



Supporting Information

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**Platinum-Catalyzed Formation of Cyclic
Ketone-Fused Indoles from N-(2-Alkynylphenyl)lactams**

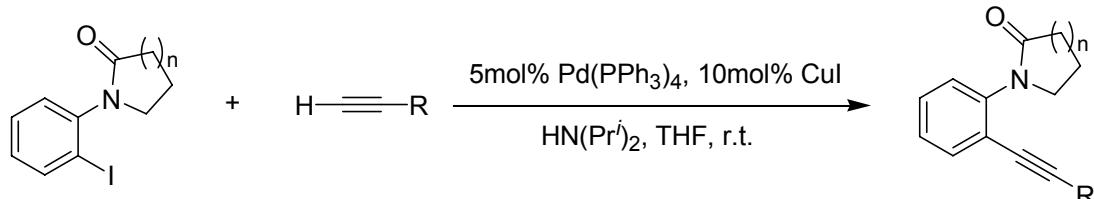
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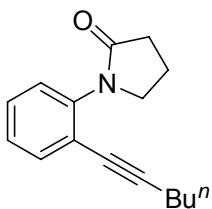
General: Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous tetrahydrofuran and anhydrous dichloromethane in Pure-Pac™ from Aldrich were used directly without further treatment. Commercially available reagents were used without further purification. The NMR data were the same as reported. 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). ^1H 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 electron spray method.

The Sonagashira reaction precursors for lactams **7k**, **7l**, and **7m** were prepared according to a known procedure (U. Ladziata, A. Y. Koposov, K. Y. Lo, J. Willging, V. N. Nemykin, V. V. Zhdankin, *Angew. Chem., Int. Ed.*, **2005**, *44*, 7127) from the corresponding substituted 2-iodoanilines. 2-iodo-6-methoxyaniline (Y. Kondo, S. Kojima, T. Sakamoto, *J. Org. Chem.* **1997**, *62*, 6507), 4-bromo-2-iodoaniline (D. Roche, K. Prasad, O. Repic, T. J. Blacklock, *Tetrahedron Lett.*, **2000**, *41*, 2083, and ethyl 4-amino-3-iodobenzate (M. M. Slutsky, T. V. Jones, G. N. Tew, *J. Org. Chem.* **2007**, *72*, 342) were prepared according to literature procedures.

General procedure A: preparation of *N*-(2-Alkynylphenyl) Lactams

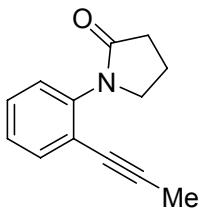


To a solution of the lactam in THF were added sequentially the alkyne (2.0~3.0 eq.), a catalyst [$\text{Pd}(\text{PPh}_3)_4$ or $\text{PdCl}_2(\text{PPh}_3)_2$, 5 mol %], CuI (10 mol %), and $\text{HN}(\text{iPr})_2$ (4 eq.) at room temperature. The resulting mixture was stirred at room temperature for the time indicated. After all the lactam was consumed, the mixture was concentrated under vacuum, and the resulting residue was purified through silica gel flash column chromatography with hexanes/ethyl acetate (1:1).



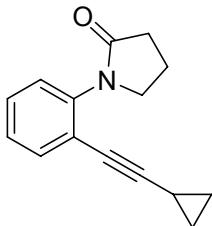
3

Compound **3** was isolated in 97% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2.5 h. ^1H NMR (400MHz, CDCl_3) δ : 7.46 (dd, 1H, J = 1.6, 7.6 Hz), 7.33-7.20 (m, 3H), 3.87 (t, 2H, J = 7.0 Hz), 2.56 (t, 2H, J = 8.1 Hz), 2.43 (t, 2H, J = 6.1 Hz), 2.19 (quintet, 2H, J = 7.5 Hz), 1.62-1.55 (m, 2H), 1.52-1.43 (m, 2H), 0.95 (t, 3H, J = 7.3 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.7, 140.1, 133.4, 128.4, 127.6, 127.3, 122.1, 95.4, 50.1, 31.4, 30.7, 22.0, 19.2, 19.1, 13.6; IR (neat): 3063, 2928, 2862, 2230, 1699, 1494, 1451, 1404, 1050, 875, 824; MS (ES $^+$) Calculated for $[\text{C}_{16}\text{H}_{19}\text{NONa}]^+$ 264.1; Found: 263.9.



7a

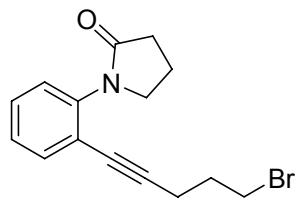
Compound **7a** was isolated in 70% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and excess of propyne. Reaction time: 12 h. ^1H NMR (500MHz, CDCl_3) δ : 7.46 (dd, 1H, J = 1.5, 8.0 Hz), 7.33~7.21 (m, 3H), 3.87 (t, 2H, J = 7.0 Hz), 2.57 (t, 2H, J = 7.0 Hz), 2.18 (quintet, 2H, J = 7.0 Hz), 2.08 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.8, 140.2, 133.4, 128.5, 127.6, 127.3, 121.9, 90.8, 76.4, 50.2, 31.5, 19.3, 4.5; IR (neat): 2925, 2253, 1688, 1607, 1493, 1451, 1050, 824; MS (ES $^+$) Calculated for $[\text{C}_{13}\text{H}_{13}\text{NONa}]^+$: 222.0; Found: 221.8.



7b

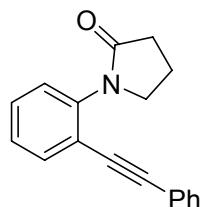
Compound **7b** was isolated in 85% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of ethynylcyclopropane. Reaction time: 3 h. ^1H NMR

(400MHz, CDCl₃) δ: 7.44 (dd, 1H, *J* = 1.2, 7.6 Hz), 7.32~7.18 (m, 3H), 3.85 (t, 2H, *J* = 7.2 Hz), 2.56 (t, 2H, *J* = 7.6 Hz), 2.23~2.15 (m, 2H), 1.48~1.42 (m, 1H), 0.91~0.86 (m, 2H), 0.79~0.75 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 174.7, 140.2, 133.2, 128.4, 127.5, 127.2, 121.8, 98.6, 72.3, 50.1, 31.5, 19.2, 8.7, 0.26; IR (neat): 2926, 2229, 1700, 1596, 1493, 1450, 1403, 1304, 1257, 1050, 957, 874, 824; MS (ES⁺) Calculated for [C₁₅H₁₅NONa]⁺ 248.1; Found: 248.1.



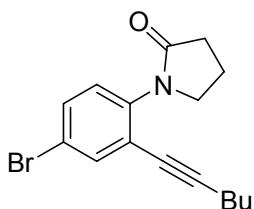
7c

Compound **7c** was isolated in quantitative yield following the general procedure A using Pd(PPh₃)₄ as catalyst and 3 eq. of 5-bromo-1-pentyne. Reaction time: 3 h. ¹H NMR (400MHz, CDCl₃) δ: 7.46 (dd, 1H, *J* = 7.6, 1.6 Hz), 7.35~7.31 (m, 1H), 7.26~7.21 (m, 2H), 3.86 (t, 2H, *J* = 6.8 Hz), 3.59 (t, 2H, *J* = 6.4 Hz), 2.64 (t, 2H, *J* = 6.8 Hz), 2.59 (t, 2H, *J* = 8.4 Hz), 2.26~2.21 (m, 2H), 2.15~2.08 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 174.7, 140.3, 133.5, 128.8, 127.5, 127.4, 121.6, 92.8, 78.4, 50.3, 32.4, 31.4, 31.3, 19.2, 18.2; IR (neat): 3016, 2925, 1699, 1607, 1494, 1451, 1050, 875, 824; MS (ES⁺) Calculated for [C₁₅H₁₆BrNONa]⁺ 328.0; Found: 328.0.



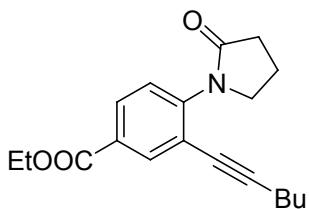
7e

Compound **7e** was isolated in 71% yield following the general procedure A using Pd(PPh₃)₂Cl₂ as catalyst and 2 eq. of phenylacetylene. Reaction time: 4h. ¹H NMR (400MHz, CDCl₃) δ: 7.60 (dd, 1H, *J* = 1.6, 7.6 Hz), 7.51~7.47 (m, 2H), 7.41~7.27 (m, 6H), 3.95 (t, 2H, *J* = 6.9 Hz), 2.60 (t, 2H, *J* = 8.0 Hz), 2.27~2.19 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 174.8, 140.3, 133.3, 131.4, 129.3, 128.6, 128.4, 127.7, 127.4, 123.0, 121.2, 94.2, 86.0, 50.3, 31.5, 19.3; IR (neat): 3063, 2926, 2861, 1698, 1497, 1448, 1404, 1050, 875, 824; MS (ES⁺) Calculated for [C₁₈H₁₅NONa]⁺ 284.1; Found: 283.9.



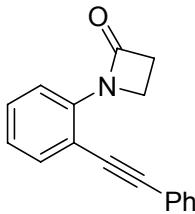
7l

Compound **7l** was isolated in 77% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2h. ^1H NMR (400MHz, CDCl_3) δ : 7.59 (d, 1H, J = 2.4 Hz), 7.20 (dd, 1H, J = 8.4, 2.4 Hz), 7.13 (d, 1H, J = 8.4 Hz), 3.85 (t, 2H, J = 6.8 Hz), 2.55 (t, 2H, J = 8.0 Hz), 2.43 (t, 2H, J = 7.2 Hz), 2.19 (quintet, 2H, J = 7.6 Hz), 1.61-1.54 (m, 2H), 1.51-1.42 (m, 2H), 0.95 (t, 3H, J = 7.2 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.8, 139.1, 136.0, 131.6, 129.0, 123.9, 120.5, 97.1, 76.1, 49.9, 31.3, 30.6, 22.0, 19.2, 19.1, 13.6; IR (neat): 2928, 2862, 2226, 1699, 1489, 1455, 1404, 1304, 1050, 876, 824; MS (ES $^+$) Calculated for $[\text{C}_{16}\text{H}_{18}\text{BrNONa}]^+$ 342.1; Found: 341.8.

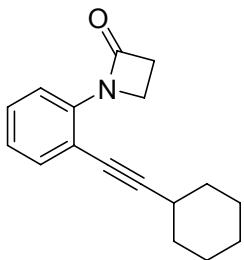


7m

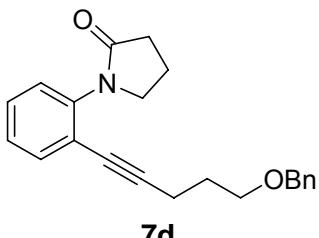
Compound **7m** was isolated in 80% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2h. ^1H NMR (400MHz, CDCl_3) δ : 8.13 (d, 1H, J = 2.0 Hz), 7.96 (dd, 1H, J = 8.4, 1.6 Hz), 7.37 (d, 1H, J = 8.4 Hz), 4.37 (q, 2H, J = 7.2 Hz), 3.93 (t, 2H, J = 7.2 Hz), 2.58 (t, 2H, J = 8.0 Hz), 2.45 (t, 2H, J = 7.2 Hz), 2.21 (quintet, 2H, J = 7.6 Hz), 1.63-1.56 (m, 2H), 1.52-1.45 (m, 2H), 1.40 (t, 3H, J = 7.2 Hz), 0.95 (t, 3H, J = 7.2 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ : 174.7, 165.5, 143.9, 134.9, 129.4, 129.2, 127.3, 121.7, 96.7, 76.8, 61.2, 49.8, 31.5, 30.6, 22.0, 19.3, 14.3, 13.6; IR (neat): 2927, 1715, 1604, 1495, 1452, 1229, 1097, 1050, 875, 824; MS (ES $^+$) Calculated for $[\text{C}_{19}\text{H}_{23}\text{NO}_3\text{Na}]^+$ 336.2; Found: 335.8.



This compound was isolated in quantitative yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of phenylacetylene. Reaction time: 3 h. ^1H NMR (500MHz, CDCl_3) δ : 8.09 (d, 1H, J = 8.5 Hz), 7.51~7.47 (m, 3H), 7.38~7.35 (m, 3H), 7.30 (t, 1H, J = 7.5 Hz), 7.07 (t, 1H, J = 7.5 Hz), 4.25 (t, 2H, J = 4.5 Hz), 3.13 (t, 2H, J = 4.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ : 166.1, 139.1, 134.0, 131.1, 129.4, 128.6, 128.5, 123.9, 123.0, 120.9, 111.4, 93.7, 86.8, 42.3, 37.4; IR (neat): 2926, 2253, 1746, 1607, 1494, 1451, 1050, 824; MS (ES $^+$) Calculated for $[\text{C}_{17}\text{H}_{13}\text{NONa}]^+$ 270.1; Found: 270.3.



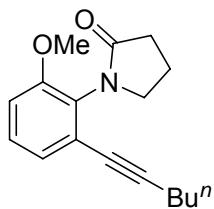
This compound was isolated in 75% yield following the general procedure A using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of cyclohexylacetylene. Reaction time: 3 h. ^1H NMR (400MHz, CDCl_3) δ : 8.01 (d, 1H, J = 8.0 Hz), 7.49 (d, 1H, J = 8.0 Hz), 7.22 (t, 1H, J = 8.0 Hz), 6.99 (t, 1H, J = 8.0 Hz), 4.15 (t, 2H, J = 4.4 Hz), 3.09 (t, 2H, J = 4.4 Hz), 2.60~2.50 (m, 1H), 1.96~1.87 (m, 2H), 1.80~1.70 (m, 2H), 1.62~1.46 (m, 3H), 1.42~1.30 (m, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ : 166.1, 138.8, 134.3, 128.4, 123.9, 120.9, 112.6, 98.9, 77.7, 42.2, 37.2, 32.4, 30.0, 25.8, 25.0. IR (neat): 2927, 2253, 1738, 1607, 1494, 1451, 1050, 907, 824, 732; MS (ES $^+$) Calculated for $[\text{C}_{17}\text{H}_{19}\text{NONa}]^+$ 276.1; Found: 276.1.



7d

A solution of *N*-(2-iodophenyl)- γ -lactam (0.143g, 0.5 mmol) and 5-benzyloxy-1-pentyne (0.174g, 1 mmol) in piperidines (2 mL) was added $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (11 mg, 0.015 mmol) and CuI (6 mg, 0.03 mmol). The resulting mixture was heated to 70°C, and the reaction

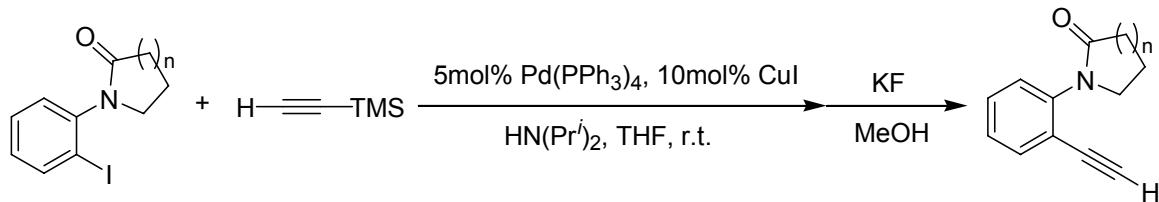
finished in 30 min. The resulting mixture was concentrated, and the residue was purified via silica gel flash column chromatography to give compound **7d** in 96% yield. ¹H NMR (500MHz, CDCl₃) δ: 7.43 (dd, 1H, J = 7.7, 1.3 Hz), 7.34~7.19 (m, 8H), 4.52 (s, 2H), 3.83 (t, 2H, J = 7.0 Hz), 3.61 (t, 2H, J = 6.3 Hz), 2.56~2.52 (m, 4H), 2.17~2.11 (m, 2H), 1.92~1.87 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 174.6, 140.0, 138.3, 133.3, 128.5, 128.3, 127.5, 127.5, 127.5, 127.2, 121.8, 94.6, 77.5, 72.9, 68.7, 50.1, 31.4, 28.9, 19.1, 16.4; IR (neat): 3020, 1686, 1494, 1451, 1216, 1050, 875, 824; MS (ES⁺) Calculated for [C₂₂H₃₃NO₂Na]⁺ 356.2; Found: 356.1.



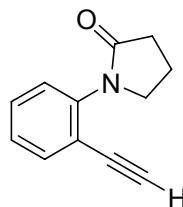
7k

A solution of *N*-(6-methoxy-2-iodophenyl)- γ -lactam (0.200g, 0.63 mmol) and 1-hexyne (0.103g, 1.26 mmol) in piperidines (2 mL) was added Pd(PPh₃)₂Cl₂ (13 mg, 0.019 mmol) and Cul (12 mg, 0.063 mmol) under N₂. The resulting mixture was heated to 70°C, and the reaction finished in 30 min. The resulting mixture was concentrated, and the residue was purified via silica gel flash column chromatography to give compound **7k** in 68% yield. ¹H NMR (400MHz, CDCl₃) δ: 7.19 (t, 1H, J = 8.0 Hz), 7.04 (dd, 1H, J = 7.6, 1.2 Hz), 6.87 (dd, 1H, J = 8.4, 0.8 Hz), 3.80 (s, 3H), 3.75-3.61 (m, 2H), 2.61-2.45 (m, 2H), 2.40 (t, 2H, J = 7.2 Hz), 2.27-2.12 (m, 2H), 1.60-1.42 (m, 4H), 0.94 (t, 3H, J = 7.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 174.6, 155.6, 128.5, 128.4, 124.5, 124.5, 111.3, 94.4, 76.8, 55.7, 48.8, 30.9, 30.6, 21.8, 19.2, 19.0, 13.4; IR (neat): 2932, 2862, 2230, 1704, 1574, 1472, 1296, 1100, 1051, 1015, 875, 824; MS (ES⁺) Calculated for [C₁₇H₂₁NO₂Na]⁺ 294.2; Found: 294.2.

General procedure B: preparation of ethynyl lactams

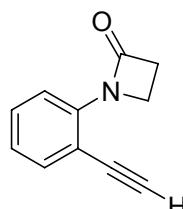


To a solution of the lactam and (trimethylsilyl)acetylene (1.5 eq.) in THF were added $\text{PdCl}_2(\text{PPh}_3)_2$ (0.05 eq.), CuI (0.1 eq.), and $\text{HN}(i\text{Pr})_2$ (5.0 eq.). The resulting mixture was stirred at room temperature for 2.5 h. When the lactam was consumed, the mixture was concentrated under vacuum. The resulting residue was dissolved in MeOH and treated with $\text{KF}\cdot\text{H}_2\text{O}$ (3eq.) at room temperature for 10h. The reaction mixture was concentrated under vacuum, and the residue was purified through silica gel flash column chromatography with hexanes/ethyl acetate (1:1).



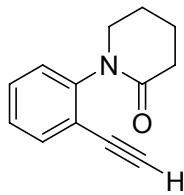
7j

Compound **7j** was isolated in 73% yield following the general procedure B. ^1H NMR (400MHz, CDCl_3) δ : 7.56 (dd, 1H, J = 1.4, 7.6 Hz), 7.42-7.38 (m, 1H), 7.30-7.24 (m, 2H), 3.88 (t, 2H, J = 7.0 Hz), 3.28 (s, 1H), 2.57 (t, 2H, J = 8.0 Hz), 2.24-2.17 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.8, 140.8, 134.0, 129.8, 127.6, 127.3, 82.0, 80.3, 50.2, 31.4, 19.1; IR (neat): 3220, 3063, 2926, 2103, 1699, 1491, 1449, 1405, 1306, 1119, 1050, 875, 824; MS (ES^+) Calculated for $[\text{C}_{12}\text{H}_{11}\text{NONa}]^+$ 208.1; Found: 208.0.



10

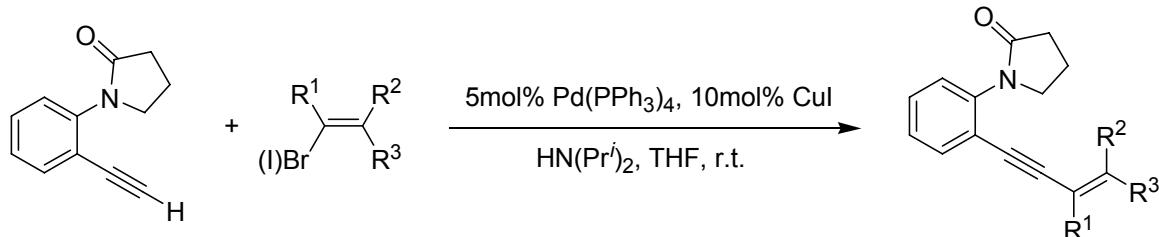
Compound **9** was isolated in 80% yield following the general procedure B. ^1H NMR (400MHz, CDCl_3) δ : 8.05 (d, 1H, J = 8.4 Hz), 7.44 (d, 1H, J = 7.6 Hz), 7.32 (t, 1H, J = 7.2 Hz), 7.04 (t, 1H, J = 7.6 Hz), 4.17 (t, 2H, J = 4.8 Hz), 3.28 (s, 1H), 3.11 (t, 2H, J = 4.8 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ : 166.1, 139.7, 134.8, 129.8, 123.9, 120.9, 110.3, 82.2, 81.1, 42.2, 37.2; IR (neat): 2253, 1746, 1596, 1492, 1446, 1363, 1050, 907, 730; MS (ES^+) Calculated for $[\text{C}_{11}\text{H}_9\text{NONa}]^+$ 194.1; Found 194.0.



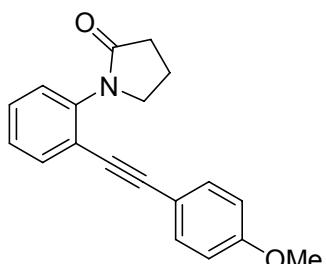
12

Compound **12** was isolated in 53% yield following the general procedure B. ^1H NMR (500MHz, CDCl_3) δ : 7.58 (d, 1H, J = 7.5 Hz), 7.40 (t, 1H, J = 7.5 Hz), 7.28 (t, 1H, J = 7.5 Hz), 7.22 (d, 1H, J = 7.5 Hz), 3.61 (bs, 2H), 3.22 (s, 1H), 2.57 (t, 2H, J = 6.5 Hz), 1.99~1.92 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 169.9, 145.4, 133.9, 130.1, 128.1, 127.4, 120.9, 81.2, 80.2, 51.2, 32.7, 23.5, 21.4; IR (neat): 2926, 2253, 1607, 1494, 1451, 1050, 907, 732; MS (ES $^+$) Calculated for $[\text{C}_{13}\text{H}_{13}\text{NONa}]^+$: 222.0; Found: 221.7.

General procedure C: preparation of lactam with alkenyl or aryl substituents at its alkyne terminus



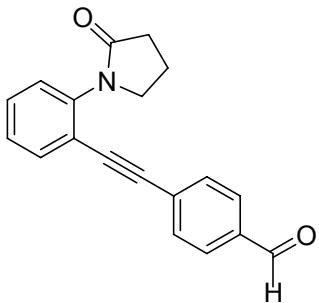
To a solution of alkyne **7j** (1.0 eq.) and an alkenyl/aryl halide (2.0 eq.) were added a catalyst [$\text{Pd}(\text{PPh}_3)_4$ or $\text{PdCl}_2(\text{PPh}_3)_2$, 5 mol %], CuI (10 mol %), and $\text{HN}(\text{iPr})_2$ (3.0~5.0 eq.). The resulting mixture was stirred at room temperature for the indicated time. After **7j** was consumed, the reaction mixture was concentrated under vacuum. The residue was purified through silica gel flash column chromatography with hexanes/ethyl acetate (1:1).



7f

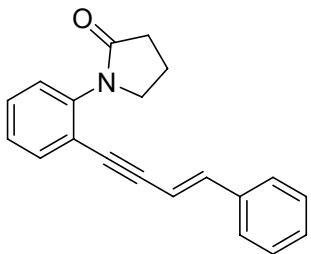
Compound **7f** was isolated in 97% yield following the general procedure C using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of 4-iodoanisole. Reaction time: 5 h. ^1H NMR (400MHz,

CDCl_3) δ : 7.57 (dd, 1H, J = 1.2, 8.0 Hz), 7.44~7.26 (m, 5H), 6.90~6.86 (m, 2H), 3.95 (t, 2H, J = 6.8 Hz), 3.83 (s, 3H), 2.59 (t, 2H, J = 7.6 Hz), 2.26~2.18 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.8, 159.8, 140.1, 133.1, 132.9, 128.9, 127.7, 127.3, 121.5, 115.1, 114.1, 94.3, 84.8, 55.3, 50.3, 31.6, 19.3; IR (neat): 2926, 2212, 1699, 1606, 1512, 1494, 1451, 1403, 1250, 1050, 875, 824; MS (ES^+) Calculated for $[\text{C}_{19}\text{H}_{17}\text{NONa}]^+$ 314.1; Found: 314.1.



7g

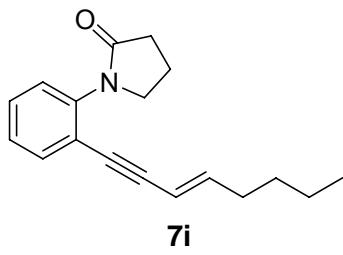
Compound **7g** was isolated in 80% yield following the general procedure C using $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ as catalyst and 2 eq. of 4-bromobenzaldehyde. Reaction time: 2.5 h. ^1H NMR (400MHz, CDCl_3) δ : 10.03 (s, 1H), 7.88 (d, 2H, J = 8.8 Hz), 7.65~7.62 (m, 3H), 7.46~7.43 (m, 1H), 7.36~7.33 (m, 2H), 3.95 (t, 2H, J = 6.8 Hz), 2.62 (t, 2H, J = 8.0 Hz), 2.29~2.21 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 191.3, 174.7, 140.6, 135.7, 133.5, 131.9, 130.0, 129.6, 129.2, 127.6, 127.5, 120.5, 93.1, 90.1, 50.5, 31.5, 19.4; IR (neat): 2925, 2216, 1699, 1601, 1494, 1451, 1403, 1050, 875, 824; MS (ES^+) Calculated for $[\text{C}_{19}\text{H}_{15}\text{NONa}]^+$ 312.1; Found: 311.9.



7h

Compound **7h** was isolated in 97% yield following the general procedure C using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and 2 eq. of β -bromostyrene. Reaction time: 4h. ^1H NMR (400MHz, CDCl_3) δ : 7.54 (dd, 1H, J = 1.2, 7.6 Hz), 7.44~7.24 (m, 8H), 6.96 (d, 1H, J = 16.0 Hz), 6.38 (d, 1H, J = 16.0 Hz), 3.92 (t, 2H, J = 6.8 Hz), 2.61 (t, 2H, J = 8.0 Hz), 2.27~2.20 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 174.8, 141.6, 140.2, 136.1, 133.3, 129.2, 128.8, 128.7, 127.6, 127.3, 126.4, 121.3, 107.8, 93.7, 88.3, 50.3, 31.5, 19.3; IR (neat): 2925,

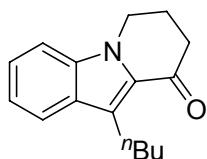
2195, 1699, 1607, 1492, 1451, 1403, 1050, 875, 824; MS (ES⁺) Calculated for [C₂₀H₁₇NONa]⁺ 310.1; Found: 310.0.



Compound **7i** was isolated in 99% yield following the general procedure C using Pd(PPh₃)₄ as catalyst and 2 eq. of 1-iodo-1-hexene. Reaction time: 2.5 h. ¹H NMR (400MHz, CDCl₃) δ: 7.48 (d, 1H, *J* = 7.6 Hz), 7.35~7.21 (m, 3H), 6.21 (dt, 1H, *J* = 7.2, 15.6 Hz), 5.68 (dt, 1H, *J* = 1.6, 15.6 Hz), 3.88 (t, 2H, *J* = 6.8 Hz), 2.57 (t, 2H, *J* = 6.8 Hz), 2.24~2.13 (m, 4H), 1.46~1.30 (m, 4H), 0.91 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (125 MHz, CDCl₃) δ: 174.8, 145.7, 140.1, 133.2, 128.8, 127.6, 127.3, 121.5, 109.4, 93.3, 84.4, 50.2, 32.9, 31.5, 30.8, 22.2, 19.3, 13.8; IR (neat): 2926, 2253, 1681, 1607, 1493, 1451, 1050, 824; MS (ES⁺) Calculated for [C₁₃H₁₃NONa]⁺ 290.1; Found: 290.3.

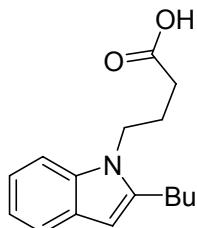
General procedure D: PtCl₄/PtCl₂-catalyzed reaction of lactams

To a 0.01 M solution of the lactam in anhydrous 1,2-dichloroethane was added 10 mol % of PtCl₄ or PtCl₂ (if specified) under an atmosphere of O₂. The resulting mixture was refluxing for the indicated time. Upon the completion of the reaction, the solvent was removed under vacuum, and the residue was purified via silica gel flash column chromatography (eluent: hexanes/ethyl acetate = 3:1) to yield the desired product.



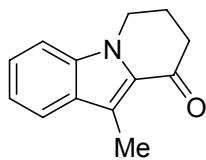
Compound **4** was isolated in 83% yield following the general procedure D. Reaction time: 1 h. ¹H NMR (400MHz, CDCl₃) δ: 7.73 (dt, 1H, *J* = 8.0, 0.8 Hz), 7.40~7.30 (m, 2H), 7.14 (dd, 1H, *J* = 6.8, 1.2 Hz), 4.22 (t, 2H, *J* = 6.0 Hz), 3.18 (t, 2H, *J* = 8.0 Hz), 2.72 (t, 2H, *J* = 6.4 Hz), 2.39~2.33 (m, 2H), 1.68~1.61 (m, 2H), 1.46~1.37 (m, 2H), 0.93 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (125 MHz, CDCl₃) δ: 191.1, 136.3, 129.1, 126.9, 125.9, 125.2, 121.6,

120.1, 110.1, 41.5, 38.1, 32.9, 24.6, 23.1, 22.8, 14.0; IR (neat): 3016, 2926, 2860, 1669, 1495, 1050, 875, 824; MS (ES⁺) Calculated for [C₁₆H₁₉NONa]⁺ 264.1; Found: 264.3.



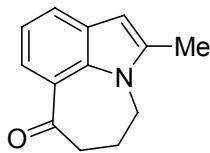
6

Compound **6** was isolated as the side product during the reaction of **3**. ¹H NMR (500MHz, CDCl₃) δ: 7.51 (d, 1H, J = 7.6 Hz), 7.27 (d, 1H, J = 8.0 Hz), 7.14-7.10 (m, 1H), 7.07-7.03 (m, 1H), 7.25 (d, 1H, J = 0.8 Hz), 4.14 (t, 2H, J = 7.6 Hz), 2.71 (t, 2H, J = 7.6 Hz), 2.41 (t, 2H, J = 7.2 Hz), 2.08 (quintet, 2H, J = 7.2 Hz), 1.76-1.69 (m, 2H), 1.46 (sextet, 2H, J = 7.6 Hz), 0.97 (t, 3H, J = 7.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 177.8, 140.9, 136.5, 128.2, 120.6, 119.8, 119.3, 108.9, 99.1, 41.9, 30.7, 30.7, 26.3, 24.9, 22.6, 13.9; IR (neat): 2926, 2861, 1706, 1699, 1608, 1495, 1452, 1258, 1232, 1050, 875, 824; MS (ES⁺) Calculated for [C₁₆H₂₁NONa]⁺ 282.2; Found: 282.0.



8a

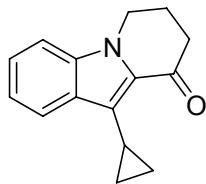
Compound **8a** was isolated in 83% yield following the general procedure D. Reaction time: 1.5 h. ¹H NMR (400MHz, CDCl₃) δ: 7.70 (d, 1H, J = 8.4 Hz), 7.40~7.25 (m, 2H), 7.15 (t, 1H, J = 6.8 Hz), 4.19 (t, 2H, J = 5.6 Hz), 2.72 (t, 2H, J = 6.4 Hz), 2.68 (s, 3H), 2.38~2.32 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 191.4, 136.3, 129.4, 127.3, 125.9, 121.4, 120.1, 119.7, 110.0, 41.4, 37.9, 23.1, 120.3; IR (neat): 2926, 2253, 1661, 1539, 1494, 1452, 1376, 1332, 1234, 1107, 1050, 824; MS (ES⁺) Calculated for [C₁₃H₁₃NONa]⁺ 222.0; Found: 221.7.



9a

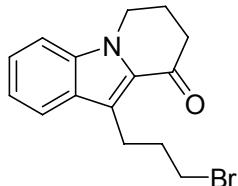
Compound **9a** was isolated as the side product during the reaction of **7a**. ¹H NMR (500MHz, CDCl₃) δ: 7.95 (dd, 1H, J = 1.0, 7.5 Hz), 7.72 (dd, 1H, J = 1.0, 7.5 Hz), 7.13 (t,

1H, $J = 7.5$ Hz), 6.39 (s, 1H), 4.24~4.22 (m, 2H), 3.12~3.09 (m, 2H), 2.45 (s, 3H), 2.37~2.32 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 198.9, 137.6, 134.6, 130.2, 125.7, 124.1, 120.6, 118.9, 101.5, 47.7, 44.6, 21.7, 13.6; IR (neat): 2925, 1660, 1607, 1494, 1451, 1261, 1050, 875, 824; MS (ES $^+$) Calculated for $[\text{C}_{13}\text{H}_{13}\text{NONa}]^+$ 222.1; Found: 222.0.



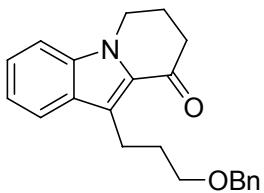
8b

Compound **8b** was isolated in 71% yield following the general procedure D. Reaction time: 14 h. ^1H NMR (400MHz, CDCl_3) δ : 7.66 (d, 1H, J = 5.2 Hz), 7.35~7.24 (m, 2H), 7.06 (dd, 1H, J = 6.8, 1.6 Hz), 4.16 (t, 2H, J = 5.6 Hz), 2.97~2.90 (m, 1H), 2.73 (t, 2H, J = 6.4 Hz), 2.36~2.30 (m, 2H), 1.16~1.06 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 190.9, 136.5, 130.4, 125.8, 125.7, 125.0, 122.4, 120.1, 110.3, 41.4, 38.1, 22.9, 8.9, 7.9; IR (neat): 3012, 2926, 1661, 1532, 1336, 1193, 1114, 1049, 1024, 875, 824; MS (ES $^+$) Calculated for $[\text{C}_{15}\text{H}_{15}\text{NONa}]^+$ 248.1; Found: 247.8.



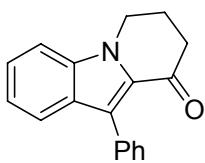
8c

Compound **8c** together with the corresponding chloride was isolated as an inseparable mixture in a combined 88% yield following the general procedure D. Reaction time: 11 h. ^1H NMR (500MHz, CDCl_3) δ : 7.79 (d, 1H, J = 8.1 Hz), 7.41~7.38 (m, 1H), 7.34~7.32 (m, 1H), 7.19~7.15 (m, 1H), 4.22 (t, 2H, J = 5.9 Hz), 3.44 (t, 2H, J = 6.8 Hz), 3.31 (t, 2H, J = 7.2 Hz), 2.72 (t, 2H, J = 6.5 Hz), 2.39~2.34 (m, 2H), 2.28~2.22 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 191.1, 136.2, 129.2, 126.9, 126.8, 126.1, 122.7, 122.6, 121.4, 121.4, 120.5, 120.5, 110.2, 110.2, 45.0, 41.5, 37.9, 33.9, 33.6, 33.3, 23.4, 23.0, 22.1; IR (neat): 2927, 2254, 1664, 1536, 1494, 1452, 1050, 908, 824; MS (ES $^+$) Calculated for $[\text{C}15\text{H}16\text{BrNONa}]^+$ 328.0; Found: 328.2; for $[\text{C}15\text{H}16\text{ClrNONa}]^+$ 284.1, Found: 283.8.



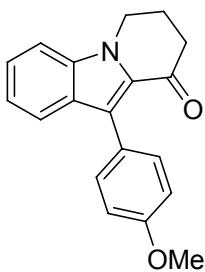
8d

Compound **8d** was isolated in 81% yield following the general procedure D. Reaction time: 2 h. ^1H NMR (400MHz, CDCl_3) δ : 7.75 (d, 1H, J = 8.4 Hz), 7.39~7.24 (m, 7H), 7.15~7.11 (m, 1H), 4.49 (s, 2H), 4.19 (t, 2H, J = 6.0 Hz), 3.53 (t, 2H, J = 6.4 Hz), 3.27 (t, 2H, J = 7.6 Hz), 2.70 (t, 2H, J = 6.4 Hz), 2.37~2.31 (m, 2H), 2.05~1.98 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 191.0, 138.8, 136.2, 129.1, 128.3, 127.6, 127.3, 127.0, 126.0, 124.2, 121.6, 120.3, 110.1, 72.7, 70.1, 41.4, 37.9, 30.4, 23.0, 21.4; IR (neat): 2927, 2253, 1663, 1536, 1495, 1453, 1108, 908, 824; MS (ES $^+$) Calculated for $[\text{C}_{22}\text{H}_{33}\text{NO}_2\text{Na}]^+$ 356.2; Found: 356.0.



8e

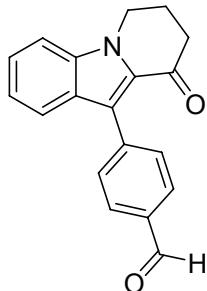
Compound **8e** was isolated in 64% yield following the general procedure D. Reaction time: 23 h. ^1H NMR (400MHz, CDCl_3) δ : 7.71 (d, 1H, J = 8.0 Hz), 7.60 (d, 2H, J = 7.2 Hz), 7.48~7.34 (m, 5H), 7.17 (dd, 1H, J = 1.2, 6.0 Hz), 4.32 (t, 2H, J = 5.6 Hz), 2.76 (t, 2H, J = 6.0 Hz), 2.48~2.38 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 189.7, 136.5, 133.5, 130.5, 128.3, 127.8, 127.3, 126.7, 126.2, 123.1, 122.5, 121.3, 110.2, 41.8, 38.1, 22.9; IR (neat): 2317, 1674, 1494, 1457, 1052, 874, 824; MS (ES $^+$) Calculated for $[\text{C}_{18}\text{H}_{15}\text{NONa}]^+$ 284.1; Found: 284.3.



8f

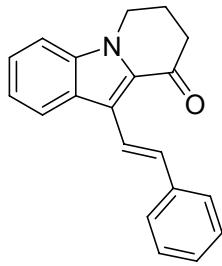
Compound **8f** was isolated in 73% yield following the general procedure D. Reaction time: 11 h. ^1H NMR (500MHz, CDCl_3) δ : 7.71 (d, 1H, J = 8.0 Hz), 7.56~7.53 (m,2H),

7.42~7.37 (m, 2H), 7.16 (t, 1H, J = 8.0 Hz), 7.01~6.98 (m, 2H), 4.29 (t, 2H, J = 6.0 Hz), 3.87 (s, 3H), 2.74 (t, 2H, J = 6.0 Hz), 2.43~2.38 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 189.8, 158.8, 136.5, 131.6, 128.2, 126.7, 126.1, 125.7, 122.9, 122.5, 121.1, 113.4, 110.1, 55.2, 41.8, 38.1, 22.9; IR (neat): 2926, 2253, 1669, 1608, 1494, 1451, 1248, 1050, 824; MS (ES $^+$) Calculated for $[\text{C}_{19}\text{H}_{17}\text{NONa}]^+$ 314.1; Found: 314.0.



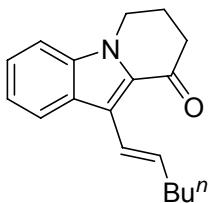
8g

Compound **8g** was isolated in 70% yield following the general procedure D. Reaction time: 34 h. ^1H NMR (400MHz, CDCl_3) δ : 10.06 (s, 1H), 7.96~7.94 (m, 2H), 7.77~7.75 (m, 2H), 7.69~7.68 (m, 2H), 7.47~7.41 (m, 2H), 7.23~7.19 (m, 1H), 4.33 (t, 2H, J = 5.6 Hz), 2.76 (t, 2H, J = 6.0 Hz), 2.47~2.41 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 192.1, 189.8, 140.3, 136.5, 135.0, 131.2, 129.3, 128.5, 126.4, 126.3, 121.9, 121.8, 121.3, 110.4, 41.9, 37.9, 22.8; IR (neat): 3049, 2925, 1694, 1673, 1602, 1495, 1451, 1403, 1331, 1208, 1094, 1050, 942, 875, 823; MS (ES $^+$) Calculated for $[\text{C}_{19}\text{H}_{15}\text{NONa}]^+$ 312.1; Found: 311.9.



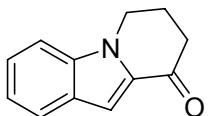
8h

Compound **8h** was isolated in 60% yield following the general procedure D. Reaction time: 14 h. ^1H NMR (400MHz, CDCl_3) δ : 8.27 (d, 1H, J = 16.8 Hz), 8.18 (d, 1H, J = 8.0 Hz), 7.62 (d, 1H, J = 7.6 Hz), 7.45~7.35 (m, 2H), 7.27~7.23 (m, 1H), 4.24 (t, 2H, J = 5.6 Hz), 2.77 (t, 2H, J = 6.0 Hz), 2.42~2.35 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 190.9, 138.2, 137.1, 131.6, 129.4, 128.6, 127.4, 126.5, 126.2, 124.6, 123.3, 122.5, 121.6, 120.2, 110.5, 41.6, 38.0, 22.8; IR (neat): 3055, 2927, 2246, 1661, 1596, 1510, 1494, 1448, 1408, 1367, 1336, 1303, 1276, 1247, 1194, 1109, 1010, 956, 909, 875, 824; MS (ES $^+$) Calculated for $[\text{C}_{20}\text{H}_{17}\text{NONa}]^+$ 310.1; Found: 310.0.



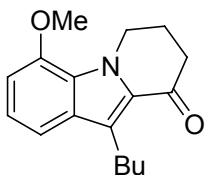
8i

Compound **8i** was isolated in 52% yield following the general procedure D. Reaction time: 48 h. ^1H NMR (400MHz, CDCl_3) δ : 8.02 (d, 1H, J = 8.4 Hz), 7.44~7.25 (m, 3H), 7.20~7.16 (m, 1H), 6.53 (dt, 1H, J = 7.2, 16.4 Hz), 4.21 (t, 2H, J = 6.0 Hz), 2.73 (t, 2H, J = 6.4 Hz), 2.39~2.31 (m, 4H), 1.56~1.51 (m, 2H), 1.47~1.38 (m, 2H), 0.95 (t, 3H, J = 7.2 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ : 190.9, 136.9, 135.3, 128.4, 126.0, 124.8, 123.3, 123.0, 121.1, 120.8, 110.3, 41.5, 38.0, 33.6, 31.8, 29.7, 22.8, 22.4, 14.0; IR (neat): 2955, 2926, 1667, 1607, 1494, 1451, 1050, 874, 824; MS (ES $^+$) Calculated for $[\text{C}_{18}\text{H}_{21}\text{NONa}]^+$ 290.2; Found: 290.0.



8j

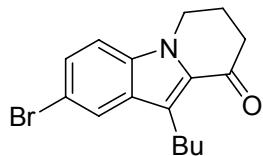
Compound **8j** was isolated in 73% yield using PtCl_4 as catalyst and in 95% yield using PtCl_2 following the general procedure D. Reaction time: 22 h. ^1H NMR (400MHz, CDCl_3) δ : 7.72 (d, 1H, J = 8.4 Hz), 7.39~7.33 (m, 2H), 7.31 (s, 1H), 7.16 (dd, 1H, J = 5.6, 1.6 Hz), 4.23 (t, 2H, J = 6.0 Hz), 2.74 (t, 2H, J = 7.2 Hz), 2.42~2.36 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 190.2, 137.3, 133.7, 126.6, 125.6, 123.4, 121.1, 110.3, 105.6, 41.3, 37.0, 23.2; IR (neat): 2925, 2348, 1671, 1472, 1050; MS (ES $^+$) Calculated for $[\text{C}_{12}\text{H}_{11}\text{NONa}]^+$ 208.1; Found: 208.0.



8k

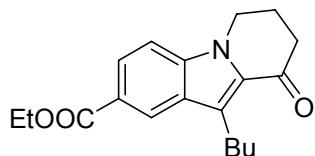
Compound **8k** was isolated in 89% yield following the general procedure D. Reaction time: 1 h. ^1H NMR (400MHz, CDCl_3) δ : 7.28 (d, 1H, J = 8.0 Hz), 7.00 (t, 1H, J = 8.0 Hz), 6.71 (t, 1H, J = 7.6 Hz), 4.68 (t, 2H, J = 6.0 Hz), 3.92 (s, 3H), 3.14 (t, 2H, J = 7.6 Hz), 2.66 (t, 2H, J = 6.8 Hz), 2.29 (quintet, 2H, J = 6.4 Hz), 1.66-1.58 (m, 2H), 1.45-1.36 (m, 2H), 0.93 (t, 3H, J = 7.6 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ : 191.3, 148.2, 129.5, 128.7, 127.0, 125.5, 120.1, 113.8, 105.3, 55.4, 44.8, 37.9, 32.8, 24.8, 23.6, 22.9, 14.0; IR (neat):

2927, 2861, 1659, 1537, 1495, 1451, 1241, 1051, 1016, 874, 824; MS (ES⁺) Calculated for [C₁₇H₂₁NO₂Na]⁺ 294.2; Found: 294.0.



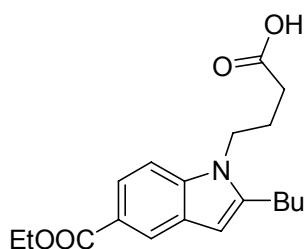
8l

Compound **8l** was isolated in 71% yield following the general procedure D. Reaction time: 1 h. ¹H NMR (400MHz, CDCl₃) δ: 7.84 (d, 1H, J = 1.6 Hz), 7.42 (dd, 1H, J = 8.8, 1.6 Hz), 7.19 (d, 1H, J = 8.8 Hz), 4.18 (t, 2H, J = 6.0 Hz), 3.11 (t, 2H, J = 7.6 Hz), 2.72 (t, 2H, J = 6.4 Hz), 2.36 (quintet, 2H, J = 6.4 Hz), 1.65-1.57 (m, 2H), 1.40 (quintet, 2H, J = 7.6 Hz), 0.94 (t, 3H, J = 7.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 190.9, 134.8, 129.7, 128.7, 128.4, 124.3, 123.9, 113.3, 111.7, 41.6, 37.9, 32.8, 24.5, 22.9, 22.8, 14.0; IR (neat): 2927, 2860, 1668, 1538, 1452, 1405, 1343, 1118, 1051, 904, 872, 824; MS (ES⁺) Calculated for [C₁₆H₁₈BrNONa]⁺ 242.1; Found: 241.6.

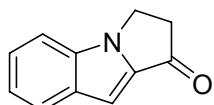


8m

Compound **8m** was isolated in 65% yield following the general procedure D. Reaction time: 1 h. ¹H NMR (400MHz, CDCl₃) δ: 8.50 (s, 1H), 8.04 (d, 1H, J = 8.8 Hz), 7.32 (d, 1H, J = 8.8 Hz), 4.42 (q, 2H, J = 7.6 Hz), 4.24 (t, 2H, J = 6.0 Hz), 3.20 (t, 2H, J = 7.6 Hz), 2.74 (t, 2H, J = 6.4 Hz), 2.38 (quintet, 2H, J = 6.0 Hz), 1.69-1.61 (m, 2H), 1.48-1.39 (m, 5H), 0.95 (t, 3H, J = 7.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 190.8, 167.2, 138.3, 130.1, 126.9, 126.6, 126.4, 124.8, 122.6, 109.8, 60.8, 41.8, 37.9, 33.0, 24.6, 22.8, 14.4, 14.0; IR (neat): 2928, 2860, 1691, 1665, 1540, 1495, 1452, 1385, 1312, 1250, 1122, 1050, 875, 824; MS (ES⁺) Calculated for [C₁₉H₂₃NO₃Na]⁺ 336.2; Found: 335.8.

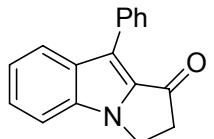


This compound was isolated as the side product during the reaction of **7m**. ¹H NMR (400MHz, CDCl₃) δ: 8.29 (d, 1H, *J* = 1.6 Hz), 7.86 (dd, 1H, *J* = 8.8, 1.6 Hz), 7.28 (d, 1H, *J* = 8.8 Hz), 6.35 (s, 1H), 4.38 (q, 2H, *J* = 7.2 Hz), 4.16 (t, 2H, *J* = 7.6 Hz), 2.72 (t, 2H, *J* = 7.6 Hz), 2.42 (t, 2H, *J* = 6.8 Hz), 2.08 (quintet, 2H, *J* = 7.2 Hz), 1.79-1.71 (m, 2H), 1.52-1.39 (m, 5H), 0.98 (t, 3H, *J* = 7.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 178.2, 167.9, 142.5, 139.1, 127.6, 122.7, 122.2, 121.6, 108.4, 100.5, 60.5, 42.2, 30.7, 30.5, 26.3, 24.8, 22.6, 14.4, 13.9; IR (neat): 2928, 2861, 1699, 1611, 1494, 1452, 1309, 1299, 1256, 1230, 1096, 1050, 874, 824; MS (ES⁺) Calculated for [C₁₉H₂₃NO₃Na]⁺ 354.2; Found: 353.9.

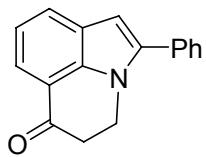


11

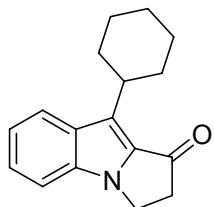
Compound **11** was isolated in 85% yield using PtCl₄ as catalyst and in 99% yield using PtCl₂ following the general procedure D. Reaction time: 8 h in the case of PtCl₄; 24 h in the case of PtCl₂. ¹H NMR (500MHz, CDCl₃) δ: 7.75 (d, 1H, *J* = 8.5 Hz), 7.41 (d, 1H, *J* = 8.5 Hz), 7.35 (t, 1H, *J* = 8.5 Hz), 7.18 (t, 1H, *J* = 8.5 Hz), 6.99 (s, 1H), 4.39 (t, 2H, *J* = 6.0 Hz), 3.18 (t, 2H, *J* = 6.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ: 192.8, 135.3, 135.2, 132.1, 125.1, 124.1, 121.5, 110.4, 98.8, 39.9, 39.4; IR (neat): 2253, 1713, 1540, 1494, 1451, 1050, 907, 824, 732; MS (ES⁺) Calculated for [C₁₁H₉NONa]⁺ 194.1; Found: 194.0.



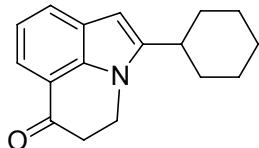
This compound was isolated in 67% yield along with 33% of the Friedel-Crafts acylation product (shown below) following the general procedure D. Reaction time: 4 h. ¹H NMR (400MHz, CDCl₃) δ: 8.01 (d, 1H, *J* = 8.0 Hz), 7.86 (d, 2H, *J* = 8.0 Hz), 7.48 (t, 2H, *J* = 7.6 Hz), 7.42~7.30 (m, 3H), 7.26~7.18 (m, 1H) 4.38 (t, 2H, *J* = 6.0 Hz), 3.21 (t, 2H, *J* = 6.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ: 192.0, 135.2, 132.6, 130.8, 130.3, 129.2, 128.5, 127.2, 125.4, 123.2, 121.8, 116.9, 110.5, 39.8, 39.5; IR (neat): 2253, 1705, 1553, 1494, 1451, 1050, 907, 824, 730; MS (ES⁺) Calculated for [C₁₇H₁₃NONa]⁺ 270.1; Found: 270.1.



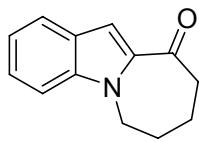
¹H NMR (400MHz, CDCl₃) δ: 7.83 (dd, 1H, *J* = 6.0, 0.8 Hz), 7.73 (dd, 1H, *J* = 6.4, 0.8 Hz), 7.56~7.42 (m, 5H), 7.22 (t, 1H, *J* = 6.0 Hz), 6.66 (s, 1H), 4.45 (t, 2H, *J* = 5.2 Hz), 3.10 (t, 2H, *J* = 5.2 Hz); ¹³C NMR (125 MHz, CDCl₃) δ: 192.8, 141.9, 141.1, 131.6, 128.8, 128.7, 128.4, 127.9, 126.6, 120.5, 118.6, 118.2, 102.7, 42.8, 38.2; IR (neat): 2926, 2253, 1682, 1592, 1494, 1454, 1050, 907, 731; MS (ES⁺) Calculated for [C₁₇H₁₃NONa]⁺ 270.1; Found: 270.1.



This compound was isolated in 67% yield along with 33% of the Friedel-Crafts acylation product (shown below) following the general procedure D. Reaction time: 2 h. ¹H NMR (500MHz, CDCl₃) δ: 7.84 (d, 1H, *J* = 8.5 Hz), 7.37~7.32 (m, 2H), 7.14 (t, 1H, *J* = 6.5 Hz), 4.36 (t, 2H, *J* = 6.5 Hz), 3.20 (m, 1H), 3.18 (t, 2H, *J* = 6.5 Hz), 1.99~1.92 (m, 2H), 1.86~1.76 (m, 5H), 1.47~1.41 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 192.6, 134.8, 131.2, 130.7, 125.1, 124.1, 123.1, 120.3, 110.4, 39.9, 39.4, 35.7, 33.2, 27.0, 26.0; IR (neat): 2928, 2253, 1699, 1558, 1494, 1450, 1307, 1050, 824; MS (ES⁺) Calculated for [C₁₇H₁₉NONa]⁺ 276.1; Found: 276.3.

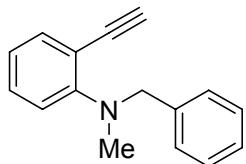


¹H NMR (500MHz, CDCl₃) δ: 7.72 (d, 1H, *J* = 7.6 Hz), 7.63 (d, 1H, *J* = 7.6 Hz), 7.13 (t, 1H, *J* = 7.6 Hz), 6.31 (s, 1H), 4.36 (t, 2H, *J* = 6.8 Hz), 3.07 (t, 2H, *J* = 6.8 Hz), 2.73~2.67 (m, 1H), 2.06~2.03 (m, 2H), 1.91~1.88 (m, 2H), 1.82~1.78 (m, 1H), 1.56~1.32 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) δ: 192.9, 147.7, 139.9, 127.9, 126.1, 119.8, 117.5, 117.4, 97.9, 41.5, 38.1, 35.8, 33.0, 26.5, 26.0; IR (neat): 2930, 2253, 1681, 1594, 1494, 1451, 1050, 907, 731, 650; MS (ES⁺) Calculated for [C₁₇H₁₉NONa]⁺ 276.1; Found: 276.1.



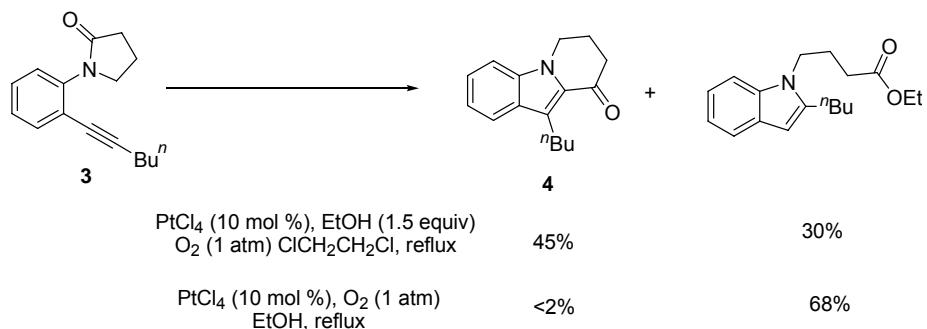
13

Compound **13** was isolated in 45% yield using PtCl_2 as catalyst following the general procedure D. Reaction time: 36 h. ^1H NMR (500 MHz, CDCl_3) δ : 7.69 (d, 1H, J = 8.0 Hz), 7.38~7.25 (m, 1H), 7.26 (d, 1H, J = 8.5 Hz), 7.15~7.12 (m, 1H), 4.38 (t, 2H, J = 6.0 Hz), 2.89~2.86 (m, 2H), 2.16~2.11 (m, 2H), 2.00~1.97 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 194.7, 139.2, 138.8, 126.3, 125.3, 123.1, 120.6, 110.2, 108.8, 43.2, 40.5, 26.8, 20.7; IR (neat): 2928, 2253, 1699, 1558, 1494, 1450, 1050, 824; MS (ES $^+$) Calculated for $[\text{C}_{13}\text{H}_{13}\text{NONa}]^+$ 222.0; Found: 222.1.



¹H NMR (500 MHz, CDCl₃) δ: 7.49 (dd, 1H, *J* = 1.5, 7.5 Hz), 7.37~7.23 (m, 6H), 6.92~6.88 (m, 2H), 4.47 (s, 2H), 3.34 (s, 1H), 2.74 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 154.7, 138.7, 135.0, 129.7, 128.3, 128.2, 126.9, 120.8, 118.3, 114.6, 82.8, 82.6, 60.2, 39.3; IR (neat): 2295, 1607, 1494, 1451, 1049, 907, 824; MS (ES⁺) Calculated for [C₁₆H₁₆N]⁺ 222.1; Found: 222.1.

Mechanism study: trapping acylium C and/or the preceding zwitterionic intermediate.



Experiment 1: Compound **3** was used as substrate, and the reaction was run according to the general procedure D except with the addition of 1.5 equiv. of EtOH. Reaction time: 1h. The ethyl ester was isolated in 30% yield along with product **4** (45% yield). For the ethyl ester: ^1H NMR (400MHz, CDCl_3) δ : 7.53 (d, 1H, J = 7.6 Hz), 7.29 (d, 1H, J = 7.6

Hz), 7.13 (dt, 1H, J = 7.6, 1.2 Hz), 7.06 (dt, 1H, J = 7.6, 1.2 Hz), 7.26 (s, 1H), 4.17-4.11 (m, 4H), 2.72 (t, 2H, J = 7.6 Hz), 2.34 (t, 2H, J = 7.2 Hz), 2.07 (quintet, 2H, J = 7.2 Hz), 1.78-1.70 (m, 2H), 1.48 (sextet, 2H, J = 7.6 Hz), 1.26 (t, 3H, J = 7.2 Hz), 0.98 (t, 3H, J = 7.6 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ : 172.8, 141.0, 136.5, 128.1, 120.5, 119.7, 119.2, 109.0, 98.9, 60.6, 42.0, 31.2, 30.6, 26.3, 25.1, 22.6, 14.2, 14.0; IR (neat): 3056, 2958, 2932, 1733, 1464, 1315, 1161, 1033, 874; MS (ES $^+$) Calculated for $[\text{C}_{18}\text{H}_{25}\text{NO}_2\text{Na}]^+$ 310.2; Found: 310.1.

Experiment 2: Compound **3** was used as substrate, and the reaction was run according to the general procedure D except that EtOH was used as solvent. Reaction time: 1h. The ethyl ester was isolated in 68% yield.

File: hxg-gzmade

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp. 25.0 C. / 298.1 K

Operator: walkup

File: hxg-gzmade

INOVA-500 "redjacket"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 8012.8 Hz

8 repetitions

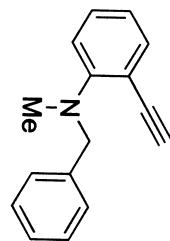
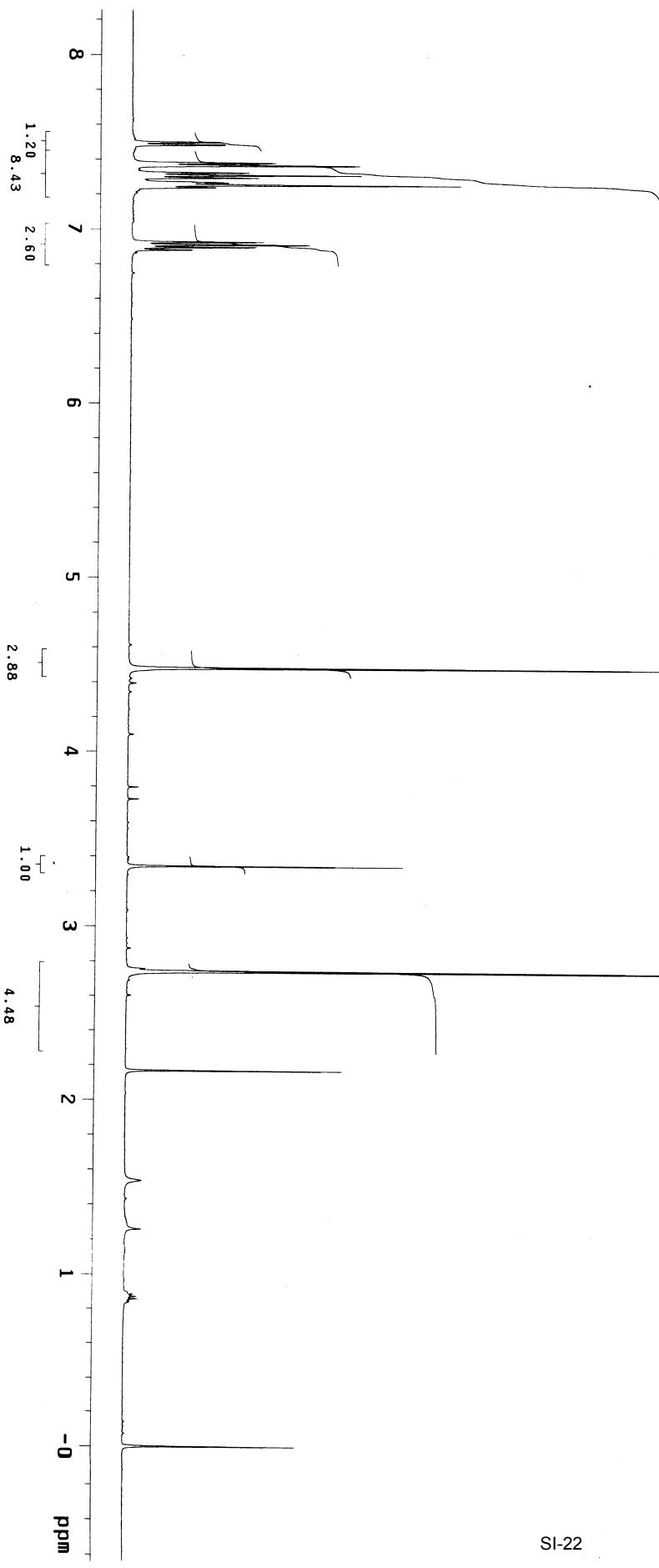
OBSERVE H1, 499.7991874 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrSys/data/auto_2007.06.25_11
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdc13

Temp: 25.0 C / 298.1 K

Operator: walkup

VNMRS-500 "nmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acc. time 1.300 sec

Width 30487.8 Hz

384 repetitions

OBSERVE C13, 125.6746044 MHz

DECUPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

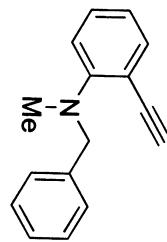
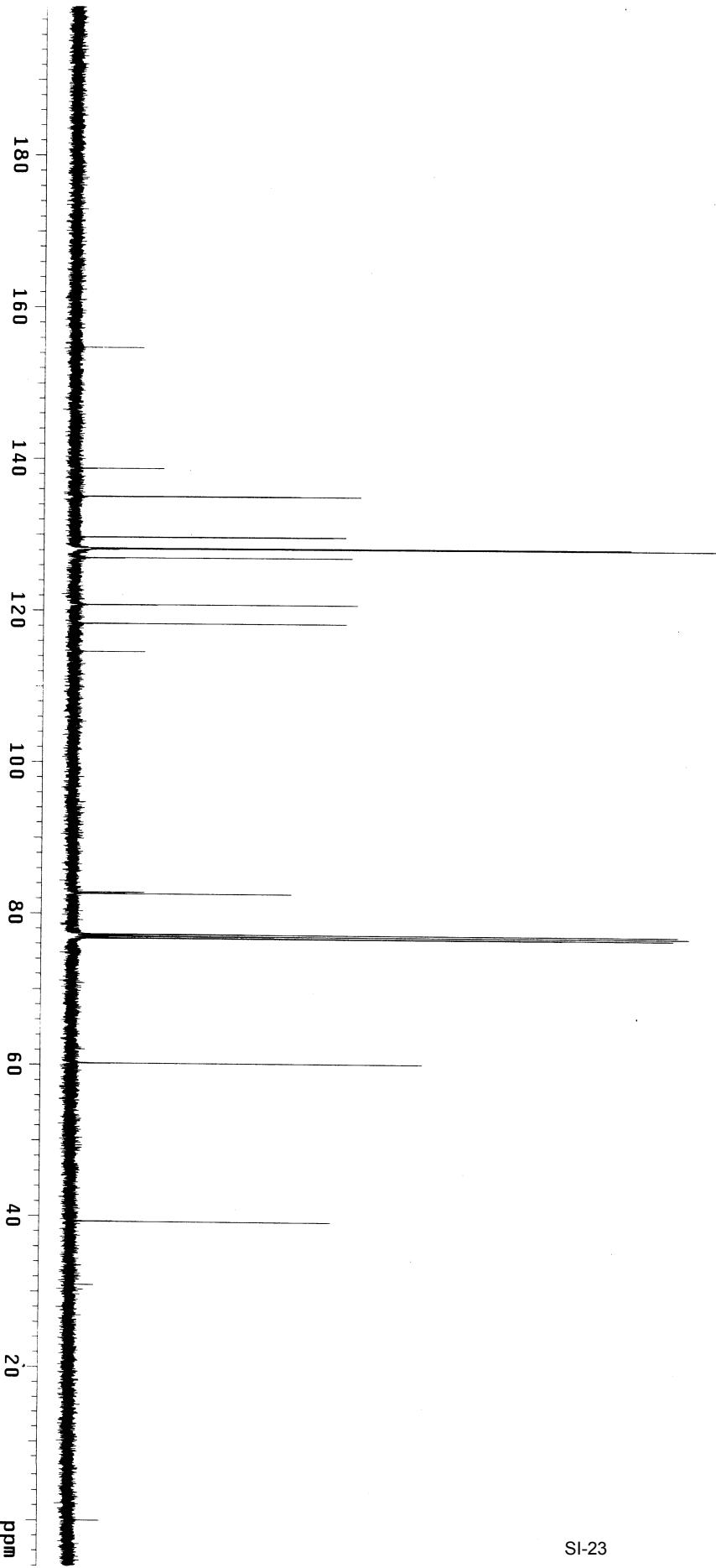
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

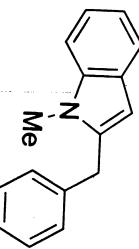
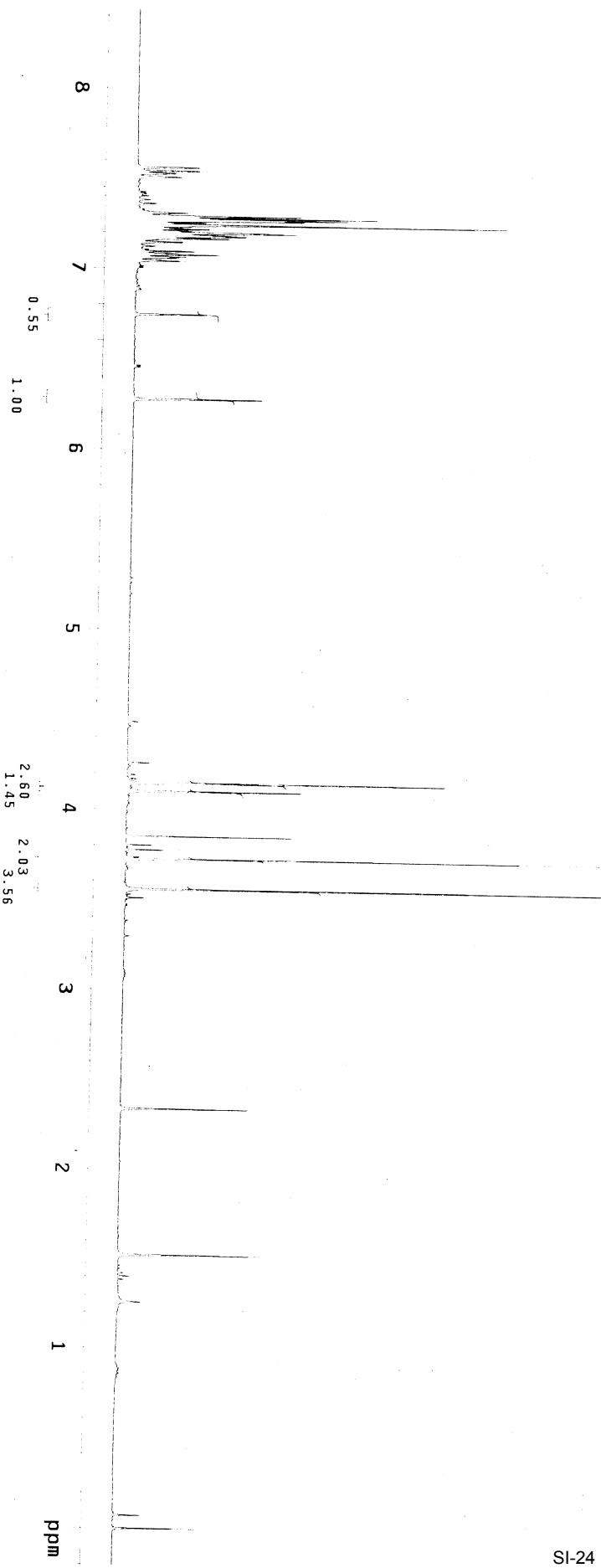
Total time 19 min, 42 sec



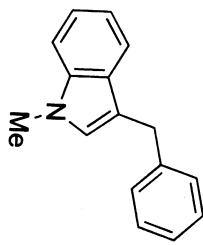
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Automation directory: /home/walkup/vnmrjsys/data/auto_2007.07.01_07
Filelist: S_20070701_002/fid
Sample id: S_20070701_002
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Pulse Sequence: s2pul
 Solvent: cdc13
 Temp: 25.0 C / 298.1 K
 Operator: walkup
 File: cdc13_01
 VNMRSS-400 "NMR400"

Relax. delay 1.000 sec
 pulse 45.0 degrees
 Acq. time 2.049 sec
 width 6410.3 Hz
 8 repetitions
 OBSERVE H1, 399.86556793 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 0 min, 30 sec



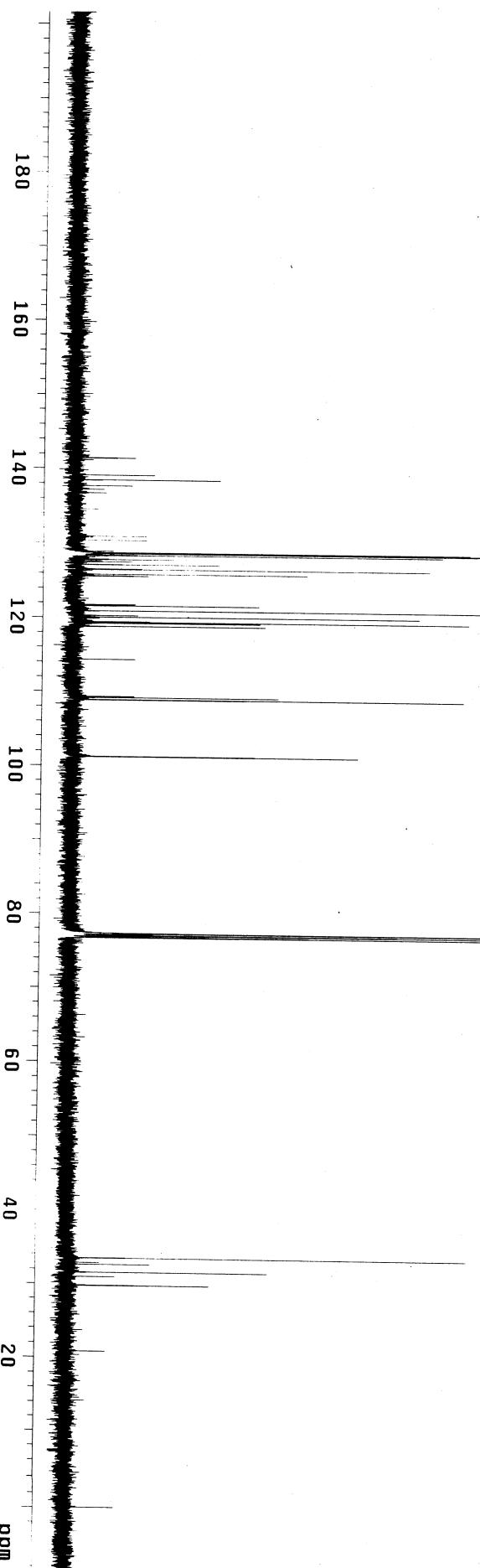
and

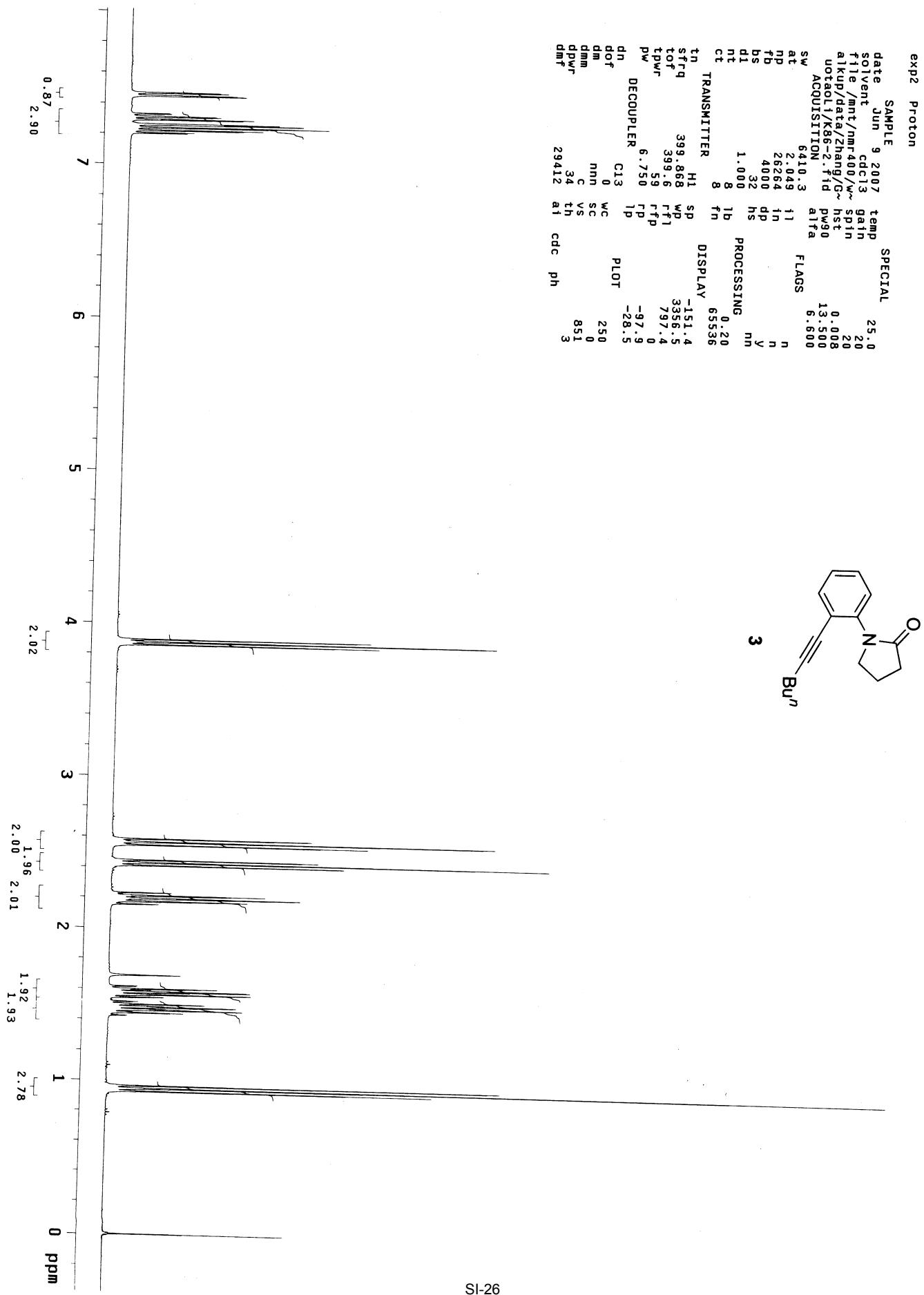


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Sample id : tmpstudy

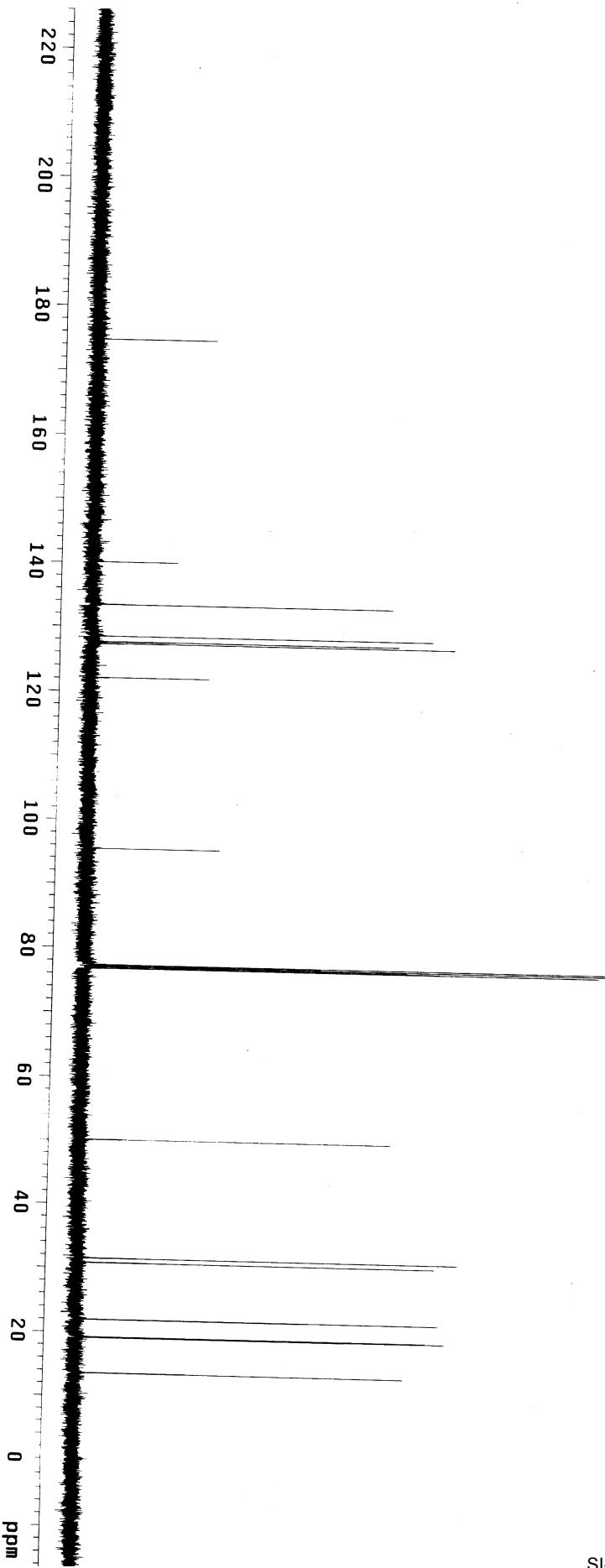
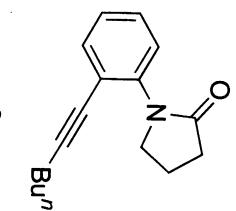
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: walkup
VNMRs-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
768 repetitions
OBSERVE C13, 125.6746067 MHz
DECOPLE H1, 499.8016822 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec





exptl Carbon
 SAMPLE date Jun 9 2007 temp SPECIAL 25.0
 solvent cdc13 gain 30
 ffile exp spin not used
 ACQUISITION sw 0.008
 30487.8 pw90 15.300
 at 1.300 a1fa 10.000
 np 79298
 f1b 17000 flags n
 bs 64 in n
 d1 1.000 d90 v
 nt 1.024 hs nn
 ct 128 PROCESSING nn
 TRANSMITTER lb 0.50
 tn C13 fn not used
 sfrq 125.688
 torf 1255.3 sp DISPLAY -2080.2
 tpwr pw 4.9 wp 30487.3
 7.650 rfp 1175.6
 rfp 9676.9
 decoupler dn H1 rp -38.6
 dof 0 1p PLOT -250.8
 dim yyy
 dmm w 250
 upwr 39 sc 23094
 dmrf 12200 vs 68
 ai cdc ph



File: hxg-4-25H

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: wakup

File: hxg-4-25H

INNOVA-500 "sittingbull1"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 8012.8 Hz

8 repetitions

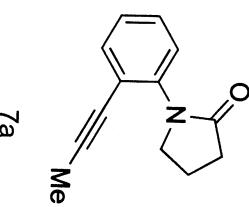
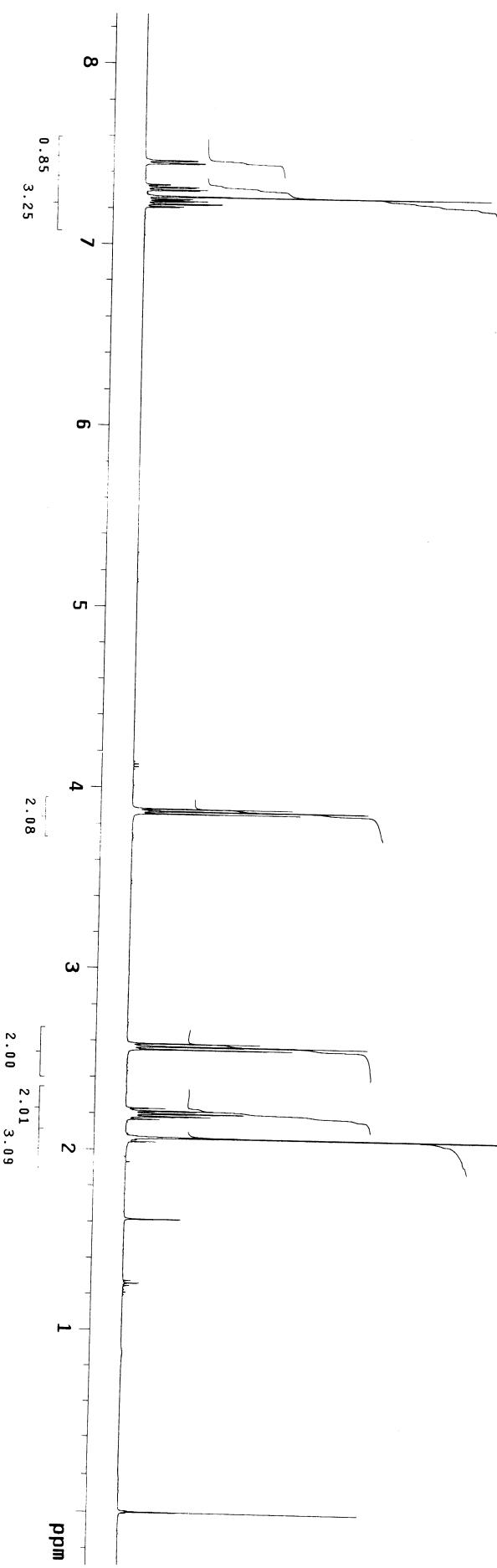
OBSERVE H1, 499.7991817 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_12

File : exp

Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: walkup

VNMR-500

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3048.78 Hz

512 repetitions

OBSERVE C13, 125.6746053 MHz

DECOPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

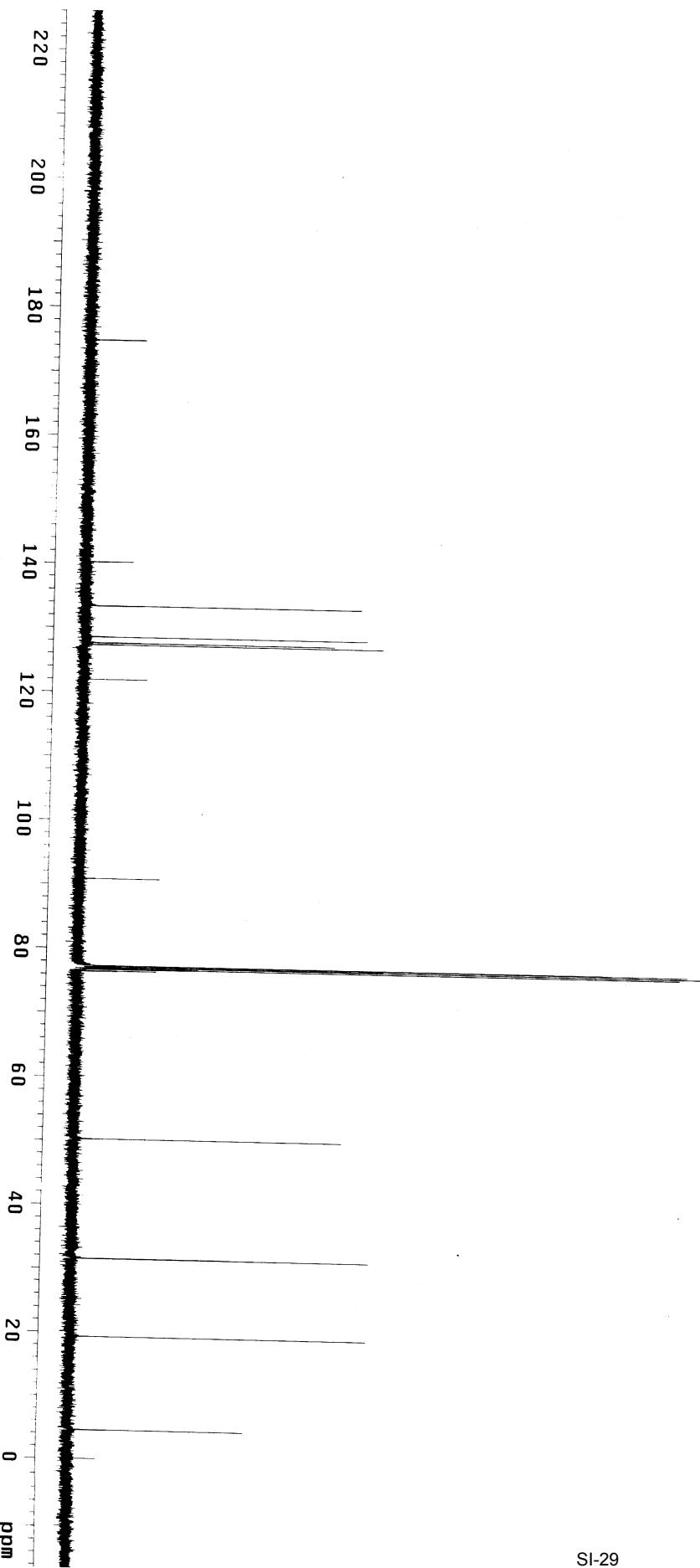
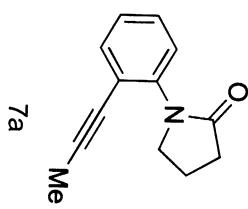
WALT-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

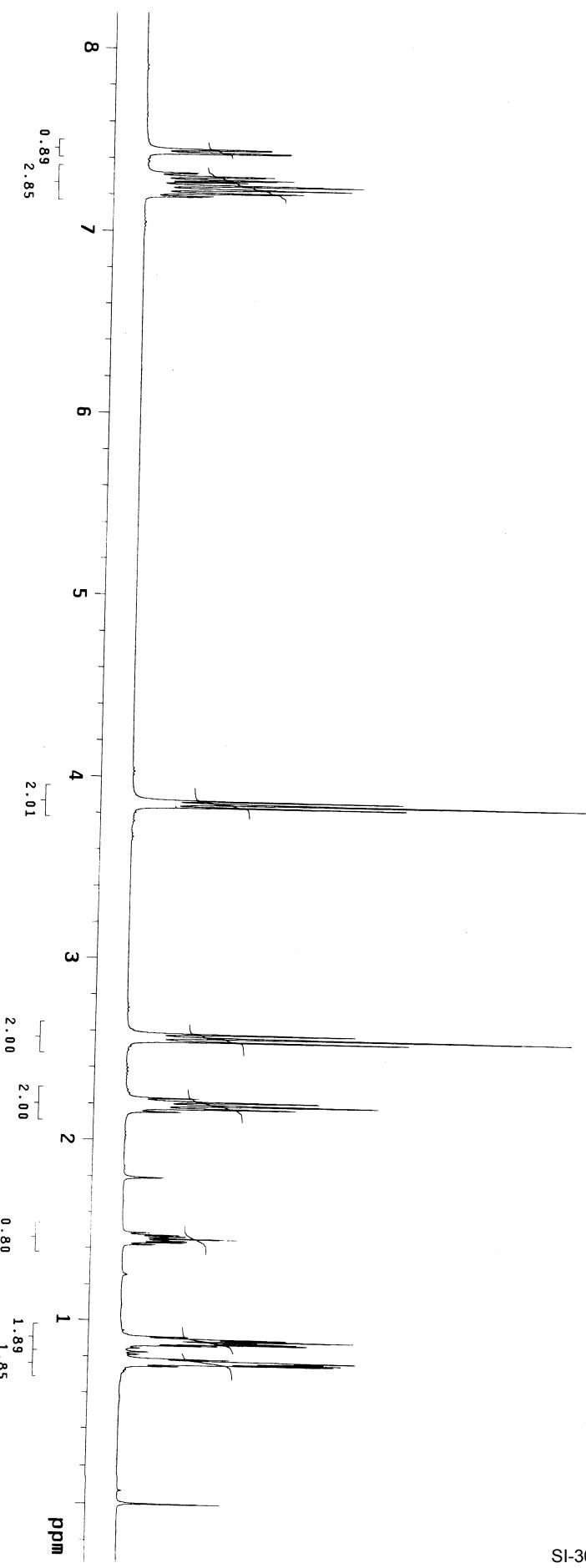
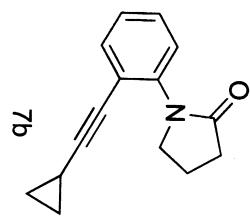
FT size 131072

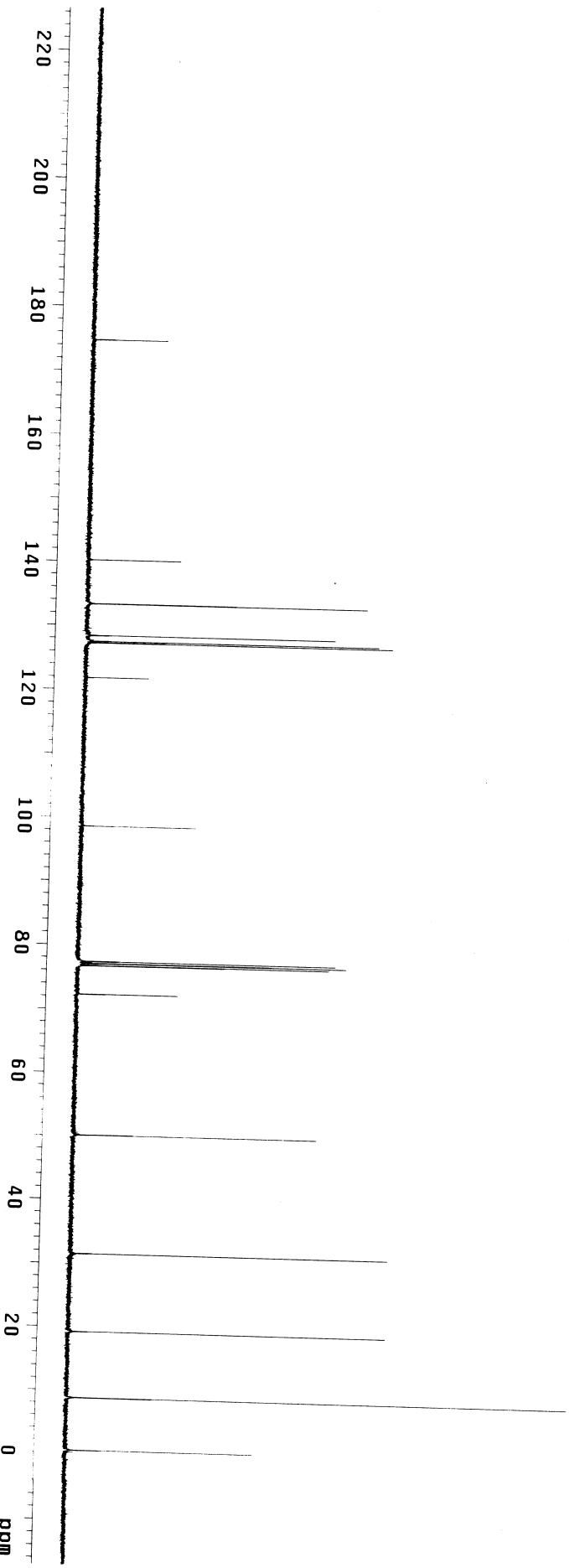
Total time 39 min, 24 sec



exp2 proton

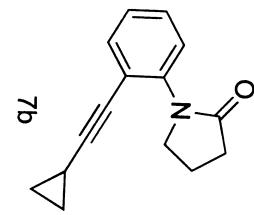
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alnup/dta/Zhang/G~		0.008
uotaol/K157-2.fid		0.008
ACQUISITION		13.500
sw	6410.3	pw90 6.600
at	2.049	alpha
nd	26264	FLAGS n
fo	4000	n
bs	400	y
d1	32	hs
nt	1.000	PROCESSING nn
ct	8	0.20
TRANSMITTER	8	DISPLAY 65536
tn	H1	sp -131.3
sfrq	399.868	wp 3409.2
trf	399.6	rf1 794.8
tpwr	59	rfp 0
pw	6.750	rp -22.1
DECOUPLER	1p	lp -19.9
dn	C13	PLOT
dof	0	250
dm	nnn	sc 0
dmm	c	vs 1044
dpr	34	th 2
	29412	ai cdc ph

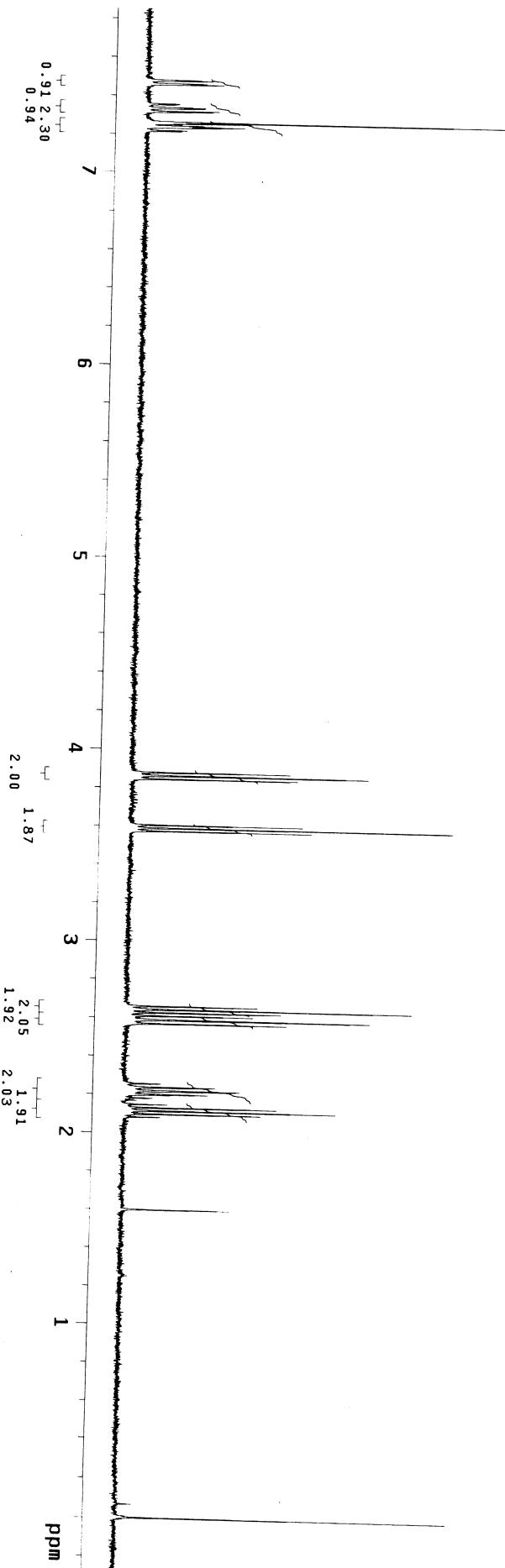




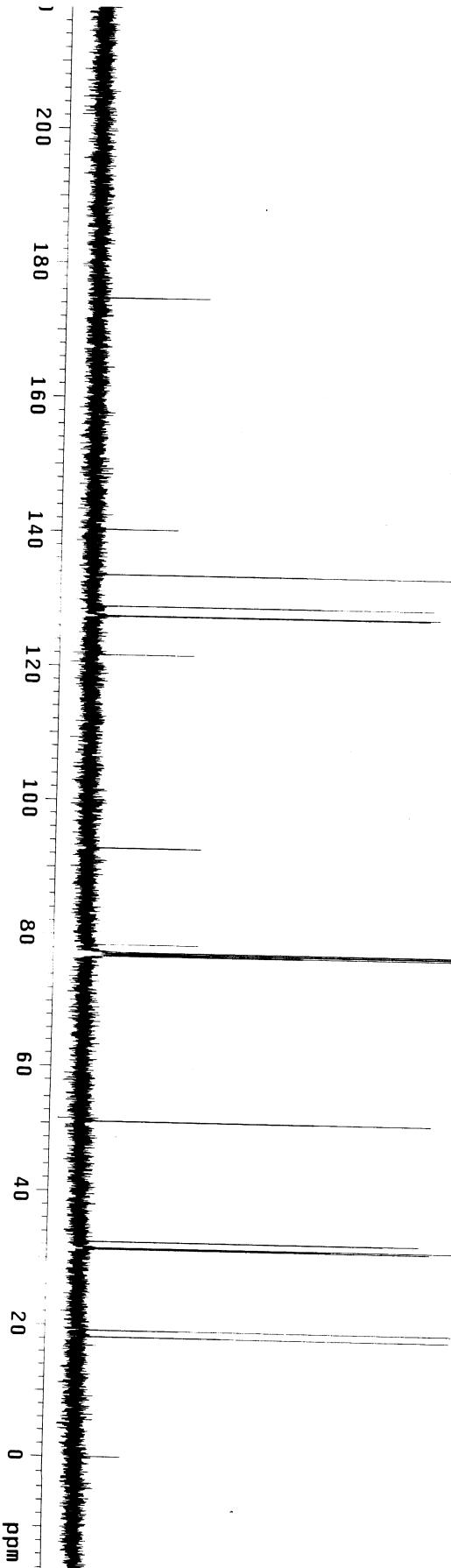
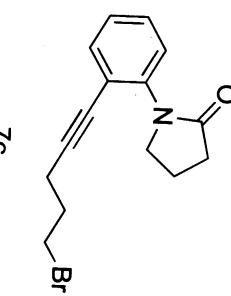
exp5 Carbon

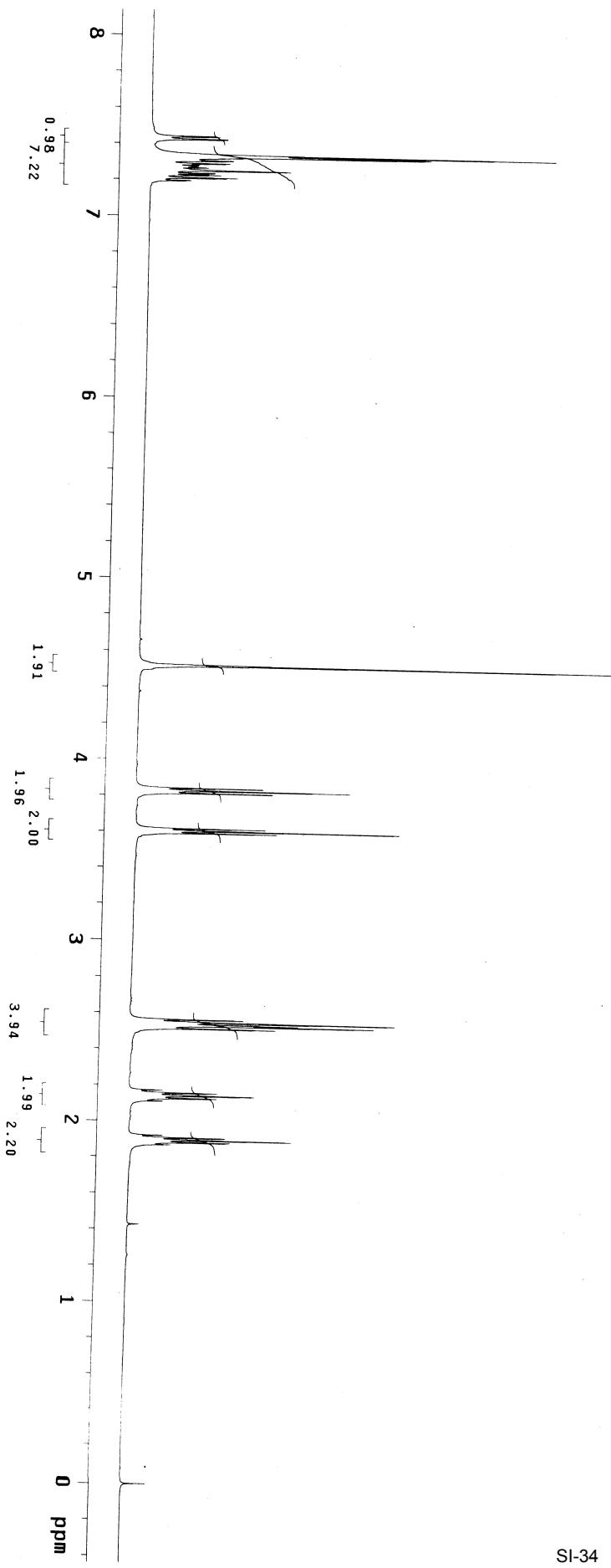
SAMPLE	2007	temp	25.0	SPECIAL	25.0
date	Jun	cdcl3	3.0	file	3.0
solvent		gain	20	/mnt/nmr00/w~	20
file		spin	0.008	alkup/data/Zhang/G~	0.008
alnkp		hst	11.900	uotaol1/K157-2carb~	11.900
alnkp		pw90	10.000	on.fid	10.000
alnkp		alnfa		alnfa	
ACQUISITION				FLAGS	
sw	24509.8	11	n		
at	1.300	in	n		
np	63750	dp	y		
fb	1700	hs	nn		
bs	64			PROCESSING	0.50
ai	1.000	1b	not used		
nt	3200	fn		DISPLAY	
ct	3200			SP	-1725.1
TRANSMITTER				wd	24509.1
tn	C13			rf1	9467.9
sfrq	100.557			rf2	7742.1
tof	1042.8			rp	179.7
tpwr	53			pp	
pw	5.950	1p			-199.3
DECOUPLER				PLOT	
dn	H1	wc			250
dof	0	sc			0
dm	yyy	vs			53937
dmm	w	th			7
dpr	41	ai			
dmt	9500	cdcl			
		ph			





expt Carbon
 SAMPLE 2007 temp 25.0
 solvent cdc13 gain 30
 file exp spin not used
 ACQUISITION 30487.8 hst 0.008
 sw 15.300 pw90 15.300
 at 1.300 np alfa 10.000
 np 79298 alfa
 fb 17000 flags
 js 8 in n
 j1 1.000 dp y
 j1 1024 hs nn
 ;t 392 PROCESSING 1.00
 TRANSMITTER 1b in not used
 C13 fn
 ;frq 125.688
 ;of 1255.3 sp
 ;pwr 49 wp -2077.9
 ;w 7.650 rfp 30487.3
 DECOUPLER H1 rfp 1755.3
 in 0 ip 9676.9
 of 1p 59.7
 mm yvv
 pw 39 wc 25.0
 mft 12200 sc 0
 pwr vs 66063
 ai cdc ph 9

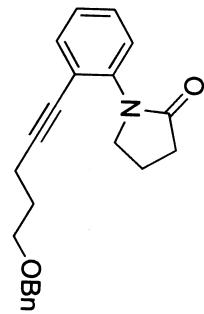


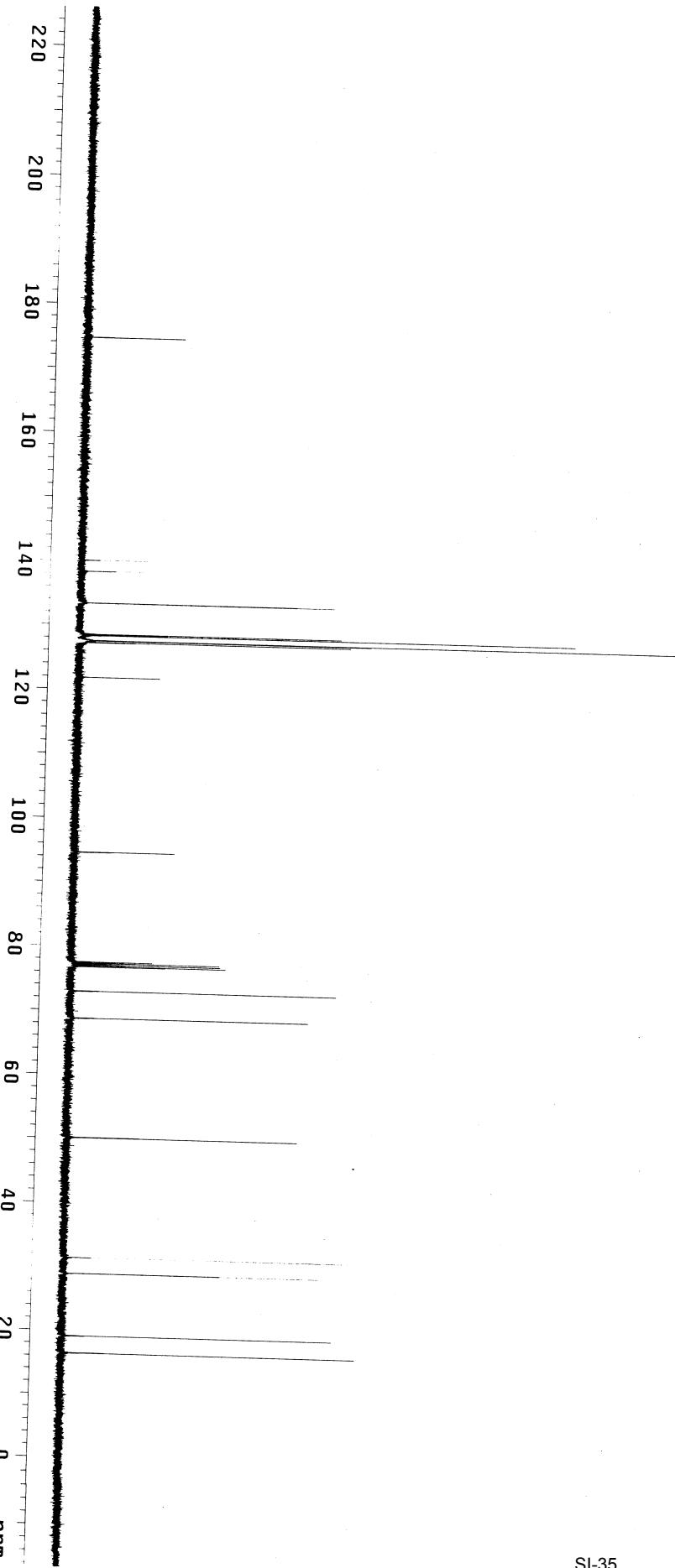


```

exp2 proton
SAMPLE SPECIAL
date Jun 2007 temp 25.0
Solvent cdc13 gain 30
file /mnt/nm/500/w~ spin not used
alikup/data/Zhang/w~ hst 0.008
1/K205.fid pw0 14.700
ACQUISITION 8012.8 alfa 6.600
at 2.049 11
np 32830 in
fo 4000 dip
bs 8 hs y
d1 1.000 PROCESSING nn
nt 8 fb 0.20
ct 8 fn DISPLAY 65536
TRANSMITTER H1 sp -220.3
tn 499.802 wp 4286.9
strq 499.7 rf1 1011.4
trf 499.7 rfp 54
tpwr 7.350 rp -174.5
pw 7.350 ip -13.0
DECOUPLER C13 PLOT
dn dof 250
dm nnn 0 wc
omm sc
dpwr c vs
dmf 34 th 38
32258 ai cdc ph 2

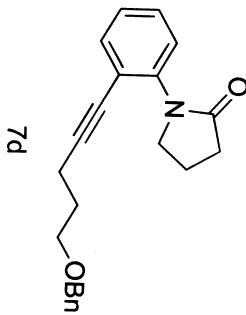
```





exp3 Carbon

SAMPLE	2007	temp	25.0	SPECIAL
date	Jun	gain	30	
Solvent	cdcl3	spin	not used	
file		not used		
ACQUISITION	exp	0.008		
sw	300.87.8	0.008		
at	1.300	15.300		
np	79298	pw0		
fb	17000	alfa	10.000	FLAGS
bs	16	in	n	
dd	1.000	dip	n	
nt	1.024	hs	y	
ct	80	PROCESSING	nn	
TRANSMITTER		1b	0.50	
tn	C13	fn	not used	
sfrq	125.688	sp	DISPLAY	
tof	1255.3	sp	-2092.3	
tpwr	49	wp	30487.3	
pw	7.650	rf1	11769.7	
DECOUPLER		rfp	9676.9	
dn	H1	rp	150.5	
dof	0	1p	-247.3	PLOT
dm	yyy			
dmm	wc			
dpr	39	sc		
dmf	12200	vs		
		th		
ai	cdcl	ph	7	



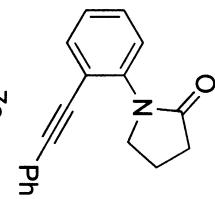
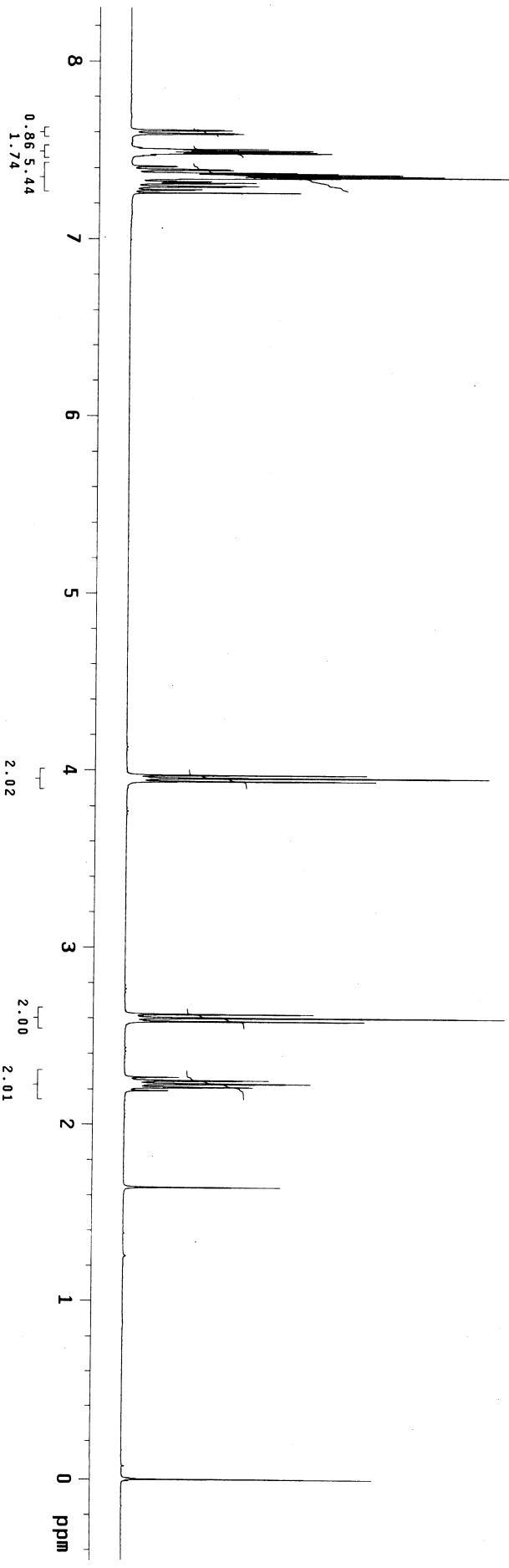
exp2 Proton

Proton

SAMPLE SPECIAL

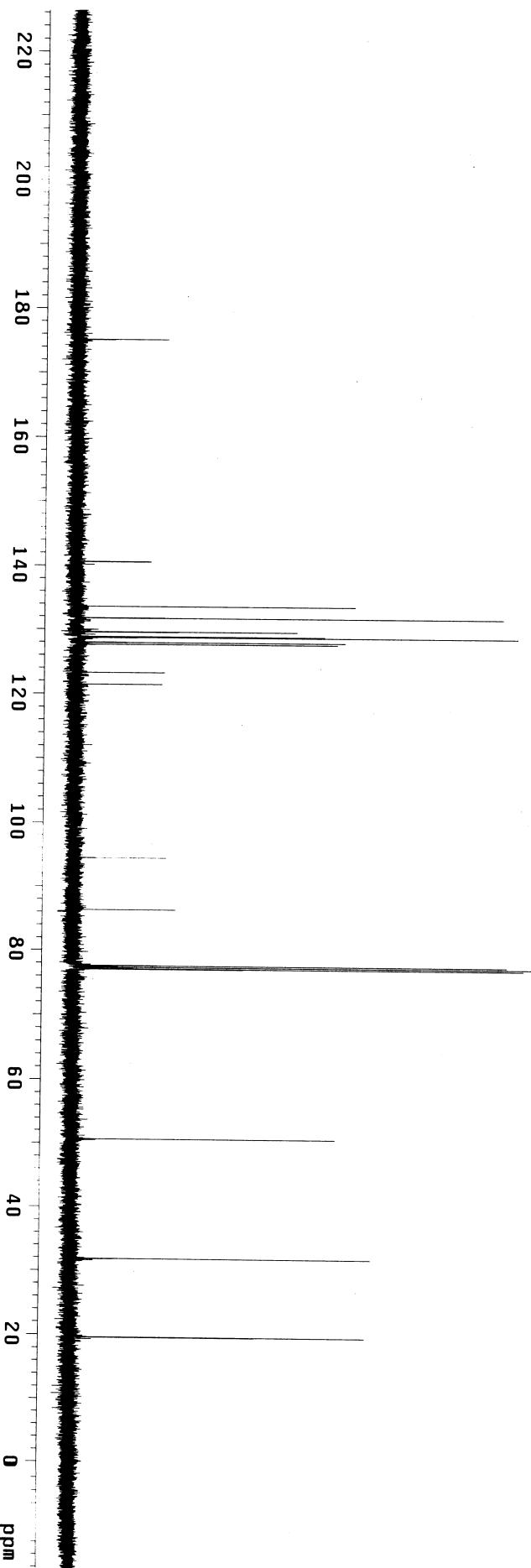
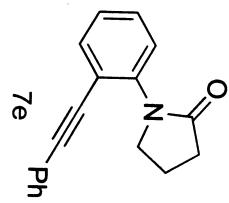
	date	Jun 10 2007	temp	25.0
solvent	cdc13	gain	20.0	0.0088
file	/mnt/nmr400/wr	spin	0.0088	13.5000
alikup/datta/Zhang/G~	hst			
uotab1/K63-2.fid	pw90			
ACQUISITION	alra			
sw	6410.3			6.6000
at	2.049	11		
np	26264	in		
fb	4000	dp		
bs	32	hs		
dl	1.000			
PROCESSING	FLAGS			
	nn			

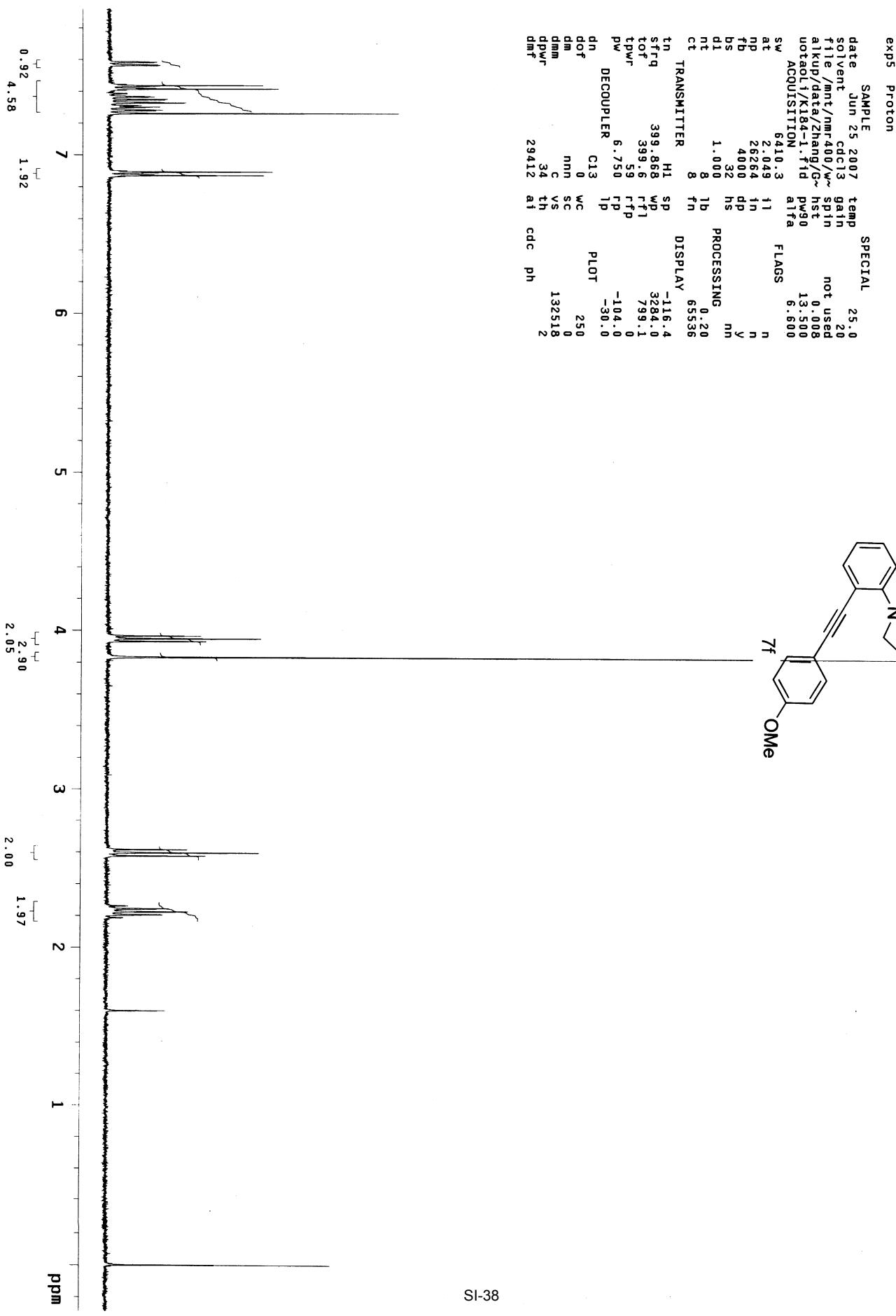
ct	nt	8	8	1b	0.20
ct	fn	8	fn	65536	65536
TRANSMITTER				DISPLAY	
tn		H1	sp	-183.1	
sfrq		399.868	wp	350.5	
tof		399.6	r ¹ p	799.7	
tpwr		59	r ¹ p	0	
6.750		r ¹ p	-93.8	-28.2	
DECOPPLER		1p			
dn	C13		PLOT		
dof	0	wc		250	
dmm	nnn	sc		0	
dmm	c	vs		487	
d ¹ pwr	34	th		3	
d ¹ m ¹	29412	ai	cdc	ph	

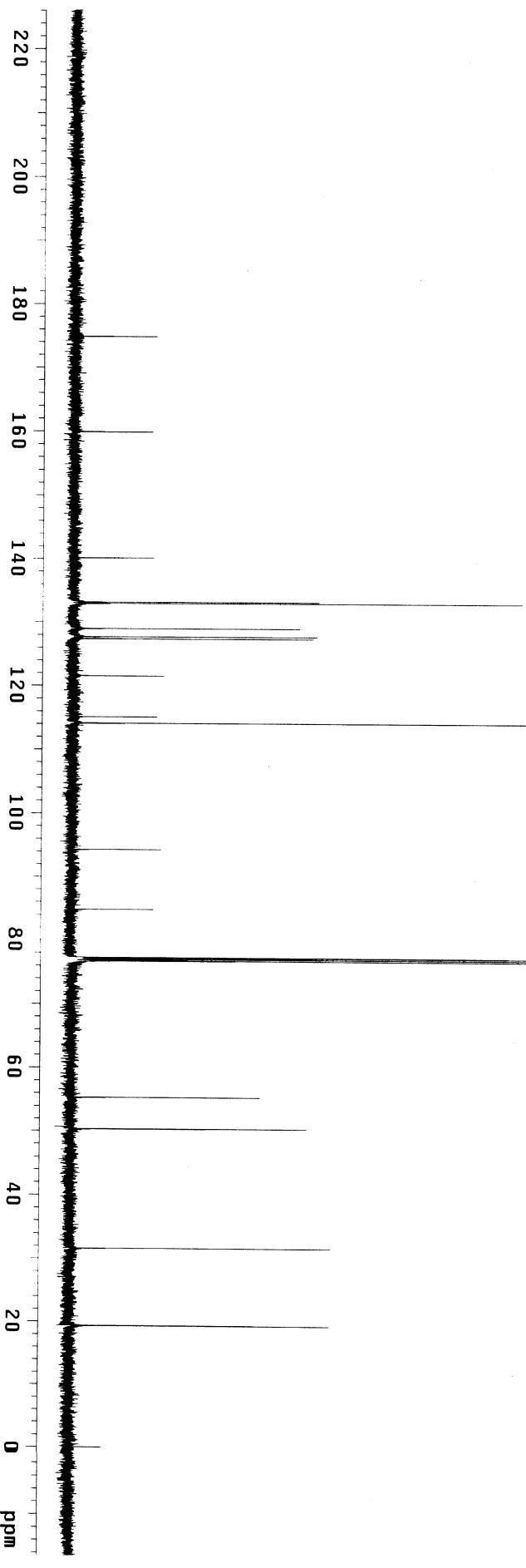


expt Carbon

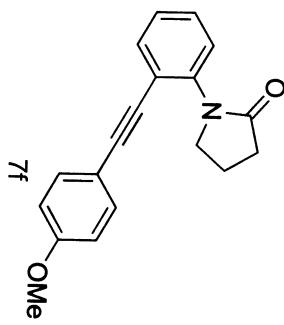
SAMPLE	9	temp	25.0
date	Jun 9 2007	cdcl3	30
solvent		gain	not used
file	/home/walkup/	spin	0.008
data/Zhang/L1/K63-~		hst	
2cation.fid		pwg0	15.300
ACQUISITION		alffa	10.000
sw	30487.8	FLAGS	
at	1.300	11	n
np	79.98	in	n
fb	17000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	0.50
nt	1.024	1b	not used
ct	64	fn	
TRANSMITTER	C13	SP	-2047.6
sfrq	125.688	wp	30487.3
tot	1255.3	rfl	2048.0
tpwr	49	rfp	0
pw	7.650	rp	-20.9
DECOUPLER		tp	-309.3
dn	H1	PLOT	
dof	0	250	
dm	w	sc	0
dmm	w	vs	17188
dppr	39	th	8
dmt	12200	ai	cdcl3
		ph	

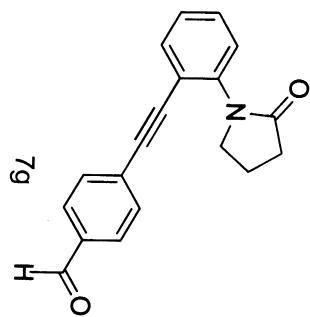
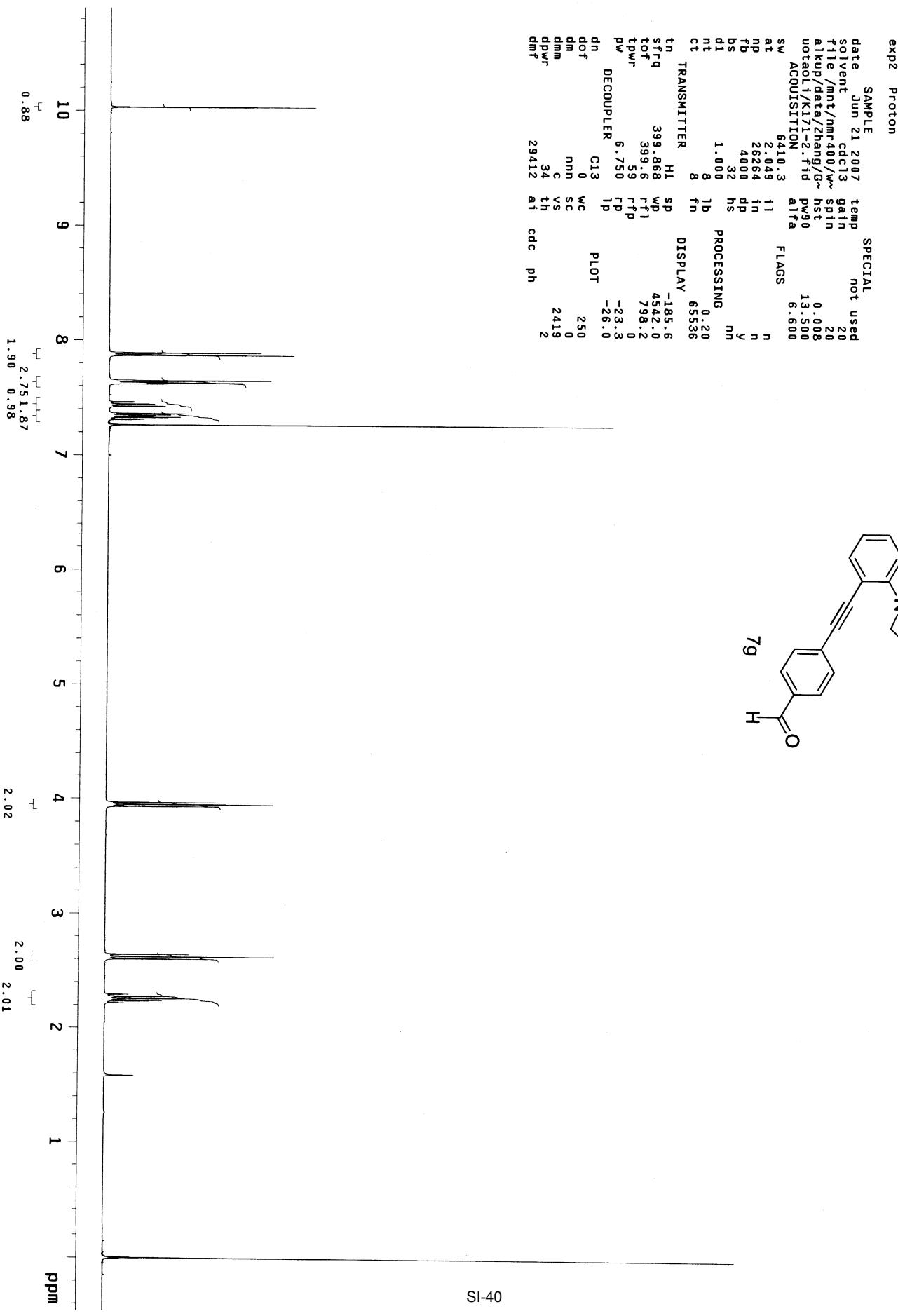


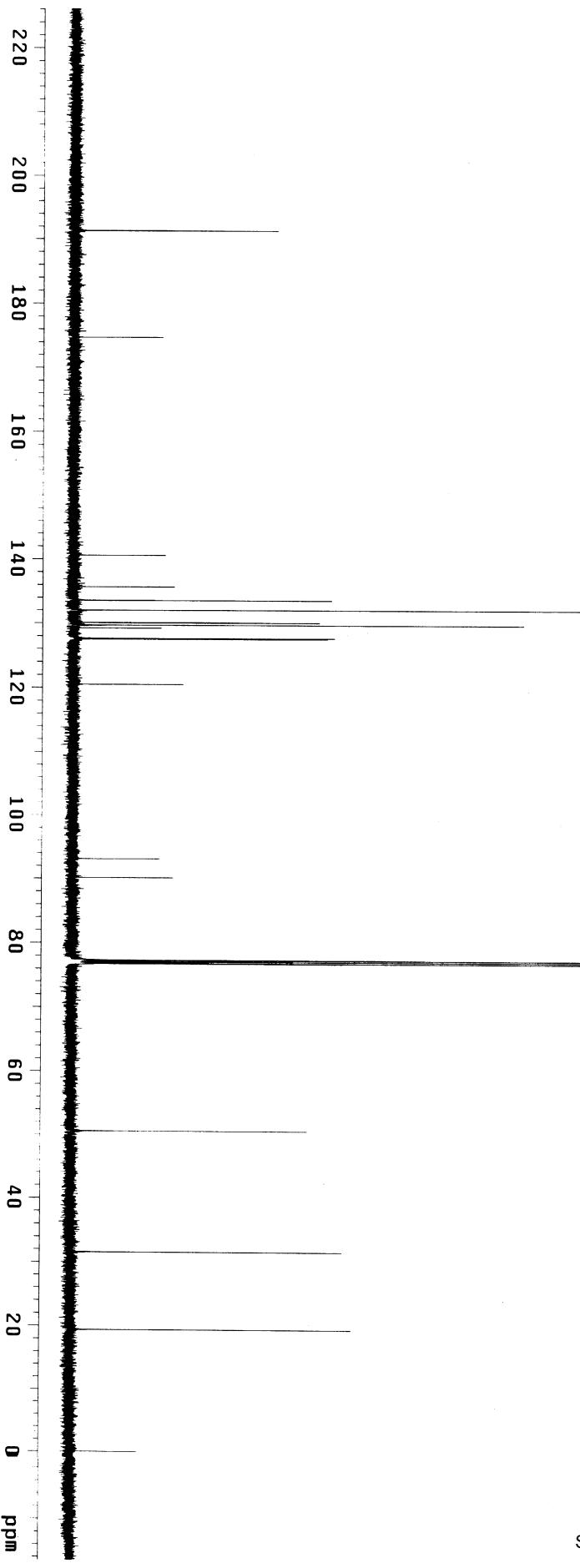




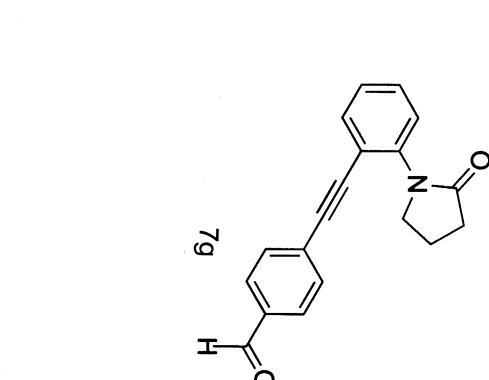
exptl Carbon
 SAMPLE Jun 26 2007 temp 25.0
 solvent cdc13 gain 30
 t1le acquisition exp spin not used
 sw 30487.8 pw90 0.008
 at 1.300 a1fa 15.300
 np 79298 a1fa 10.000
 fb 17000 flags n
 bs 11 n
 d1 8 n
 d1 1.000 dp y
 nt 1024 hs mn
 ct 456 lb
 tn 125.688 C13 fn
 sfrq 1255.3 not used
 tof 49 sp 1.00
 tpwr 7.650 w1 1.00
 pw r11
 DECOUPLER H1 rfp
 dn 0 ip
 dof 0 ip
 dm 39 wyy
 dmm 39 sc
 dpwr 12200 vs 250
 dm 12200 th 0
 a1 cdc ph 46606
 9

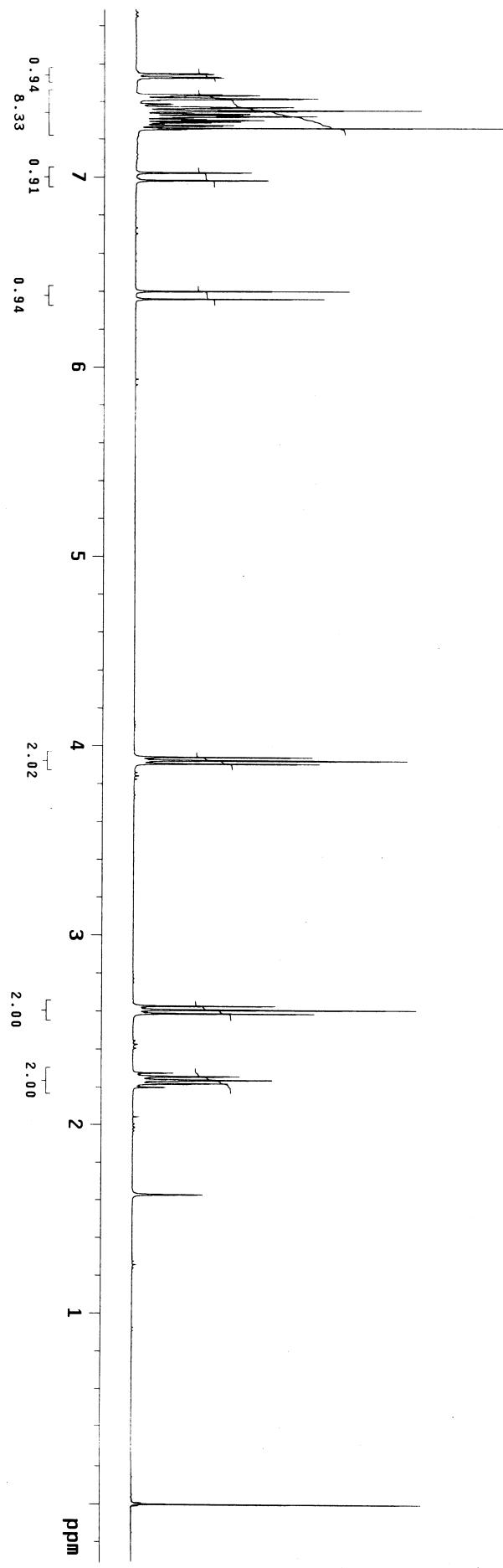






expt Carbon
 SAMPLE Jun 21 2007 temp 25.0
 date cdc13 gain 30
 solvent /home/walkup/~/ spin not used
 vnmrsys/data/auto_ hst 0.008
 2007-06-21_03/Aug_~ pw90 15.300
 01/data/cdcl3_01.f~ alfa 10.000
 id
 ACQUISITION 11
 sw 30487.8 n
 at 1.300 in
 np 79298 dp
 fb 1700 hs
 bs 64 1b
 d1 1.000 fn
 nt 1800 not used
 ct 1800 sp
 TRANSMITTER wp
 tn C13 rf1
 sfrq 125.688 rfp
 tof 125.3 rp
 tpwr 4.9 lp
 pw 7.650 PLOT
 DECOUPLER wc 250
 dn H1 sc 0
 dof 0 vs
 dm yyy 69796
 dmm th 8
 dpwr 39 a1 cdc ph
 dmt 12200

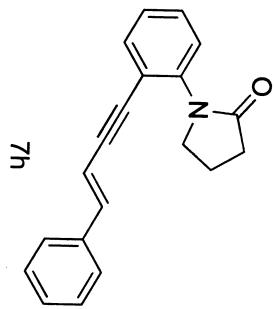




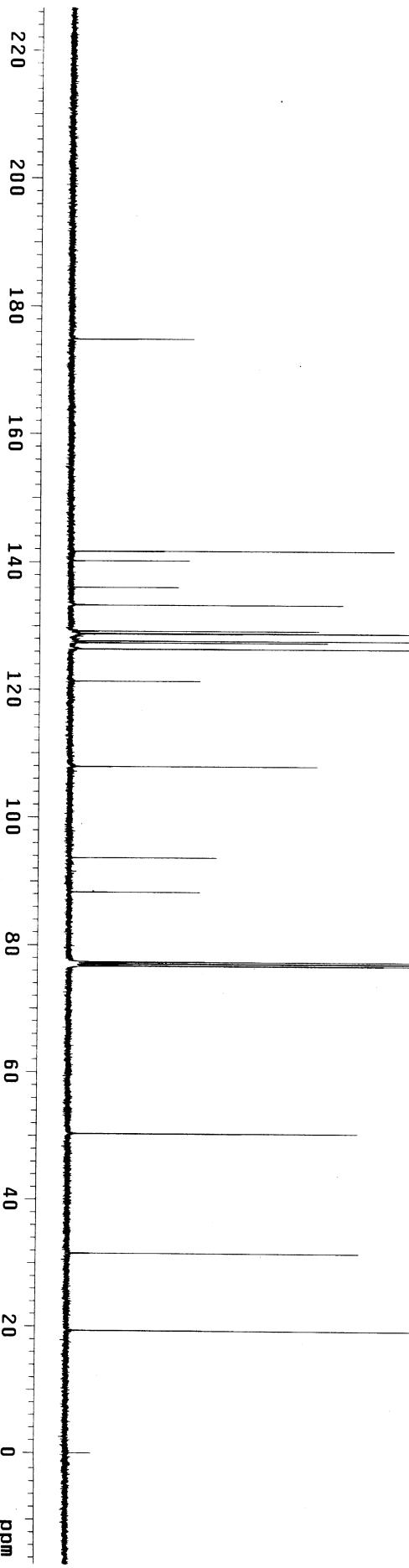
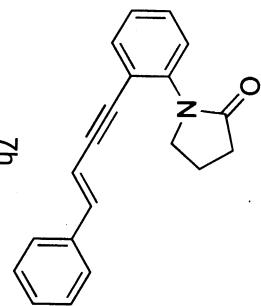
```

exp2 proton
SAMPLE Jun 11 2007 temp 25.0
date cdc13 gain 20
solvent /mnt/mm400/w spin 20
file /mnt/mm400/w hst 0.008
alikup/data/Zhang/G~ hst 13.500
uotao1/K114-2.fid pw90
ACQUISITION alfa 6.600
sw 6.410.3
at 2.049 11 n
np 26.64 1m n
fb 4000 dp y
bs 1.32 hs m
d1 1.000 1b 0.20
nt 8 fn 65536
ct 8
TRANSMITTER H1 sp -124.8
tn 399.868 wp 3278.3
sfrq 399.6 rf1 800.1
tof 559 rfp 0
tpwr 6.750 rp -98.4
pw 1p -27.5
DECOUPLER C13 PLOT 250
dn 0 wc 0
dor 0 nnn 0
dm 0 sc 0
dmm 0 vs 1177
dpr 34 th 3
dmt 29412 at cdc ph

```



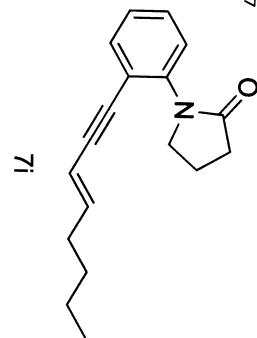
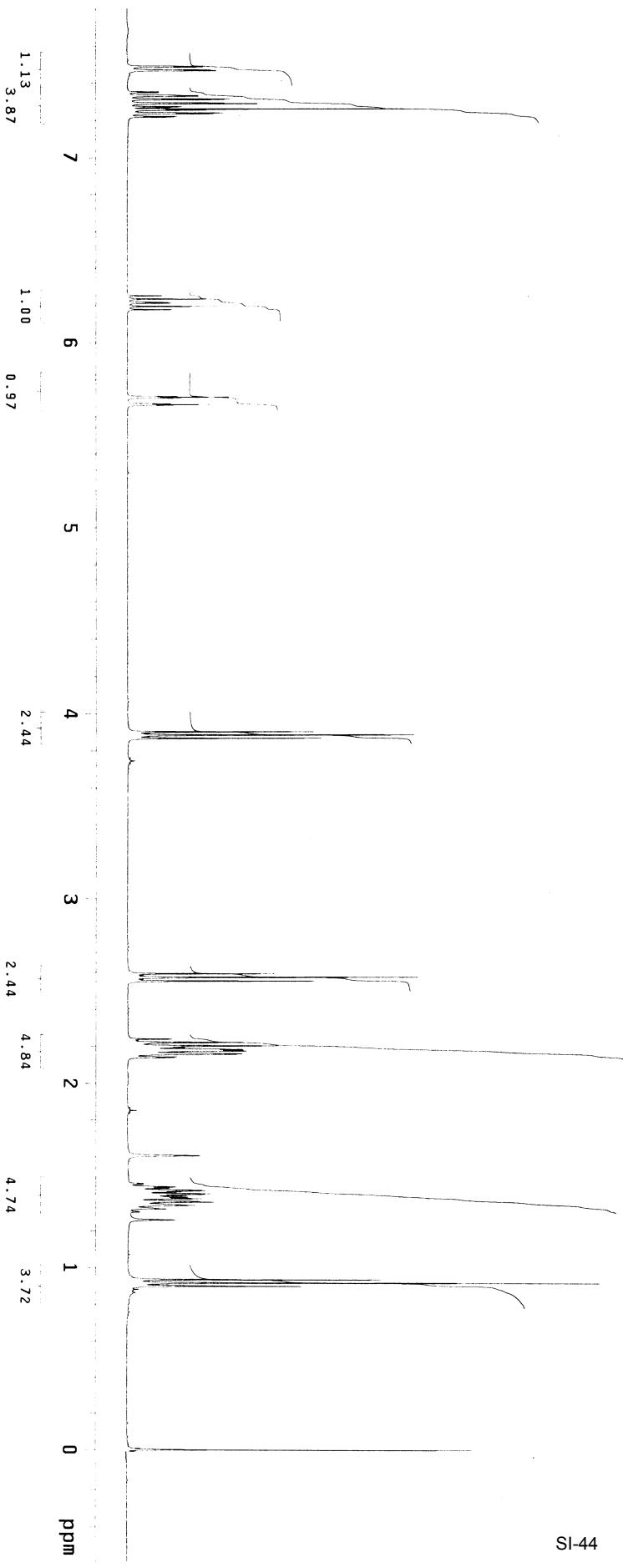
exp2 Carbon
SAMPLE Jun 11 2007 **temp** 25.0
date cdc13 **gain** 25.0
solvent /home/walkup/~/ **spin** 30
file /home/walkup/~/ **nmr400** 20
nmr400/walkup/data/~ **hst** 0.008
/Zhang/GuoaoLi/K1~ **pwg0** 11.900
14-2carbon.fid **alfa** 10.000
ACQUISITION
sw 2450.8 **tt** 11 **SPECIAL**
at 1.300 **in** n
np 63750 **dp** n
fb 17000 **hs** y
bs 64 **PROCESSING**
di 1.000 **lb** 0.50
nt 12000 **fn** not used
ct 64 **DISPLAY**
TRANSMITTER sp -1720.6
tn C13 **wp** 24509.1
sfrq 100 557 **rf1** 9463.4
tof 1042.8 **rfp** 7742.1
tpwr 53 **rp** 88.9
pw 5.950 **1p** -198.6
DECOPPLER
dn H1 **wc** 250
dof 0 **sc** 0
dov yyy
dm vs 141715
dpr w 12
dpr th
dpr a1 cdc ph
dpr 9500



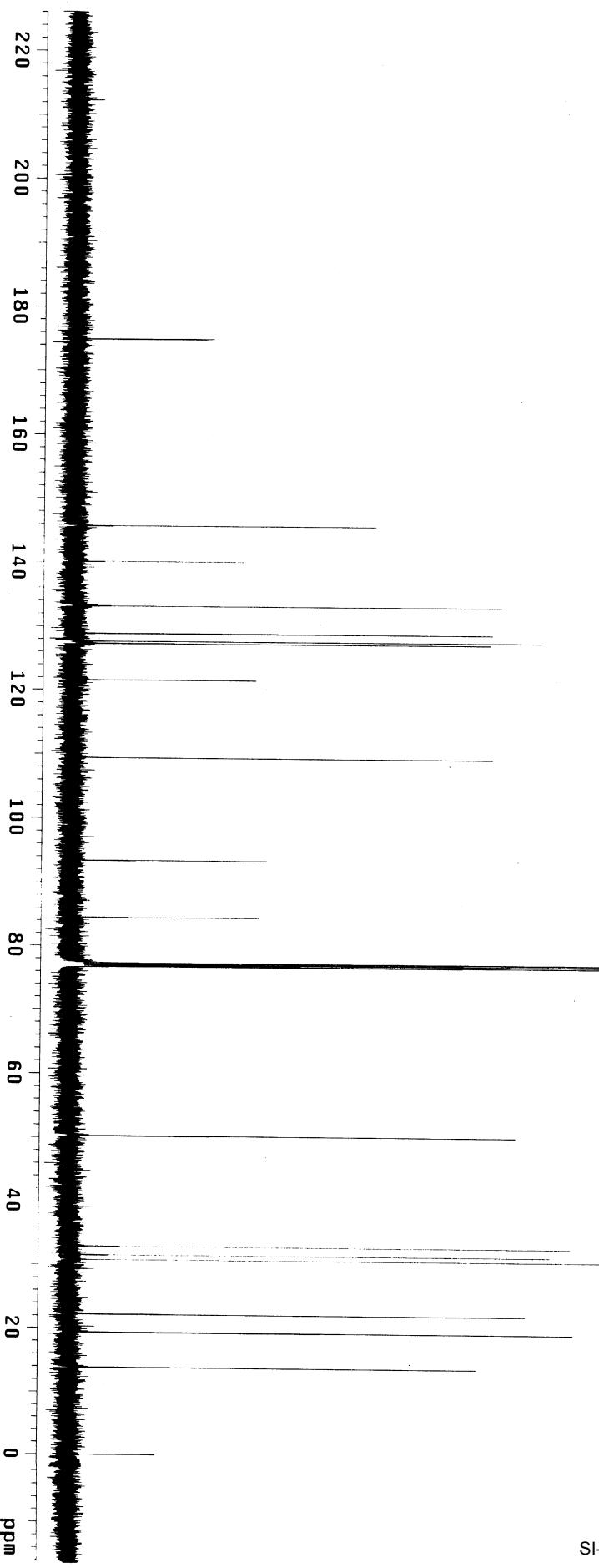
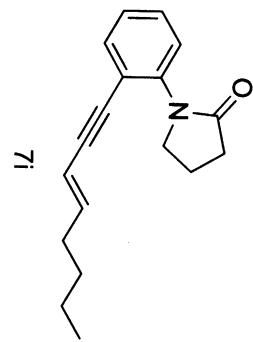
Automation directory: /home/walkup/vnrrsys/data/auto_2007.06.27
File : s_20070627_001/data/cdc13_01.fid
Sample id : s_20070627_001

Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: walkup
File: cdc13_01
VNMR-400 "NMR400"

Relax. delay 1.000 sec
pulse 45.0 degrees
Acq. time 2.049 sec
width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8656721 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



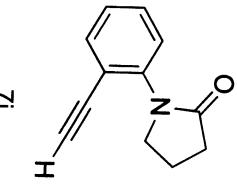
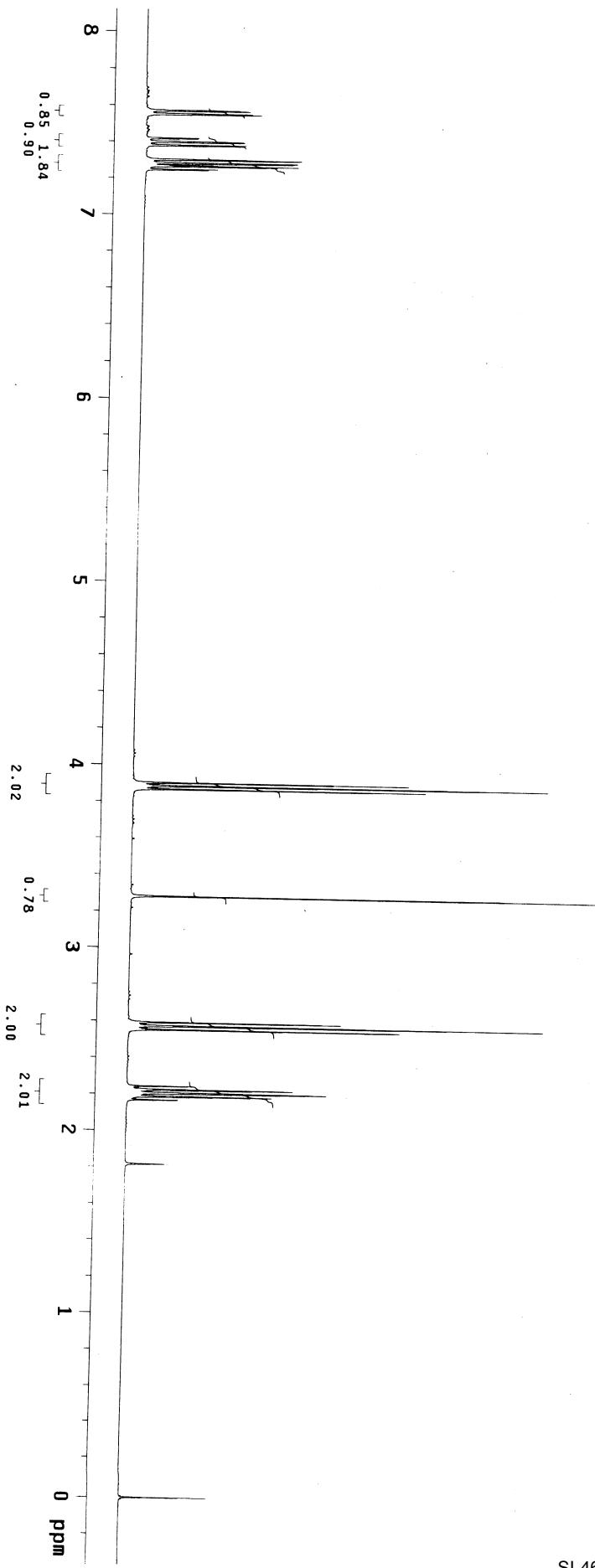
expt Carbon
SAMPLE Jun 14 2007 **temp** 25.0
date Jun 14 2007 **solvent** cdc13 **gain** 30
file /home/walkip/~/spin **not used**
data/ZhangXiaogen~ hst **0.003**
gHuang/hxg-4-2C.f1~ pw90 **15.300**
d alfa **10.000**
ACQUISITION
sw 30487.8 **11** **FLAGS** **SPECIAL**
at 1.300 **in** **25.0**
np 70298 **dp** **30**
fb 1700 **hs** **not used**
bs 64 **PROCESSING**
di 1.000 **1b** **0.50**
nt 2000 **fn** **0.50**
ct 1280 **not used**
TRANSMITTER **DISPLAY**
tn sp **-2077.4**
sfrq C13 **30487.3**
sf 125.688 **r1** **11754.9**
tof 1235.3 **rfp** **9676.9**
tpwr 49 **rp** **-92.9**
pw 7.650 **1p** **-264.2**
DECOUPLER **PLOT**
dn H1 **wc** **250**
dof 0 **sc** **0**
dm vyy **vs** **67983**
dmm vyy **th** **9**
dpr 39 **ai** **cdc** **ph**
dmt 12200

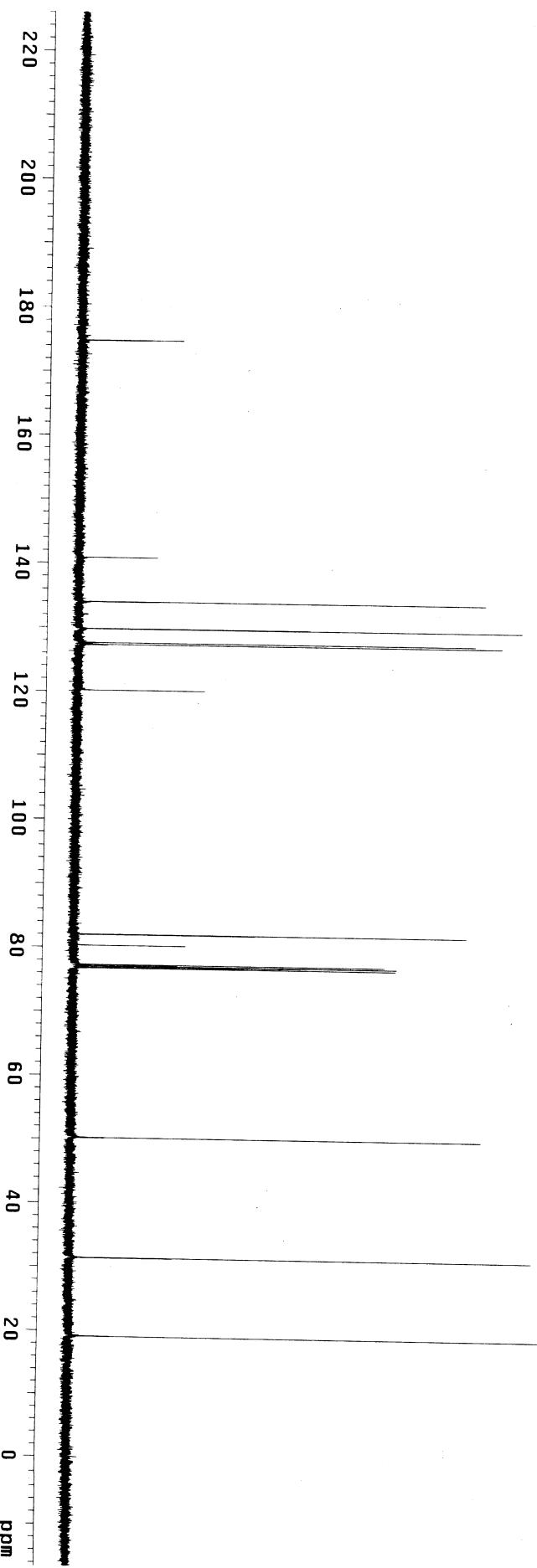


STANDARD 1H OBSERVE - profile

exp2 Proton

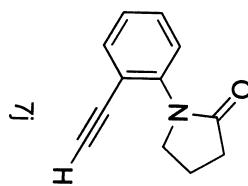
SAMPLE	9	2007	temp	25.0
date	Jun	cdcl3	gain	20
solvent			not used	
file	/mnt/nmr/00/w~	spin	0.008	
aliquup	/data/Zhang/G~	hst	13.500	
uotaol1/K942.fid		pw90	6.600	
ACQUISITION		atfa		
sw	6410.3	FLAGS		
at	2.049	11	n	
np	26264	in	n	
fb	4000	dp	y	
bs	32	hs	nn	
d1	1.000	1b	PROCESSING	0.20
nt	8	fn	DISPLAY	65536
ct				
TRANSMITTER				
tn	H1	sp		
sfrq	399.868	wp	-148.9	
tof	399.6	r71	3395.9	
tpwr	59	r7p	794.8	
pw	6.750	rp	0	
DECOUPLER		1p	-97.6	
d1	C13	PLOT	-26.3	
dof	0	250		
dm	nnn	sc		
dmm	sc	vs		
dppr	34	th	339	
	29412	ai	cdc	2
			ph	





exp1 Carbon

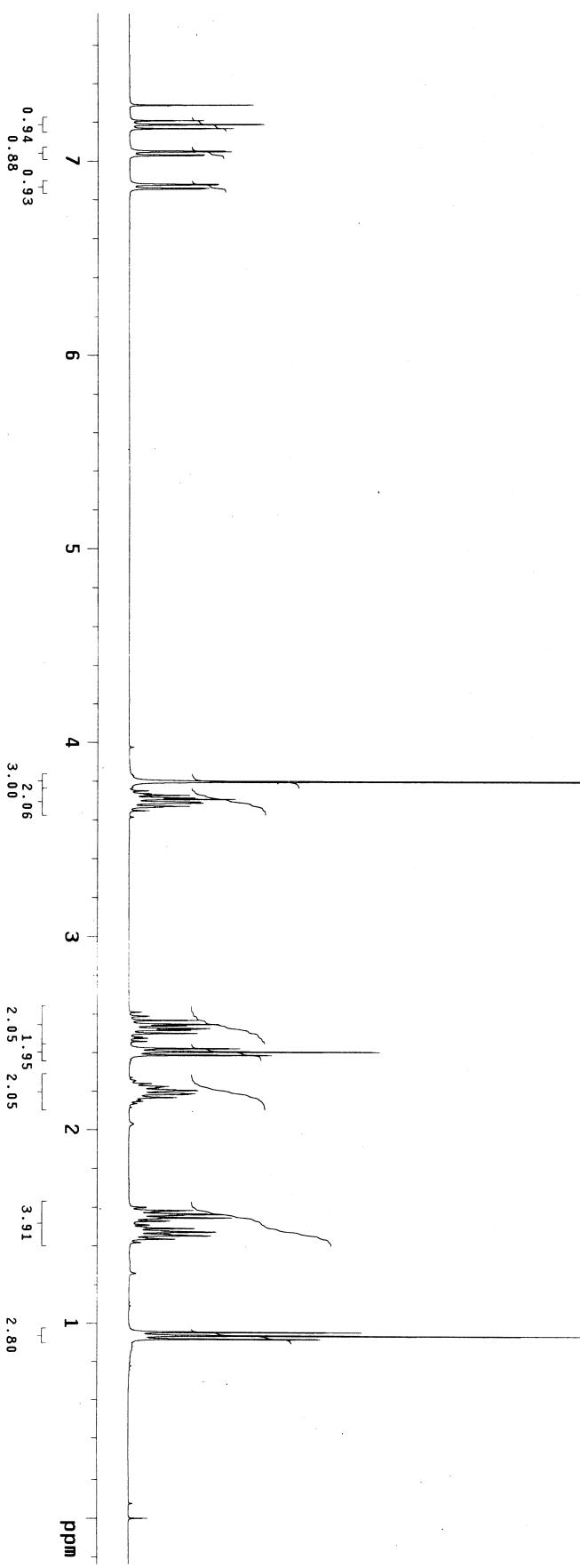
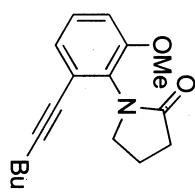
date	Jun 9 2007	temp	25.0
solvent	cdcl3	gain	30
rt		spin	not used
ACQUISITION		hist	0.008
sw	30487.8	pw90	15.300
at	1.300	alpha	10.000
pp	79298	FLAGS	n
fb	17000	d1	n
bs	64	nt	n
d1	1.000	dt	y
nt		hs	nn
ct	1024	PROCESSING	nn
TRANSMITTER	128	lb	0.50
tn	C13	fn	not used
sfrq	125.688	taf	DISPLAY
taf	125.688	sp	-2085.3
tpwr	49	wp	30487.3
pw	7.650	r1	11762.8
DECOUPLER	H1	r1p	9676.9
dn	0	rp	-26.5
dof		1p	-287.0
dm	yyy	PLOT	-287.0
dmm	wc	250	
dpmr	sc	0	
dmf	vs	14226	
	th	11	
	ai	cdcl	
		ph	

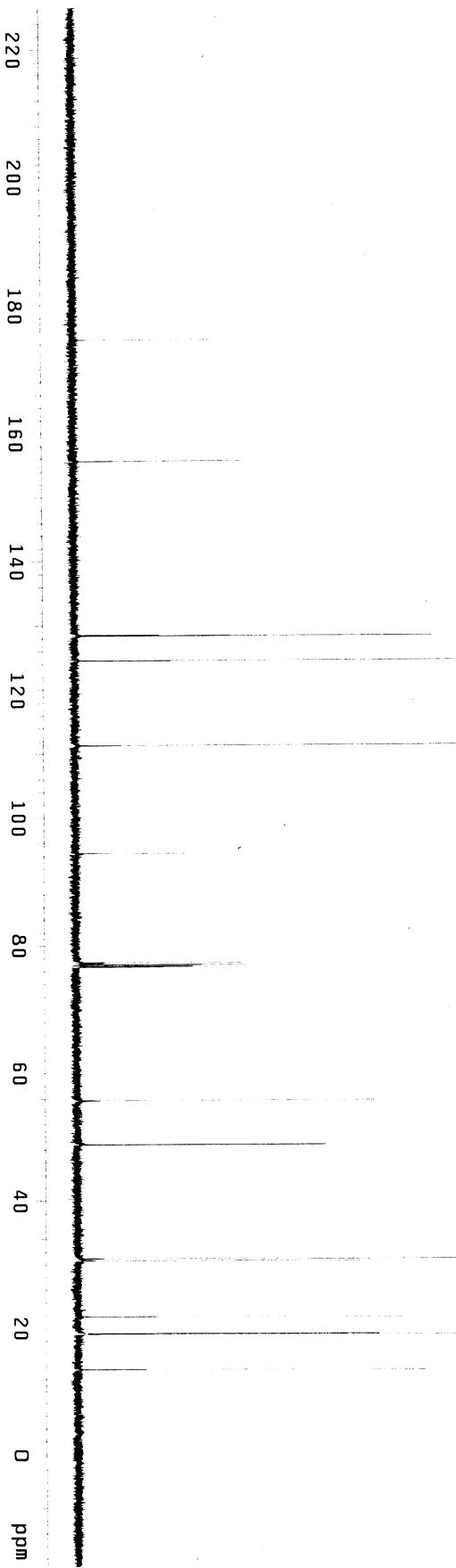


STANDARD 1H OBSERVE - profile

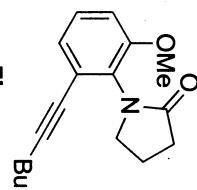
exp1 Proton

SAMPLE	4	2007	temp	24.0
solvent	cdcl3	gain	30	
file	/home/walkup/	spin	not used	
nmr000/walkup/data~		hst	0.008	
/zhang/nuotao1/k2~		pw90	13.500	
90-2b.fid		alfa	6.600	
ACQUISITION	6410.3	11	FLAGS	
sw	2.049	in		n
at	2.624	dp		n
np	4000			y
fb				nn
bs	8		PROCESSING	0.20
d1	1.000	1b		65536
nt	8	fn		
ct			DISPLAY	-96.4
TRANSMITTER		sp		
tn	H1	wp		3201.4
sfrq	399.868	rf1		788.8
tof	399.6	rfp		157.0
tpwr	559	rp		-21.6
pw	6.750	lp		
DECOPPLER			PLOT	
dn	C13	wc		250
dof	0	sc		0
dm	mn	vs		57
dmm	c	th		2
dpmr	34	ai	cdc	ph
	29412			



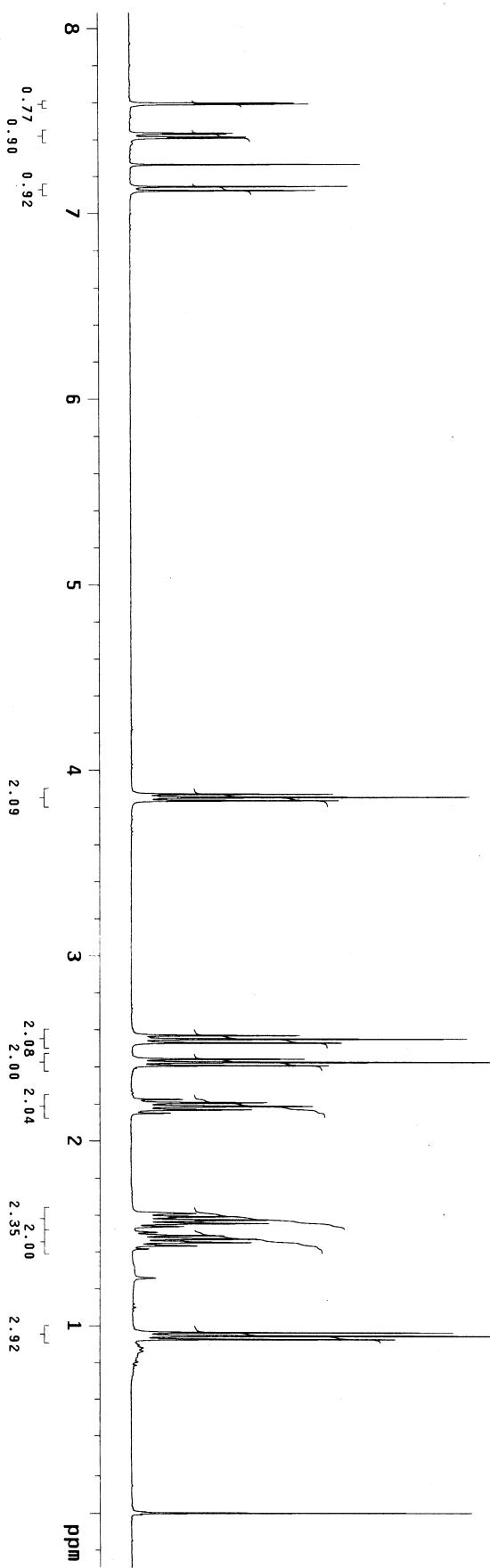
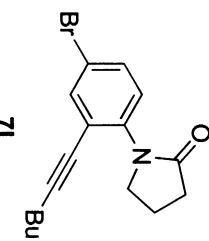


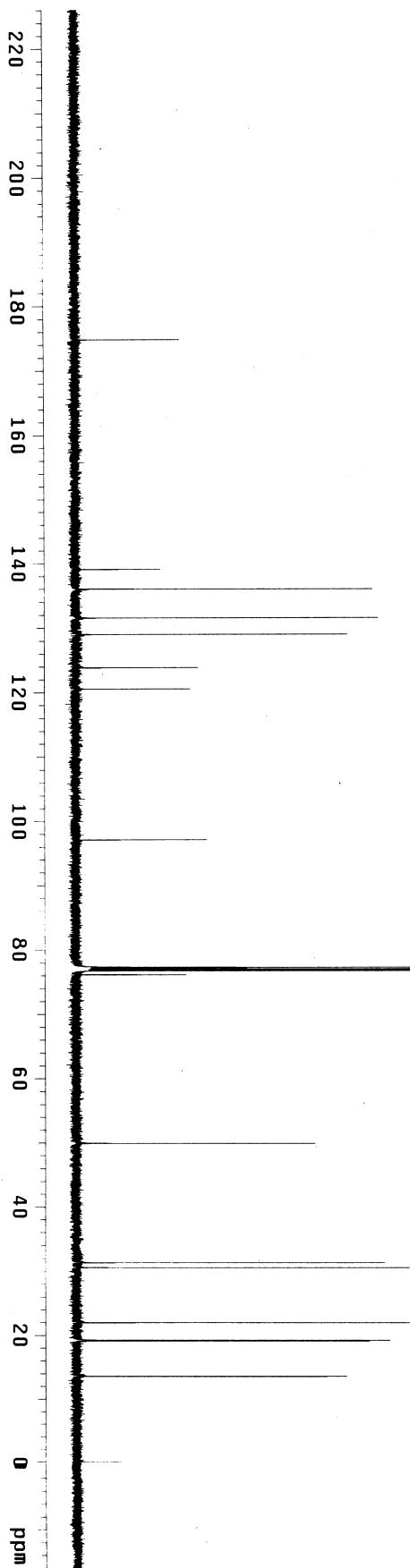
exptl		Carbon		SPECIAL		SAMPLE		date		Aug 4 2007		temp		24.0			
				solvent		cdcl3		gain								30.0	
				file		/home/walkup/		spin								not used	
				vnmrss		/data/autorange		hst								0.008	
				07.08.04_001		08.08.20		pw90								11.000	
				070804_001		/data/cd~		alfa								10.000	
				c13		01_fid		FLAGS									
				ACQUISITION		24:09.8		in		n							
				art		1.300		dp		y							
				np		63750		hs		nn							
				fb		17000		fb		PROCESSING		0.50					
				bs		64		1b		not used							
				d1		1.000		fn		DISPLAY							
				nt		3200		nt		-1733.3							
				ct		64		sp		-1733.3							
				TRANSMITTER		C13		wp		24509.1							
				tn		100.557		rf1		9476.0							
				sf rq		1042.8		rfp		7742.1							
				to f		1042.8		rp		-152.2							
				tp wr		53		tp		-155.4							
				pw		5.950		wc		210							
				DECOUPLER		H1		sc		17540							
				dn		0		vs		68							
				dof		yy		th		cdc							
				dm		w		ph		ph							
				dpm		41		ai									
				dmf		9500											



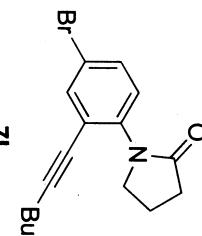
expt Proton

SAMPLE	Jul 30 2007	temp	25.0
solvent	cdcl ₃	gain	60
file	/mnt/nmr400/wr	spin	not used
alikup/data/Zhang/G-		hist	0.008
uotao01/283-3.fid		pv90	13.500
sw		alfa	6.600
ACQUISITION	6410.3	FLAGS	
at	2.049	i1	
np	26264	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	0.20
nt	8	1b	
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-120.1
sfrq	399.868	wp	3352.8
tof	399.6	rf1	798.0
tpwr	59	rp	0
pw	6.750	ip	172.6
DECOUPLER	C13	PLOT	-33.4
dn	0	WC	250
dof	0	NN	0
dmm	vs	SC	21
dpm	34	TH	3
dpwr	29412	CDCL	PH



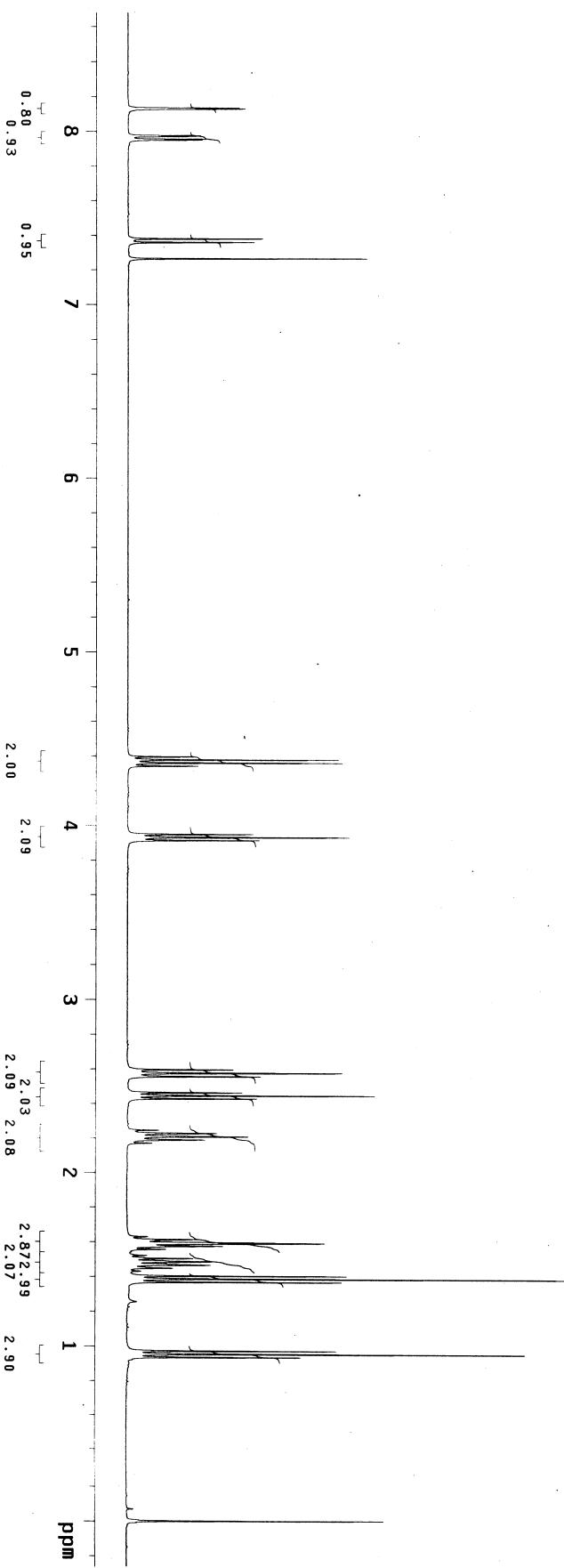
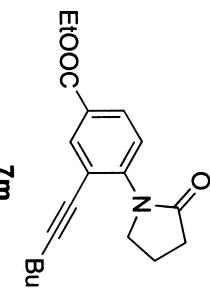


expt Carbon
 SAMPLE Jul 31 2007 temp 25.0
 solvent cdc13 special 25.0
 title exp gain 30
 ACQUISITION hst spin 0.008
 sw 30487.8 pw90 11.300
 at 1.300 alfa 10.000
 np 79298
 tb 17000
 bs 64
 d1 1.000 in n
 d1 2000 hs y
 nt 1600 nn
 ct 1600
 TRANSMITTER C13 1b PROCESSING 0.50
 tn 125.688 fn not used
 sfrq 1255.3 sp DISPLAY -2080.7
 tof 1255.3
 tpow 7.49 wp 30487.3
 pw 7.650 rf1 11758.1
 rrp 9876.9
 DECOUPLER H1 rp -111.1
 dn 0 ip -279.4
 dof 1p
 dim yyy PLOT 250
 dim w
 dppr 39 sc 58435
 dmtr 12200 vs 6
 th ai cdc ph
 ai



STANDARD	1H	OBSERVE	-	profile
STANDARD	1H	OBSERVE	-	profile
STANDARD	1H	OBSERVE	-	profile

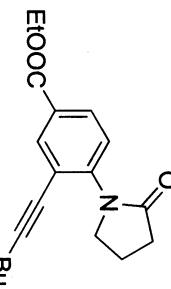
SAMPLE		date	Aug 3 2007	temp	25.0	SPECIAL
solvent		cdcl3	gain	30	30	
file		/home/walkup/	spin	not used	not used	
nmr400/walkup/data		hst	0.088	0.088	0.088	
/zhang/GuoRao1/K2~		pw90	13.500	13.500	13.500	
/zhang/GuoRao1/K2~		alra	6.6000	6.6000	6.6000	
ACQUISITION		FLAGS				
sw	64.0	3	11	r		
at	2.049		in	r		
np	20264		dp	y		
fb	4000		hs			
bs	8					
di	1.000	1b		0.2		
nt	8	fn		653365		
ct	8					
TRANSMITTER		DISPLAY				
tn	h1	sp	-105.4			
sfreq	399.868	wp	3576.2			
tof	339.6	r ^f 1	797.8			
tpwr	5.59	r ^f 0	151.0			
pw	6.750	tp	-30.4			
DECOUPLER		PLOT				
dn	C13	wc	250			
dof	0	sc	0			
dm	nnn	vs	699			
dmm	c	th	3			
dprw	34	cdc				
dmf	29412	ph				





exp1 Carbon

SAMPLE	3	2007	temp	25.0	SPECIAL
date	Aug		gain	30	
Solvent	cdcl3		not used		
file	/home/walkup/	~	spin		
vmrsys	/data/auto/	~	hst		
2007.08.03	0.3s	2007.08.03	prg0	0.008	
803_011	/data/cdc13/	~	alfa	11.900	
01.fid				10.000	
ACQUISITION	i1		FLAGS		
sw	2409.8	in	n		
at	1.300	dp	y		
np	63750	hs	nn		
fb	1700				
bs	64	1b	0.50		
d1	1.000	fn	not used		
nt	12000				
ct	64	sp	-1719.1		
TRANSMITTER		wp	2459.1		
tn		r _f 1	9461.9		
sf1-q	100.557	r _{fp}	7747.1		
tof	1042.8	r _p	-171.9		
tpur	53	1p	-202.9		
pw	5, 950				
DECOUPLER		wc	250		
dn		h1	sc		
dof		0	vs		
dm		vvv	th		
dmm		41	ai		
dprw			cdc		
	9500		ph		



Automation directory: /home/walkup/nmrsys/data/auto_2007.06.01_34
File : s_2007001_001.fid
Sample id : s_2007001_001

Pulse Sequence: s2pul

Solvent: cdc13

Temp. 25.0 C / 298.1 K

Operator: walkup

File: cdc13_02

UNMR-400 "NMR400"

Relax. delay 1.000 sec

pulse 45.0 degrees

Acq. time 2.049 sec

Width 64.0.3 Hz

8 repetitions

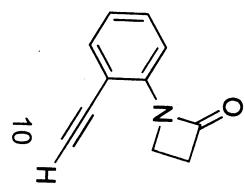
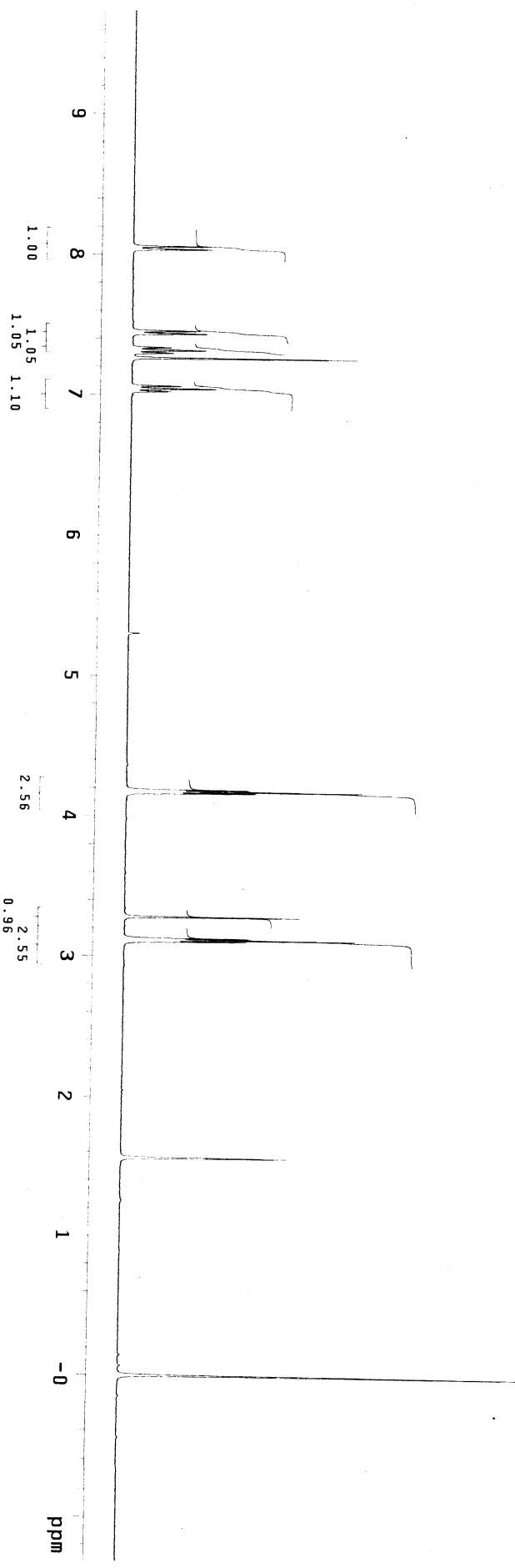
OBSERVE H1 399.8656717 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.10_01
File : Aug_02/data/cdc13_01.fid
Sample Id : Aug_02

Pulse Sequence: s2pul

Solvent: cdc13
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdc13_01
VNMRs-500 "nmr500"

Relax. delay 1.000 sec
pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz

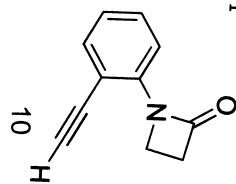
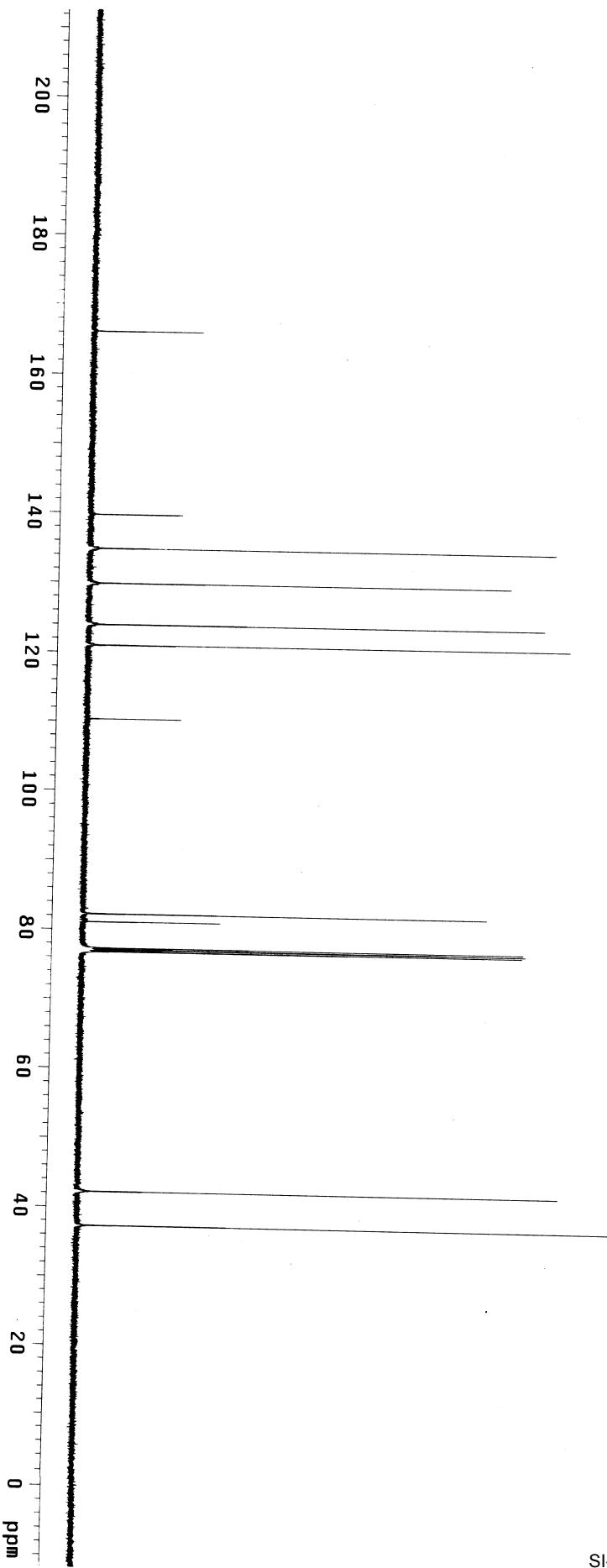
1728 repetitions
OBSERVE C13, 125.676077 MHz

DECOUPLE H1, 499.8016822 MHz
power 39 dB

continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz

FT size 131072
Total time 1 hr, 18 min, 49 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_15
File : Aug.01/data/cdc13_01.fid
Sample Id : Aug.01

Pulse Sequence: szpul

Solvent: cdc13

Temp: 25.0 C / 298.1 K

Operator: walkup

File: cdc13_01

VNMRs-500 "nmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 8012.8 Hz

8 repetitions

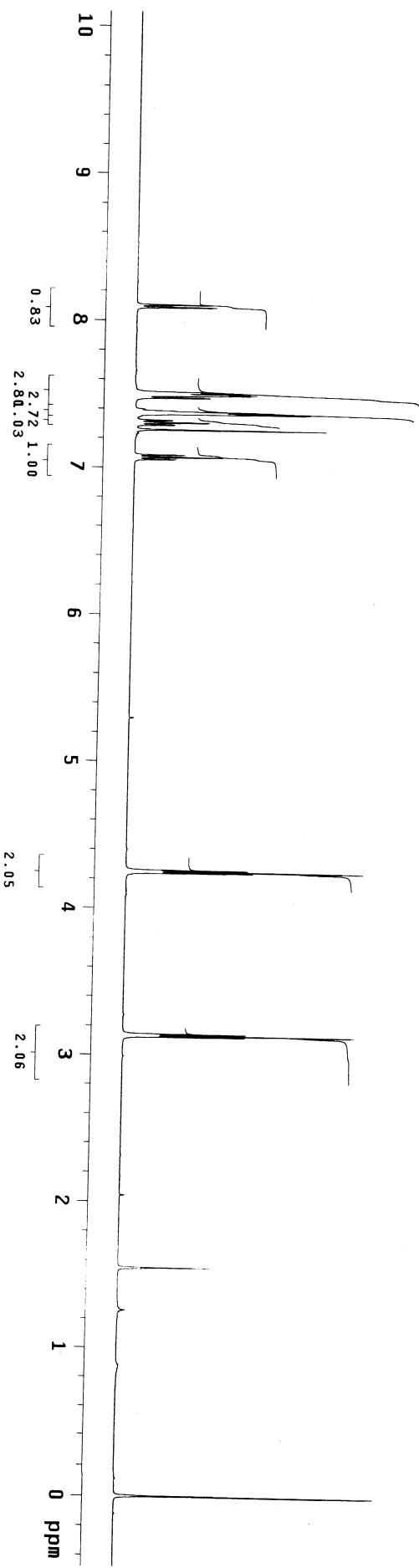
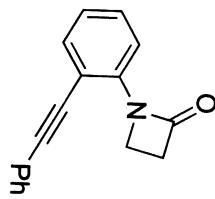
OBSERVE H1, 499.7991839 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 6536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_15

File : exp

Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdc13

Temp. 25.0 C / 298.1 K

Operator: walkup

VNMR-500 "nmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3048.8 Hz

576 repetitions

OBSERVE C13, 135.6746039 MHz

DECOUPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

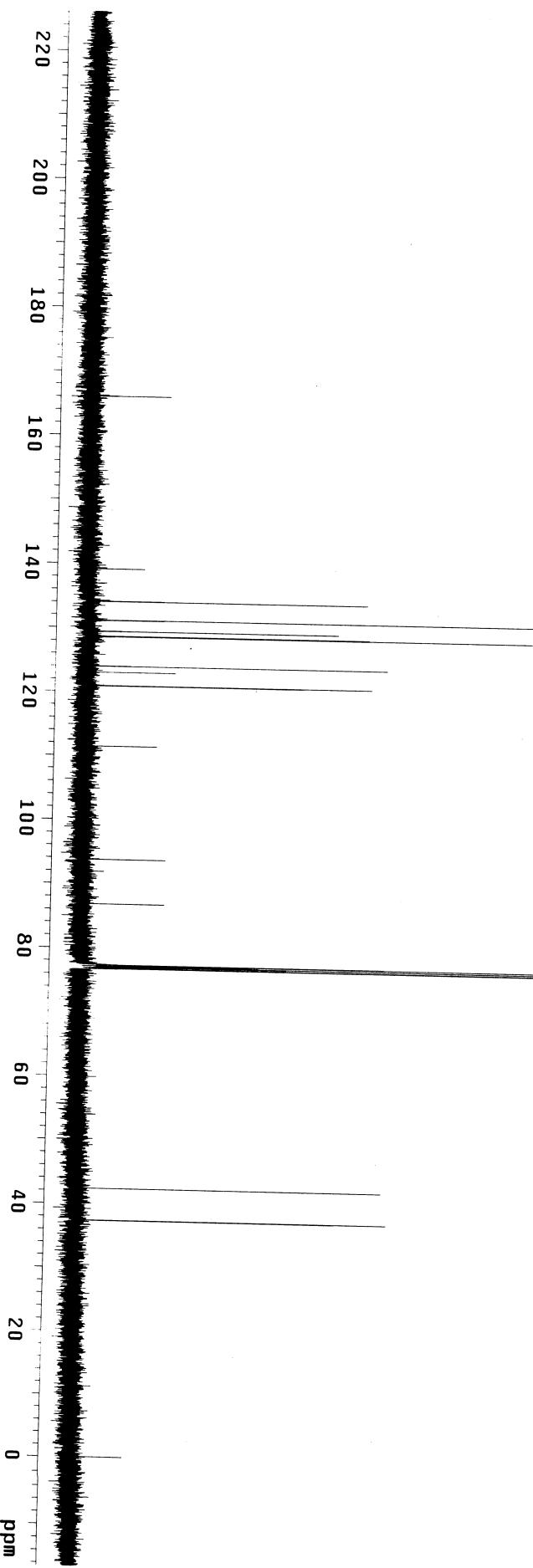
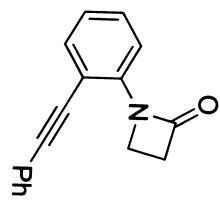
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

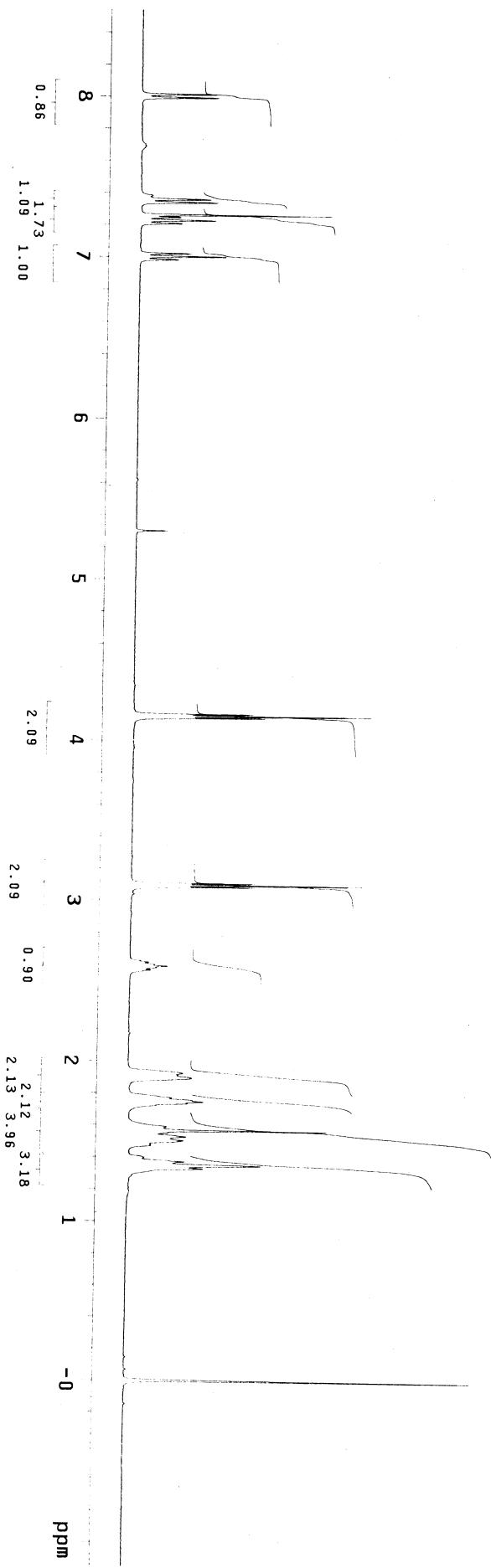
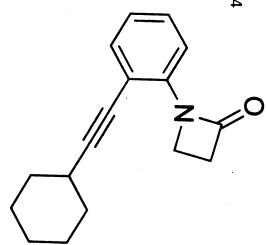
Total time 39 min, 24 sec



Automation directory: /home/walkup/vnmrjsys/data/auto_2007.06.01_34
File : s_20070601_003\data\cdcl3_03.fid
Sample id : s_20070601_003

Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_03
VNMR-S-400 "NMR400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8656715 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



File: hgx-3-145C

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: walkup

File: hgx-3-145C

INOVA-500 "sittingbull"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3047.8 Hz

2240 repetitions

OBSERVE C13, 125.6746013 MHz

DECOPPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

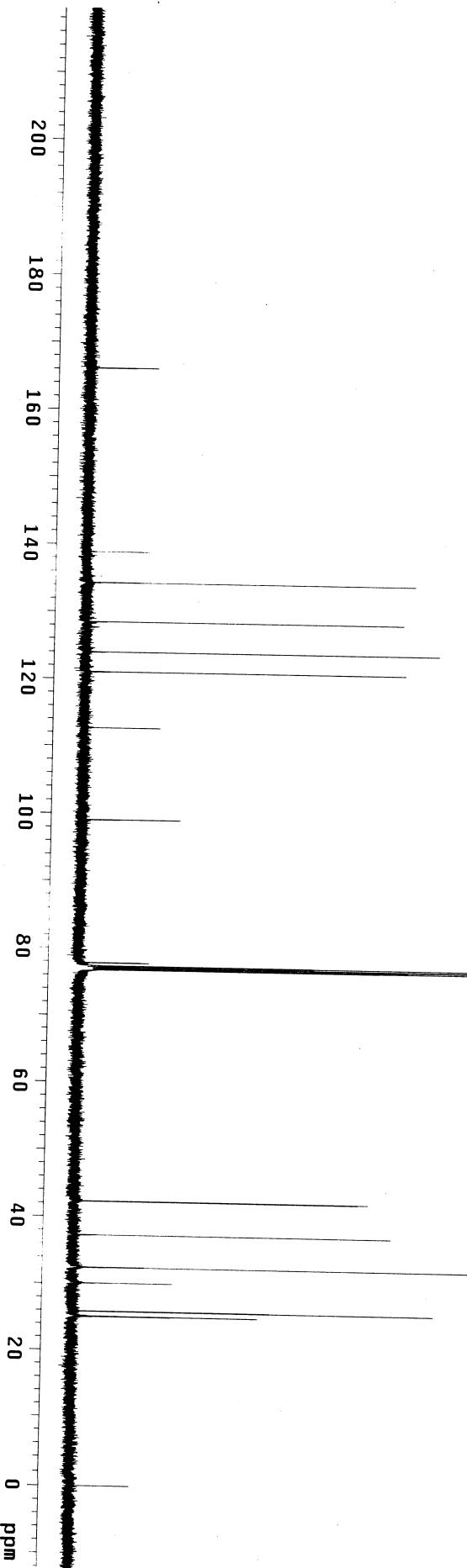
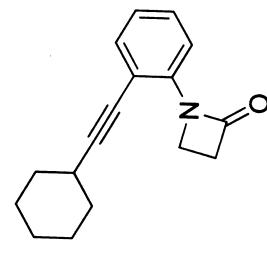
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 2 hr, 33 min, 57 sec



File: hxg-3-159H

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 22.0 C / 295.1 K

Operator: walkup

File: hxg-3-159H

INOVA-500 "redjacket"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 8012.8 Hz

8 repetitions

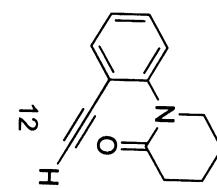
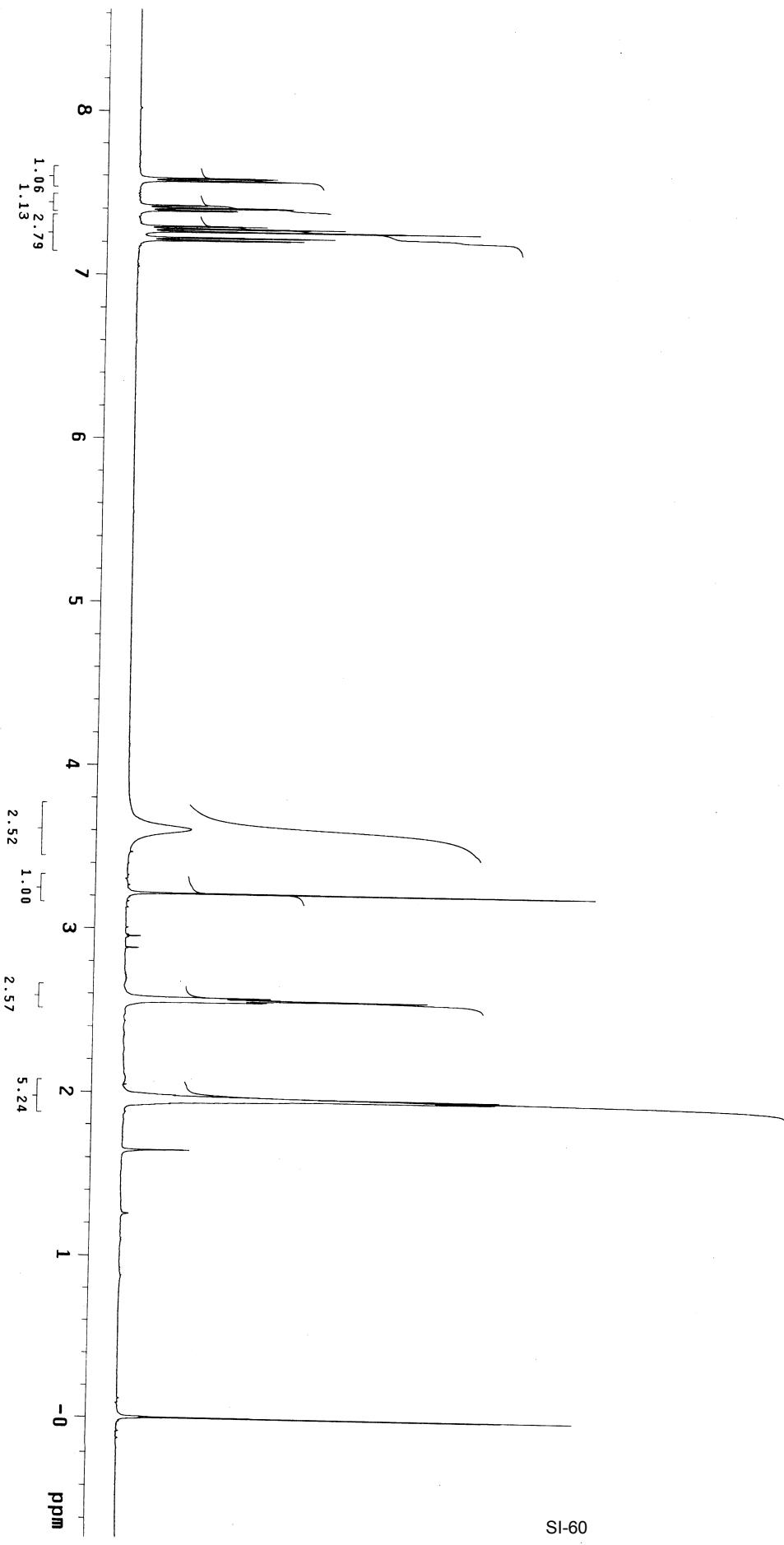
OBSERVE H1, 499.7991815 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



File: hxg-3-159C

Pulse Sequence: s2pul

Solvent: cdcl₃

Temp. 22.0 C / 295.1 K

Operator: walkup

File: hxg-3-159C

"redjacket"

Relax. delay 1.000 sec

pulse 45.0 degrees

width 30437.8 Hz

Acq. time 1.300 sec

512 repetitions

OBSERVE C13, 125.674667 MHz

DECUPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

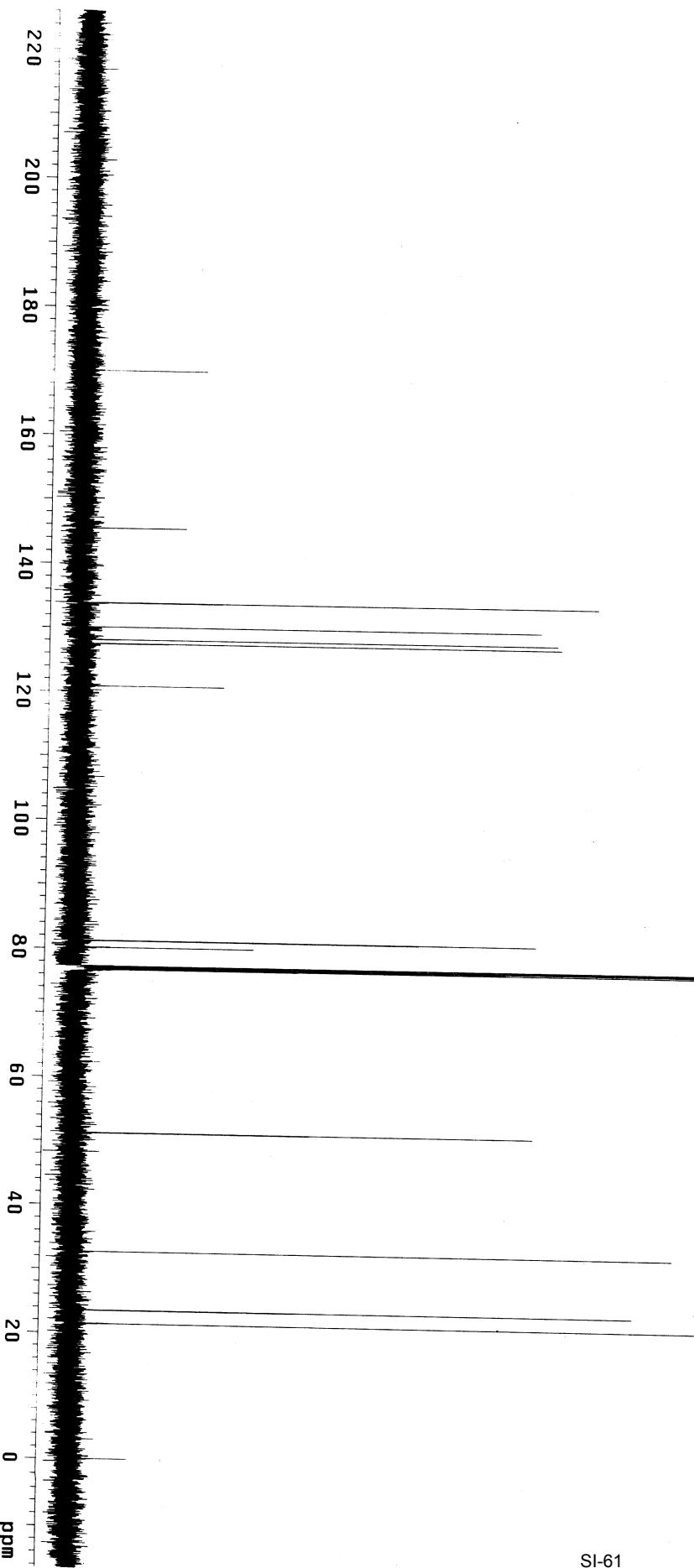
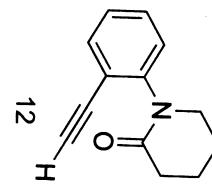
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

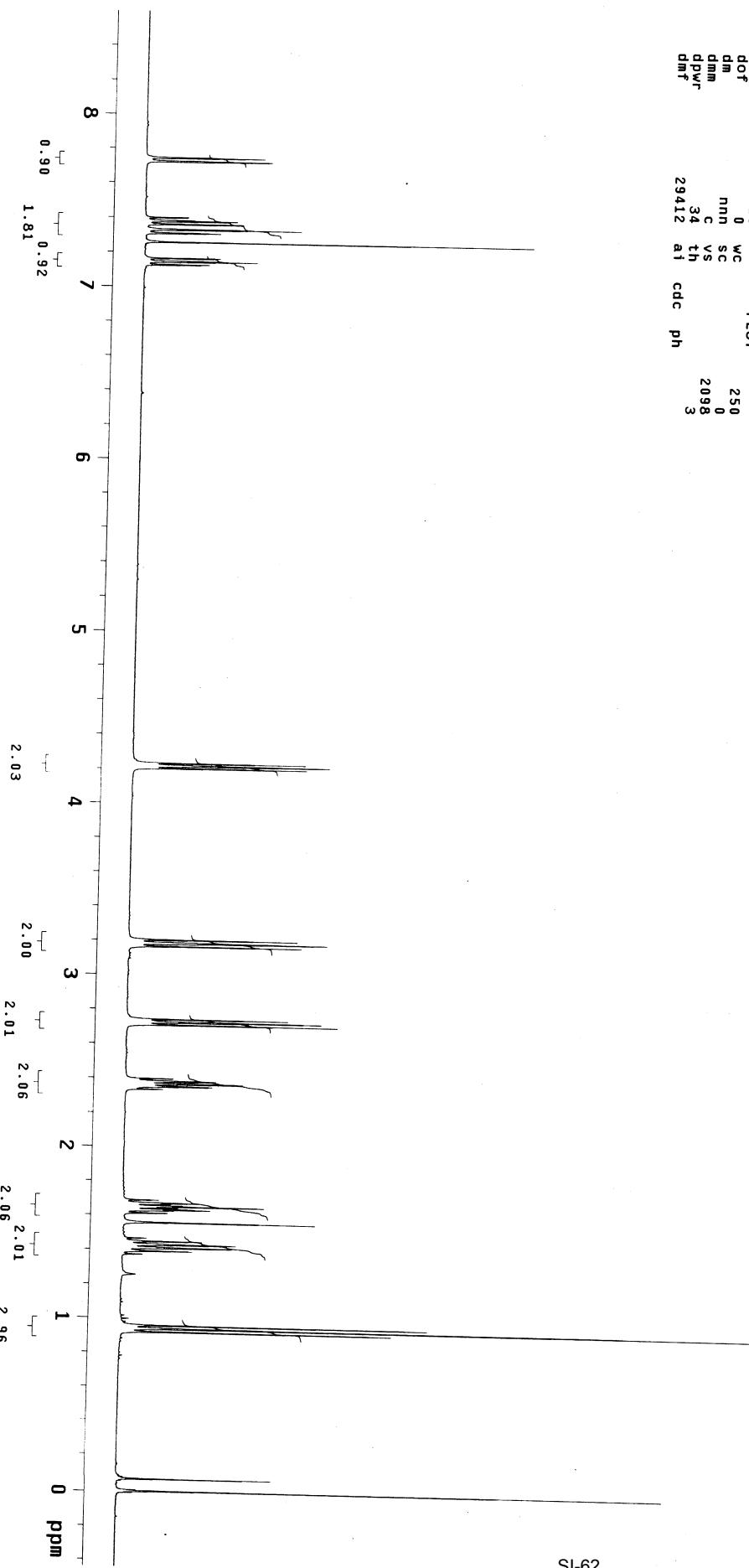
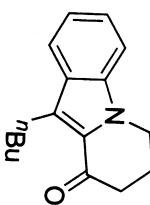
Total time 19 min, 42 sec



exp2 Proton

Proton

SAMPLE		date	Jun 8 2007	temp	SPECIAL
solvent		ccl3	ccl3	gain	25.
file	/mnt/umr040/gw~			22	
alkup	/data/Zhang/G~			0.00	
uotao1	/K102-1.fid			0.00	
ACQUISITION				13.50	
Sw		6410.3		pw90	6.600
at		2.049	11		
np		26264	in		
fb		4000	dp		
bs		32	hs		
d1		1.000			
nt		8	1b		
ct		8	fn		
TRANSMITTER		DISPLAY		FLAGS	
tn		H1	sp	-176.3	
sfq		399.868	wp	364.2	
tof		399.6	rft1	799.9	
tpwr		59	rfp	0	
pw	6.750	rP		-96.4	
DECOPPLER		1P		-29.6	
dn	C13		PLT		
dof	0	wc			
dm	nnn	sc		250	
dmm	c	vs		0	
dPWR	34	th		2098	
dmf	29412	ai	cdc	ph	3

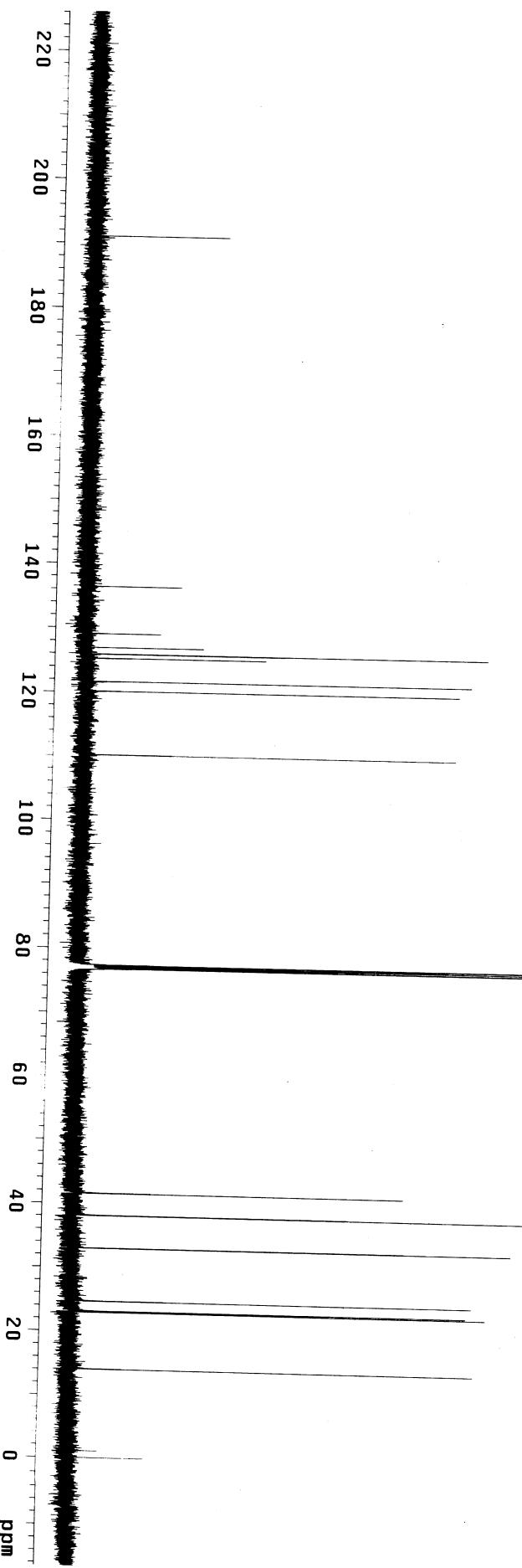
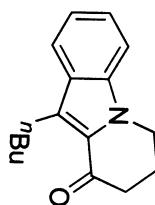


T1 EXPERIMENT
T1 EXPERIMENT
T1 EXPERIMENT

exp1 Carbon

	SAMPLE	SPECIAL
date	Jun 8 2007	temp 25.0
solvent	cdcl3	gain 30
file		not used
ACQUISITION	exp	spin
sw	30487.8	hst
at		pwg90
pp	1.300	0.008
fb	7938	15.300
bs	17000	10.000
d1	64	aifa
nt	1.000	FLAGS
ct	2000	n
TRANSMITTER	hs	n
tn	1408	PROCESSING
sfrq	125.688	1b 0.50
tot	1255.3	fn not used
tpwr	49	DISPLAY
pw	7.650	-2077.0
DECOUPLER	rpi	30487.3
dn	H1	1174.4
dof	0	rfp 9676.9
dm	1p	-11.3
dmm	yyy	1p
dppwr	w	PLOT -336.9
dmpf	39	250
dmf	sc	0
th	vs	92547
ai	cdc	68
	ph	

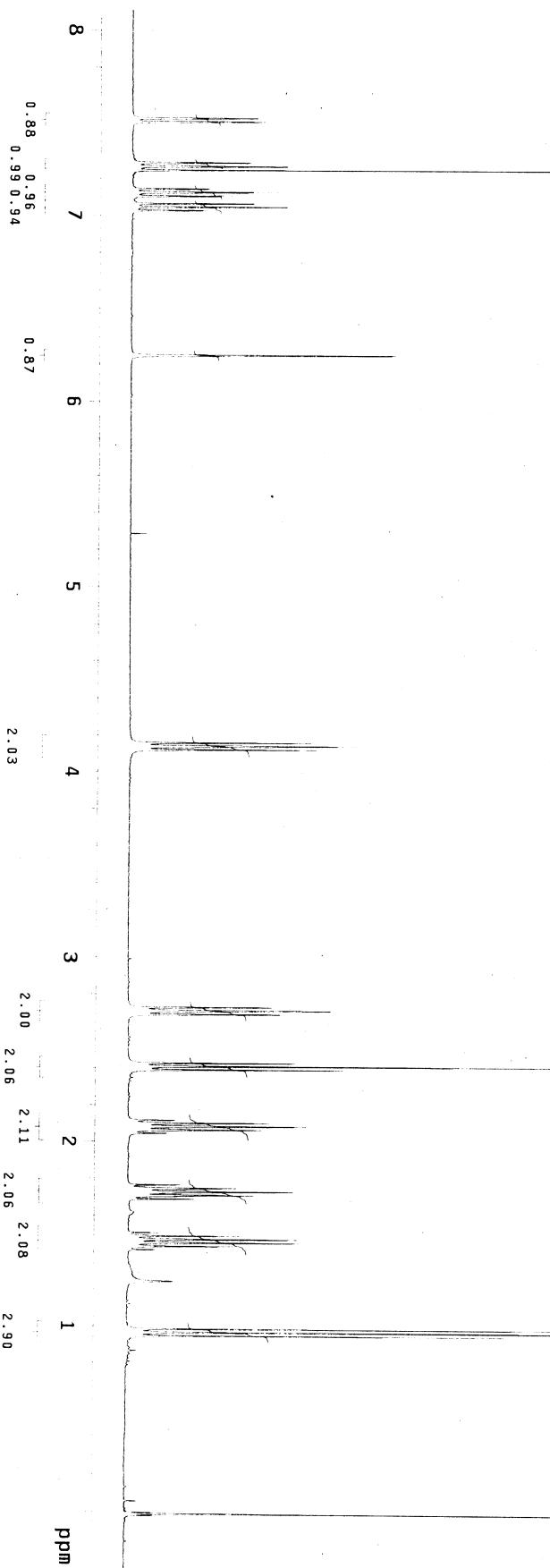
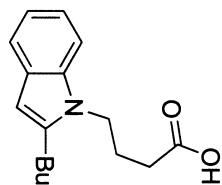
4



STANDARD 1H OBSERVE - profile

expt Proton

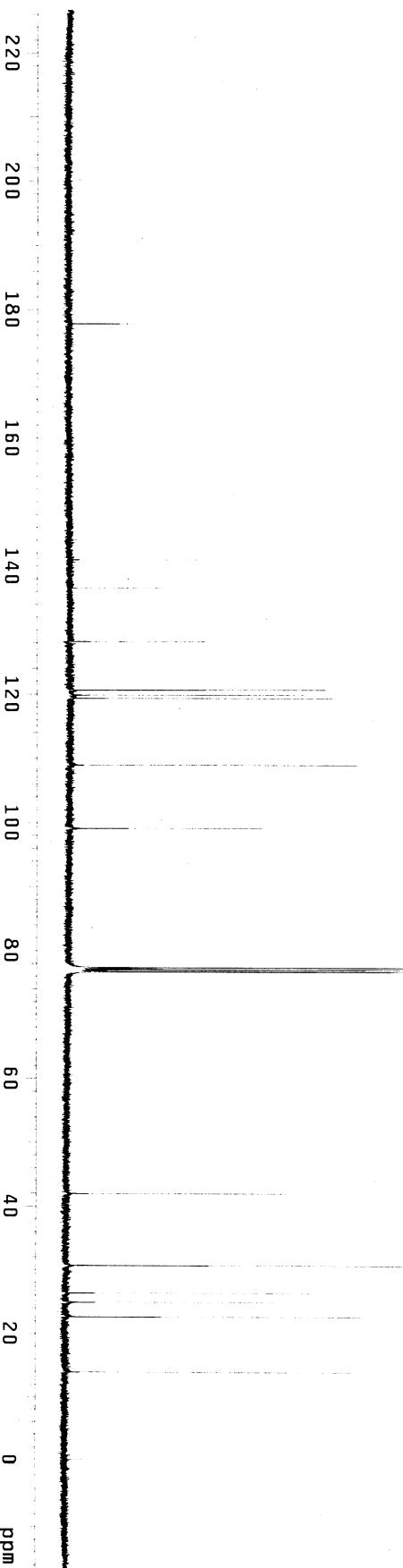
SAMPLE	2	2007	temp	25.0
solvent	Aug	cdcl3	gain	30
file			spin	not used
ACQUISITION			hst	0.008
sw	6.41.0.3	pw0	13.500	
at	2.049	alfa	6.600	
np	26264	FLAGS		
fb	4000	i1	n	
bs	8	in	n	
d1	1.000	dp	y	
nt	8	hs	nn	
ct				
TRANSMITTER	8	PROCESSING	0.20	
tn		lb		
strq	399.868	fn	65536	
tof	399.6	sp	DISPLAY	-132.2
tpwr	59	wp		3375.1
pw	6.750	rf1		805.9
DECOUPLER		rfp		0
dn	C13	rp	76.7	
dof	0	lp	-24.8	
dm	nmn	plot		
dmm	c	wc		250
dpwf	34	sc		0
dmf	29412	vs		1296
		th		12
		ai	cdcl	12
			ph	



STANDARD 1H OBSERVE - profile

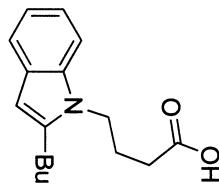
expt Noesyid

ACQUISITION	8012.8	DECOUPLER	C13	ACQUISITION ARRAYS
sw	1.998	dm	nnn	array (selpw,selpw~
at	32018	dm	nnn	r,selshape)
np	2600	SAMPLE	nnn	arraydm
fb	4	date	2 2007	
bs	-2	solvent	cccl3	selpw
ss	1.000	file	SPECIAL	1 44937.1
d1	10240	temp	25.0	2 44957.1
nt	64	gain	30	
ct		spin	not used	selpw
TRANSMITTER		not used	1	7
tn	H1	pw90	14.700	
sfrq	499.802	FLAGS	y	
tof	499.7	sspu1	y	
tpwr	54	in	n	1 NOESYID_4_16p
pw	14.700	dp	y	2 NOESYID_4_16p
mix	0.500	hs		
sweeppwr	44	PROCESSING	0.50	
sweeppw	1500.000	lb	not used	
sweeppw		fn		
swexp		not used		
swexp				
sech180				
SEL PULSE				
selfrfq	499.7	DISPLAY	-1007.2	
selfrfq		sp		
selfshape		8012.3		
selfshape		55974		
selfpw		arrayed		
selfpw		sc		
selfpw		0.00500		
selfpw		th		
GRADIENT				
gz1v11	1000	wc	250	
gt1	0.00500	hzmm	32.05	
gz1v12	4000	gt1	500.00	
gt1		is		
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gt1				
gz1v150				
gt1				
gz1v151				
gt1				
gz1v152				
gt1				
gz1v153				
gt1				
gz1v154				
gt1				
gz1v155				
gt1				
gz1v156				
gt1				
gz1v157				
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gz1v160				
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gz1v161				
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gz1v162				
gt1				
gz1v163				
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gz1v164				
gt1				
gz1v165				
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gz1v166				
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gz1v167				
gt1				
gz1v168				
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gz1v169				
gt1				
gz1v170				
gt1				
gz1v171				
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gz1v172				
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gz1v173				
gt1				
gz1v174				
gt1				
gz1v175				
gt1				
gz1v176				
gt1				
gz1v177				
gt1				
gz1v178				
gt1				
gz1v179				
gt1				
gz1v180				



expt Carbon

SAMPLE	2	2007	temp	25.0
date	Aug		solvent	cdcl3
solvent			gain	30
file	/home/walkup/	~	spin	not used
vnmr/sysdata/auto			0.008	
2007.08.02/s/2007~			11.900	
pwg0			10.000	
802_001/data/cdcl3~			alfa	
ACQUISITION	04	.fid	FLAGS	
sw	24509.8	in		n
at	24509.8	in		n
at	1.300	dp		y
np	63750	hs		nn
fb	17000		PROCESSING	0
bs	64	1b		0.50
d1	1.000	fn		not used
nt	13200		DISPLAY	
ct	13200	sp		-1718.4
TRANSMITTER		wp		24509.1
tn	C13	r _f 1		9461.2
sfrq	100.557	r _{fp}		7742.1
tof	1042.8	r _p		106.7
tpwr	53	lp		-193.1
pw	5.950		PLOT	
DECOPPLER		wc		250
dn	H1	sc		
dof	H1	vs		
dm	0			
dmm	yyy	th		
dpmr	w	ai		
dpmr	41	cdc		
dmpf	9500	ph		



Automation directory: /home/walkup/nmrssys/data/auto_2007.06.24_08
File : s_20070624_001/data/cdc13_02.fid
Sample id : s_20070624_001

Pulse Sequence: s2pul

Solvent: cdc13

Temp: 25.0 C / 298.1 K

Operator: walkup

File: cdc13_02

VNMRs-400 "NMR400"

Relax. delay 1.000 sec

pulse 45.0 degrees

Acq. time 2.049 sec

width 6410.3 Hz

8 repetitions

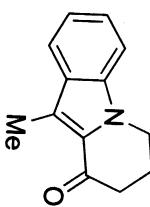
OBSERVE H1, 399.8656762 MHz

DATA PROCESSING

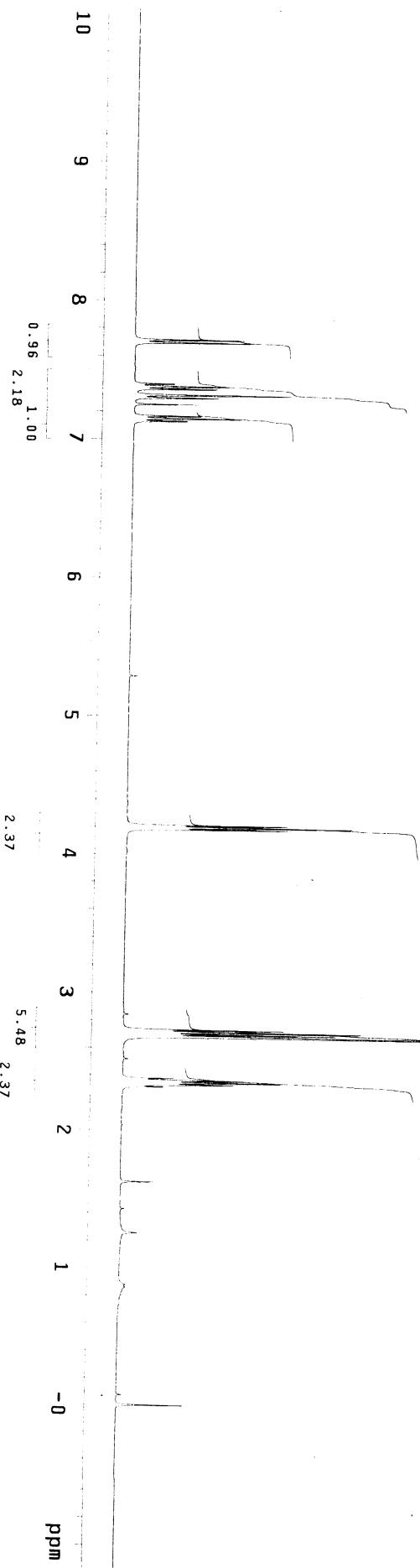
Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



8a



File: hxg-4-27C

Pulse Sequence: s2pu1

Solvent: cdcl₃

Temp: 25.0 C / 298.1 K

Operator: walkup

File: hxg-4-27C

INOVA-500 "redjacket"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3067.8 Hz

512 repetitions

OBSERVE C13, 125.6746072 MHz

DECOUPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

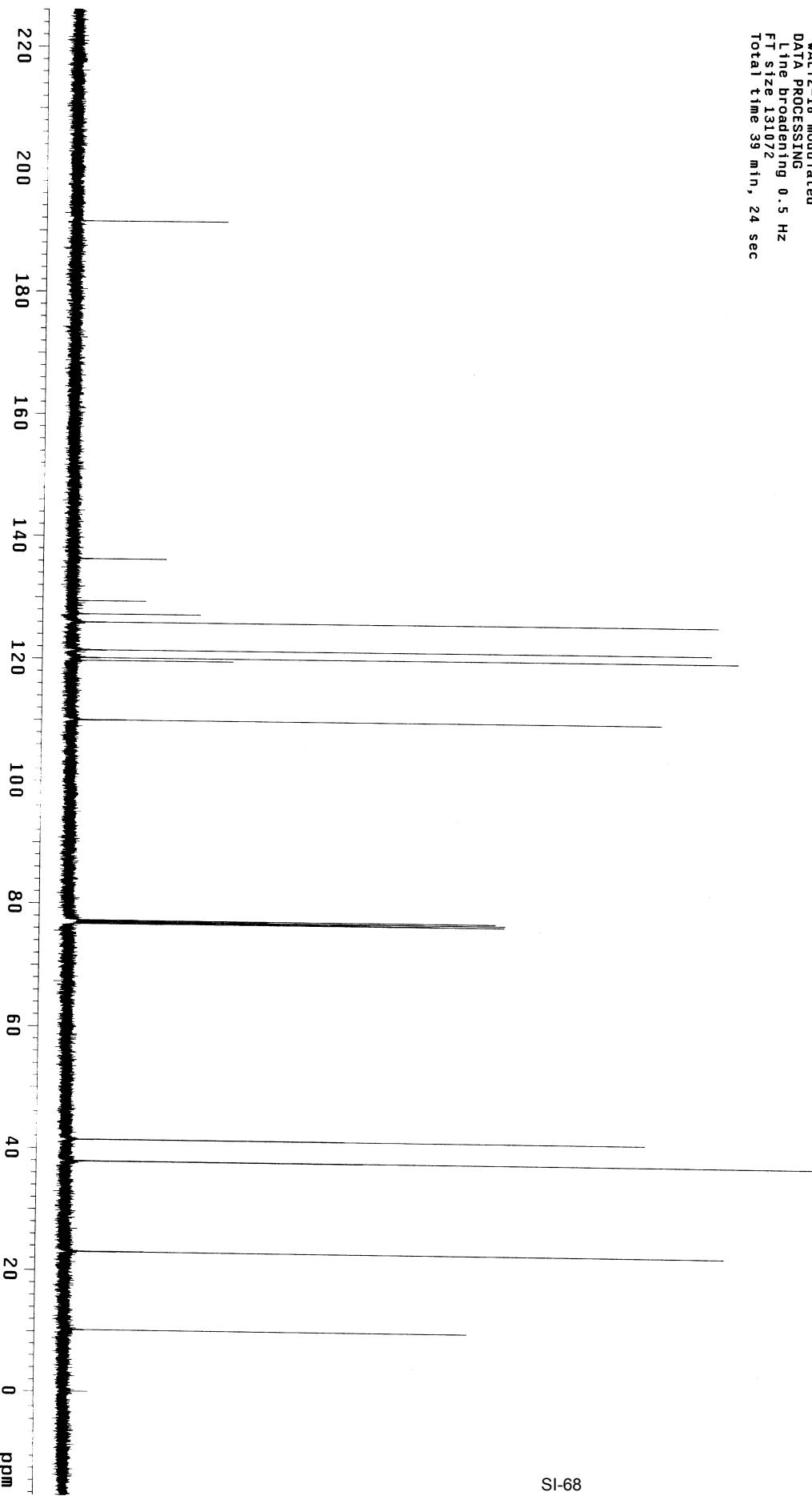
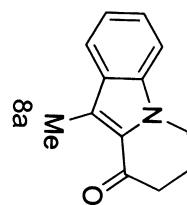
WALTZ-16 modulated

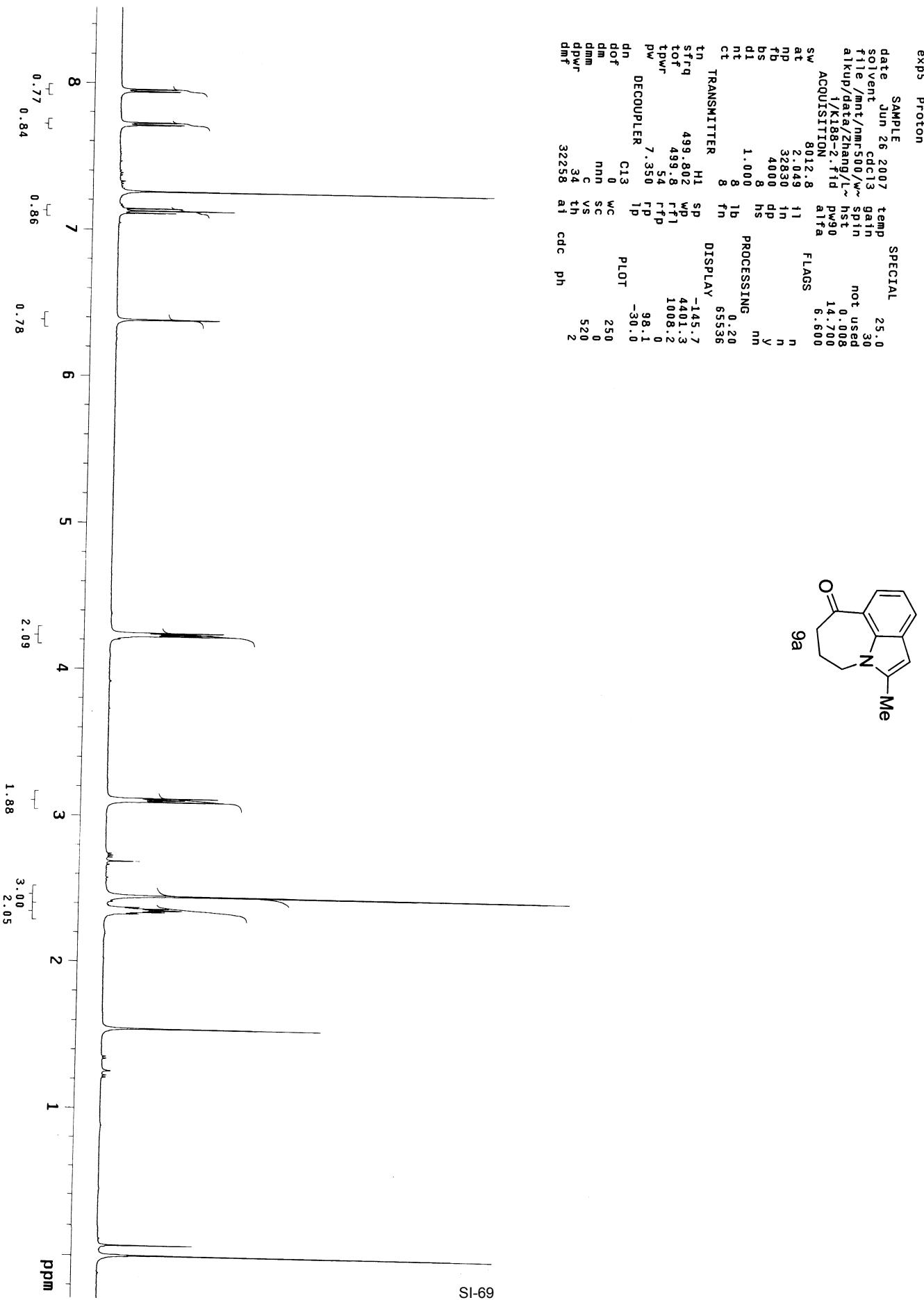
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

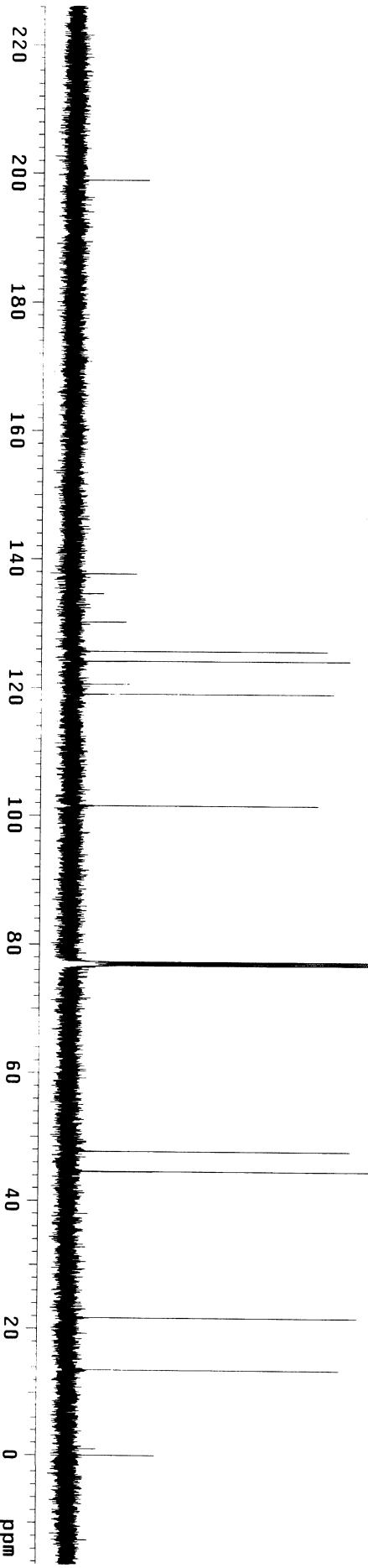
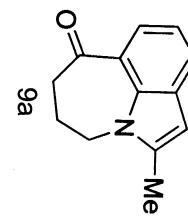
Total time 39 min, 24 sec





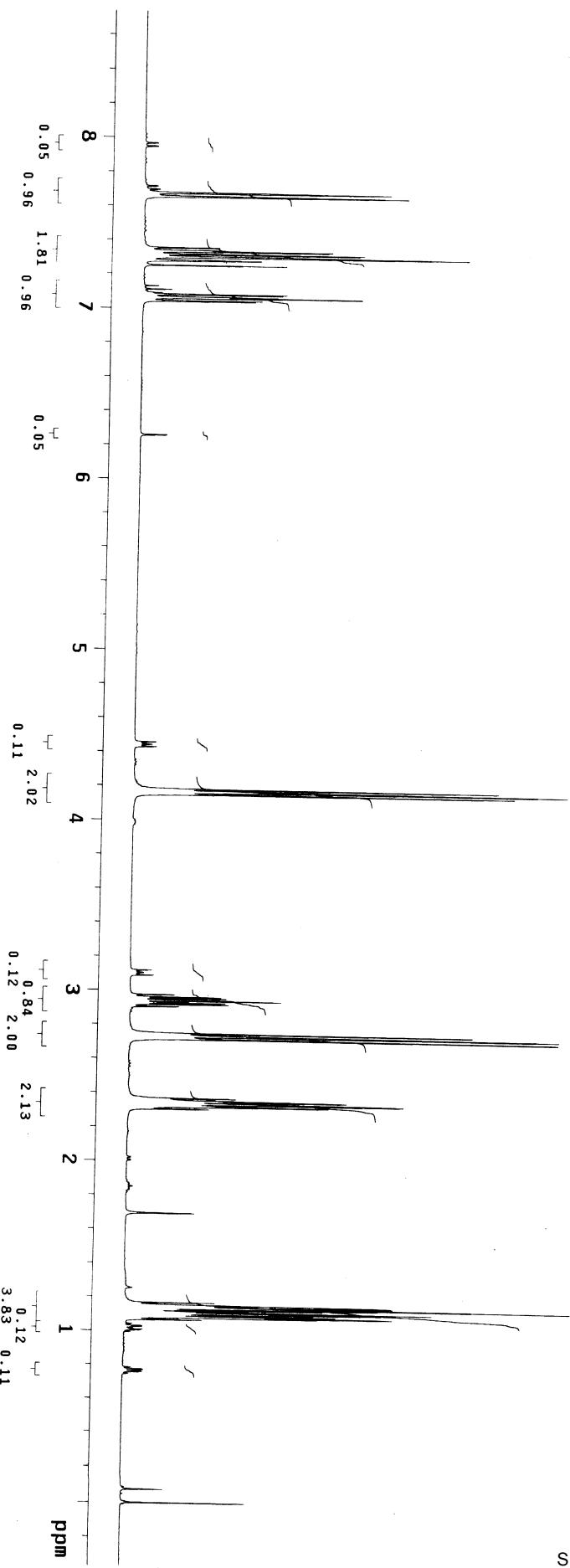
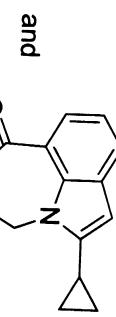
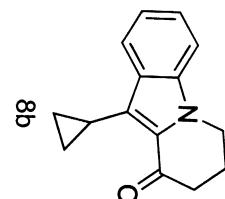
expt Carbon

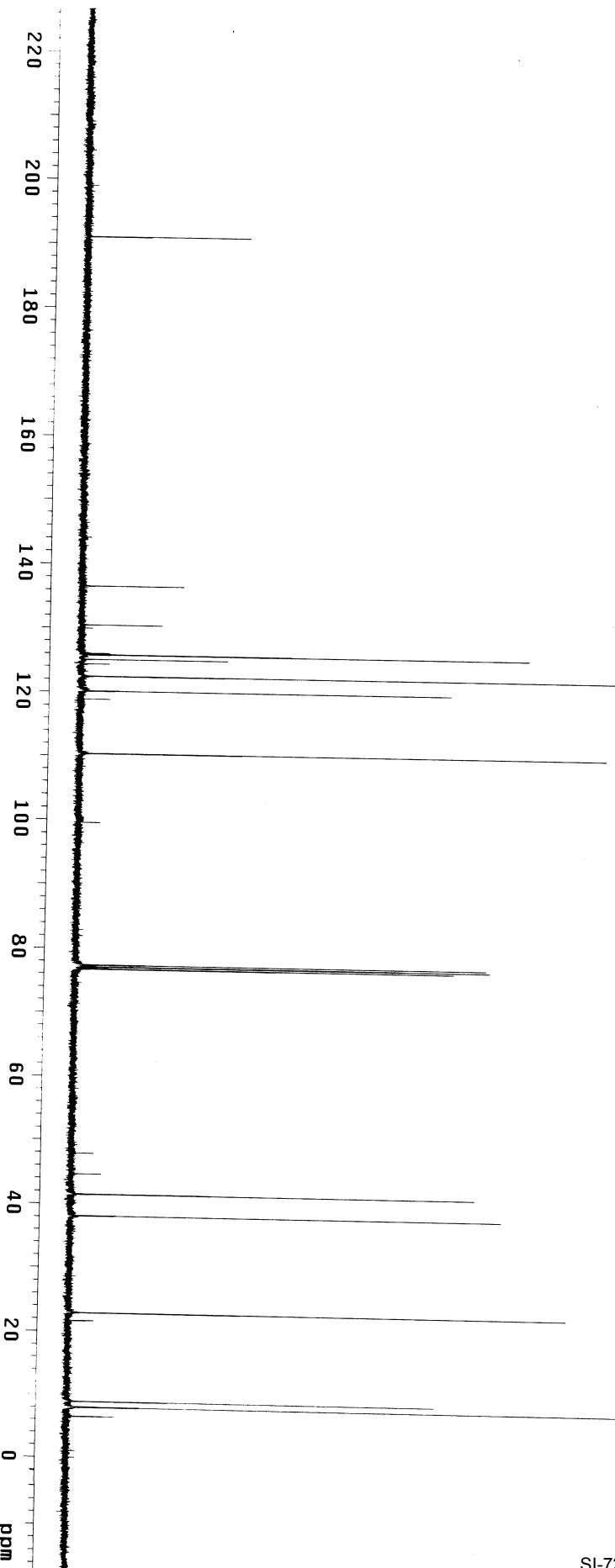
SAMPLE	JUN 26 2007	temp	25.0
SOLVENT	CDCl ₃	gain	30
FILE	exp	spin	not used
ACQUISITION	30487.8	0.008	
SW	1.300	15.300	
AT	79298	alpha	
NP		FLAGS	10.000
FB	17000	n	
BS	8	in	
D1	1.000	dp	y
NT	3000	hs	nn
CT	2440	PROCESSING	0.50
TN	C13	1b	not used
SFRQ	125.688	fn	
TOF	1255.3	SP	DISPLAY
TPWR	7.49	WP	-207.5
PW	7.650	RT1	30487.3
DECOPPLER	H1	RTP	11753.9
DN	0	RP	9676.9
D0F	0	1P	63.7
DM	Y/Y	PLOT	-286.5
DMM	WC	250	
DPPW	39	SC	
DMF	12200	VS	124604
		TH	4
		AI	
		CDC	
		PH	



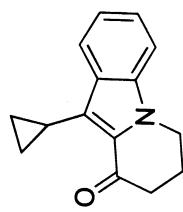
exp2 proton

SAMPLE	temp	SPECIAL	
date	Jun 19 2007	25.0	
solvent	cdcl3	20	
rtle /mnt/nmr400/v~	gain	20	
alikup/data/hang~	spin	0.008	
uotao1/K16~.fid	pro90	13.500	
ACQUISITION	altra	6.600	
sw	2.649	11	
at	2.664	in	
np	4000	dp	
fb	32	hs	
bs	1.000	1b	
di	8	fn	
nt	ct	PROCESSING	
ct	tn	DISPLAY	
TRANSMITTER	H1	SP	
sfrq	399.868	wp	-149.8
totf	399.6	rt1	3643.9
tpwr	59	rtfp	803.2
pw	6.750	rp	0
DECOUPLER	C13	1p	-19.5
dn	0	PLOT	-27.8
dof	wc	250	
dm	nnn	sc	
dmm	c	vs	
dppwr	34	th	1084
dmrf	29412	ai	2
		cdcl3	
		ph	

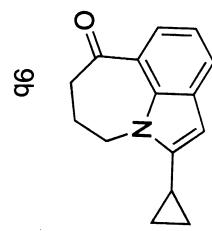




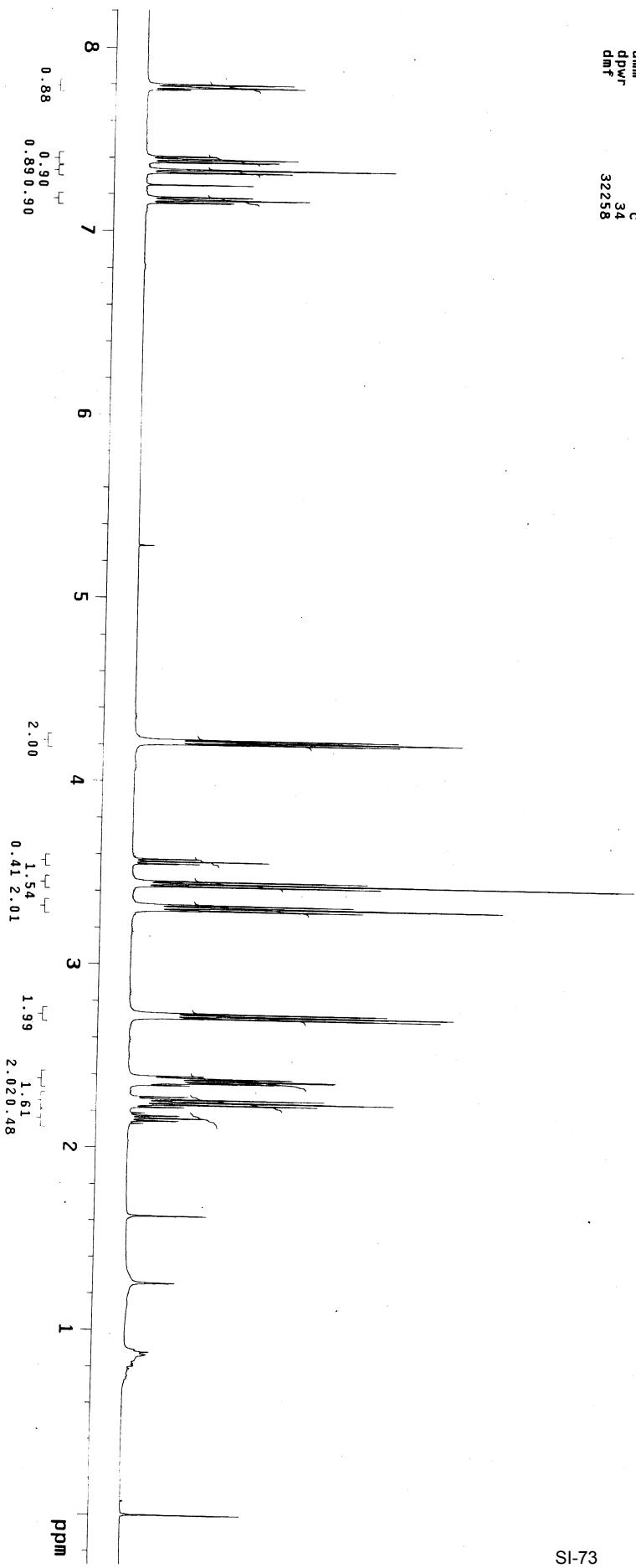
exp5 Carbon
 SAMPLE
 date Jun 19 2007 temp 25.0
 solvent cdc13 gain 30
 file /mnt/nmr400/w~ spin 20
 alkupdata/Zhang/G~ host 0.008
 uotaol1/K160-1carb~ p90 11.900
 on.fid p90 10.000
 a1fa
 ACQUISITION
 sw 24509.8 i1
 at 1.300 in n
 np 63750 dp n
 fb 17000 hs y
 bs 1.32 processing nn
 d1 1.000 1b 0.50
 nt 4000 fn not used
 ct 1408 DISPLAY
 TRANSMITTER
 tn C13 sp -1725.1
 sfrq 100.557 wp 24509.1
 tot 1042.8 r71 9467.9
 tpwr 53 rfp 7742.1
 pw 5.950 rp 175.8
 DECOUPLER
 dn H1 plot -196.9
 dof 0 250
 dm 0 sc
 dmm vs 8777.1
 dpwr 41 th 8
 dmrf 9500 a1 cdc ph



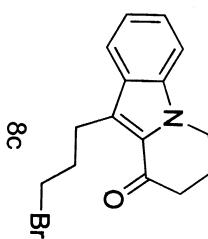
and



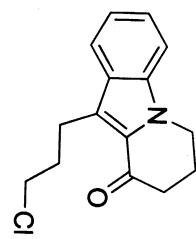
9b

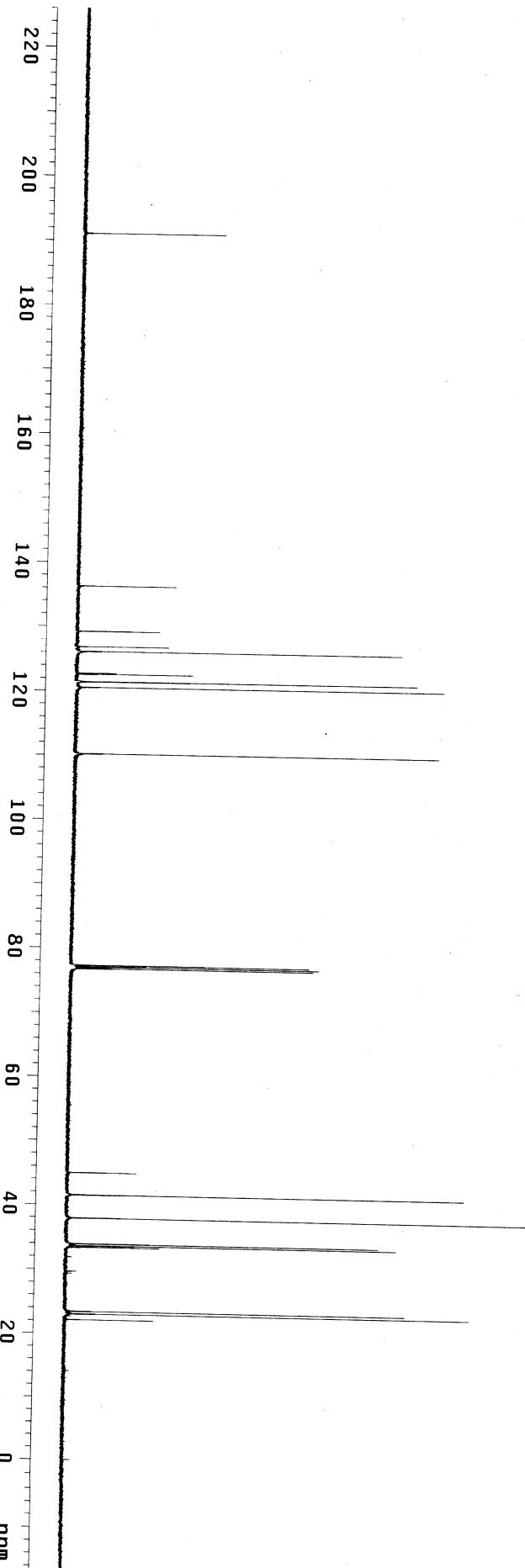


SAMPLE		date	Jun 26 2007	temp	25.
ACQUISITION		solvent	cdcl3	gain	not use
		file	/home/walkup/	spin	not use
		vnmrss	/data/autorange	host	0.00
		02/07.06.26.03/Aug~	pw90	14.70	14.70
		02/data/cdc13_01.t~	alra	6.601	6.601
		id		FLAGS	
sw	8012.8	i1	in		
at	2.049	i1	in		
np	3233.0	d1	hs		
fb	400	32	1b		
bs		2	fn		
ss		1.000			
d1		8	sp		
nt			wp		
ct			rfl		
TRANSMITTER					
tn		H1	rfp		
sfrq	499.802	r1p			
tof	499.7	1p			
tpwr	54				
pw	7.350	wc			
DECOPPLER		sc			
dn	C13	vs			
dof	0	0			
dm	nnn	th			
dmm	C	a1			
dppr	34	cdc			
dmf	32258	ph			
		308.2			



pág





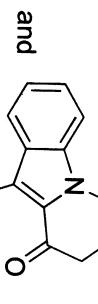
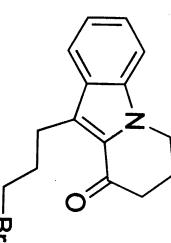
```

exp3  Carbon
      SAMPLE  temp  SPECIAL
date  Jul1  2007  25.0
solvent  cdc13  gain  30
file /home/vakup/~/  spin  not used
vnmrsys/data/auto~  0.008
2007.07.01 01/Aug~  pw90  15.300
01/data/cdc13_01.f~  a1fa  10.000
                                id

ACQUISITION  i1  FLAGS
sw  30487.8  in  n
at  1.300  dp  n
np  79398  hs  y
fb  17000
bs  1b
d1  64
d1  1.000  fn  0.50
nt  4000  not used
ct  1536  DISPLAY
      sp  -2083.0
      wp  30487.3
tn  C13  r11  11760.4
sfrq  125.688  rfp  9676.9
tof  1255.3  rp  156.6
tpwr  49  lp  -279.7
pw  7.650  PLOT
      wc  250
      H1  sc  0
      d1f  vs  17141
      dm  yy  2
      dmm  th
      dmpw  a1  cdc
      dmfp  ph  39
                                12200

```

Carbon

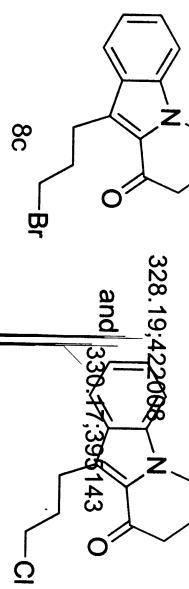


and

K186 31 (0.573) Cm (2:44)

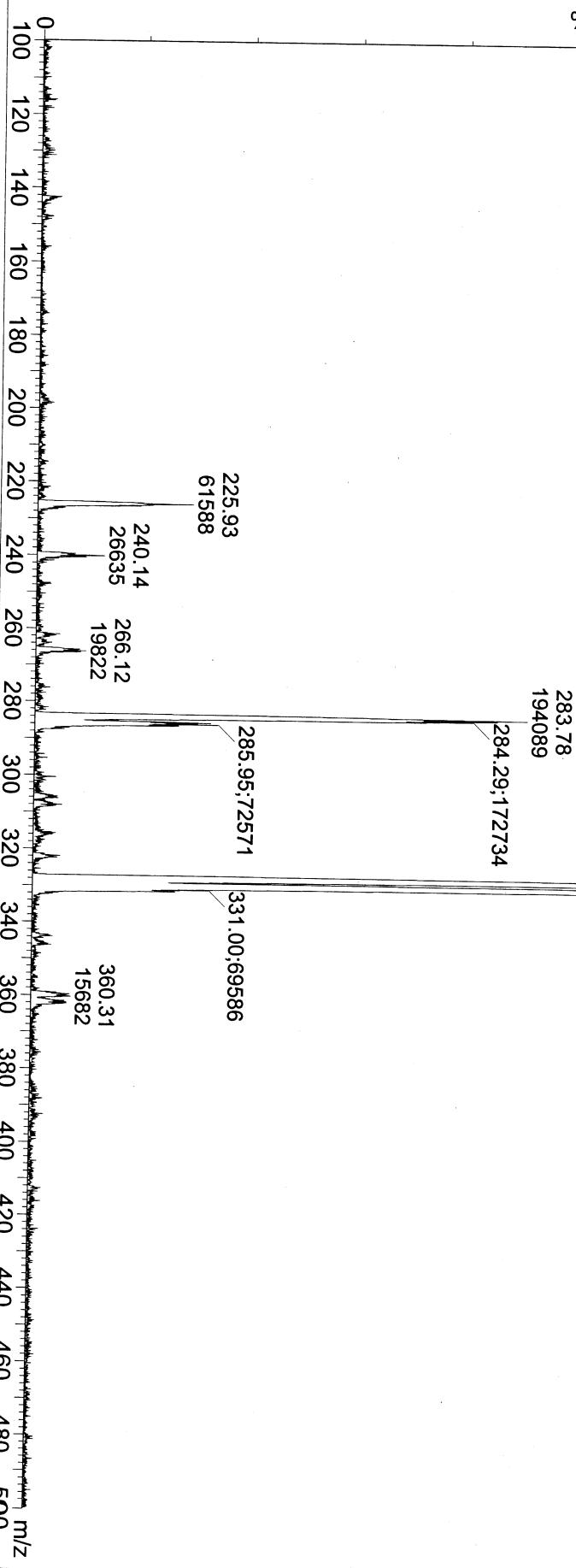
MAA266

27-Jun-2007
Scan ES+
4.22e5



8c
Br

%

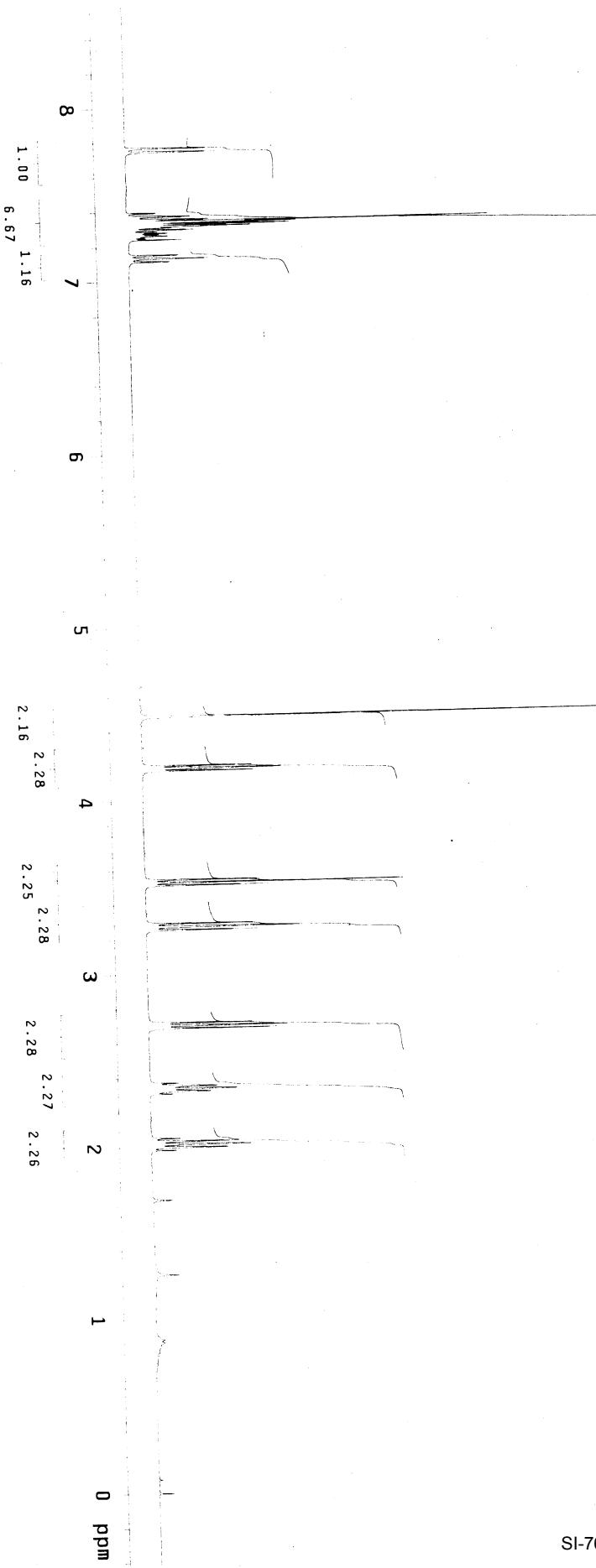
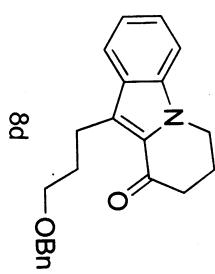


SI-75

Automation directory: /home/walkup/vnmrsys/data/auto_2007.07.01_07
File : s_200701_01/data/cdc13_01.fid
Sample id : s_200701_01

Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: walkup
File: cdc13_01
VNMRs-400 "NMR400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8656807 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.07.01_06
F1le : exp
Sample id : tmpstudy

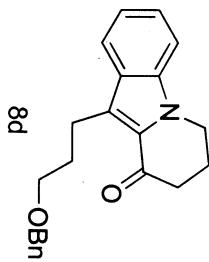
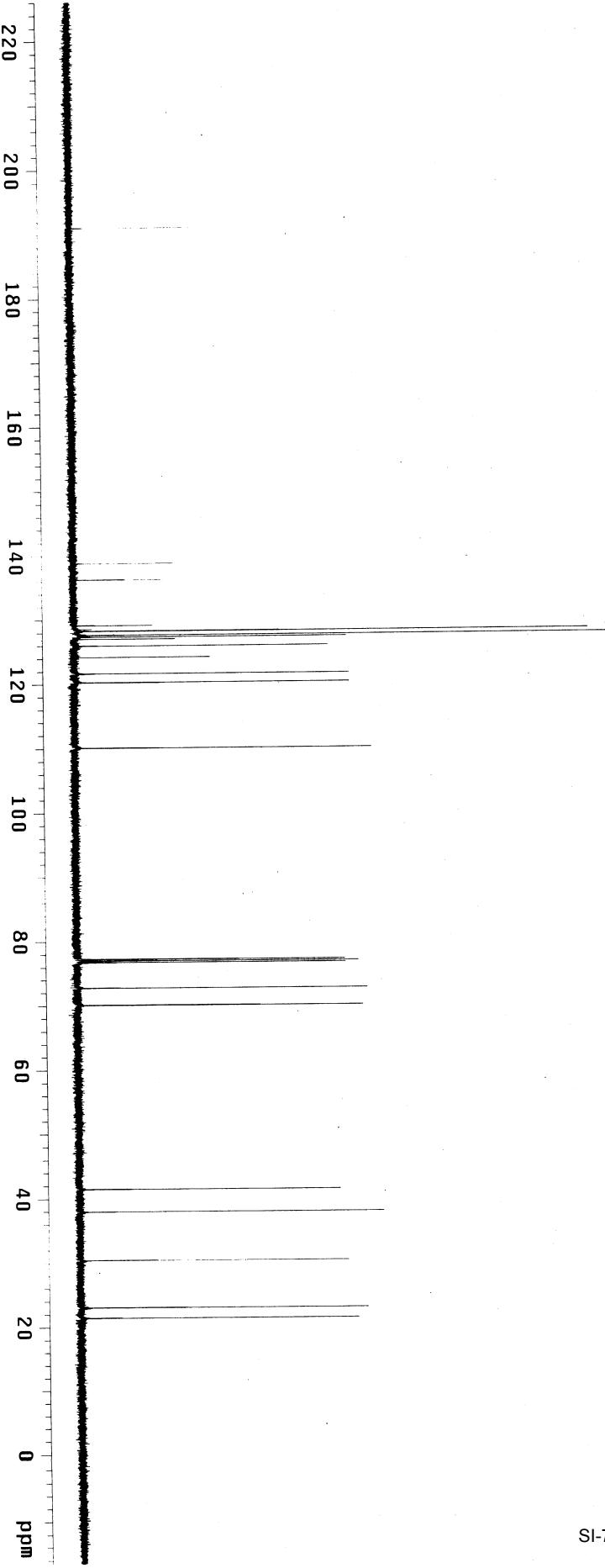
Pulse Sequence: s2pul

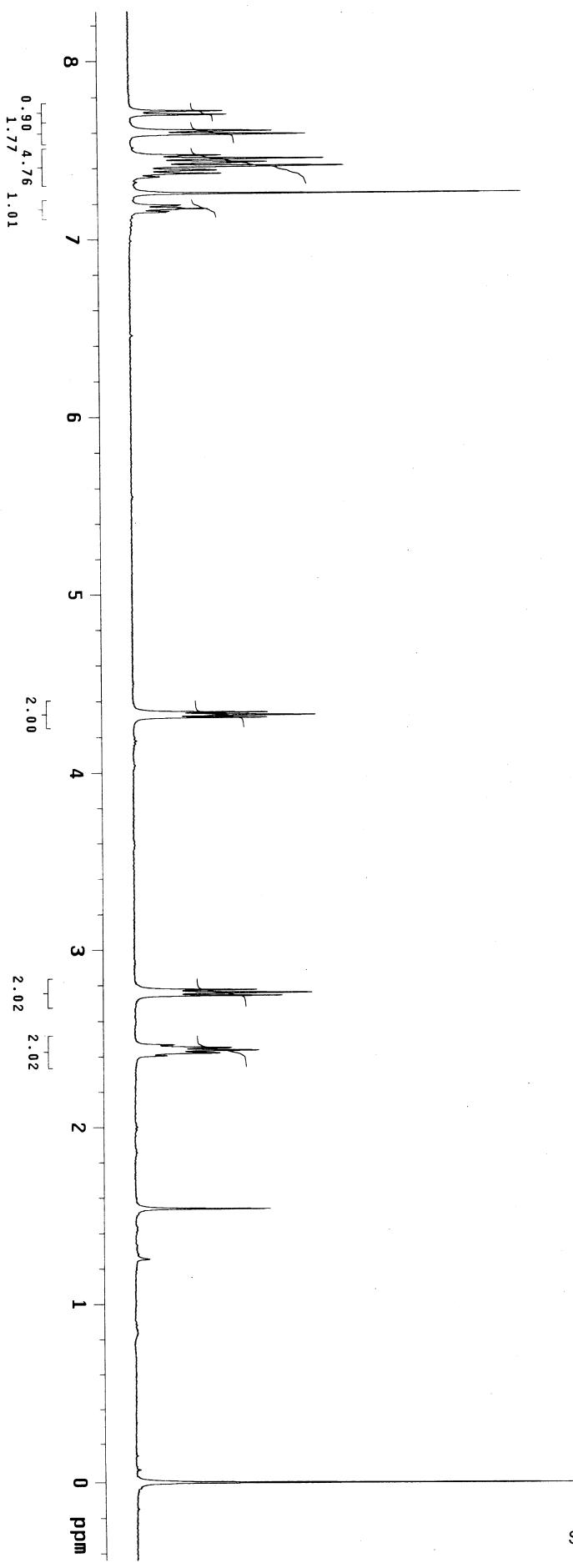
Solvent: cdc13
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMR-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz

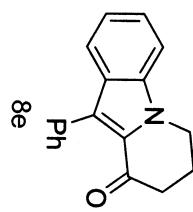
256 repetitions
OBSERVE C13, 125.6746100 MHz
DECOUPLE H1, 499.8016822 MHz

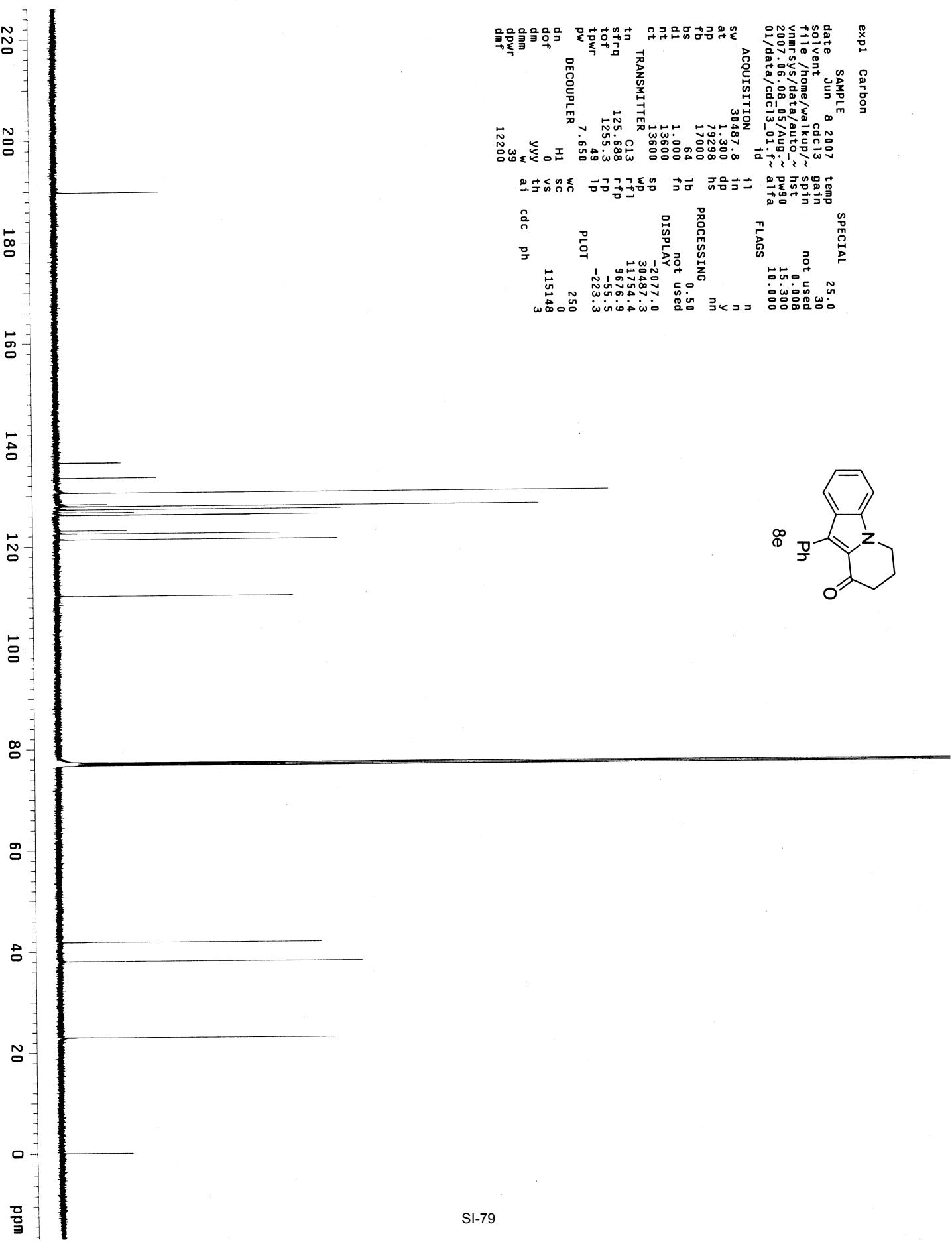
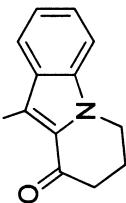
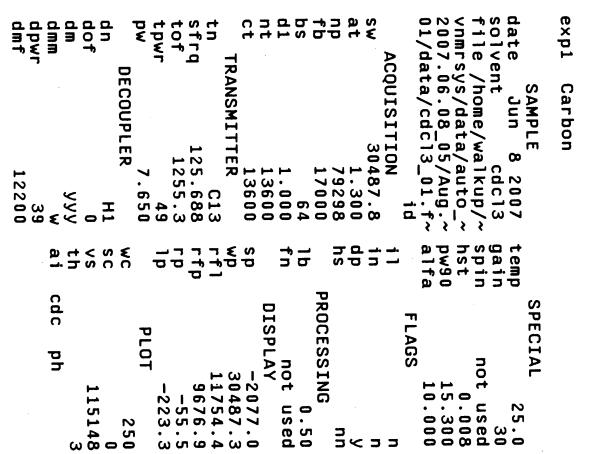
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec





SAMPLE		SPECIAL		25.0
date	Jun 4 2007	temp	gain	20
solvent	cdcl3	not used		
file	/mnt/nmr400/w~	0.008		
al/kup/da/zhang/G~	hst	13.500		
uotao/1/K93-2.fid	pwg0	6.600		
ACQUISITION	alta			
sw	6410.3		FLAGS	
at	2.049	11		n
np	26264	in		n
fb	4000	dp		y
bs	32	hs		nn
d1	1.000		PROCESSING	0.20
nt	8	1b		65535
ct		fn		
TRANSMITTER		DISPLAY		
tn		HI	sp	-176.5
trq	399.868		wp	3487.2
tof	399.6		r ^f 1	800.7
tpw	59		r ^f p	0.7
pw	6.750		rp	163.2
DECOPPLER		1p		-19.3
			PLOT	
d13	C13			
dof	0		wc	250
dm	nnn		sc	0
dmm	c		vs	4555.0
dpwr	34		th	
dmr	29412		cdc	2
			ph	





Automation directory: /home/walkup/vnmrsys/data/auto_2007.08

File: Aug_02/data/cdcl3_01.fid

Sample Id: Aug_02

Pulse Sequence: s2pul

Solvent: cdcl3

Temp: 25.0 C / 298.1 K

Operator: walkup

F1/e: cdcl3_01

VNMRs-500 "nmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.449 sec

With 8012.8 Hz

8 repetitions

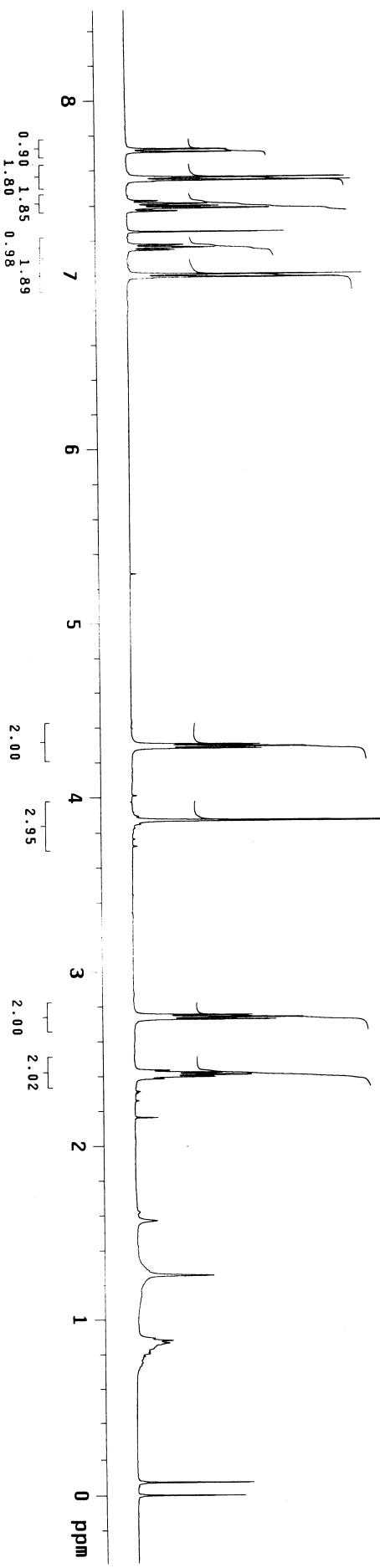
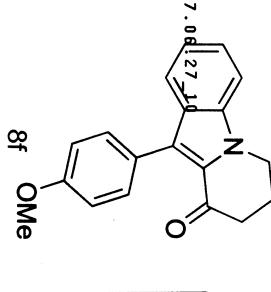
OBSRVE H1, 499.7991883 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrjsys/data/auto_2007.06
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

Aq. time 1.300 sec

Width 3487.8 Hz

1024 repetitions

OBSERVE C13, 125.6746067 MHz

DECOUPLE H1, 499.8016822 MHz

Pover 39 dB

continuously on

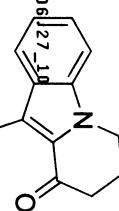
WALTZ-16 modulated

DATA PROCESSING

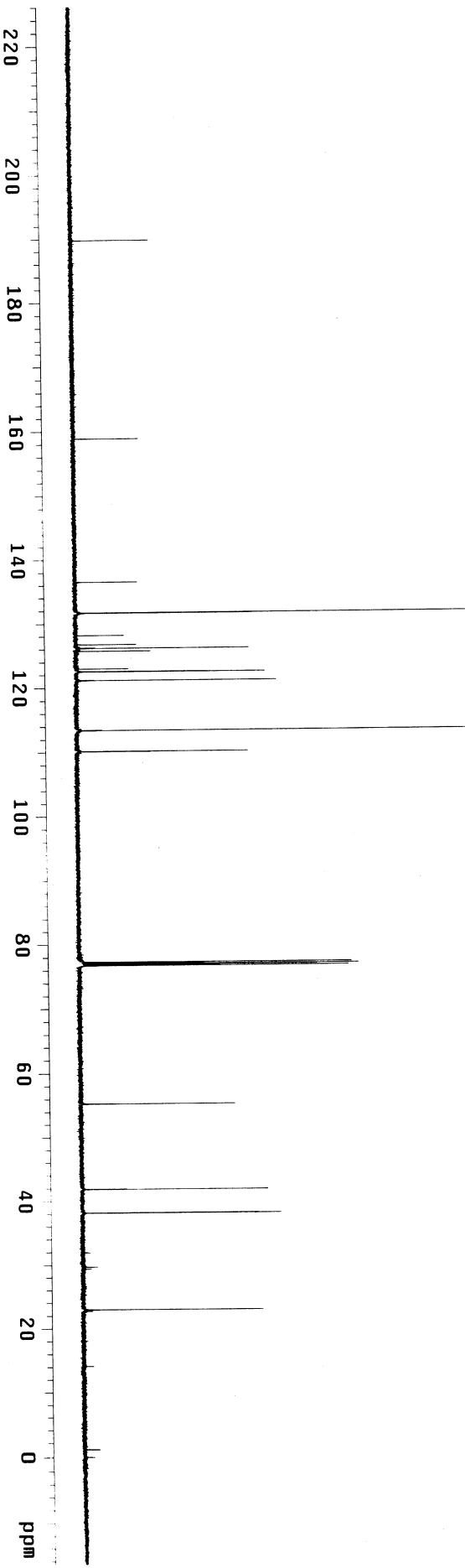
Line broadening 0.5 Hz

FT Size 131072

Total time 39 min, 24 sec



8f CMe



STANDARD 1H OBSERVE - profile

exp2 Protom

SAMPLE	date	temp	SPECIAL
solvent	Jun 25 2007	25.0	
file /mnt/nmr400/w~	cdc13	not used	
alkai/data/Zhang/G~	spin	not used	
utoaol1/k175-2	hst	0.008	
ACQUISITION	pw90	13.500	
	alpha	6.600	

```

SPECIAL 25.0
        not used
        not used
        0.008
        13.500
        6.600

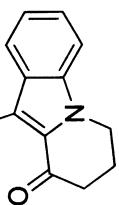
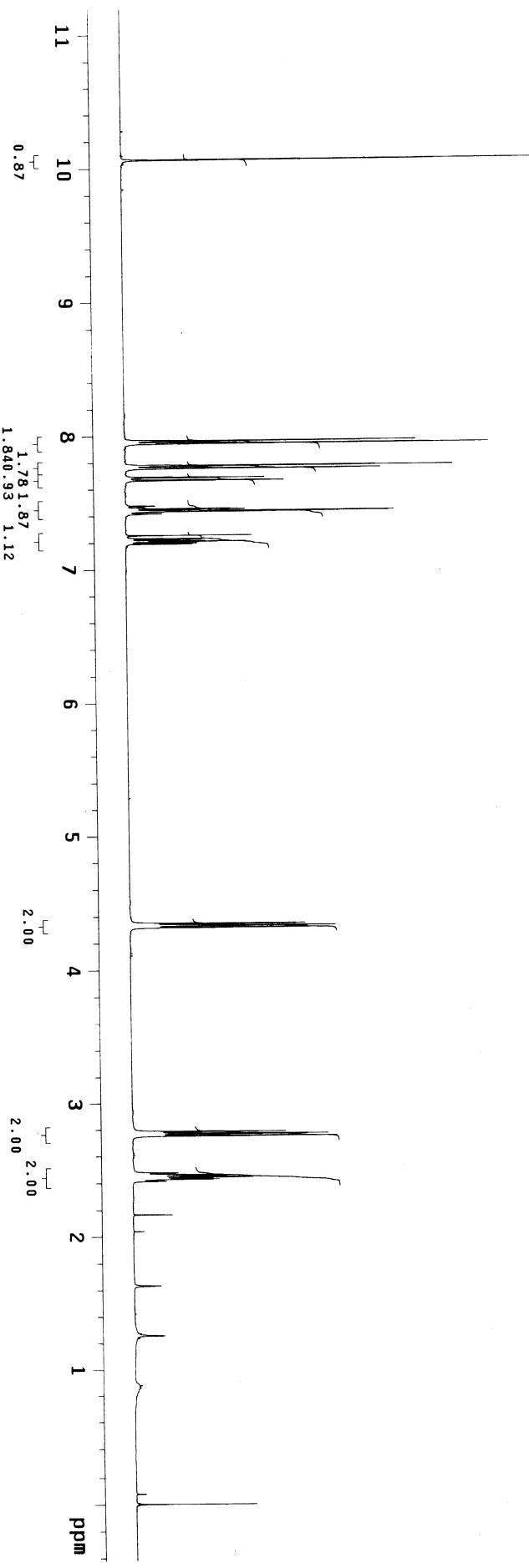
FLAGS n
      n
      y
      nn

PROCESSING 0.20
            65536

DISPLAY -68.6

```

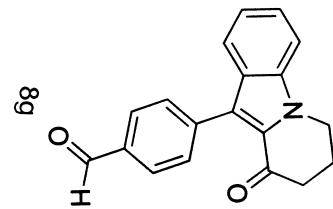
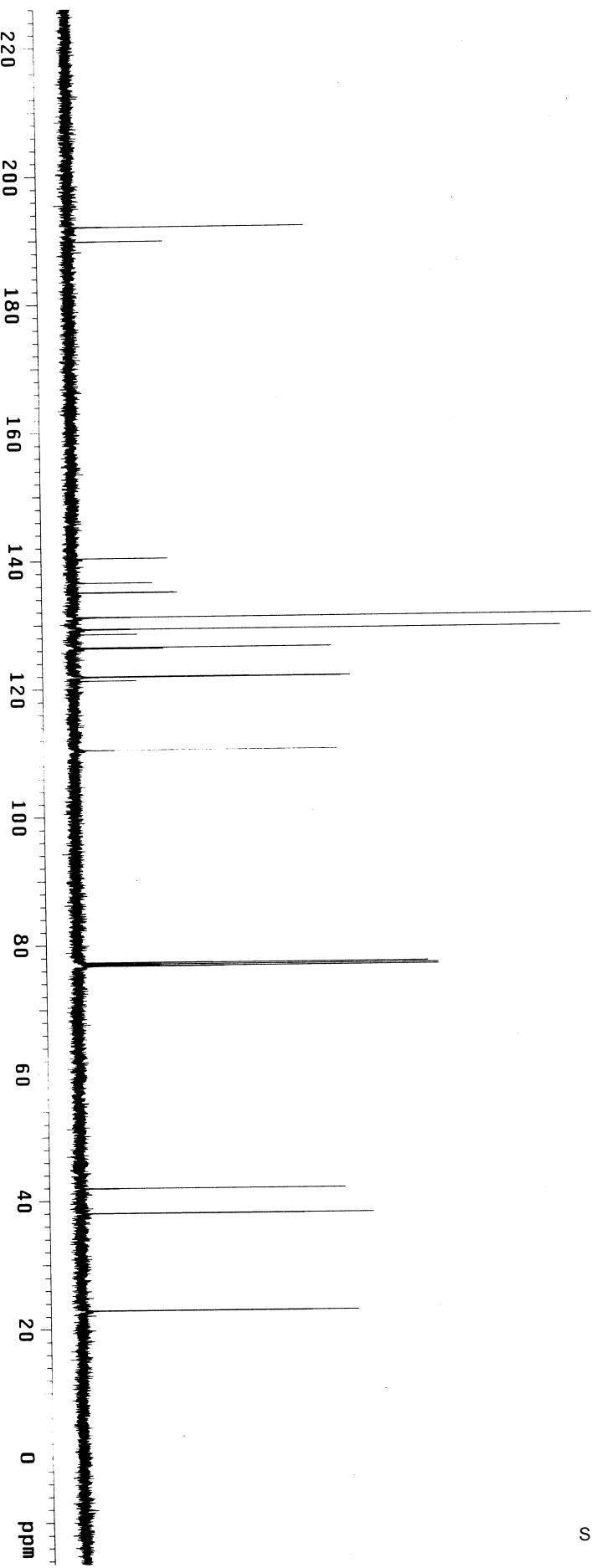
TRANSMITTER		PLOT	
tn	HI	wp	4644.1
sfrq	399.868	rft	801.5
tof	399.6	rpf	0
tpwr	59	rp	-105.6
pw	6.750	tp	-32.0
DECOPPLER			
dn	C13	250	
dof	wc	0	
dm	sc		
dmm	nnn		
dmpw	vs		
dmf	c		
	th		
29412	34	2	
	ai	cdc	
		ph	



8g

exp1 Carbon

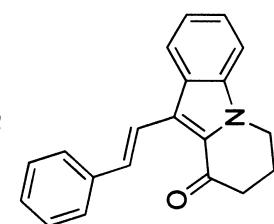
SAMPLE	temp	25.0	
date	Jun 26 2007	30	
solvent	cdcl3	gain	not used
file	exp	spin	0 008
sw	ACQUISITION	hst	0 008
at	30487.8	pwg0	15.300
np	1.300	alfa	10.000
fb	79298	FLAGS	
bs	17000	11	n
d1	8	in	n
nt	1.000	dp	y
ct	1024	hs	nn
tn	64	PROCESSING	1.00
sfrq	C13	1b	not used
tn	125.688	fn	DISPLAY
tn	1255.3	sp	-2082.6
tpwr	49	wp	30487.3
pw	7.650	r11	11761.0
		rfp	9676.9
DECOUPLER			53.5
dn	H1	rp	-252.5
dotf	0	1p	
dm	yyy	PLOT	
dmm	w	250	
dprw	sc	18919	
dmtf	12200	vs	7
		th	
		a1	cdc ph



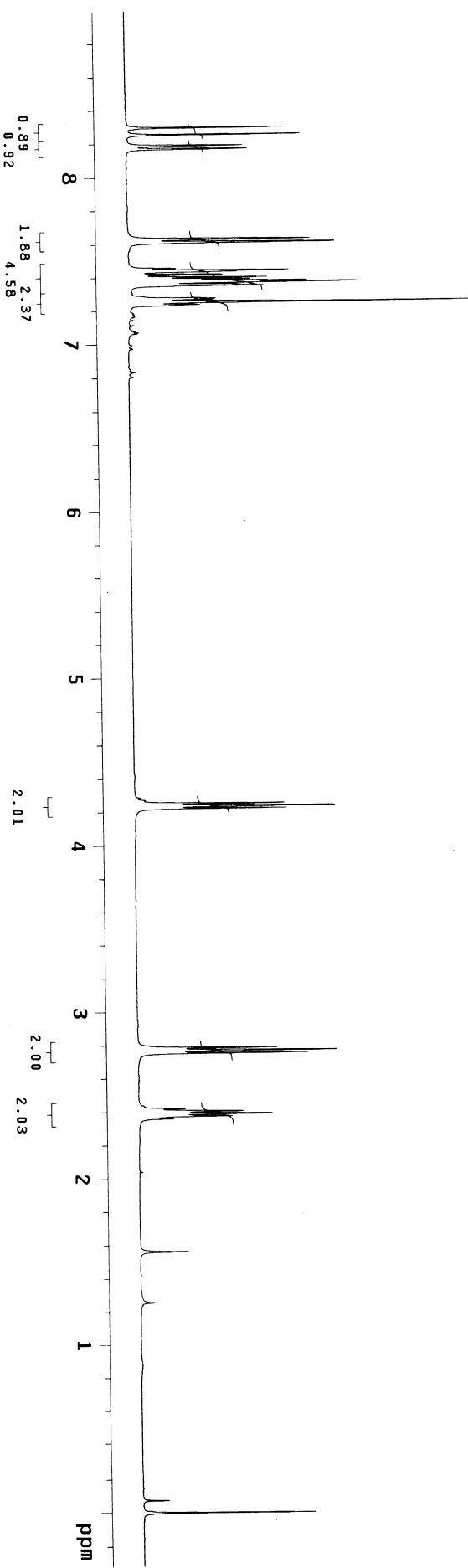
STANDARD 1H OBSERVE - profile

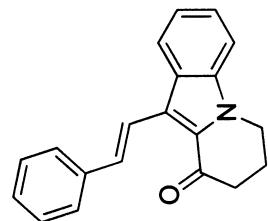
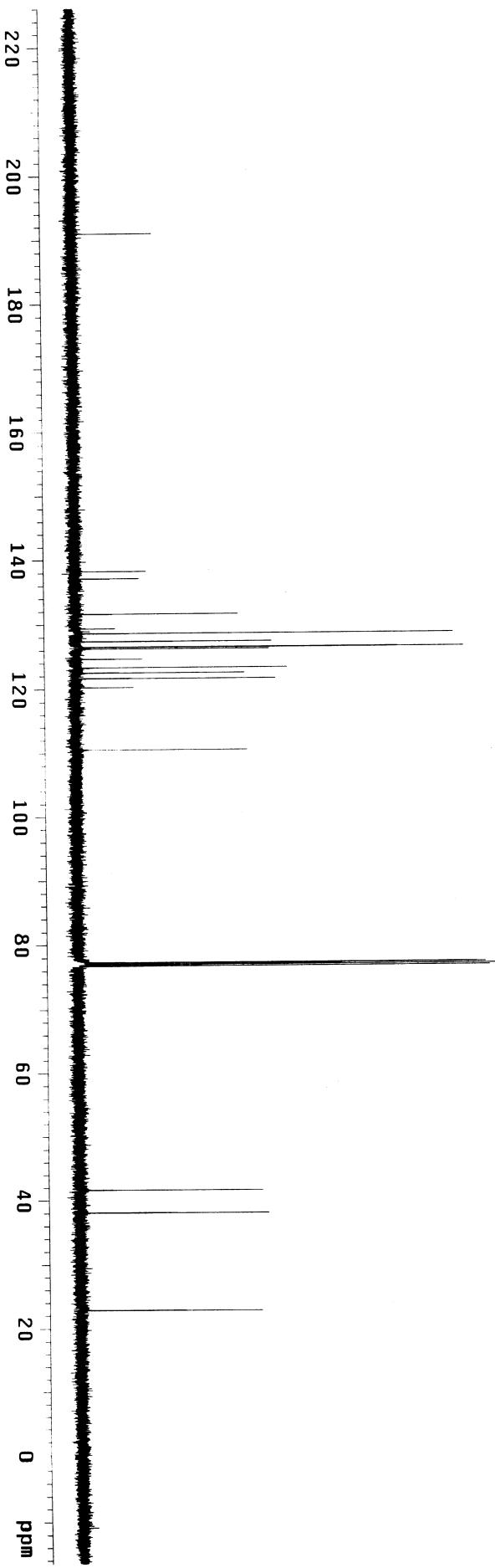
expt1 Proton

SAMPLE	Jun 21 2007	temp	25.0
solvent	cdcl ₃	gain	20
file	/mnt/nmr400/w~	spin	not used
akup	/data/Zhang/G~	hst	0.008
utao1/J/K167-2.fid		pwg0	13.500
ACQUISITION		alra	6.600
sw	6110.3	FLAGS	
at	2.049	11	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	0.20
nt	8	1b	
ct	fn	65536	
TRANSMITTER	H1	DISPLAY	
tn	sp	-129.1	
sfrq	399.868	wp	3725.5
tot	399.6	r1	802.8
tpwr	59	rtp	0
pw	6.750	rp	-20.7
DECOUPLER	1p		-22.2
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dm	c	vs	2325
dpmr	34	th	2
dmrf	29412	ai	cdc ph



8h





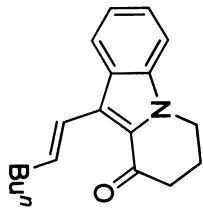
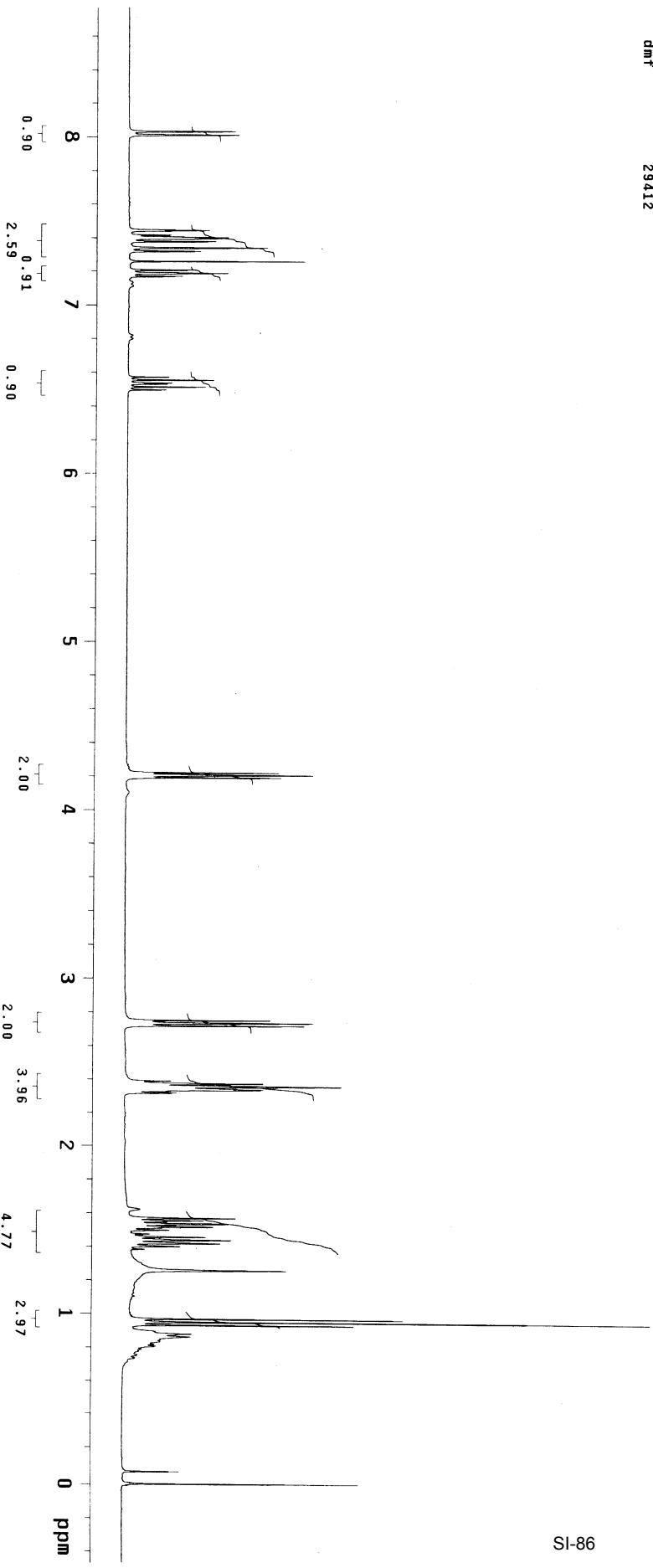
STANDARD 1H OBSERVE – profile

exp2 proton

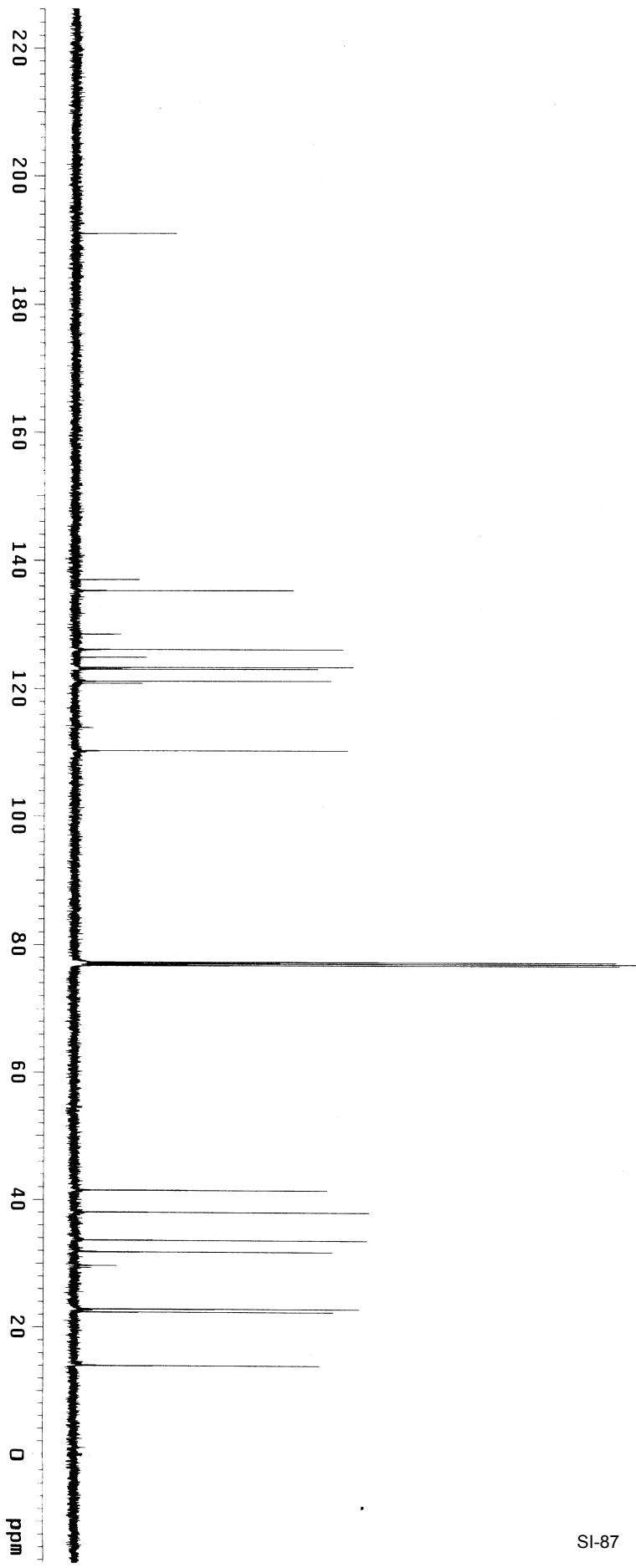
Proton

	SAMPLE	SPECIAL	
date	Jun 25 2007	temp	25.0
solvent	cdcl3	gain	not used
file	/mnt/nmr400/w^~	spin	not used
alkup	/data/ZhangG-	hst	0.008
uotaolLi/K174-1.fid		pw90	13.500

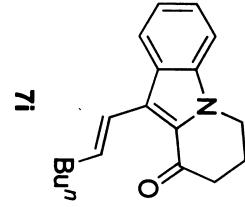
ACQUISITION		alfa	
sw	6410.3	FLAGS	6.600
at	2.049	11	n
np	26264	in	y
fb	4000	dp	y
bs	32	hs	nn
ss	2		
di	1.000	1b	0.20
nt	8	fn	65536
ct	8		
TRANSMITTER		DISPLAY	
tn	H1	sp	-188.0
sfrq	399.868	wp	3694.8
torf	399.6	rfp1	800.5
tpwr	.59	rfp	0.0
pw	6.750	rp	-106.1
		1p	-30.5
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
din	nnn	vs	1902
dim	c	th	3
dpmr	34	ai	cdc
dmf	29412	cd	ph

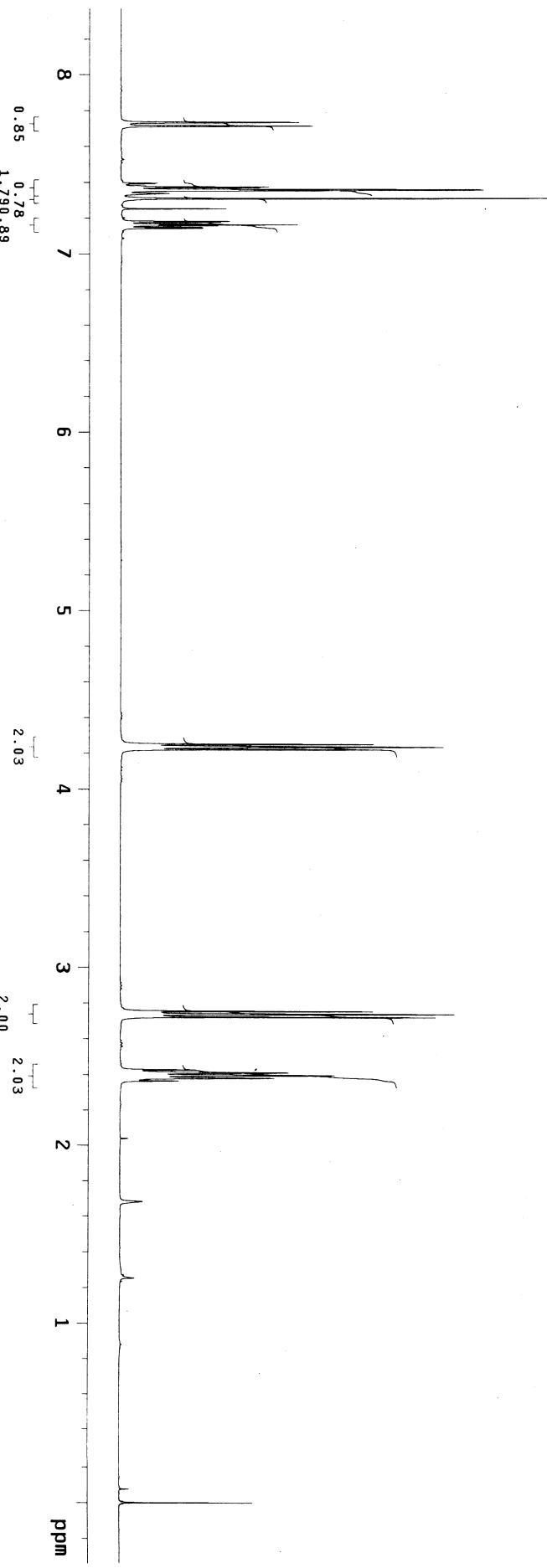


4



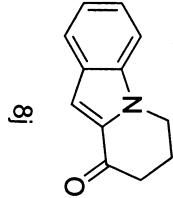
expt Carbon
 SAMPLE Jun 26 2007 temp 25.0
 solvent cdc13 gain 30
 f1le ACQUISITION exp spin not used
 sw 30487.8 pw90 0.008
 at 1.300 pw15.300
 np 79208 a1fa 10.000
 fb 17000 11 FLAGS n
 bs 1700 8 in n
 di 1.000 ap y
 nt 1024 hs nn
 ct 672 1b PROCESSING 1.00
 TRANSMITTER C13 fn not used
 tn 125.688 1b DISPLAY 1.00
 sfrq 125.3 sp -2077.4
 toff 1255.3 sp 30487.3
 tpowr 49 wP 11754.9
 pw 7.650 rP 9676.9
 DECOUPLER H1 rP 64.0
 dn 0 1P -273.7
 dof 0
 dm 0
 dmm 39 wC 250
 dpwr 12200 vs 0
 dmrf 12200 th 46287
 a1 cdc 68
 ph





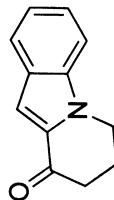
exp2 Proton

SAMPLE	8	2007	temp	25.0
date	Jun	cdd13	gain	20
solvent			spin	20
file	/mnt/mnr400nm			0.008
alikup	data/Zhang/G~			13.500
uotao1/K104-1.fid				6.600
ACQUISITION				
sw	6410.3		FLAGS	
at	2.049	11		n
np	2664	1n		n
fb	4000	dp		y
bs	32	hs		mn
dl			PROCESSING	0.20
nt	8	1b		
ct		fn		65536
TRANSMITTER			DISPLAY	
tn		H1	SP	-136.7
sfrq		399.868	WP	3483.9
tot		399.6	RT1	802.3
tpwr		59	RTP	0
pw	6.750			-97.6
DECOUPLER				-28.7
dn	C13		IP	
dof	0	WC	PLOT	250
dm	nnn	SC		0
dmm	C	VS		637
dmpw	34	TH		2
	29412	AI	CDC	PH

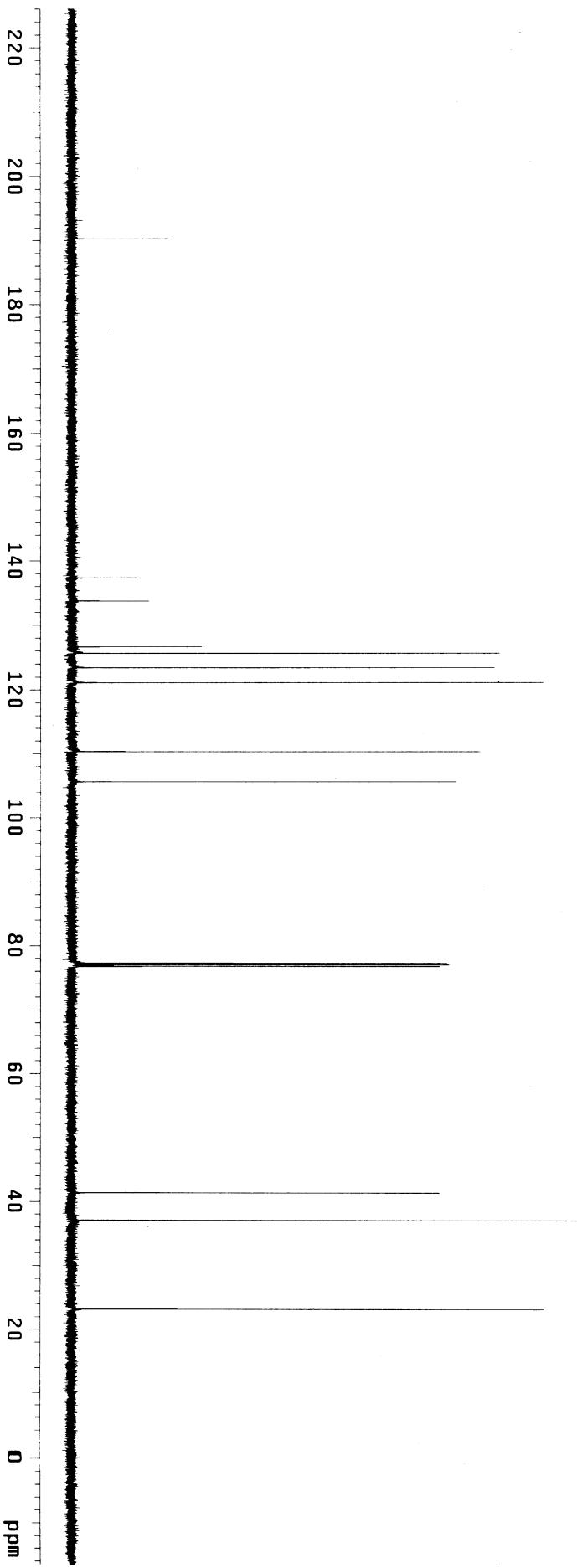


T1 EXPERIMENT
T1 EXPERIMENT
T1 EXPERIMENT

exp1 Carbon
SAMPLE

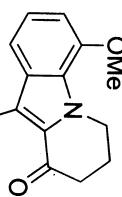


8

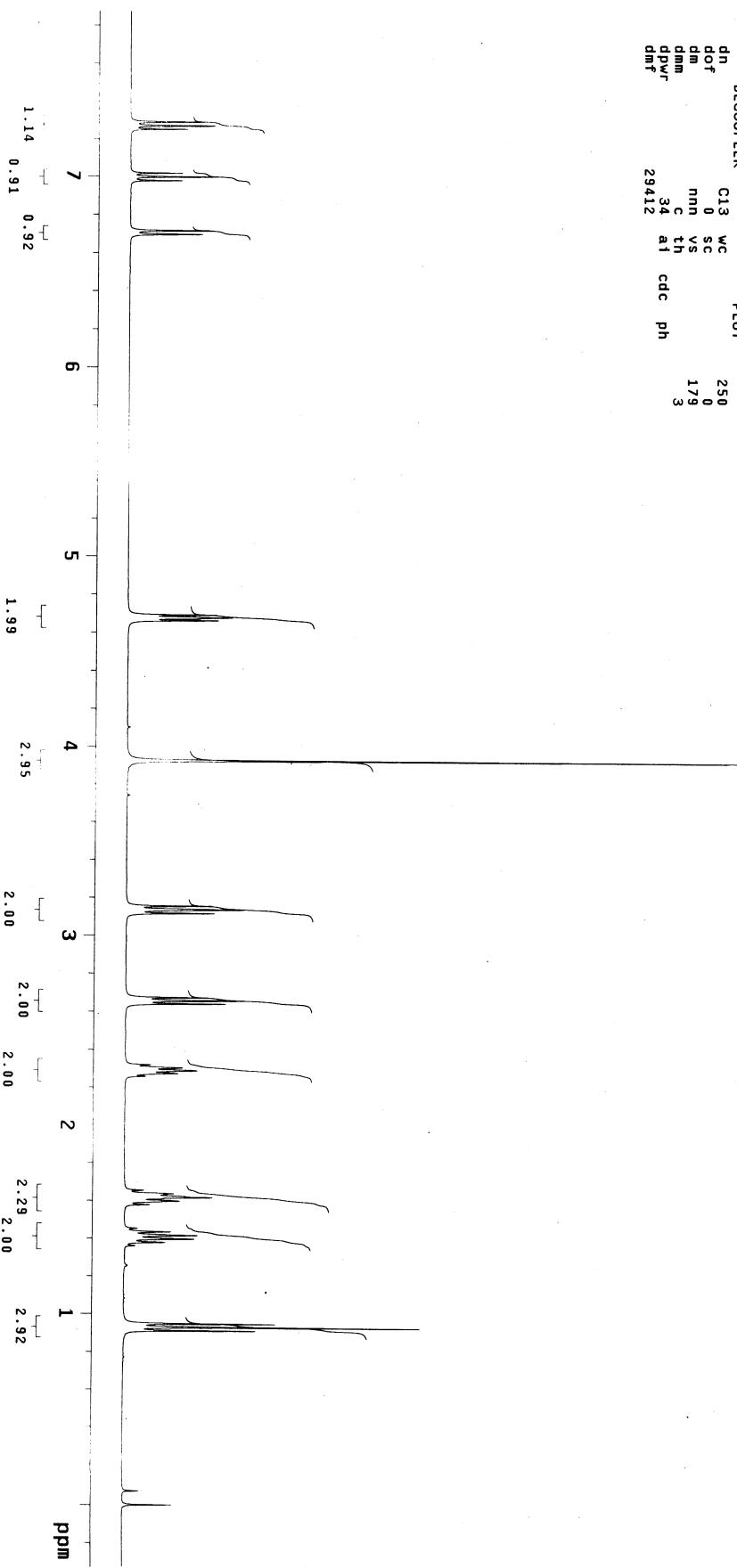


STANDARD 1H OBSERVE – profile

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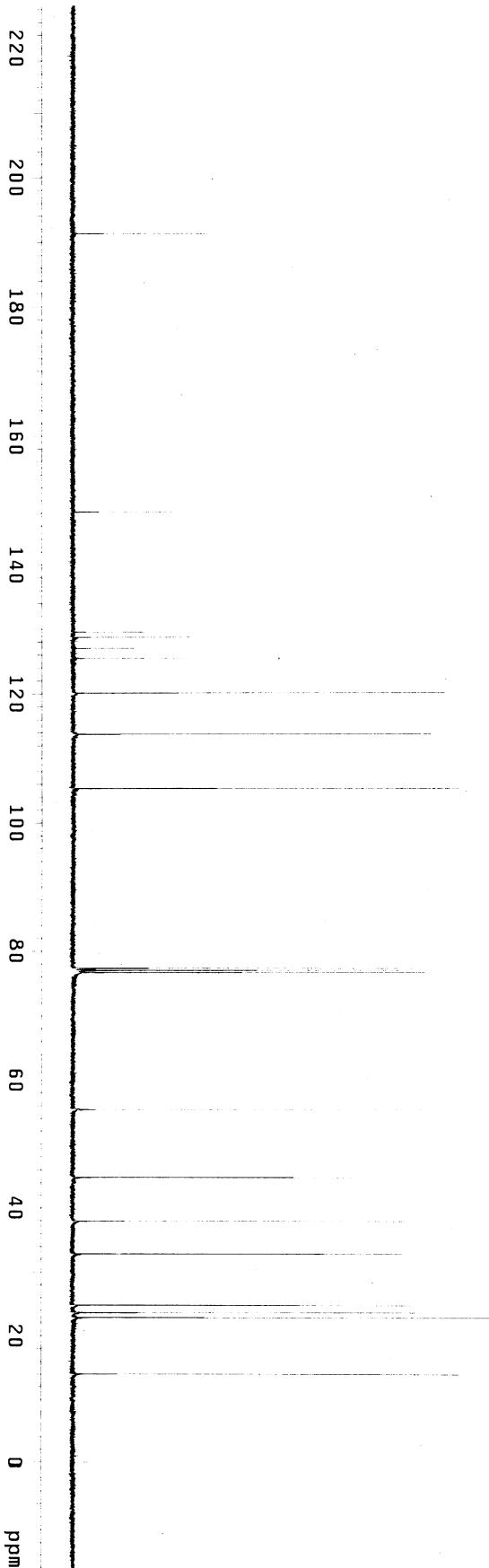
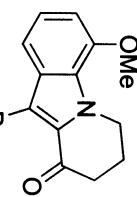


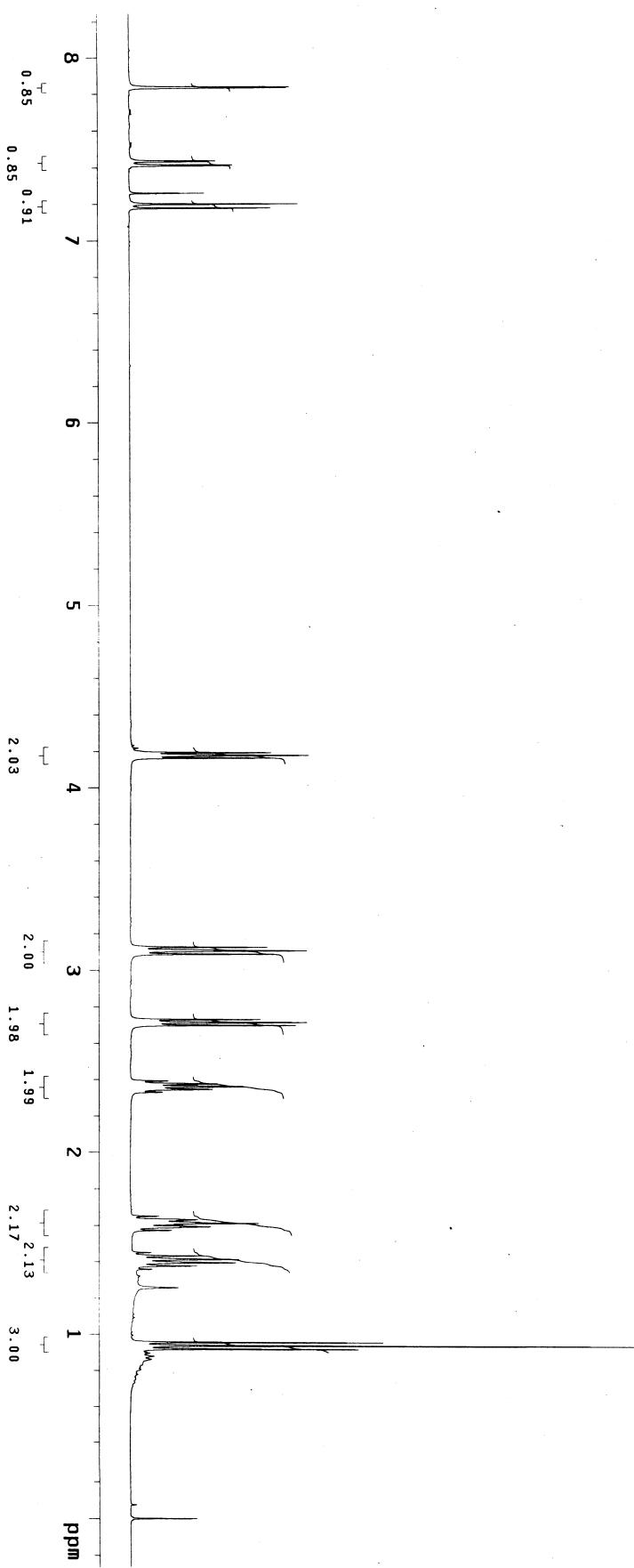
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STANDARD 1H OBSERVE - profile

SAMPLE		date	Aug 2 2007	temp	25.0	SPECIAL
Solvent		cdcl3	gain	30	file	
ACQUISITION		exp	spin	not used	0.008	
SW		24.09.8	hst	0.008	pw90	
AT		1.330	11.900	11.900	alfa	
NP		63750	10.000	10.000		
FB		17000	11	n	FLAGS	
BS		16	in	n		
DI		1.000	dp	y		
CT		32960	hs	nn		
TRANSMITTER		1b	0.50	0.50	PROCESSING	
SFREQ		100.557	not used	not used	DISPLAY	
TOF		1042.8	sp	-1721.4		
TPWR		53	sp	-24509.1		
PW		5.950	wp	9464.2		
DECOPPLER		5.950	rfp	7742.1		
D1		H1	rfp	179.4		
D0F		0	ip	-192.2		
D0M		VVV	PLT	-192.2		
D0MM		W	250	250		
D0PWR		SC	56722	56722		
DMT		41	5	5		
ATI		9500	VS			
CDCL3		th				
PH		ai				

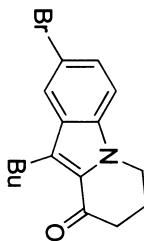


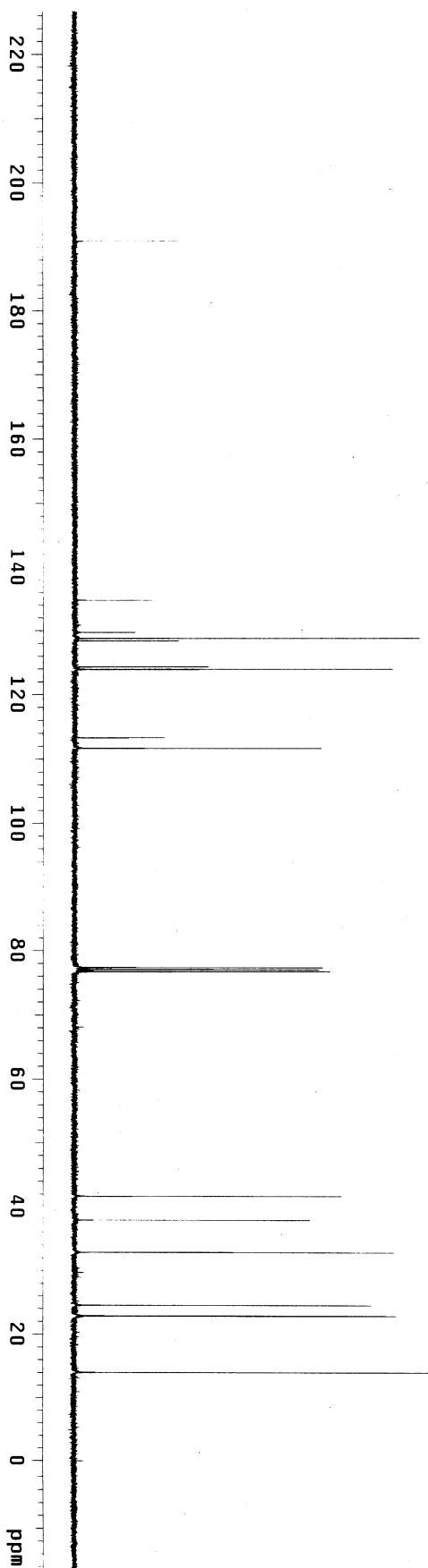


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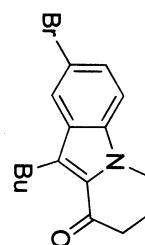
expt1 proton
SAMPLE          JUN 31 2007 temp 25.0
date           solvent cdcl3 gain 30
file /mnt/nmr400/vnmr spin not used
fileup/databk/Zhang/GC/rotor pg90 0.008
datafile1/2841.fid het 13.500
ACQUISITION    alfa 6.600
sw 6410.3      at 2.049 i1 n
np 26264      in 26264 n
fb 4000      dp 400 y
bs 8          hs nn
d1 1.000      PROCESSING 0.20
nt 8          1b 65536
ct 8          fn
TRANSMITTER    H1 sp DISPLAY -106.6
tn 399.868      wp rfp 3403.3
sfrq          399.868      rfp 798.9
t0f 399.6      59 0
tpwr 6.750      75.2
pw 1p      -13.5
DECOPPLER      C13 PLOT 250
dn 0          wc 0
dof 0          nnn 0
dmn 0          sc 0
dmm 0          vs 183
dpr 34          th 6
dmr 29412      a1 cdc ph

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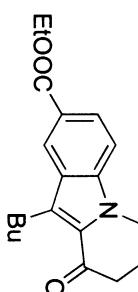




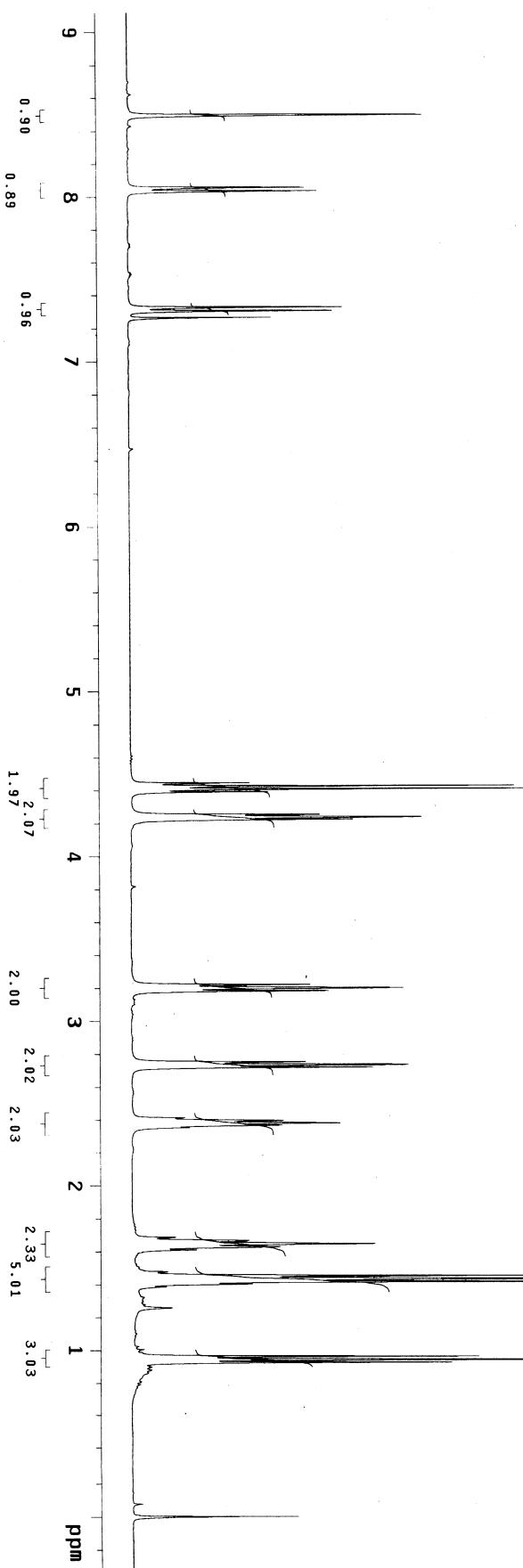
expt	Carbon	SAMPLE	SPECIAL
date		Jul 31 2007	temp 25.0
solvent		cdcl3	gain 60
file		/mnt/nmr400/w~	spin not used
alku/		data/Zhang/G-	hist 0.008
utoaol1/k284-1carb~		pw90	11.900
alfa		10.000	
ACQUISITION	on. fid	FLAGS	
sw	245.09.8	i1	
at	1.300	in	n
np	6.3750	dp	y
fb	17.000	hs	nn
bs	64	PROCESSING	
d1	1.000	1b	0.50
nt	12.000	fn	not used
ct	12.00	DISPLAY	
TRANSMITTER		sp	-1721.4
tn	C13	wp	245.09.1
sfrq	100.557	rf1	9464.2
tof	1042.8	rfp	7742.1
tpwr	53	rp	115.9
pw	5.950	1p	-195.5
DECOPPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yy	vs	1417
dm	w	th	6
dppr	41	cdcl	ph
	9500		

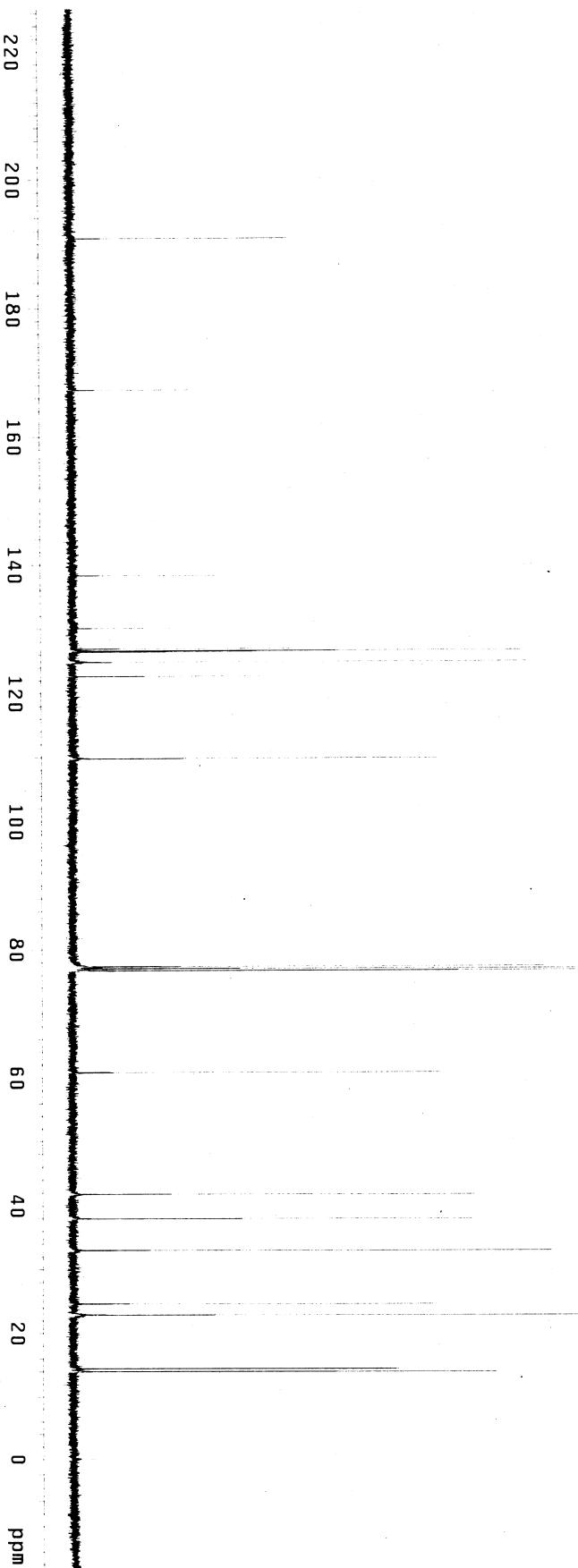


exp1		proton	
	SAMPLE	SPECIAL	
date	AUG_1 2007	temp	25.6
solvent	cdcl3	spin	31
so	/mnt/nmr400/wm	spin	not used
ai	ai	spin	0.0000
lup	/data/Zhang/G	hst	0.0000
utoa01	/K265-1.fid	pw90	13.5000
ACQUISITION		alfa	6.6000
sw	64.0-3	FLAGS	
at	2.049	11	1
np	2.049	11	1
fb	4000	dp	1
bs	8	hs	1
d1	1.000	PROCESSING	0.2
nt	1.000	nn	0.2
ct	8	fn	65533
TRANSMITTER		DISPLAY	
tn	H1	sp	-125.6
srq	399.868	wp	3770.6
tof	399.6	rf1	776.0
tpw	559	rfp	81.0
pw	6.750	rp	-25.0
DECOUPLER	C13	1p	
dn	0	PLT	251.0
dof	nnn	sc	57.0
dm	c	vs	
dppr	34	th	
dmr	29412	cdc	ph



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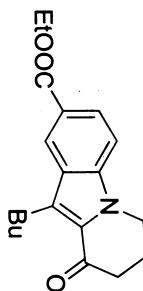




```

expi Carbon
      date     SAMPLEx      SPECIALx      25.0
      aug 1 2007      tempx      25.0
      solvent      tdc13      gainx      30.0
      trile/home/walkup/~      spinx      not used
      vnmrjs/data/auto/~      spinx      not used
      2007.08.01/s 2007.08.01      pw90      0.008
      801_001/data/cdc13~      pw90      11.900
      801_001/data/cdc13~      alfa      10.000
      ACCUStION      01.fid      FLAGS      n
      SW      24509.8      n
      at      1.300      n
      np      63750      n
      fb      17000      n
      bs      64      n
      d1      1.000      fn      not used
      nt      20000      sp      -1720.6
      ct      20000      sp      -1720.6
      TRANSMITTER      C13      wp      24509.1
      tn      100      557      rf1      9463.4
      sfrq      1042.8      rf1      7742.1
      tof      1042.8      rp      111.3
      tlow      53      rp      -185.6
      pw      5.950      wc      250
      DECOUPLER      H1      sc      0
      dn      0      vs      8264.0
      dof      yyy      th      5
      dm      w      ai      cdc      8264.0
      dmm      w      ai      ph      5
      dmr      41      ai
      dnrt      9500

```



8

expt proton

SAMPLE cdc13 temp 25.0
 date Aug 3 2007 gain 30
 solvent /home/walkup/.~ spin not used
 file /home/walkup/.~ nmr400/walkup/data~ hst 0.008
 /Zhang/Guotao1/12~ pw90 13.500
 96.3.fid alfa 6.600

ACQUISITION FLAGS

sw 6410.3 i1 n
 at 2.049 in n
 np 26264 dp y
 fb 4000 hs nn
 bs 1.000 1b 0.20
 di 1.000 1 fn 65536
 nt 1 ct DISPLAY -108.2

TRANSMITTER

tn H1 wp 361.9.5
 sfrq 399.868 rf1 800.5
 tof 399.6 r1p 0
 tpwr 59 rp 155.8
 pw 6.750 1p -33.1

DECOPPLER

C13 PLOT

250

sc 0

vs 300

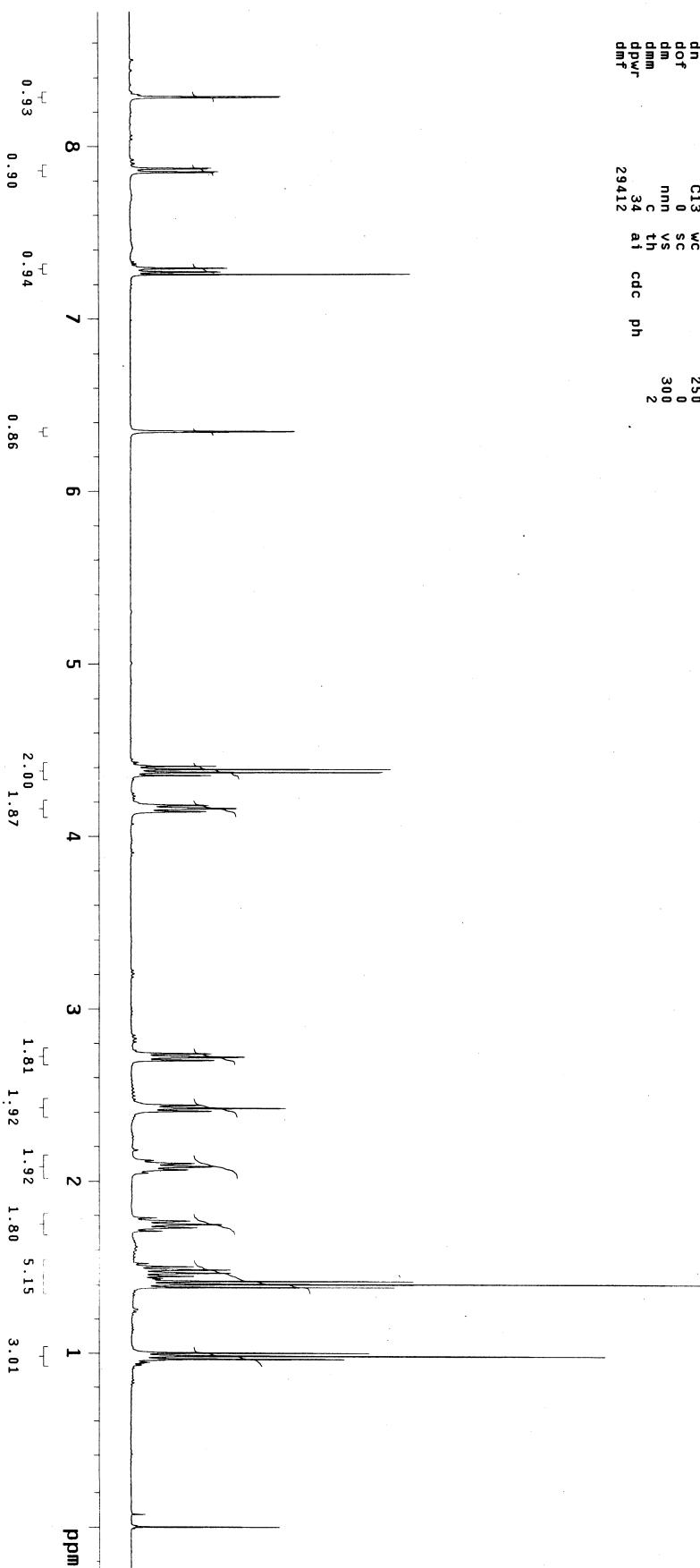
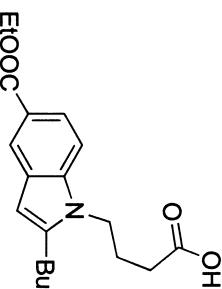
nnn 2

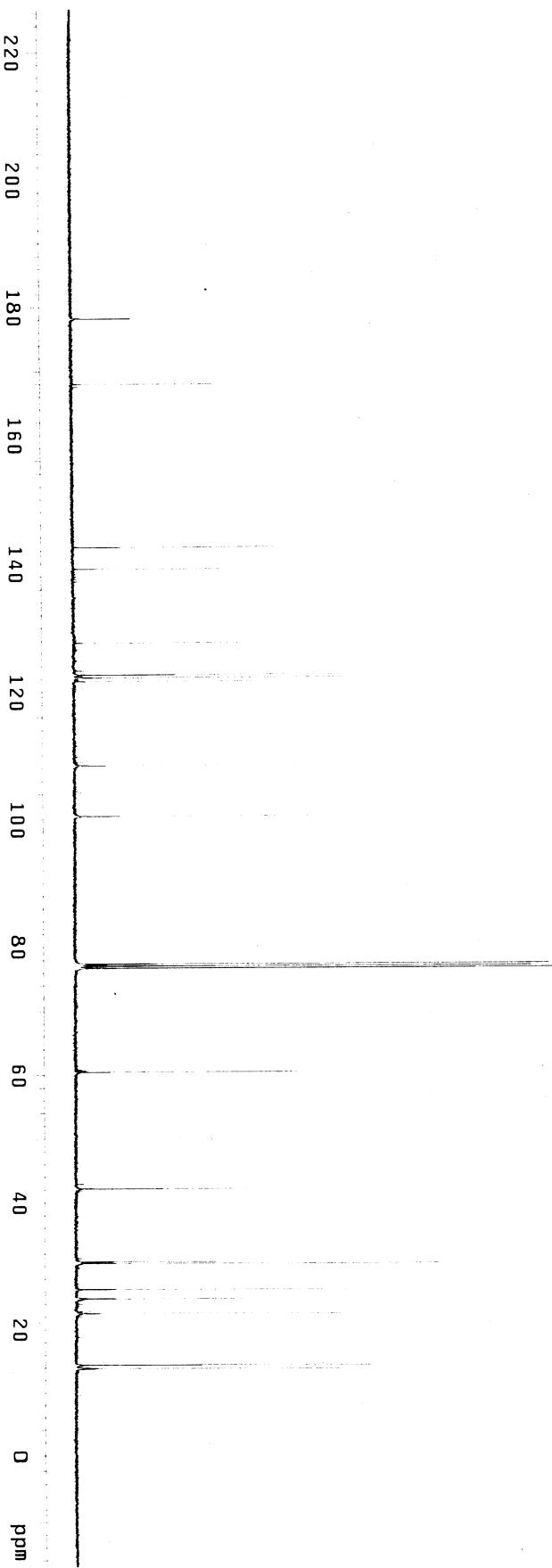
dmm 34

a1

cdc ph

29412



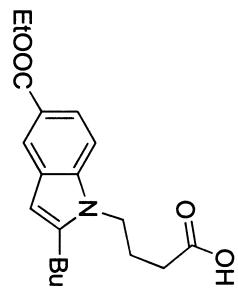


```

exptl  Carbon
      SAMPLE  temp  SPECIAL  25.0
      date  Aug 3 2007  temp  30
      solvent  cdcl3  gain  not used
      file /home/walkup/~ spin  0.008
      vnmr/svs/data/auto/  spin  11.900
      2007_08_03_16/s_20~ pw90  10.000
      070803_001/data/cd~ alfa  10.000
      c13_01.fid  FLAGS

      ACQUISITION  11  n
      sw  24509.8  in  n
      at  1.300  dp  v
      np  63750  hs  nn
      17000
      tb  64  1b  PROCESSING  0.50
      bs  1.000  fn  not used
      d1  12000
      nt  12000  DISPLAY  -1719.9
      ct  12000  sp  24509.1
      TRANSMITTER  wp  24509.1
      tn  C13  r1  9462.7
      sfrq  100.557  rfp  7742.1
      tof  1042.8  rp  -172.9
      tpowr  53  1p  -193.0
      pw  5.950  PLOT  250
      DECOUPLER  wc  250
      dn  H1  sc  8228.7
      dof  0  vs  8228.7
      dm  yyy  th  8
      dmm  41  ai  cdc  ph
      dpwr  9500

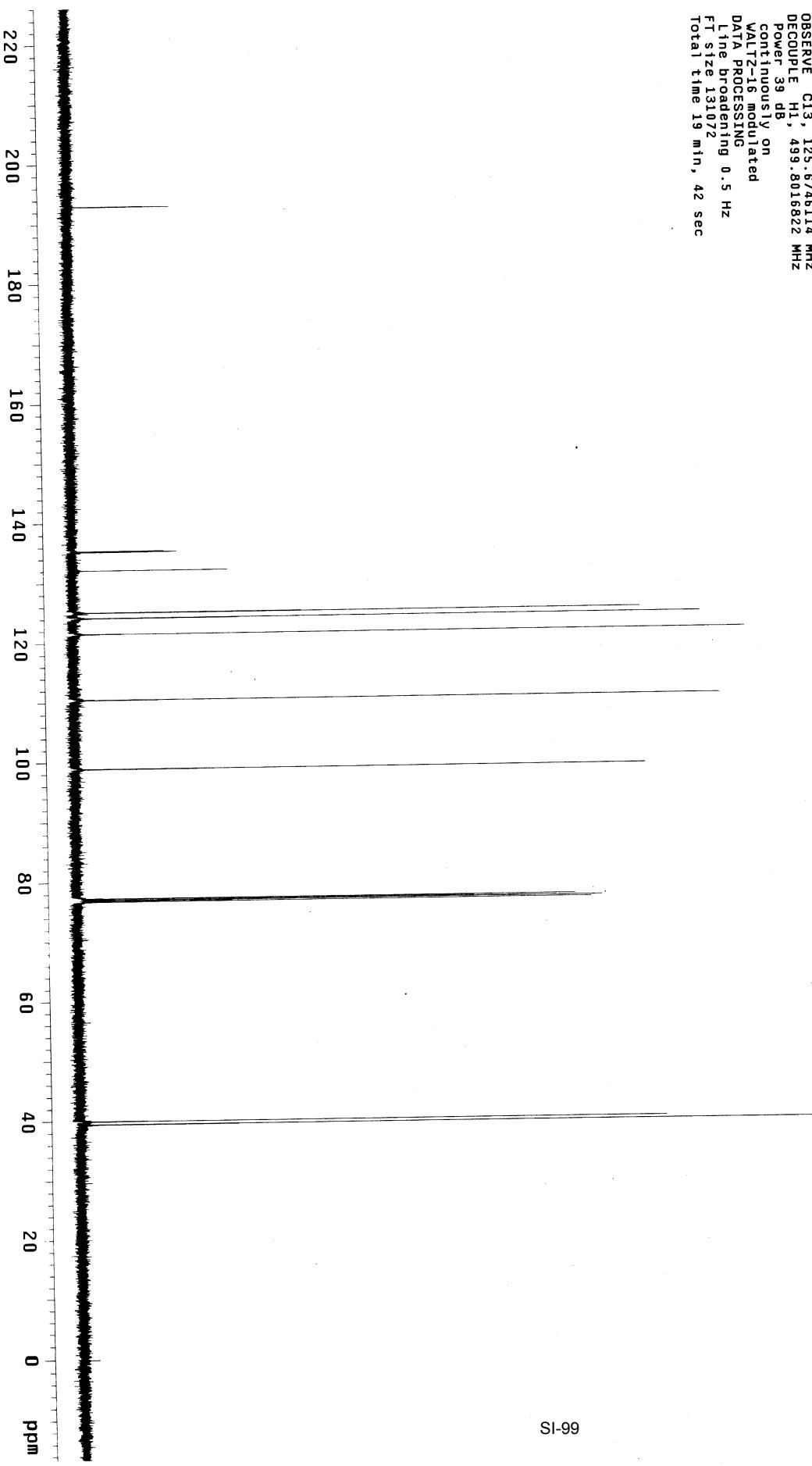
```




```
Automation directory: /home/valku/vnmSys/data/auto/2007-06-12
File: /home/walkup/data/Zhang/xiaogenghuang/hxg-3-15C.rid
File id: tmppwalkup
```

Pulse Sequence: s2pul
 Solvent: cdc13
 Temp. 22.0 C / 295.1
 Operator: walkup
 File: hgx-3-157C
 VNMRs-500 "nmr500"

Relax. delay 1.000 sec
 pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 30.087.8 Hz
 448 repetitions
 OBSERVE H13, 125.646114 MHz
 DECOUPLE H1, 499.8016822 MHz
 Power 39 dB
 continuously on
 WALZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 13.072
 Total time 19 min, 42 sec

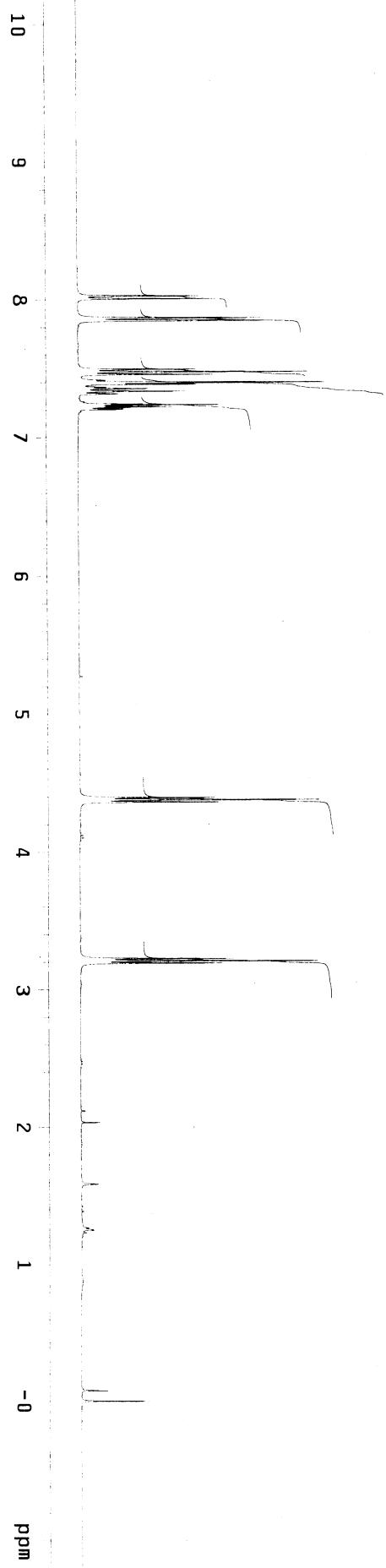
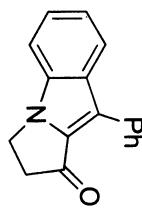


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_26
File: s_20070611_001\data\cdcl3_01.fid
Sample id: s_20070611_001

Pulse Sequence: s2pul

Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_01
VNMRs-400 "NMR400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.019 sec
Width 640.3 Hz
8 repetitions
OBSERVE H1 399.8656811 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



File: hxg-3-155-2C

Pulse Sequence: s2pul

Solvent: cdc13

Temp: 25.0 C / 298.1 K

Operator: walkup

File: hxg-3-155-2C

"redJacket"

INOVA-500

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 30087.8 Hz

600 repetitions

OBSERVE C13, 125.6746105 MHz

DECOPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

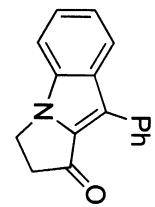
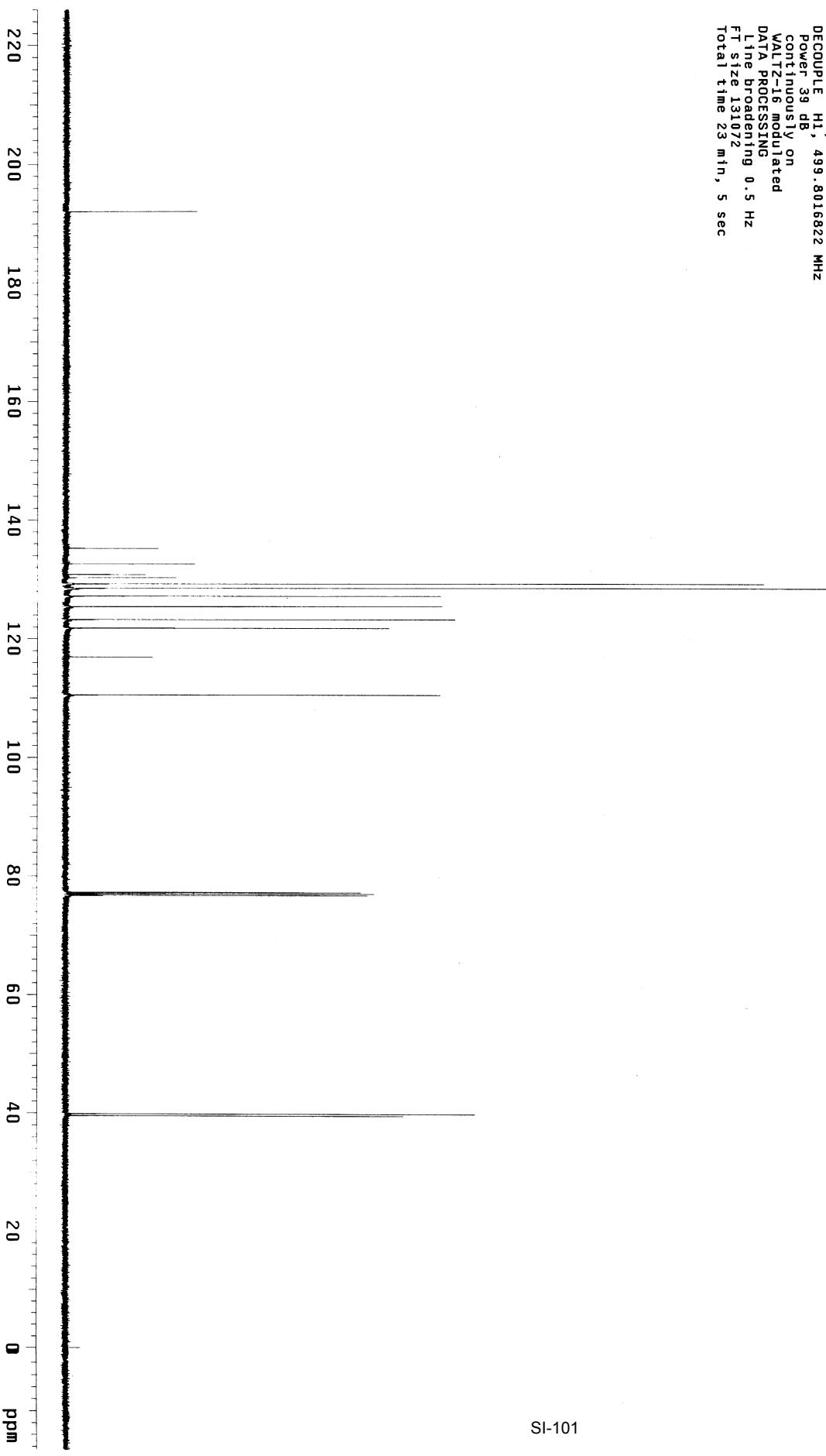
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

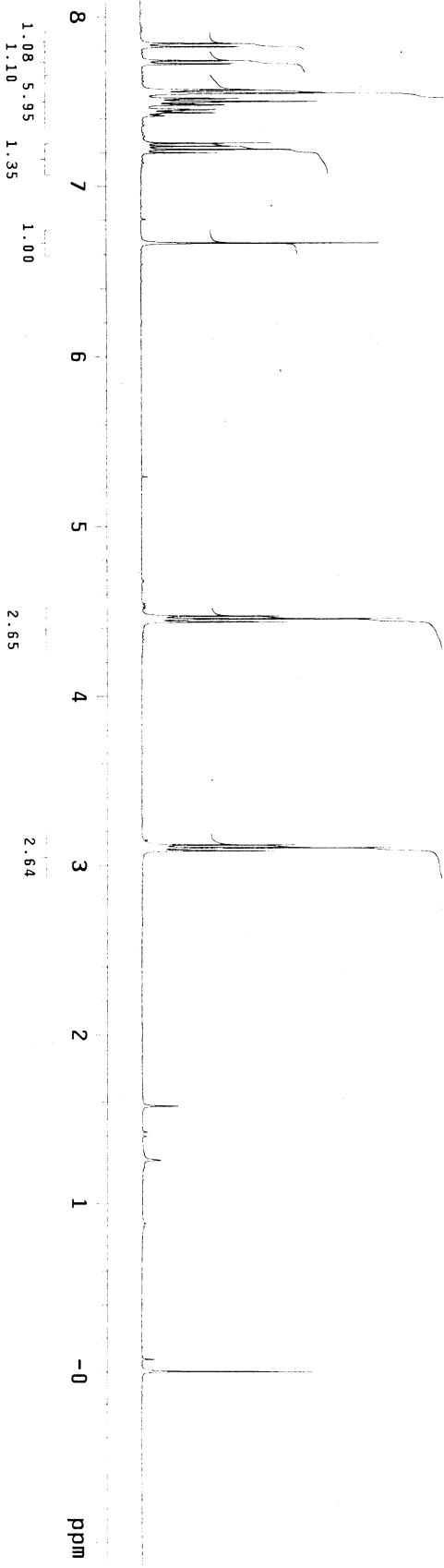
Total time 23 min, 5 sec



Automation directory: /home/walkup/vnmrjsys/data/auto_2007.06.11_26
File : s_20070611_002.data/cdcl3_02.fid
Sample id : s_20070611_002

Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_02
VNMR3-400 "NMR400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
With 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8656747 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_10
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdc13
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

800 repetitions

OBSERVE Cl3, 125.6746053 MHz

DECUPLE H1, 499.8016822 MHz

power 39 dB

continuously on

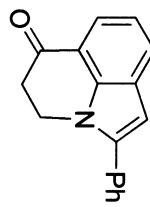
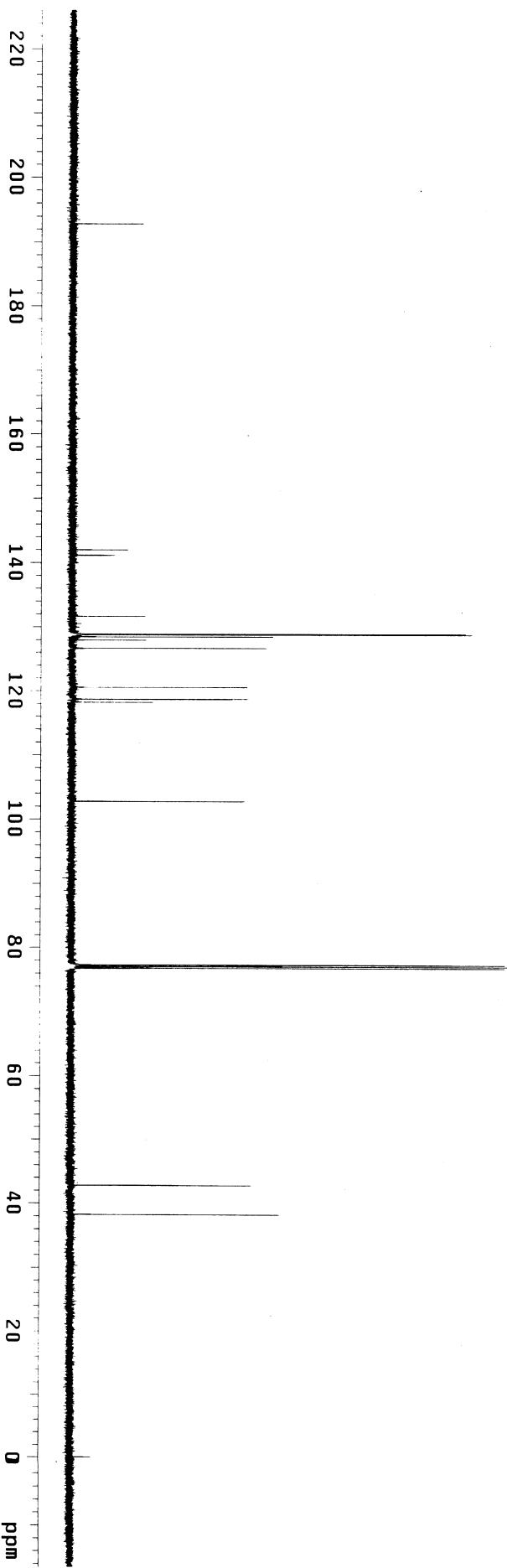
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

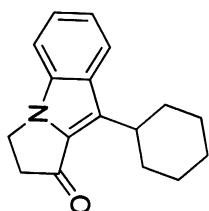
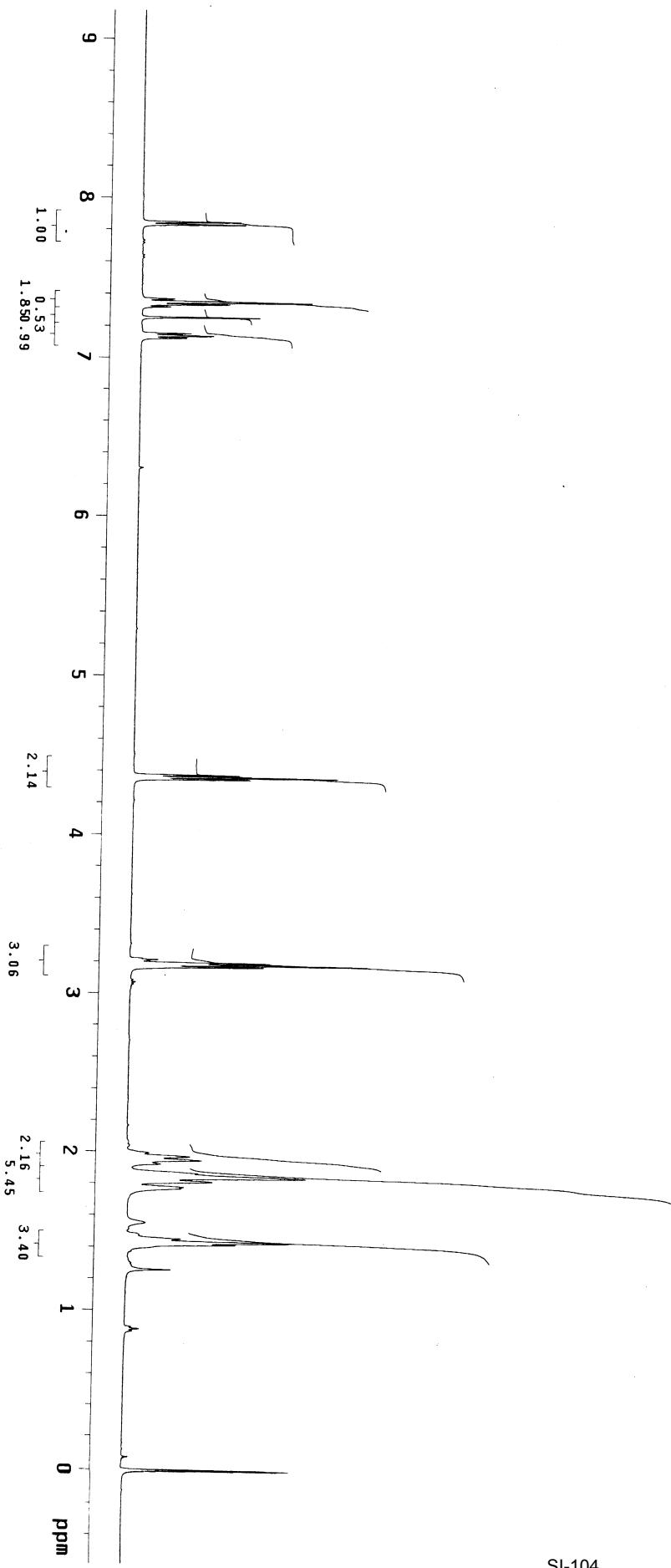
Total time 30 min, 47 sec



File: hxg-3-158-2H

Pulse Sequence: s2pul
Solvent: cdc13
Temp. 25.0 C / 298.1 K
Operator: walkup
File: hgx-3-158-2H
INOVA-500 "redjacket"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 80.2.8 Hz
 8 repetitions
 OBSERVE H1 499.7991854 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT 65536
 Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_13

File : exp

Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: ccl4:13

Temp: 25.0 C / 298.1 K

Operator: walkup

VNMRS-500 "vnmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3048.8 Hz

384 repetitions

OBSERVE C13, 125.6746039 MHz

DECOPPLE H1, 499.8016822 MHz

Power 3.9 dB

continuously on

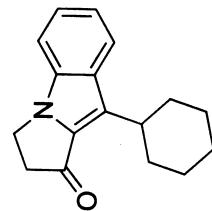
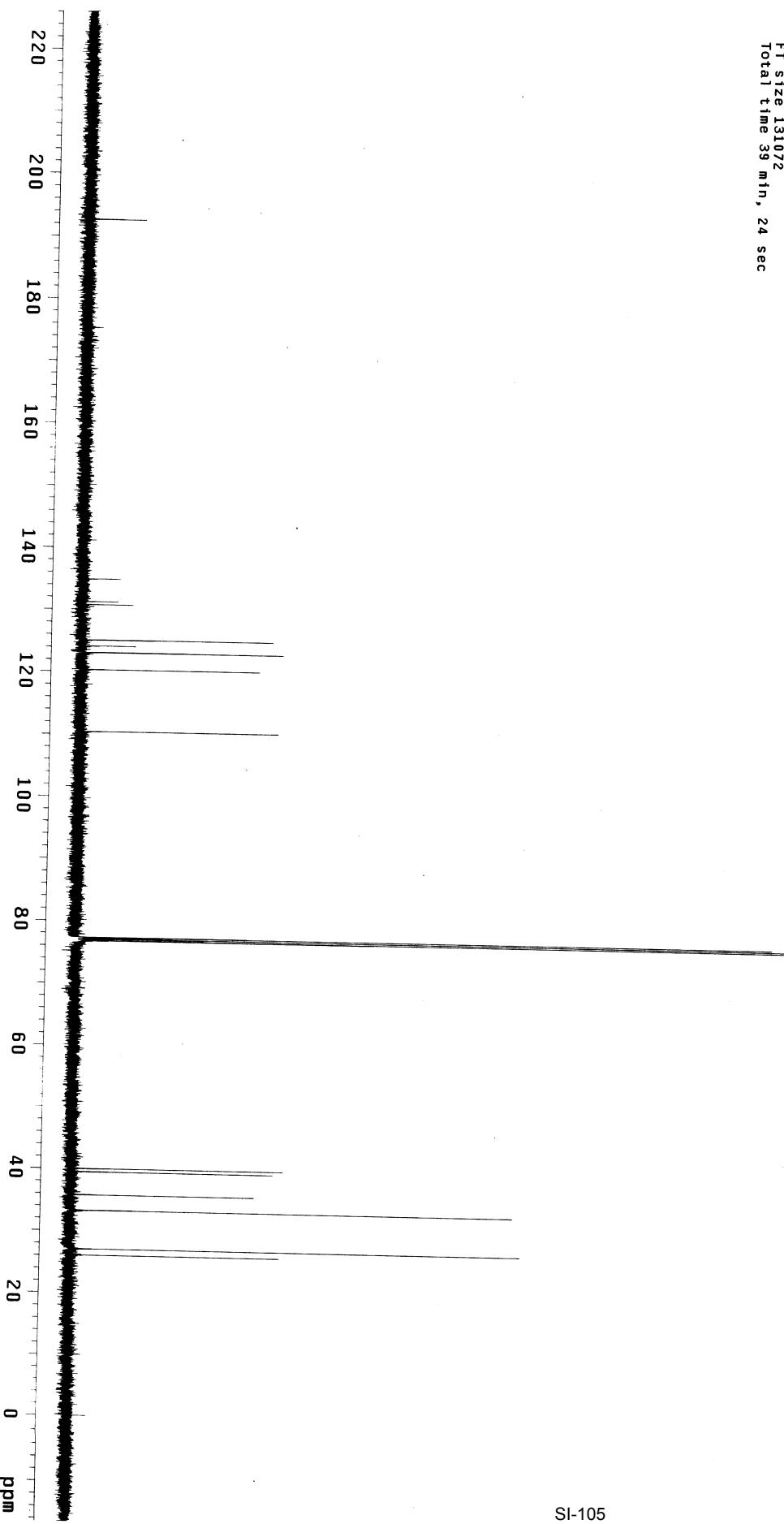
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 39 min, 24 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_14
File : s_20070611_001/data/cdc13_02.fid
Sample id : s_20070611_01

Pulse Sequence: s2pu1

Solvent: cdc13

Temp: 25.0 C / 298.1 K

Operator: walkup

File: cdc13_02

VNMR-400 "NMR400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.049 sec

Width 64.0.3 Hz

8 repetitions

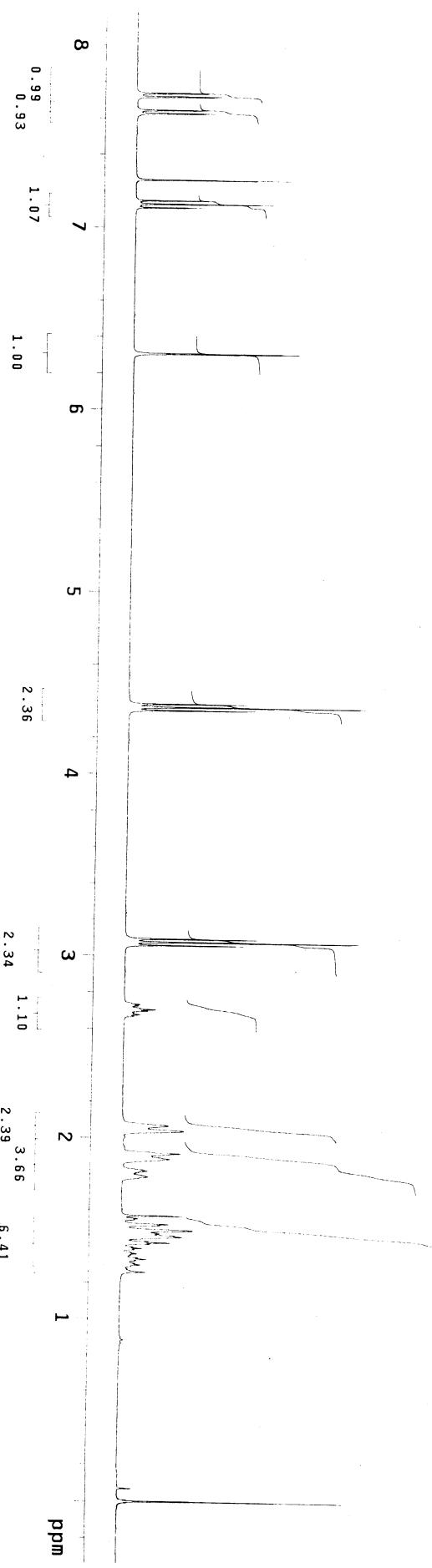
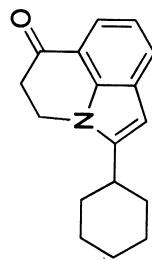
OBSERVE H1 399.8656733 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min, 30 sec



Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_08

File : exp

Sample id : tmpstudy

Pulse Sequence: s2pu1

Solvent: cdc13

Temp. 25.0 C / 298.1 K

Operator: walkup

VNMR-500 "nmr500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 3048.8 Hz

2368 repetitions

OBSERVE C13, 125.676039 MHz

DECOPPLE H1, 499.8016822 MHz

Power 39 dB

continuously on

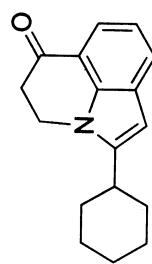
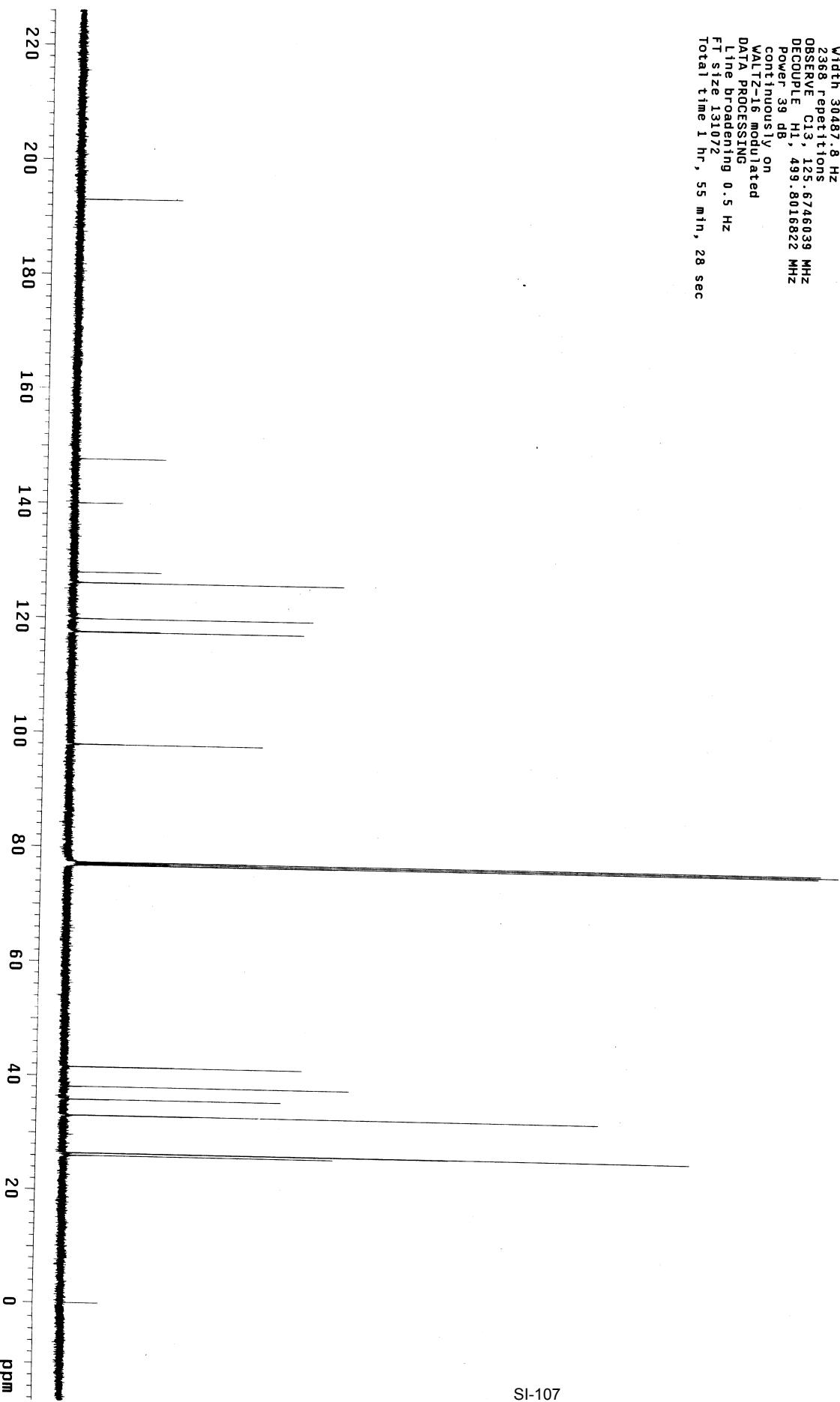
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 1 hr, 55 min, 28 sec



File: hxg-4-13H

Pulse Sequence: s2pul1

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: walkup

File: hxg-4-13H

JOVA-500 "redjacket"

Relax. delay 1.000 sec

Pulse 45.0 degrees

1cg. time 2.049 sec

Width 8016.8 Hz

Repetitions 1

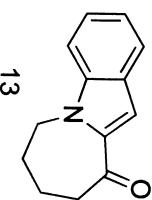
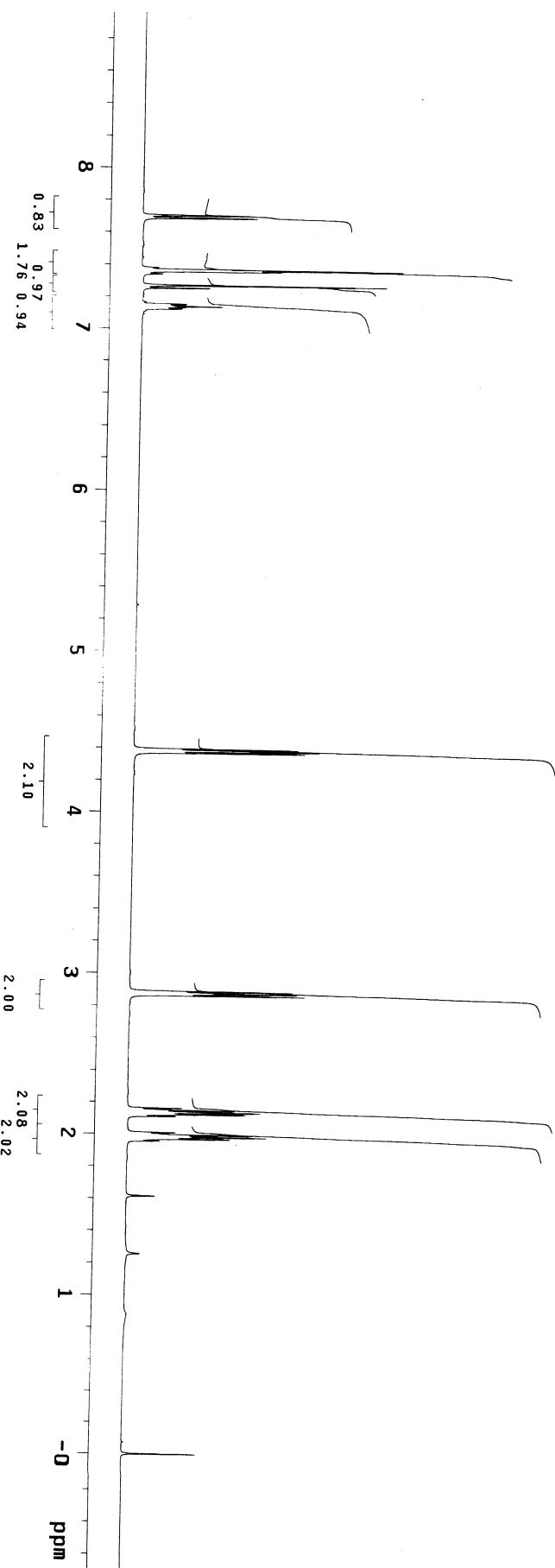
Service HI 499.7991866 MHz

TA PROCESSING

Line broadening 0.2 Hz

Size 65536

Total time 0 min, 30 sec

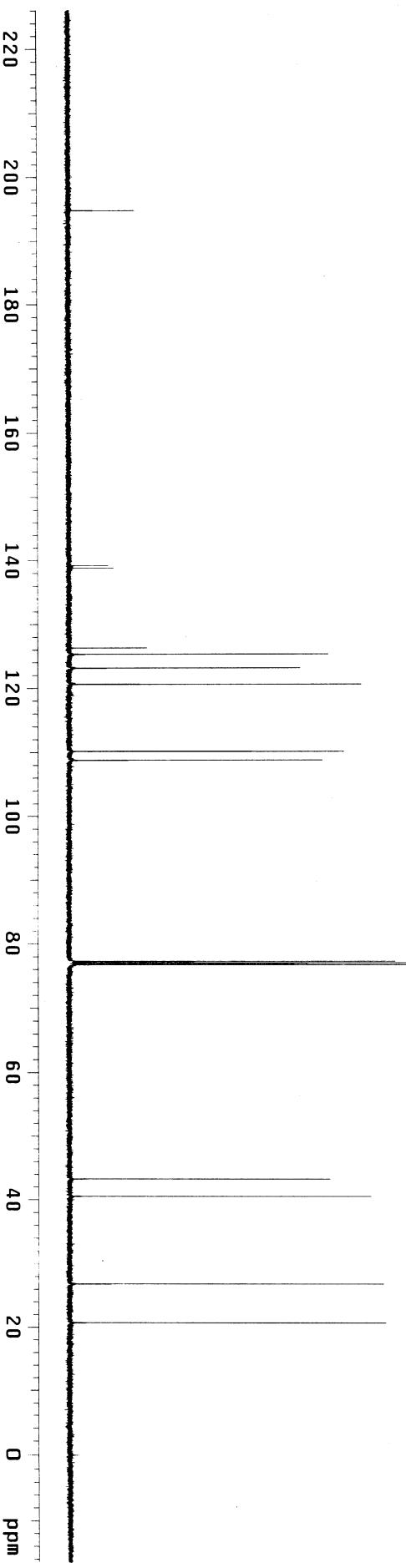
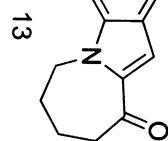


Automation directory: /home/walkup/vnmrjsys/data/auto_2007.06.25_14
File : exp
Sample Id : tmpstudy

Pulse Sequence: s2pu1

Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: walkup
VNMR-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30.87.8 Hz
640 repetitions
OBSERVE C13, 125.6746072 MHz
DECOUPLE H1, 499.8016822 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec

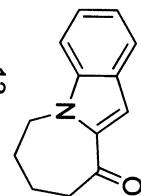
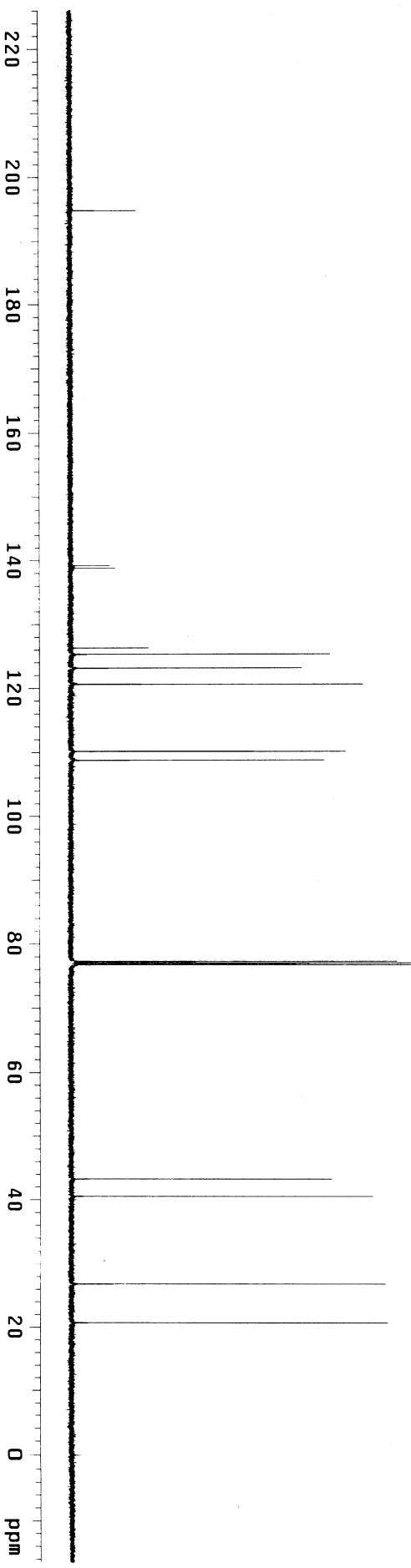


Automation directory: /home/walkup/vnmrSys/data/auto_2007.06.25_14
File : exp

Sample id : tmpstudy

Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: walkup

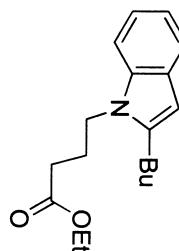
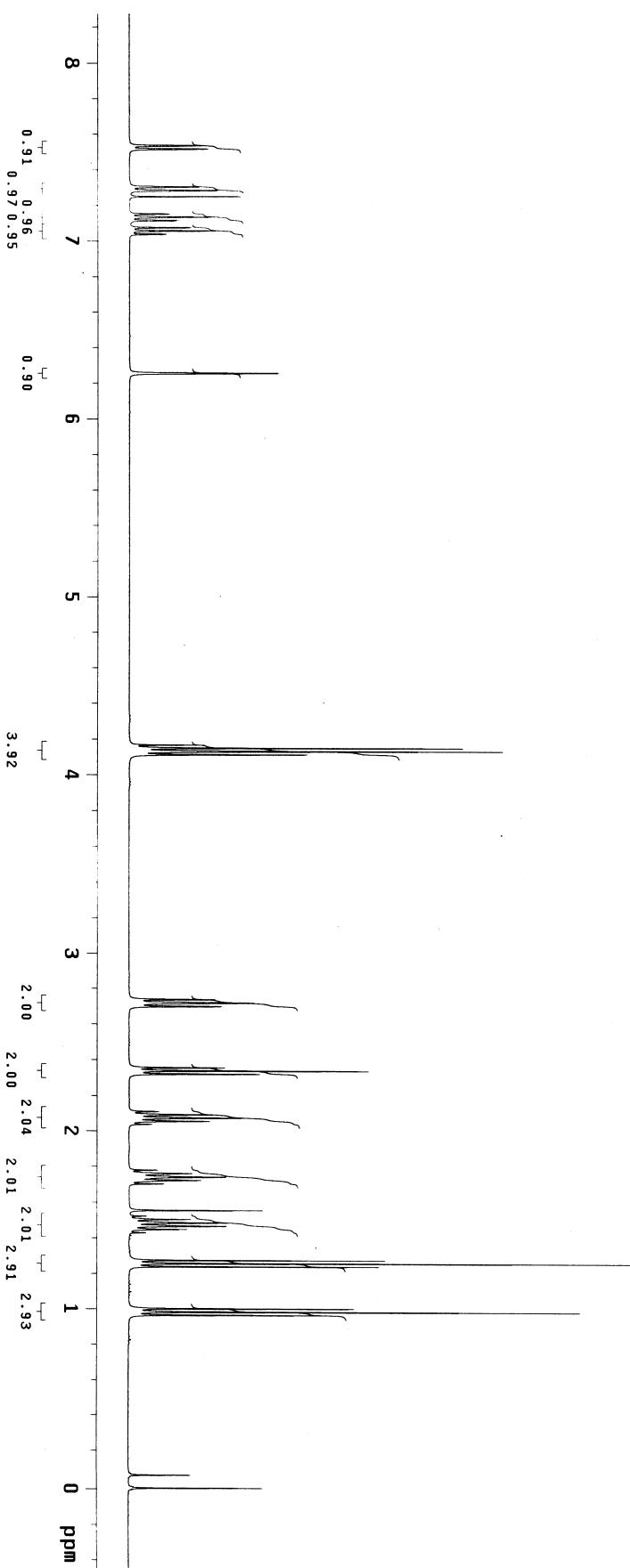
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
640 repetitions
OBSEERVE C13, 125.67416072 MHz
DECOPLE H1, 499.8016822 MHz
Power 39 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec



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SI-110

expt		SAMPLE		SPECIAL		25.3	
date	Aug 31 2007	temp		not use			
solvent	cdcl3	gain		0.0			
file	home/walkup/	spin		0.0			
nmr 400/walkup/data		hst		0.0			
/Zhang/Guorao1/k3~		pw90		13.40			
77-1b.fid		alfa		6.60			
ACQUISITION		FLAGS					
sw	6410.3	11					
at	2.049	15					
np	26264	dp					
fb	4000	hs					
bs	8						
d1	1.000	1b					
nt	8	fn					
ct	8	sp					
TRANSMITTER		PROCESSING		n			
th	HI	wp					
strq	399.866	r ^f 1					
tof	399.6	r ^f 2					
tpwr	59	rp					
pw	6.700	1p					
DECOUPLER		DISPLAY		-182.805.			
dn	C13	wc					
dof	0	sc					
dm	nnn	vs					
dim	c	th					
djw	34	ai					
dmf	29412	cdc					
		ph					



expt Carbon

SAMPLE	31	2007	temp	25.0
date			gain	30
solvent	cdcl ₃		not used	
file	/home/walkup/	spin	0.008	
vrms/s	0.02	hst	15.300	
01/data/cdc13_02.f~	0.02	pmso	10.000	
id		alfa		
ACQUISITION	30.87.8	i1	n	
sw		in	n	
at	1.300	dp	y	
np	79298	hs	nn	
fb	17000			
bs	64	1b	0.50	
d1	1.000	fn	not used	
nt	3000			
ct	64	sp	-2001.2	
TRANSMITTER			30457.3	
tn	C13	rf1	11758.6	
sfrq	125.687	rfp	9676.9	
tofr	1255.2	rp	-56.9	
tpwr	49	lp	-264.0	
pw	7.650		PLOT	
DECOUPLER		wc	250	
dn	H1	sc	0	
dof	0	vs	26406	
dm	yyy	th	14	
dmm	w	a1	cdc	
dpwr	39		ph	
	12200			

