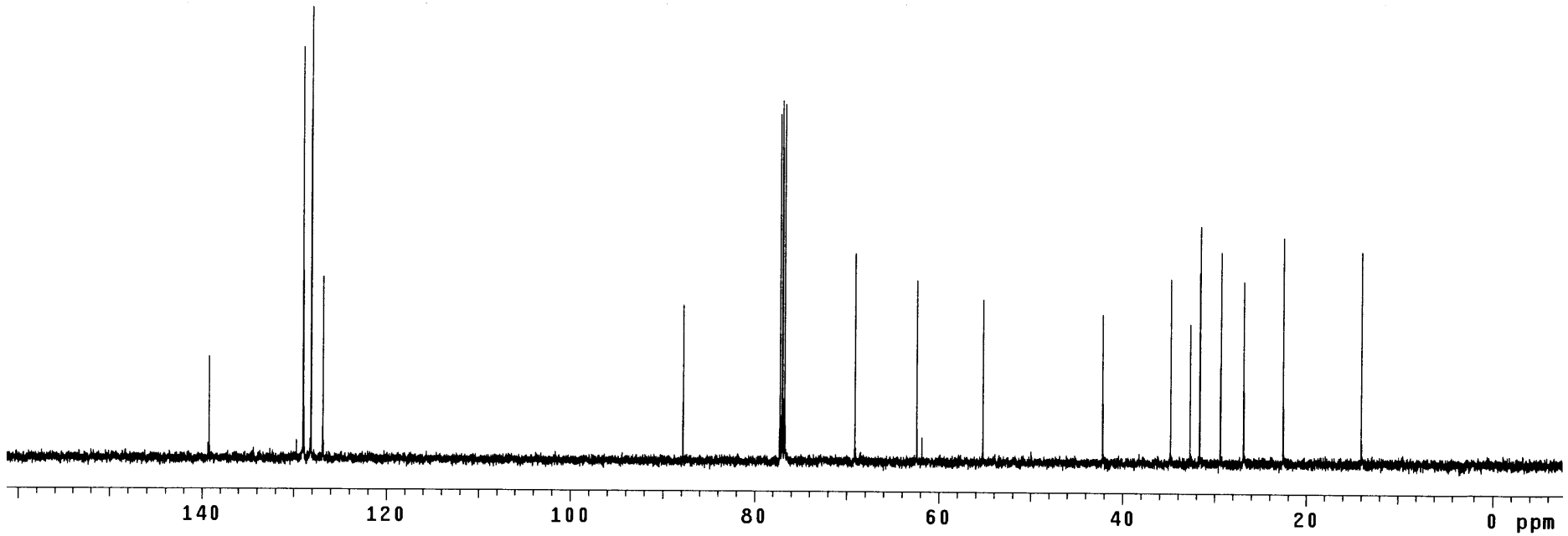
Pulse Sequence: s2pul
Solvent: cdcl3

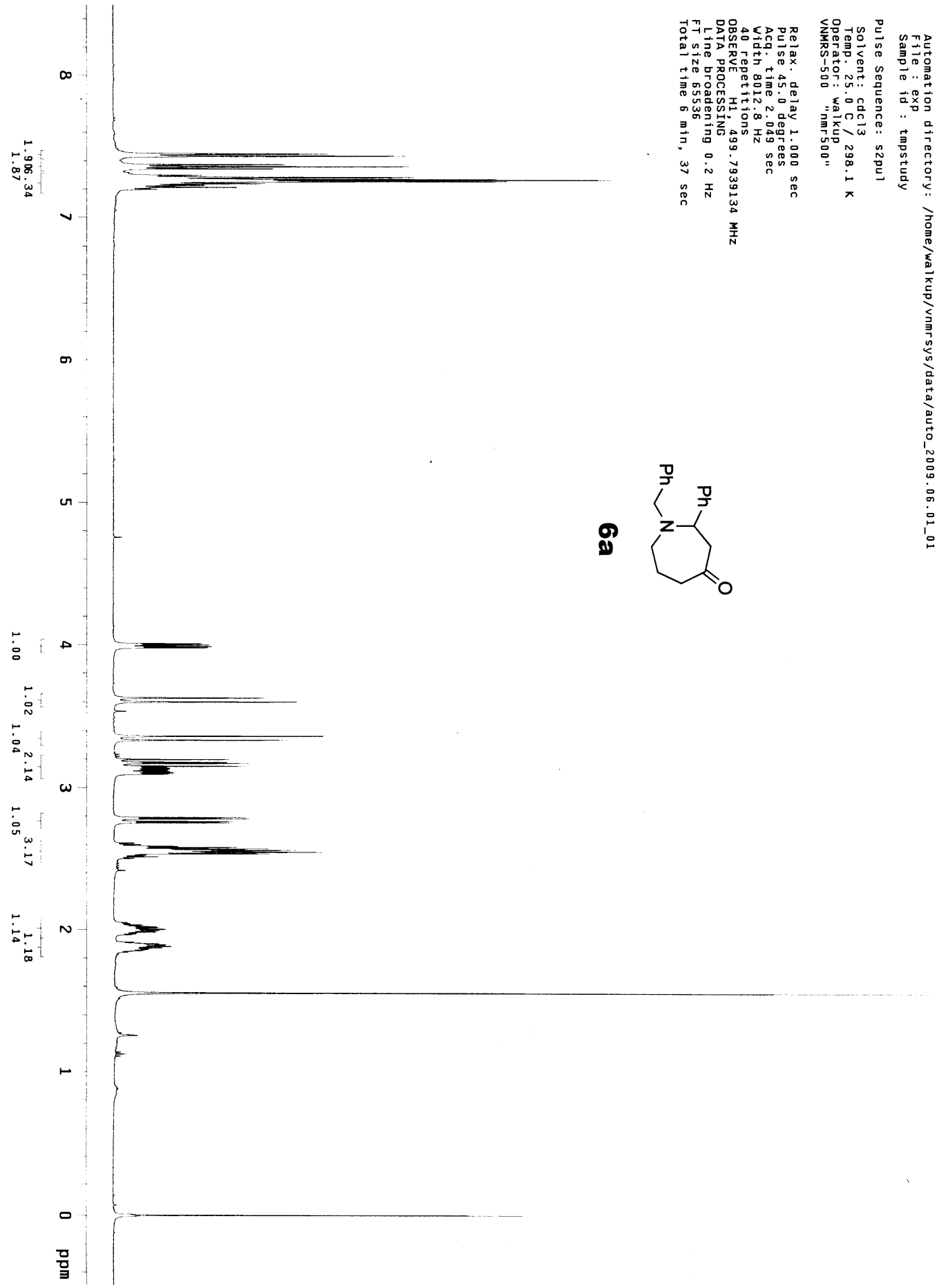
Temp. 24.8 C / 297.9 K
Operator: lcui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
76 repetitions
OBSERVE C13, 125.6889858 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



11



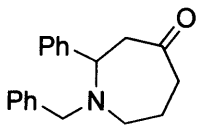

Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.01_01
File : exp
Sample id : tmpstudy
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
40 repetitions
OBSERVE H1, 499.7939134 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min, 37 sec
Ph
Ph
N
O
6a
8
7
6
5
4
3
2
1
0
ppm
1.90
6.34
1.87
1.00
1.02
2.14
1.04
1.05
3.17
1.18
1.14

licui-66-1-1H

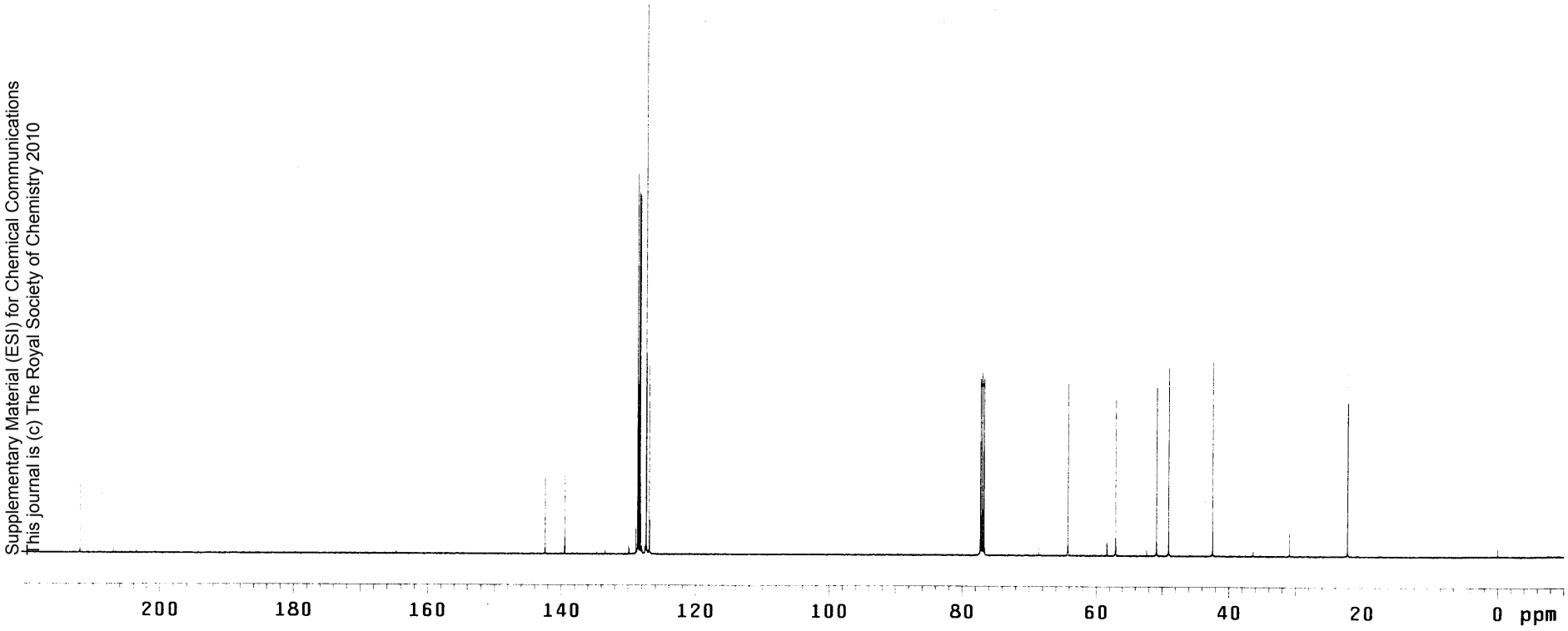
Automation directory: /home/walkup/vnmrsys/data/auto_2009.05.21
File : exp
Sample id : tmpstudy

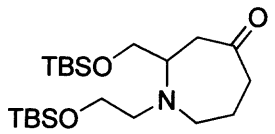
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
5896 repetitions
OBSERVE C13, 125.6732813 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec



6a





6b

STANDARD CARBON PARAMETERS

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

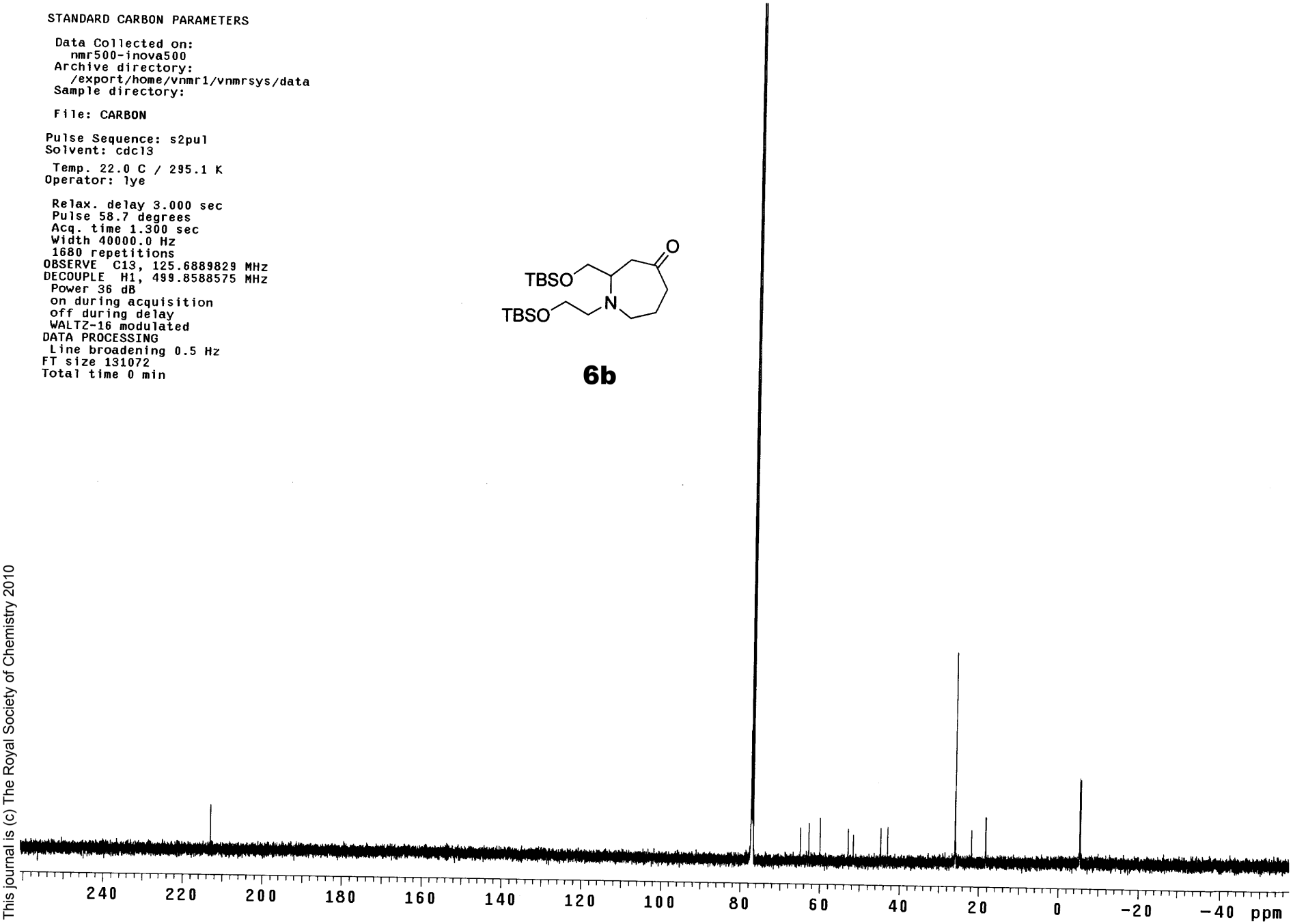
File: CARBON

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
1680 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

6b



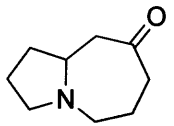
licui-53-1-F1

Archive directory:
Sample directory:

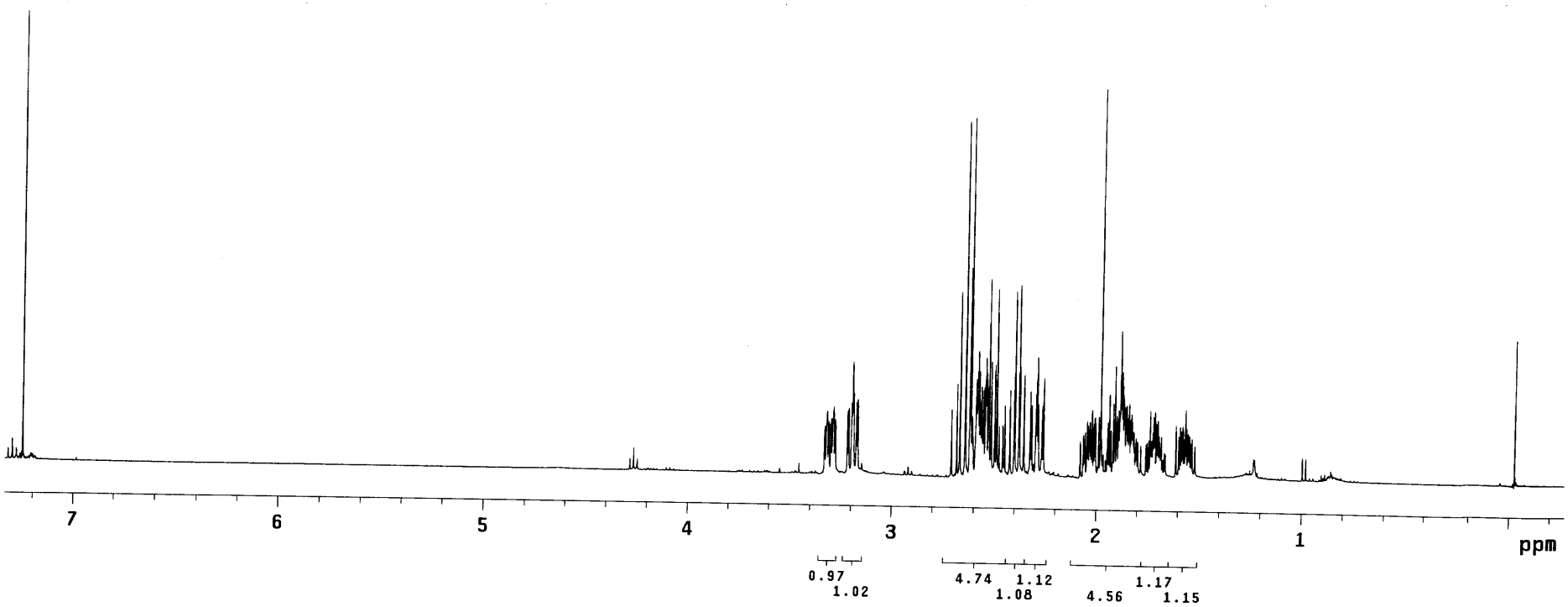
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: licui-53-1-F1
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
28 repetitions
OBSERVE H1, 400.0565537 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 6 min, 31 sec



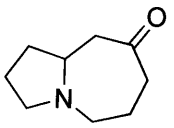
6c



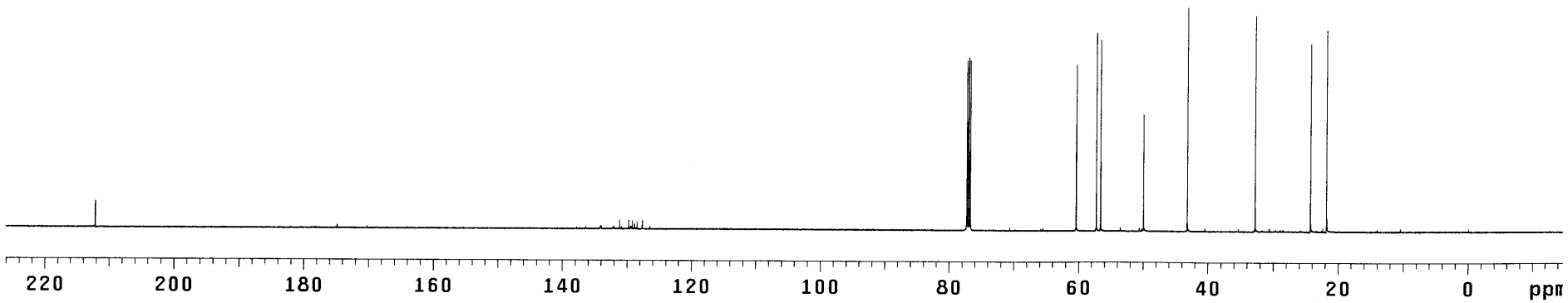
Archive directory:
Sample directory:

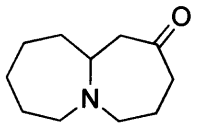
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: licui-53-1-F1-13Ca
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
17364 repetitions
OBSERVE C13, 125.6732809 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec

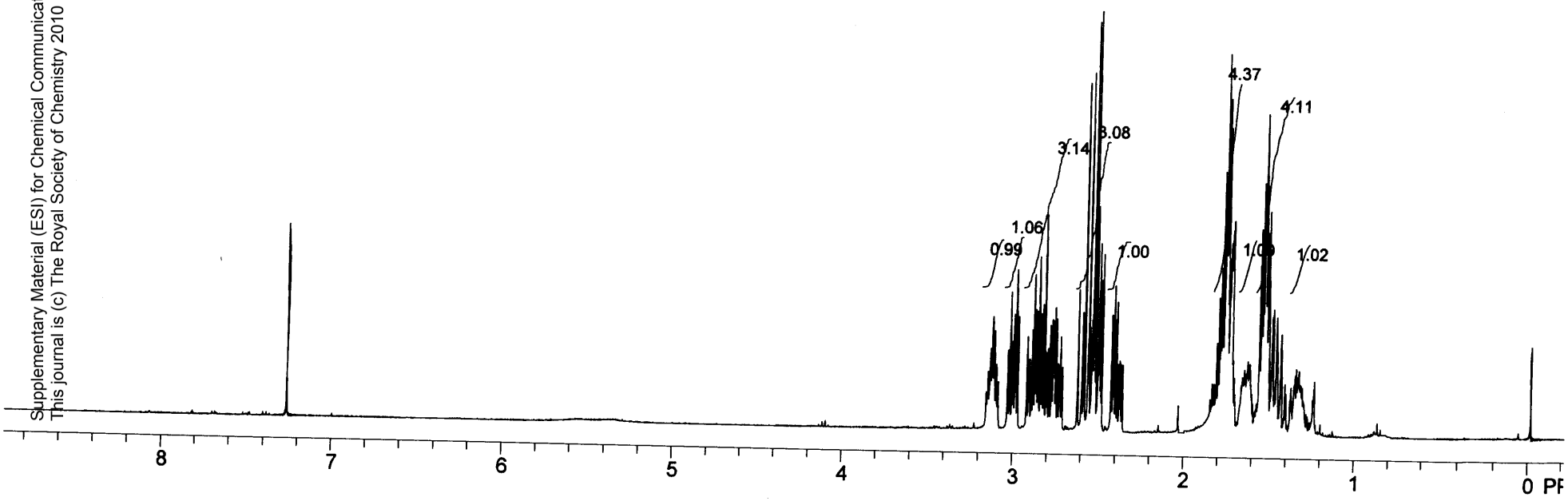


6c





6d



STANDARD CARBON PARAMETERS

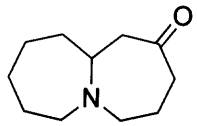
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

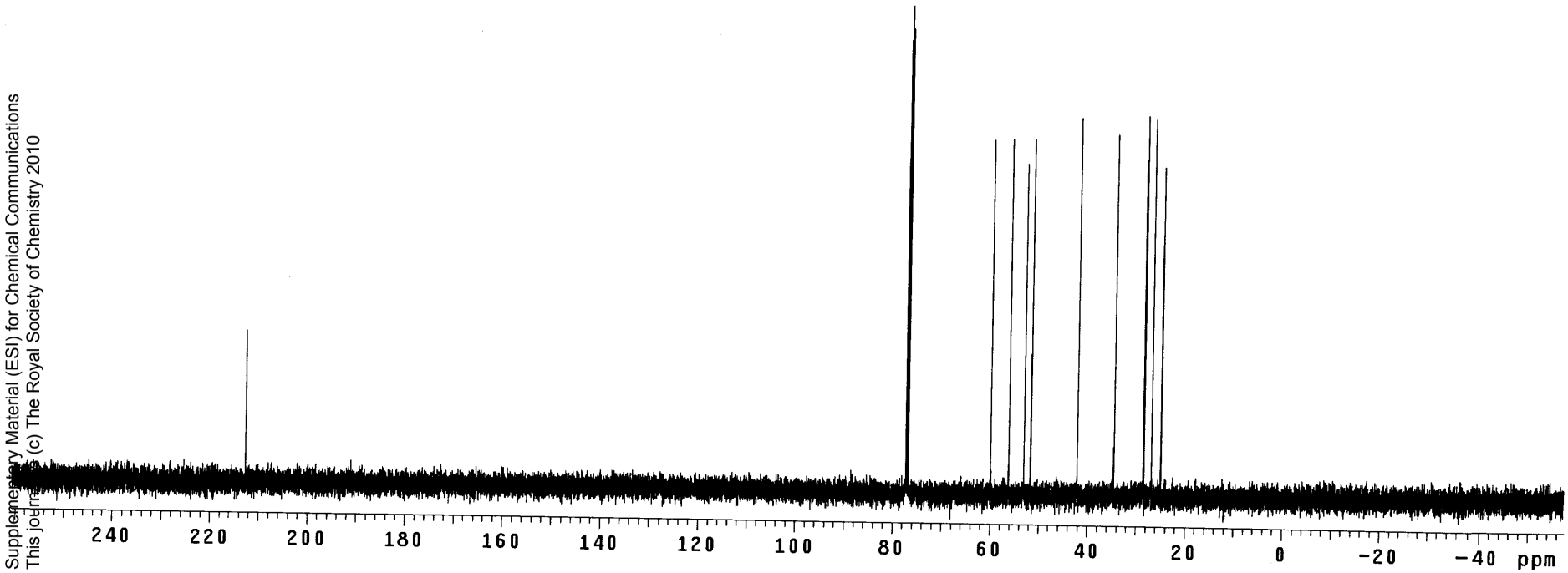
Pulse Sequence: s2pul
Solvent: cdcl3

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
72 repetitions
OBSERVE C13, 125.6889890 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



6d

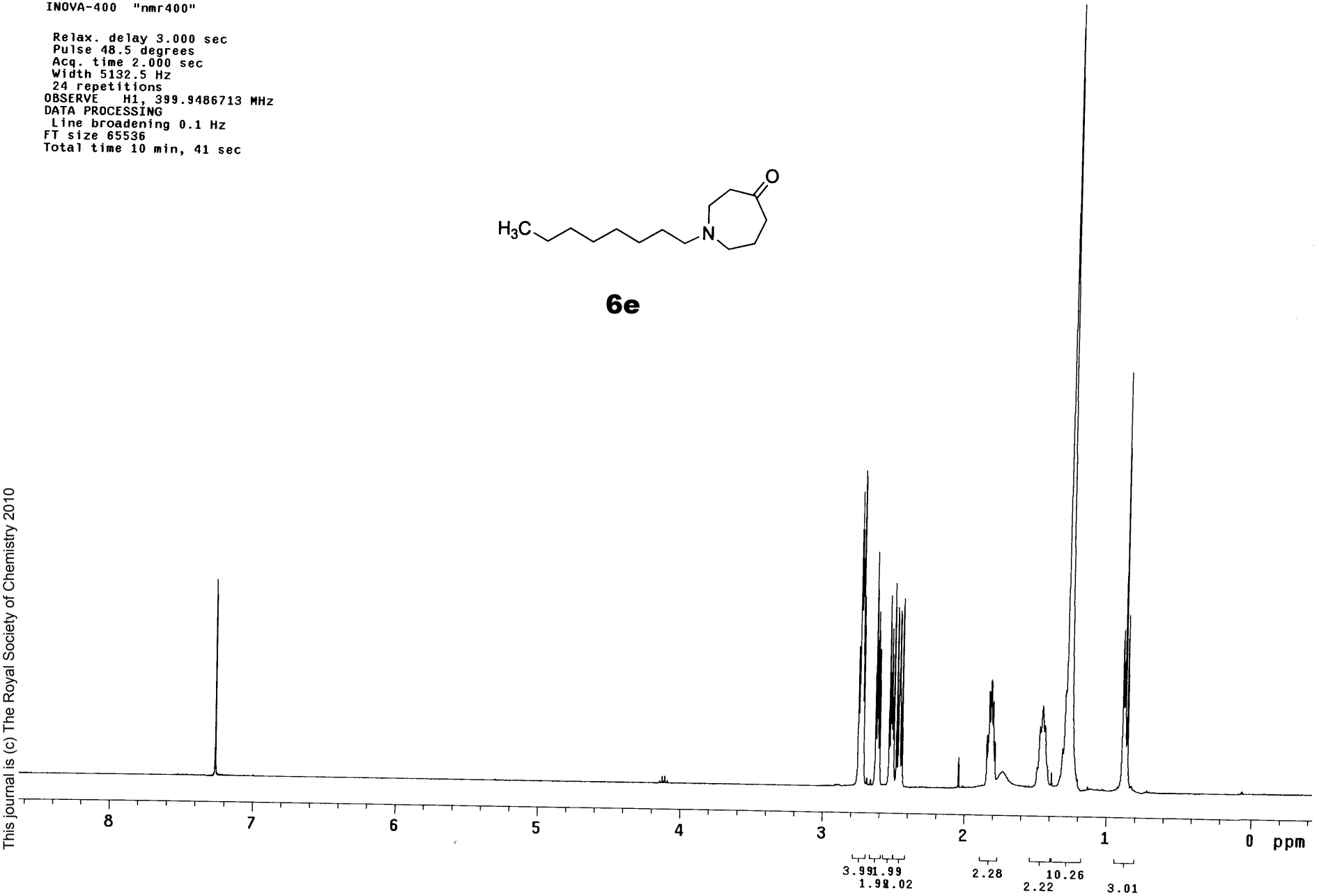


licui-106-1-1H

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
24 repetitions
OBSERVE H1, 399.9486713 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec

6e



licui-103-1-13C

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

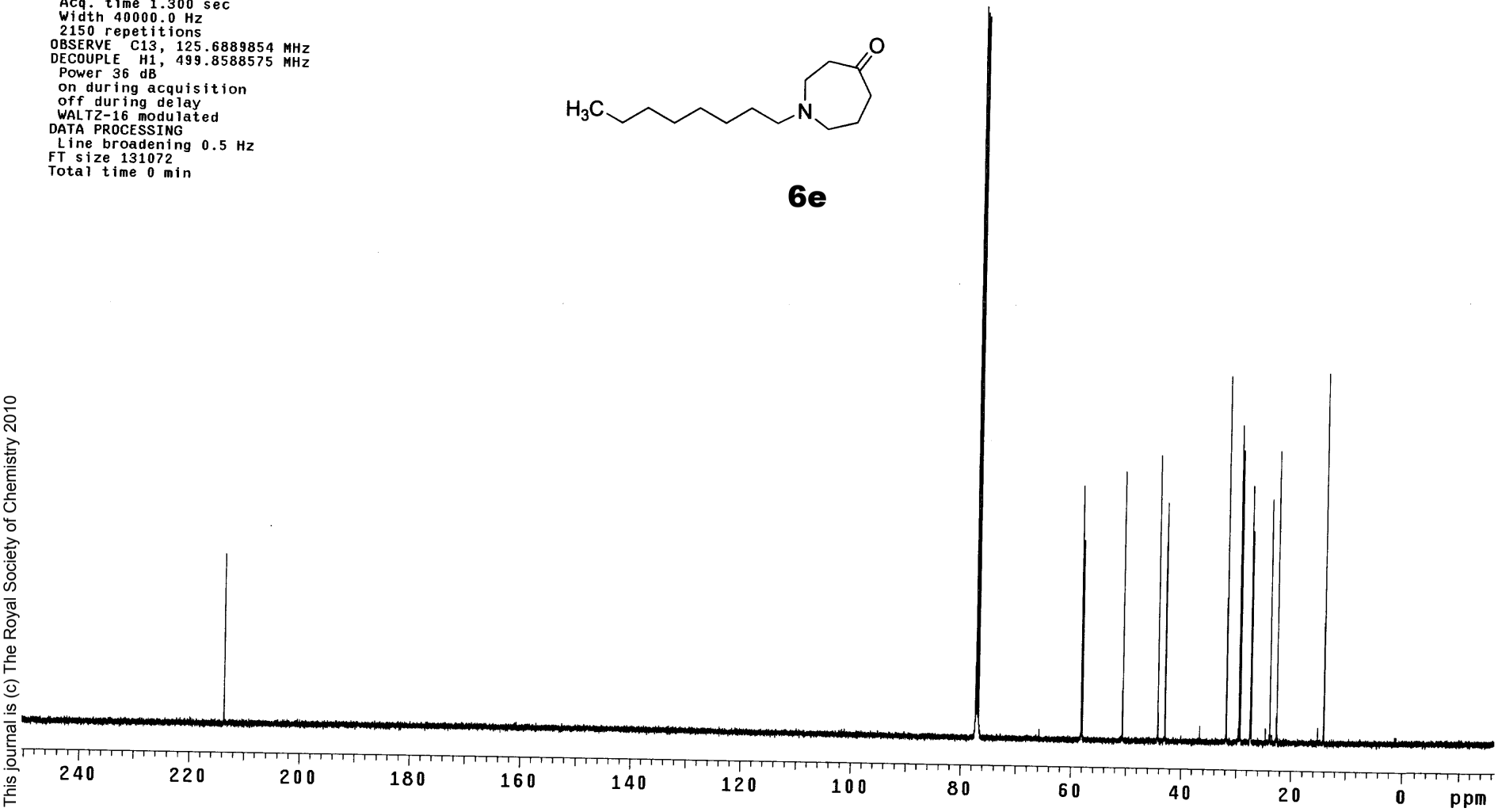
File: CARBON

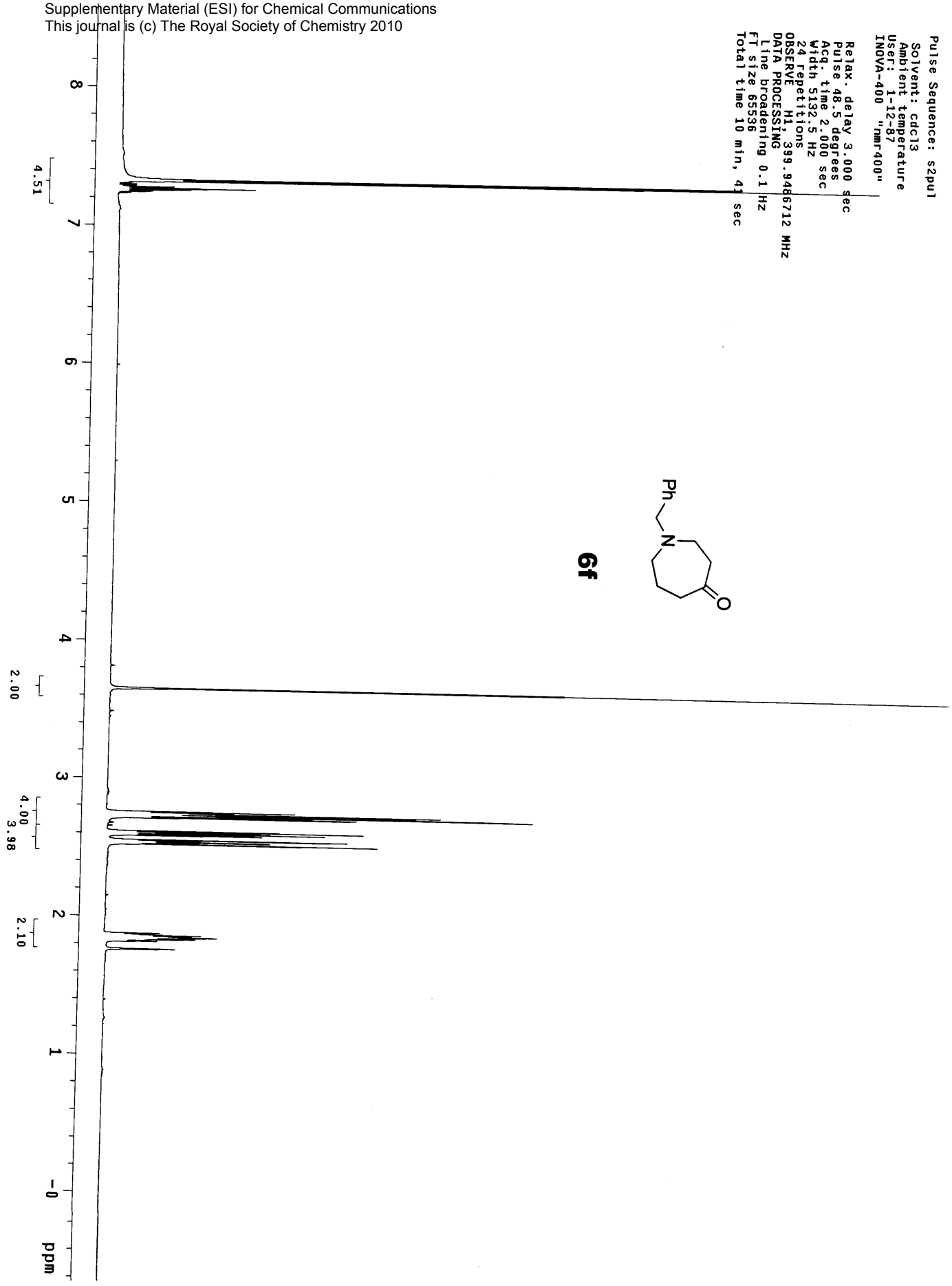
Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 22.0 C / 295.1 K
Operator: lcui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
2150 repetitions
OBSERVE C13, 125.6889854 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

6e




licui-112-1-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "nmr400"
Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
width 5132.5 Hz
24 repetitions
OBSERVE H1, 399.9486712 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec
Ph
N
O
6f
4.51
2.00
4.00
3.98
2.10
8
7
6
5
4
3
2
1
-0
ppm

licui-109-1-13C

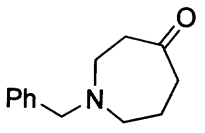
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

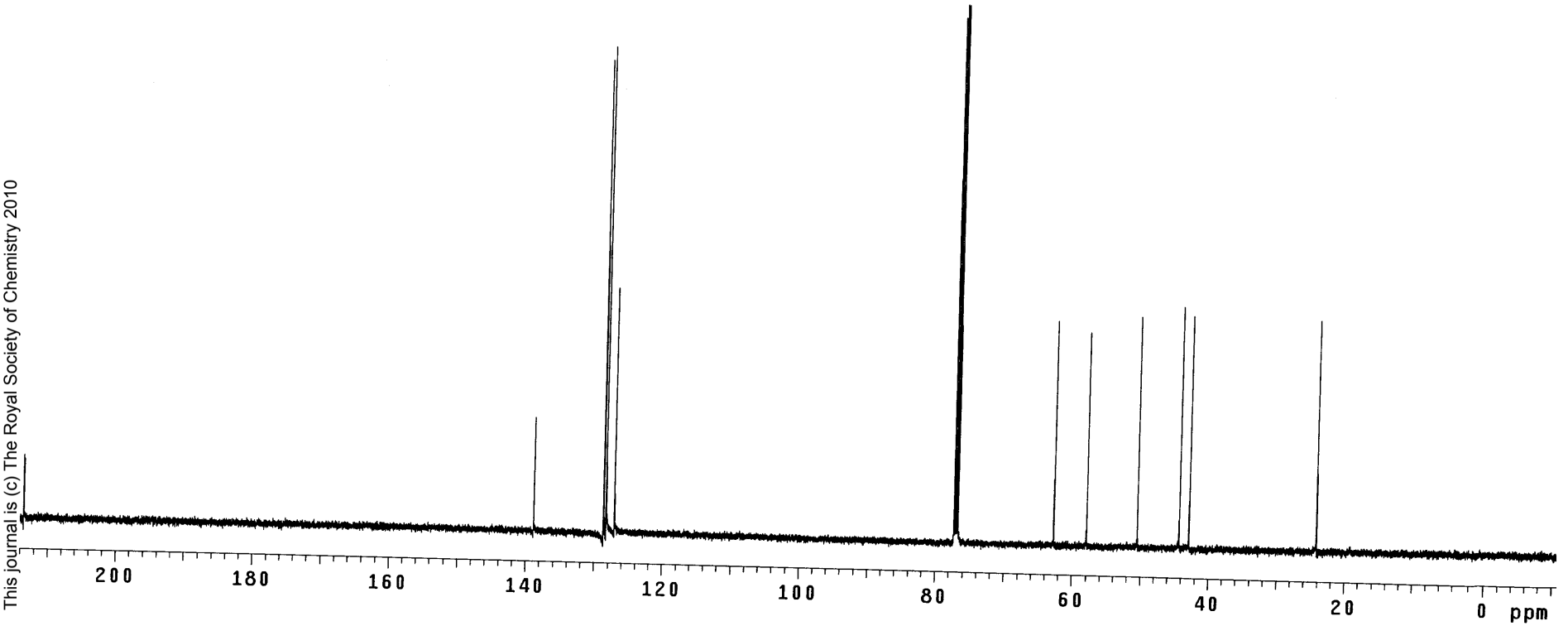
Pulse Sequence: s2pul
Solvent: cdcl3

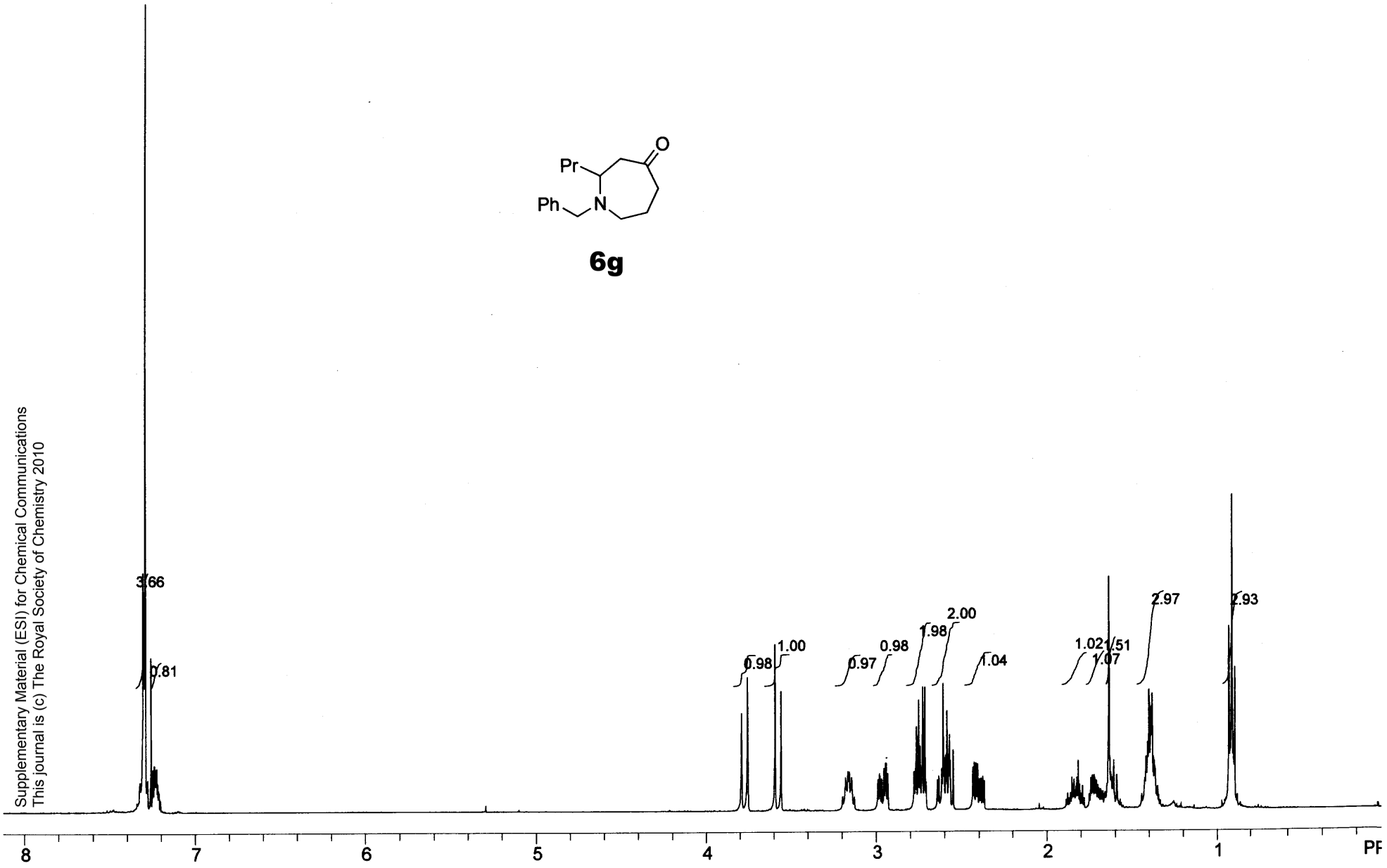
Temp. 25.0 C / 298.1 K
Operator: lcui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 28258.6 Hz
640 repetitions
OBSERVE C13, 125.6889858 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



6f





STANDARD CARBON PARAMETERS

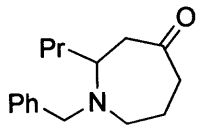
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

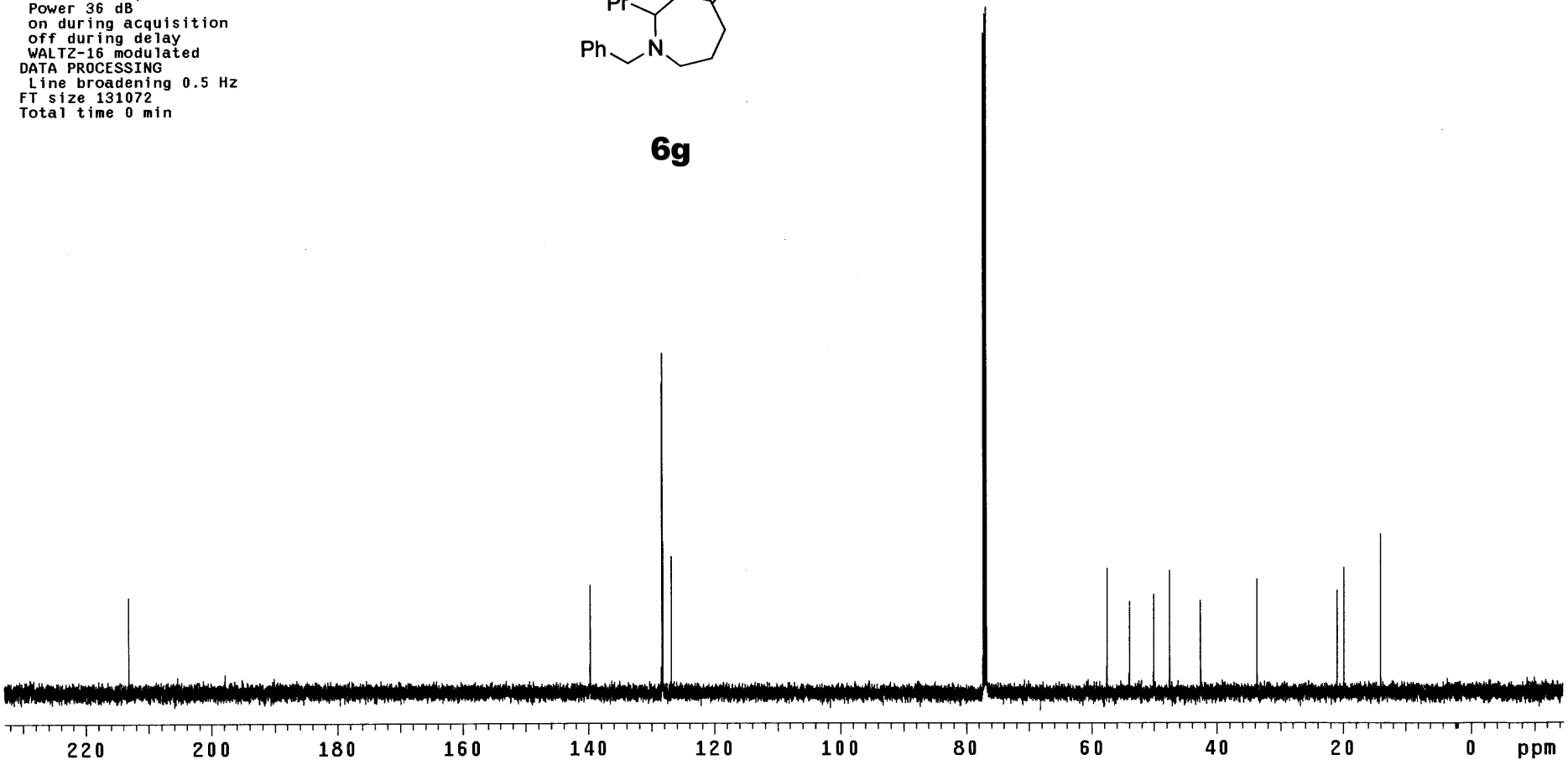
Pulse Sequence: s2pul
Solvent: cdcl3

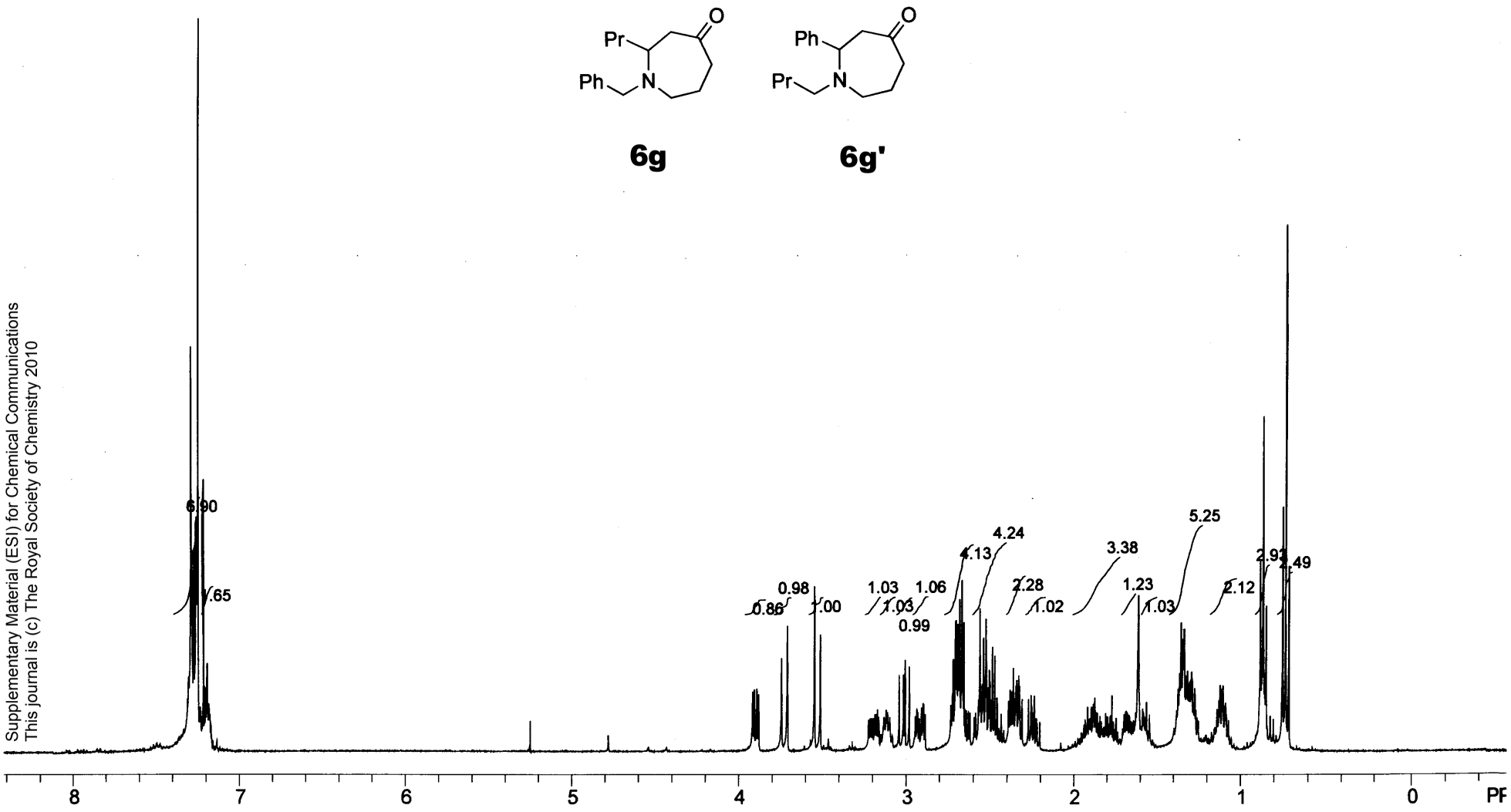
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
216 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



6g





ylw-1-231-mixture-c

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

File: CARBON

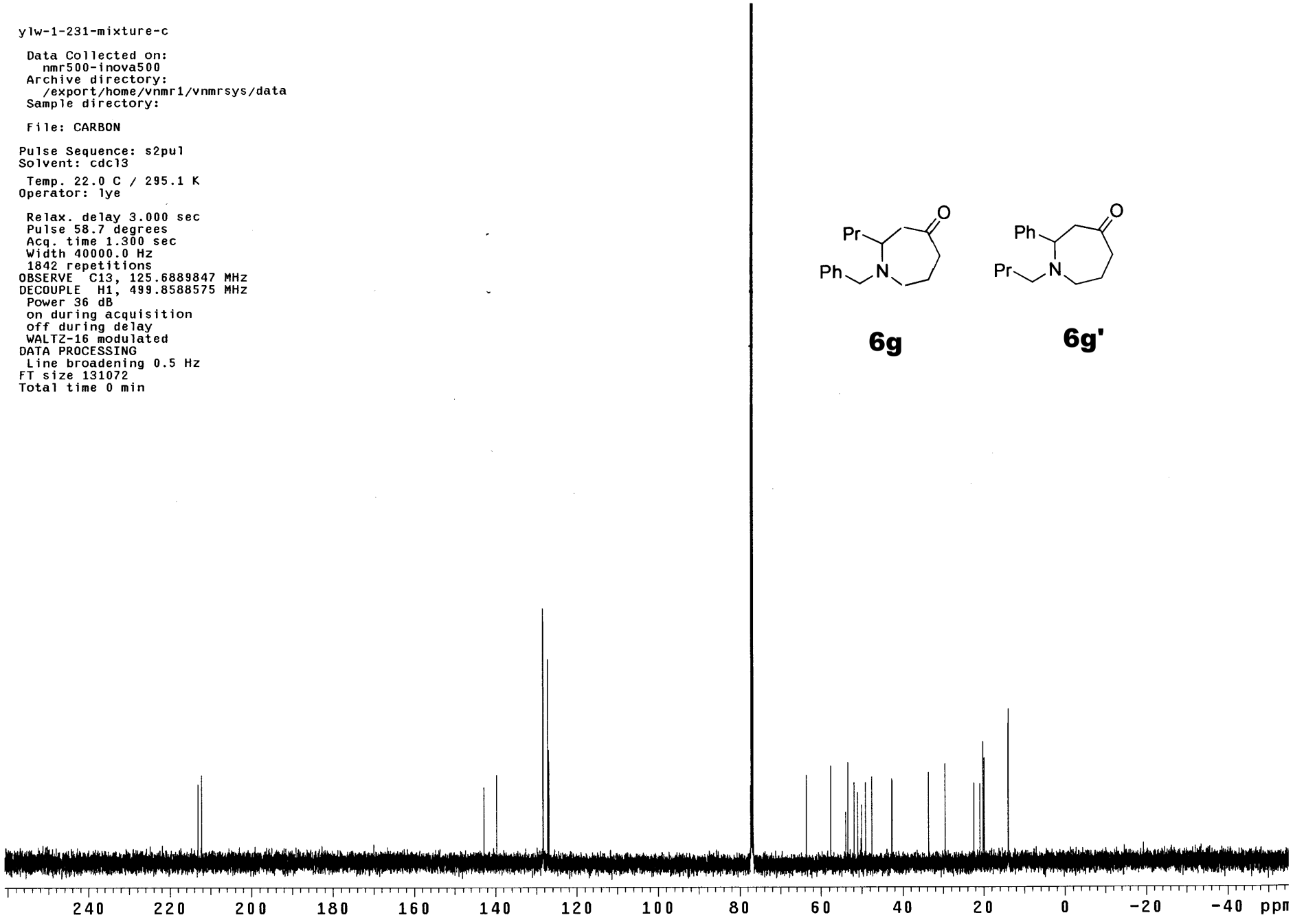
Pulse Sequence: s2pul
Solvent: cdcl3

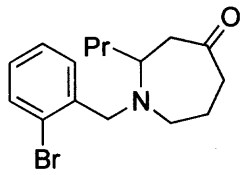
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1842 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

6g

6g'





6h

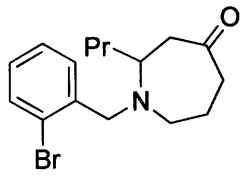
ylw-1-252-c

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

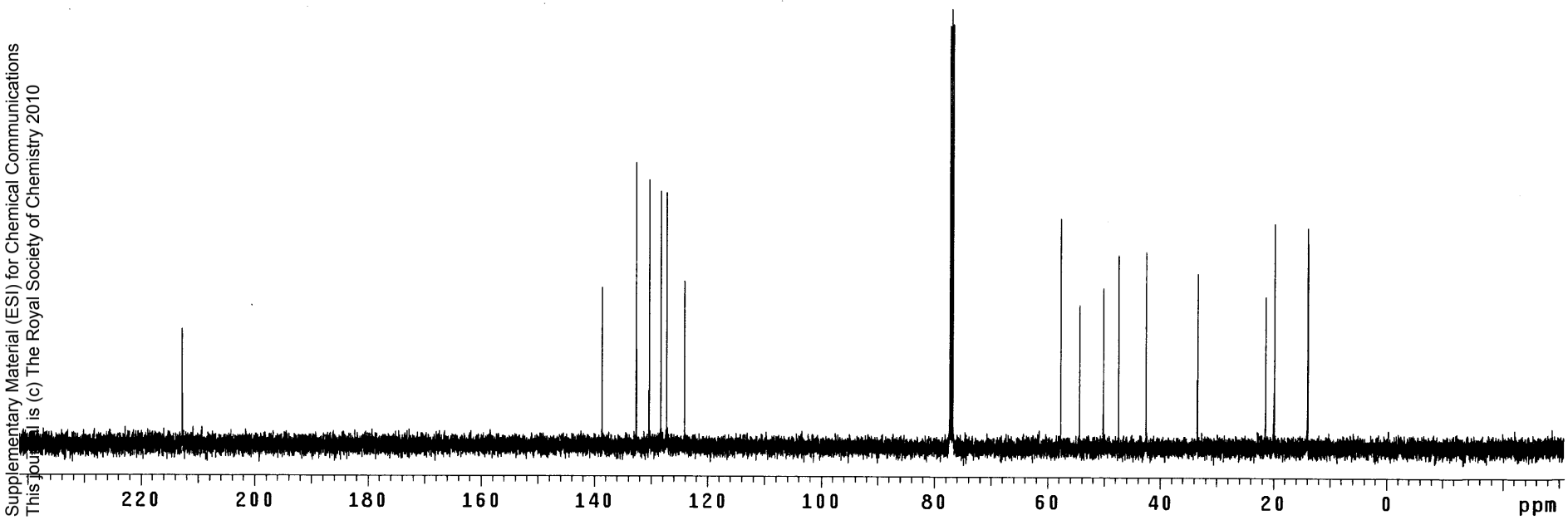
File: CARBON

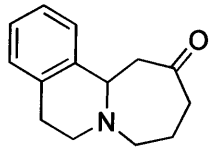
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
126 repetitions
OBSERVE C13, 125.6889896 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



6h





6i

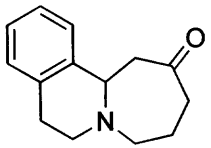
STANDARD CARBON PARAMETERS

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

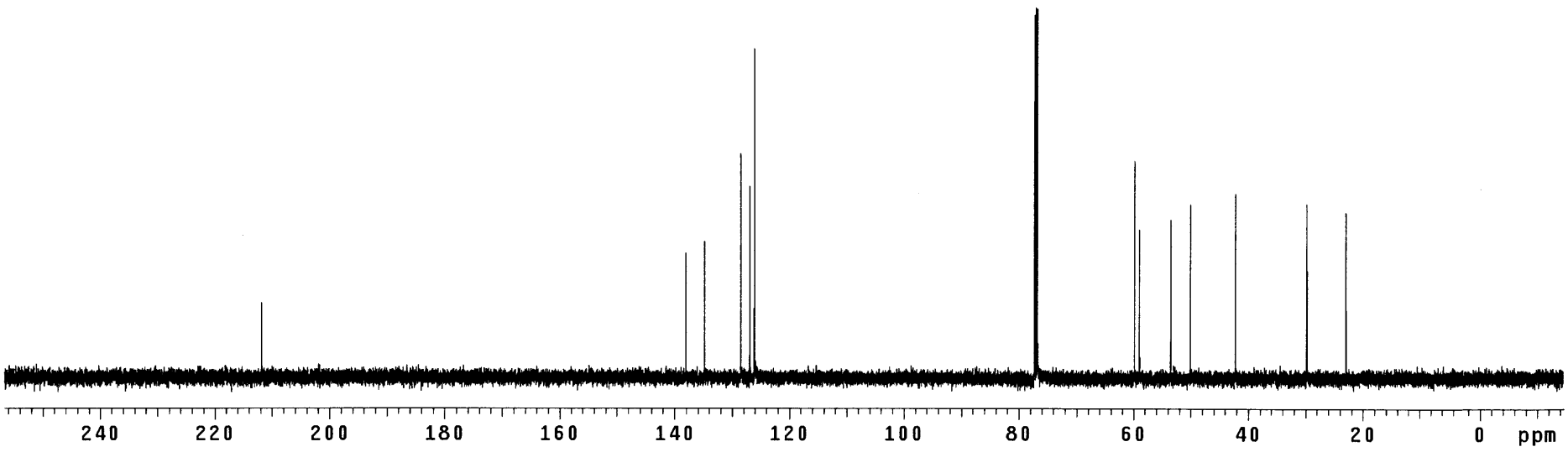
File: CARBON

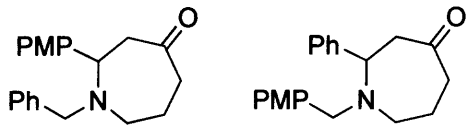
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
92 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

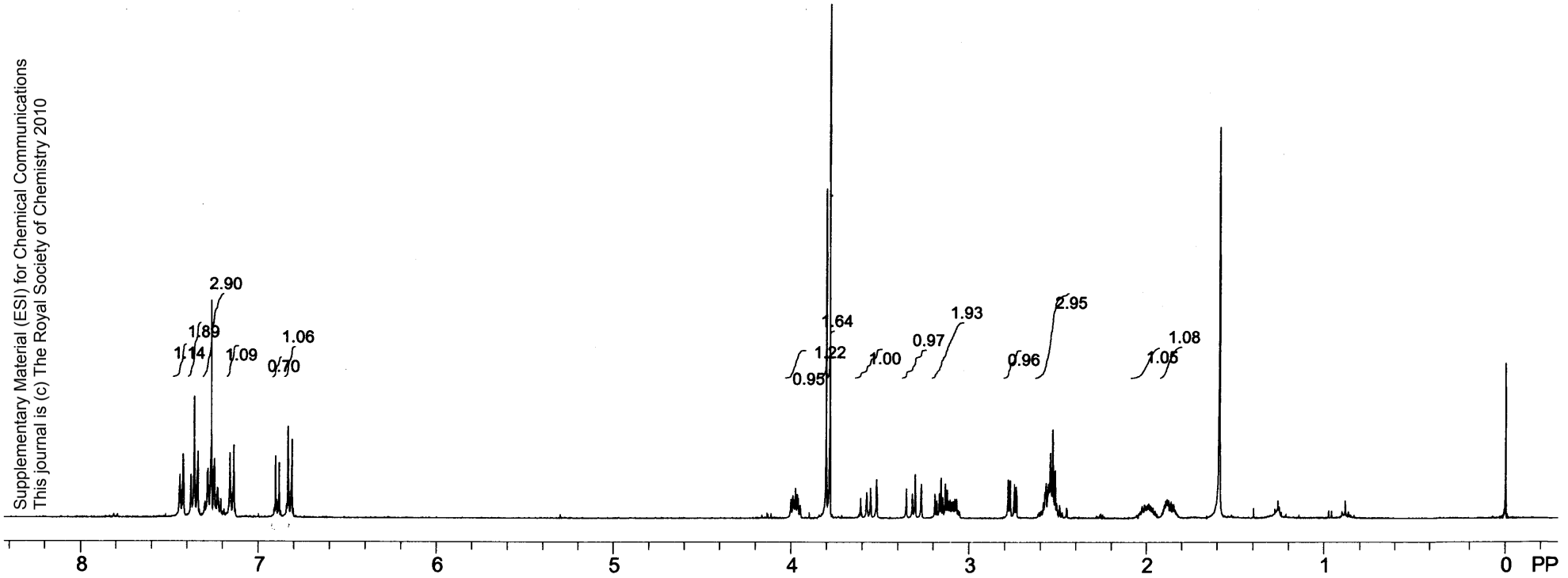


6i





6j **6j'**



STANDARD CARBON PARAMETERS

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

File: CARBON

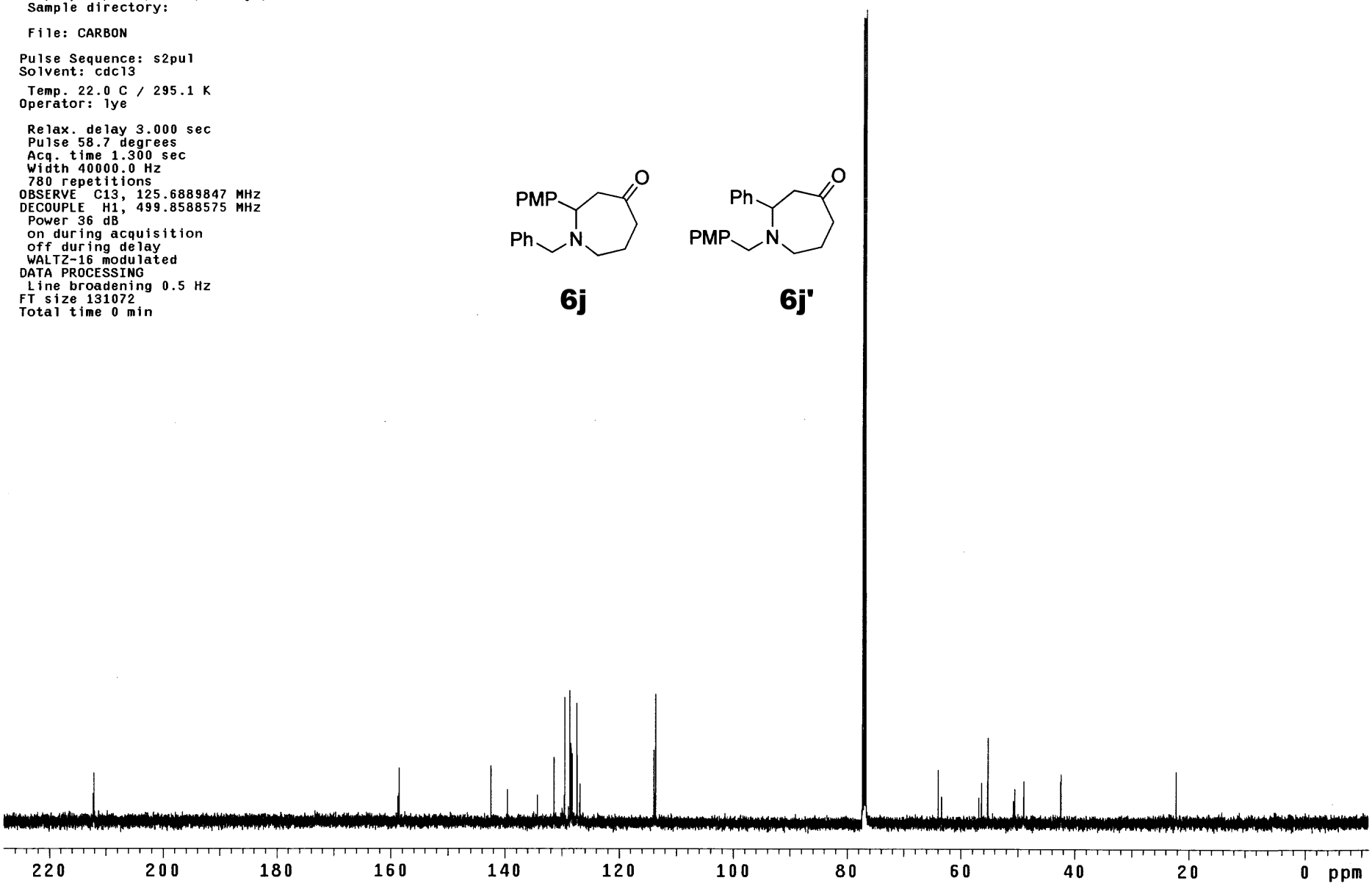
Pulse Sequence: s2pul
Solvent: cdcl3

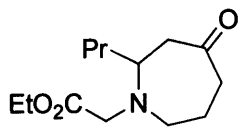
Temp. 22.0 C / 295.1 K
Operator: lye

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
780 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

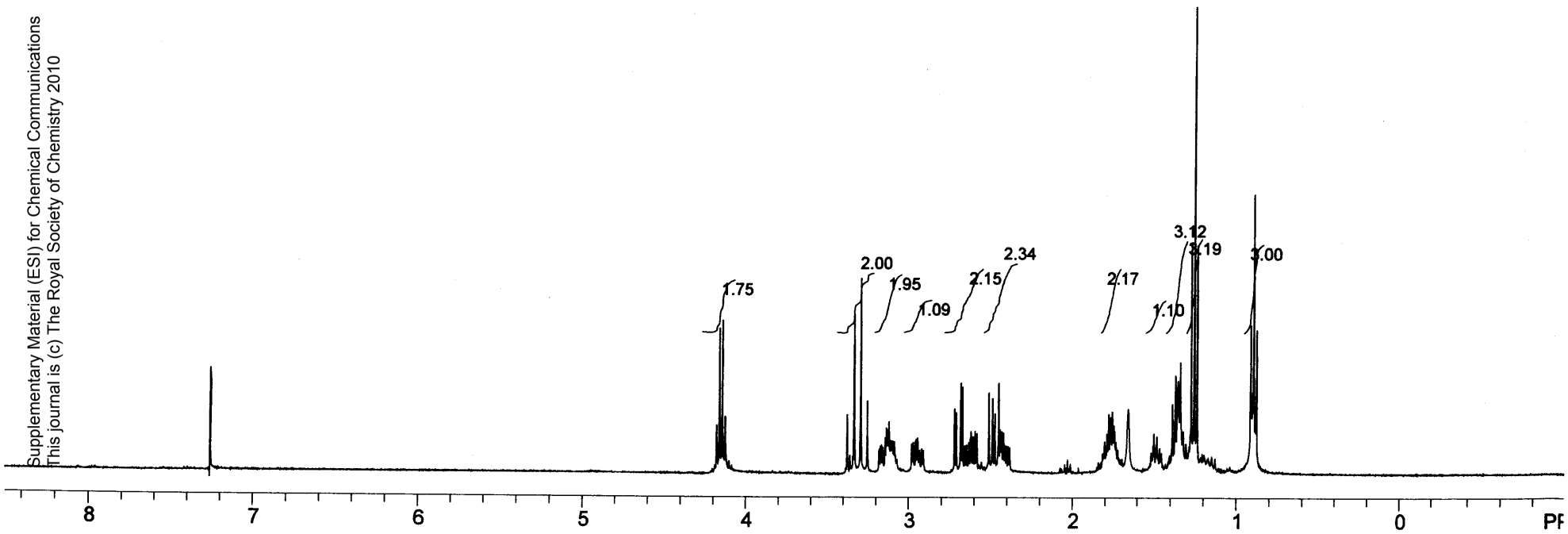
6j

6j'

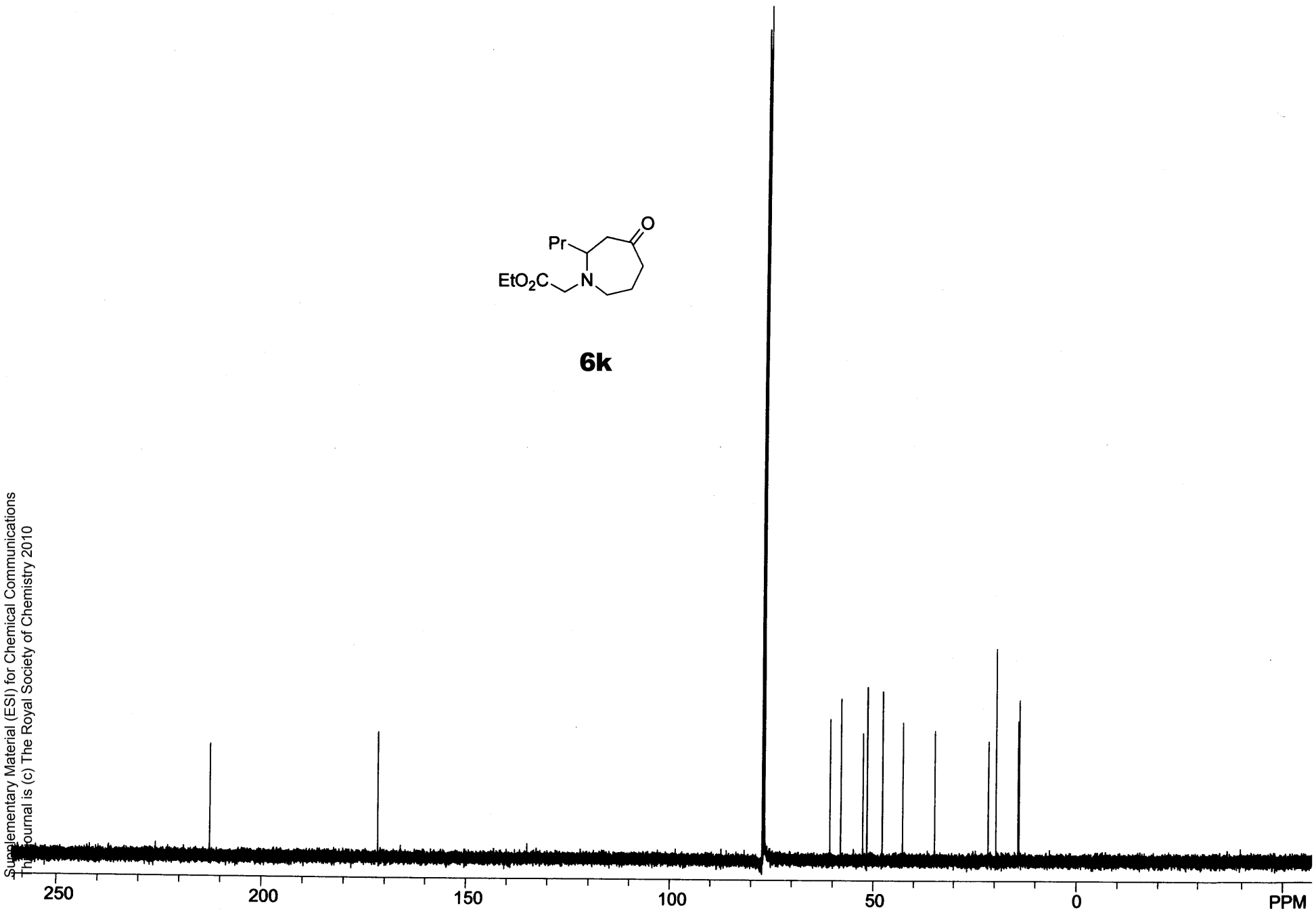


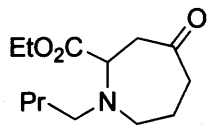


6k

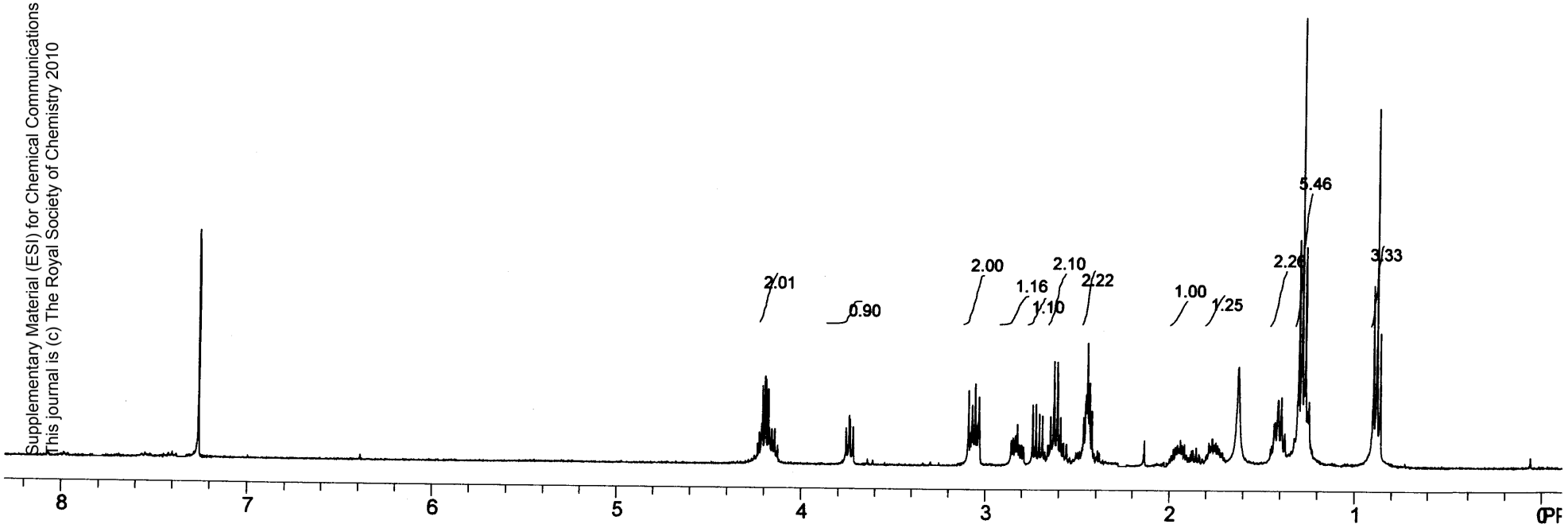


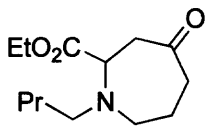
6k





6k'





6k'

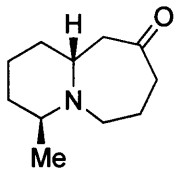
licui-87-1-1H

Archive directory:
Sample directory:

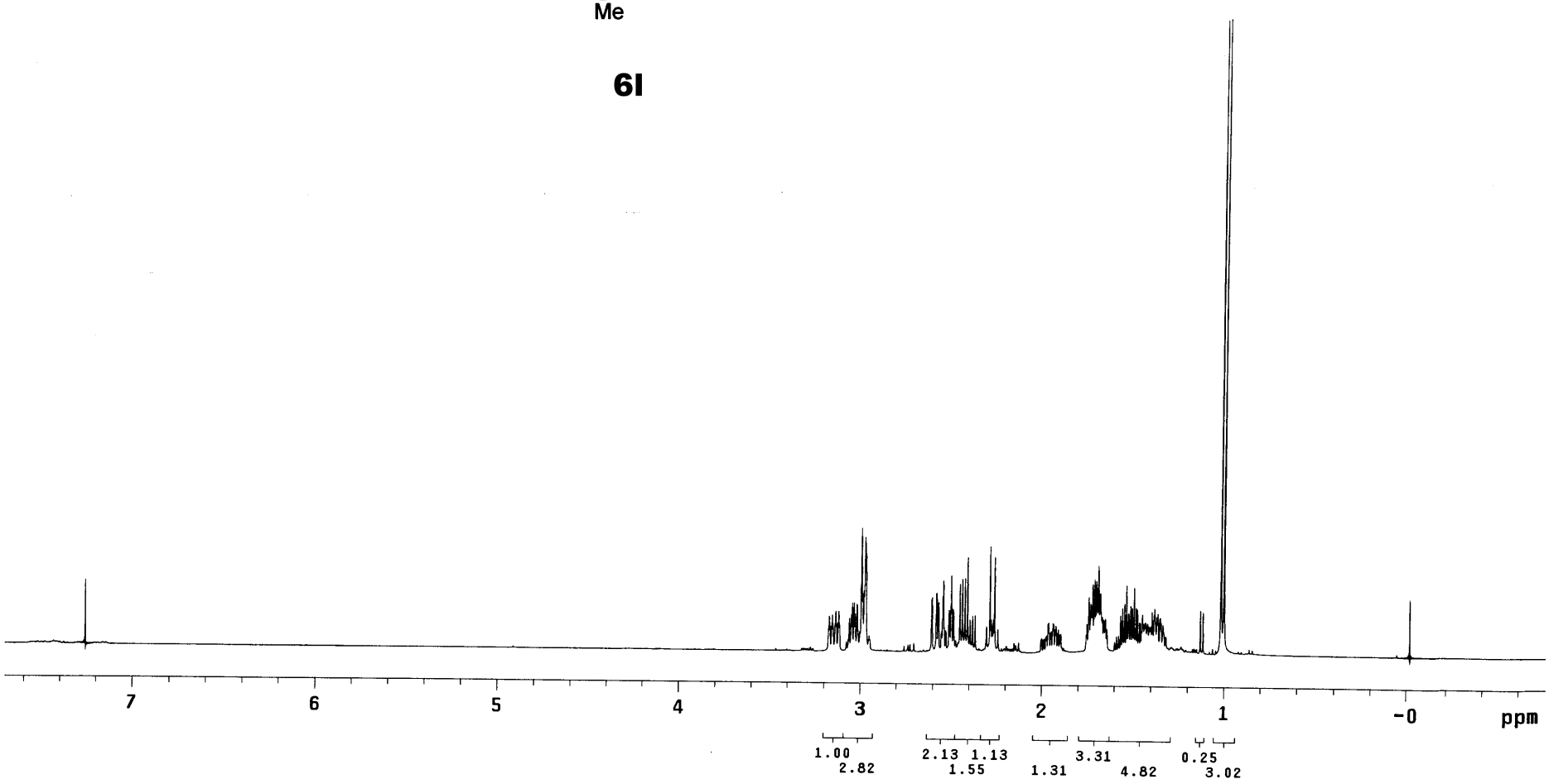
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: licui-87-1-1H
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
28 repetitions
OBSERVE H1, 400.0568165 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 6 min, 31 sec



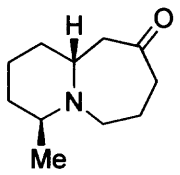
6l



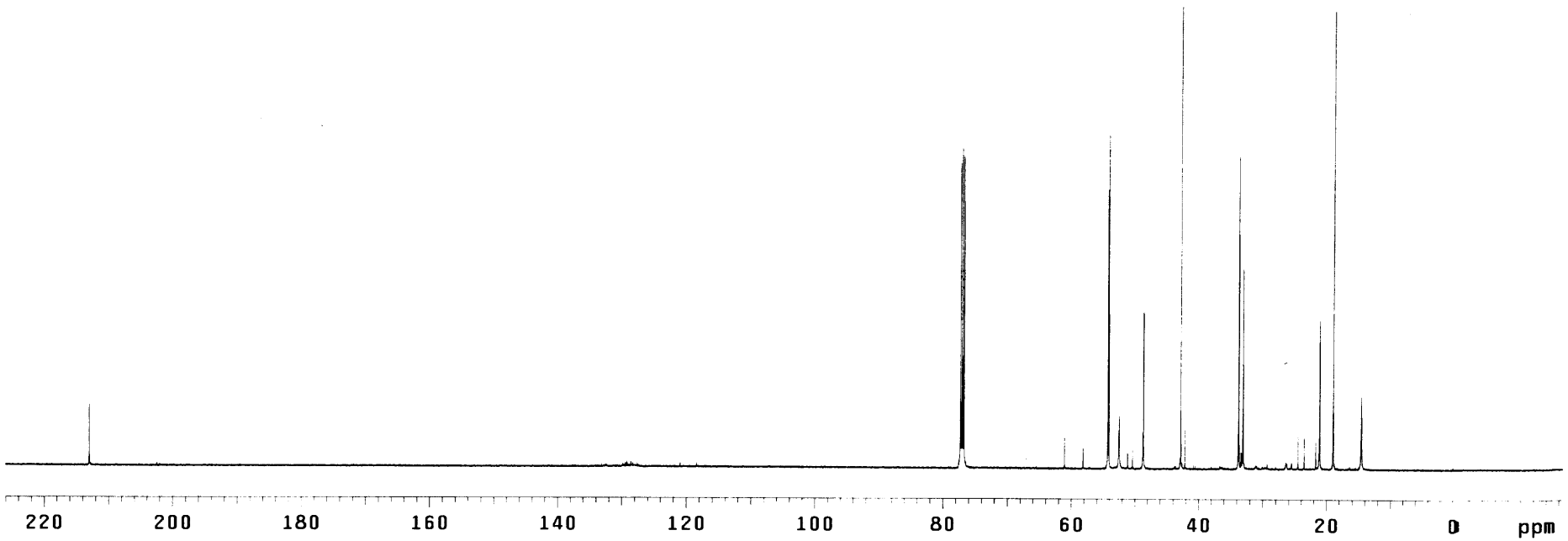
Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.08_04
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: walkup
VNMRS-500 "nmr500"

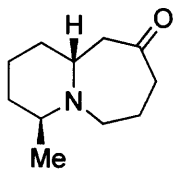
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
17508 repetitions
OBSERVE C13, 125.6732827 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec



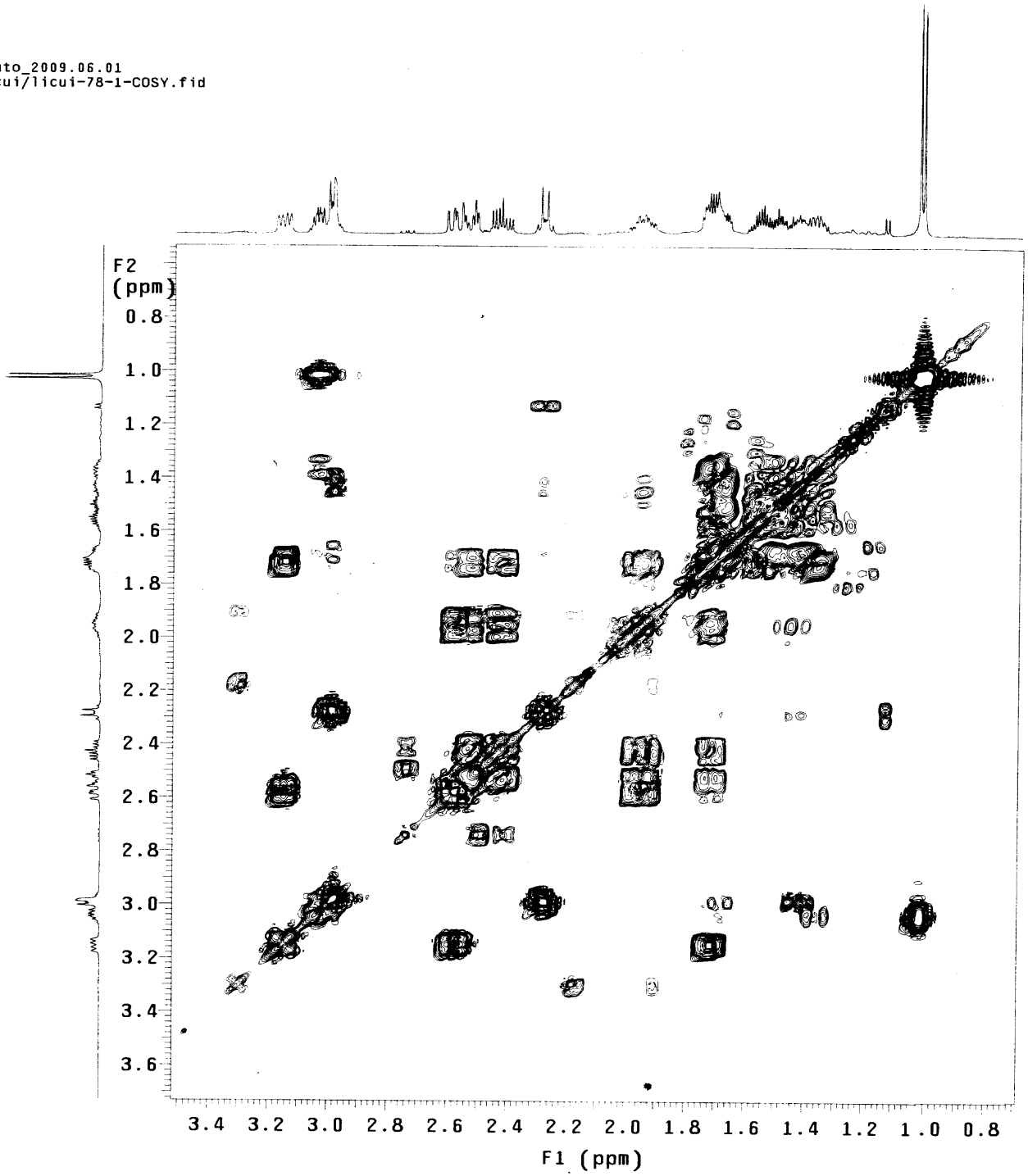
6l



```
  Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.01
  File : /home/walkup/vnmrsys/data/ARGENTA/Zhang/licui/licui-78-1-COSY.fid
  Sample id : s_20090601_001

Pulse Sequence: gCOSY
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: walkup
File: licui-78-1-COSY
VNMRS-500  "nmr500"

 Relax. delay 1.301 sec
 Acq. time 0.128 sec
 Width    8012.8 Hz
 2D Width 8012.8 Hz
 8 repetitions
 512 increments
OBSERVE   H1, 499.7939123 MHz
DATA PROCESSING
 Sine bell 0.064 sec
F1 DATA PROCESSING
 Sine bell 0.128 sec
FT size 8192 x 8192
Total time 1 hr, 40 min, 58 sec
```



6l

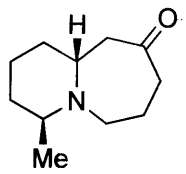


licui-80-1

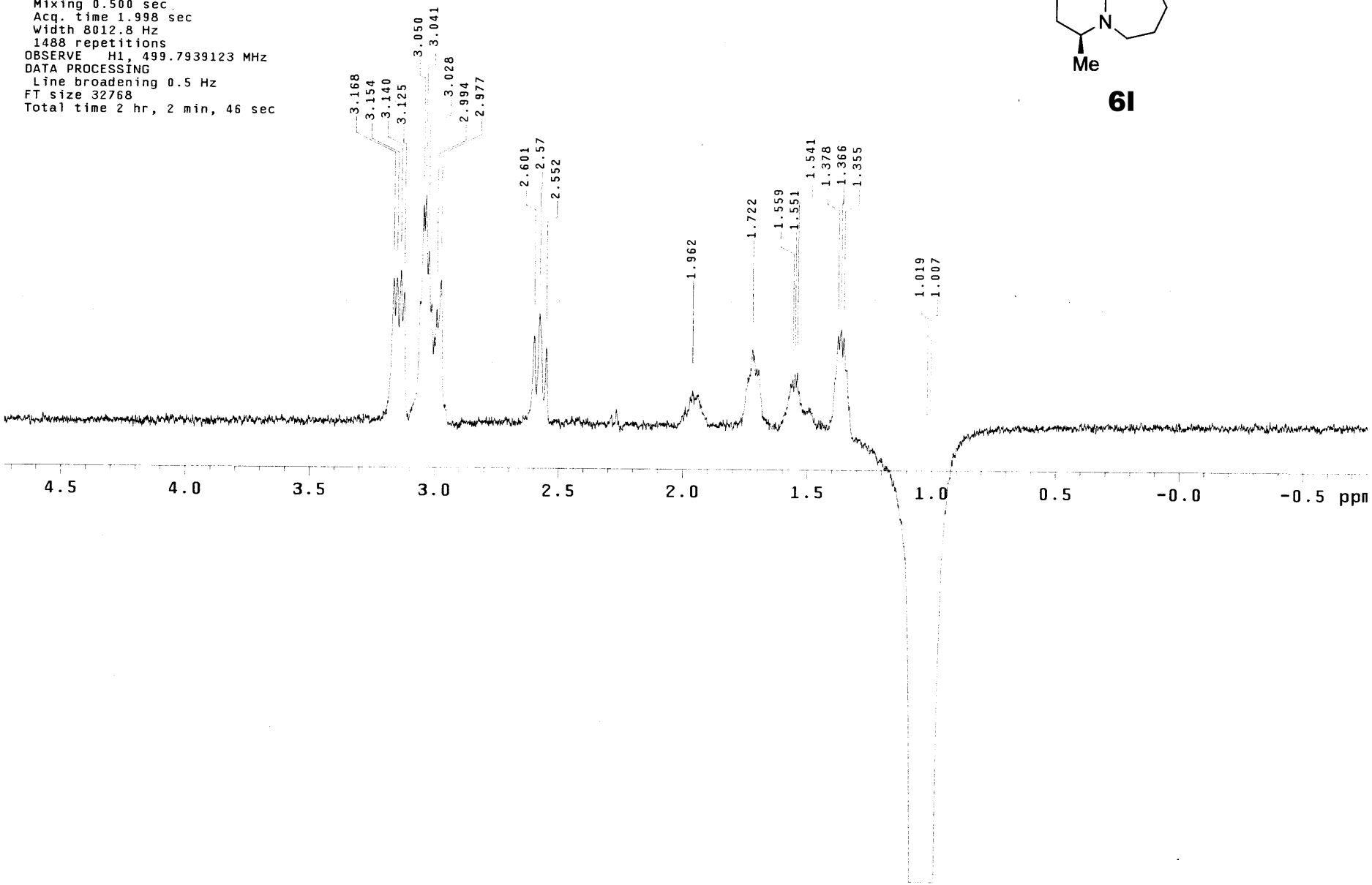
Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.02
File : exp
Sample id : tmpstudy

Pulse Sequence: NOESY1D
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.500 sec
Acq. time 1.998 sec
Width 8012.8 Hz
1488 repetitions
OBSERVE H1, 499.7939123 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 2 hr, 2 min, 46 sec



6l

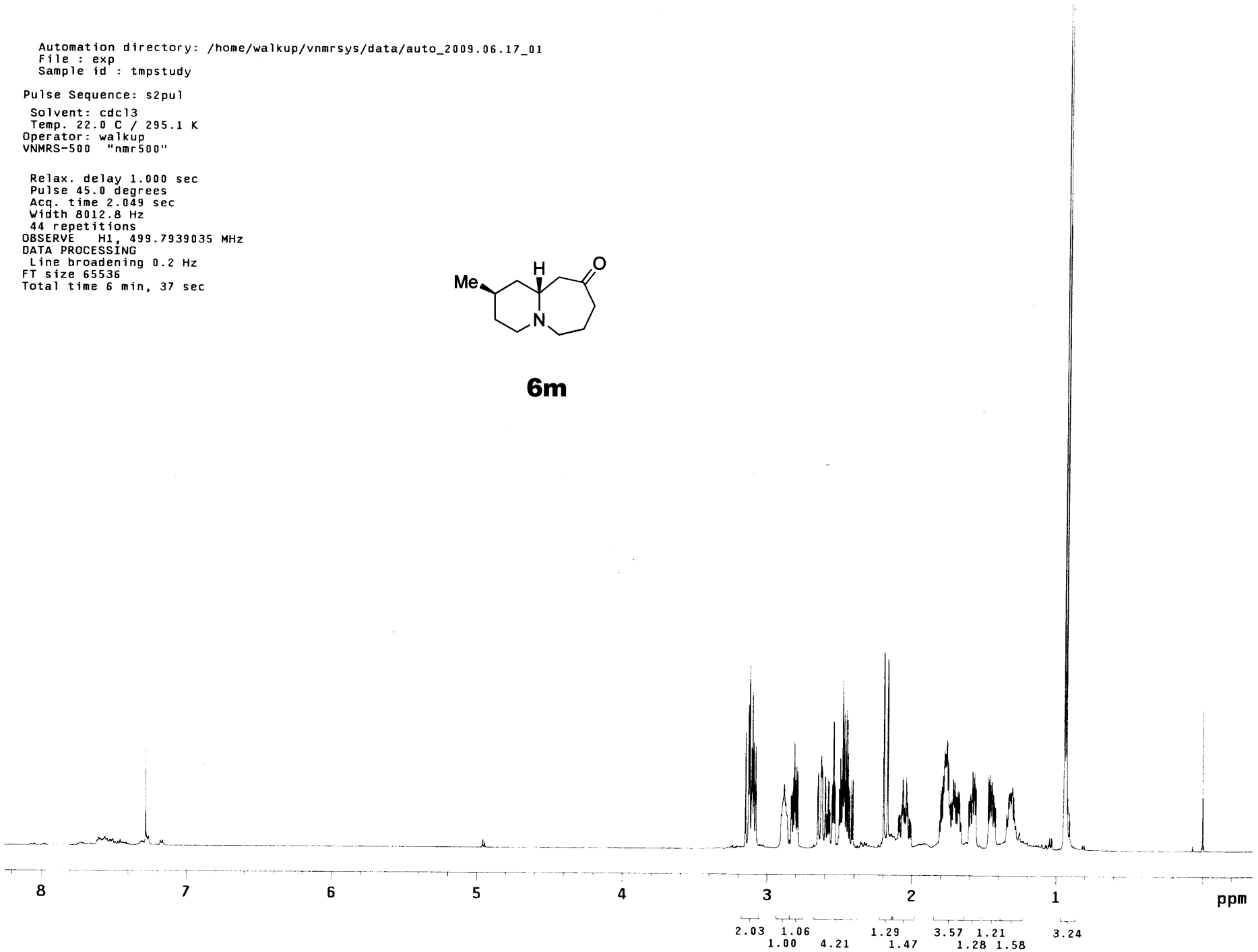


Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.17_01
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
44 repetitions
OBSERVE H1, 499.7939035 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min, 37 sec

6m

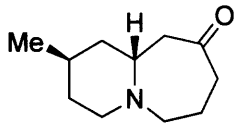


licui-95-1-13C

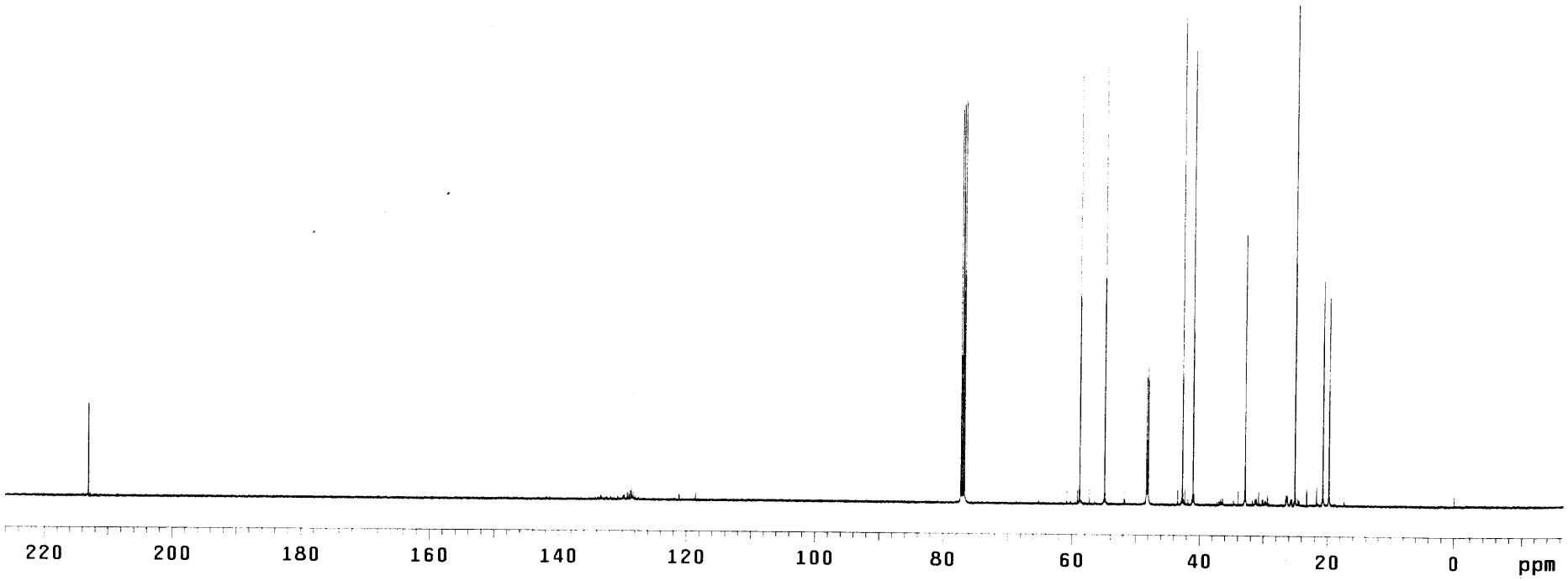
Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.18_03
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

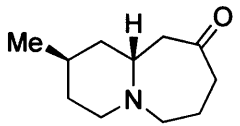
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
9348 repetitions
OBSERVE C13, 125.6732818 MHz
DECOUPLE H1, 499.7964114 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec



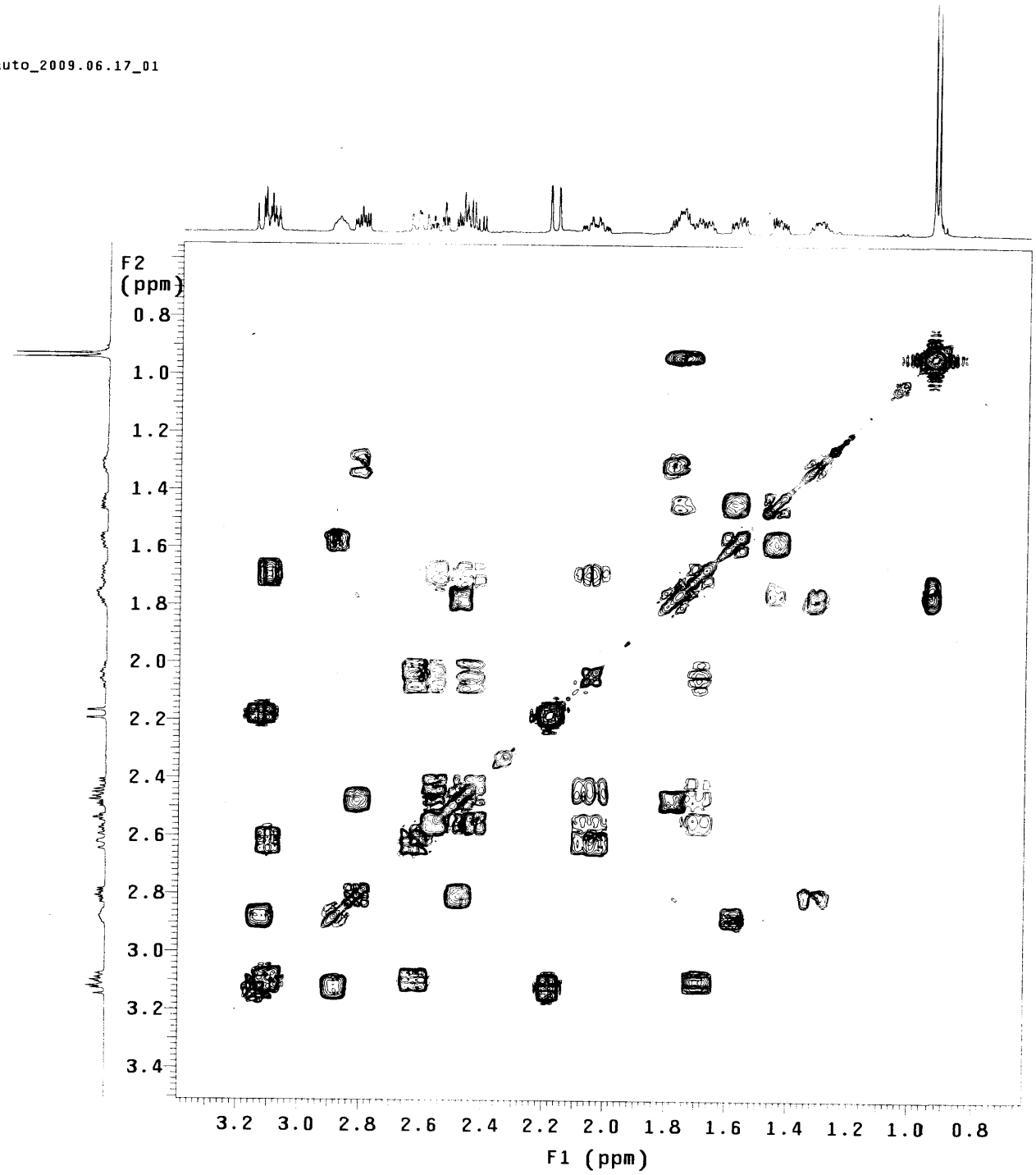
6m



```
  Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.17_01
  File : s_20090617_001/data/cdcl3_01.fid
  Sample id : s_20090617_001

Pulse Sequence: gCOSY
 Solvent: cdcl3
 Temp. 22.0 C / 295.1 K
Operator: walkup
File: cdcl3_01
VNMRS-500  "nmr500"

 Relax. delay 1.301 sec
 Acq. time 0.128 sec
 Width    8012.8 Hz
 2D Width 8012.8 Hz
 8 repetitions
 512 increments
OBSERVE   H1, 499.7939056 MHz
DATA PROCESSING
 Sine bell 0.064 sec
F1 DATA PROCESSING
 Sine bell 0.128 sec
FT size 8192 x 8192
Total time 1 hr, 40 min, 58 sec
```



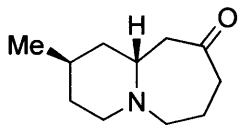
6m



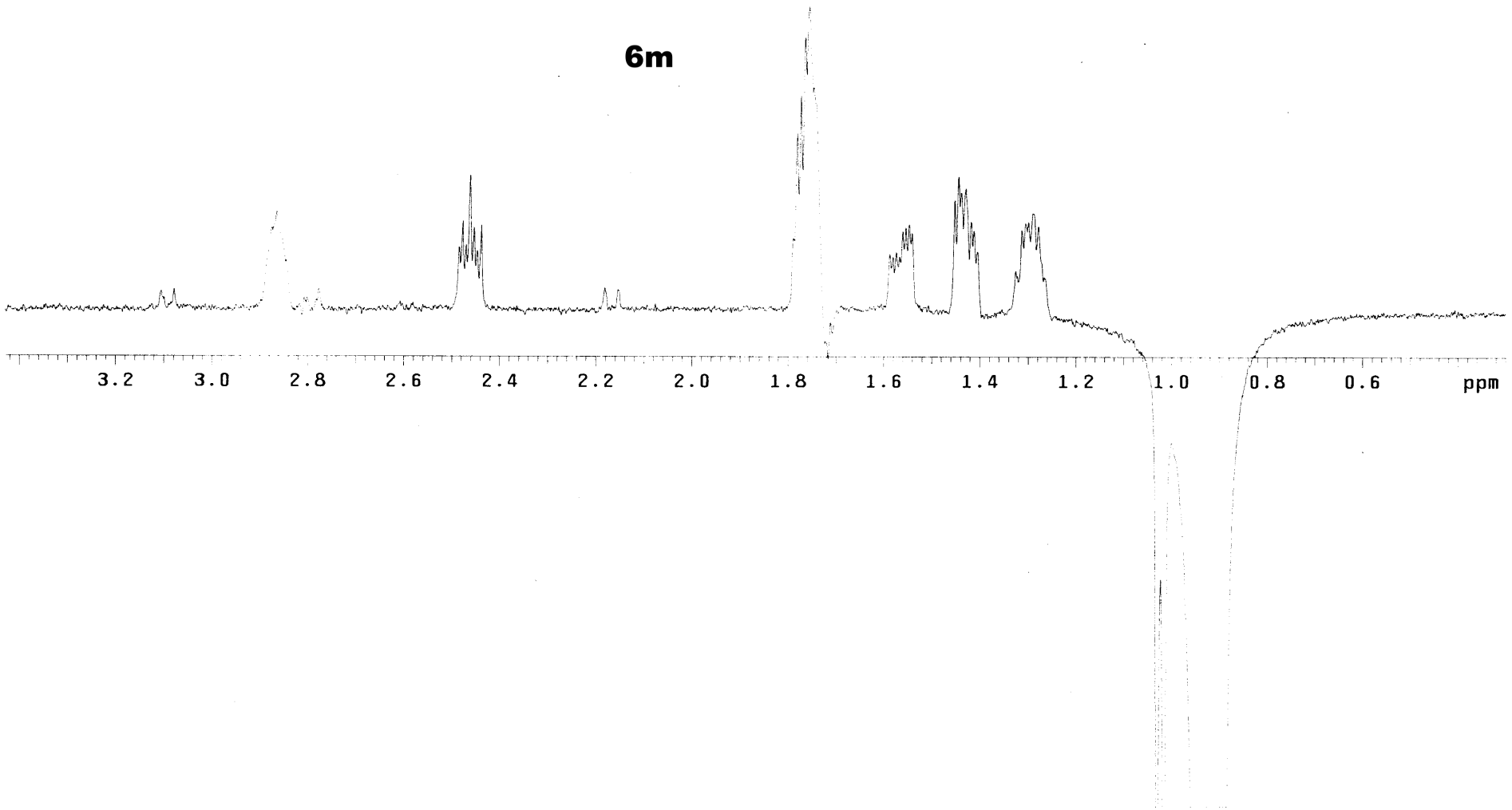
Automation directory: /home/walkup/vnmrsys/data/auto_2009.06.18_04
File : exp
Sample id : tmpstudy

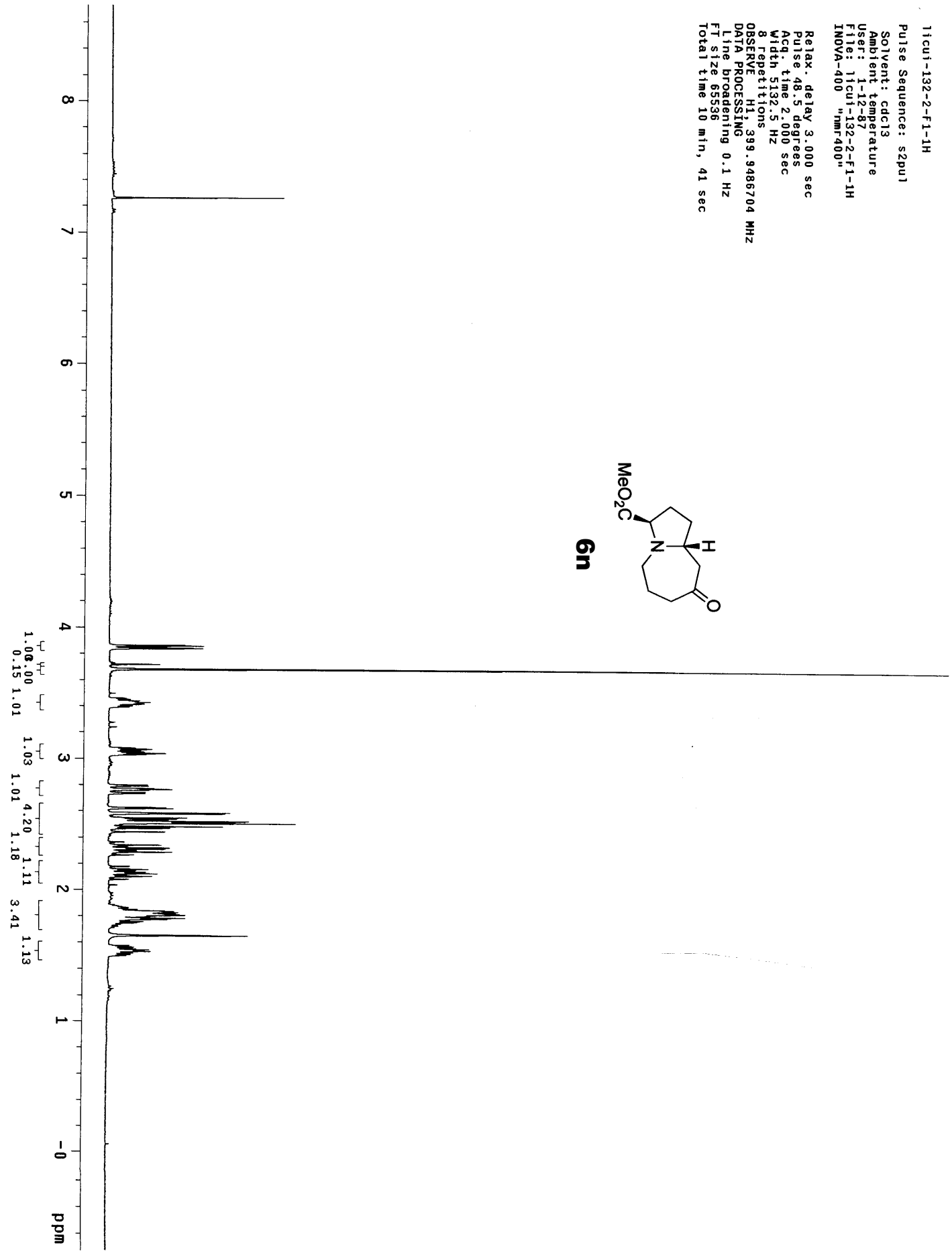
Pulse Sequence: NOESY1D
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.500 sec
Acq. time 1.998 sec
Width 8012.8 Hz
1952 repetitions
OBSERVE H1, 499.7939123 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 2 hr, 8 min, 24 sec



6m




licui-132-2-f1-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: licui-132-2-f1-1H
INOVA-400 "nmf400"
Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
8 repetitions
OBSERVE H1, 399.9486704 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec
MeO2C
N
H
O
6n
8
7
6
5
4
3
2
1
-0
ppm
1.00 1.03 4.20 1.11 1.13
0.15 1.01 1.01 1.18 3.41

licui-132-2-13C

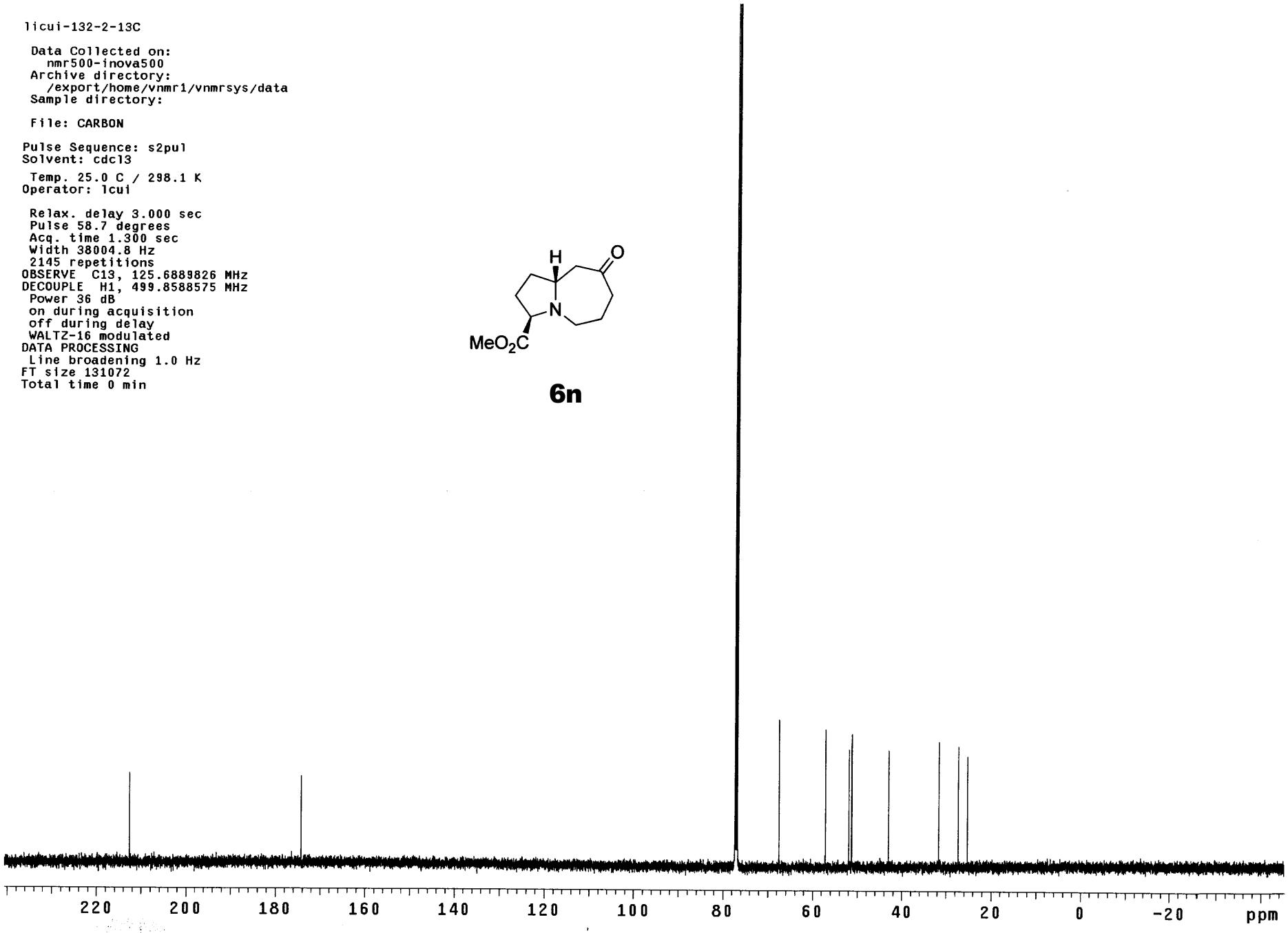
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

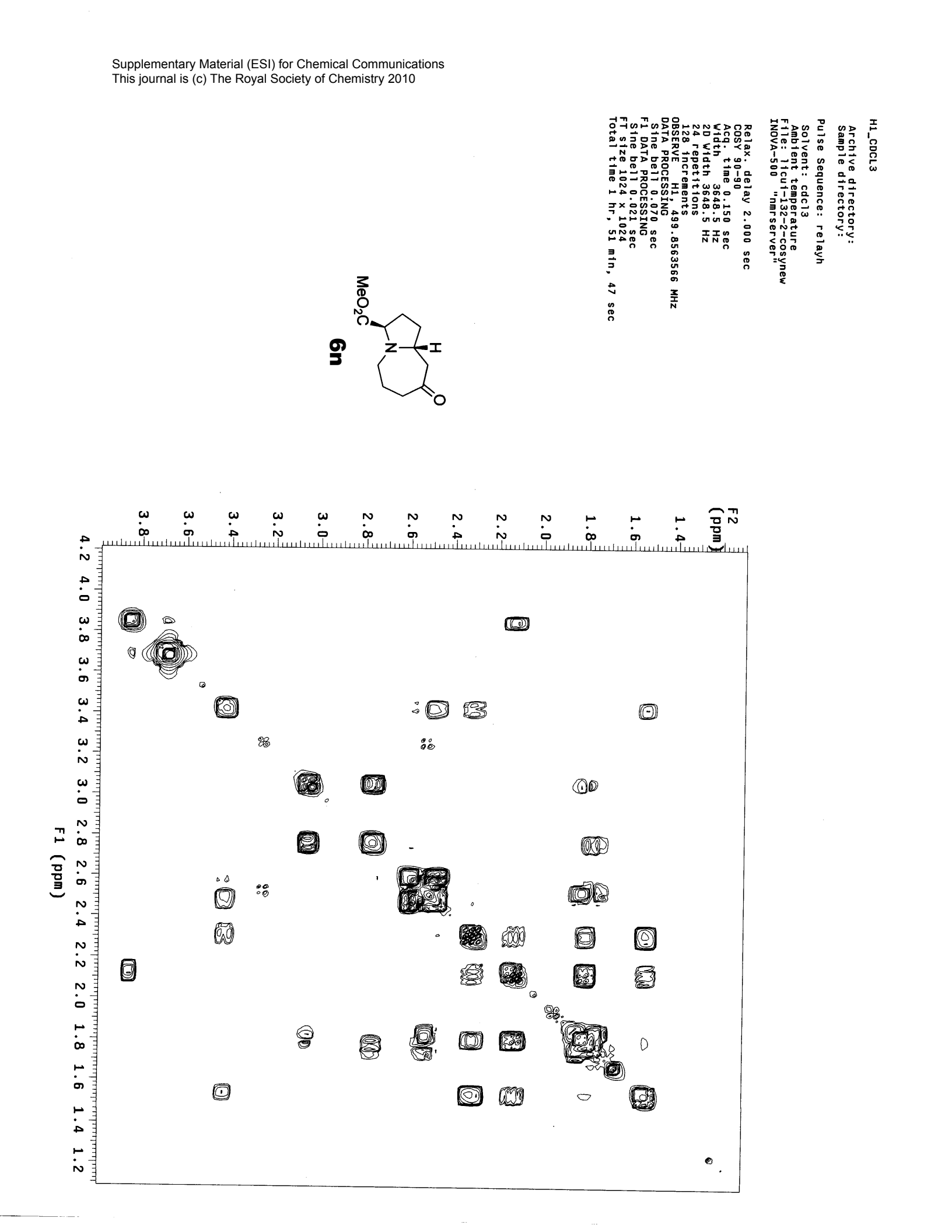
File: CARBON

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

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 38004.8 Hz
2145 repetitions
OBSERVE C13, 125.6889826 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

6n




H1_CDCL3
Archive directory:
Sample directory:
Pulse Sequence: relayh
Solvent: cdcl3
Ambient temperature
File: licu1-132-2-cosynew
INOVA-500 "nmrserver"
Relax. delay 2.000 sec
COSY 90-90
Acq. time 0.150 sec
Width 3648.5 Hz
2D Width 3648.5 Hz
24 repetitions
128 increments
OBSERVE H1, 499.8563566 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 1024 x 1024
Total time 1 hr, 51 min, 47 sec
MeO2C
N
H
O
6n
F2 (ppm)
F1 (ppm)

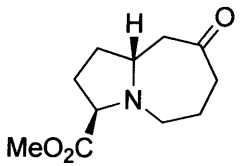
STANDARD PROTON PARAMETERS
Selective band center: 3.43 (ppm); width: 37.5 (Hz)

Archive directory:
Sample directory: lcui

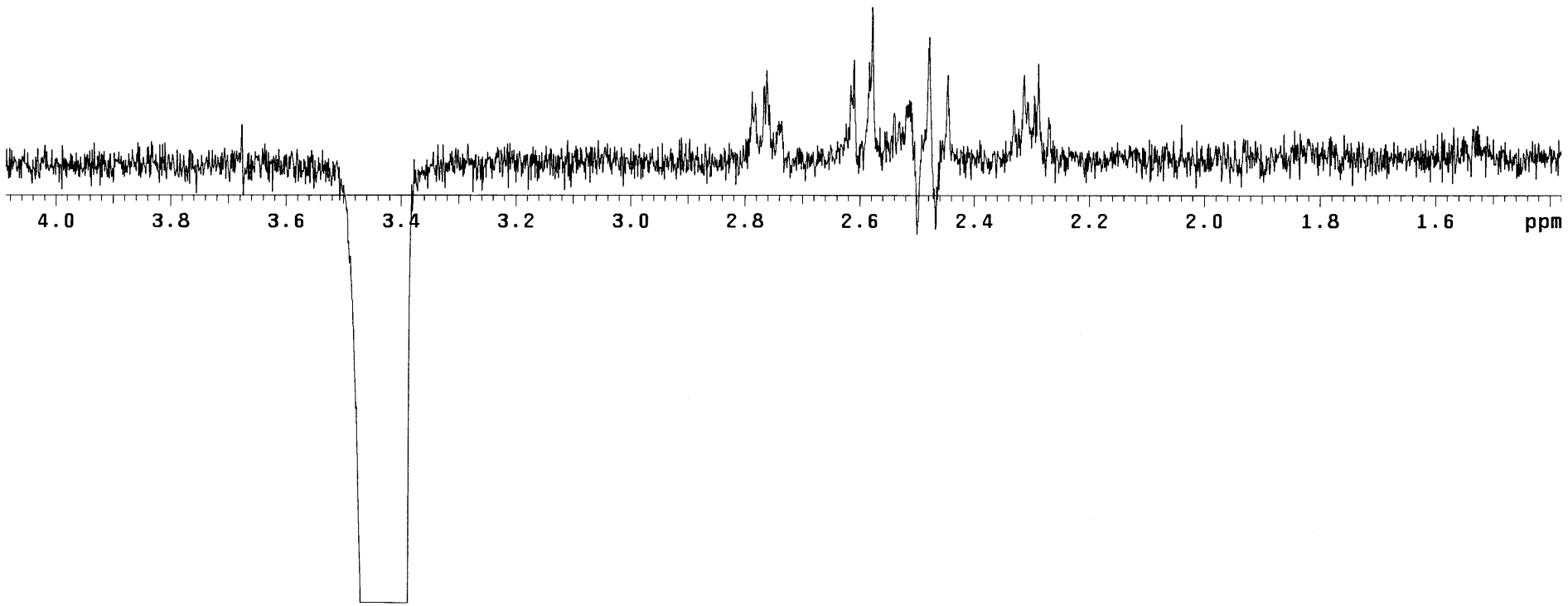
Pulse Sequence: NOESY1D

Solvent: CDCl3
Temp. 22.0 C / 295.1 K
File: licui-1012-noesy-34
INOVA-500 "nmrserver"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.049 sec
Width 3998.8 Hz
116 repetitions
OBSERVE H1, 499.8563615 MHz
DATA PROCESSING
FT size 16384
Total time 13 min, 17 sec



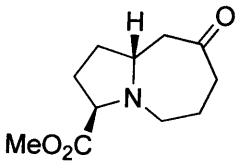
6n



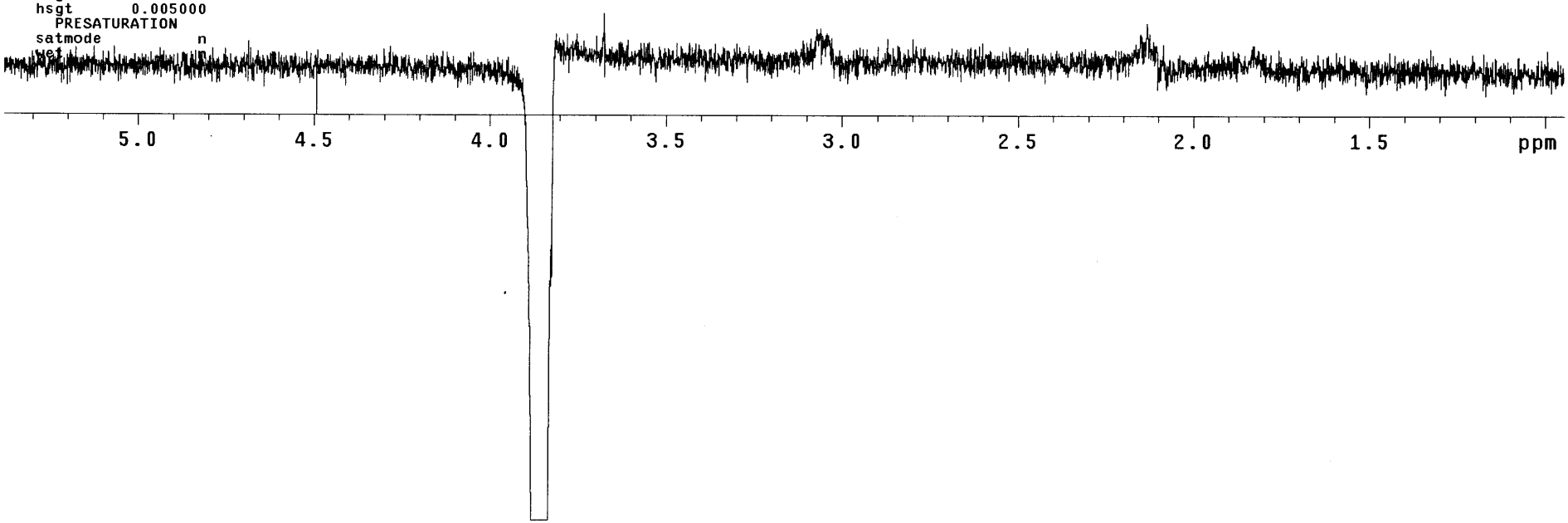
STANDARD PROTON PARAMETERS
Selective band center: 3.86 (ppm); wid
th: 24.9 (Hz)

exp2 NOESY1D

ACQUISITION		DECOUPLER	
sw	3998.8	dn	C13
at	2.049	dm	nnn
np	16384	SAMPLE	
fb	2000	date	Oct 13 2009
bs	4	solvent	CDCl3
ss	2	file	exp
d1	1.000	SPECIAL	
nt	256	temp	22.0
ct	220	gain	46
TRANSMITTER		spin	0
tn	H1	pw90	9.100
sfrq	499.859	FLAGS	
tof	-249.9	sspul	y
tpwr	53	il	n
pw	9.100	in	n
NOESY		dp	y
mixN	0.500	hs	nn
sweeppwr	39	PROCESSING	
sweeppw	1500.000	fn	not used
sweepshp	sech180	DISPLAY	
DPFGSE		sp	472.7
selshapeA	NOESY1D_~	wp	2217.1
	3_86p	vs	30518
selpwrA	-7	sc	0
selpwA	144586.5	wc	250
gzlvlA	3902	hzmm	8.87
gtA	0.001000	is	33.57
selshapeB	NOESY1D_~	rfl	3382.3
	3_86p	rfp	3629.0
selpwrB	-7	th	14
selpwB	144586.5	ins	100.000
gzlvlB	5853	ai cdc ph	
gtB	0.001000		
gstabAB	0.000500		
GRADIENT			
gzlvlC	-975		
gtC	0.001000		
gstab	0.000500		
hsglvl	4878		
hsgt	0.005000		
PRESATURATION			
satmode	n		
wet			



6n



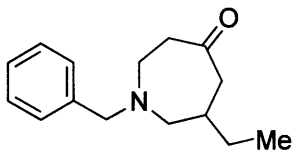
licui-3-12-1-1H

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

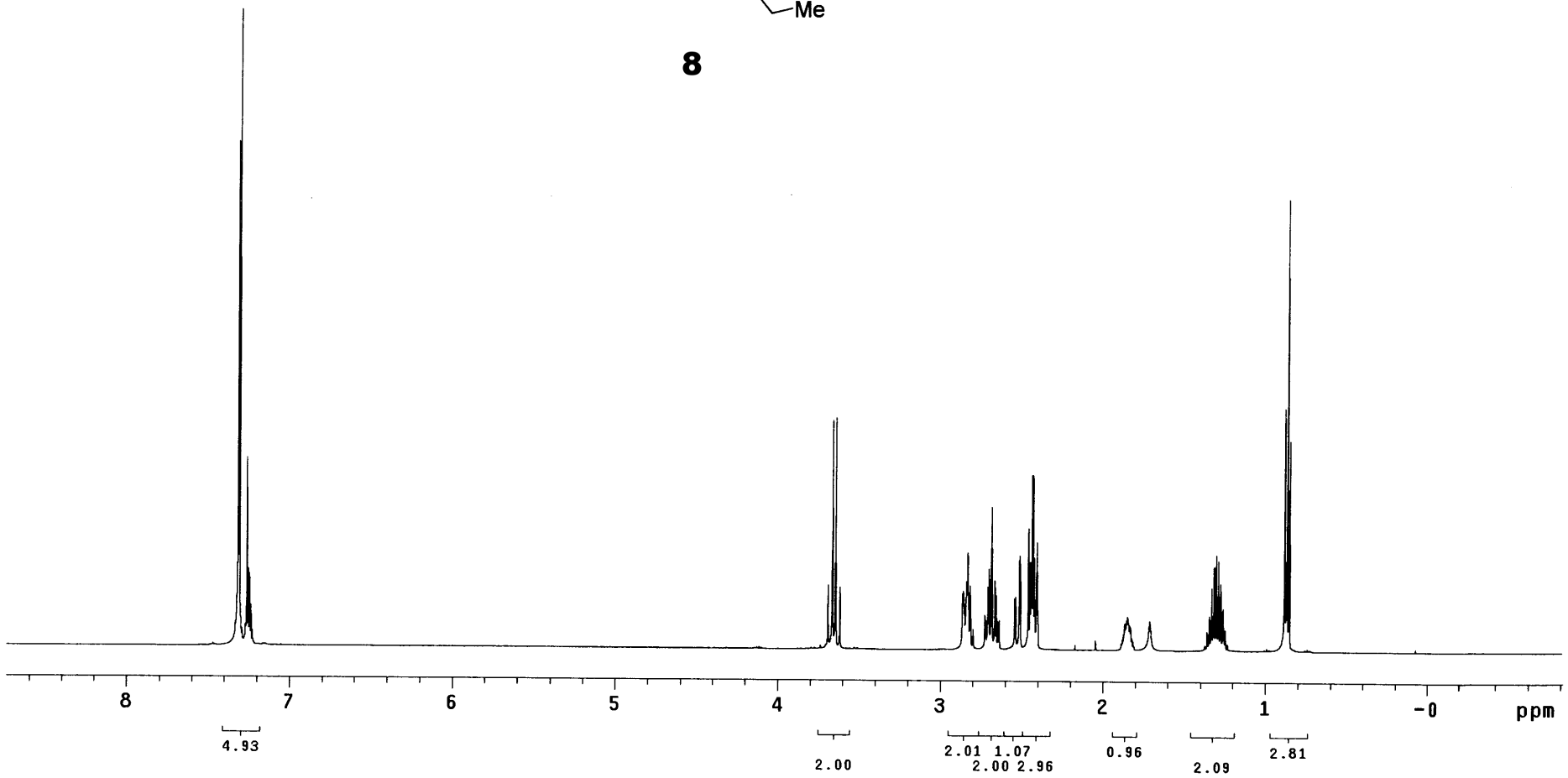
File: licui-3-12-1-1H

Pulse Sequence: s2pul
Solvent: cdcl3
Operator: licui

Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
20 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min



8



licui-3-12-1-13C

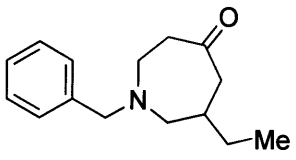
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: licui-3-13-1-13C

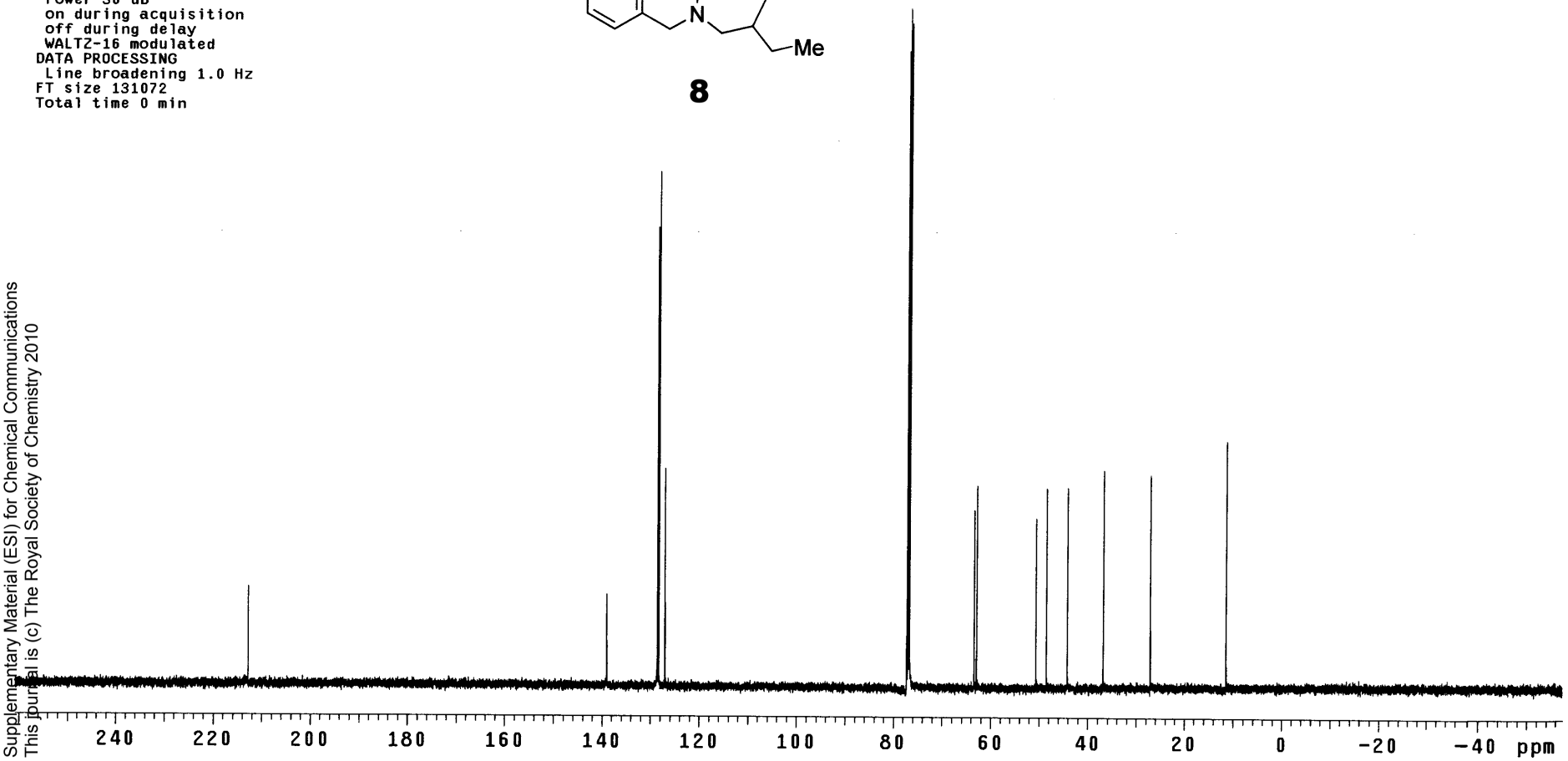
Pulse Sequence: s2pul
Solvent: cdcl3

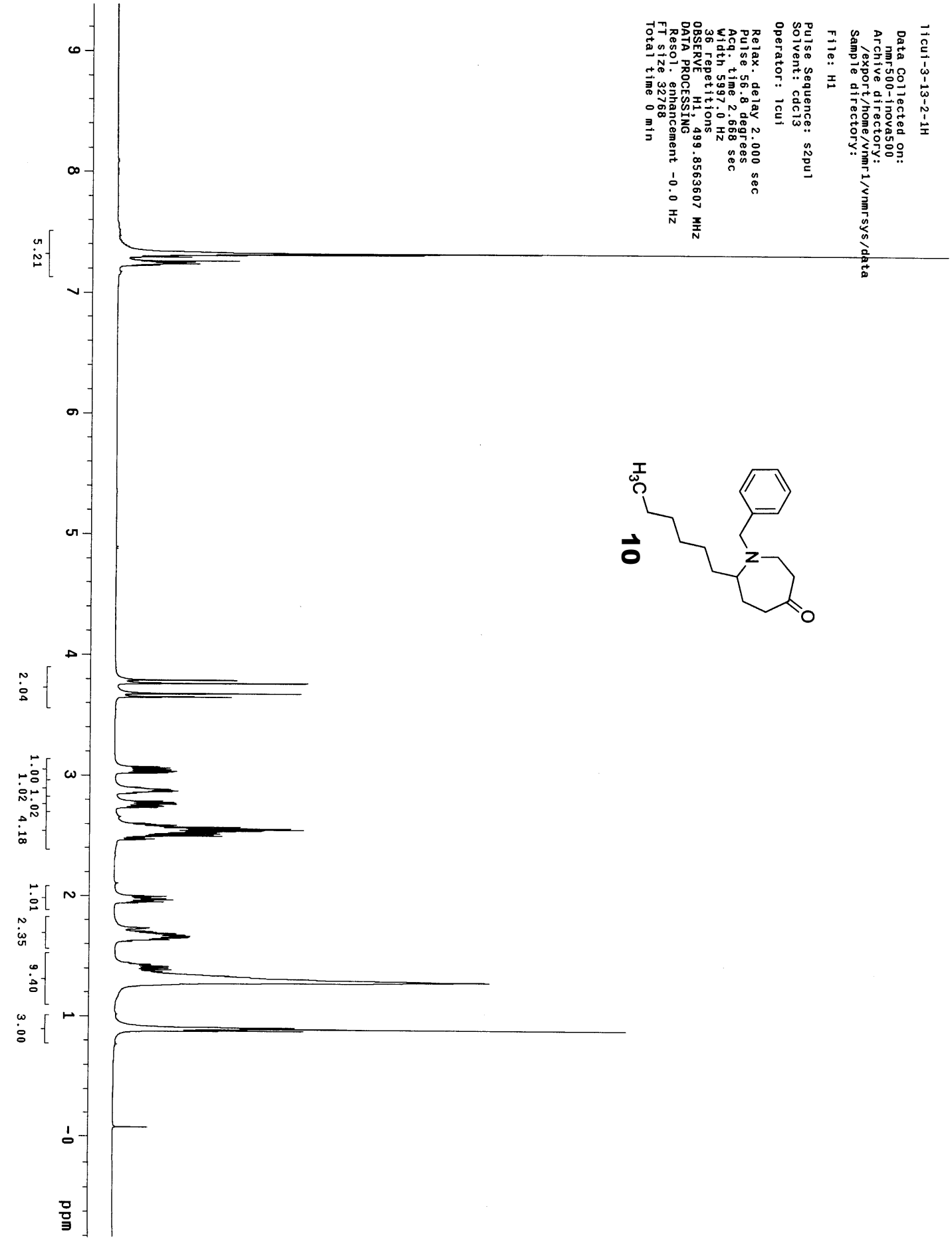
Temp. 25.0 C / 298.1 K
Operator: licui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
520 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 1.0 Hz
FT size 131072
Total time 0 min



8




1icui-3-13-2-1H
Data Collected on:
nmr500-1nova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:
File: H1
Pulse Sequence: s2pul
Solvent: cdcl3
Operator: 1cui
Relax. delay 2.000 sec
Pulse 56.8 degrees
Acq. time 2.668 sec
Width 5997.0 Hz
36 repetitions
OBSERVE H1, 499.8563607 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
Total time 0 min
H3C
N
O
10
ppm
5.21
2.04
1.00
1.02
1.02
4.18
1.01
2.35
9.40
3.00

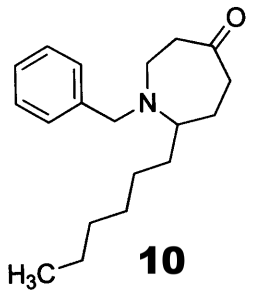
liuci-106-1-13C

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

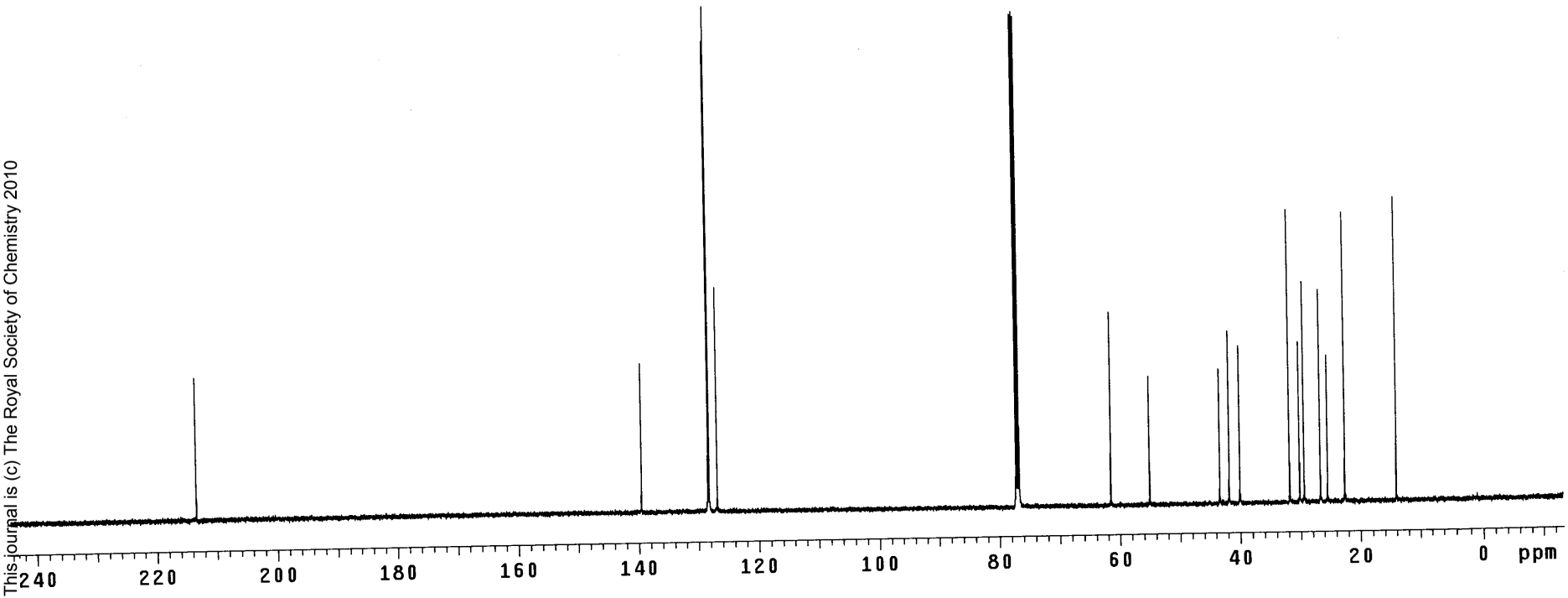
File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 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
1348 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 1.0 Hz
FT size 131072
Total time 0 min



10

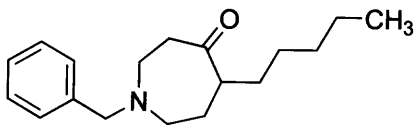


licui-3-18-1-1H

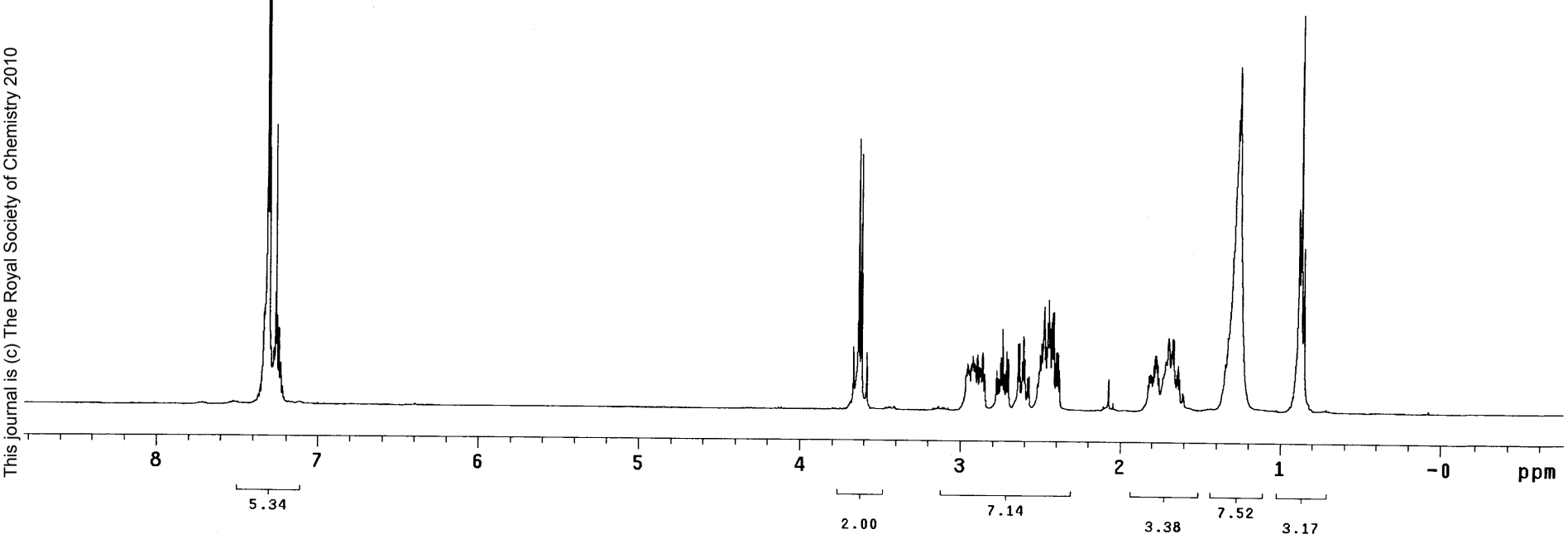
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: licui-3-18-1-1H
INOVA-400 "nmr400"

Relax. delay 3.000 sec
Pulse 48.5 degrees
Acq. time 2.000 sec
Width 5132.5 Hz
16 repetitions
OBSERVE H1, 399.9486712 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec



12



licui-3-18-1-F2-13C

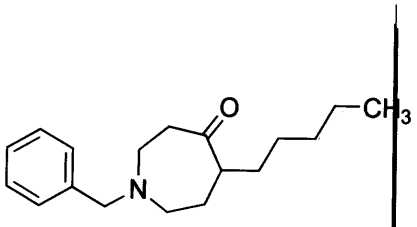
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

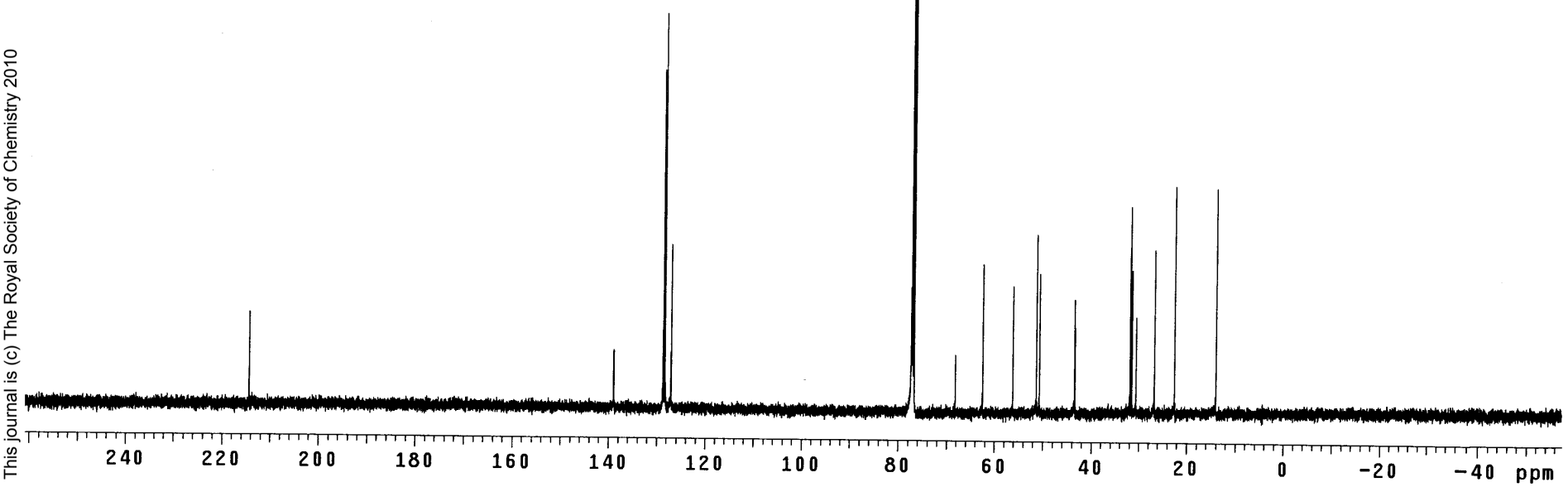
Pulse Sequence: s2pul
Solvent: cdcl3

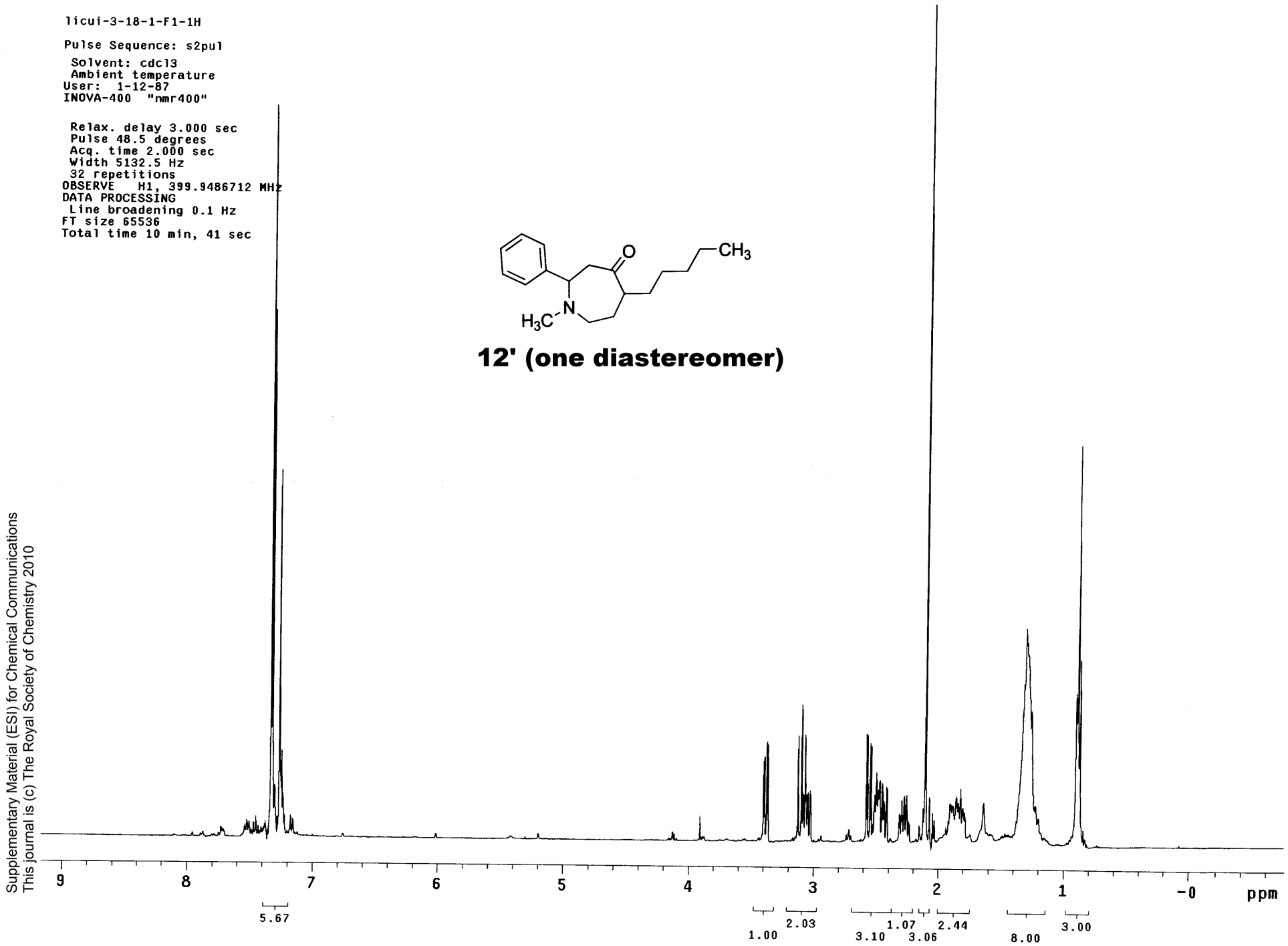
Temp. 25.0 C / 298.1 K
Operator: licui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
718 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 1.0 Hz
FT size 131072
Total time 0 min



12





liuci-106-1-13C

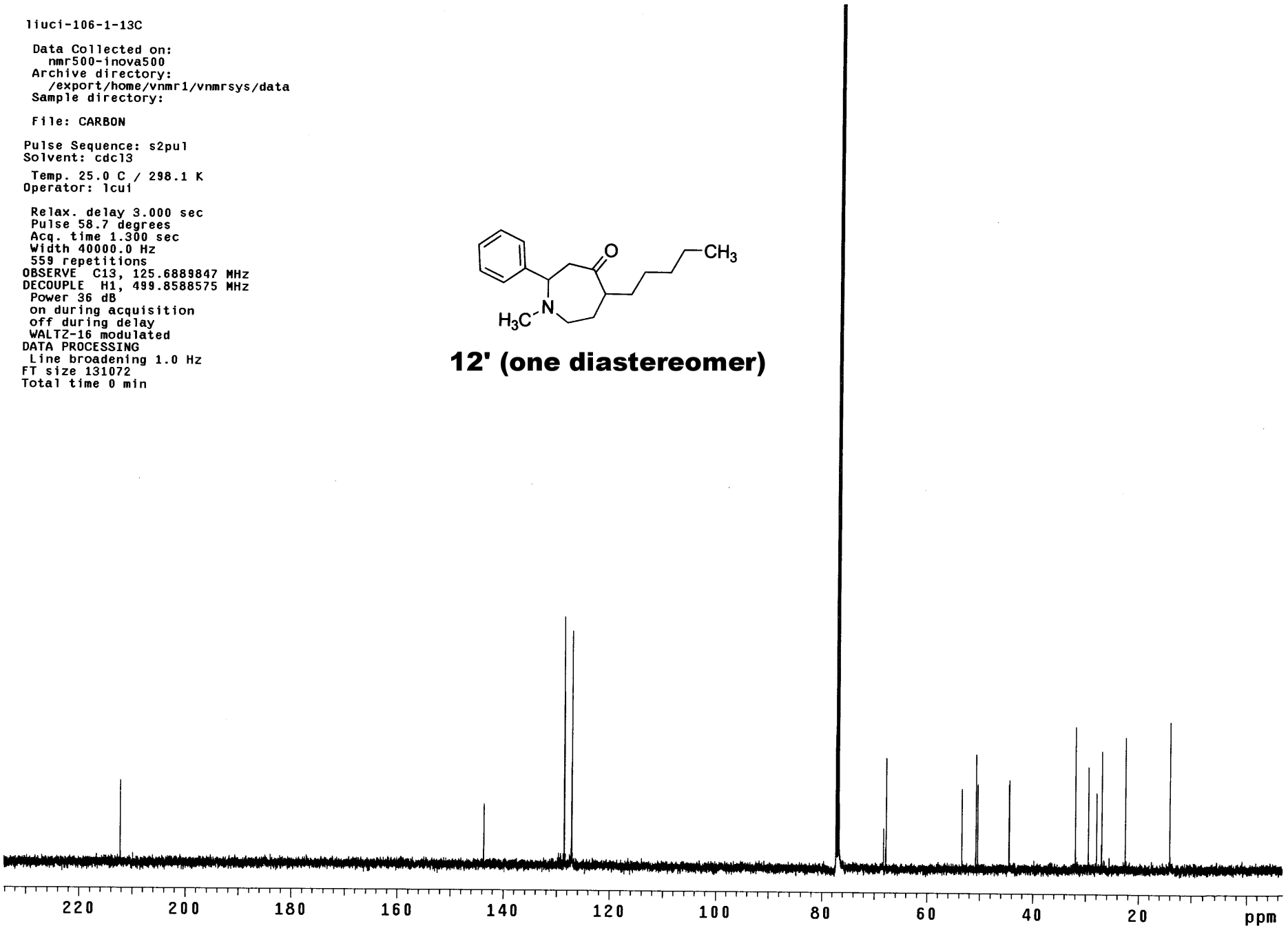
Data Collected on:
nmr500-inova500
Archive directory:
/export/home/vnmr1/vnmrsys/data
Sample directory:

File: CARBON

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 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
559 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 1.0 Hz
FT size 131072
Total time 0 min

12' (one diastereomer)



H1_CDCL3

Data Collected on:
nmr500-inova500
Archive directory:

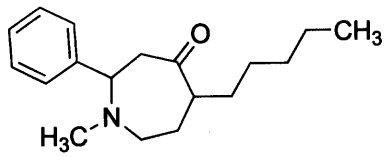
Sample directory:

File: relayh

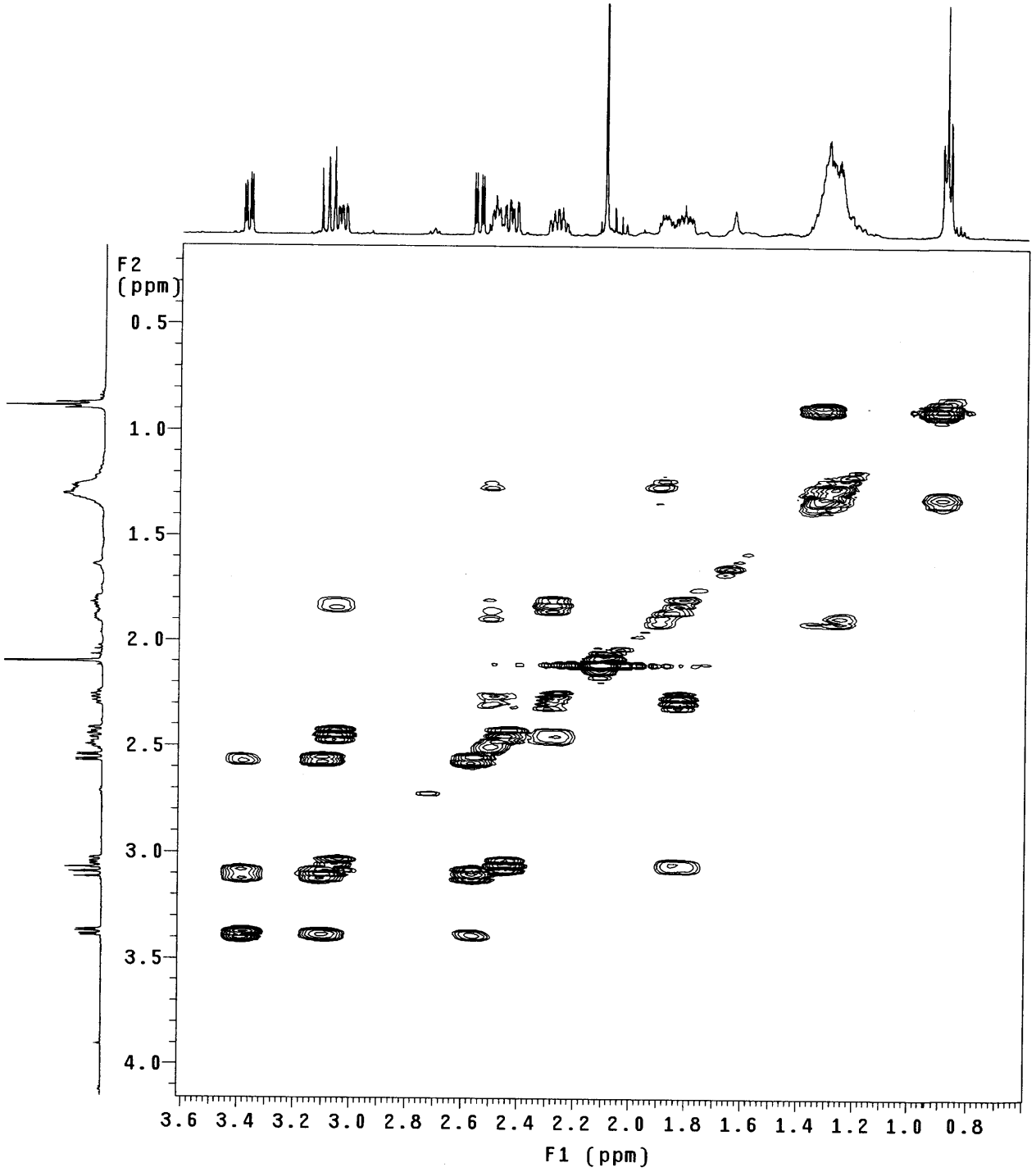
Pulse Sequence: relayh
Solvent: cdcl3

Operator: lcui

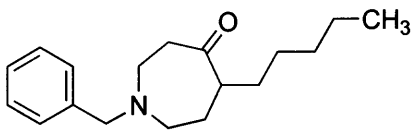
Relax. delay 2.000 sec
COSY 90-90
Acq. time 0.150 sec
Width 4412.6 Hz
2D Width 4412.6 Hz
12 repetitions
256 increments
OBSERVE H1, 499.8563582 MHz
DATA PROCESSING
Sine bell 0.075 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 2048 x 1024
Total time 0 min



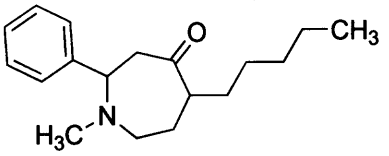
12' (one diastereomer)



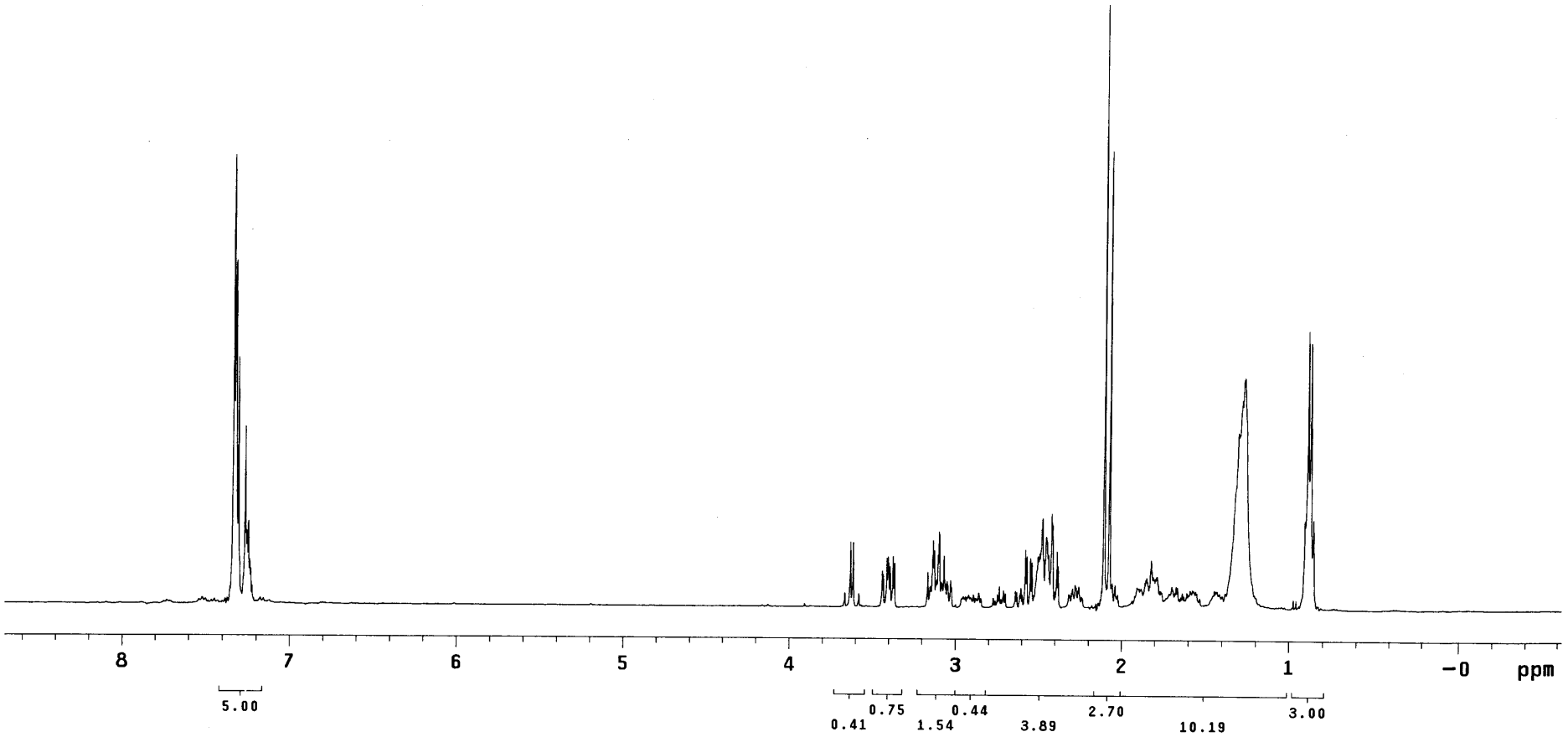
```
Z*SPEC IDG400-5FE
UCSB

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
User:  1-12-87
File: licui-3-18-T8
INOVA-500  "nmrserver"

 Relax. delay 3.000 sec
 Pulse 48.5 degrees
 Acq. time 2.000 sec
 Width 5132.5 Hz
 4 repetitions
OBSERVE   H1, 399.9486712 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 65536
Total time 10 min, 41 sec
```



12



12'



licui-3-18-T9-13C

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: lcui

Relax. delay 3.000 sec
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1217 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

12

12'