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Supporting Information

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

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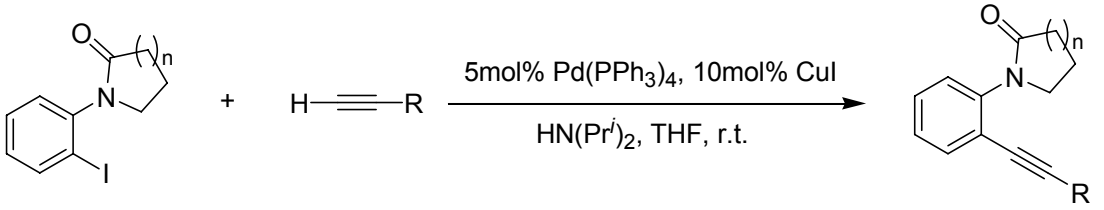
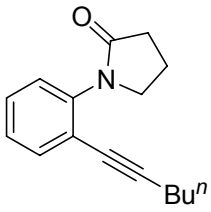
General: Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous tetrahydrofuran and anhydrous dichloromethane in Pure-Pac™ from Aldrich were used directly without further treatment. Commercially 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). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian 500 MHz Unity plus spectrometer and a Varian 400 MHz spectrometer using residue solvent peaks as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Waters micromass ZQ detector using 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 Let.*, **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

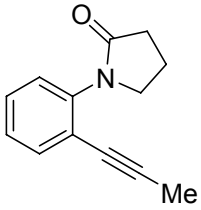


To a solution of the lactam in THF were added sequentially the alkyne (2.0~3.0 eq.), a catalyst [$Pd(PPh_3)_4$ or $PdCl_2(PPh_3)_2$, 5 mol %], CuI (10 mol %), and $HN(iPr)_2$ (4 eq.) at room temperature. The resulting mixture was stirred at room temperature for the time indicated. After all the lactam 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).



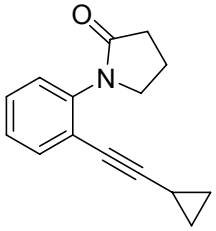
3

Compound **3** was isolated in 97% yield following the general procedure A using $Pd(PPh_3)_4$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2.5 h. ^{1}H 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 $[C_{16}H_{19}NONa]^+$ 264.1; Found: 263.9.



7a

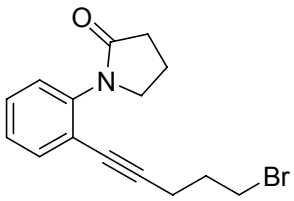
Compound **7a** was isolated in 70% yield following the general procedure A using $Pd(PPh_3)_4$ as catalyst and excess of propyne. Reaction time: 12 h. ^{1}H NMR (500MHz, $CDCl_3$) δ: 7.46 (dd, 1H, *J* = 1.5, 8.0 Hz), 7.33~7.21 (m, 3H), 3.87 (t, 2H, *J* = 7.0 Hz), 2.57 (t, 2H, *J* = 7.0 Hz), 2.18 (quintet, 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 $[C_{13}H_{13}NONa]^+$: 222.0; Found: 221.8.



7b

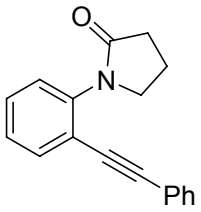
Compound **7b** was isolated in 85% yield following the general procedure A using $Pd(PPh_3)_4$ as catalyst and 2 eq. of ethynylcyclopropane. Reaction time: 3 h. ^{1}H NMR

(400MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{15}H_{15}NONa]^+$ 248.1; Found: 248.1.



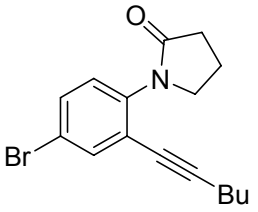
7c

Compound **7c** was isolated in quantitative yield following the general procedure A using $Pd(PPh_3)_4$ as catalyst and 3 eq. of 5-bromo-1-pentyne. Reaction time: 3 h. ^1H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{15}H_{16}BrNONa]^+$ 328.0; Found: 328.0.



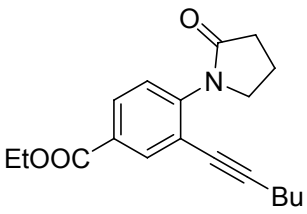
7e

Compound **7e** was isolated in 71% yield following the general procedure A using $Pd(PPh_3)_2Cl_2$ as catalyst and 2 eq. of phenylacetylene. Reaction time: 4h. ^1H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{18}H_{15}NONa]^+$ 284.1; Found: 283.9.



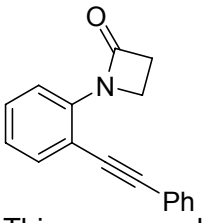
7l

Compound **7l** was isolated in 77% yield following the general procedure A using $Pd(PPh_3)_2Cl_2$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2h. ^{1}H 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 $[C_{16}H_{18}BrNONa]^+$ 342.1; Found: 341.8.

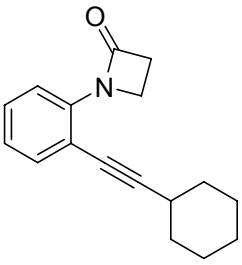


7m

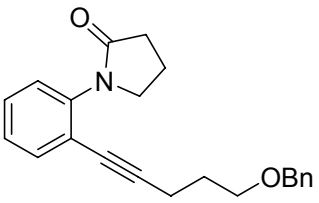
Compound **7m** was isolated in 80% yield following the general procedure A using $Pd(PPh_3)_2Cl_2$ as catalyst and 2 eq. of 1-hexyne. Reaction time: 2h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 8.13 (d, 1H, *J* = 2.0 Hz), 7.96 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.37 (d, 1H, *J* = 8.4 Hz), 4.37 (q, 2H, *J* = 7.2 Hz), 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 $[C_{19}H_{23}NO_3Na]^+$ 336.2; Found: 335.8.



This compound was isolated in quantitative yield following the general procedure A using $Pd(PPh_3)_4$ 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 $[C_{17}H_{13}NONa]^+$ 270.1; Found: 270.3.



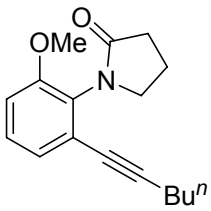
This compound was isolated in 75% yield following the general procedure A using $Pd(PPh_3)_4$ as catalyst and 2 eq. of cyclohexylacetylene. Reaction time: 3 h. ^1H NMR (400MHz, $CDCl_3$) δ: 8.01 (d, 1H, *J* = 8.0 Hz), 7.49 (d, 1H, *J* = 8.0 Hz), 7.22 (t, 1H, *J* = 8.0 Hz), 6.99 (t, 1H, *J* = 8.0 Hz), 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 $[C_{17}H_{19}NONa]^+$ 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 $Pd(PPh_3)_2Cl_2$ (11 mg, 0.015 mmol) and CuI (6 mg, 0.03 mmol). The resulting mixture was heated to 70°C, and the reaction

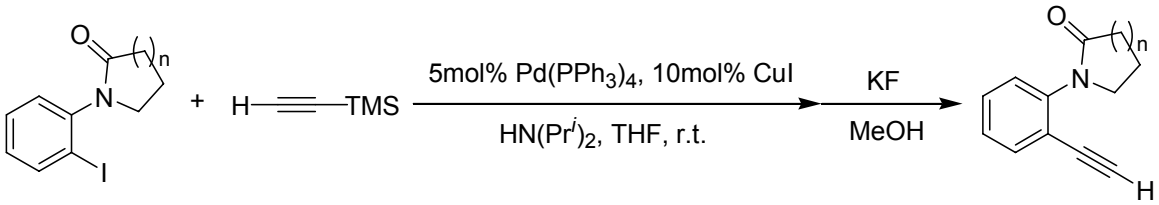
finished in 30 min. The resulting mixture was concentrated, and the residue was purified via silica gel flash column chromatography to give compound **7d** in 96% yield. ^{1}H NMR (500MHz, $CDCl_3$) δ: 7.43 (dd, 1H, *J* = 7.7, 1.3 Hz), 7.34~7.19 (m, 8H), 4.52 (s, 2H), 3.83 (t, 2H, *J* = 7.0 Hz), 3.61 (t, 2H, *J* = 6.3 Hz), 2.56~2.52 (m, 4H), 2.17~2.11 (m, 2H), 1.92~1.87 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.6, 140.0, 138.3, 133.3, 128.5, 128.3, 127.5, 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 $[C_{22}H_{33}NO_2Na]^+$ 356.2; Found: 356.1.



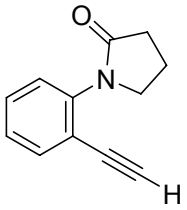
7k

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 $Pd(PPh_3)_2Cl_2$ (13 mg, 0.019 mmol) and CuI (12 mg, 0.063 mmol) under N_2. 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. ^{1}H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{17}H_{21}NO_2Na]^+$ 294.2; Found: 294.2.

General procedure B: preparation of ethynyl lactams

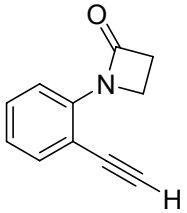


To a solution of the lactam and (trimethylsilyl)acetylene (1.5 eq.) in THF were added $PdCl_2(PPh_3)_2$ (0.05 eq.), CuI (0.1 eq.), and $HN(iPr)_2$ (5.0 eq.). The resulting mixture was stirred at room temperature for 2.5 h. When the lactam was consumed, the mixture was concentrated under vacuum. The resulting residue was dissolved in MeOH and treated with $KF{\cdot}H_2O$ (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).



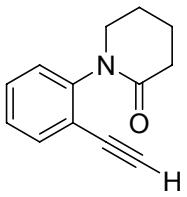
7j

Compound **7j** was isolated in 73% yield following the general procedure B. ^{1}H 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 $[C_{12}H_{11}NONa]^+$ 208.1; Found: 208.0.



10

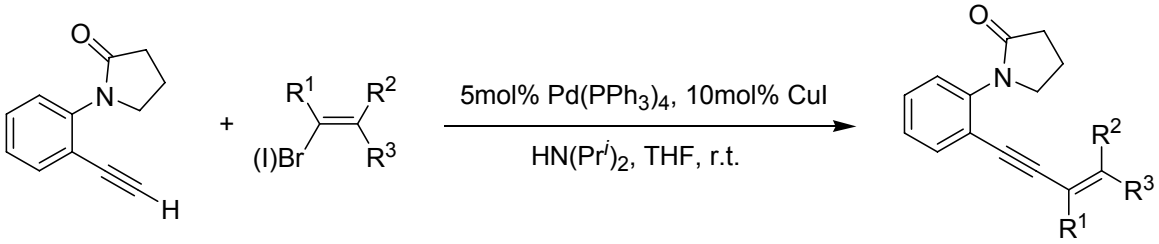
Compound **9** was isolated in 80% yield following the general procedure B. ^{1}H NMR (400MHz, $CDCl_3$) δ: 8.05 (d, 1H, *J* = 8.4 Hz), 7.44 (d, 1H, *J* = 7.6 Hz), 7.32 (t, 1H, *J* = 7.2 Hz), 7.04 (t, 1H, *J* = 7.6 Hz), 4.17 (t, 2H, *J* = 4.8 Hz), 3.28 (s, 1H), 3.11 (t, 2H, *J* = 4.8 Hz); ^{13}C NMR (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 $[C_{11}H_9NONa]^+$ 194.1; Found 194.0.



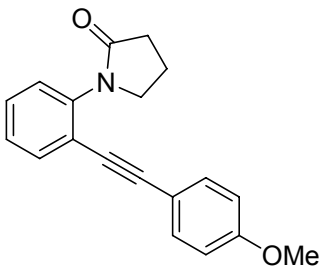
12

Compound **12** was isolated in 53% yield following the general procedure B. ^{1}H 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 $[C_{13}H_{13}NONa]^+$:222.0; Found: 221.7.

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



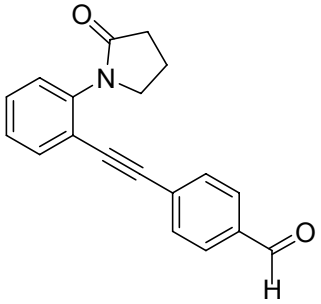
To a solution of alkyne **7j** (1.0 eq.) and an alkenyl/aryl halide (2.0eq.) were added a catalyst [$Pd(PPh_3)_4$ or $PdCl_2(PPh_3)_2$, 5 mol %), CuI (10 mol %), and $HN(iPr)_2$ (3.0~5.0 eq.). The resulting mixture was stirred at room temperature for the indicated time. After **7j** was consumed, the reaction mixture was concentrated under vacuum. The residue was purified through silica gel flash column chromatography with hexanes/ethyl acetate (1:1).



7f

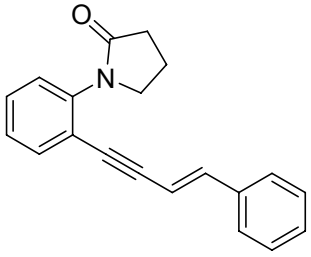
Compound **7f** was isolated in 97% yield following the general procedure C using $Pd(PPh_3)_4$ as catalyst and 2 eq. of 4-iodoanisole. Reaction time: 5 h. ^{1}H NMR (400MHz,

$CDCl_3$) 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 $[C_{19}H_{17}NONa]^+$ 314.1; Found: 314.1.



7g

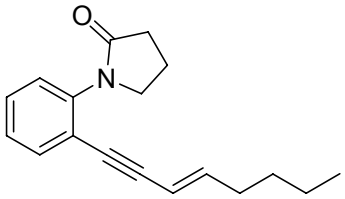
Compound **7g** was isolated in 80% yield following the general procedure C using $Pd(PPh_3)_2Cl_2$ as catalyst and 2 eq. of 4-bromobenzaldehyde. Reaction time: 2.5 h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 10.03 (s, 1H), 7.88 (d, 2H, *J* = 8.8 Hz), 7.65~7.62 (m, 3H), 7.46~7.43 (m, 1H), 7.36~7.33 (m, 2H), 3.95 (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 $[C_{19}H_{15}NONa]^+$ 312.1; Found: 311.9.



7h

Compound **7h** was isolated in 97% yield following the general procedure C using $Pd(PPh_3)_4$ as catalyst and 2 eq. of β-bromostyrene. Reaction time: 4h. ^{1}H 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 $[C_{20}H_{17}NONa]^+$ 310.1; Found: 310.0.

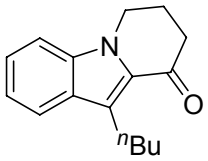


7i

Compound **7i** was isolated in 99% yield following the general procedure C using $Pd(PPh_3)_4$ as catalyst and 2 eq. of 1-iodo-1-hexene. Reaction time: 2.5 h. ^1H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{13}H_{13}NONa]^+$ 290.1; Found: 290.3.

General procedure D: $PtCl_4$/$PtCl_2$-catalyzed reaction of lactams

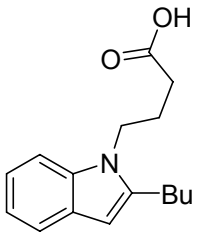
To a 0.01 M solution of the lactam in anhydrous 1,2-dichloroethane was added 10 mol % of $PtCl_4$ or $PtCl_2$ (if specified) under an atmosphere of O_2. The resulting mixture was refluxing for the indicated time. Upon the completion of the reaction, the solvent was removed under vacuum, and the residue was purified via silica gel flash column chromatography (eluent: hexanes/ethyl acetate = 3:1) to yield the desired product.



4

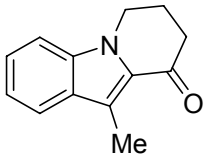
Compound **4** was isolated in 83% yield following the general procedure D. Reaction time: 1 h. ^1H NMR (400MHz, $CDCl_3$) δ: 7.73 (dt, 1H, *J* = 8.0, 0.8 Hz), 7.40~7.30 (m, 2H), 7.14 (dd, 1H, *J* = 6.8, 1.2 Hz), 4.22 (t, 2H, *J* = 6.0 Hz), 3.18 (t, 2H, *J* = 8.0 Hz), 2.72 (t, 2H, *J* = 6.4 Hz), 2.39~2.33 (m, 2H), 1.68~1.61 (m, 2H), 1.46~1.37 (m, 2H), 0.93 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{16}H_{19}NONa]^+$ 264.1; Found: 264.3.



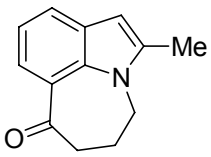
6

Compound **6** was isolated as the side product during the reaction of **3**. ^1H NMR (500MHz, $CDCl_3$) 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); ^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{16}H_{21}NONa]^+$ 282.2; Found: 282.0.



8a

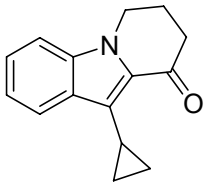
Compound **8a** was isolated in 83% yield following the general procedure D. Reaction time: 1.5 h. ^1H NMR (400MHz, $CDCl_3$) δ: 7.70 (d, 1H, *J* = 8.4 Hz), 7.40~7.25 (m, 2H), 7.15 (t, 1H, *J* = 6.8 Hz), 4.19 (t, 2H, *J* = 5.6 Hz), 2.72 (t, 2H, *J* = 6.4 Hz), 2.68 (s, 3H), 2.38~2.32 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{13}H_{13}NONa]^+$ 222.0; Found: 221.7.



9a

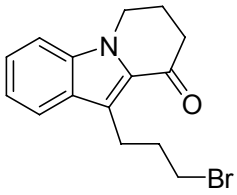
Compound **9a** was isolated as the side product during the reaction of **7a**. ^1H NMR (500MHz, $CDCl_3$) δ: 7.95 (dd, 1H, *J* = 1.0, 7.5 Hz), 7.72 (dd, 1H, *J* = 1.0, 7.5 Hz), 7.13 (t,

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 $[C_{13}H_{13}NONa]^+$ 222.1; Found: 222.0.



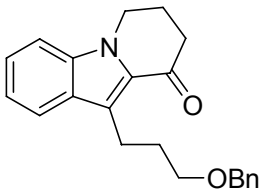
8b

Compound **8b** was isolated in 71% yield following the general procedure D. Reaction time: 14 h. ^{1}H 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 $[C_{15}H_{15}NONa]^+$ 248.1; Found: 247.8.



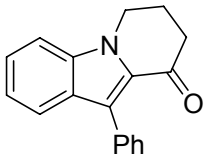
8c

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. ^{1}H 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 $[C15H16BrNONa]^+$ 328.0; Found: 328.2; for $[C15H16ClrNONa]^+$ 284.1, Found: 283.8.



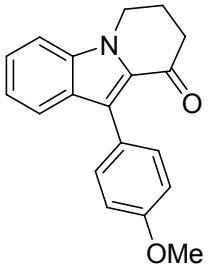
8d

Compound **8d** was isolated in 81% yield following the general procedure D. Reaction time: 2 h. ^{1}H 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 $[C_{22}H_{33}NO_2Na]^+$ 356.2; Found: 356.0.



8e

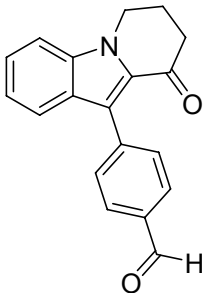
Compound **8e** was isolated in 64% yield following the general procedure D. Reaction time: 23 h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 7.71 (d, 1H, *J* = 8.0 Hz), 7.60 (d, 2H, *J* = 7.2 Hz), 7.48~7.34 (m, 5H), 7.17 (dd, 1H, *J* = 1.2, 6.0 Hz), 4.32 (t, 2H, *J* = 5.6 Hz), 2.76 (t, 2H, *J* = 6.0 Hz), 2.48~2.38 (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 $[C_{18}H_{15}NONa]^+$ 284.1; Found: 284.3.



8f

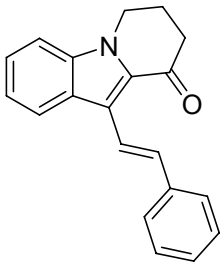
Compound **8f** was isolated in 73% yield following the general procedure D. Reaction time: 11 h. ^{1}H NMR (500MHz, $CDCl_3$) δ: 7.71 (d, 1H, *J* = 8.0 Hz), 7.56~7.53 (m,2H),

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 $[C_{19}H_{17}NONa]^+$ 314.1; Found: 314.0.



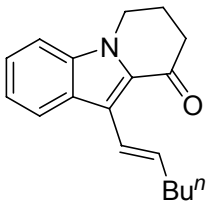
8g

Compound **8g** was isolated in 70% yield following the general procedure D. Reaction time: 34 h. ^{1}H 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 $[C_{19}H_{15}NONa]^+$ 312.1; Found: 311.9.



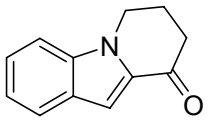
8h

Compound **8h** was isolated in 60% yield following the general procedure D. Reaction time: 14 h. ^{1}H 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 $[C_{20}H_{17}NONa]^+$ 310.1; Found: 310.0.



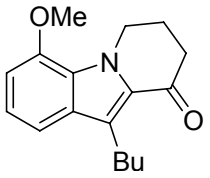
8i

Compound **8i** was isolated in 52% yield following the general procedure D. Reaction time: 48 h. ^{1}H 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 $[C_{18}H_{21}NONa]^+$ 290.2; Found: 290.0.



8j

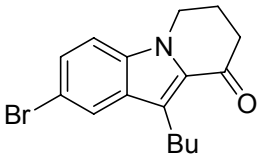
Compound **8j** was isolated in 73% yield using $PtCl_4$ as catalyst and in 95% yield using $PtCl_2$ following the general procedure D. Reaction time: 22 h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 7.72 (d, 1H, *J* = 8.4 Hz), 7.39~7.33 (m, 2H), 7.31 (s, 1H), 7.16 (dd, 1H, *J* = 5.6, 1.6 Hz), 4.23 (t, 2H, *J* = 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 $[C_{12}H_{11}NONa]^+$ 208.1; Found: 208.0.



8k

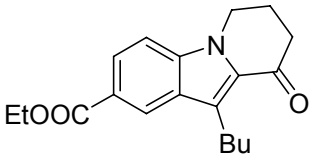
Compound **8k** was isolated in 89% yield following the general procedure D. Reaction time: 1 h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 7.28 (d, 1H, *J* = 8.0 Hz), 7.00 (t, 1H, *J* = 8.0 Hz), 6.71 (t, 1H, *J* = 7.6 Hz), 4.68 (t, 2H, *J* = 6.0 Hz), 3.92 (s, 3H), 3.14 (t, 2H, *J* = 7.6 Hz), 2.66 (t, 2H, *J* = 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 $[C_{17}H_{21}NO_2Na]^+$ 294.2; Found: 294.0.



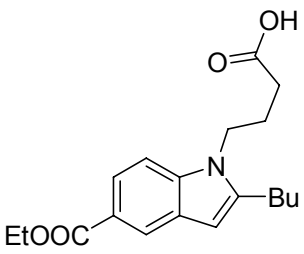
8l

Compound **8l** was isolated in 71% yield following the general procedure D. Reaction time: 1 h. ^1H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{16}H_{18}BrNONa]^+$ 242.1; Found: 241.6.

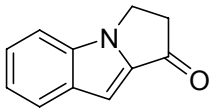


8m

Compound **8m** was isolated in 65% yield following the general procedure D. Reaction time: 1 h. ^1H NMR (400MHz, $CDCl_3$) 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); ^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{19}H_{23}NO_3Na]^+$ 336.2; Found: 335.8.

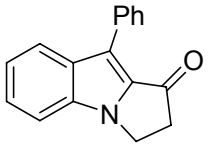


This compound was isolated as the side product during the reaction of **7m**. ^{1}H NMR (400MHz, $CDCl_3$) δ: 8.29 (d, 1H, *J* = 1.6 Hz), 7.86 (dd, 1H, *J* = 8.8, 1.6 Hz), 7.28 (d, 1H, *J* = 8.8 Hz), 6.35 (s, 1H), 4.38 (q, 2H, *J* = 7.2 Hz), 4.16 (t, 2H, *J* = 7.6 Hz), 2.72 (t, 2H, *J* = 7.6 Hz), 2.42 (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); ^{13}C NMR (100 MHz, $CDCl_3$) 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 $[C_{19}H_{23}NO_3Na]^+$ 354.2; Found: 353.9.

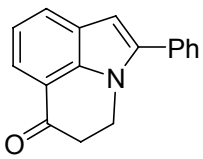


11

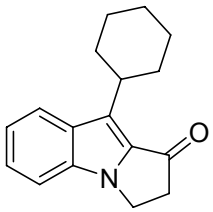
Compound **11** was isolated in 85% yield using $PtCl_4$ as catalyst and in 99% yield using $PtCl_2$ following the general procedure D. Reaction time: 8 h in the case of $PtCl_4$; 24 h in the case of $PtCl_2$. ^{1}H NMR (500MHz, $CDCl_3$) δ: 7.75 (d, 1H, *J* = 8.5 Hz), 7.41 (d, 1H, *J* = 8.5 Hz), 7.35 (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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{11}H_9NONa]^+$ 194.1; Found: 194.0.



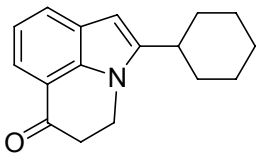
This compound was isolated in 67% yield along with 33% of the Friedel-Crafts acylation product (shown below) following the general procedure D. Reaction time: 4 h. ^{1}H NMR (400MHz, $CDCl_3$) δ: 8.01 (d, 1H, *J* = 8.0 Hz), 7.86 (d, 2H, *J* = 8.0 Hz), 7.48 (t, 2H, *J* = 7.6 Hz), 7.42~7.30 (m, 3H), 7.26~7.18 (m, 1H) 4.38 (t, 2H, *J* = 6.0 Hz), 3.21 (t, 2H, *J* = 6.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{17}H_{13}NONa]^+$ 270.1; Found: 270.1.



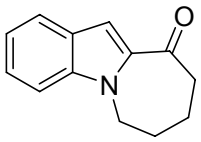
^{1}H NMR (400MHz, $CDCl_3$) δ: 7.83 (dd, 1H, *J* = 6.0, 0.8 Hz), 7.73 (dd, 1H, *J* = 6.4, 0.8 Hz), 7.56~7.42 (m, 5H), 7.22 (t, 1H, *J* = 6.0 Hz), 6.66 (s, 1H), 4.45 (t, 2H, *J* = 5.2 Hz), 3.10 (t, 2H, *J* = 5.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 192.8, 141.9, 141.1, 131.6, 128.8, 128.7, 128.4, 127.9, 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 $[C_{17}H_{13}NONa]^+$ 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.^{1}H NMR (500MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{17}H_{19}NONa]^+$ 276.1; Found: 276.3.

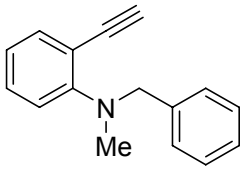


^{1}H NMR (500MHz, $CDCl_3$) δ: 7.72 (d, 1H, *J* = 7.6 Hz), 7.63 (d, 1H, *J* = 7.6 Hz), 7.13 (t, 1H, *J* = 7.6 Hz), 6.31 (s, 1H), 4.36 (t, 2H, *J* = 6.8 Hz), 3.07 (t, 2H, *J* =6.8 Hz), 2.73~2.67 (m, 1H), 2.06~2.03 (m, 2H), 1.91~1.88 (m, 2H), 1.82~1.78 (m, 1H), 1.56~1.32 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{17}H_{19}NONa]^+$ 276.1; Found: 276.1.



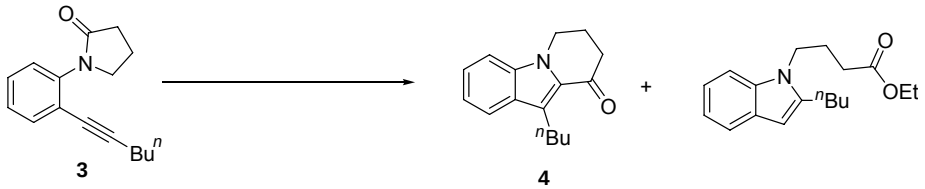
13

Compound **13** was isolated in 45% yield using $PtCl_2$ as catalyst following the general procedure D. Reaction time: 36 h.^{1}H NMR (500MHz, $CDCl_3$) δ: 7.69 (d, 1H, *J* = 8.0 Hz), 7.38~7.25 (m, 1H), 7.26 (d, 1H, *J* = 8.5Hz), 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 $[C_{13}H_{13}NONa]^+$ 222.0; Found: 222.1.



^{1}H NMR (500MHz, $CDCl_3$) 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); ^{13}C NMR (125 MHz, $CDCl_3$) 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 $[C_{16}H_{16}N]^+$ 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: ^{1}H NMR (400MHz, $CDCl_3$) δ: 7.53 (d, 1H, *J* = 7.6 Hz), 7.29 (d, 1H, *J* = 7.6

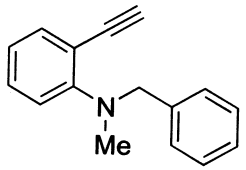
Hz), 7.13 (dt, 1H, *J* = 7.6, 1.2 Hz), 7.06 (dt, 1H, *J* = 7.6, 1.2 Hz), 7.26 (s, 1H), 4.17-4.11 (m, 4H), 2.72 (t, 2H, *J* = 7.6 Hz), 2.34 (t, 2H, *J* = 7.2 Hz), 2.07 (quintet, 2H, *J* = 7.2 Hz), 1.78-1.70 (m, 2H), 1.48 (sextet, 2H, *J* = 7.6 Hz), 1.26 (t, 3H, *J* = 7.2 Hz), 0.98 (t, 3H, *J* = 7.6 Hz); ^{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 $[C_{18}H_{25}NO_2Na]^+$ 310.2; Found: 310.1.

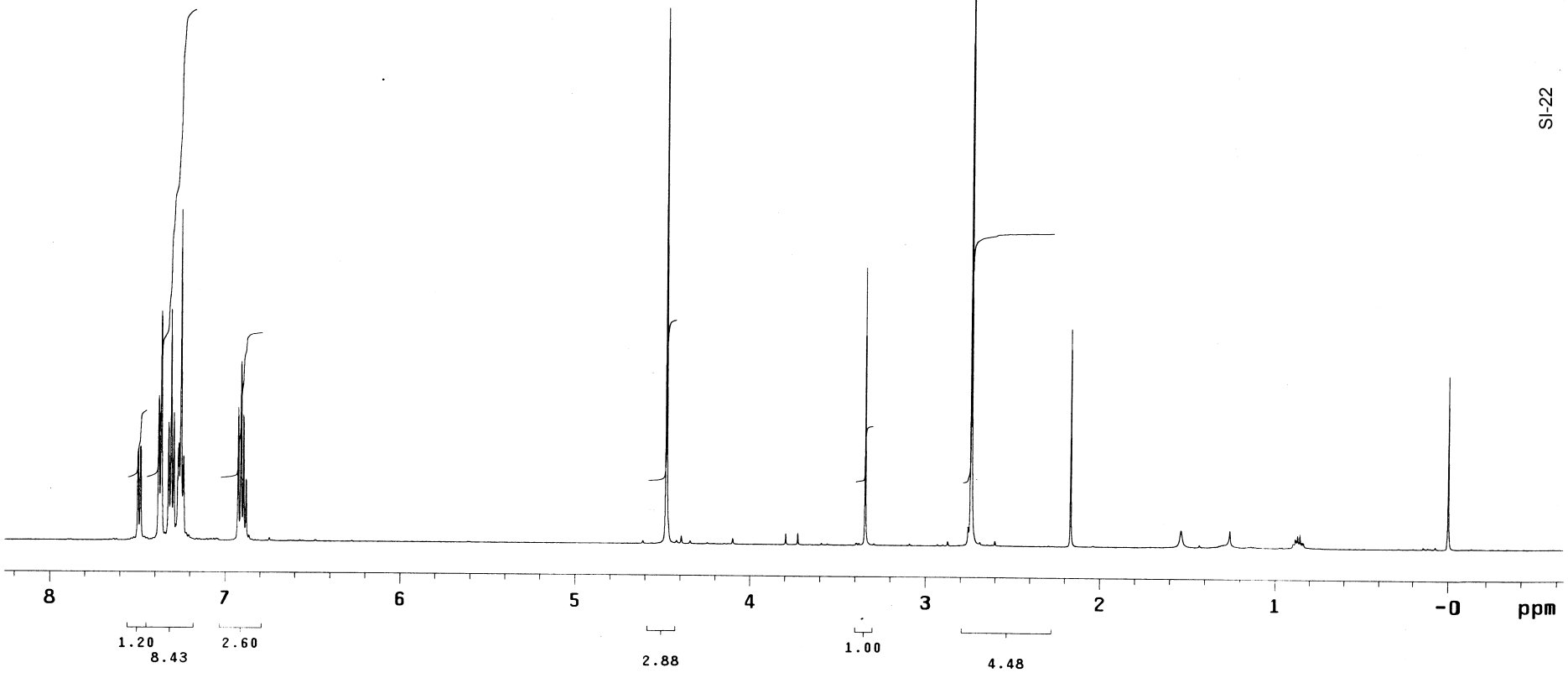
Experiment 2: Compound **3** was used as substrate, and the reaction was run according to the general procedure D except that EtOH was used as solvent. Reaction time: 1h. The ethyl ester was isolated in 68% yield.

File: hxg-gzmade

Pulse Sequence: s2pul
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



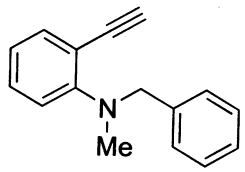


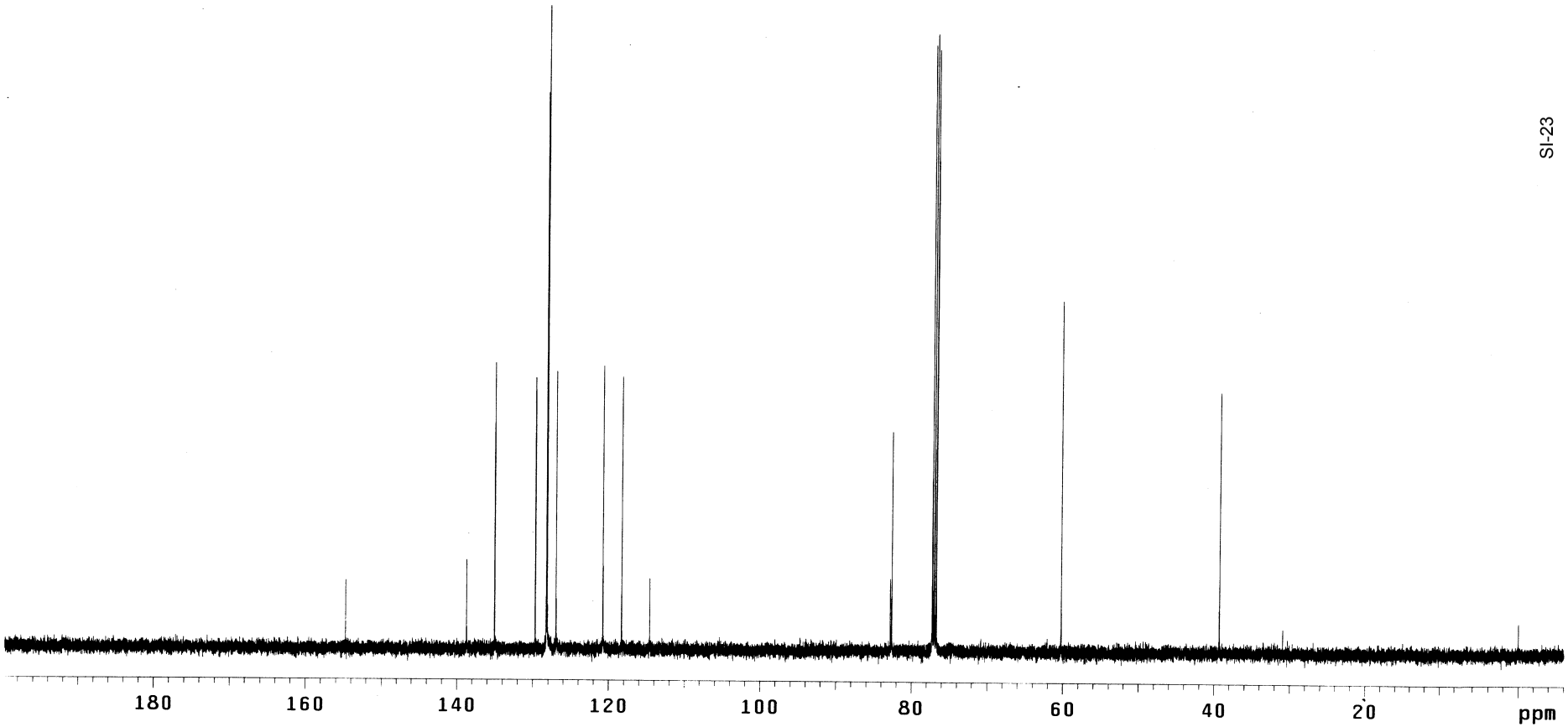
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_11
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
384 repetitions
OBSERVE C13, 125.6746044 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 19 min, 42 sec

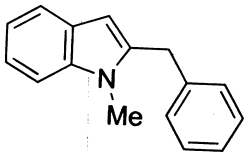




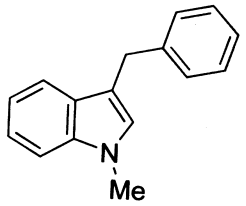
Automation directory: /home/walkup/vnmrsys/data/auto_2007.07.01_07
File : s_20070701_002/data/cdcl3_01.fid
Sample id : s_20070701_002

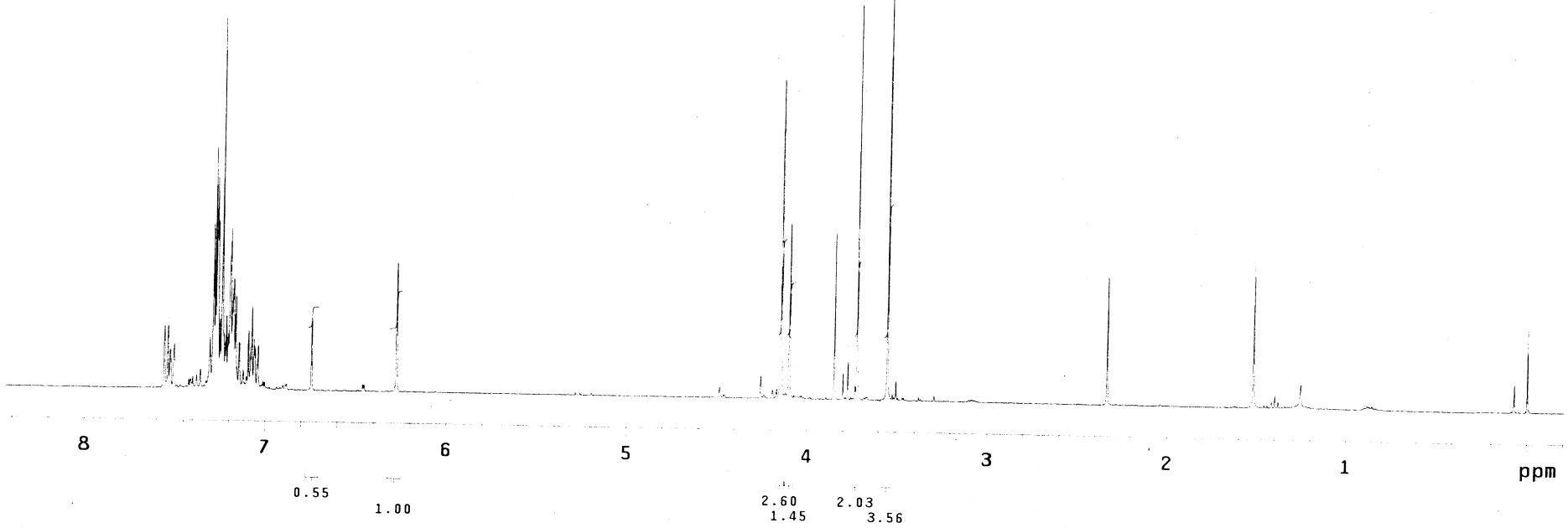
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_01
VNMRS-400 "NMR400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8656793 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



and



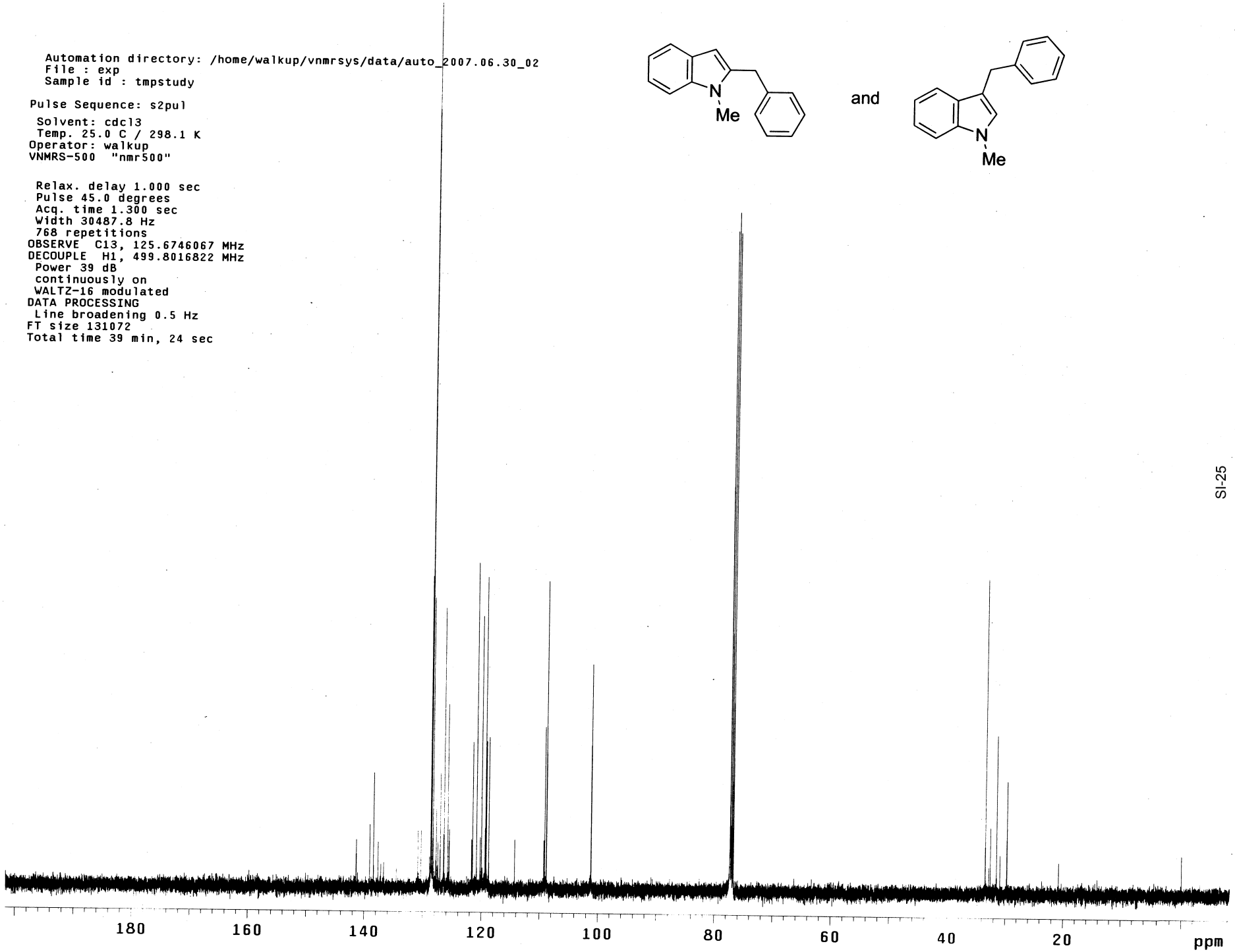


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.30_02
File : exp
Sample id : tmpstudy

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
768 repetitions
OBSERVE C13, 125.6746067 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

and



exp2 Proton

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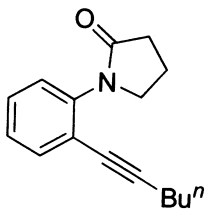
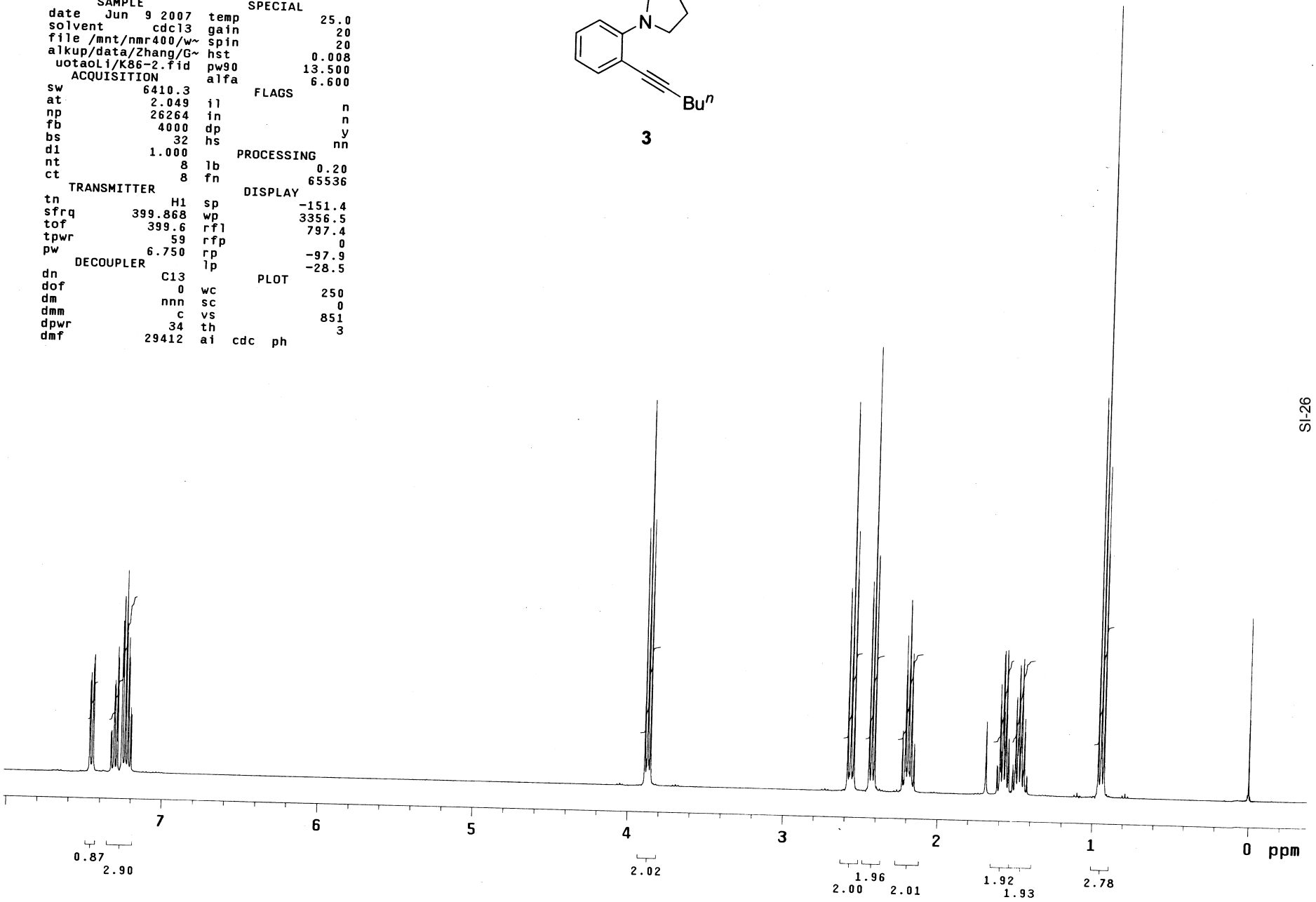
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K86-2.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
di	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-151.4
sfrq	399.868	wp	3356.5
tof	399.6	rfl	797.4
tpwr	59	rfp	0
pw	6.750	rp	-97.9
DECOUPLER		lp	-28.5
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	851
dpwr	34	th	3
dmf	29412	ai cdc ph	

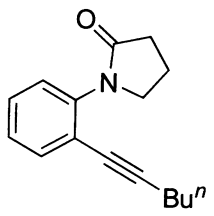


3

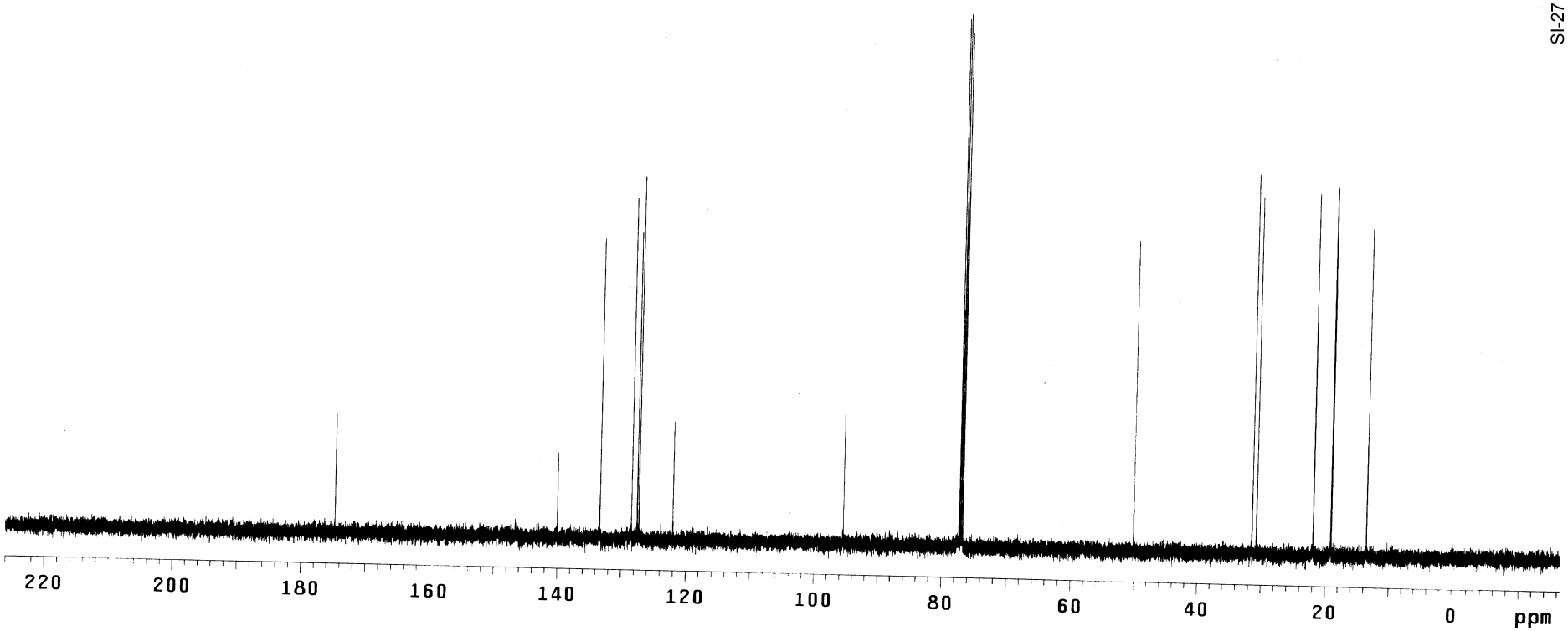


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 9 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	64	in	n
d1	1.000	dp	y
nt	1024	hs	nn
ct	128	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2080.2
tpwr	49	wp	30487.3
pw	7.650	rfl	11757.6
DECOUPLER		rfp	9676.9
dn	H1	rp	-38.6
dof	0	lp	-250.8
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	23094
		th	68
		ai	cdc ph



3

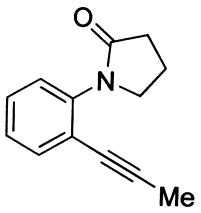


File: hxg-4-25H

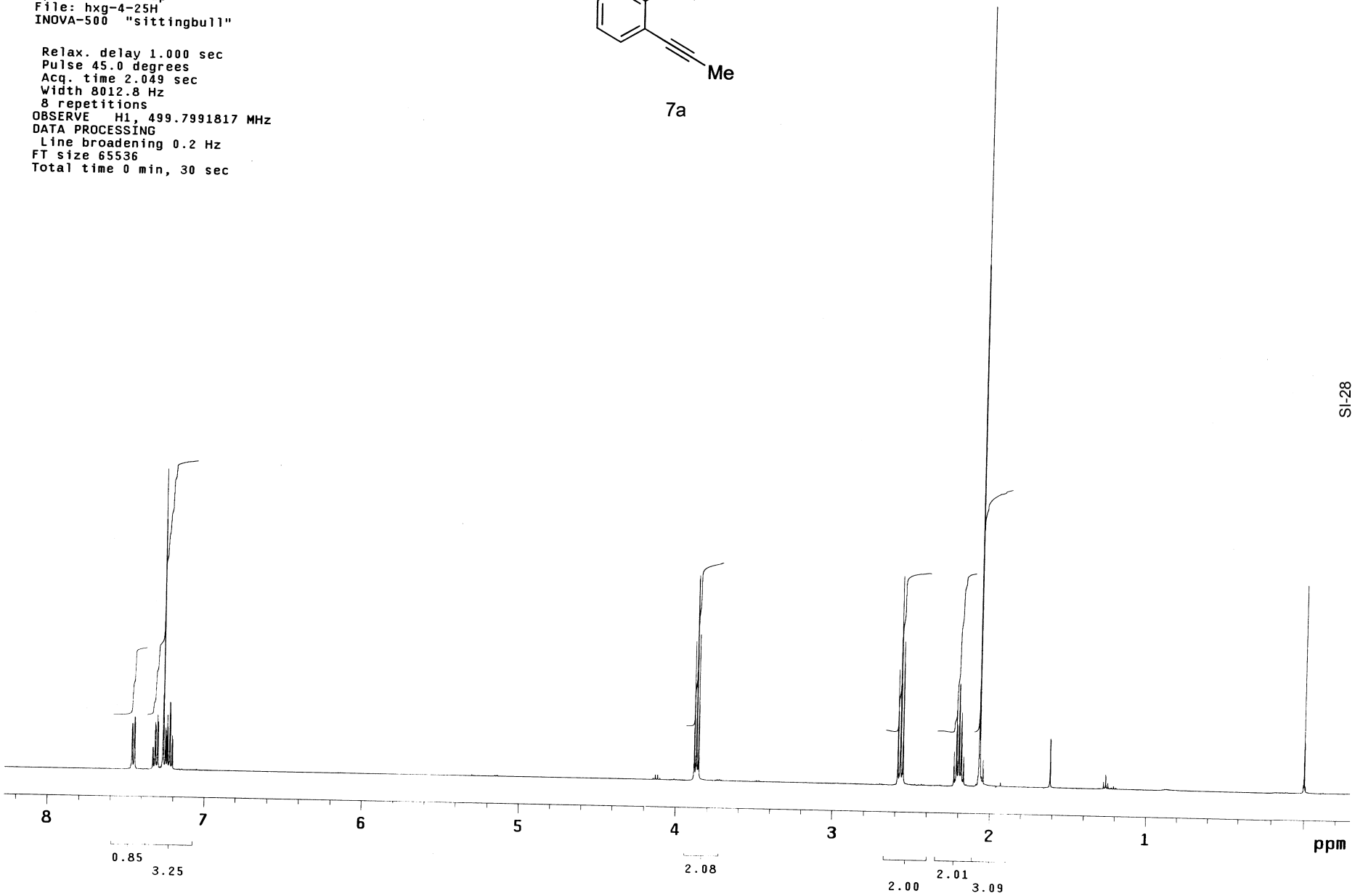
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: hxg-4-25H
INOVA-500 "sittingbull"

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



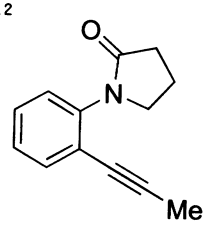
7a



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
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
512 repetitions
OBSERVE C13, 125.6746053 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



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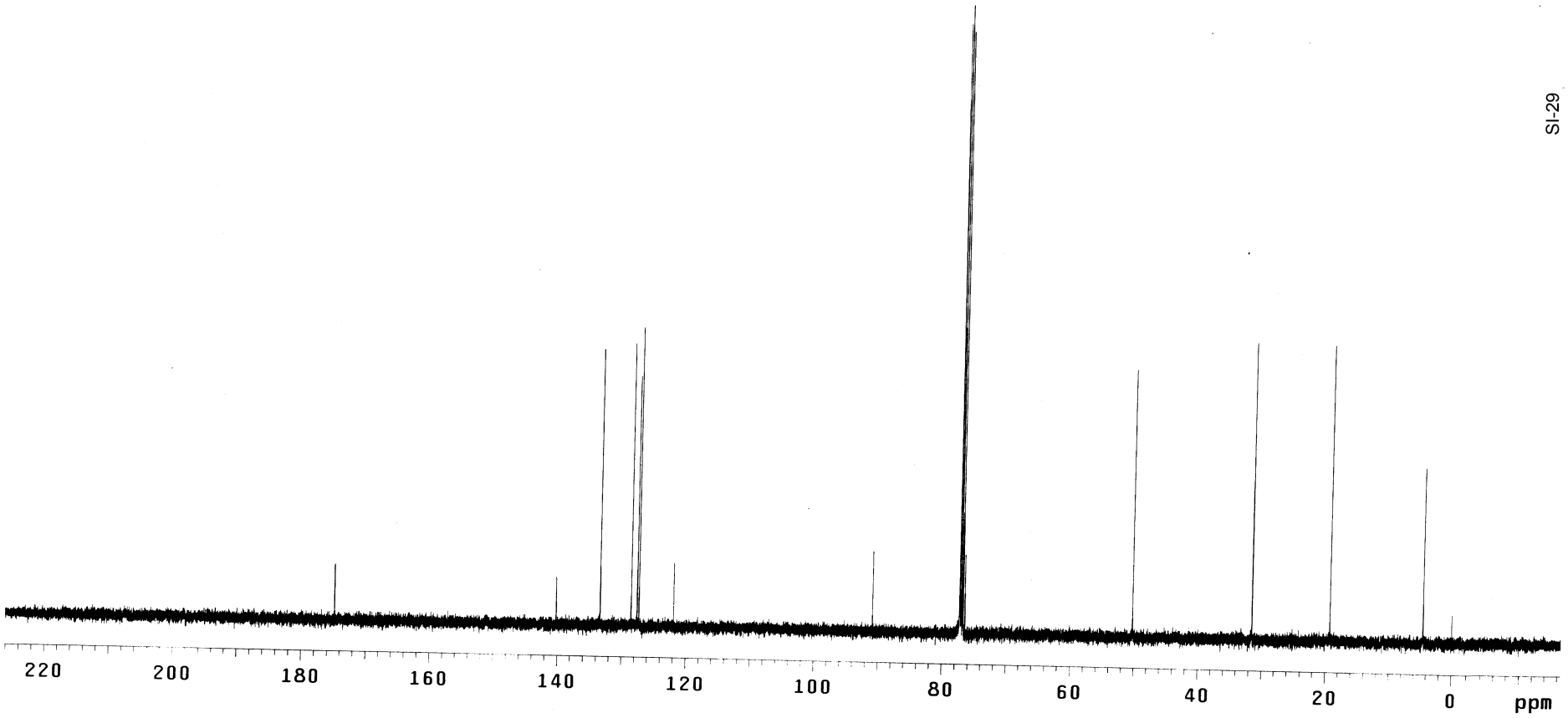
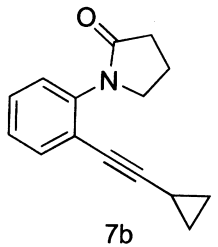
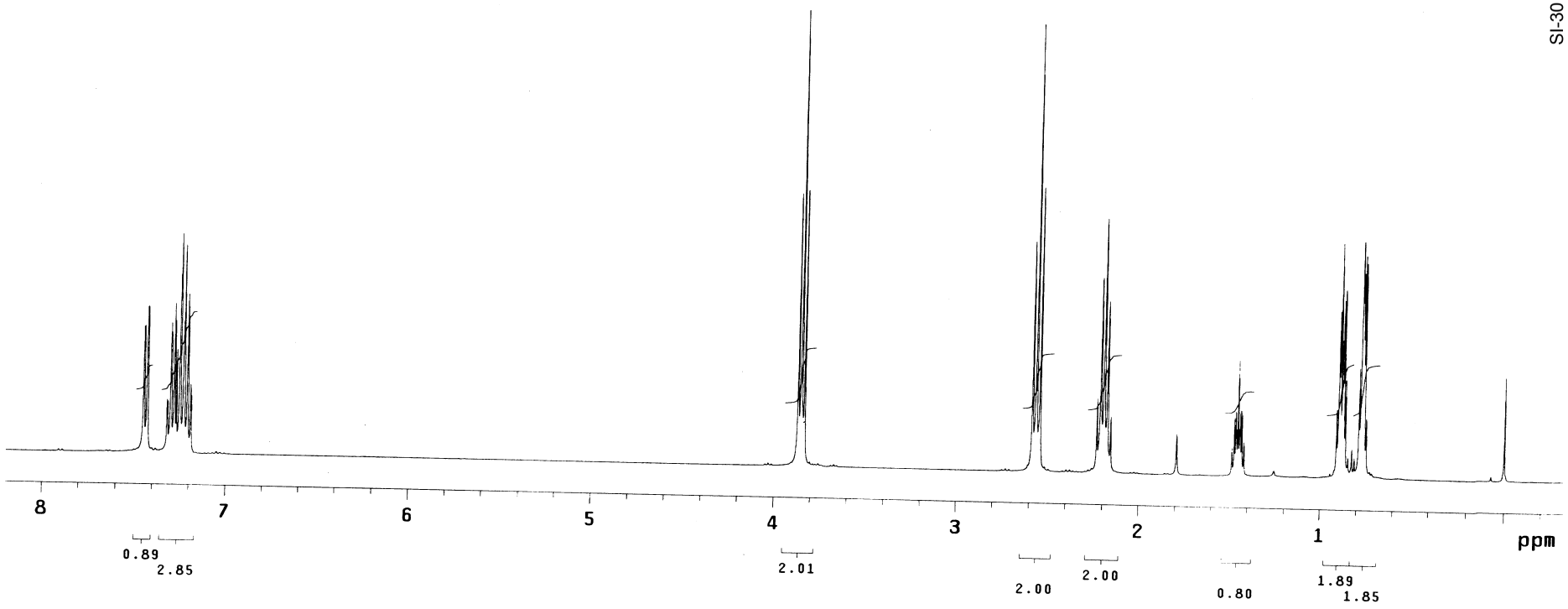


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 19 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K157-2.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	**FLAGS**	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	**PROCESSING**	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-131.3
sfrq	399.868	wp	3409.2
tof	399.6	rfl	794.8
tpwr	59	rfp	0
pw	6.750	rp	-22.1
DECOUPLER		lp	-19.9
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	1044
dpwr	34	th	2
dmf	29412	ai cdc ph	

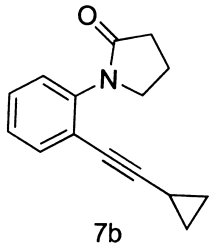


7b

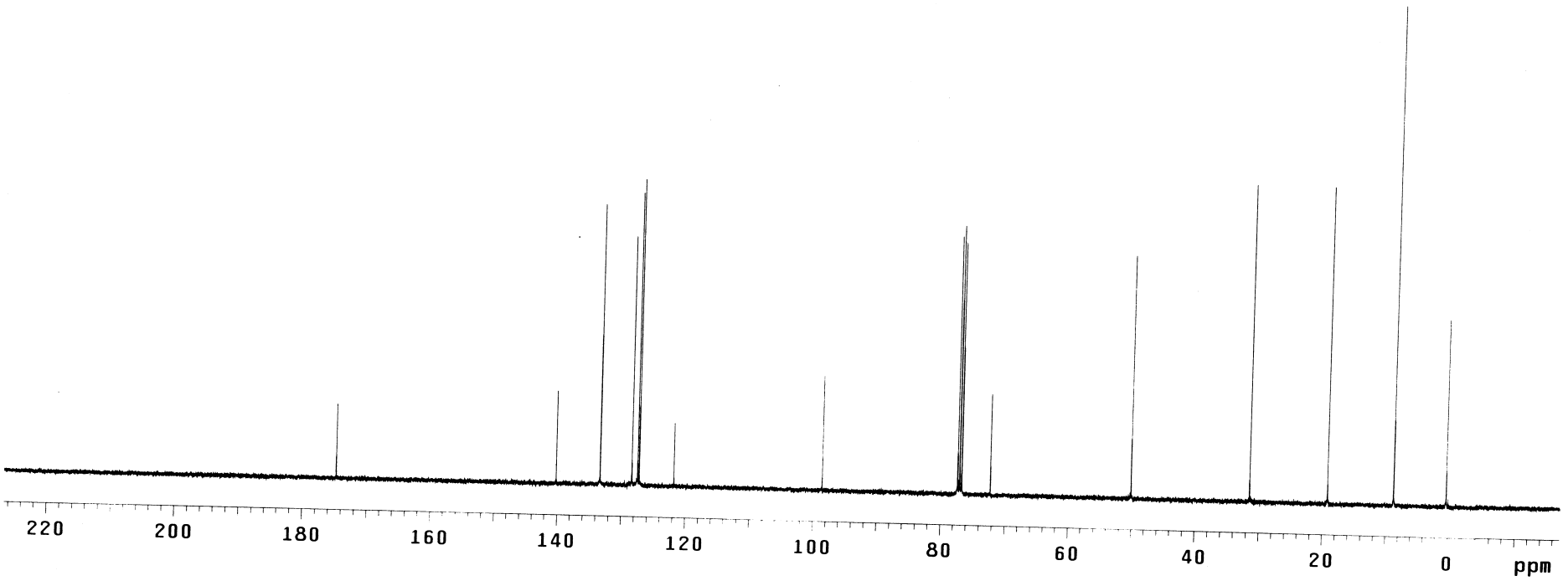


exp5 Carbon

SAMPLE		SPECIAL	
date	Jun 20 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr400/w~	spin	20
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K157-2carb~	pw90	11.900
	on.fid	alfa	10.000
ACQUISITION		**FLAGS**	
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64	**PROCESSING**	
d1	1.000	lb	0.50
nt	3200	fn	not used
ct	3200	**DISPLAY**	
TRANSMITTER		sp	-1725.1
tn	C13	wp	24509.1
sfrq	100.557	rfl	9467.9
tof	1042.8	rfp	7742.1
tpwr	53	rp	179.7
pw	5.950	lp	-199.3
DECOUPLER		**PLOT**	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	53937
dmm	w	th	7
dpwr	41	ai cdc ph	
dmf	9500		

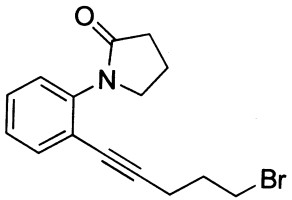


7b

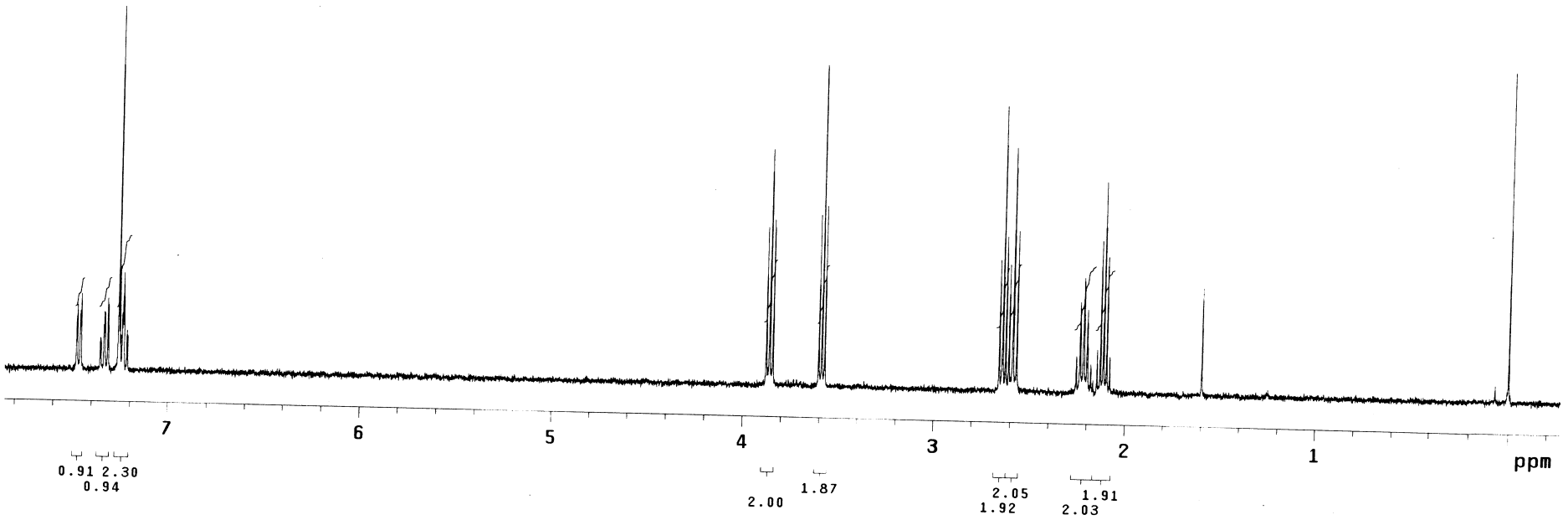


exp5 Proton

SAMPLE		SPECIAL	
date	Jun 25 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K183-1.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-108.6
sfrq	399.868	wp	3248.0
tof	399.6	rfl	798.5
tpwr	59	rfp	0
pw	6.750	rp	-109.2
DECOUPLER		lp	-21.1
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	54163
dpwr	34	th	4
dmf	29412	ai cdc ph	

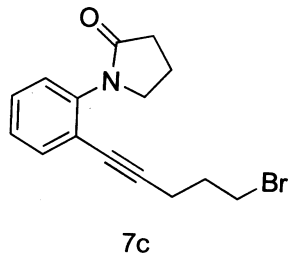


7c

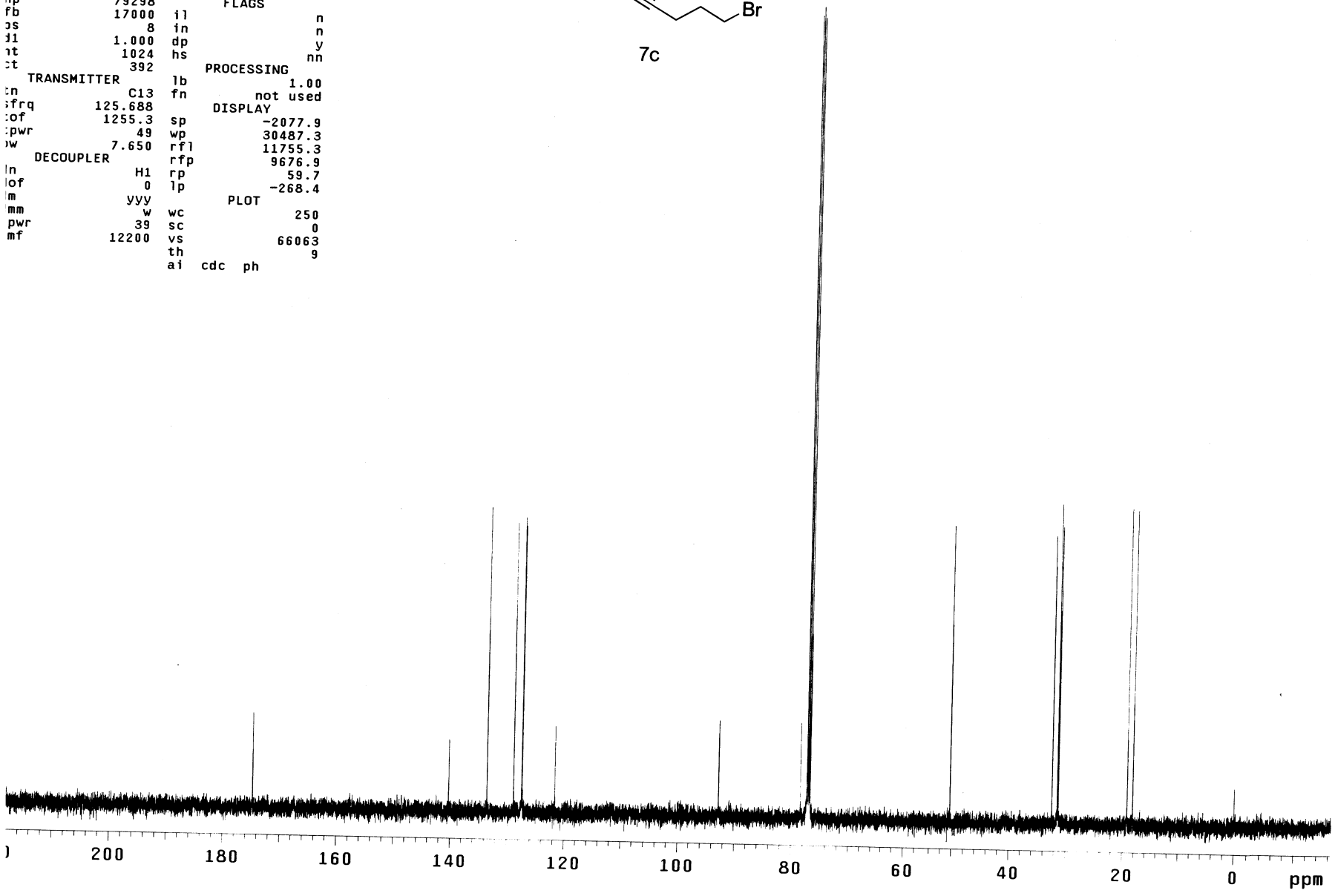


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	FLAGS	
fb	17000	il	n
bs	8	in	n
d1	1.000	dp	y
nt	1024	hs	nn
ct	392	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.688	DISPLAY	
tof	1255.3	sp	-2077.9
tpwr	49	wp	30487.3
pw	7.650	rfl	11755.3
DECOUPLER		rfp	9676.9
dn	H1	rp	59.7
dof	0	lp	-268.4
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	66063
		th	9
		ai	cdc ph

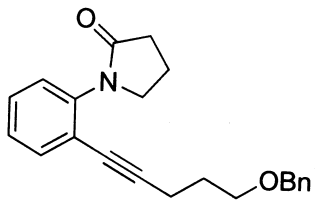


7c

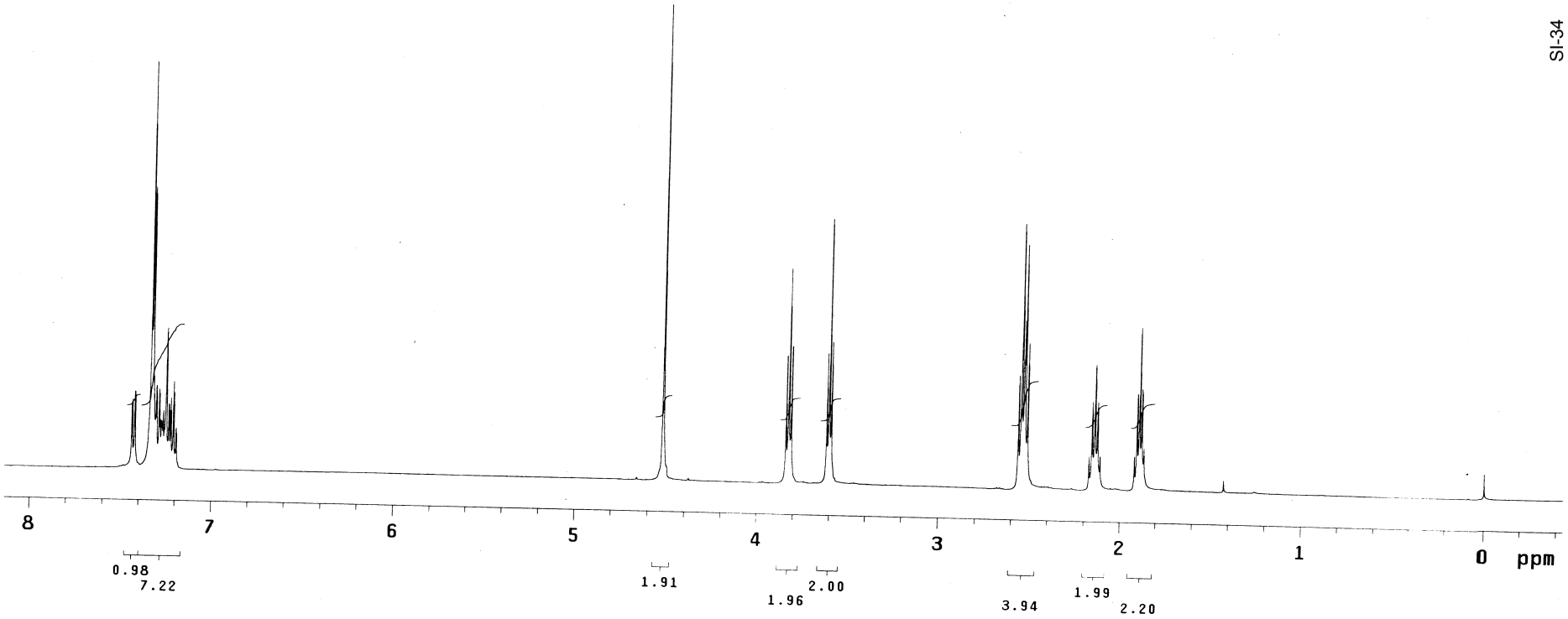


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 30 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr500/w~	spin	not used
alkup	/data/Zhang/L~	hst	0.008
	i/K205.fid	pw90	14.700
ACQUISITION		alfa	6.600
sw	8012.8	FLAGS	
at	2.049	il	n
np	32830	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-220.3
sfrq	499.802	wp	4286.9
tof	499.7	rfl	1011.4
tpwr	54	rfp	0
pw	7.350	rp	-174.5
DECOUPLER		lp	-13.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	38
dpwr	34	th	2
dmf	32258	ai cdc ph	

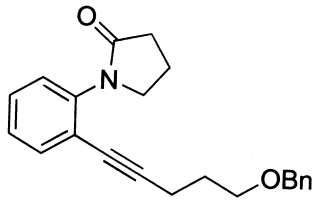


7d

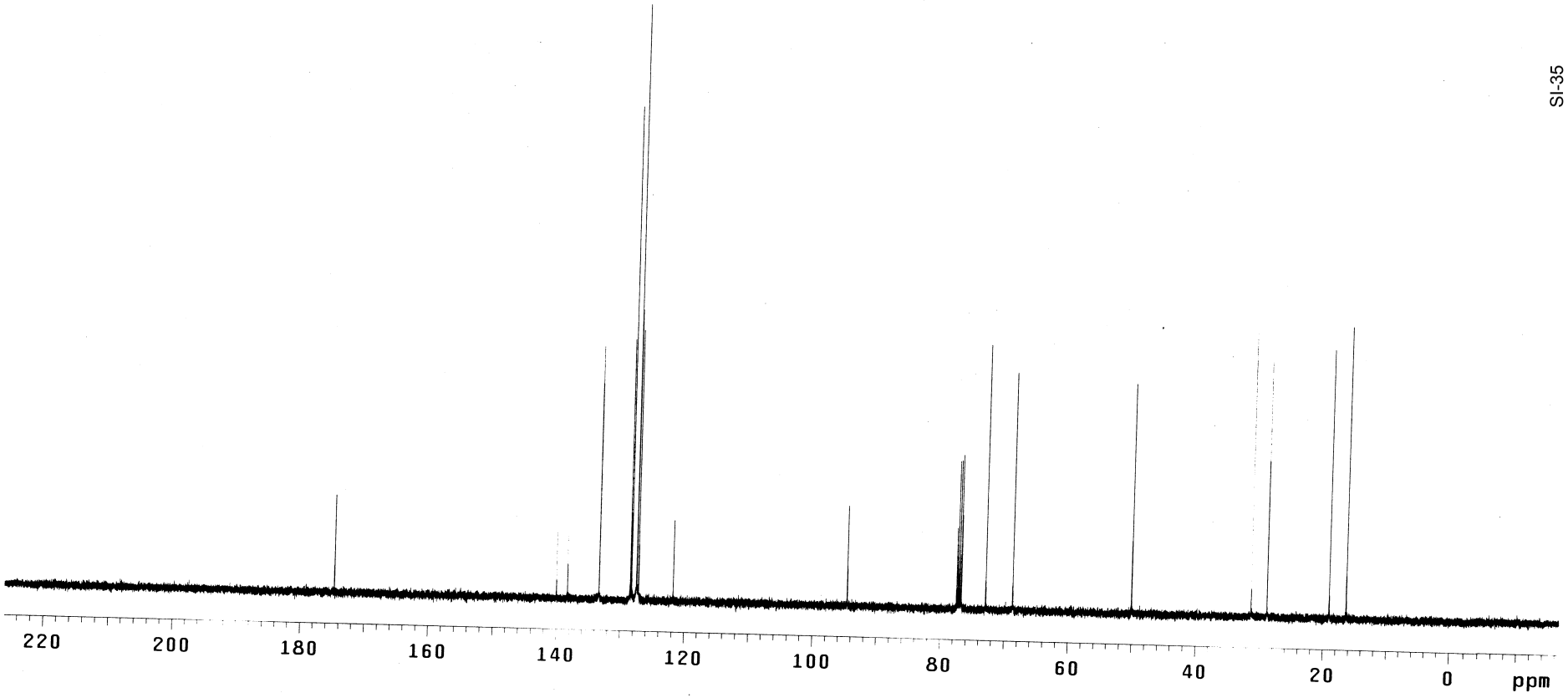


exp3 Carbon

SAMPLE		SPECIAL	
date	Jun 30 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	1024	hs	nn
ct	80	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2092.3
tpwr	49	wp	30487.3
pw	7.650	rfl	11769.7
DECOUPLER		rfp	9676.9
dn	H1	rp	150.5
dof	0	lp	-247.3
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	8429
		th	7
		ai	cdc ph

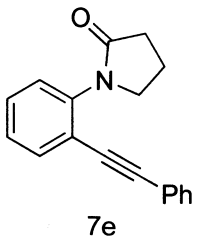


7d

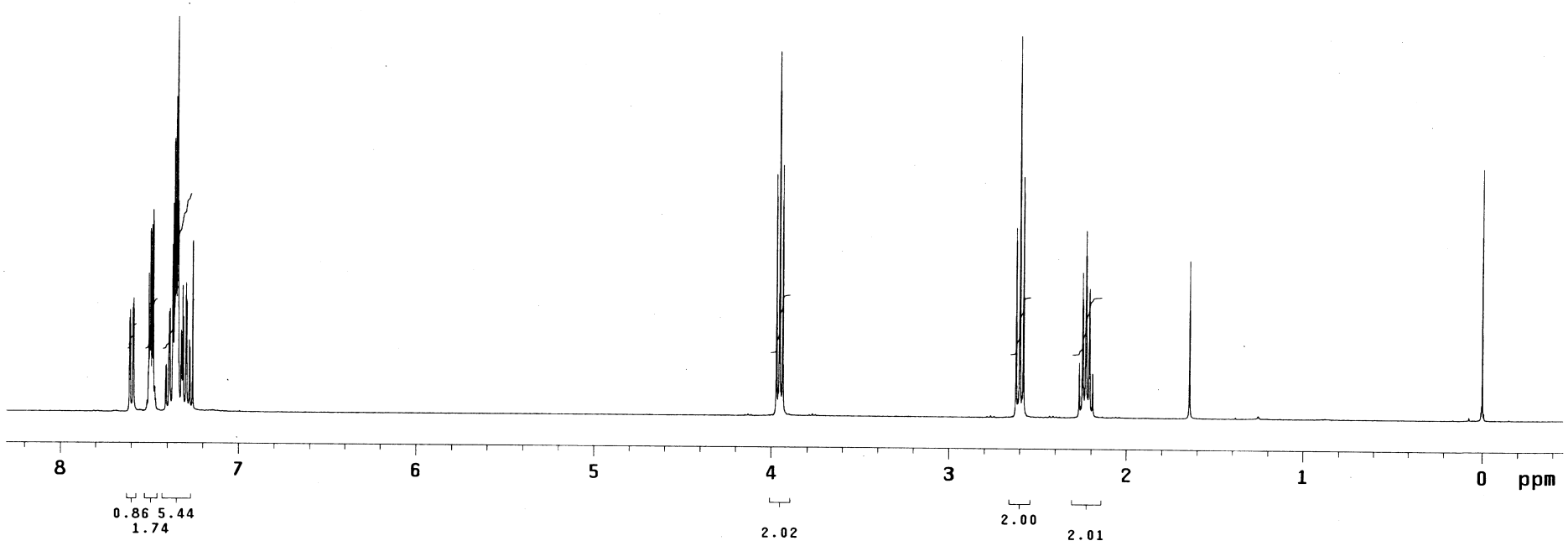


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 10 2007	temp	25.0
solvent	cdcl3	gain	26
file	/mnt/nmr400/w~	spin	20
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K63-2.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-183.1
sfrq	399.868	wp	3503.5
tof	399.6	rfl	799.7
tpwr	59	rfp	0
pw	6.750	rp	-95.8
DECOUPLER		lp	-29.2
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	487
dpwr	34	th	3
dmf	29412	ai cdc ph	

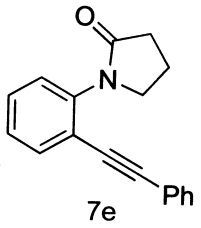


7e

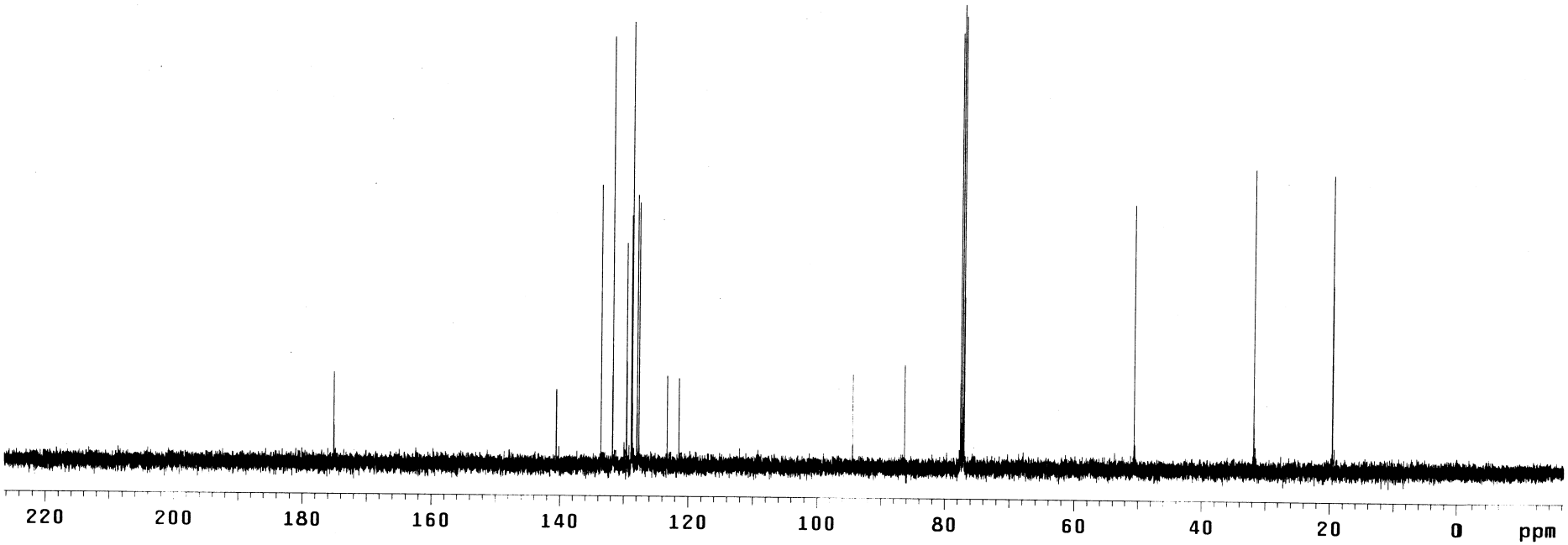


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 9 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
data	/Zhang/Li/K63-~	hst	0.008
	2carbon.fid	pw90	15.300
ACQUISITION		alfa	10.000
sw	30487.8	FLAGS	
at	1.300	il	n
np	79298	in	n
fb	17000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	1024	lb	0.50
ct	64	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-2047.6
sfrq	125.688	wp	30487.3
tof	1255.3	rfl	2048.0
tpwr	49	rfp	0
pw	7.650	rp	-20.9
DECOUPLER		lp	-309.3
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	17188
dpwr	39	th	8
dmf	12200	ai cdc ph	

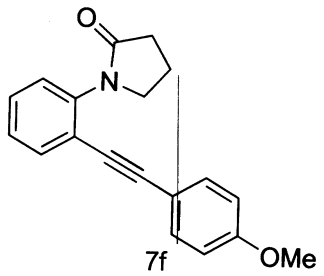


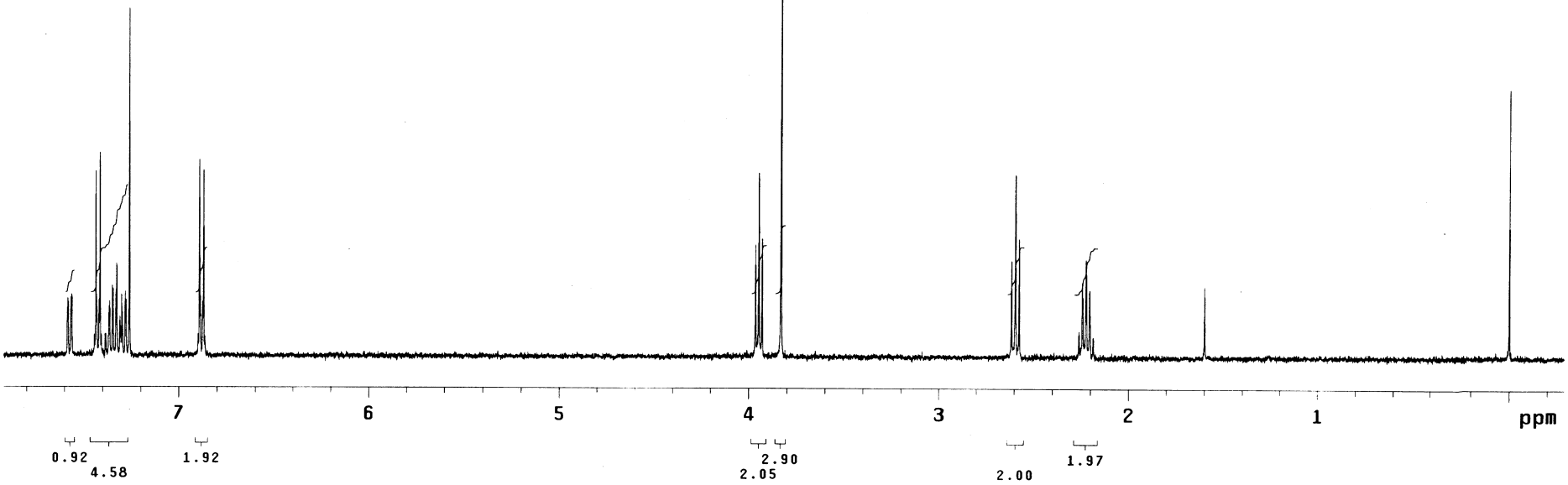
7e



exp5 Proton

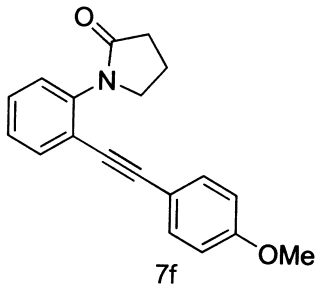
SAMPLE		SPECIAL	
date	Jun 25 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	not used
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K184-1.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-116.4
sfrq	399.868	wp	3284.0
tof	399.6	rfl	799.1
tpwr	59	rfp	0
pw	6.750	rp	-104.0
DECOUPLER		lp	-30.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	132518
dpwr	34	th	2
dmf	29412	ai cdc ph	



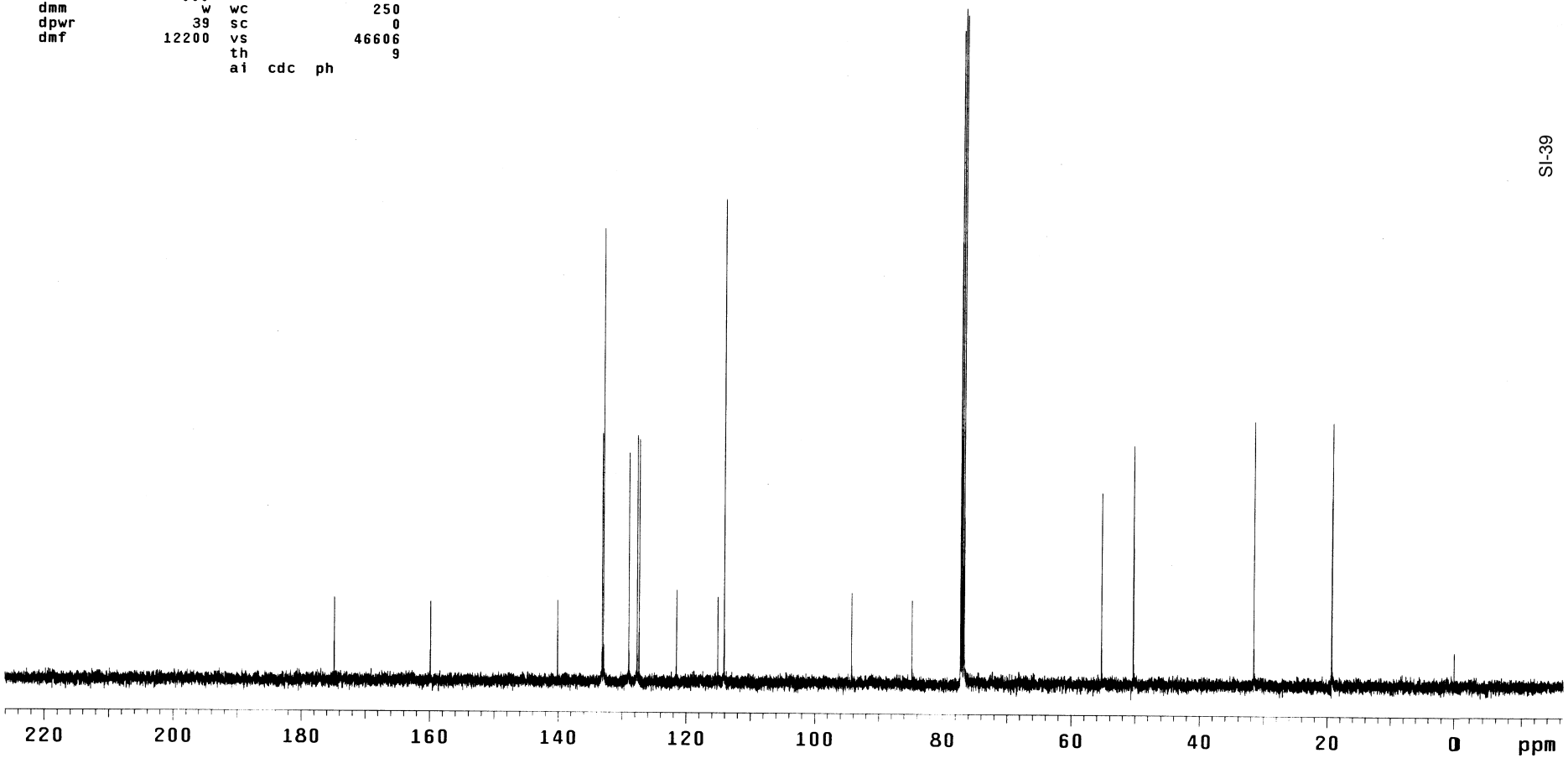


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	8	in	n
d1	1.000	dp	y
nt	1024	hs	nn
ct	456	**PROCESSING**	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2077.9
tpwr	49	wp	30487.3
pw	7.650	rfl	11755.3
DECOUPLER		rfp	9676.9
dn	H1	rp	33.0
dof	0	lp	-214.2
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	46606
		th	9
		ai	cdc ph

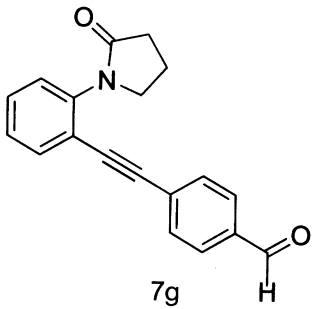


7f

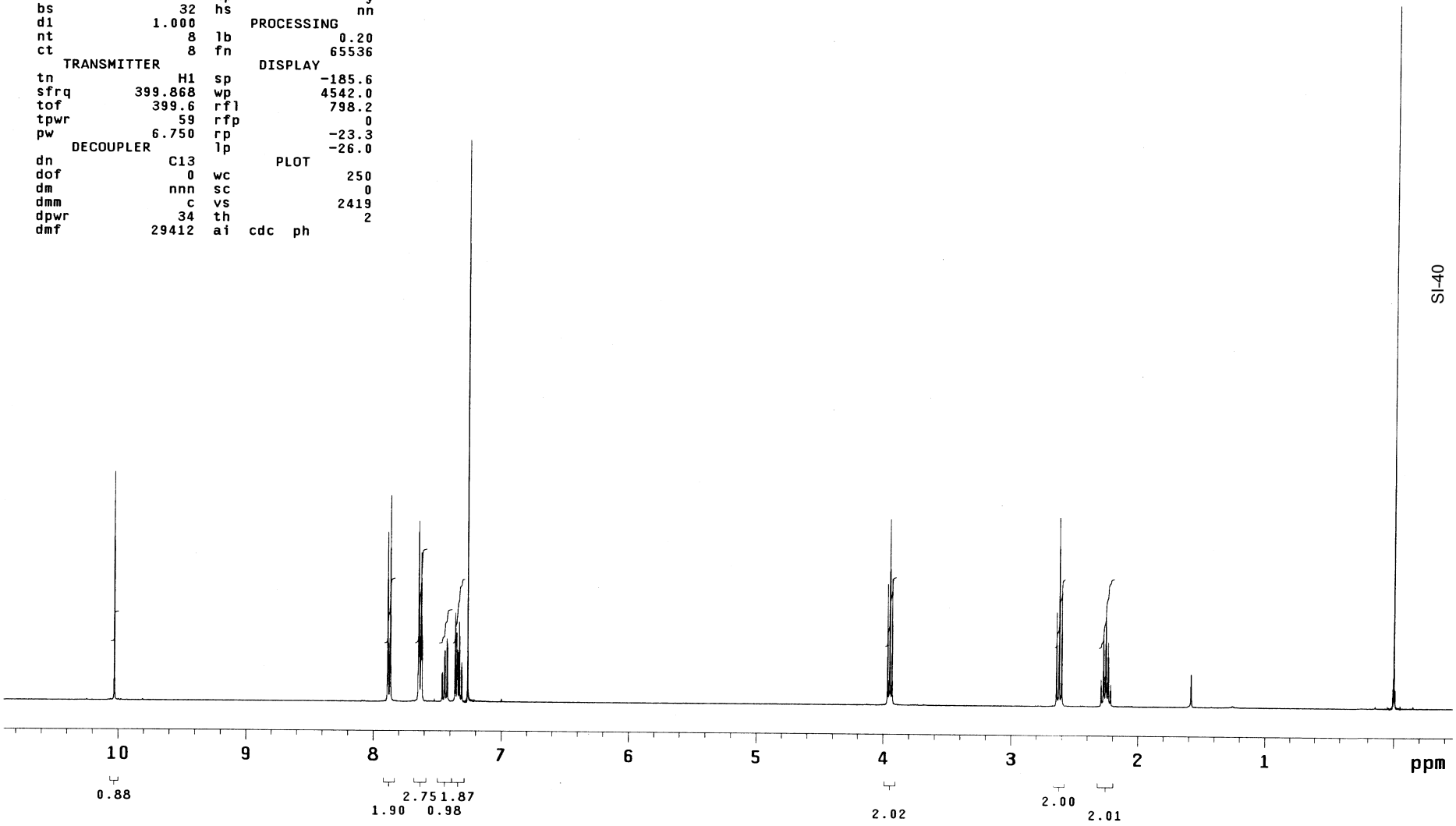


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 21 2007	temp	not used
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K171-2.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-185.6
sfrq	399.868	wp	4542.0
tof	399.6	rfl	798.2
tpwr	59	rfp	0
pw	6.750	rp	-23.3
DECOUPLER		lp	-26.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	2419
dpwr	34	th	2
dmf	29412	ai cdc ph	

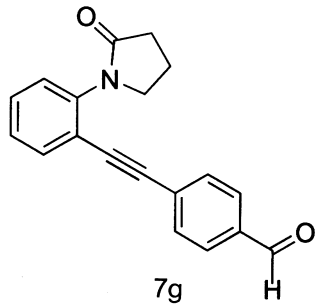


7g

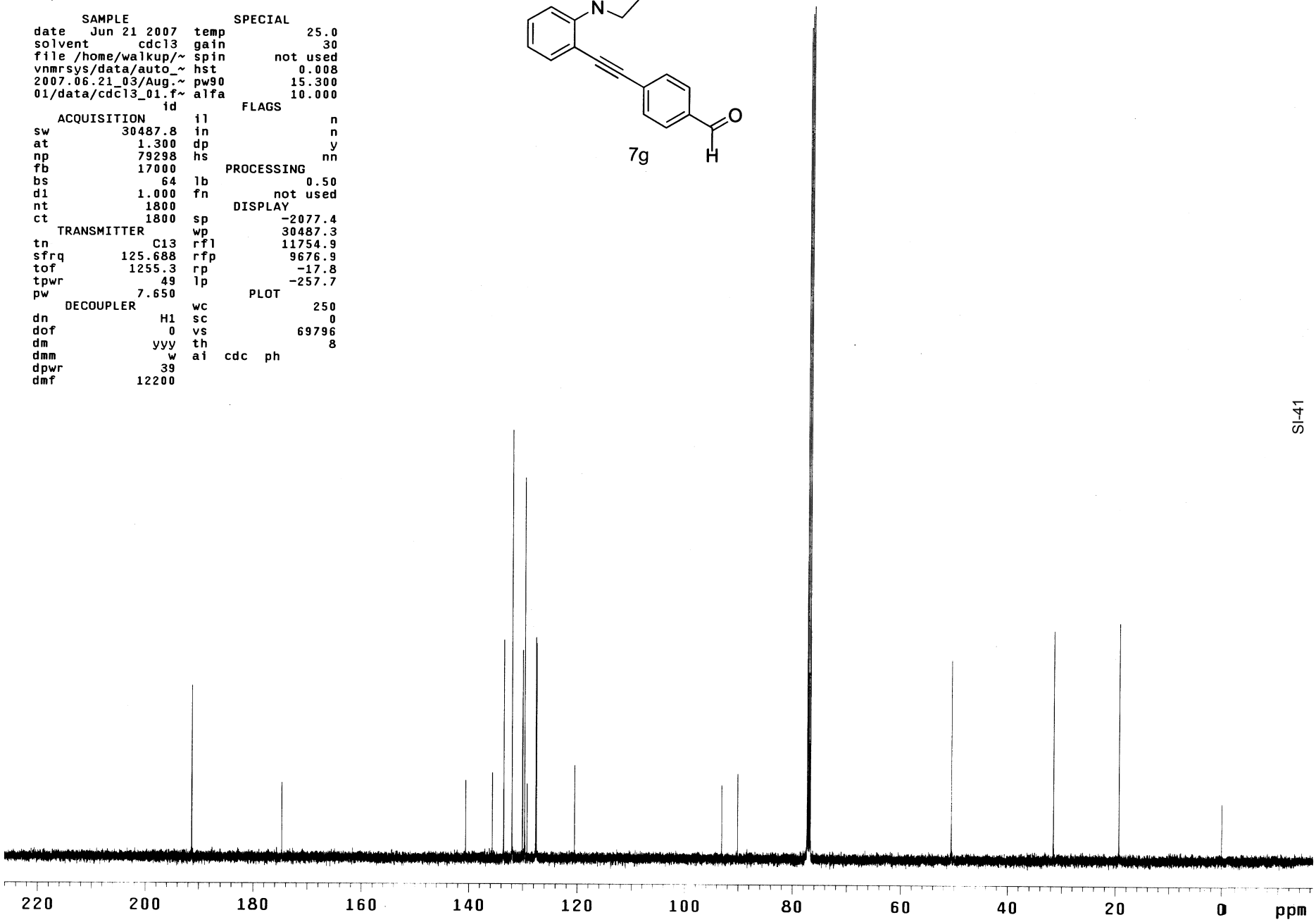


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 21 2007	temp	25.0
solvent	cdcl3	gain	30
file	/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		FLAGS	
ACQUISITION		il	n
sw	30487.8	in	n
at	1.300	dp	y
np	79298	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	1800	DISPLAY	
ct	1800	sp	-2077.4
TRANSMITTER		wp	30487.3
tn	C13	rfl	11754.9
sfrq	125.688	rfp	9676.9
tof	1255.3	rp	-17.8
tpwr	49	lp	-257.7
pw	7.650	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	69796
dm	yyy	th	8
dmm	w	ai	cdc ph
dpwr	39		
dmf	12200		

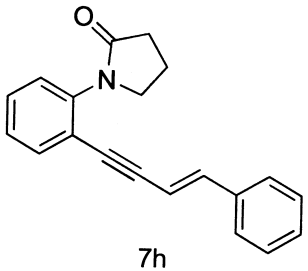


7g

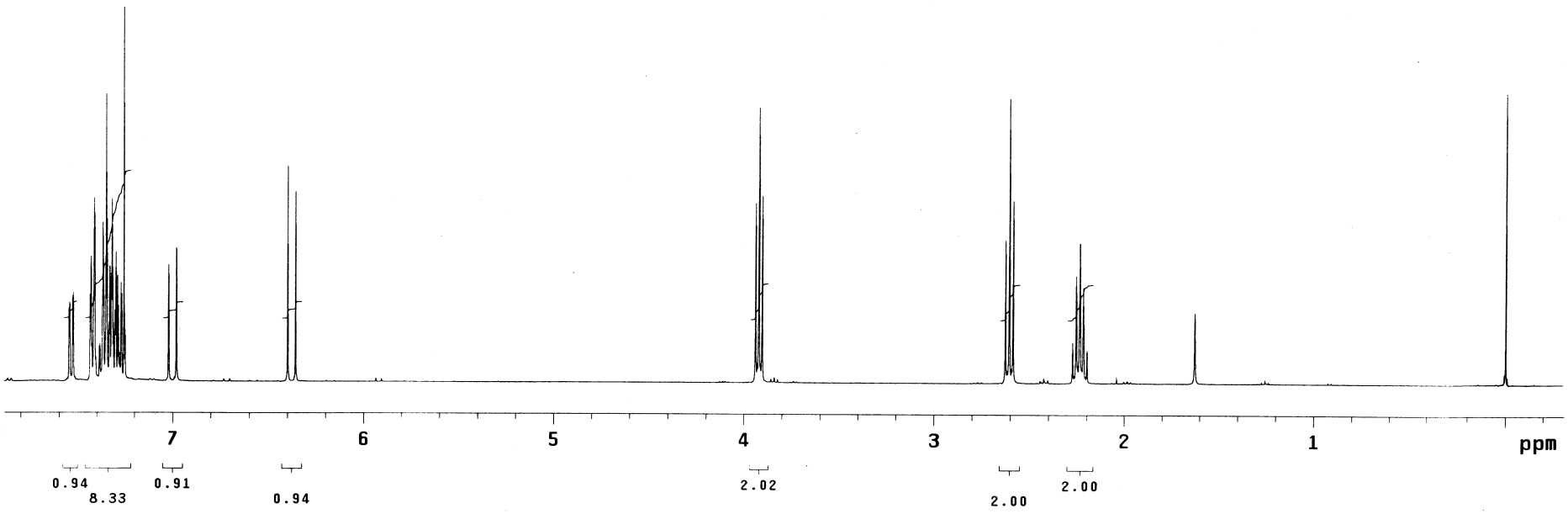


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 11 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K114-2.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-124.8
sfrq	399.868	wp	3278.3
tof	399.6	rfl	800.1
tpwr	59	rfp	0
pw	6.750	rp	-98.4
DECOUPLER		lp	-27.5
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	1177
dpwr	34	th	3
dmf	29412	ai cdc ph	

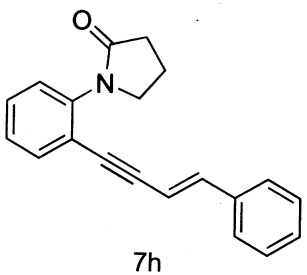


7h

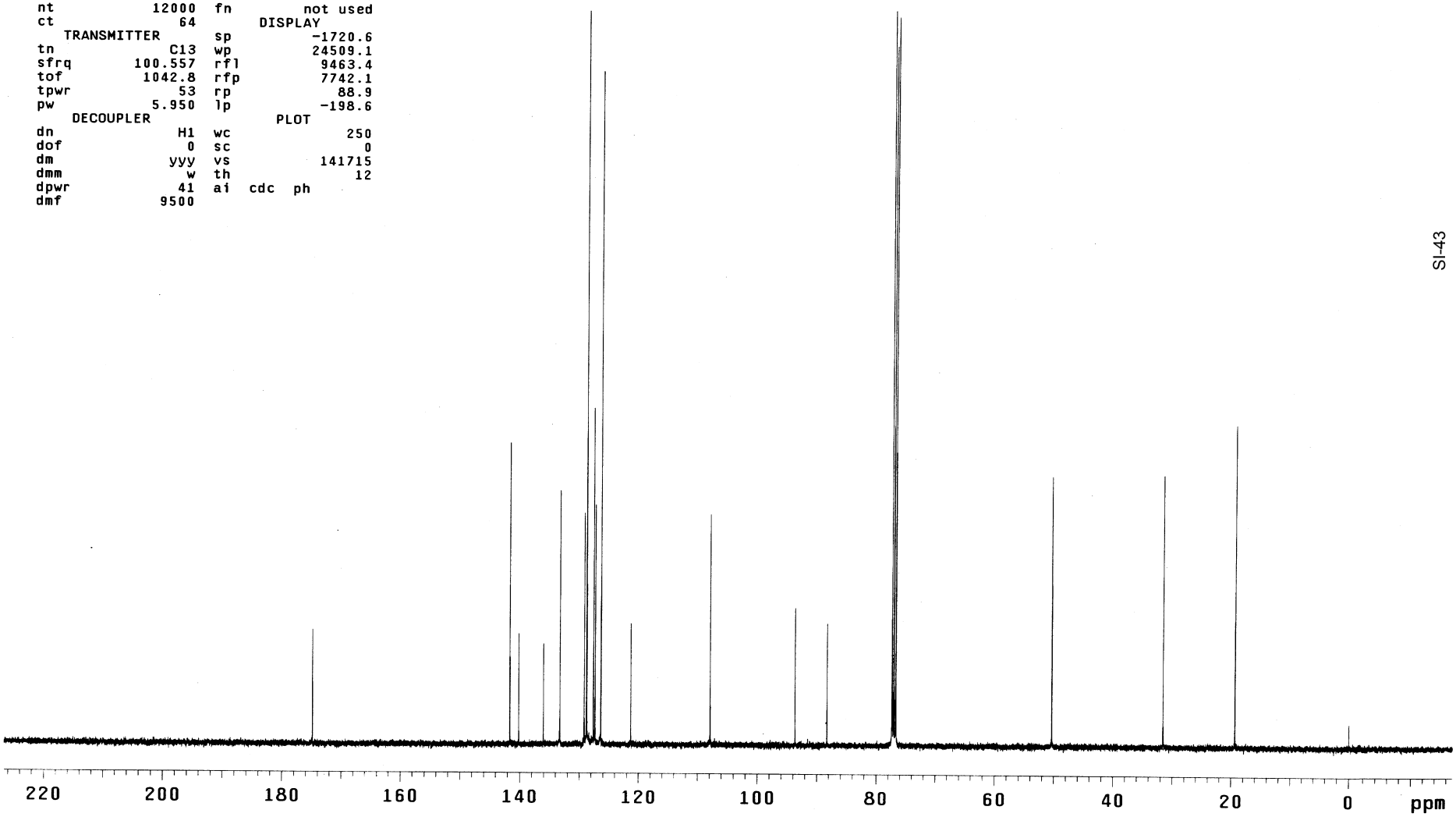


exp2 Carbon

SAMPLE		SPECIAL	
date	Jun 11 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	20
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K1~	pw90	11.900
	14-2carbon.fid	alfa	10.000
ACQUISITION		**FLAGS**	
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64	**PROCESSING**	
d1	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	rfl	9463.4
tof	1042.8	rfp	7742.1
tpwr	53	rp	88.9
pw	5.950	lp	-198.6
DECOUPLER		**PLOT**	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	141715
dmm	w	th	12
dpwr	41	ai	cdc ph
dmf	9500		



7h

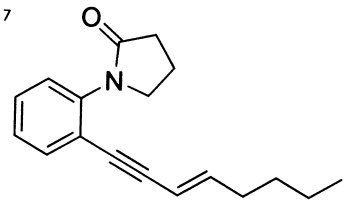


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.27
File : s_20070627_001/data/cdcl3_01.fid
Sample id : s_20070627_001

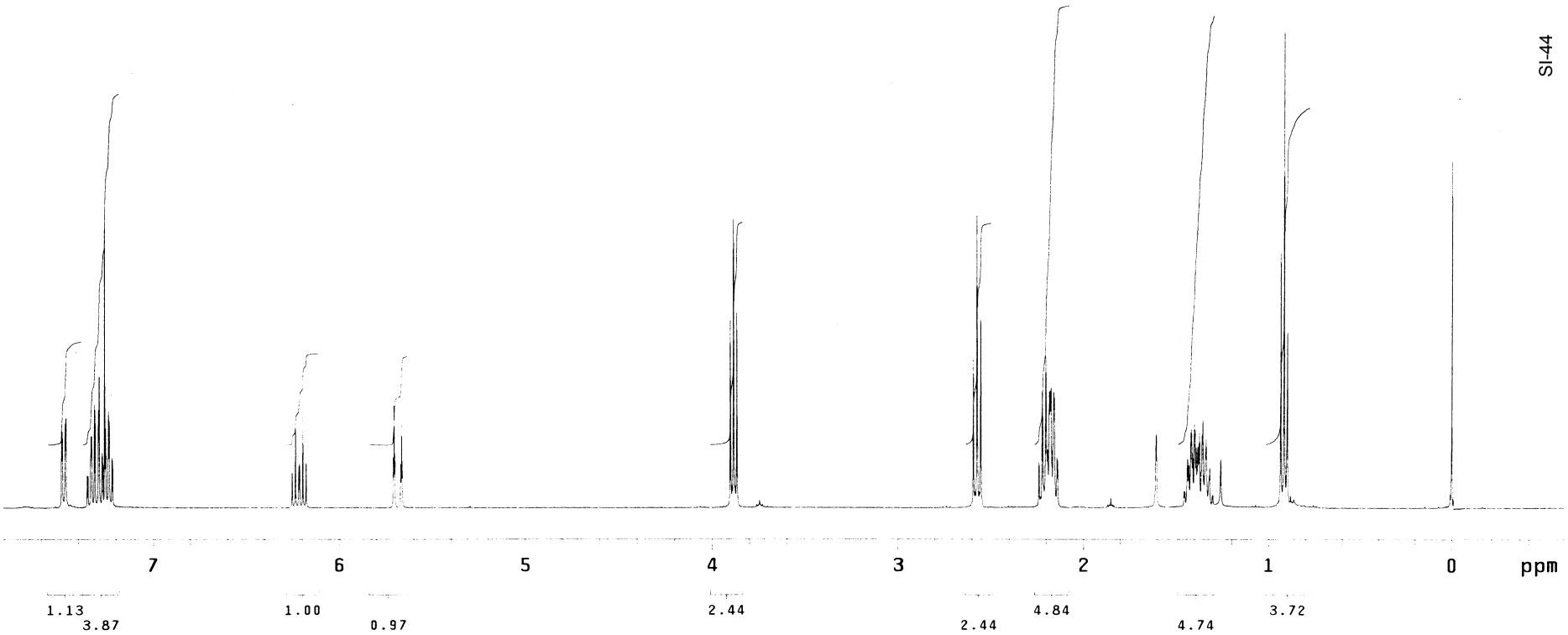
Pulse Sequence: s2pul

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 2.049 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



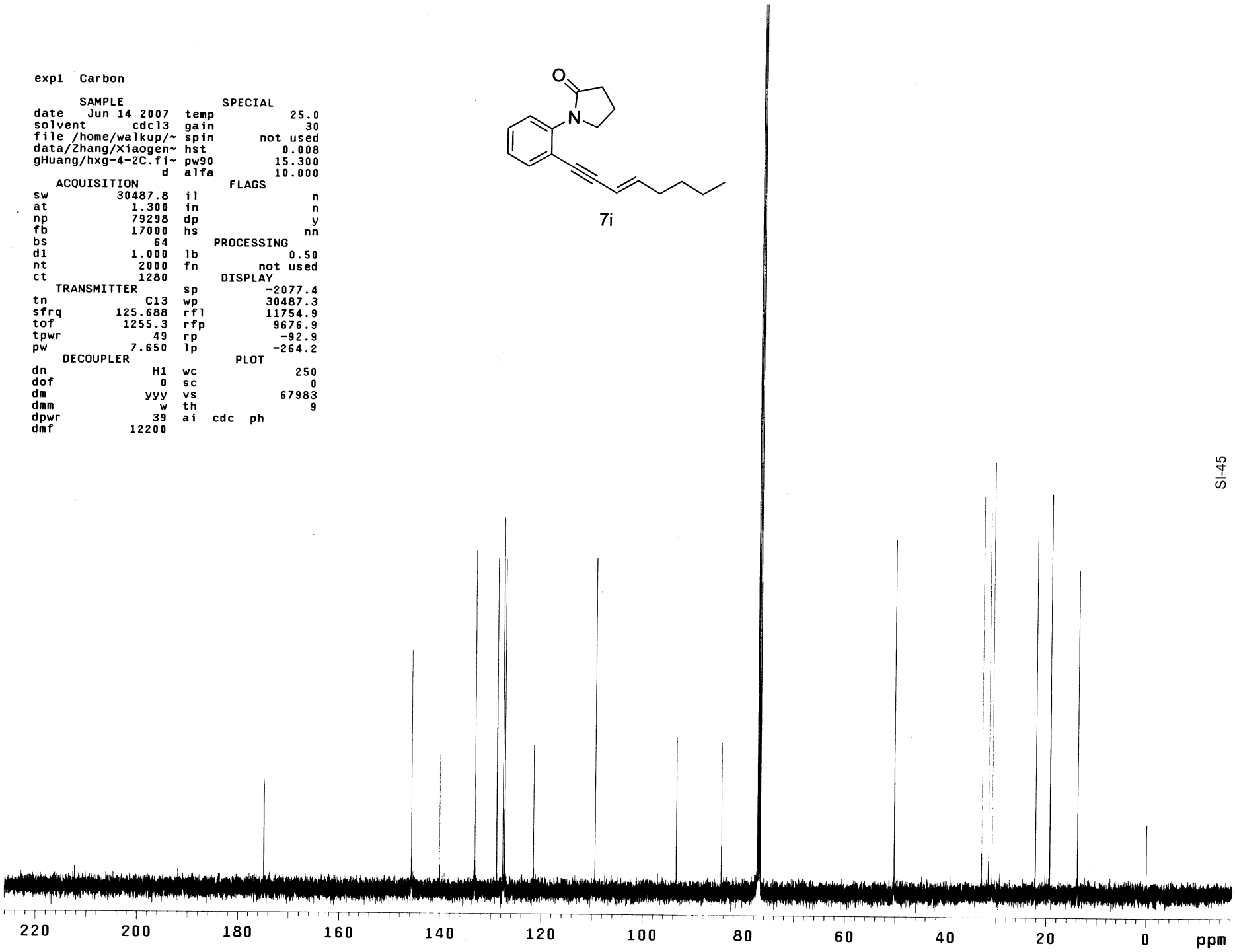
7i



exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 14 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
data/Zhang/Xiaogen~		hst	0.008
gHuang/hxg-4-2C.fi~		pw90	15.300
	d	alfa	10.000
ACQUISITION		**FLAGS**	
sw	30487.8	il	n
at	1.300	in	n
np	79298	dp	y
fb	17000	hs	nn
bs	64	**PROCESSING**	
d1	1.000	lb	0.50
nt	2000	fn	not used
ct	1280	**DISPLAY**	
TRANSMITTER		sp	-2077.4
tn	C13	wp	30487.3
sfrq	125.688	rfl	11754.9
tof	1255.3	rfp	9676.9
tpwr	49	rp	-92.9
pw	7.650	lp	-264.2
DECOUPLER		**PLOT**	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	67983
dmm	w	th	9
dpwr	39	ai cdc ph	
dmf	12200		

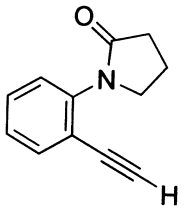
7i



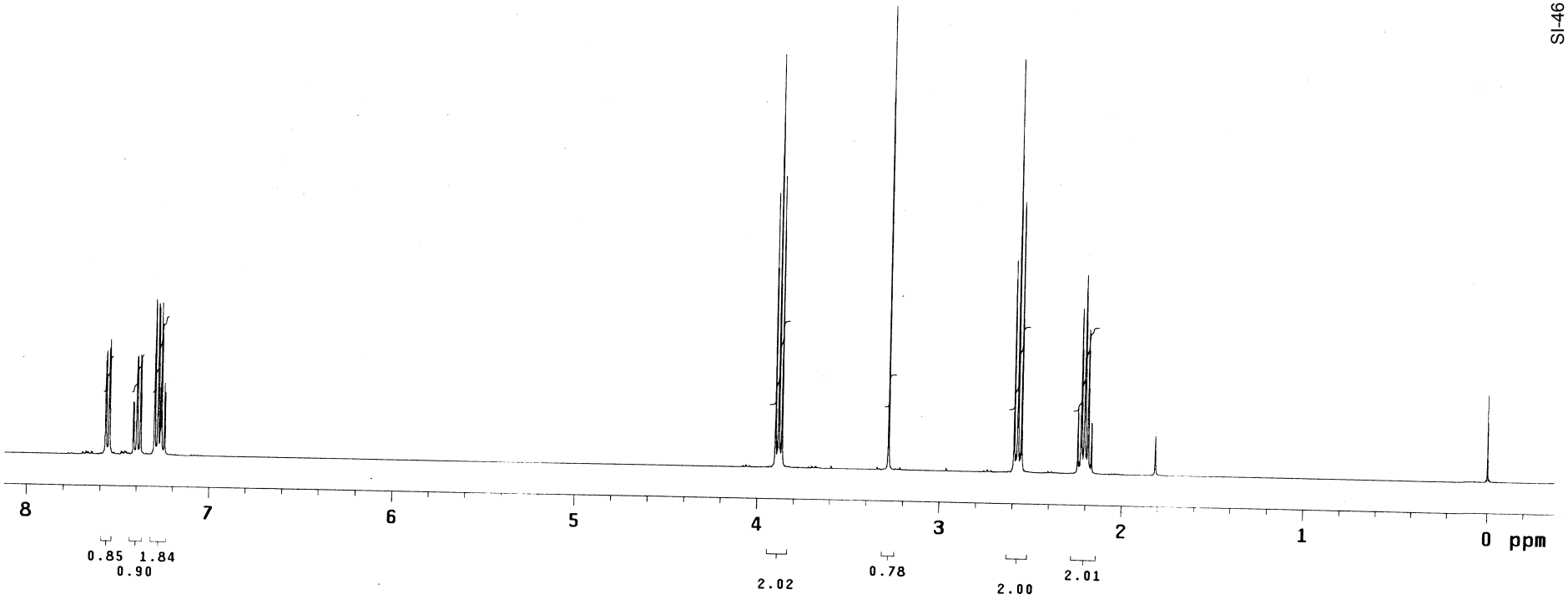
STANDARD 1H OBSERVE - profile

exp2 Proton

SAMPLE		SPECIAL	
date	Jun 9 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K94-2.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-148.9
sfrq	399.868	wp	3395.9
tof	399.6	rfl	794.8
tpwr	59	rfp	0
pw	6.750	rp	-97.6
DECOUPLER		lp	-26.3
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	339
dpwr	34	th	2
dmf	29412	ai cdc ph	



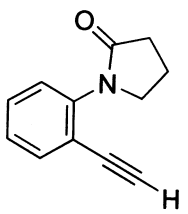
7j



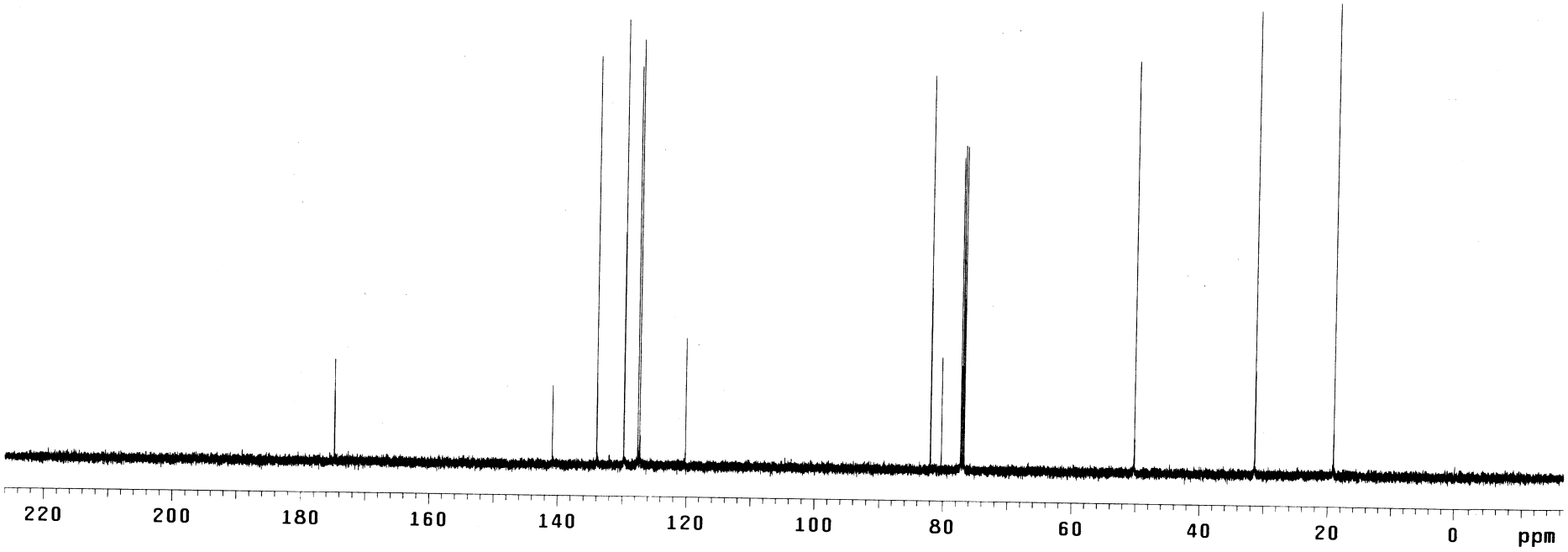
exp1 Carbon

SAMPLE	
date	Jun 9 2007
solvent	cdcl3
file	exp
ACQUISITION	
sw	30487.8
at	1.300
np	79298
fb	17000
bs	64
d1	1.000
nt	1024
ct	128
TRANSMITTER	
tn	C13
sfrq	125.688
tof	1255.3
tpwr	49
pw	7.650
DECOUPLER	
dn	H1
dof	0
dm	yyy
dmm	w
dpwr	39
dmf	12200

SPECIAL	
temp	25.0
gain	30
spin	not used
hst	0.008
pw90	15.300
alfa	10.000
FLAGS	
il	n
in	n
dp	y
hs	nn
PROCESSING	
lb	0.50
fn	not used
DISPLAY	
sp	-2085.3
wp	30487.3
rfl	11762.8
rfp	9676.9
rp	-26.5
lp	-287.0
PLOT	
wc	250
sc	0
vs	14226
th	11
ai cdc ph	



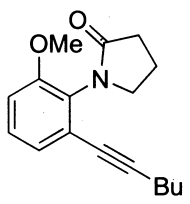
7j



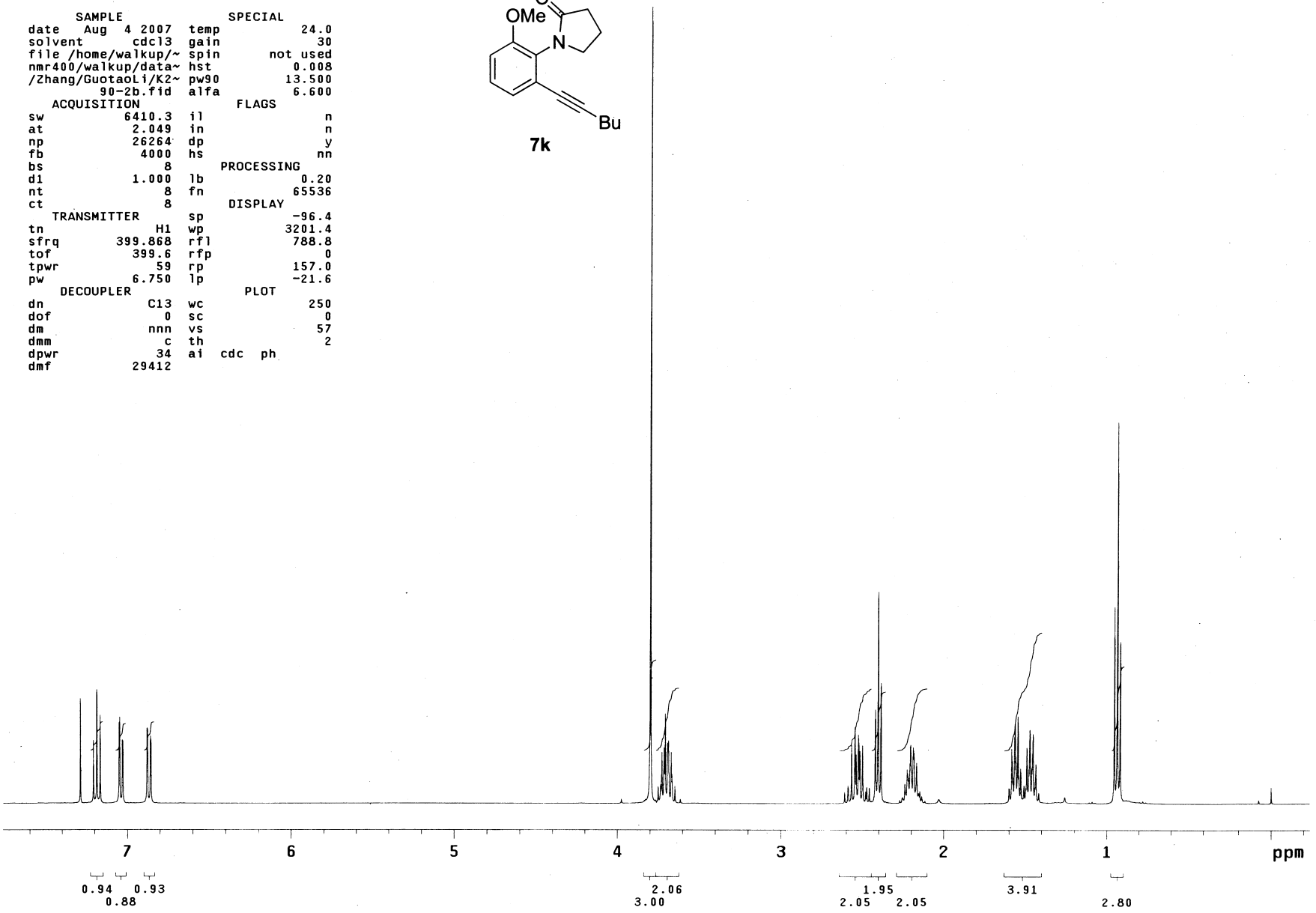
STANDARD 1H OBSERVE - profile

exp1 Proton

SAMPLE		SPECIAL	
date	Aug 4 2007	temp	24.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K2~	pw90	13.500
	90-2b.fid	alfa	6.600
ACQUISITION		FLAGS	
sw	6410.3	il	n
at	2.049	in	n
np	26264	dp	y
fb	4000	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-96.4
tn	H1	wp	3201.4
sfrq	399.868	rfl	788.8
tof	399.6	rfp	0
tpwr	59	rp	157.0
pw	6.750	lp	-21.6
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	57
dmm	c	th	2
dpwr	34	ai	cdc ph
dmf	29412		

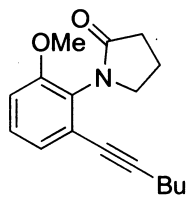


7k

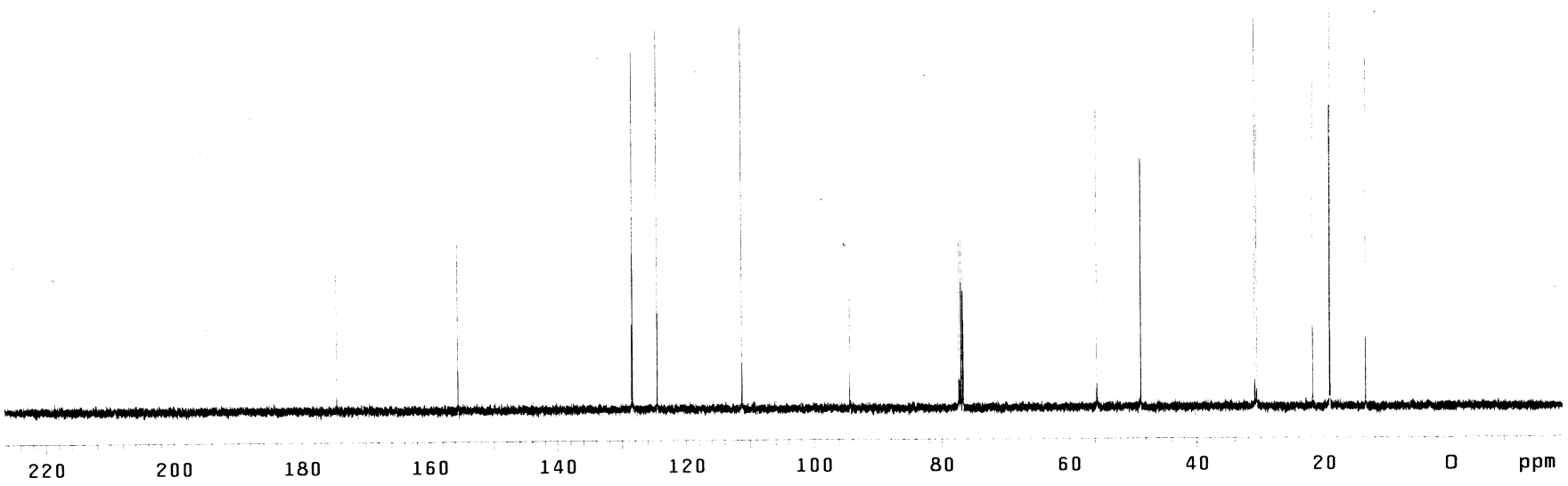


exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 4 2007	temp	24.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.04_08/s_20~	pw90	11.900
	070804_001/data/cd~	alfa	10.000
	cl3_01.fid	FLAGS	
ACQUISITION		il	n
sw	24509.8	in	n
at	1.300	dp	y
np	63750	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	3200	DISPLAY	
ct	64	sp	-1733.3
TRANSMITTER		wp	24509.1
tn	C13	rfl	9476.2
sfrq	100.557	rfp	7742.1
tof	1042.8	rp	-152.2
tpwr	53	lp	-195.4
pw	5.950	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	17546
dm	yyy	th	68
dmm	w	ai	cdc ph
dpwr	41		
dmf	9500		

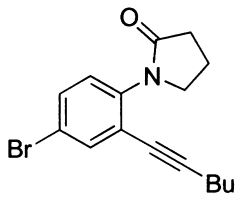


7k

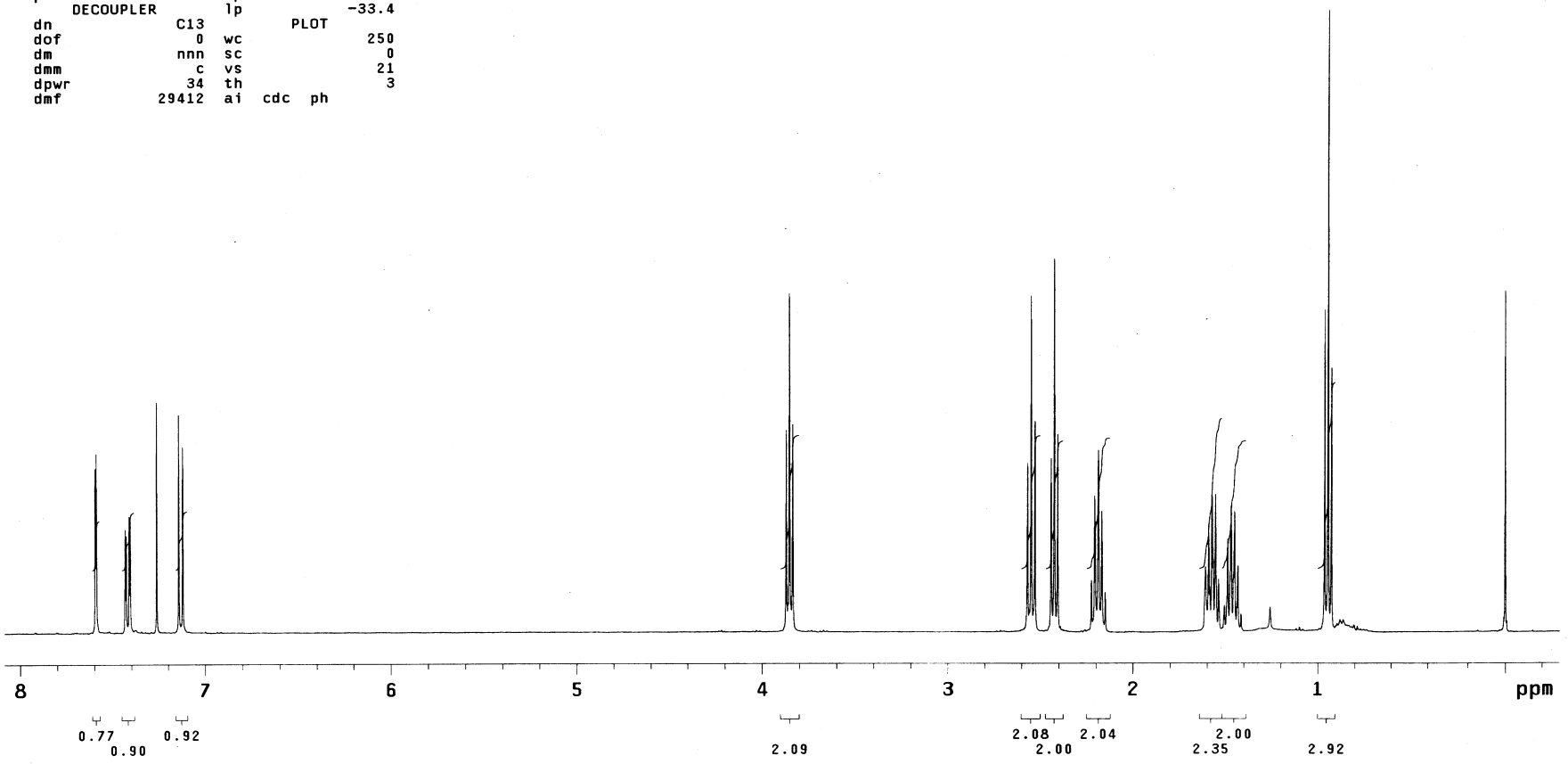


exp1 Proton

SAMPLE		SPECIAL	
date	Jul 30 2007	temp	25.0
solvent	cdcl3	gain	60
file	/mnt/nmr400/w~	spin	not used
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K283-3.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-120.1
sfrq	399.868	wp	3352.8
tof	399.6	rfl	798.0
tpwr	59	rfp	0
pw	6.750	rp	172.6
DECOUPLER		lp	-33.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	21
dpwr	34	th	3
dmf	29412	ai cdc ph	

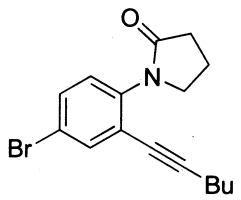


7l

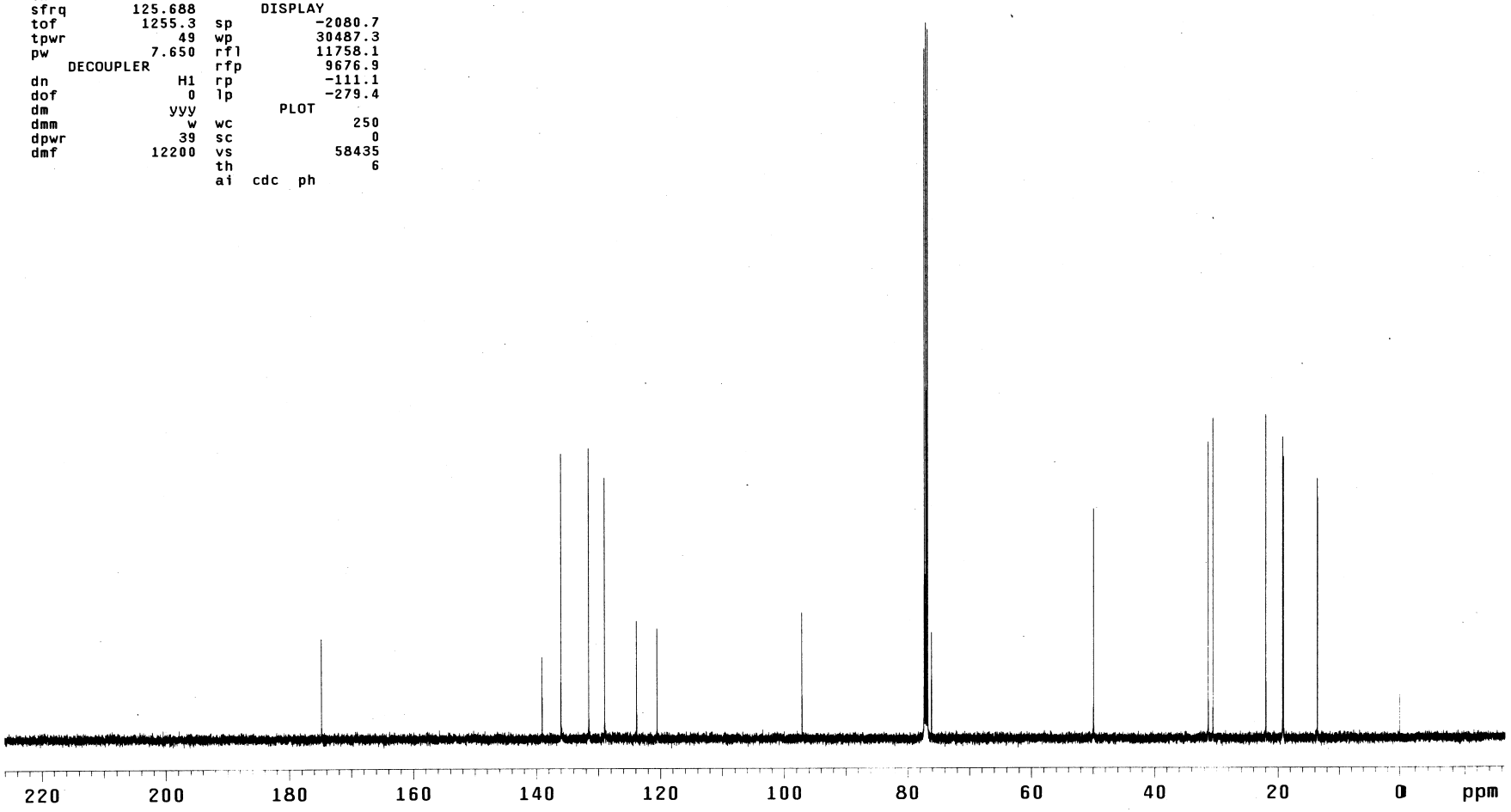


exp1 Carbon

SAMPLE		SPECIAL	
date	Jul 31 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	64	in	n
d1	1.000	dp	y
nt	2000	hs	nn
ct	1600	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2080.7
tpwr	49	wp	30487.3
pw	7.650	rfl	11758.1
DECOUPLER		rfp	9676.9
dn	H1	rp	-111.1
dof	0	lp	-279.4
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	58435
		th	6
		ai	cdc ph



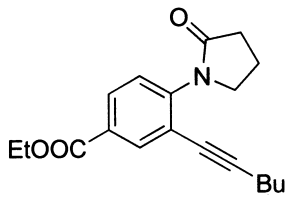
7l



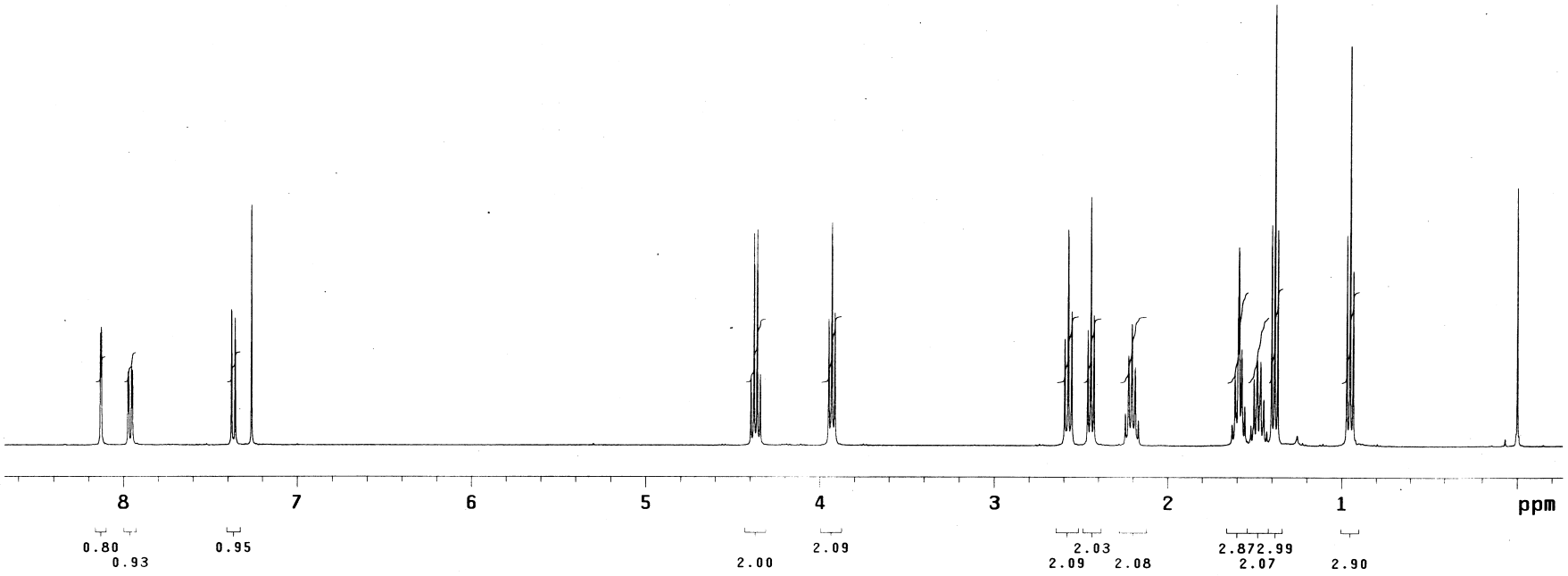
STANDARD 1H OBSERVE - profile
STANDARD 1H OBSERVE - profile
STANDARD 1H OBSERVE - profile

exp1 Proton

SAMPLE		SPECIAL	
date	Aug 3 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K2~	pw90	13.500
	85-2.fid	alfa	6.600
ACQUISITION		FLAGS	
sw	6410.3	il	n
at	2.049	in	n
np	26264	dp	y
fb	4000	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-105.4
tn	H1	wp	3576.2
sfrq	399.868	rfl	797.8
tof	399.6	rfp	0
tpwr	59	rp	151.0
pw	6.750	lp	-30.4
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	699
dmm	c	th	3
dpwr	34	ai cdc ph	
dmf	29412		



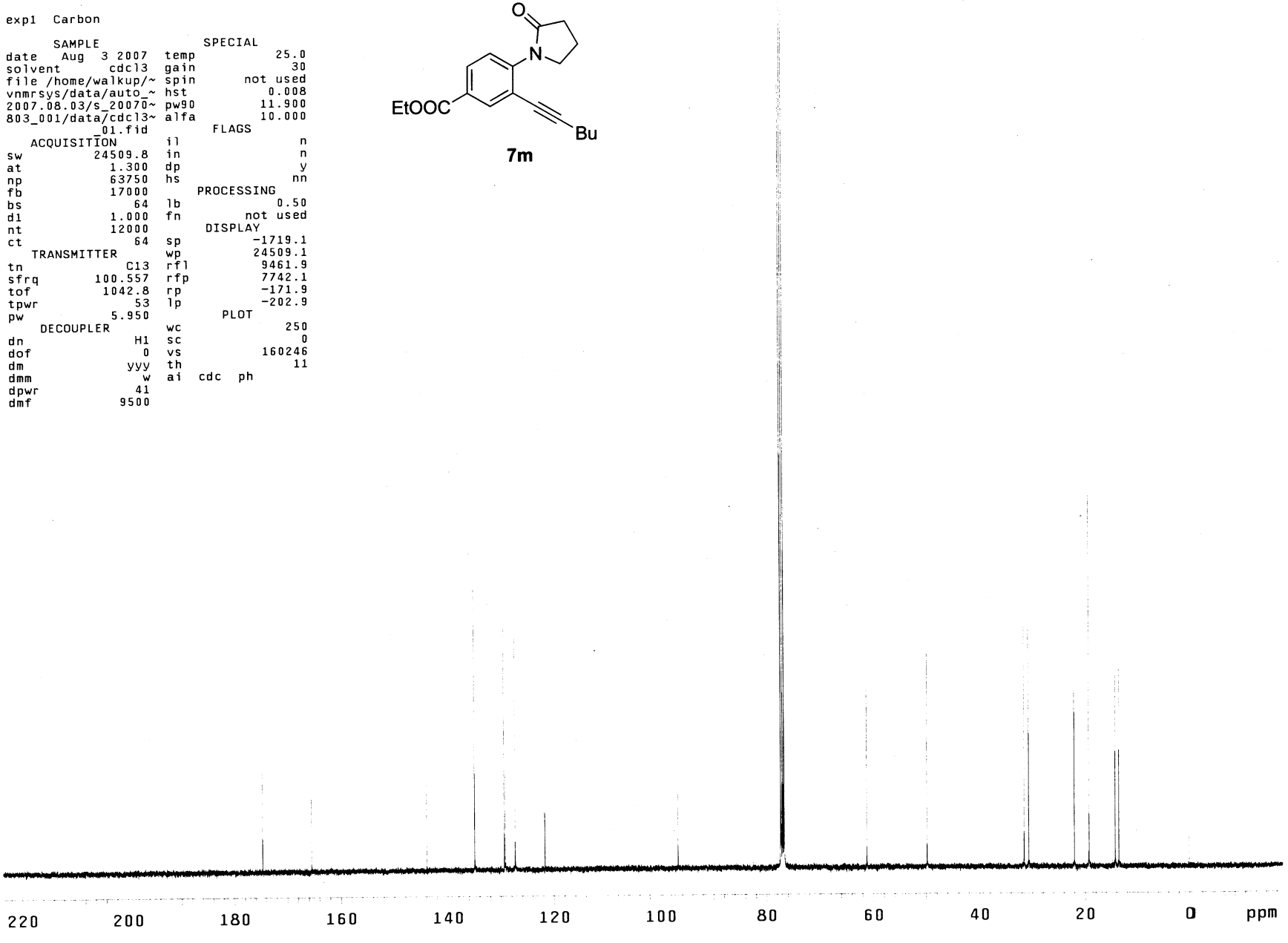
7m



exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 3 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.03/s_20070~	pw90	11.900
	803_001/data/cdcl3~	alfa	10.000
	_01.fid	FLAGS	
ACQUISITION		il	n
sw	24509.8	in	n
at	1.300	dp	y
np	63750	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	12000	DISPLAY	
ct	64	sp	-1719.1
TRANSMITTER		wp	24509.1
tn	C13	rfl	9461.9
sfrq	100.557	rfp	7742.1
tof	1042.8	rp	-171.9
tpwr	53	lp	-202.9
pw	5.950	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	160246
dm	yyy	th	11
dmm	w	ai	cdc ph
dpwr	41		
dmf	9500		

7m

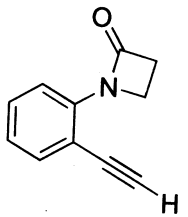


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.01_34
File : s_20070601_001/data/cdcl3_02.fid
Sample id : s_20070601_001

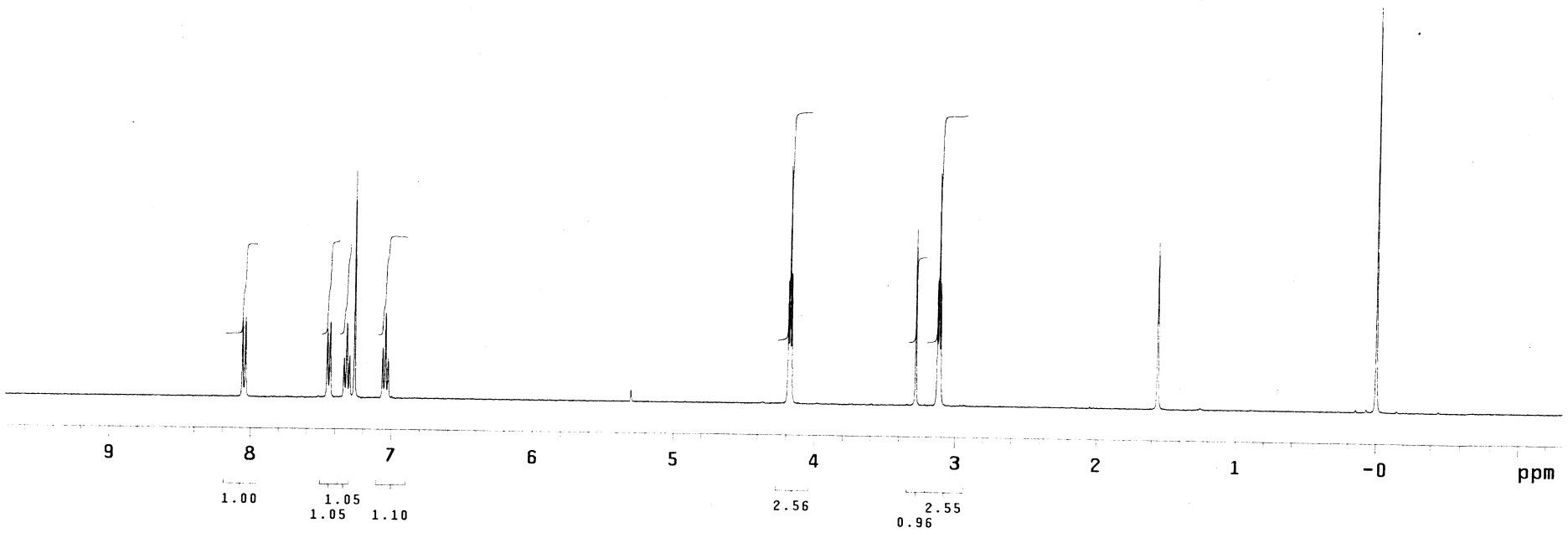
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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, 399.8656717 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



10

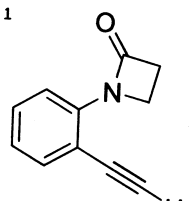


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.10_01
File : Aug.02/data/cdcl3_01.fid
Sample id : Aug.02

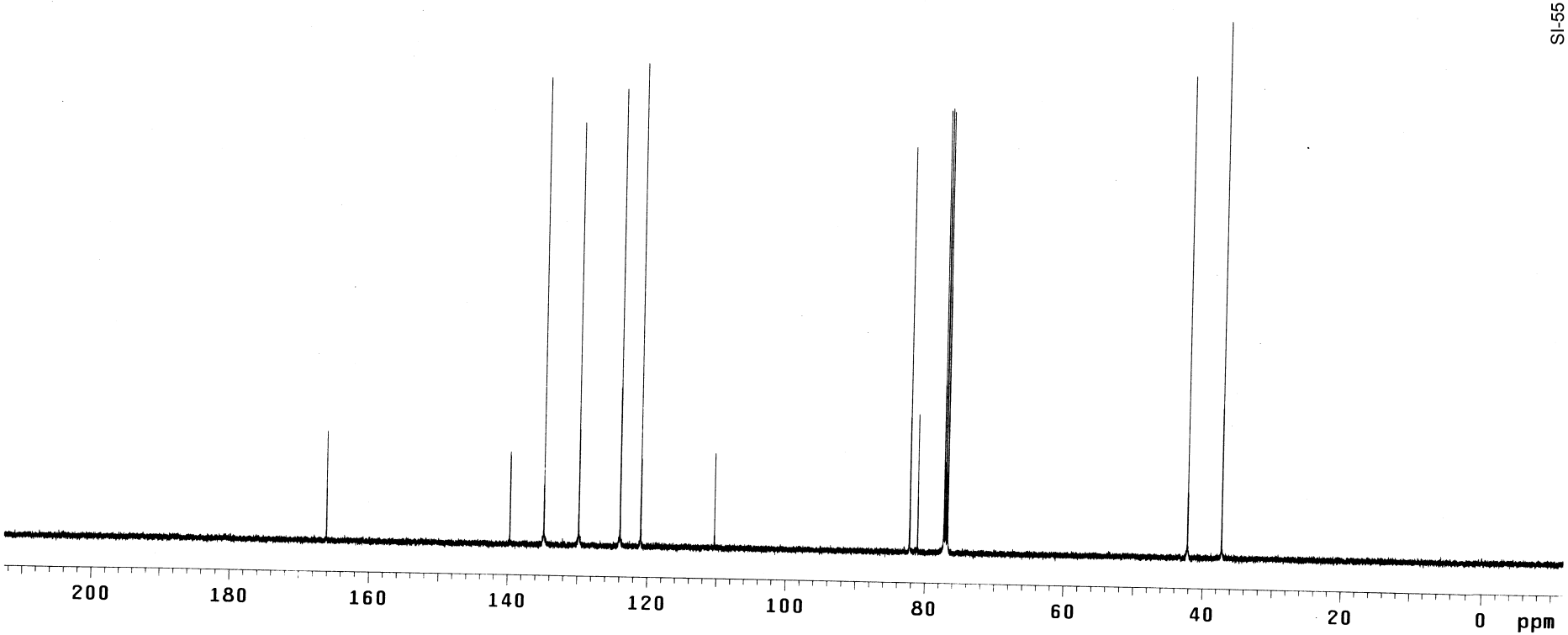
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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, 125.6746077 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



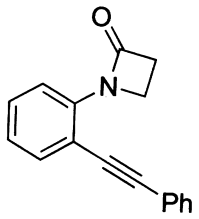
10

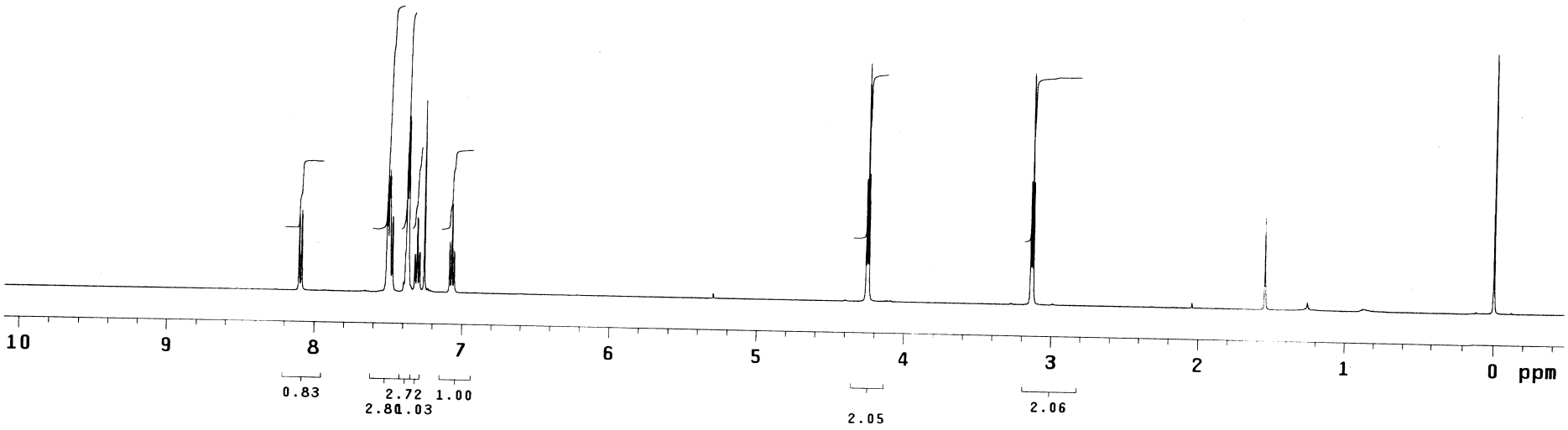


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_15
File : Aug.01/data/cdcl3_01.fid
Sample id : Aug.01

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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 65536
Total time 0 min, 30 sec

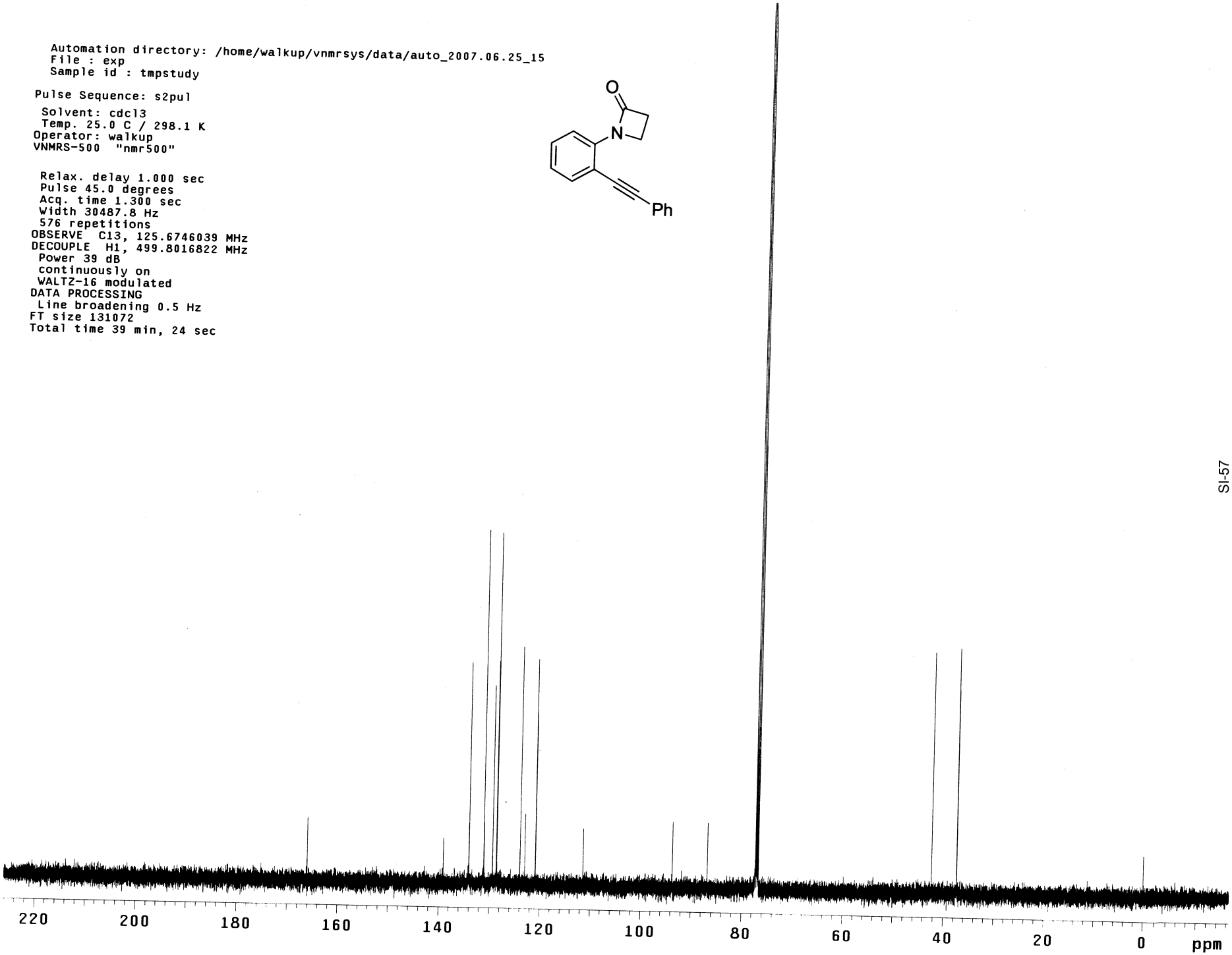




Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_15
File : exp
Sample id : tmpstudy

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

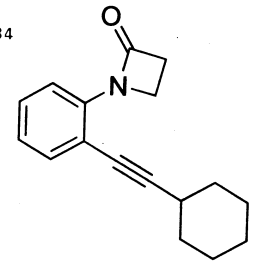
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
576 repetitions
OBSERVE C13, 125.6746039 MHz
DECOUPLE H1, 499.8016822 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec

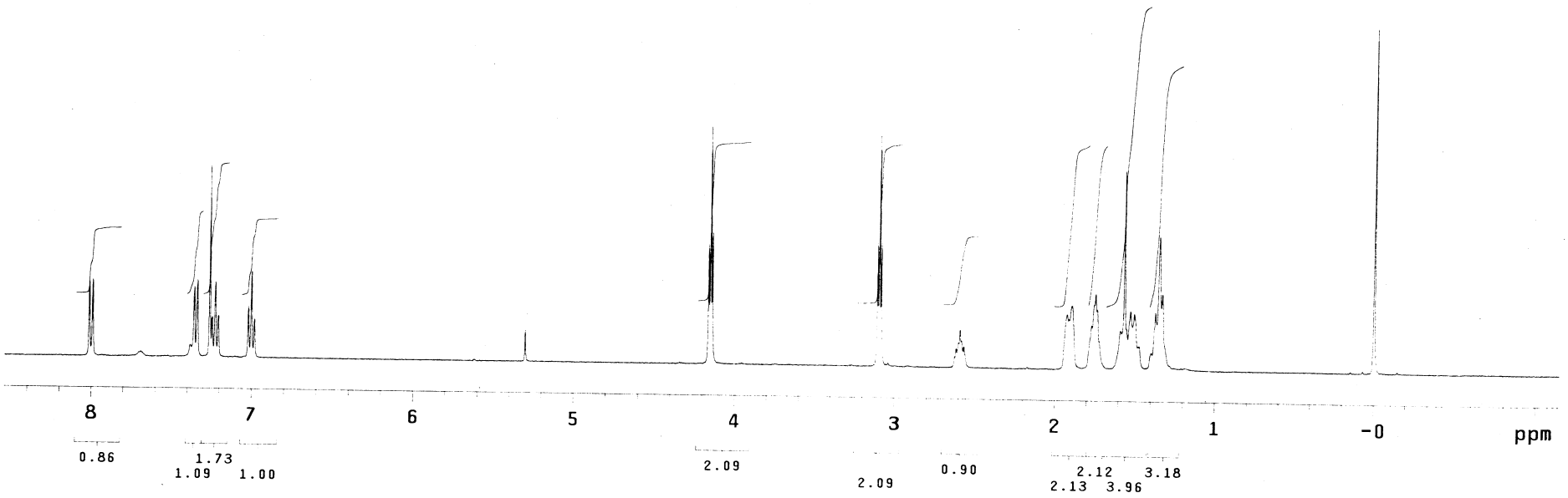


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.01_34
File : s_20070601_003/data/cdcl3_03.fid
Sample id : s_20070601_003

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_03
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, 399.8656715 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

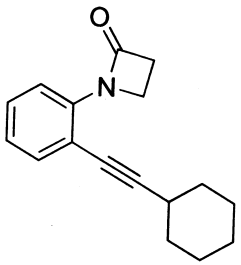


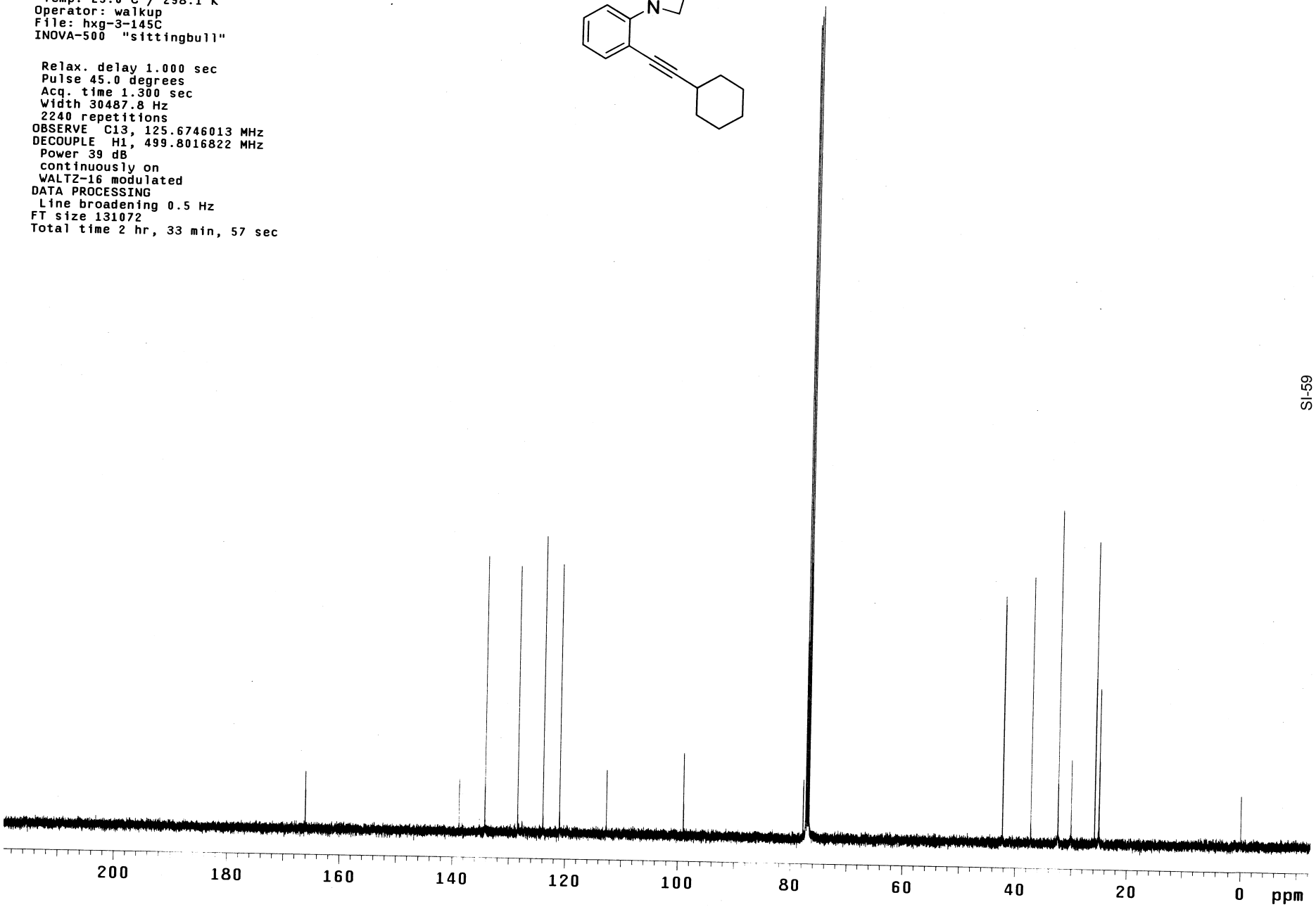


File: hxg-3-145C

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: hxg-3-145C
INOVA-500 "sittingbull"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
2240 repetitions
OBSERVE C13, 125.6746013 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 2 hr, 33 min, 57 sec



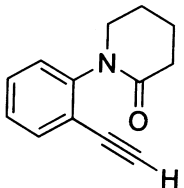


File: hxg-3-159H

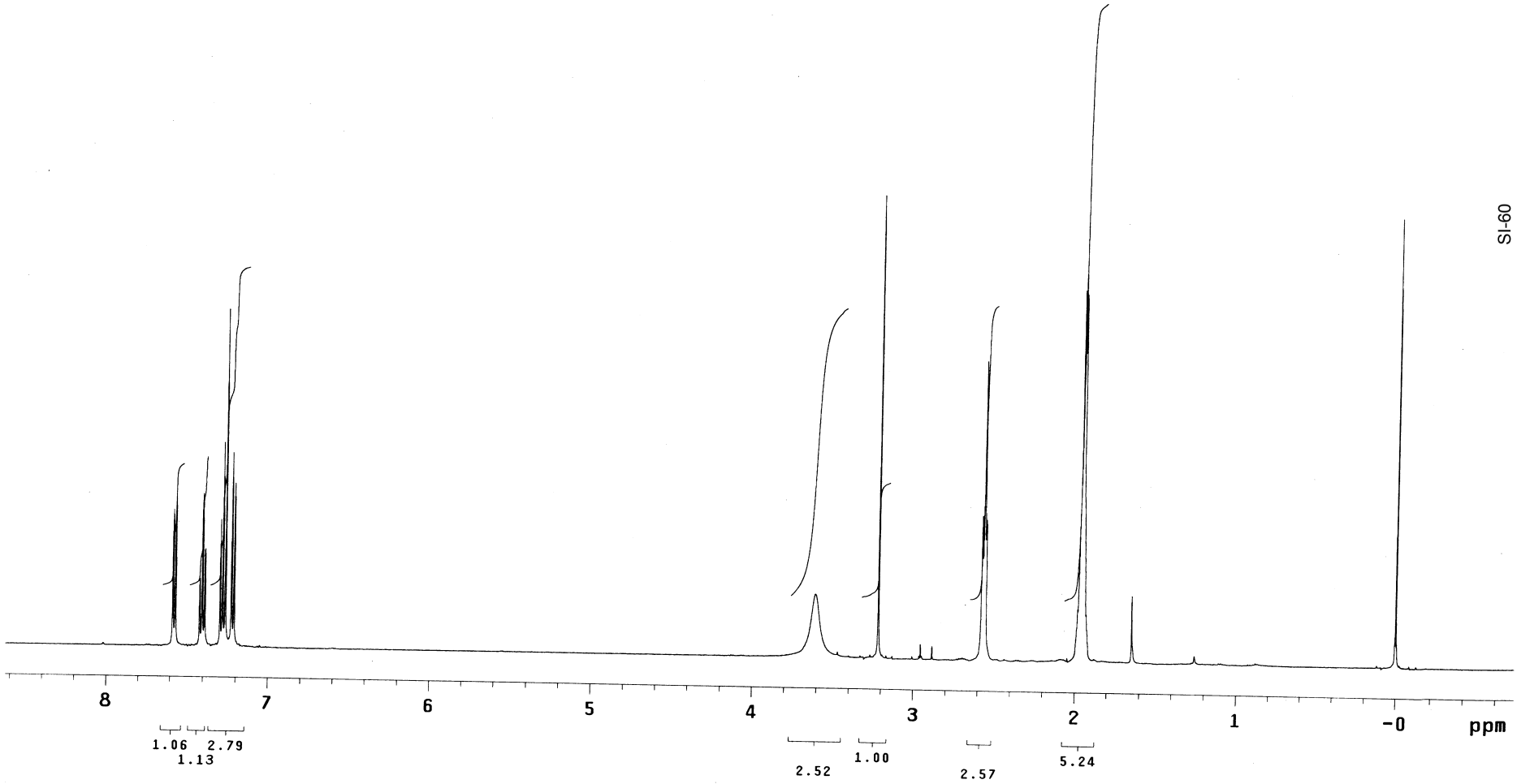
Pulse Sequence: s2pul

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



12



SI-60

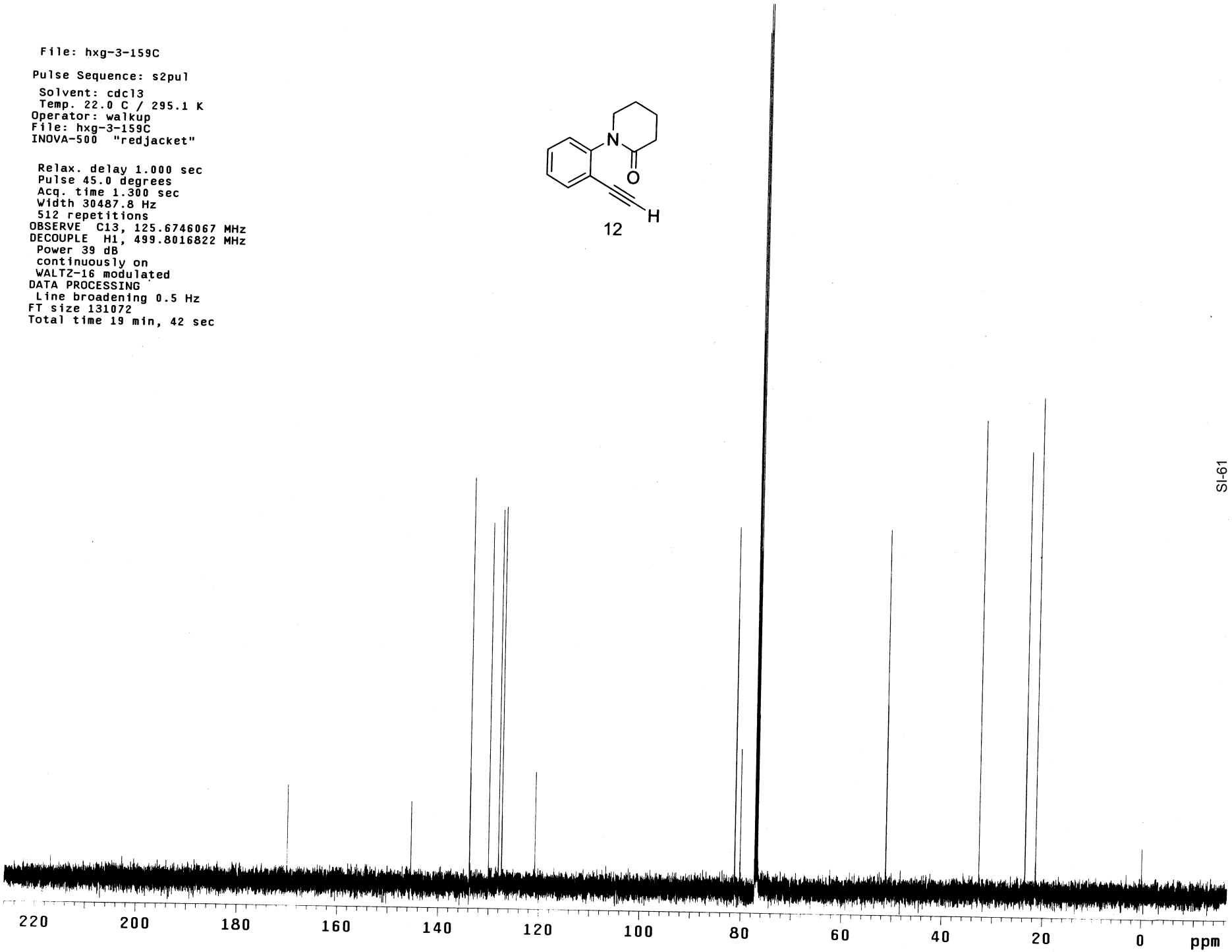
File: hxg-3-159C

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: walkup
File: hxg-3-159C
INOVA-500 "redjacket"

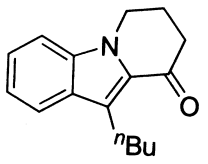
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
512 repetitions
OBSERVE C13, 125.6746067 MHz
DECOUPLE H1, 499.8016822 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 min, 42 sec

12

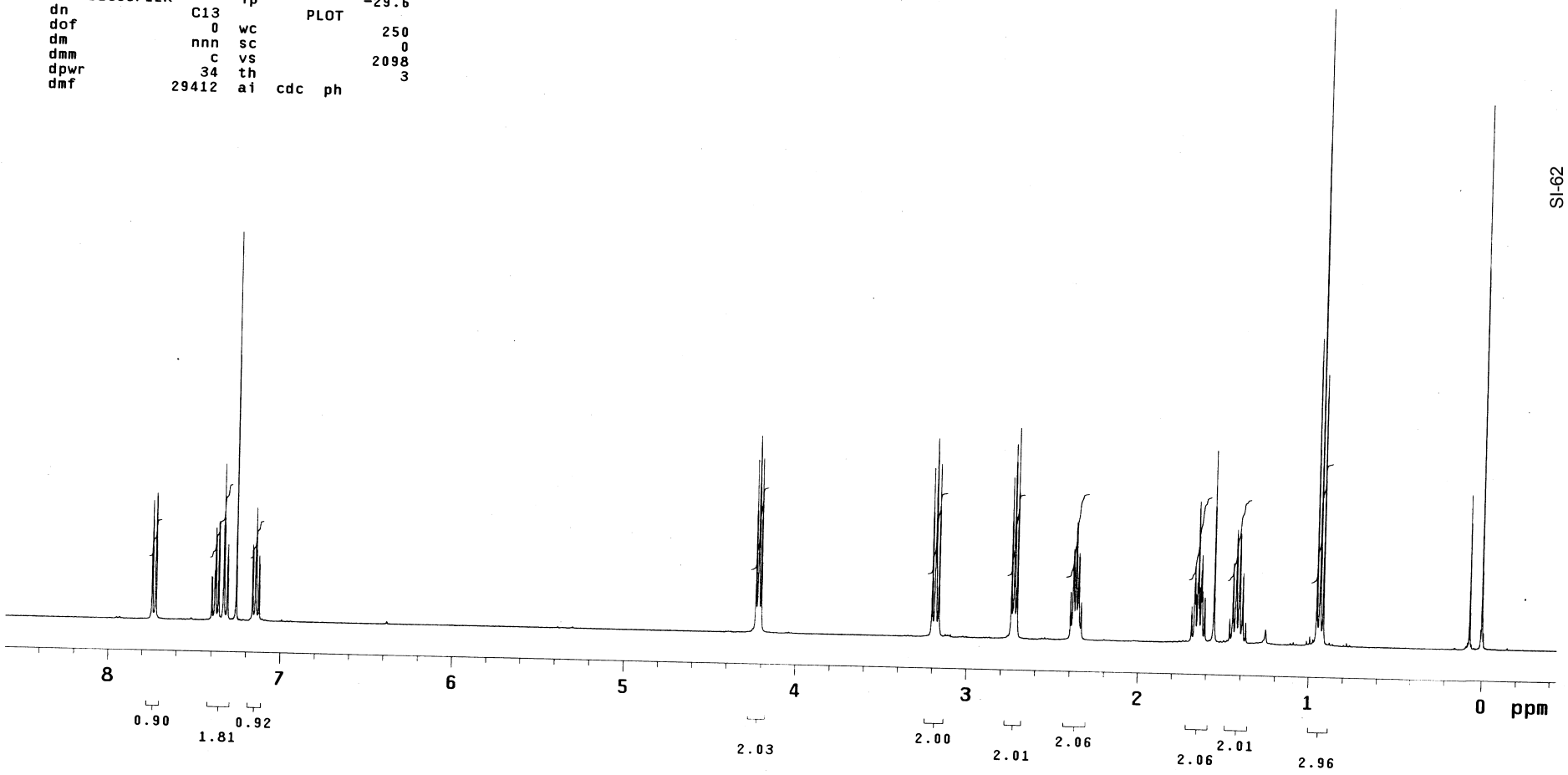


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 8 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K102-1.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-176.3
sfrq	399.868	wp	3614.2
tof	399.6	rfl	799.9
tpwr	59	rfp	0
pw	6.750	rp	-96.4
DECOUPLER		lp	-29.6
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	2098
dpwr	34	th	3
dmf	29412	ai cdc ph	



4

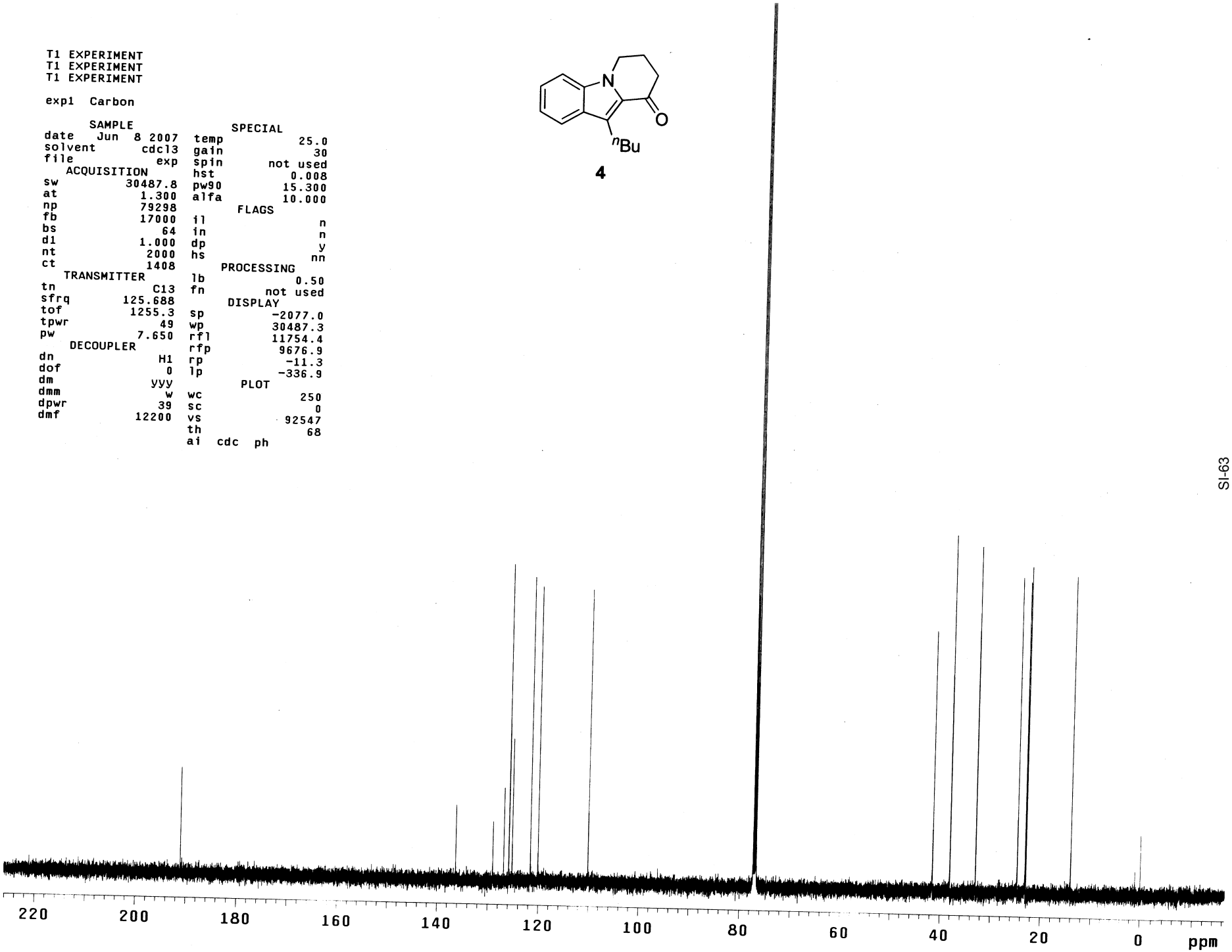


T1 EXPERIMENT
T1 EXPERIMENT
T1 EXPERIMENT

exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 8 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	64	in	n
d1	1.000	dp	y
nt	2000	hs	nn
ct	1408	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2077.0
tpwr	49	wp	30487.3
pw	7.650	rfl	11754.4
DECOUPLER		rfp	9676.9
dn	H1	rp	-11.3
dof	0	lp	-336.9
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	92547
		th	68
		ai cdc ph	

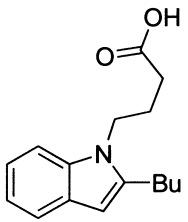
4



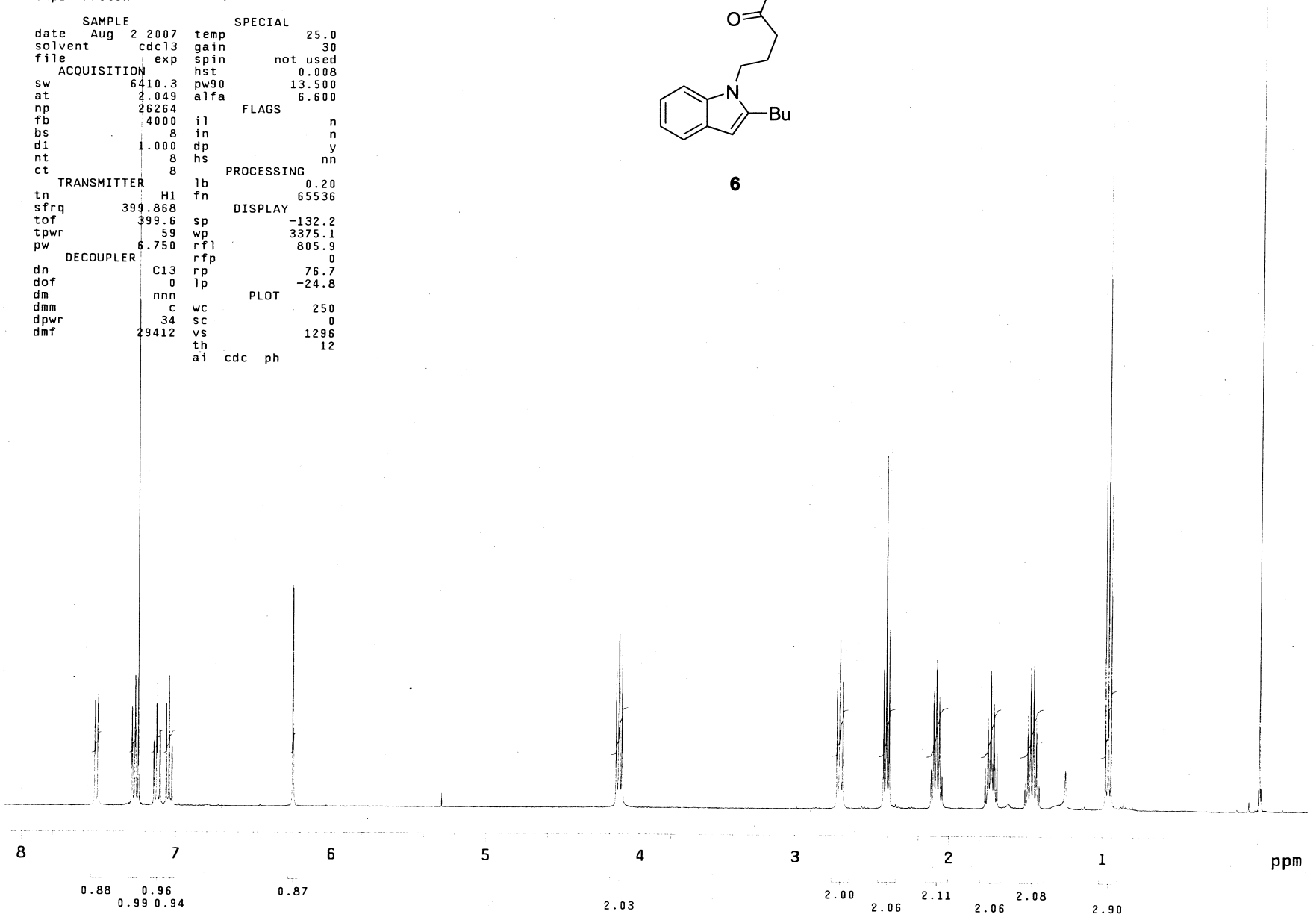
STANDARD 1H OBSERVE - profile

exp1 Proton

SAMPLE		SPECIAL	
date	Aug 2 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	6410.3	pw90	13.500
at	2.049	alfa	6.600
np	26264	FLAGS	
fb	4000	il	n
bs	8	in	n
d1	1.000	dp	y
nt	8	hs	nn
ct	8	PROCESSING	
TRANSMITTER		lb	0.20
tn	H1	fn	65536
sfrq	399.868	DISPLAY	
tof	399.6	sp	-132.2
tpwr	59	wp	3375.1
pw	6.750	rfl	805.9
DECOUPLER		rfp	0
dn	C13	rp	76.7
dof	0	lp	-24.8
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	34	sc	0
dmf	29412	vs	1296
		th	12
		ai cdc ph	



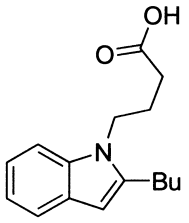
6



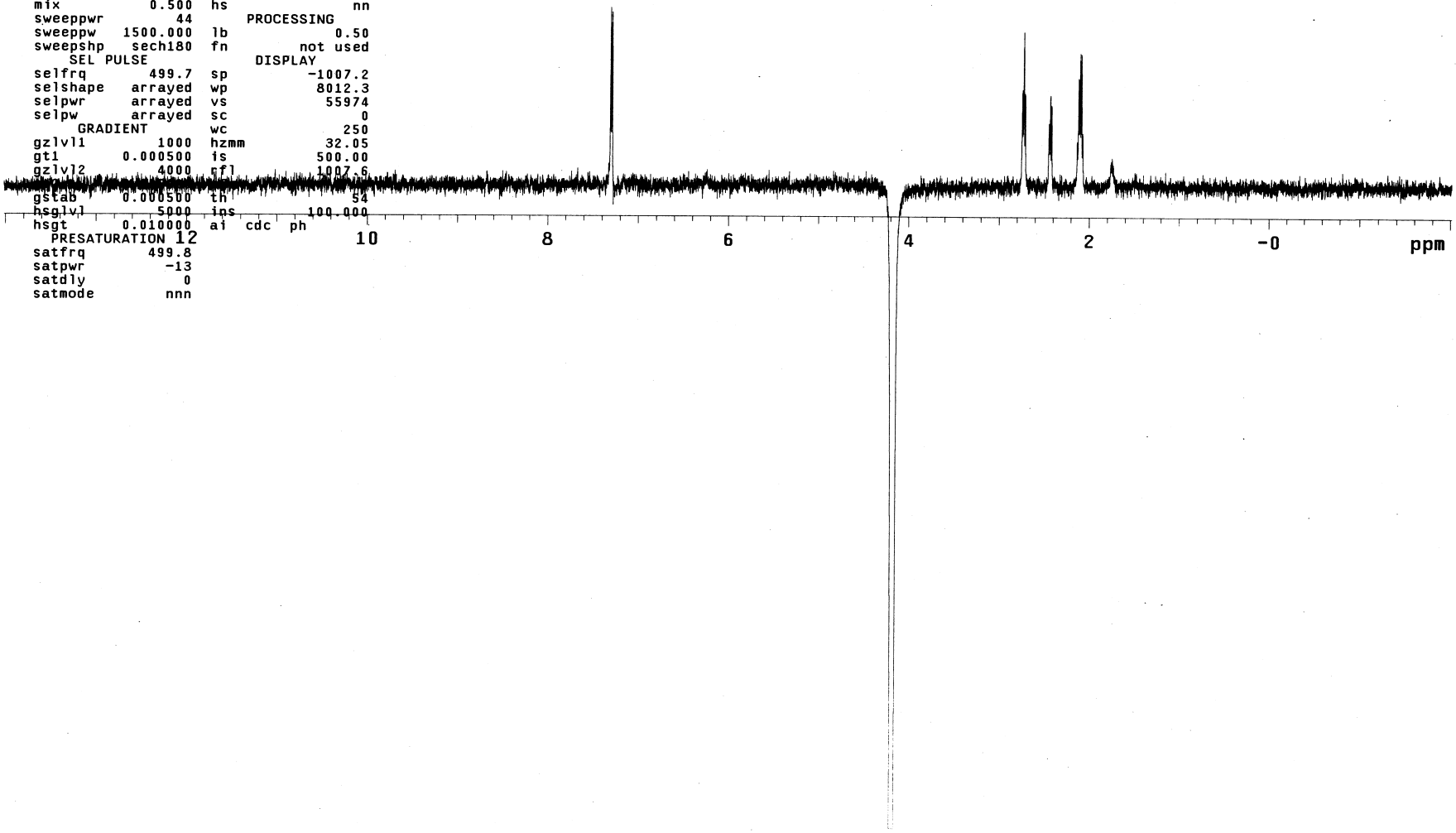
STANDARD 1H OBSERVE - profile

expl Noesy1d

ACQUISITION		DECOUPLER		ACQUISITION ARRAYS	
sw	8012.8	dn	C13	array (selpw,selpw~	
at	1.998	dm	nnn	r,selshape)	
np	32018	SAMPLE		arraydim	2
fb	2600	date	Aug 2 2007		
bs	4	solvent	cdcl3	i	selpw
ss	-2	file	exp	1	44957.1
d1	1.000	SPECIAL		2	44957.1
nt	10240	temp	25.0		
ct	64	gain	30	i	selpwr
TRANSMITTER		spin	not used	1	7
tn	H1	pw90	14.700	2	7
sfrq	499.802	FLAGS			
tof	499.7	sspul	y	i	selshape
tpwr	54	il	n	1	NOESY1D_4_16p
pw	14.700	in	n	2	NOESY1D_4_16p
NOESY		dp	y		
mix	0.500	hs	nn		
sweeppwr	44	PROCESSING			
sweeppw	1500.000	lb	0.50		
sweepshp	sech180	fn	not used		
SEL PULSE		DISPLAY			
selfrq	499.7	sp	-1007.2		
selshape	arrayed	wp	8012.3		
selpwr	arrayed	vs	55974		
selpw	arrayed	sc	0		
GRADIENT		wc	250		
gzlvl1	1000	hzmm	32.05		
gt1	0.000500	is	500.00		
gzlvl2	4000	rfl	1007.6		
[illegible]		[illegible]			
gstab	0.000500	th	54		
hsglvl	5000	ins	100.000		
hsgt	0.010000	ai cdc ph			
PRESATURATION					
satfrq	499.8				
satpwr	-13				
satdly	0				
satmode	nnn				



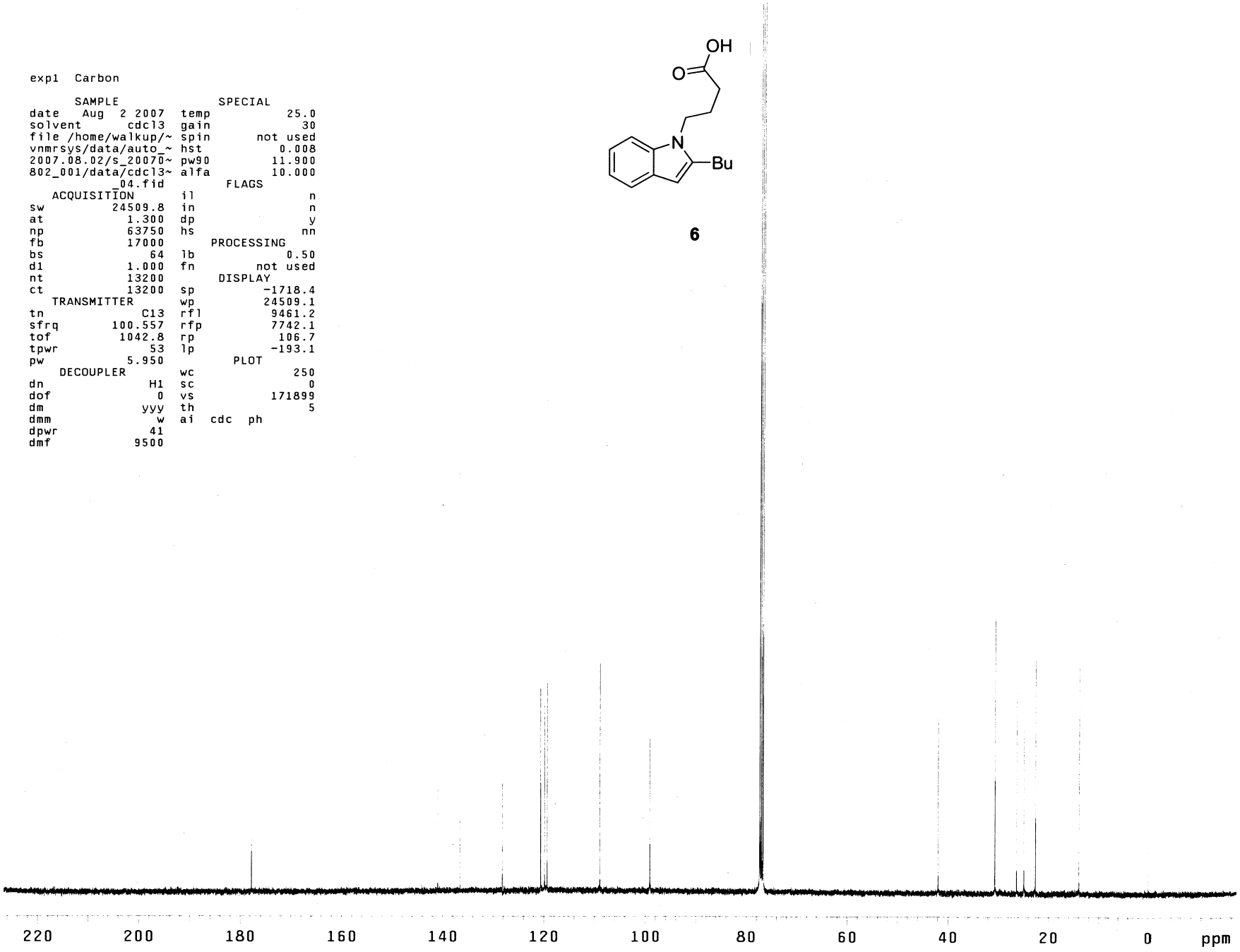
6



exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 2 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.02/s_20070~	pw90	11.900
	802_001/data/cdcl3~	alfa	10.000
	04.fid	FLAGS	
ACQUISITION		il	n
sw	24509.8	in	n
at	1.300	dp	y
np	63750	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	13200	DISPLAY	
ct	13200	sp	-1718.4
TRANSMITTER		wp	24509.1
tn	C13	rfl	9461.2
sfrq	100.557	rfp	7742.1
tof	1042.8	rp	106.7
tpwr	53	lp	-193.1
pw	5.950	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	171899
dm	yyy	th	5
dmm	w	ai cdc ph	
dpwr	41		
dmf	9500		

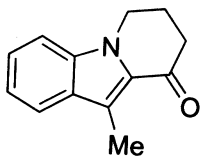
6



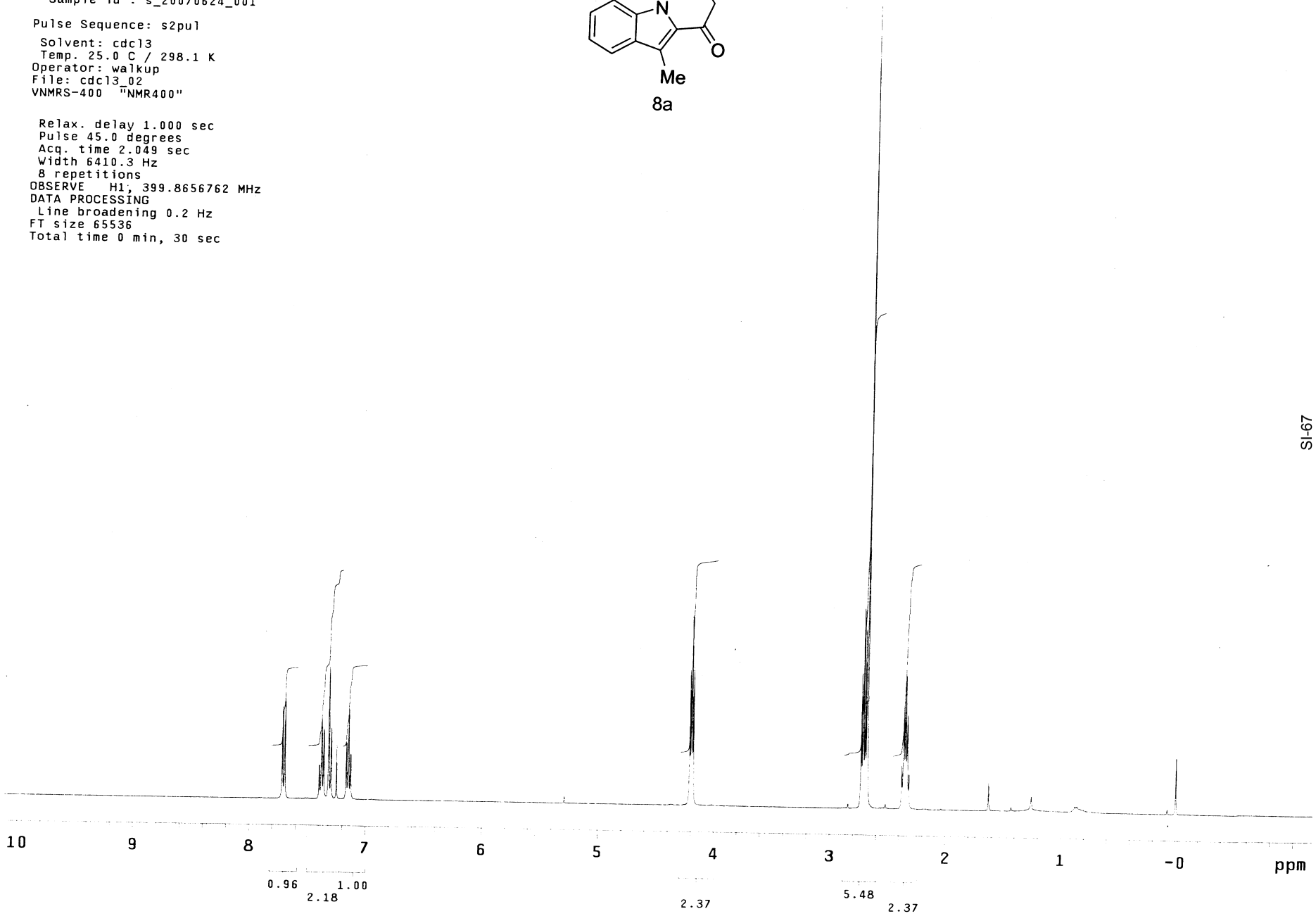
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.24_08
File : s_20070624_001/data/cdcl3_02.fid
Sample id : s_20070624_001

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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



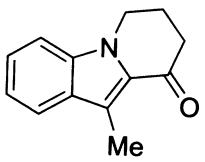
8a



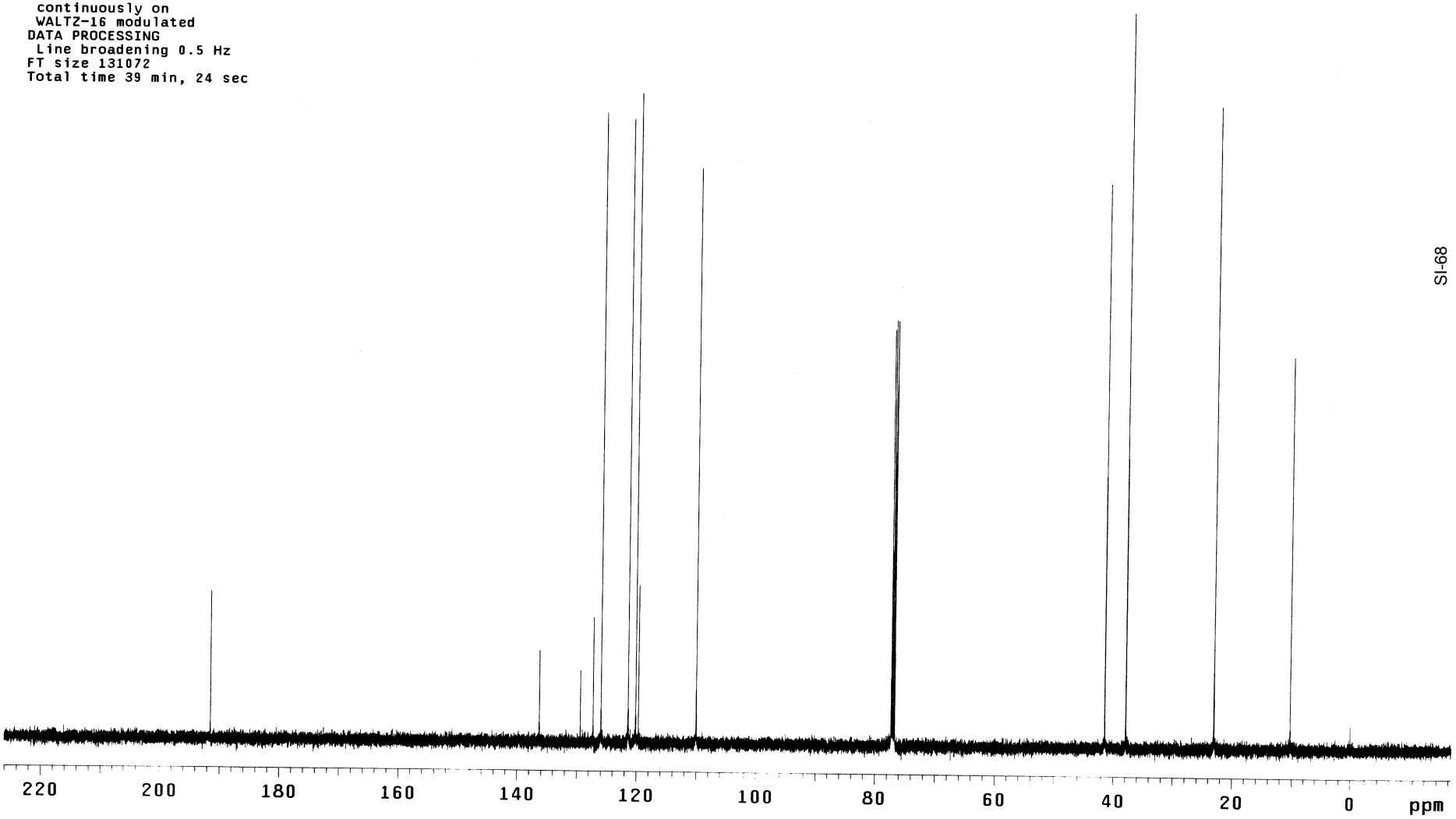
File: hxg-4-27C

Pulse Sequence: s2pul
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 30487.8 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

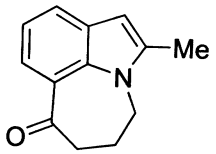


8a

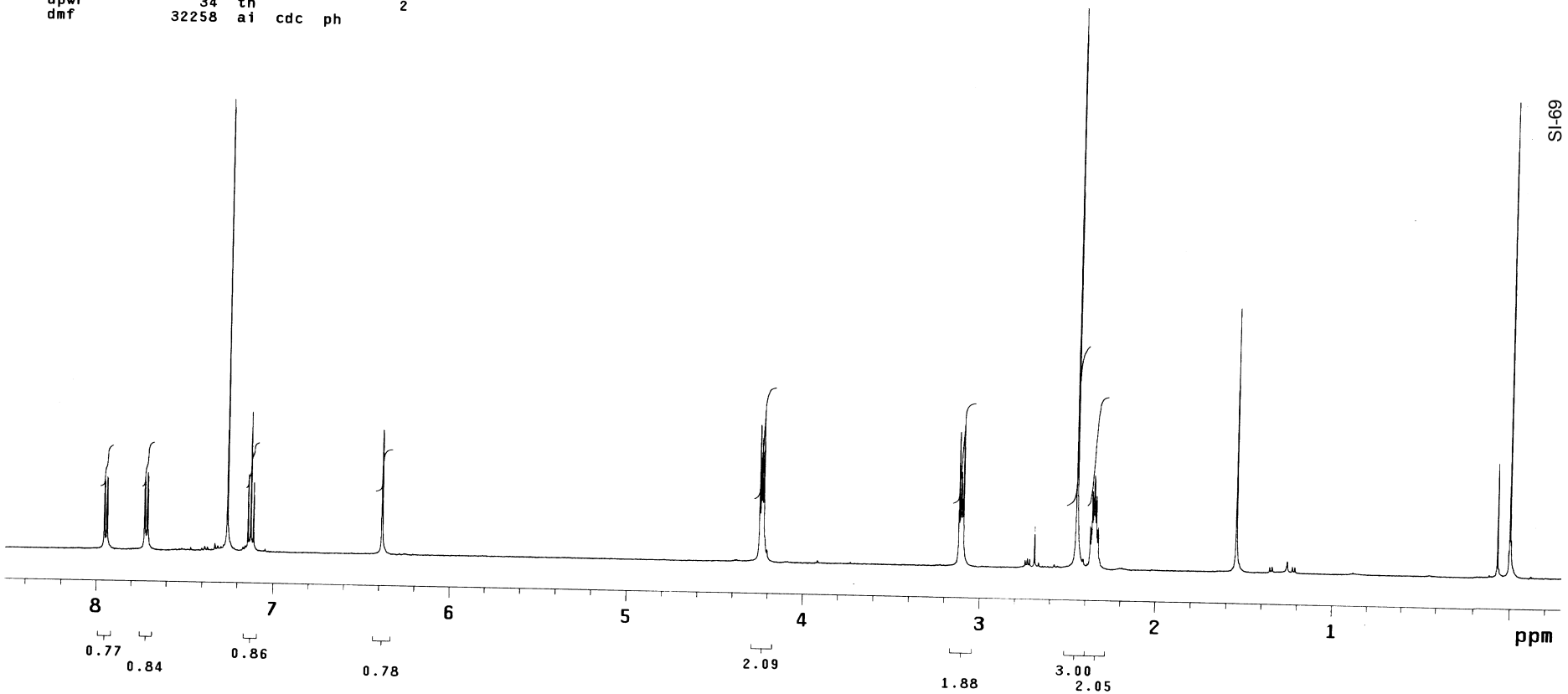


exp5 Proton

SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr500/w~	spin	not used
alkup/data/Zhang/L~		hst	0.008
	i/K188-2.fid	pw90	14.700
ACQUISITION		alfa	6.600
sw	8012.8	FLAGS	
at	2.049	il	n
np	32830	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-145.7
sfrq	499.802	wp	4401.3
tof	499.8	rfl	1008.2
tpwr	54	rfp	0
pw	7.350	rp	98.1
DECOUPLER		lp	-30.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	520
dpwr	34	th	2
dmf	32258	ai cdc ph	



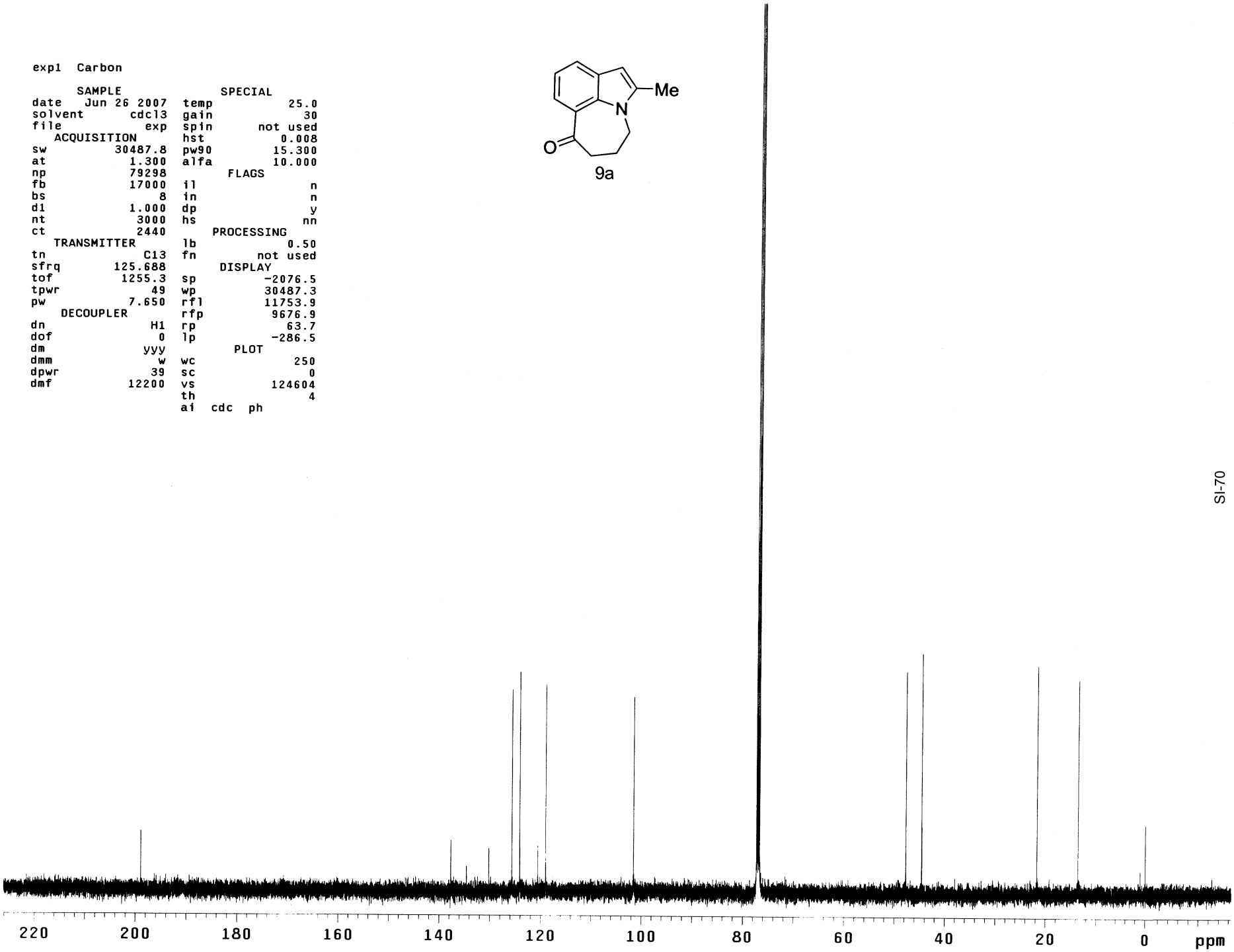
9a



exp1 Carbon

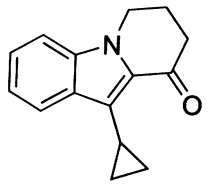
SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	FLAGS	
fb	17000	il	n
bs	8	in	n
d1	1.000	dp	y
nt	3000	hs	nn
ct	2440	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	DISPLAY	
tof	1255.3	sp	-2076.5
tpwr	49	wp	30487.3
pw	7.650	rfl	11753.9
DECOUPLER		rfp	9676.9
dn	H1	rp	63.7
dof	0	lp	-286.5
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	124604
		th	4
		ai	cdc ph

9a



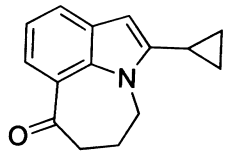
exp2 Proton

SAMPLE		SPECIAL	
date	Jun 19 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K160-1.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-149.8
sfrq	399.868	wp	3643.9
tof	399.6	rfl	803.2
tpwr	59	rfp	0
pw	6.750	rp	-19.5
DECOUPLER		lp	-27.8
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	1084
dpwr	34	th	2
dmf	29412	ai cdc ph	

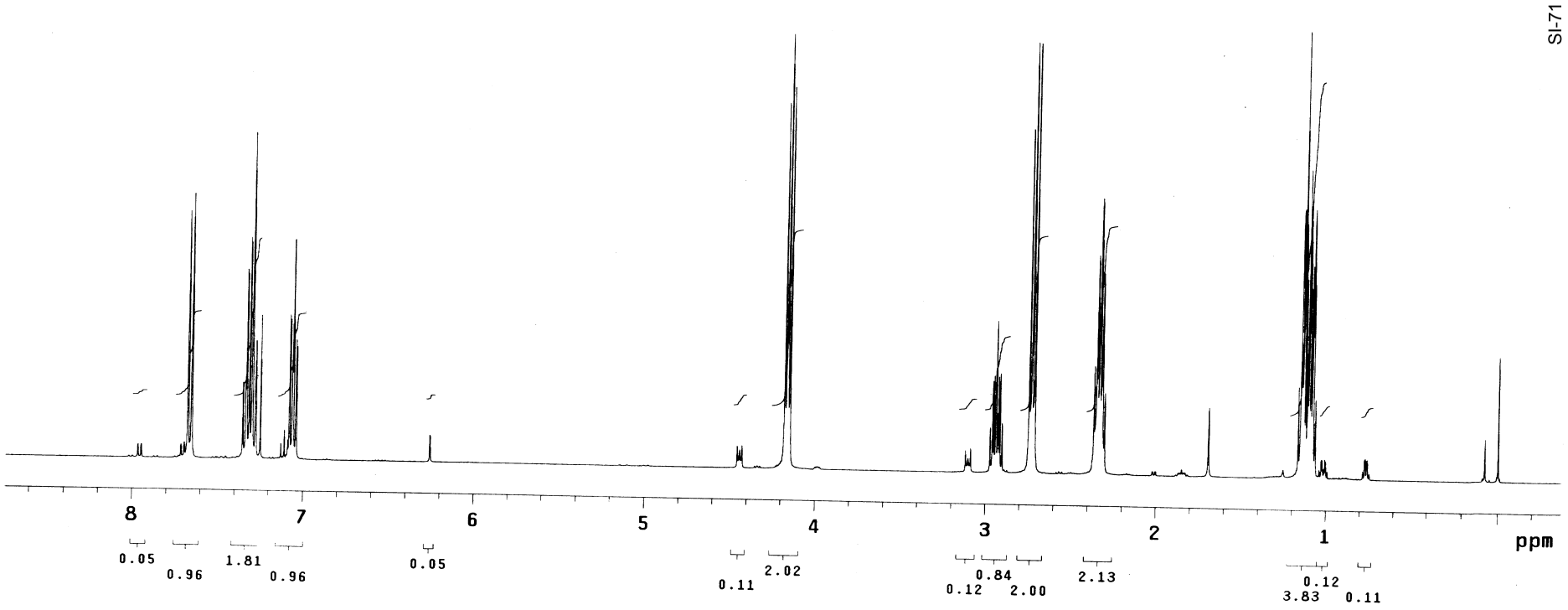


8b

and

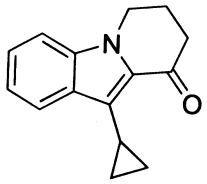


9b



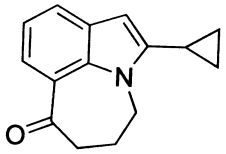
exp5 Carbon

SAMPLE		SPECIAL	
date	Jun 19 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr400/w~	spin	20
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K160-1carb~	pw90	11.900
	on.fid	alfa	10.000
ACQUISITION		**FLAGS**	
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	32	**PROCESSING**	
d1	1.000	lb	0.50
nt	4000	fn	not used
ct	1408	**DISPLAY**	
TRANSMITTER		sp	-1725.1
tn	C13	wp	24509.1
sfrq	100.557	rfl	9467.9
tof	1042.8	rfp	7742.1
tpwr	53	rp	175.8
pw	5.950	lp	-196.9
DECOUPLER		**PLOT**	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	87771
dmm	w	th	8
dpwr	41	ai cdc ph	
dmf	9500		

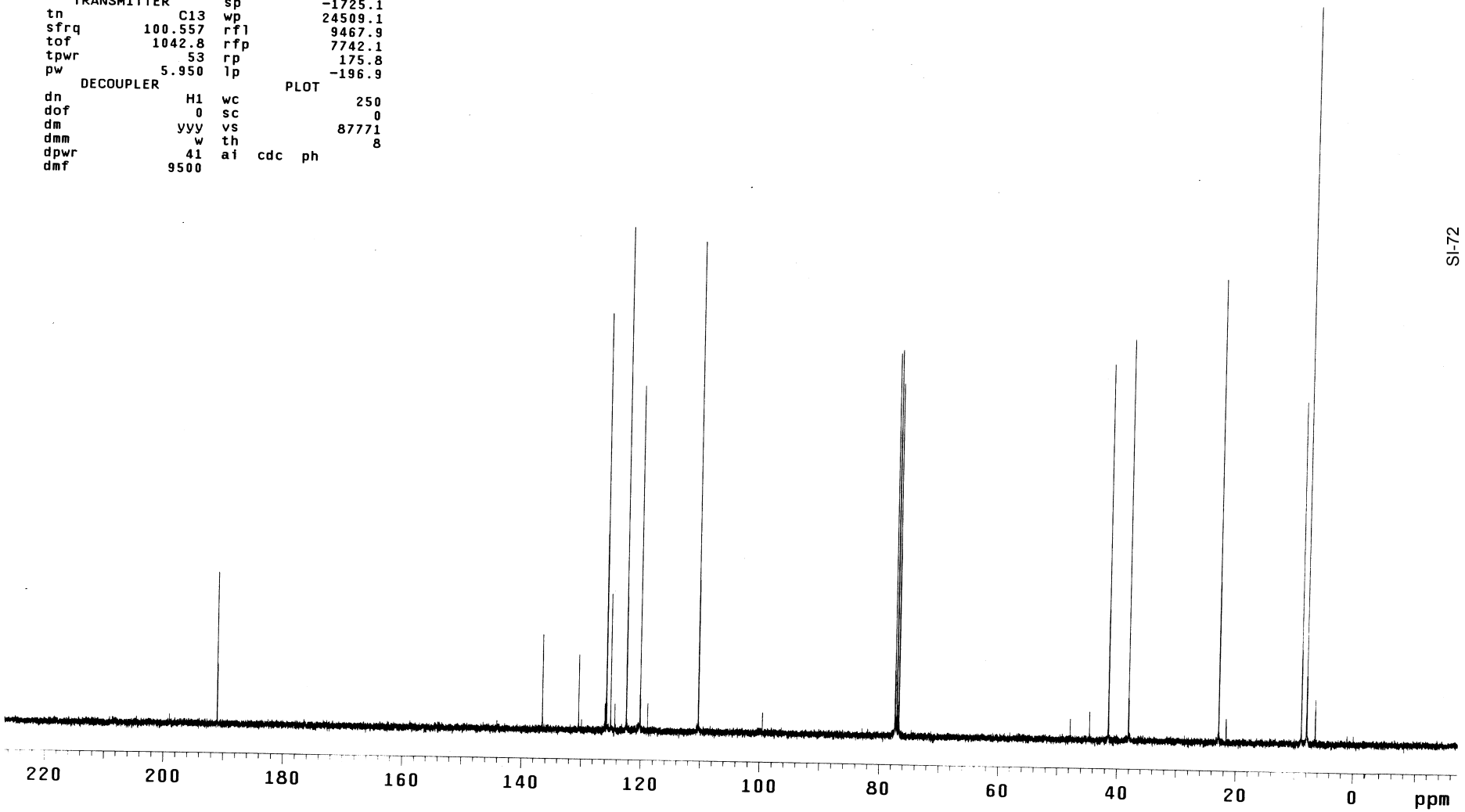


8b

and

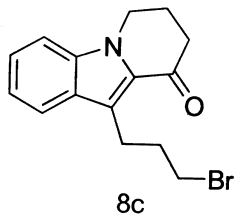


9b



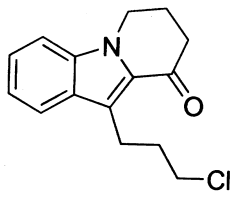
exp3 Proton

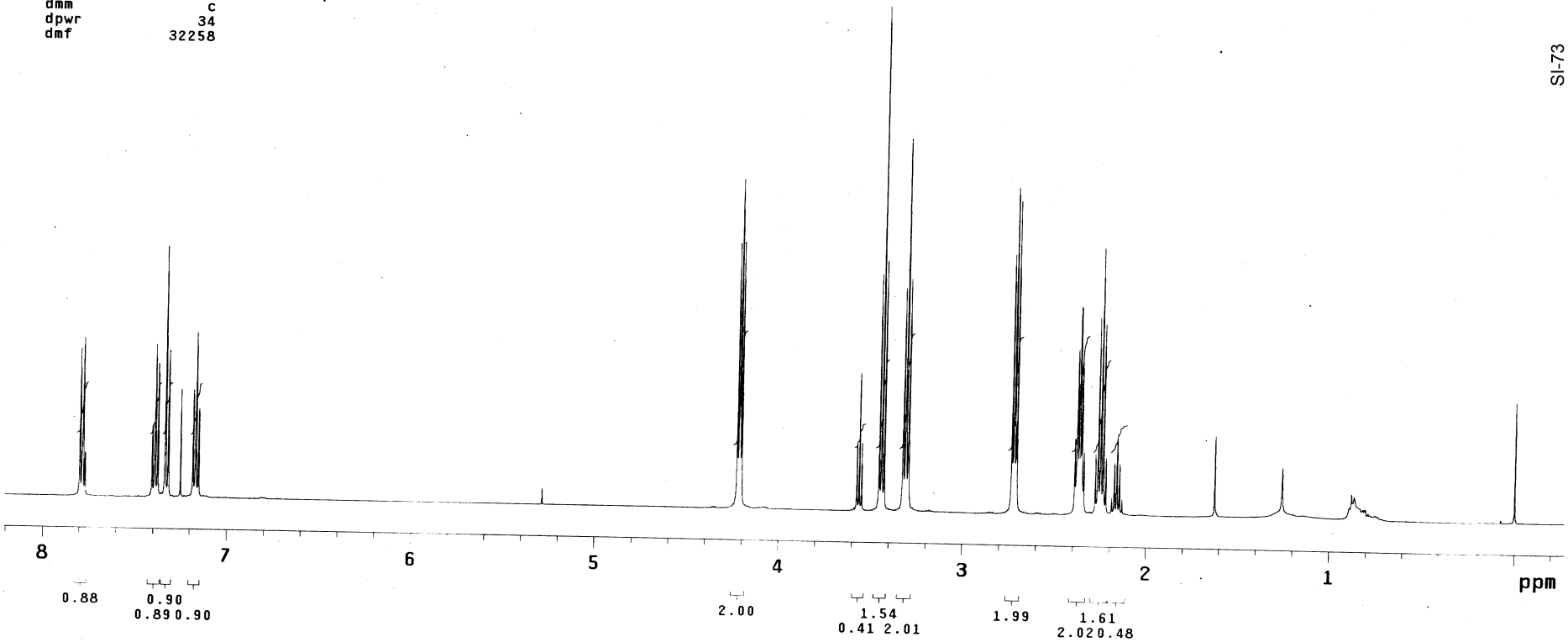
SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	not used
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.06.26_03/Aug.~	pw90	14.700
	02/data/cdcl3_01.f~	alfa	6.600
id		FLAGS	
ACQUISITION		il	n
sw	8012.8	in	n
at	2.049	dp	y
np	32830	hs	nn
fb	4000	PROCESSING	
bs	32	lb	0.20
ss	2	fn	65536
d1	1.000	DISPLAY	
nt	8	sp	-137.7
ct	8	wp	4238.5
TRANSMITTER		rfl	1011.6
tn	H1	rfp	0
sfrq	499.802	rp	96.6
tof	499.7	lp	-32.9
tpwr	54	PLOT	
pw	7.350	wc	250
DECOUPLER		sc	0
dn	C13	vs	308
dof	0	th	2
dm	nnn	ai	cdc ph
dmm	c		
dpwr	34		
dmf	32258		



8c

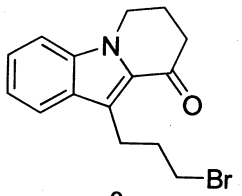
and





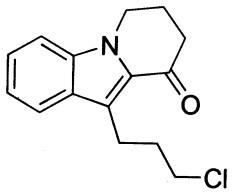
exp3 Carbon

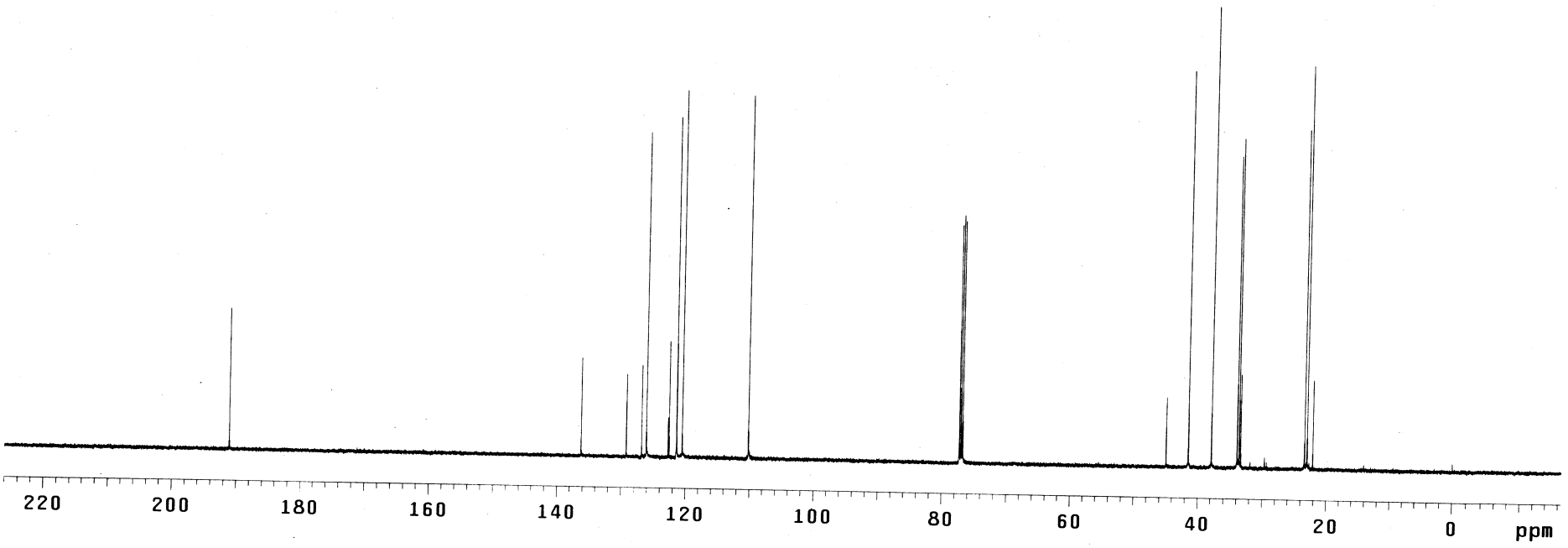
SAMPLE		SPECIAL	
date	Jul 1 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.07.01_01/Aug.~	pw90	15.300
	01/data/cdcl3_01.f~	alfa	10.000
id		FLAGS	
ACQUISITION		il	n
sw	30487.8	in	n
at	1.300	dp	y
np	79298	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	4000	DISPLAY	
ct	1536	sp	-2083.0
TRANSMITTER		wp	30487.3
tn	C13	rfl	11760.4
sfrq	125.688	rfp	9676.9
tof	1255.3	rp	156.6
tpwr	49	lp	-279.7
pw	7.650	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	17141
dm	yyy	th	2
dmm	w	ai cdc ph	
dpwr	39		
dmf	12200		

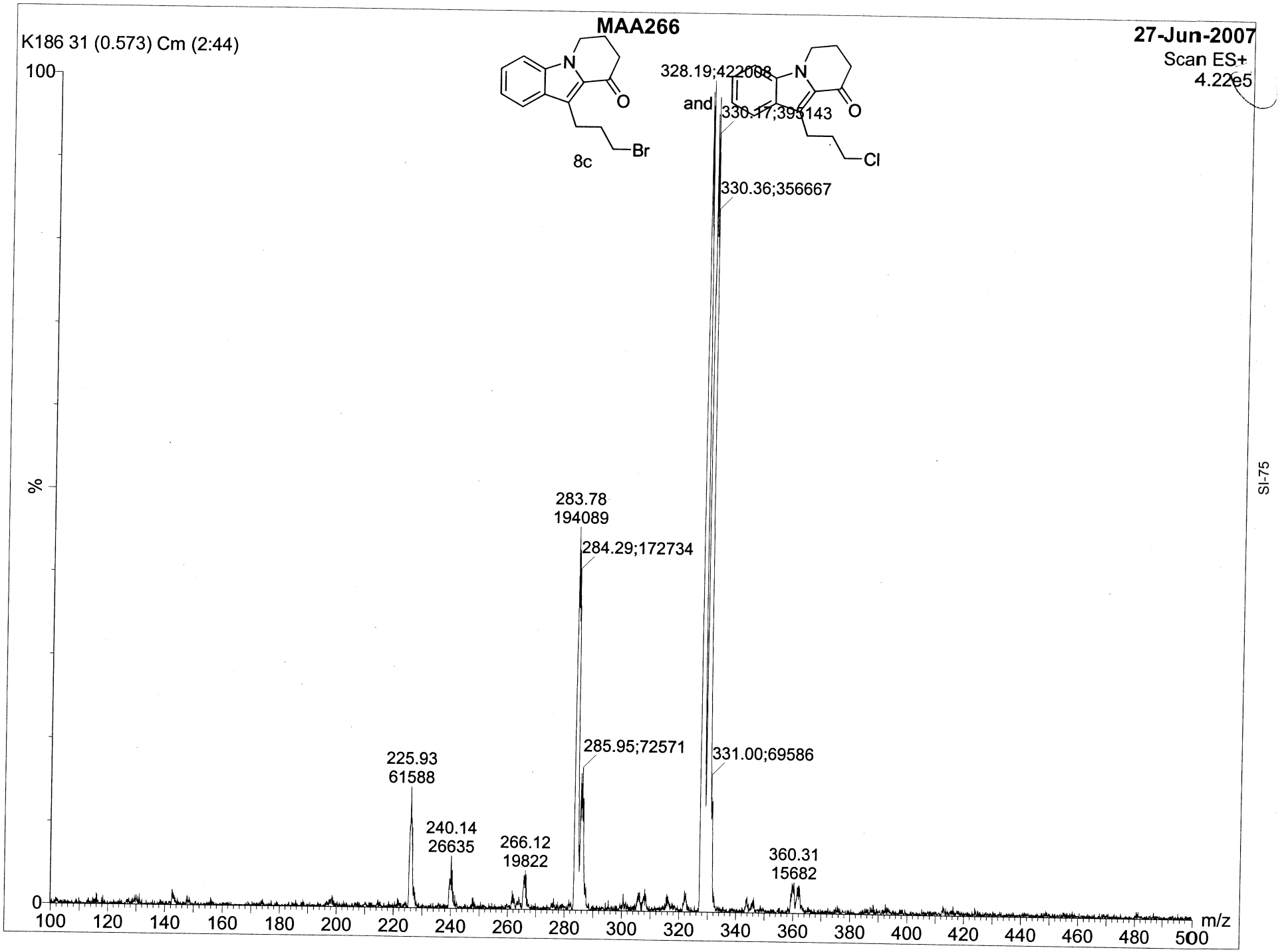


8c

and





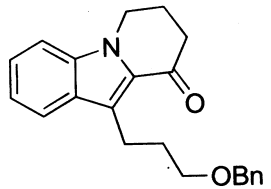

MAA266
K186 31 (0.573) Cm (2:44)
27-Jun-2007
Scan ES+
4.22e5
100
%
0
8c
Br
and
Cl
328.19;422008
330.17;395143
330.36;356667
283.78
194089
284.29;172734
285.95;72571
331.00;69586
225.93
61588
240.14
26635
266.12
19822
360.31
15682
100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500
m/z
SI-75

Automation directory: /home/walkup/vnmrsys/data/auto_2007.07.01_07
File : s_20070701_001/data/cdcl3_01.fid
Sample id : s_20070701_001

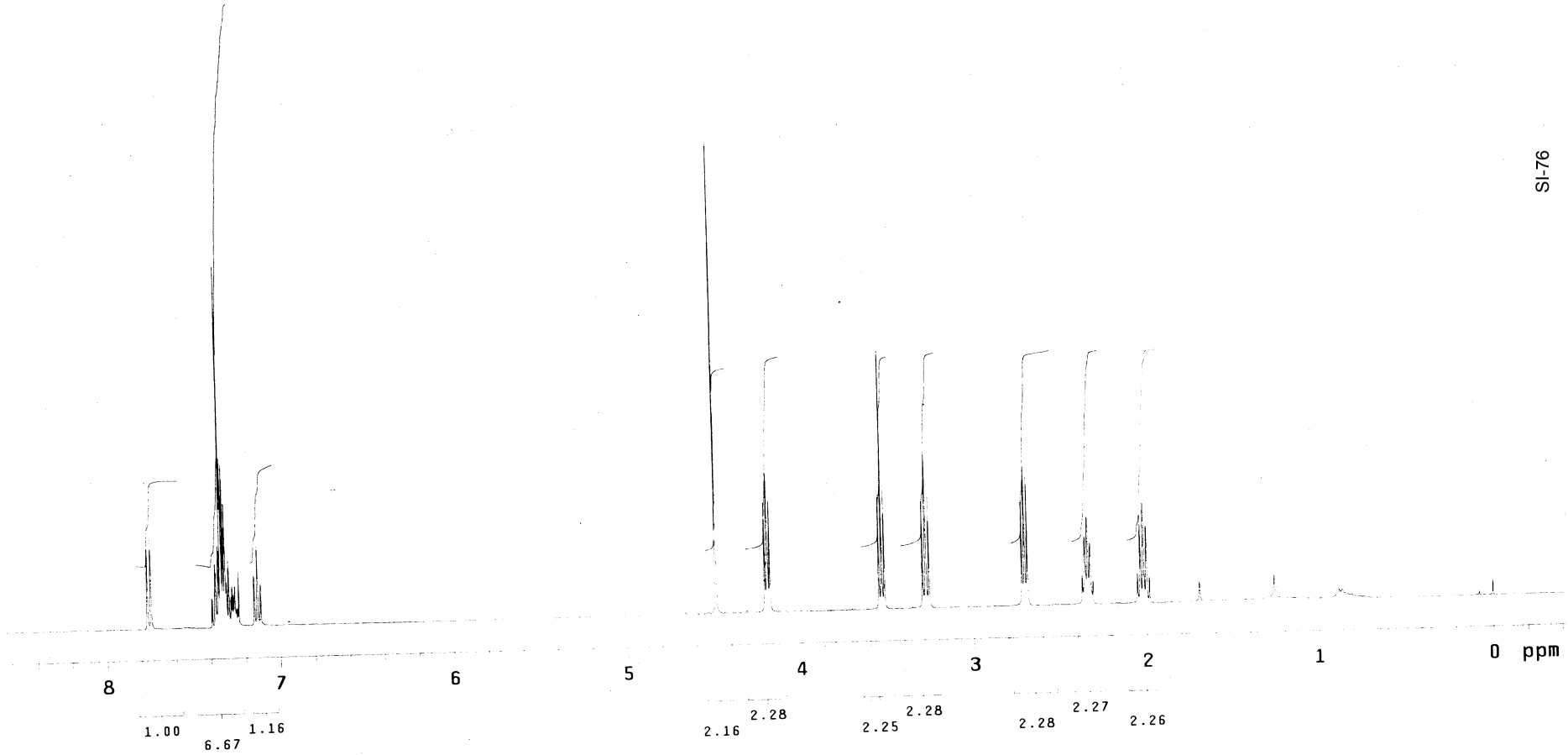
Pulse Sequence: s2pul

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 2.049 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



8d

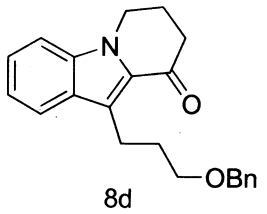


Automation directory: /home/walkup/vnmrsys/data/auto_2007.07.01_06
File : exp
Sample id : tmpstudy

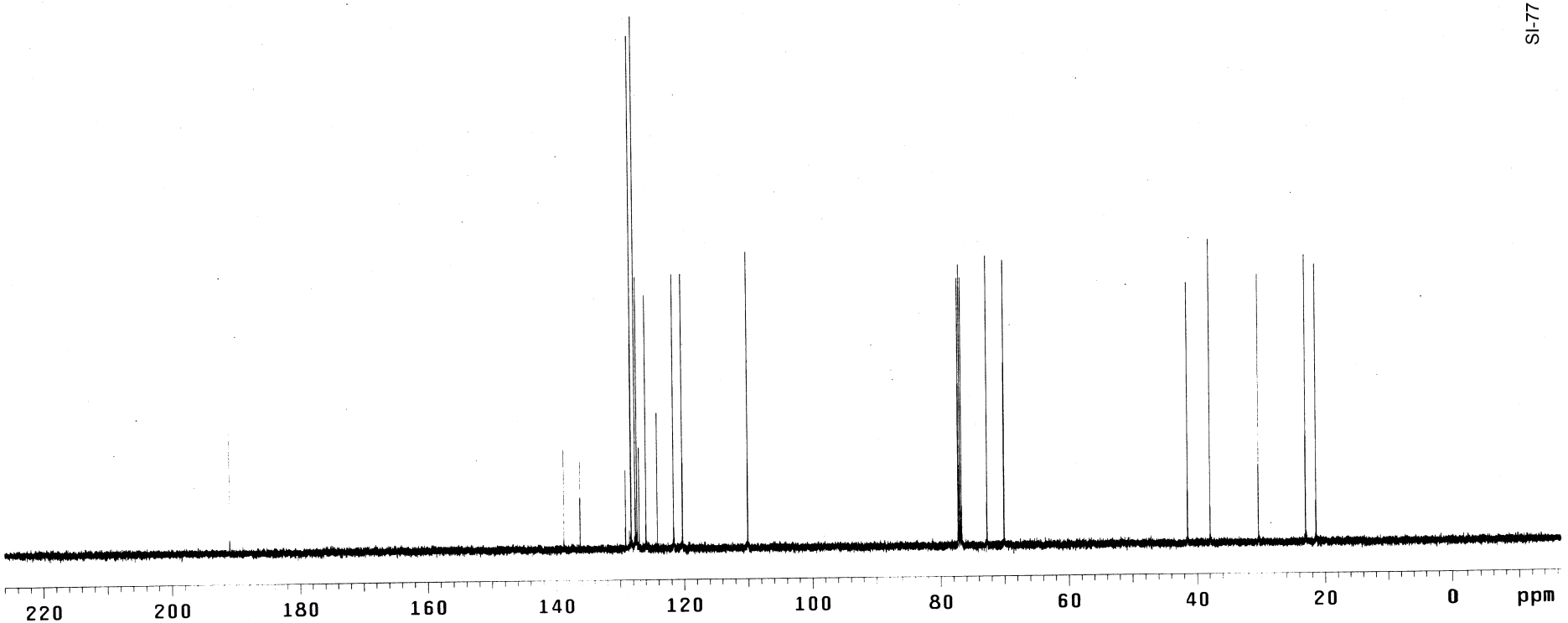
Pulse Sequence: s2pul

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 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

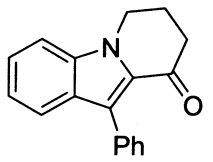


8d

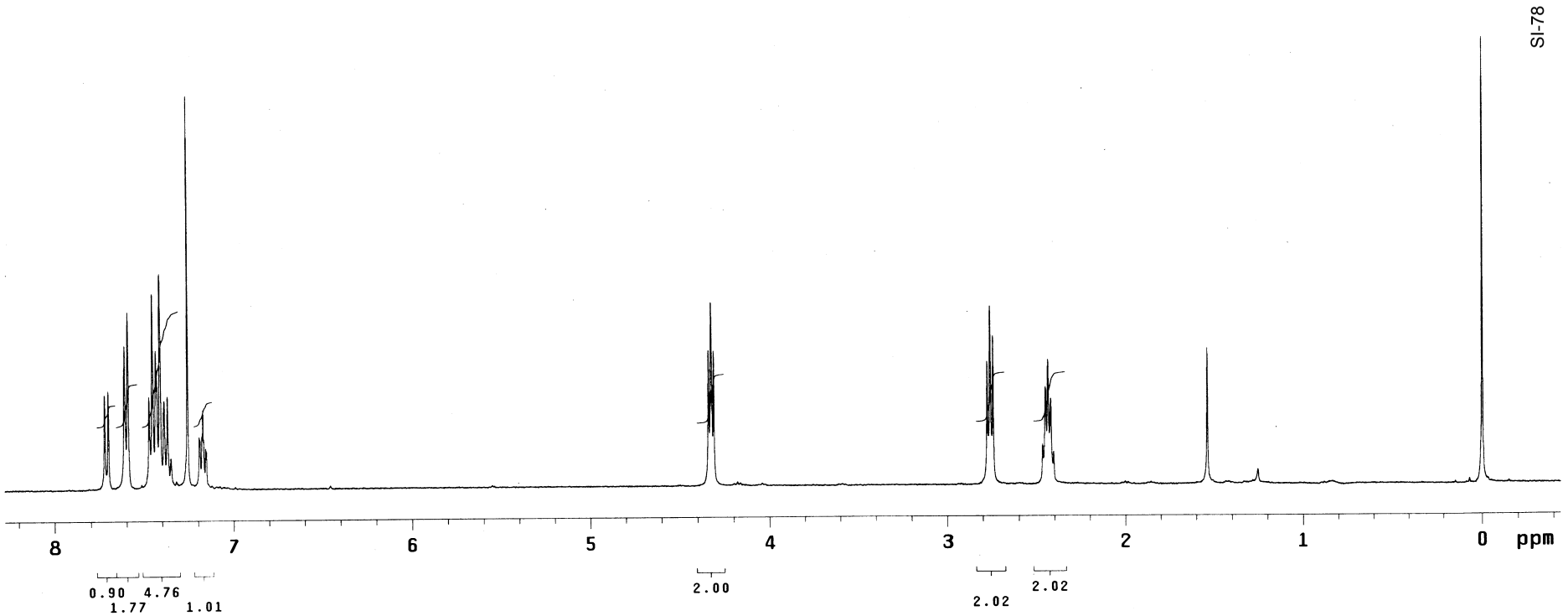


exp2 Proton

SAMPLE		SPECIAL	
date	Jun 4 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K93-2.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-176.5
sfrq	399.868	wp	3487.2
tof	399.6	rfl	800.7
tpwr	59	rfp	0
pw	6.750	rp	163.2
DECOUPLER		lp	-19.3
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	4555
dpwr	34	th	2
dmf	29412	ai	cdc ph



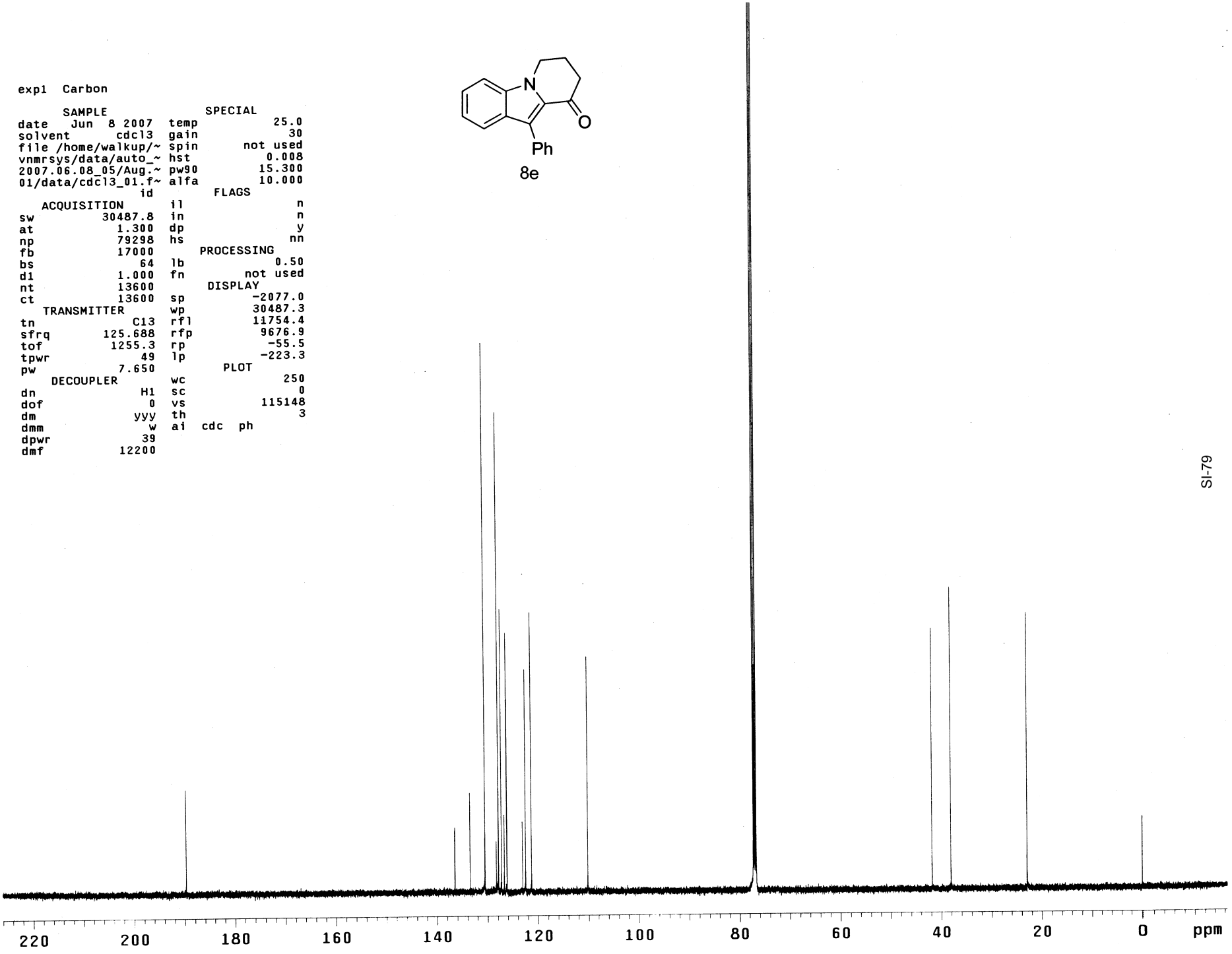
8e

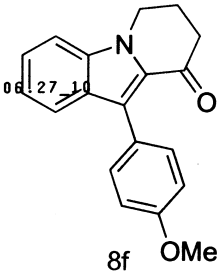


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 8 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.06.08_05/Aug.~	pw90	15.300
	01/data/cdcl3_01.f~	alfa	10.000
id		FLAGS	
ACQUISITION		il	n
sw	30487.8	in	n
at	1.300	dp	y
np	79298	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	13600	DISPLAY	
ct	13600	sp	-2077.0
TRANSMITTER		wp	30487.3
tn	C13	rfl	11754.4
sfrq	125.688	rfp	9676.9
tof	1255.3	rp	-55.5
tpwr	49	lp	-223.3
pw	7.650	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	115148
dm	yyy	th	3
dmm	w	ai	cdc ph
dpwr	39		
dmf	12200		

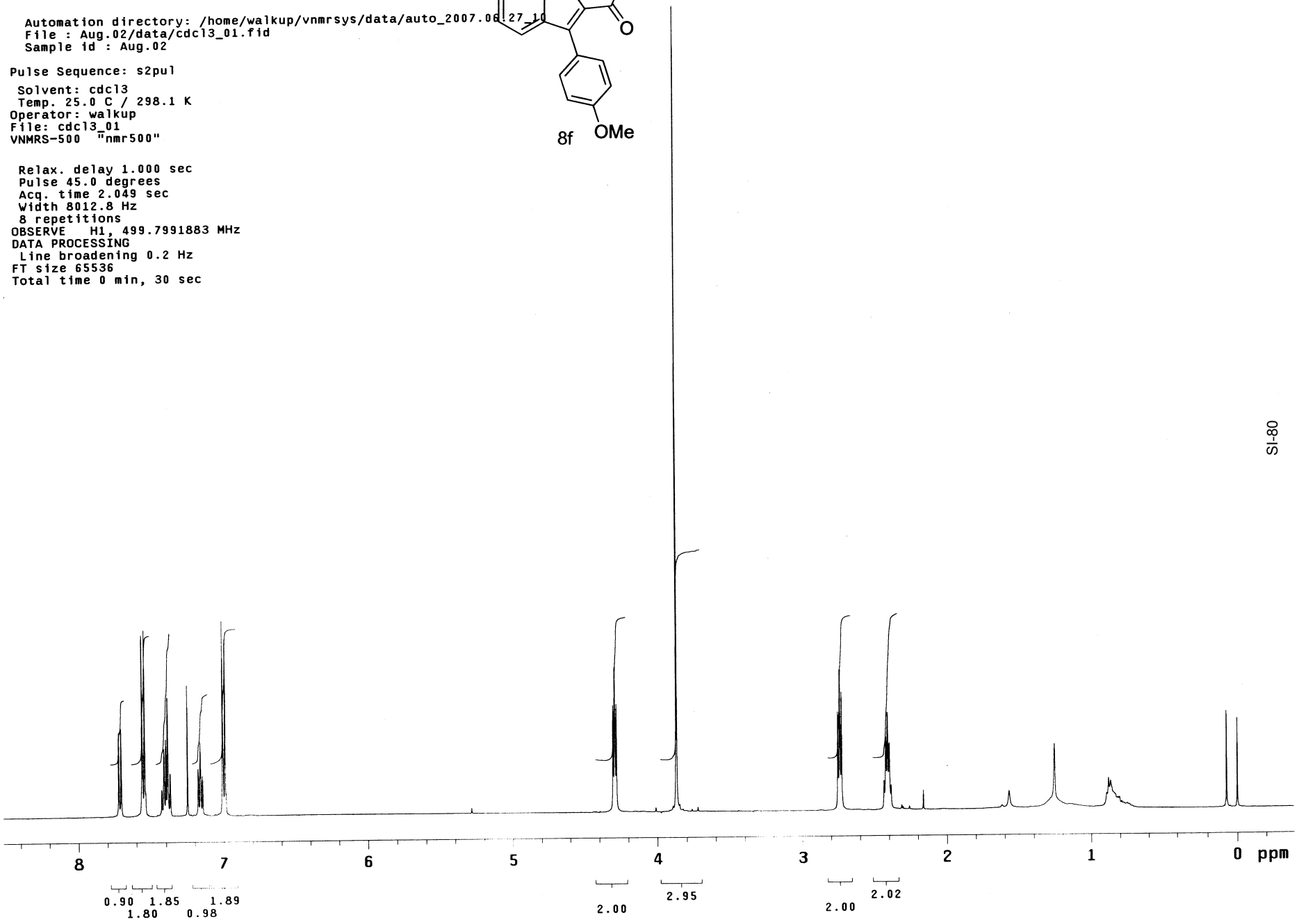
8e

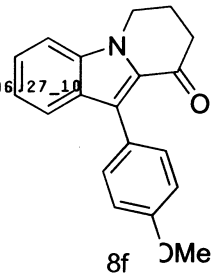




8f

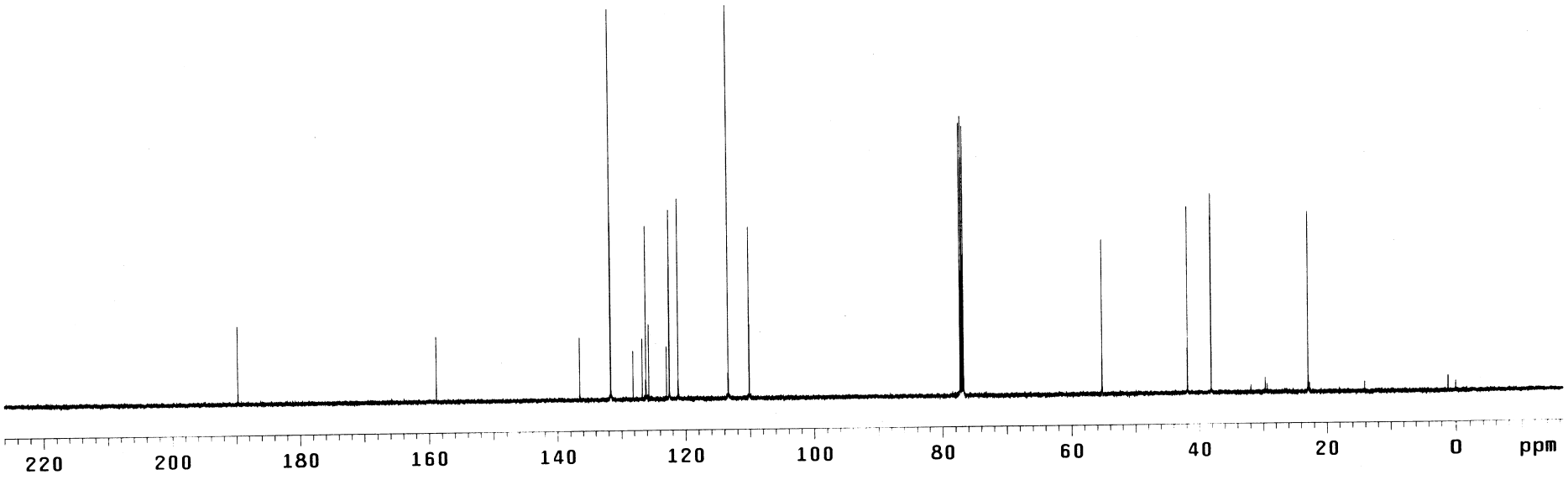
```
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.27_10
File : Aug.02/data/cdcl3_01.fid
Sample id : Aug.02

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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, 499.7991883 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec
```





8f

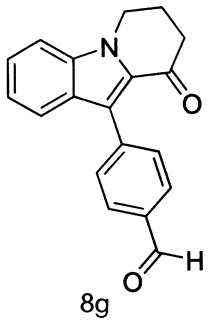
```
  Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.27_10
  File : exp
  Sample id : tmpstudy

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

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 30487.8 Hz
 1024 repetitions
OBSERVE  C13, 125.6746067 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
```

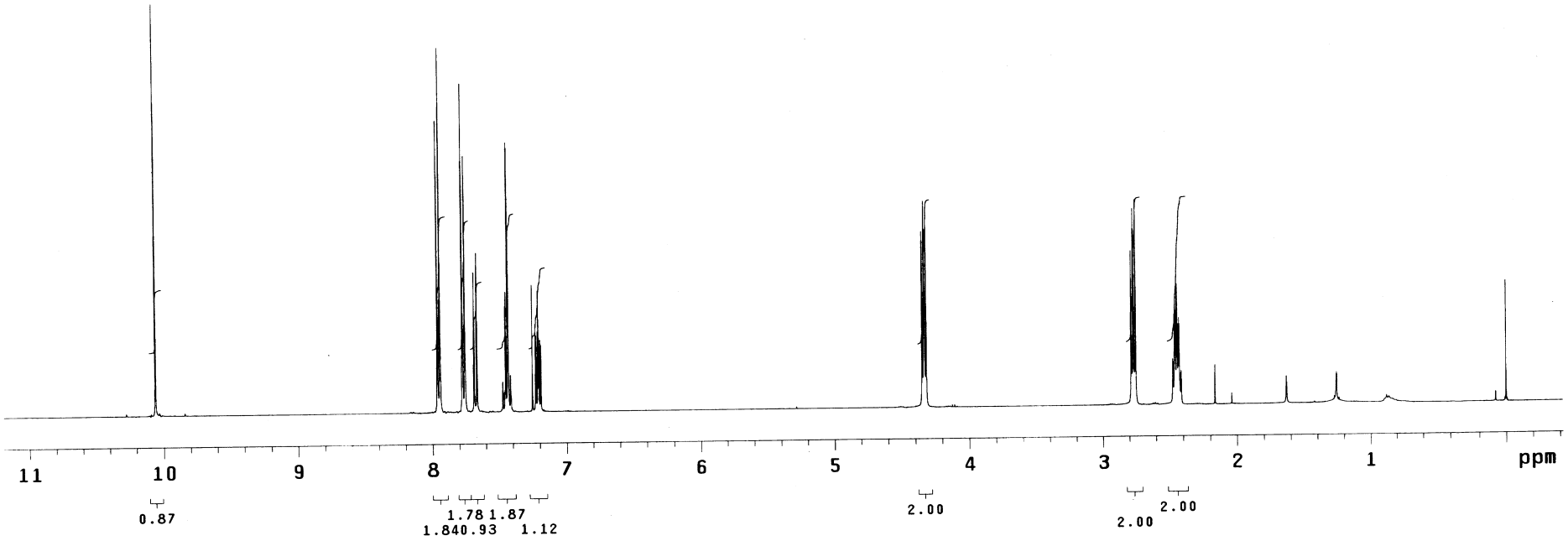
STANDARD 1H OBSERVE - profile

exp2 Proton

SAMPLE		SPECIAL	
date	Jun 25 2007	temp	25.0
solvent	cdcl3	gain	not used
file	/mnt/nmr400/w~	spin	not used
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K175-2.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
ss	2	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-168.6
tn	H1	wp	4644.1
sfrq	399.868	rfl	801.5
tof	399.6	rfp	0
tpwr	59	rp	-105.6
pw	6.750	lp	-32.0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	895
dmm	c	th	2
dpwr	34	ai cdc ph	
dmf	29412		



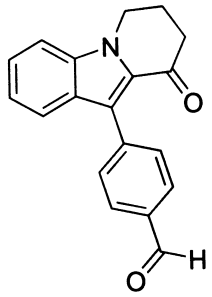
8g



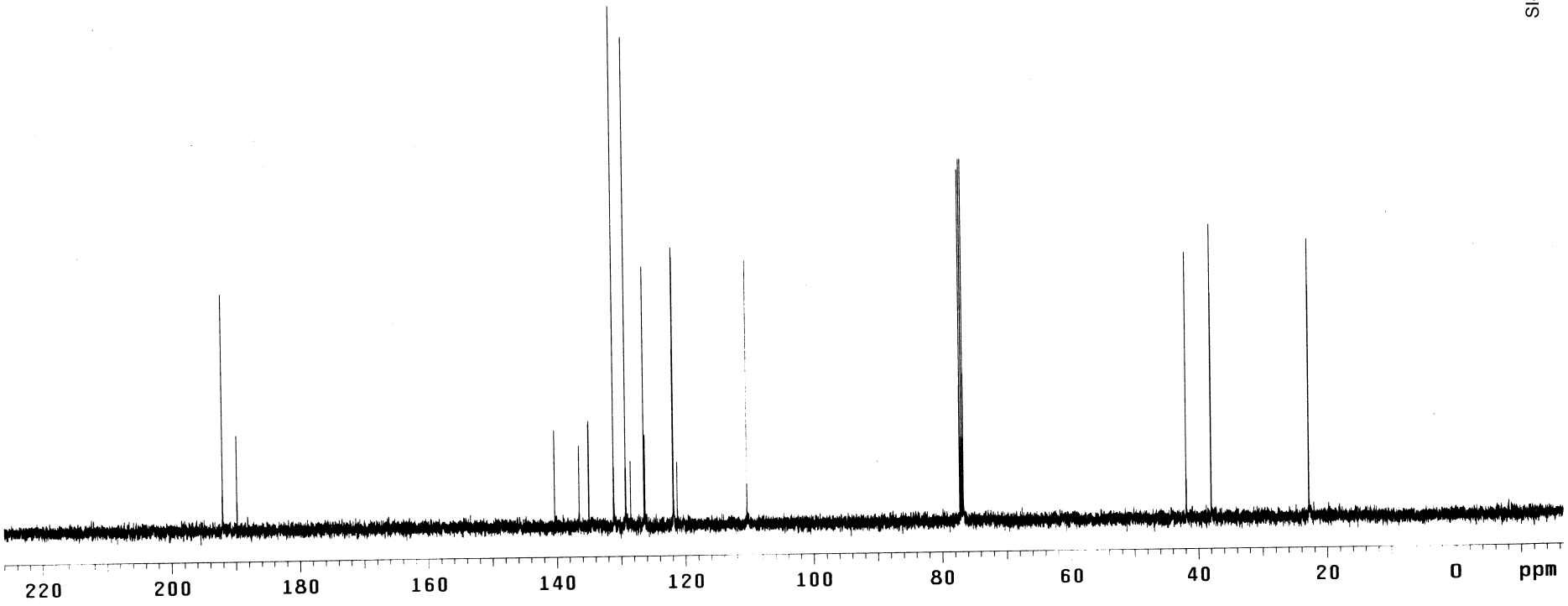
exp1 Carbon

SAMPLE	
date	Jun 26 2007
solvent	cdcl3
file	exp
ACQUISITION	
sw	30487.8
at	1.300
np	79298
fb	17000
bs	8
d1	1.000
nt	1024
ct	64
TRANSMITTER	
tn	C13
sfrq	125.688
tof	1255.3
tpwr	49
pw	7.650
DECOUPLER	
dn	H1
dof	0
dm	yyy
dmm	w
dpwr	39
dmf	12200

SPECIAL	
temp	25.0
gain	30
spin	not used
hst	0.008
pw90	15.300
alfa	10.000
FLAGS	
il	n
in	n
dp	y
hs	nn
PROCESSING	
lb	1.00
fn	not used
DISPLAY	
sp	-2082.6
wp	30487.3
rfl	11760.0
rfp	9676.9
rp	53.5
lp	-252.5
PLOT	
wc	250
sc	0
vs	18919
th	7
ai	cdc ph



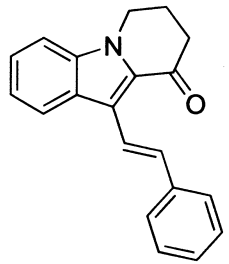
8g



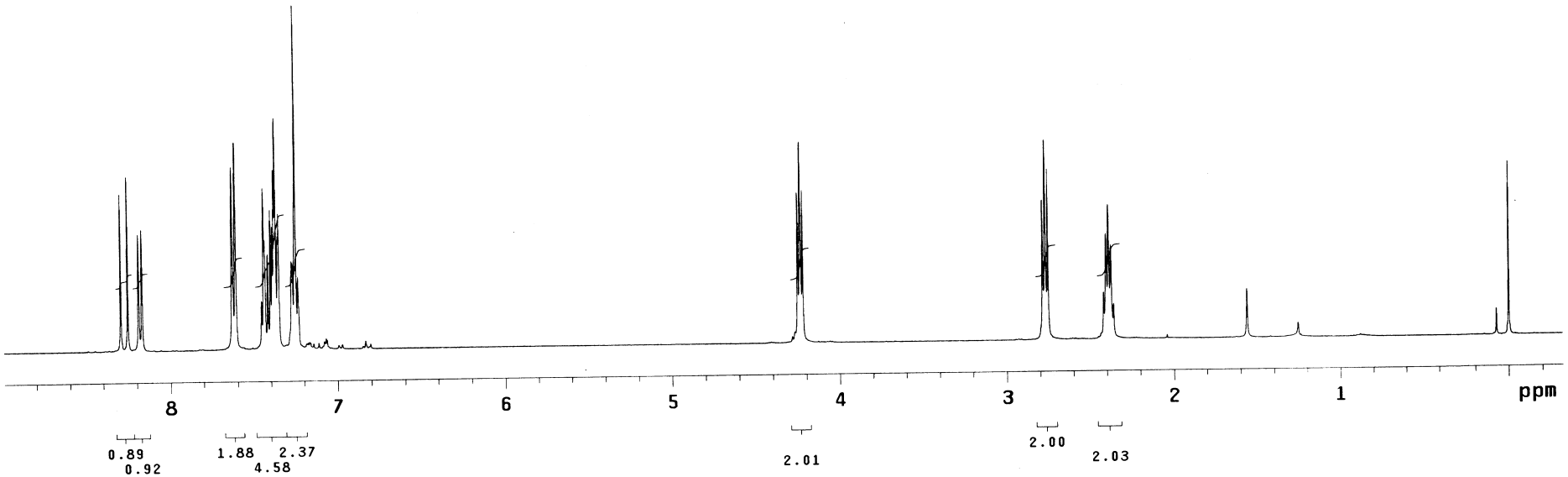
STANDARD 1H OBSERVE - profile

exp1 Proton

SAMPLE		SPECIAL	
date	Jun 21 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	not used
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K167-2.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-129.1
sfrq	399.868	wp	3725.5
tof	399.6	rfl	802.8
tpwr	59	rfp	0
pw	6.750	rp	-20.7
DECOUPLER		lp	-22.2
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	2325
dpwr	34	th	2
dmf	29412	ai cdc ph	

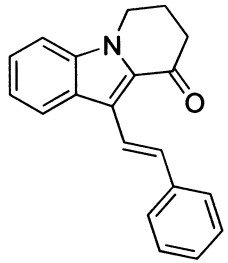


8h

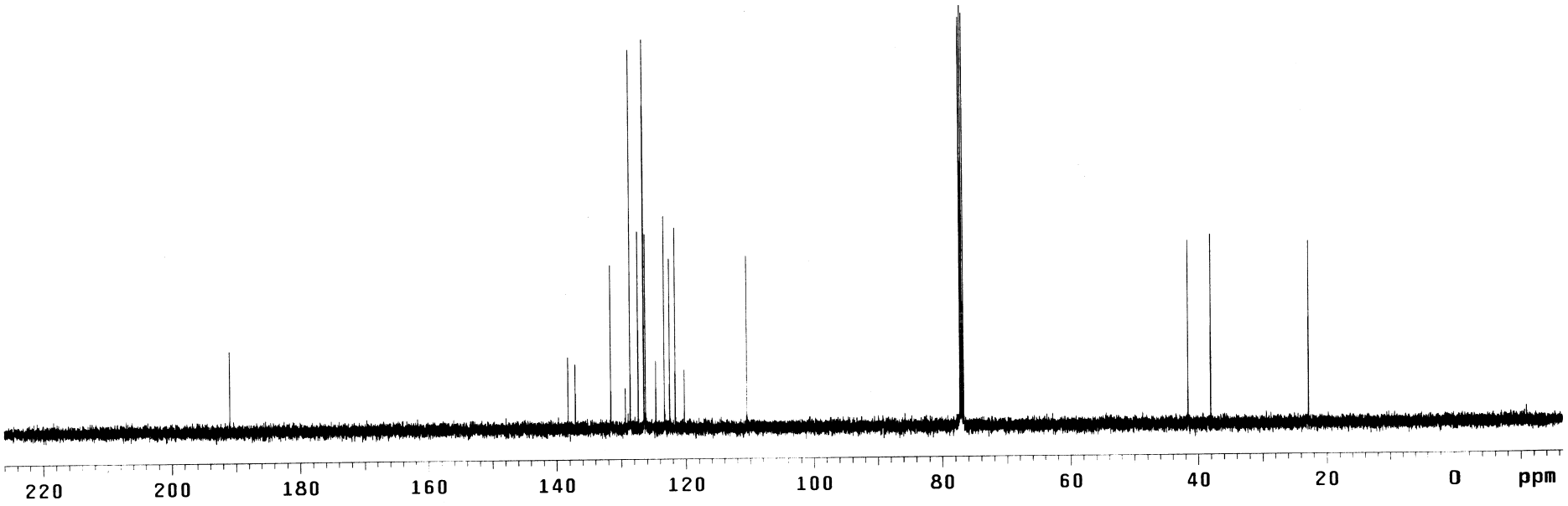


exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 21 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	**FLAGS**	
fb	17000	il	n
bs	32	in	n
d1	1.000	dp	y
nt	1024	hs	nn
ct	320	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	**DISPLAY**	
tof	1255.3	sp	-2078.8
tpwr	49	wp	30487.3
pw	7.650	rfl	11756.2
DECOUPLER		rfp	9676.9
dn	H1	rp	-7.6
dof	0	lp	-276.5
dm	yyy	**PLOT**	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	31377
		th	11
		ai	cdc ph



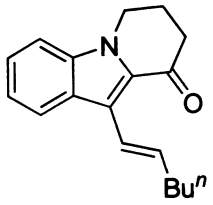
8h



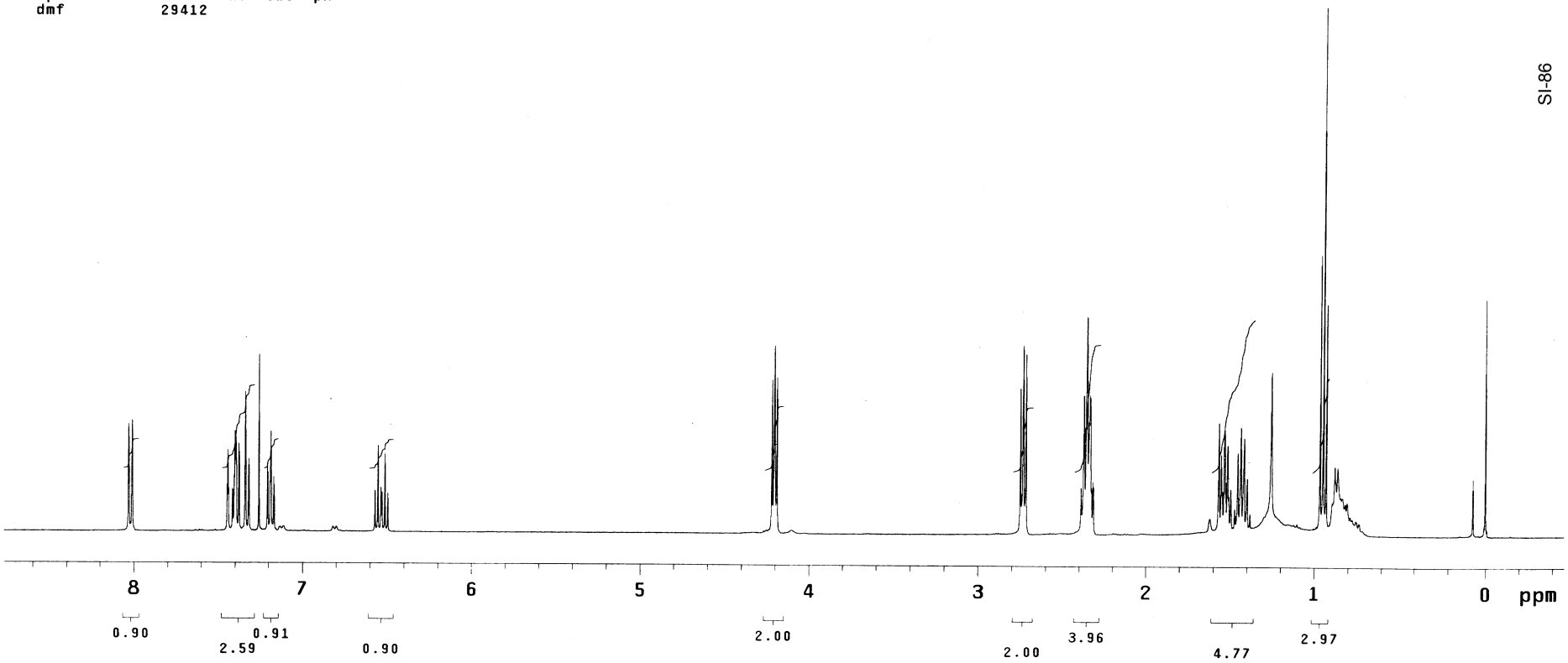
STANDARD 1H OBSERVE - profile

exp2 Proton

SAMPLE		SPECIAL	
date	Jun 25 2007	temp	25.0
solvent	cdcl3	gain	not used
file	/mnt/nmr400/w~	spin	not used
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K174-1.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
ss	2	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-188.0
tn	H1	wp	3694.8
sfrq	399.868	rfl	800.5
tof	399.6	rfp	0
tpwr	59	rp	-106.1
pw	6.750	lp	-30.5
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	1902
dmm	c	th	3
dpwr	34	ai cdc ph	
dmf	29412		

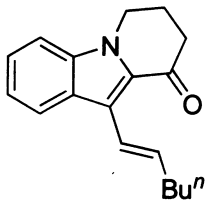


7i

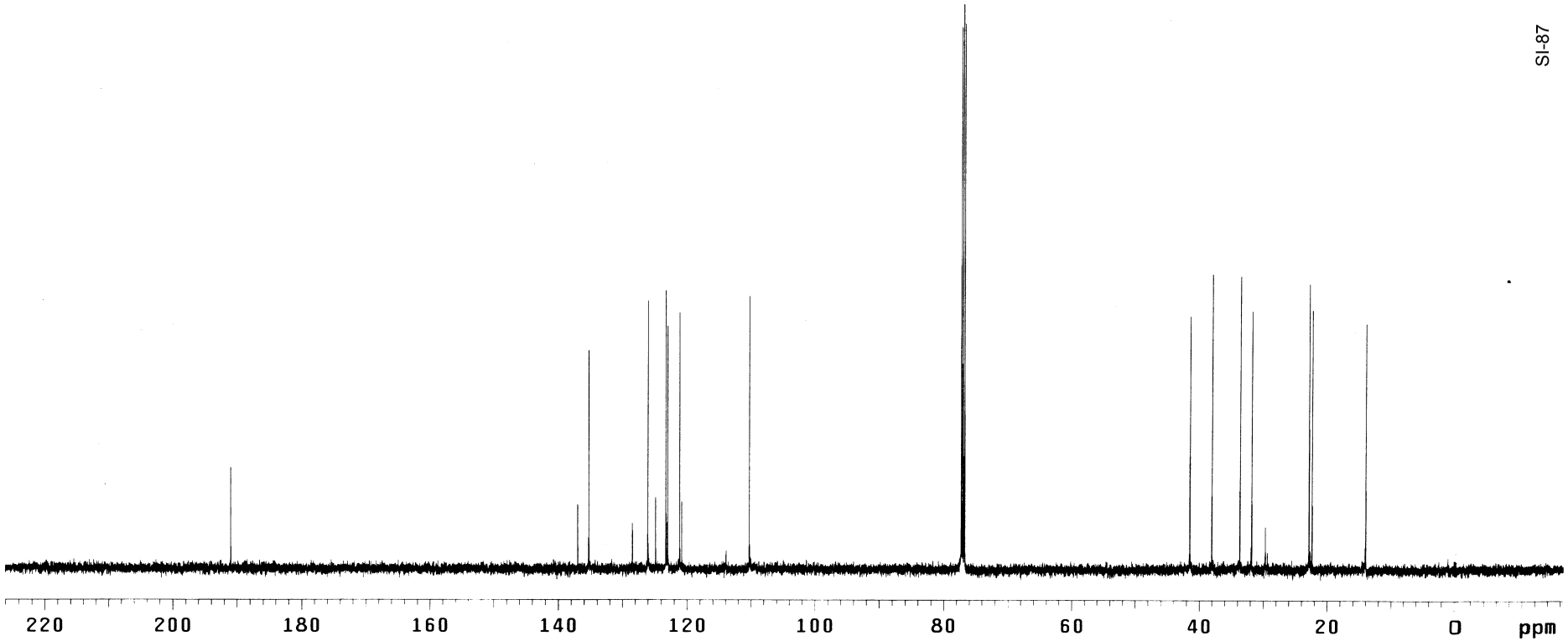


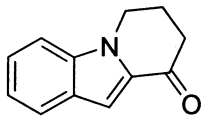
exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 26 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	FLAGS	
fb	17000	il	n
bs	8	in	n
di	1.000	dp	y
nt	1024	hs	nn
ct	672	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.688	DISPLAY	
tof	1255.3	sp	-2077.4
tpwr	49	wp	30487.3
pw	7.650	rfl	11754.9
DECOUPLER		rfp	9676.9
dn	H1	rp	64.0
dof	0	lp	-273.7
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	46287
		th	68
		ai cdc ph	



7i

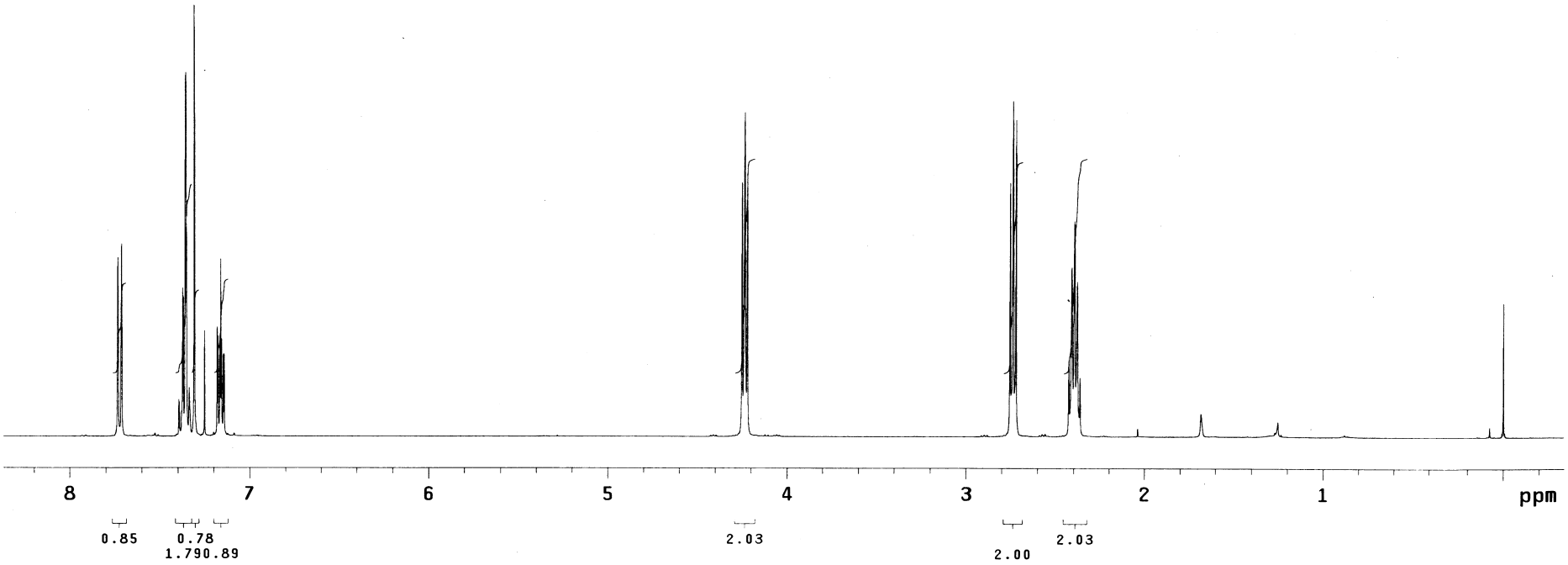




8j

exp2 Proton

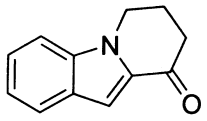
SAMPLE		SPECIAL	
date	Jun 8 2007	temp	25.0
solvent	cdcl3	gain	20
file	/mnt/nmr400/w~	spin	20
alkup/data/Zhang/G~		hst	0.008
uotaoLi/K104-1.fid		pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-136.7
sfrq	399.868	wp	3483.9
tof	399.6	rfl	802.3
tpwr	59	rfp	0
pw	6.750	rp	-97.6
DECOUPLER		lp	-28.7
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	637
dpwr	34	th	2
dmf	29412	ai cdc ph	



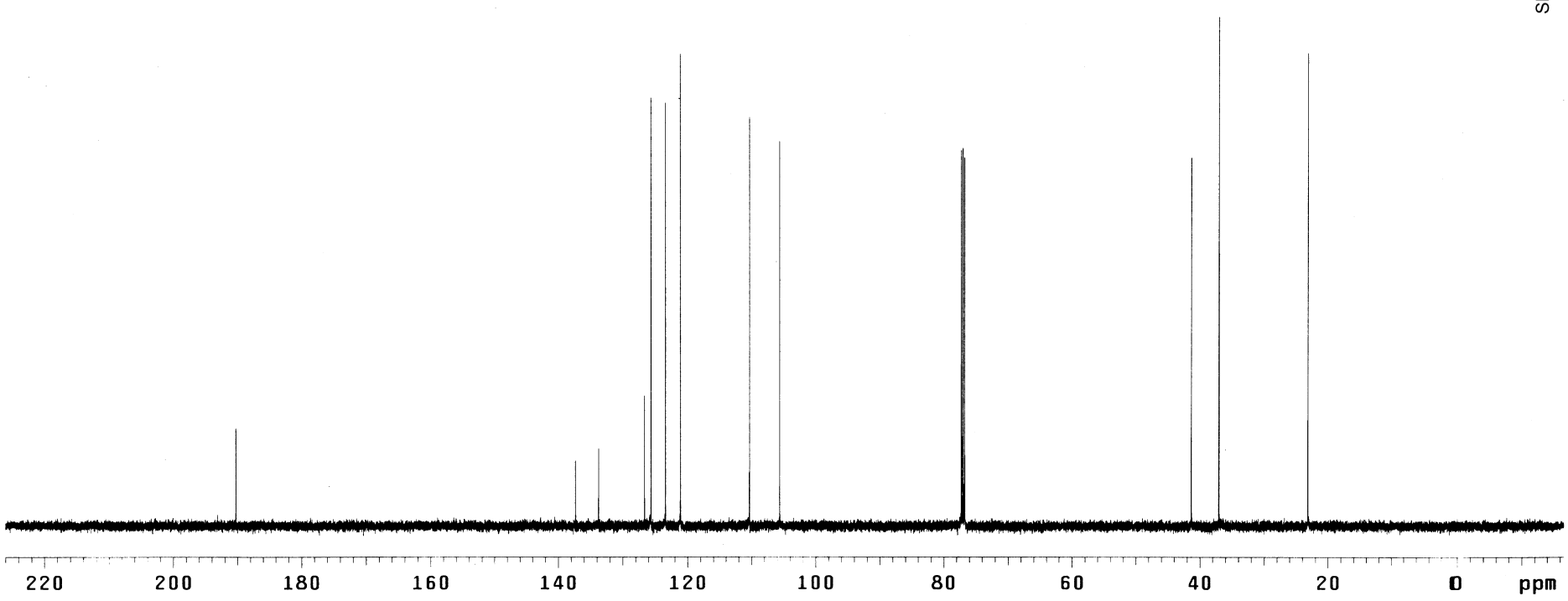
T1 EXPERIMENT
T1 EXPERIMENT
T1 EXPERIMENT

exp1 Carbon

SAMPLE		SPECIAL	
date	Jun 8 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	30487.8	pw90	15.300
at	1.300	alfa	10.000
np	79298	FLAGS	
fb	17000	il	n
bs	64	in	n
d1	1.000	dp	y
nt	2000	hs	nn
ct	192	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.688	DISPLAY	
tof	1255.3	sp	-2082.6
tpwr	49	wp	30487.3
pw	7.650	rfl	11760.0
DECOUPLER		rfp	9676.9
dn	H1	rp	-48.7
dof	0	lp	-229.4
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	39	sc	0
dmf	12200	vs	17176
		th	13
		ai cdc ph	



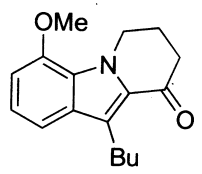
8j



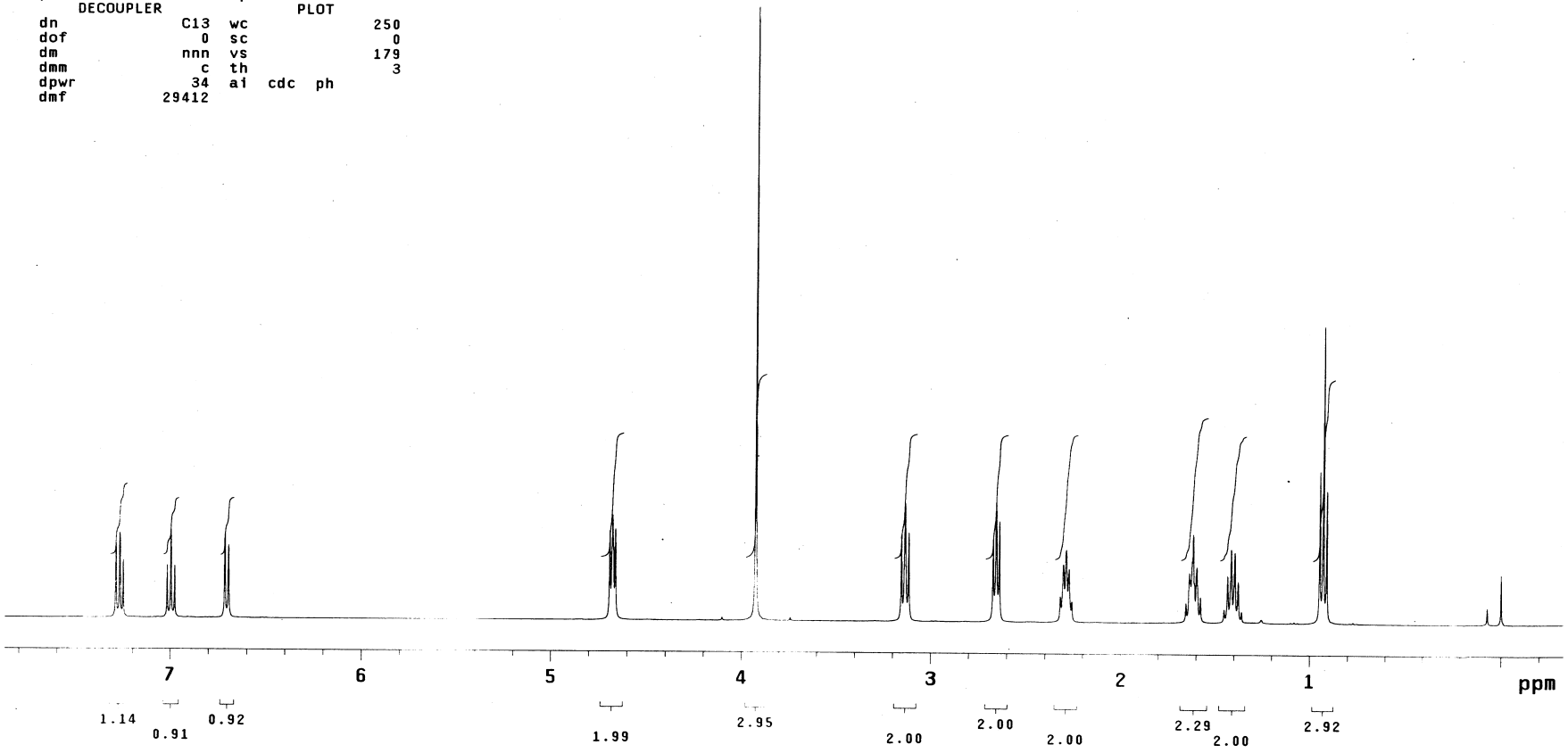
STANDARD 1H OBSERVE - profile

exp1 Proton

SAMPLE		SPECIAL	
date	Aug 2 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K2~	pw90	13.500
	92-1.fid	alfa	6.600
ACQUISITION		FLAGS	
sw	6410.3	il	n
at	2.049	in	n
np	26264	dp	y
fb	4000	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-132.2
tn	H1	wp	3280.6
sfrq	399.868	rfl	802.8
tof	399.6	rfp	0
tpwr	59	rp	154.2
pw	6.750	lp	-30.1
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	179
dmm	c	th	3
dpwr	34	ai cdc ph	
dmf	29412		



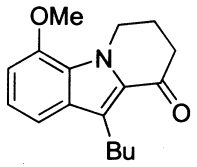
8k



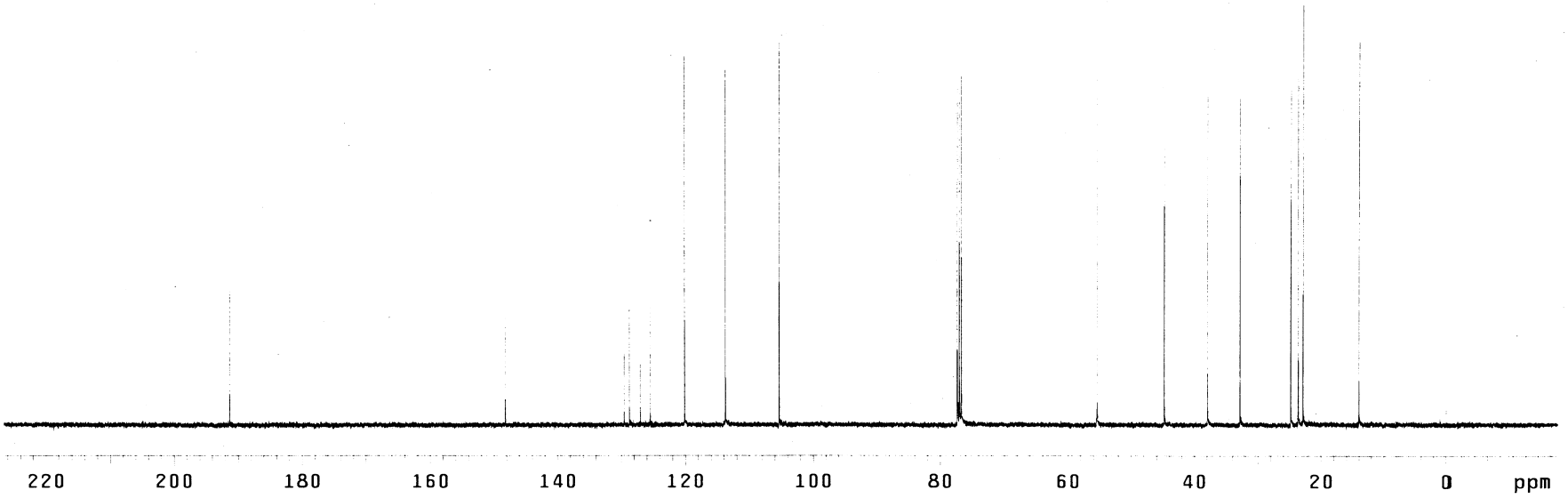
STANDARD 1H OBSERVE - profile

exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 2 2007	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	24509.8	pw90	11.900
at	1.300	alfa	10.000
np	63750	FLAGS	
fb	17000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	3200	hs	nn
ct	2960	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	100.557	DISPLAY	
tof	1042.8	sp	-1721.4
tpwr	53	wp	24509.1
pw	5.950	rfl	9464.2
DECOUPLER		rfp	7742.1
dn	H1	rp	179.4
dof	0	lp	-192.2
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	41	sc	0
dmf	9500	vs	56722
		th	5
		ai cdc ph	

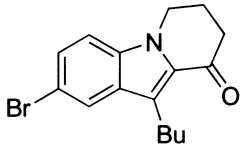


8k

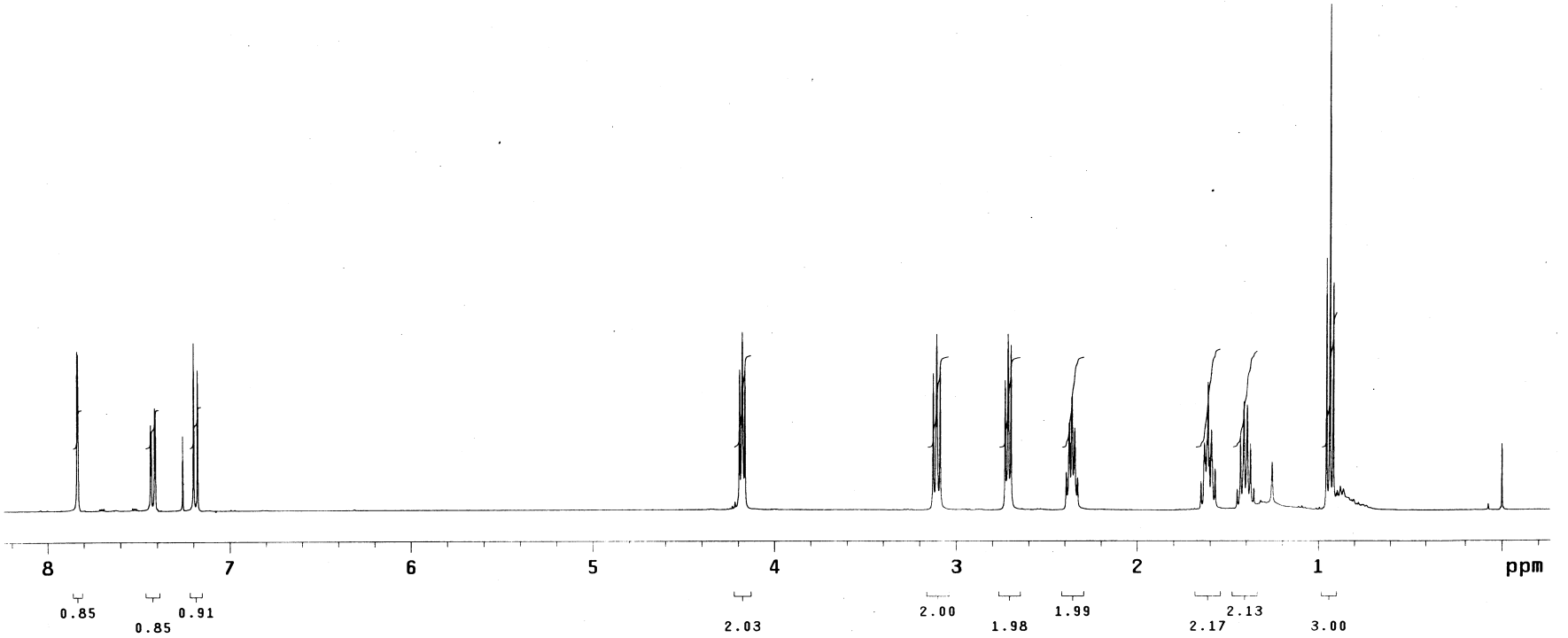


exp1 Proton

SAMPLE		SPECIAL	
date	Jul 31 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K284-1.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-106.6
sfrq	399.868	wp	3403.3
tof	399.6	rfl	798.9
tpwr	59	rfp	0
pw	6.750	rp	75.2
DECOUPLER		lp	-13.5
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	183
dpwr	34	th	6
dmf	29412	ai cdc ph	

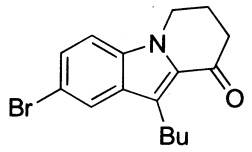


8l

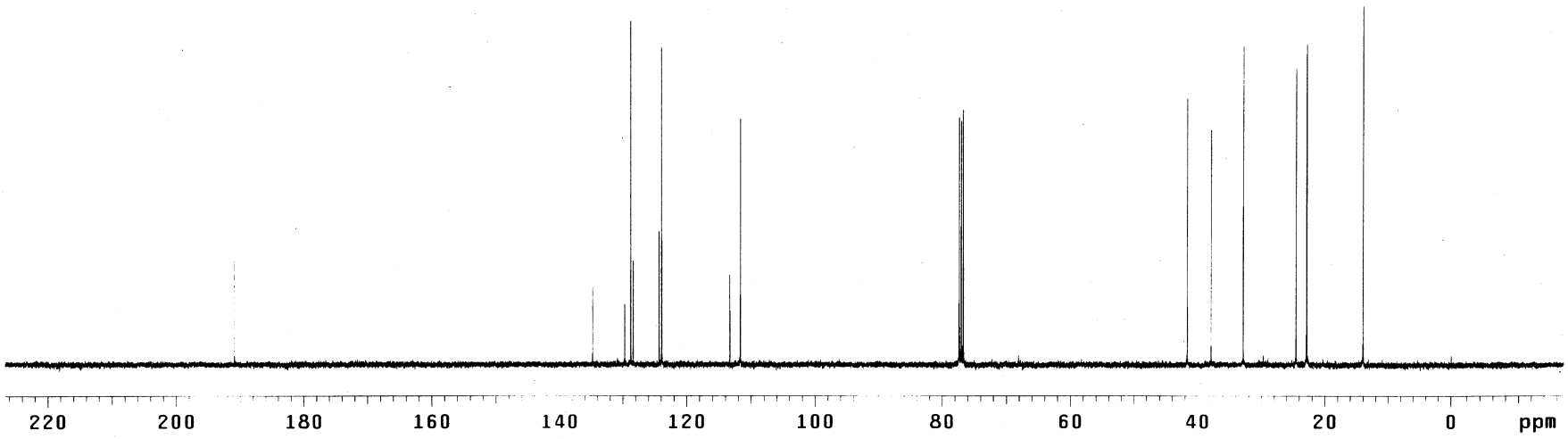


exp1 Carbon

SAMPLE		SPECIAL	
date	Jul 31 2007	temp	25.0
solvent	cdcl3	gain	60
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K284-1carb~	pw90	11.900
	on.fid	alfa	10.000
ACQUISITION		FLAGS	
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64	PROCESSING	
d1	1.000	lb	0.50
nt	1200	fn	not used
ct	1200	DISPLAY	
TRANSMITTER		sp	-1721.4
tn	C13	wp	24509.1
sfrq	100.557	rfl	9464.2
tof	1042.8	rfp	7742.1
tpwr	53	rp	115.9
pw	5.950	lp	-195.5
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	1417
dmm	w	th	6
dpwr	41	ai cdc ph	
dmf	9500		

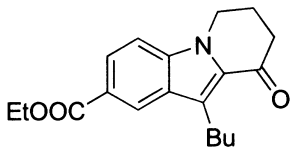


8l

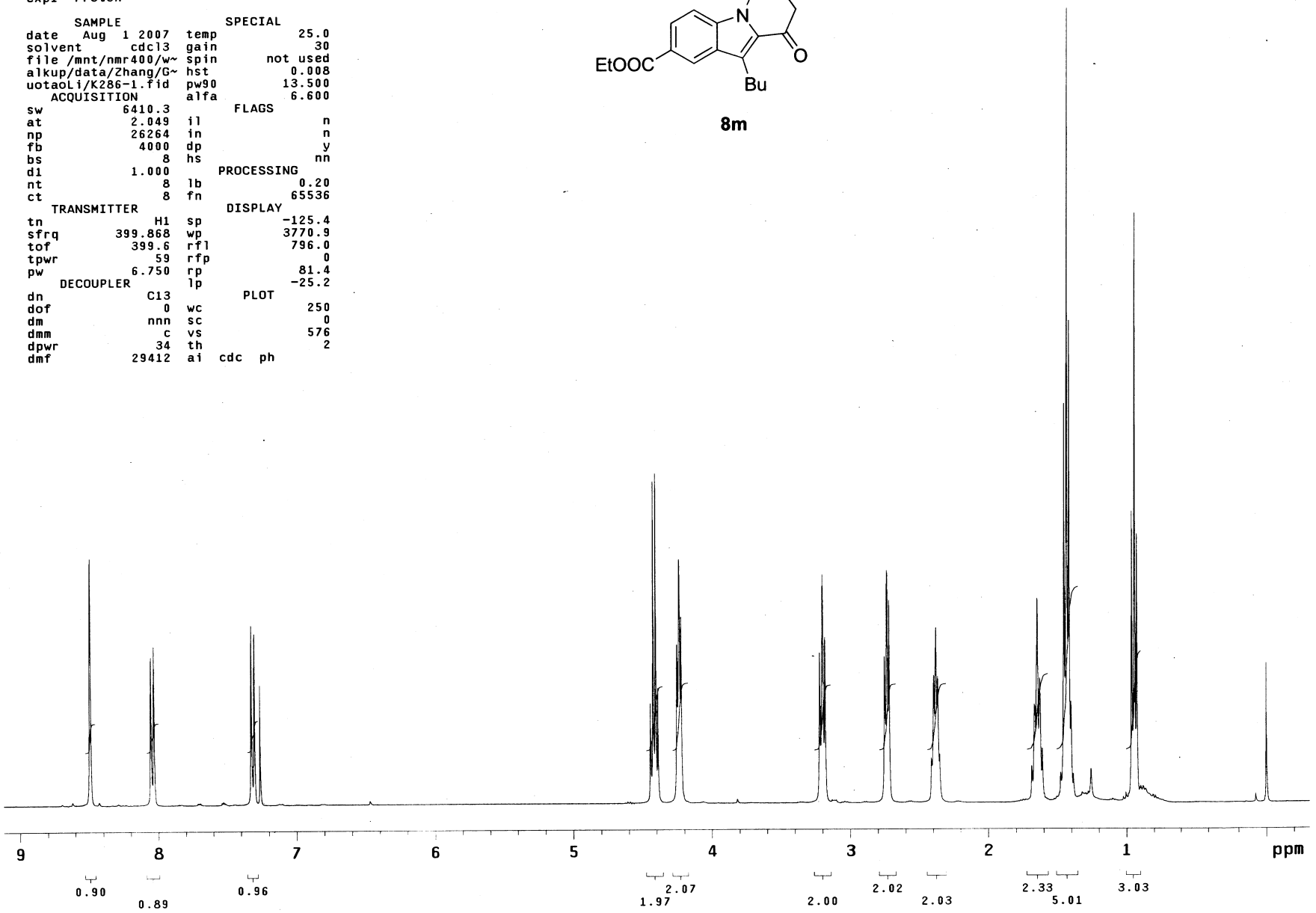


exp1 Proton

SAMPLE		SPECIAL	
date	Aug 1 2007	temp	25.0
solvent	cdcl3	gain	30
file	/mnt/nmr400/w~	spin	not used
	alkup/data/Zhang/G~	hst	0.008
	uotaoLi/K286-1.fid	pw90	13.500
ACQUISITION		alfa	6.600
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	0.20
ct	8	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-125.4
sfrq	399.868	wp	3770.9
tof	399.6	rfl	796.0
tpwr	59	rfp	0
pw	6.750	rp	81.4
DECOUPLER		lp	-25.2
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	576
dpwr	34	th	2
dmf	29412	ai cdc ph	

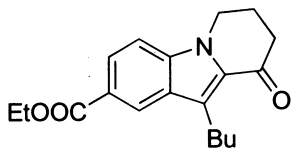


8m

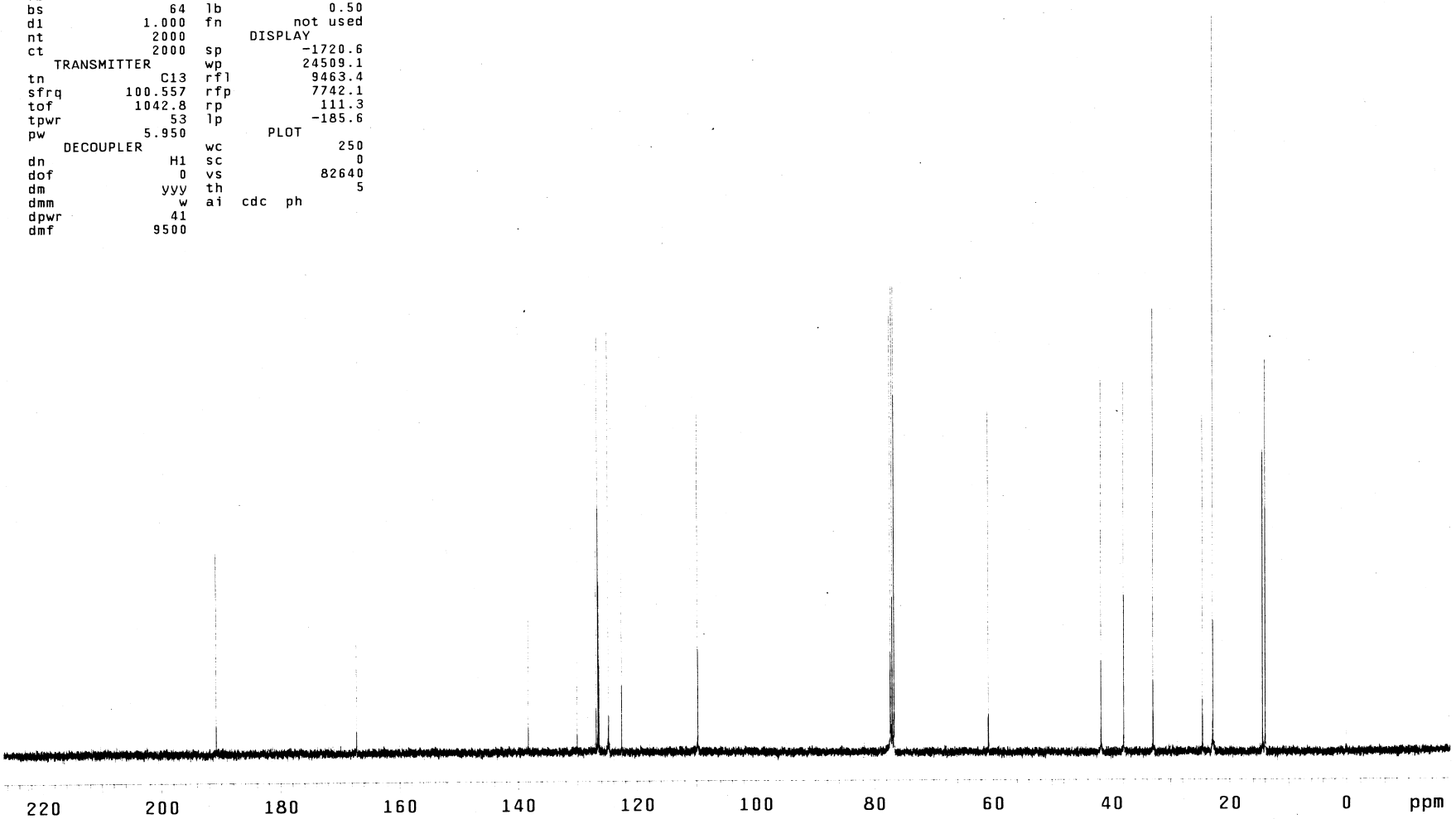


exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 1 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.01/s_20070~	pw90	11.900
	801_001/data/cdcl3~	alfa	10.000
	_01.fid	FLAGS	
ACQUISITION		il	n
sw	24509.8	in	n
at	1.300	dp	y
np	63750	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	2000	DISPLAY	
ct	2000	sp	-1720.6
TRANSMITTER		wp	24509.1
tn	C13	rfl	9463.4
sfrq	100.557	rfp	7742.1
tof	1042.8	rp	111.3
tpwr	53	lp	-185.6
pw	5.950	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	82640
dm	yyy	th	5
dmm	w	ai	cdc ph
dpwr	41		
dmf	9500		

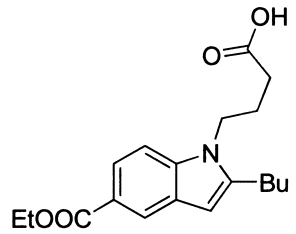


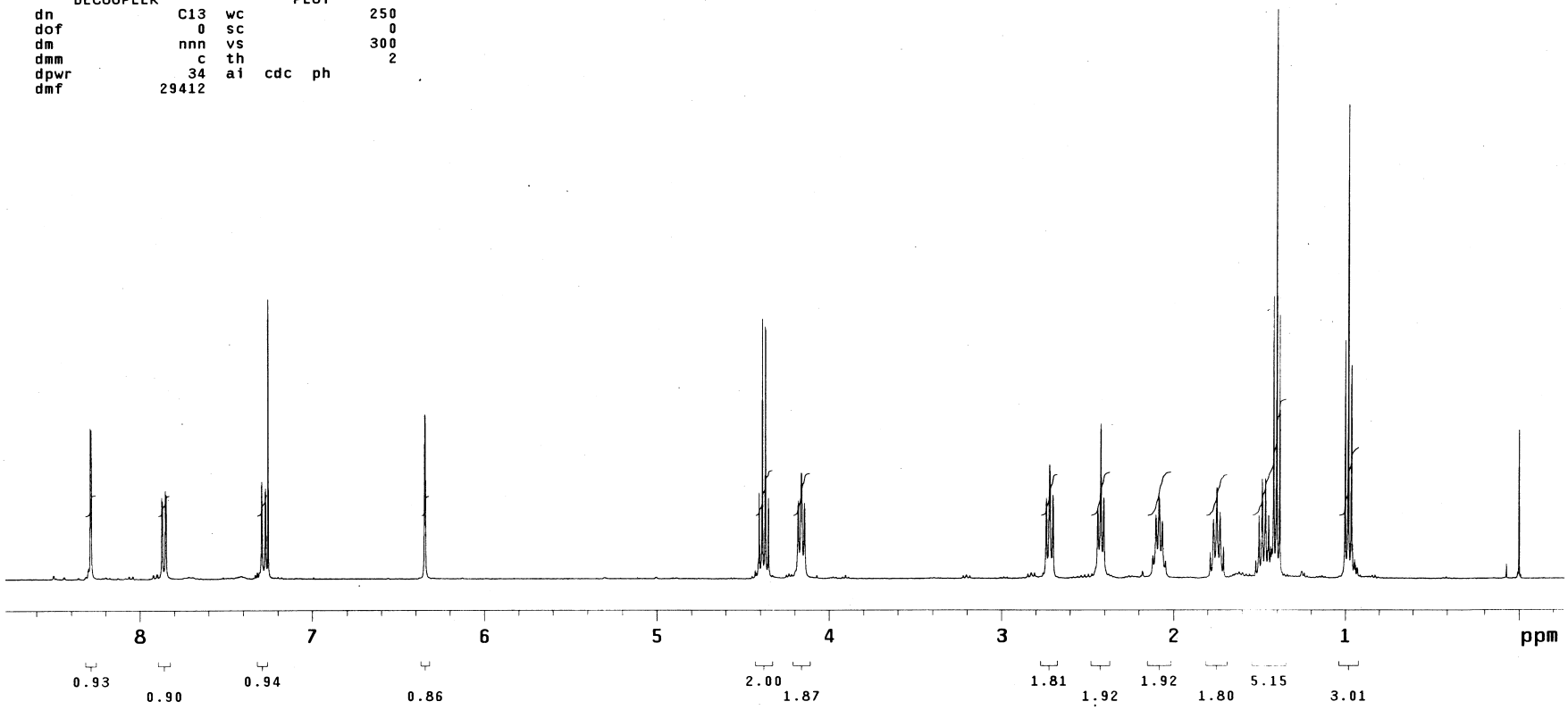
8m



exp1 Proton

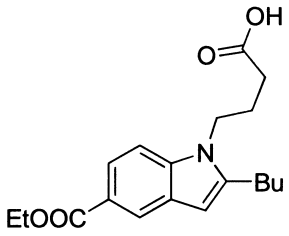
SAMPLE		SPECIAL	
date	Aug 3 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K2~	pw90	13.500
	96-3.fid	alfa	6.600
ACQUISITION		**FLAGS**	
sw	6410.3	il	n
at	2.049	in	n
np	26264	dp	y
fb	4000	hs	nn
bs	8	**PROCESSING**	
d1	1.000	lb	0.20
nt	1	fn	65536
ct	1	**DISPLAY**	
TRANSMITTER		sp	-108.2
tn	H1	wp	3619.5
sfrq	399.868	rfl	800.5
tof	399.6	rfp	0
tpwr	59	rp	155.8
pw	6.750	lp	-33.1
DECOUPLER		**PLOT**	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	300
dmm	c	th	2
dpwr	34	ai cdc ph	
dmf	29412		

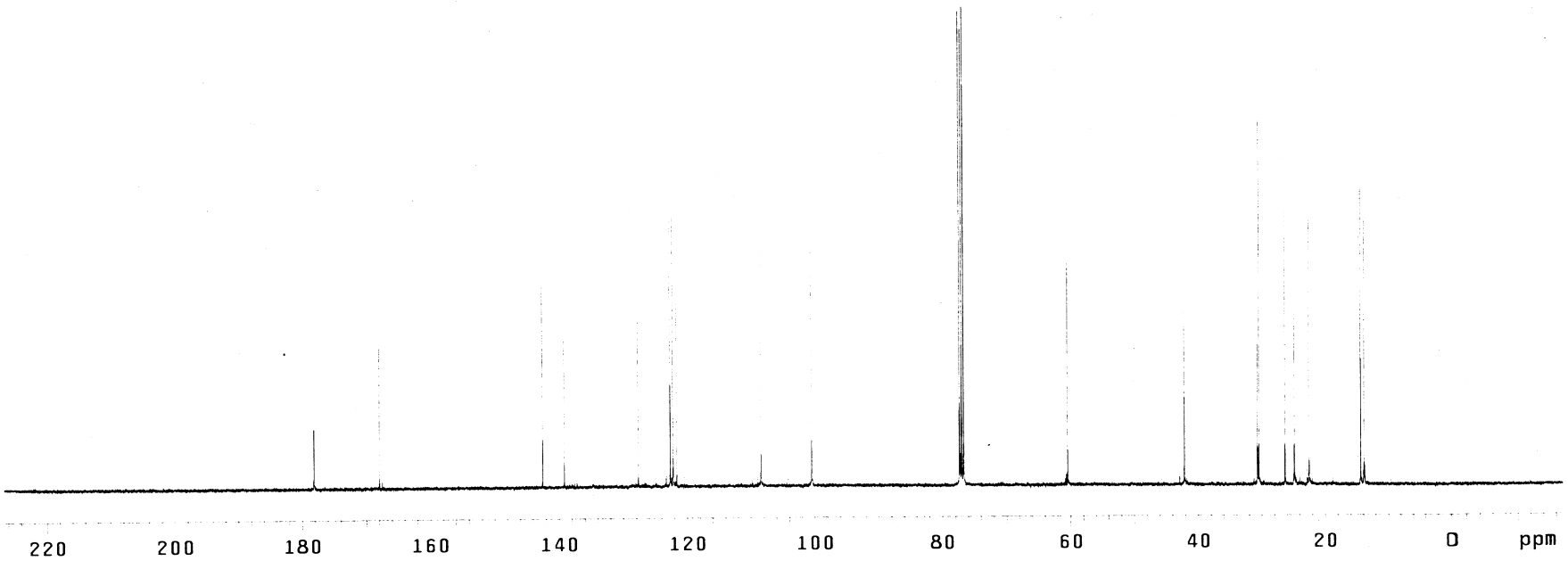




exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 3 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.03_16/s_20~	pw90	11.900
	070803_001/data/cd~	alfa	10.000
	c13_01.fid	FLAGS	
ACQUISITION		il	n
sw	24509.8	in	n
at	1.300	dp	y
np	63750	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	12000	DISPLAY	
ct	12000	sp	-1719.9
TRANSMITTER		wp	24509.1
tn	C13	rfl	9462.7
sfrq	100.557	rfp	7742.1
tof	1042.8	rp	-172.9
tpwr	53	lp	-193.0
pw	5.950	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	82807
dm	yyy	th	8
dmm	w	ai	cdc ph
dpwr	41		
dmf	9500		



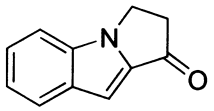


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.12
File : Aug.01/data/cdcl3_01.fid
Sample id : Aug.01

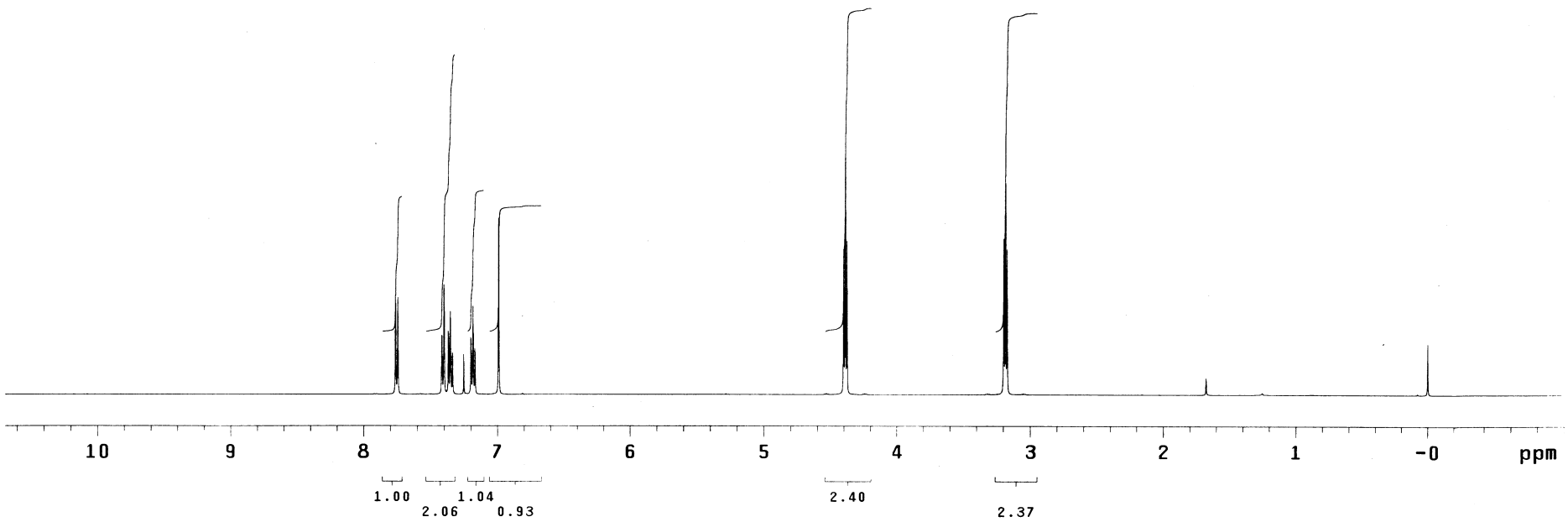
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: walkup
File: cdcl3_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, 499.7991866 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



11

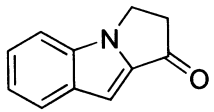


Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.12
File : /home/walkup/data/Zhang/XiaogengHuang/hxg-3-157C.fid
Sample id : tmpstudy

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: walkup
File: hxg-3-157C
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
448 repetitions
OBSERVE C13, 125.6746114 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 19 min, 42 sec



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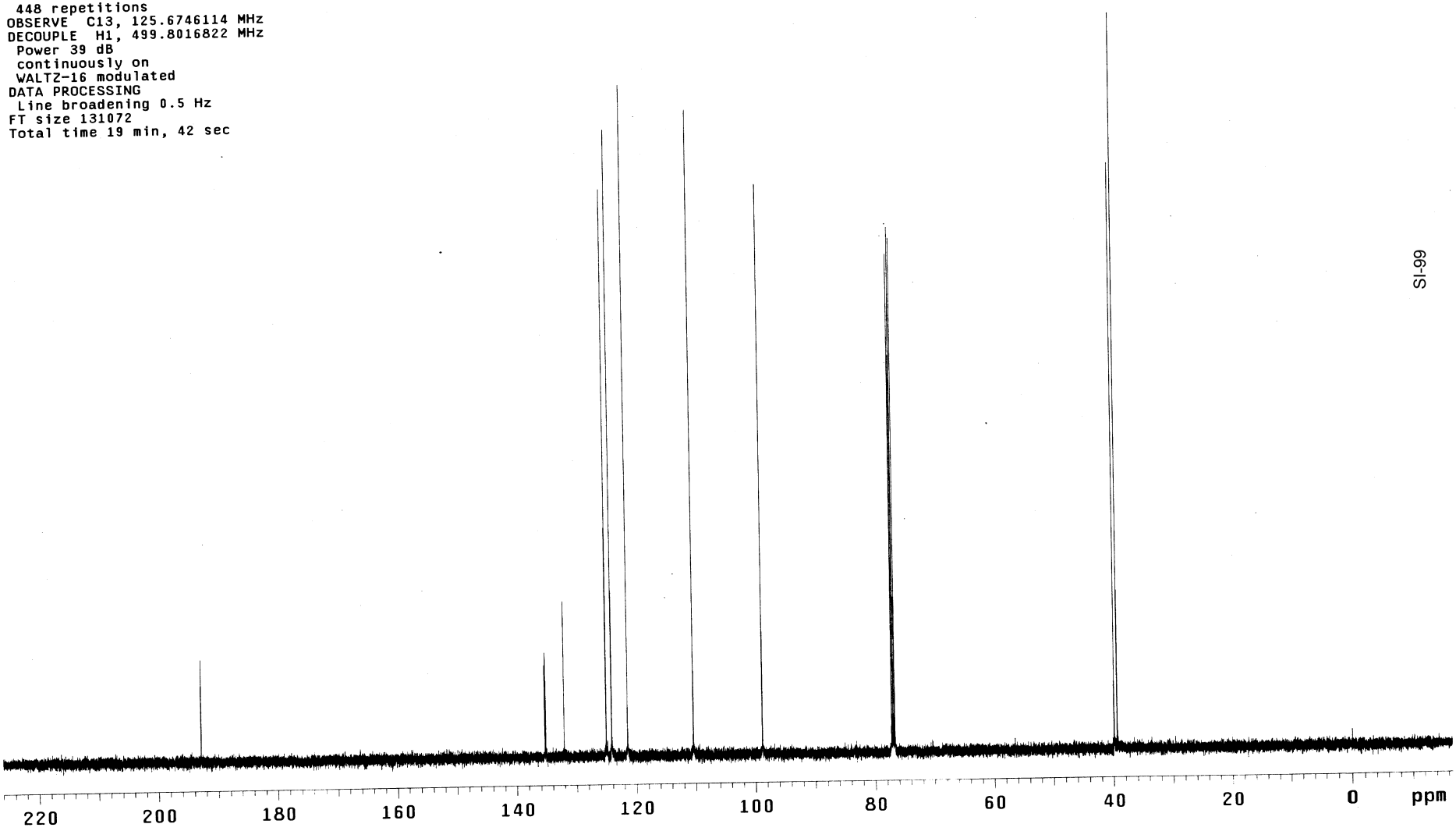
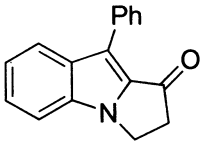
SI-99



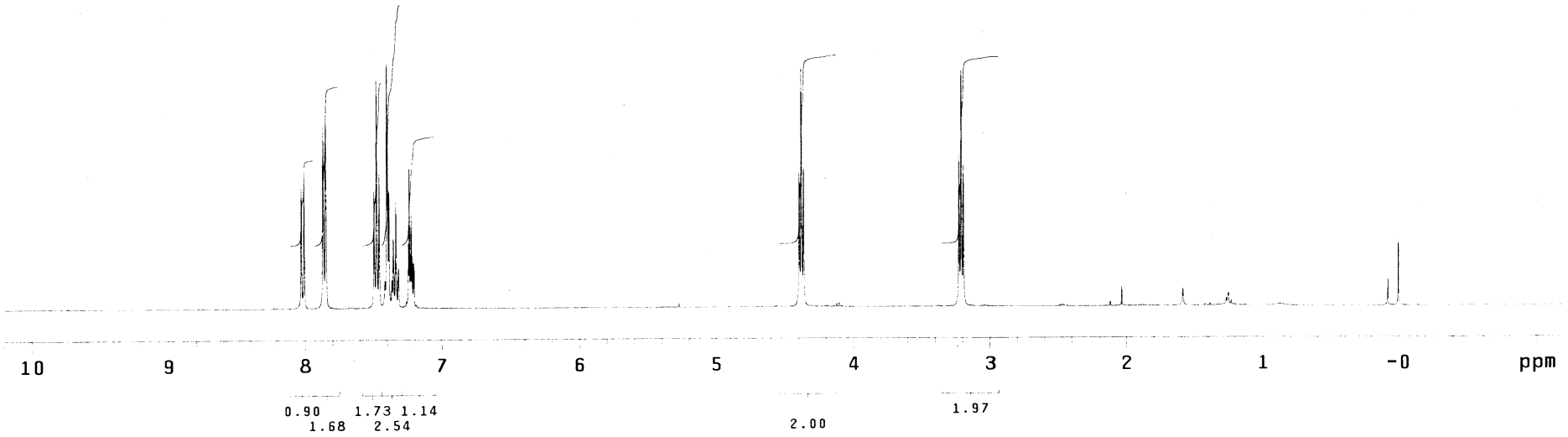


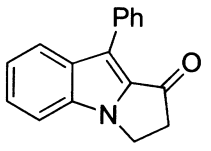

```
  Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_26
  File : s_20070611_001/data/cdcl3_01.fid
  Sample id : s_20070611_001

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 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE   H1, 399.8656811 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec
```

SI-100



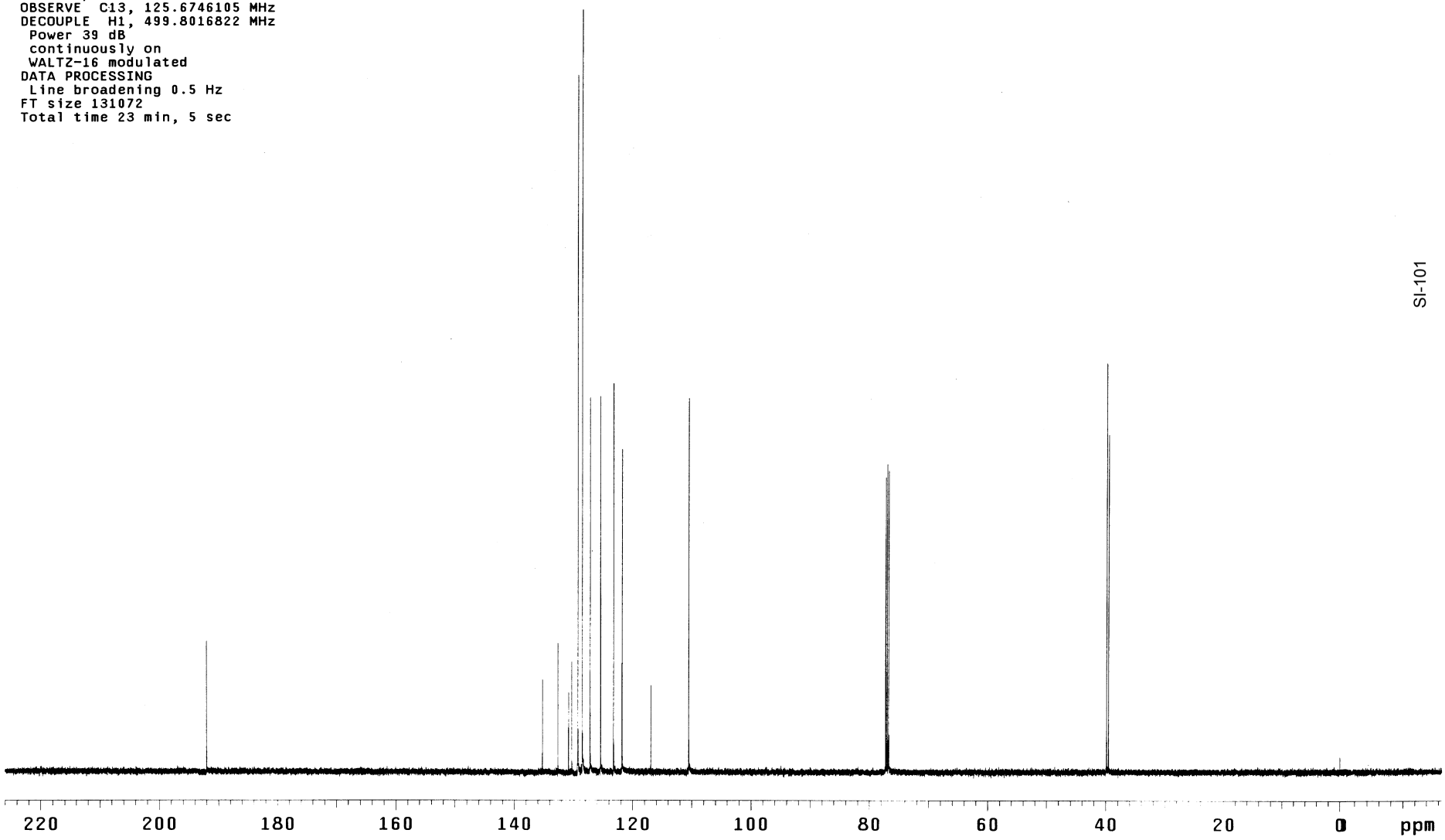


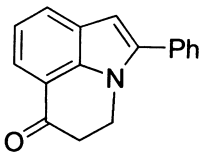
File: hxg-3-155-2C

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: hxg-3-155-2C
INOVA-500 "redjacket"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
600 repetitions
OBSERVE C13, 125.6746105 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 23 min, 5 sec

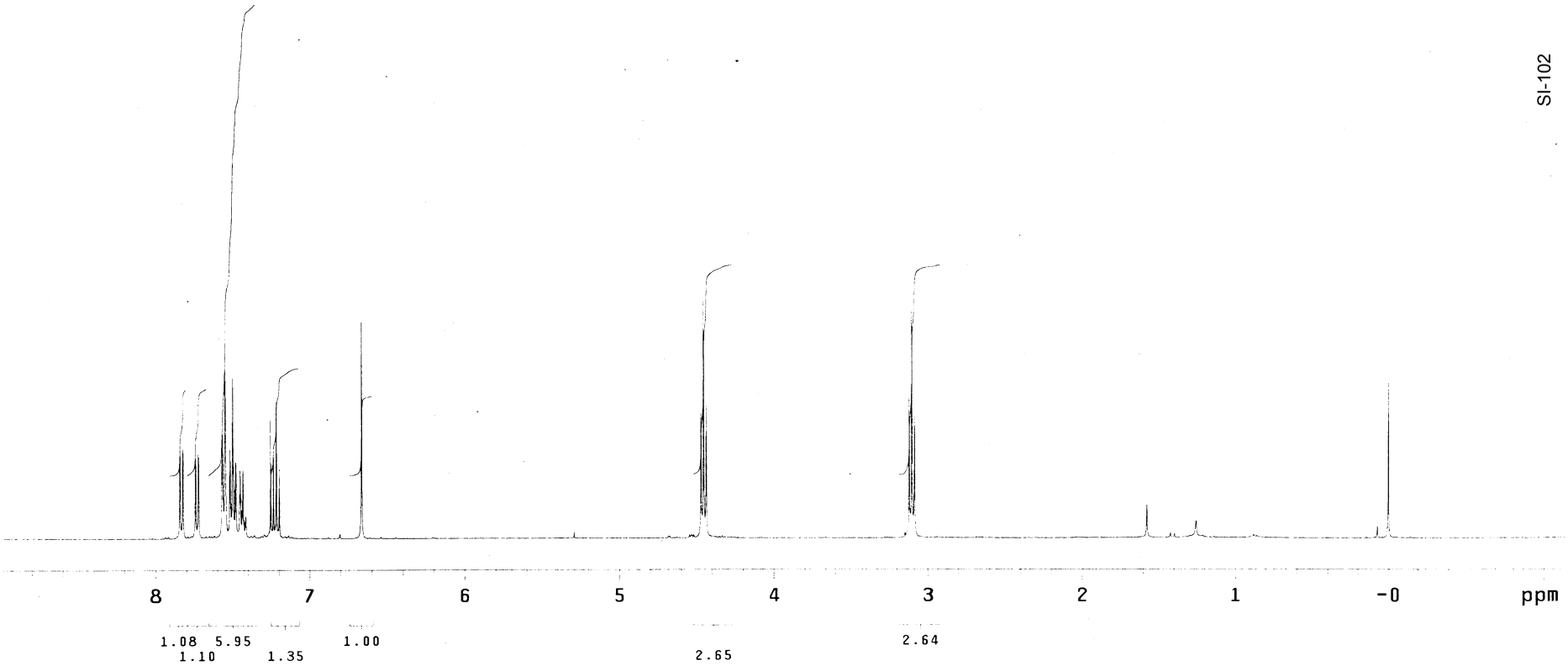


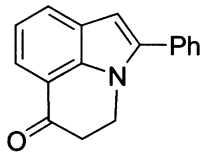



```
  Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_26
  File : s_20070611_002/data/cdcl3_02.fid
  Sample id : s_20070611_002

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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, 399.8656747 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec
```

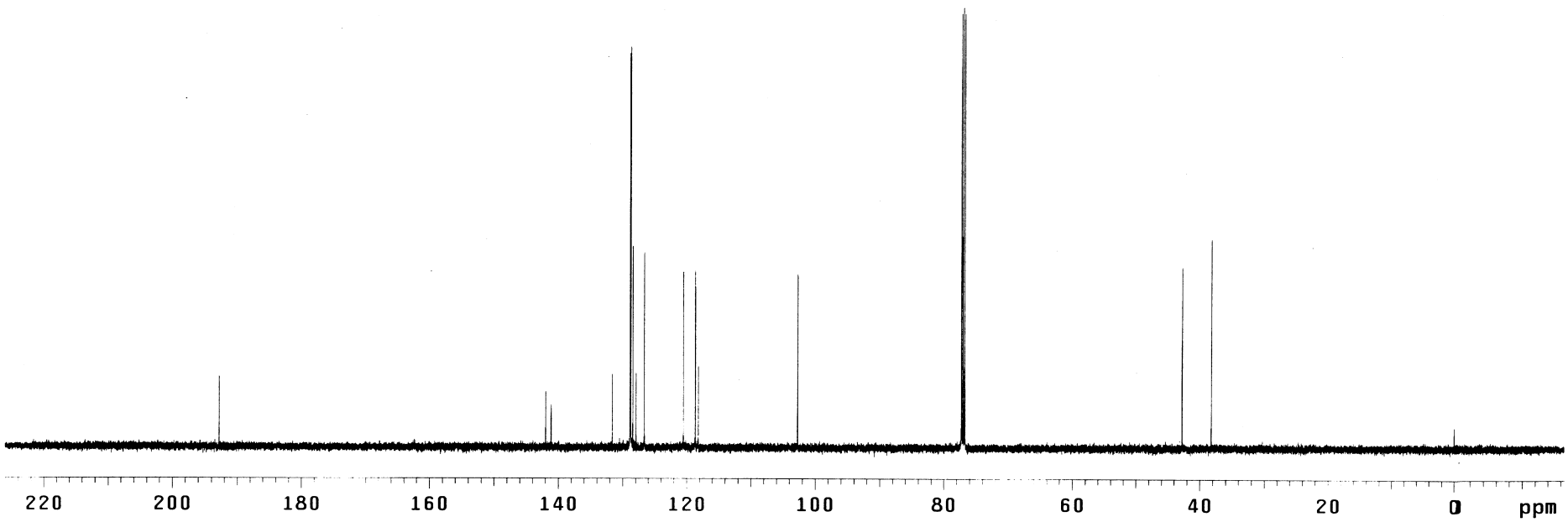





```
  Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_10
  File : exp
  Sample id : tmpstudy

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

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 30487.8 Hz
 800 repetitions
OBSERVE  C13, 125.6746053 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 30 min, 47 sec
```

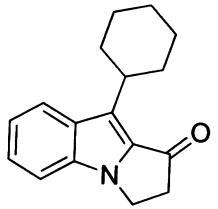



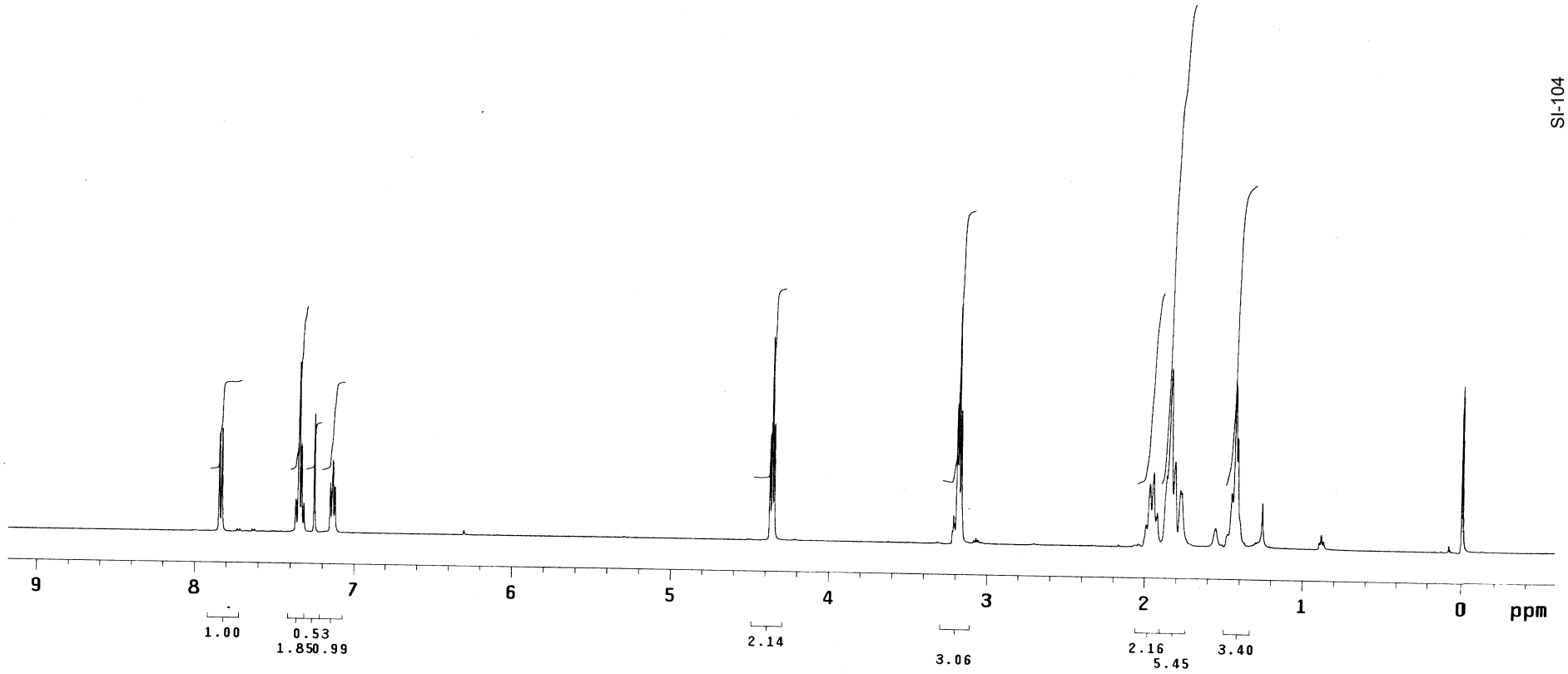
File: hxg-3-158-2H

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
File: hxg-3-158-2H
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, 499.7991854 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

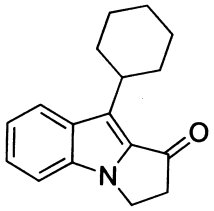


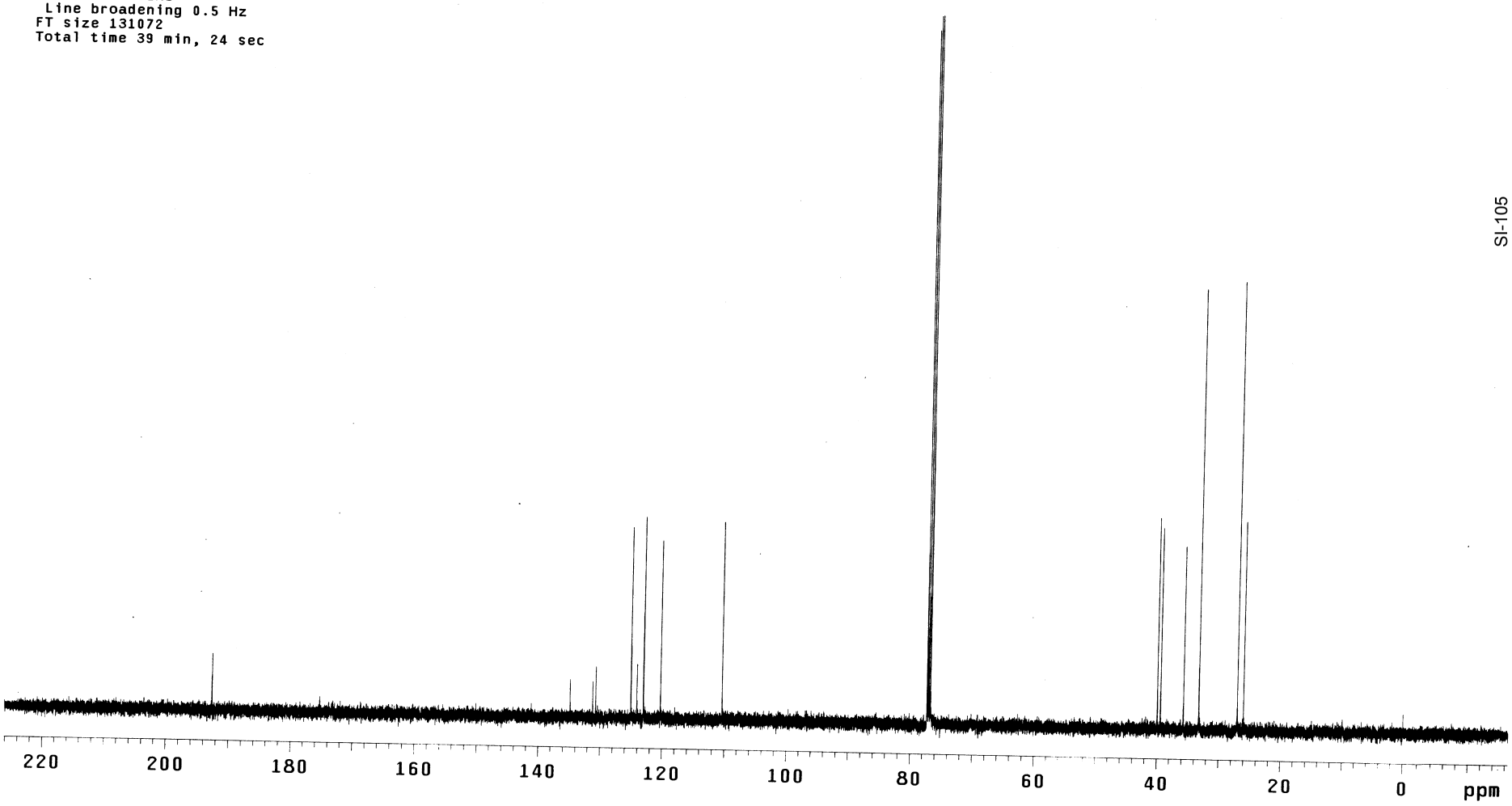


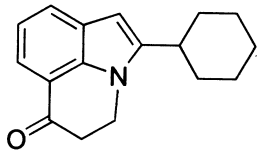
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_13
File : exp
Sample id : tmpstudy

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
384 repetitions
OBSERVE C13, 125.6746039 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



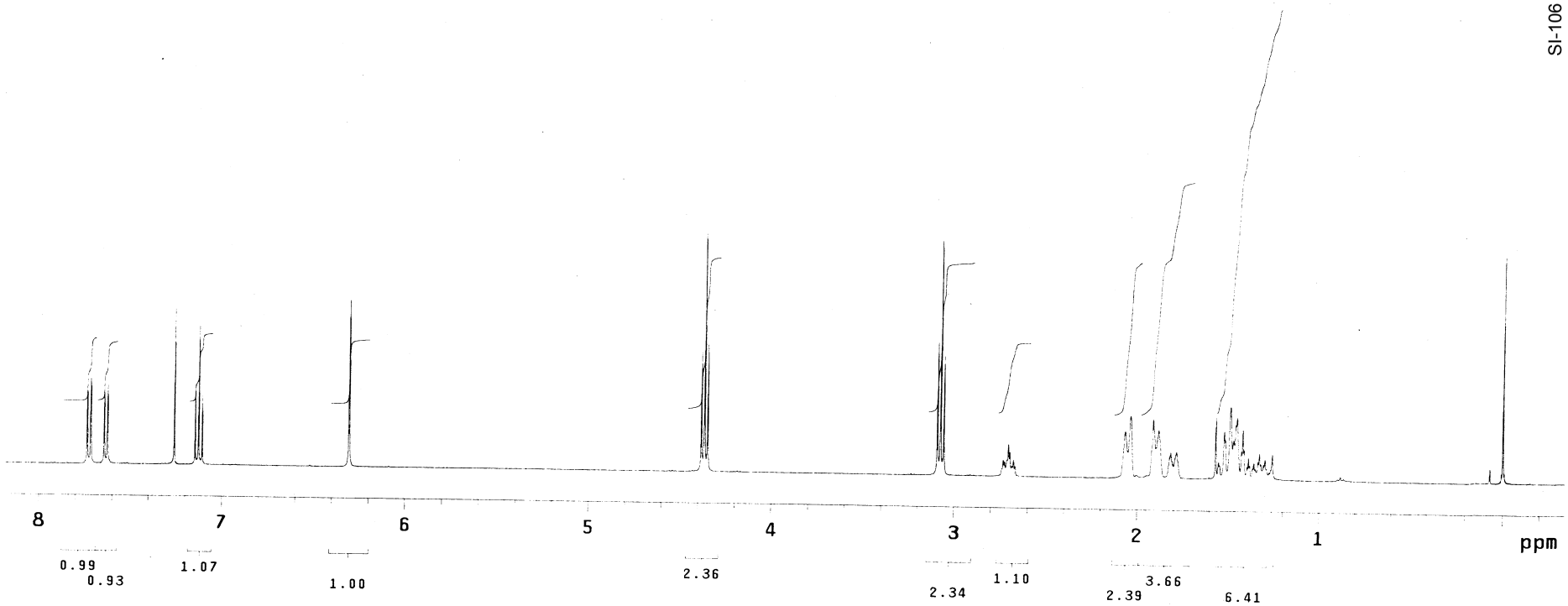


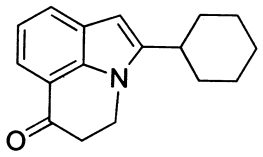



```
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.11_14
File : s_20070611_001/data/cdcl3_02.fid
Sample id : s_20070611_001

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: walkup
File: cdcl3_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, 399.8656733 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: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: walkup
VNMRS-500 "nmr500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30487.8 Hz
2368 repetitions
OBSERVE C13, 125.6746039 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 1 hr, 55 min, 28 sec

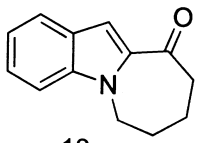
220 200 180 160 140 120 100 80 60 40 20 0 ppm

File: hxg-4-13H

ulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
perator: walkup
ile: hxg-4-13H
NOVA-500 "redjacket"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
8 repetitions
OBSERVE H1, 499.7991866 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



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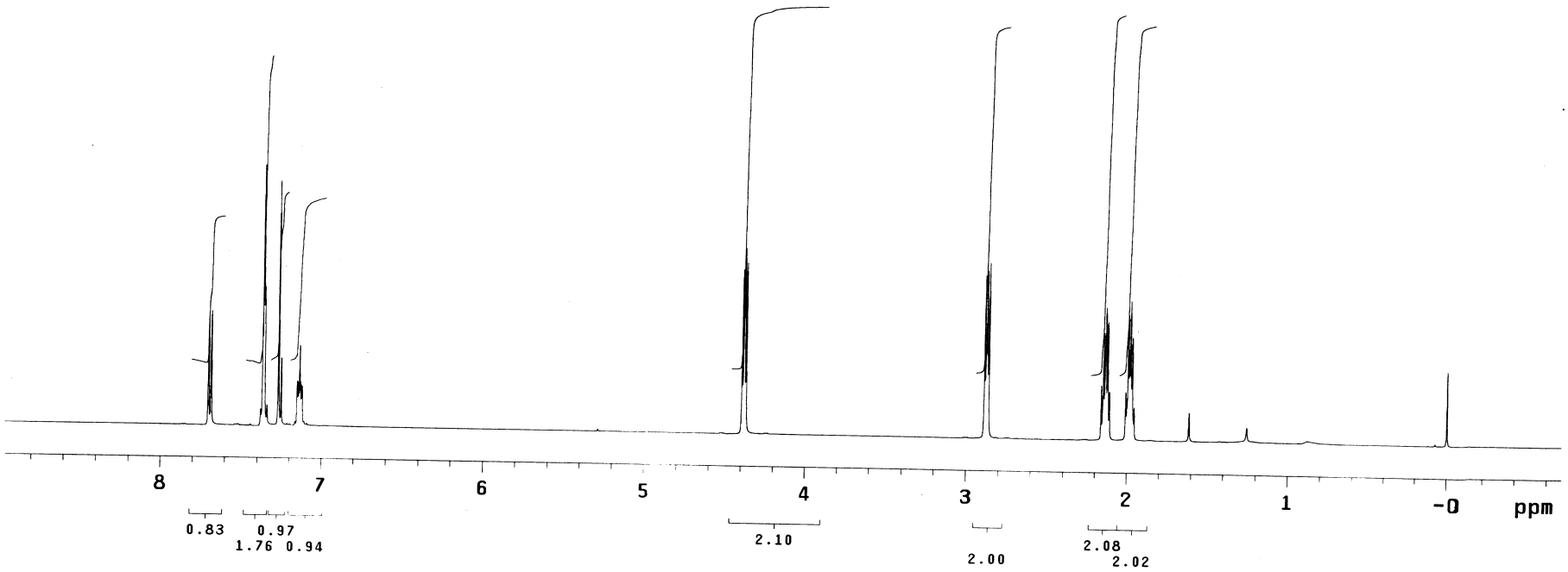
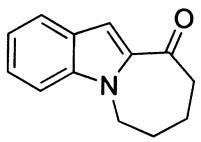
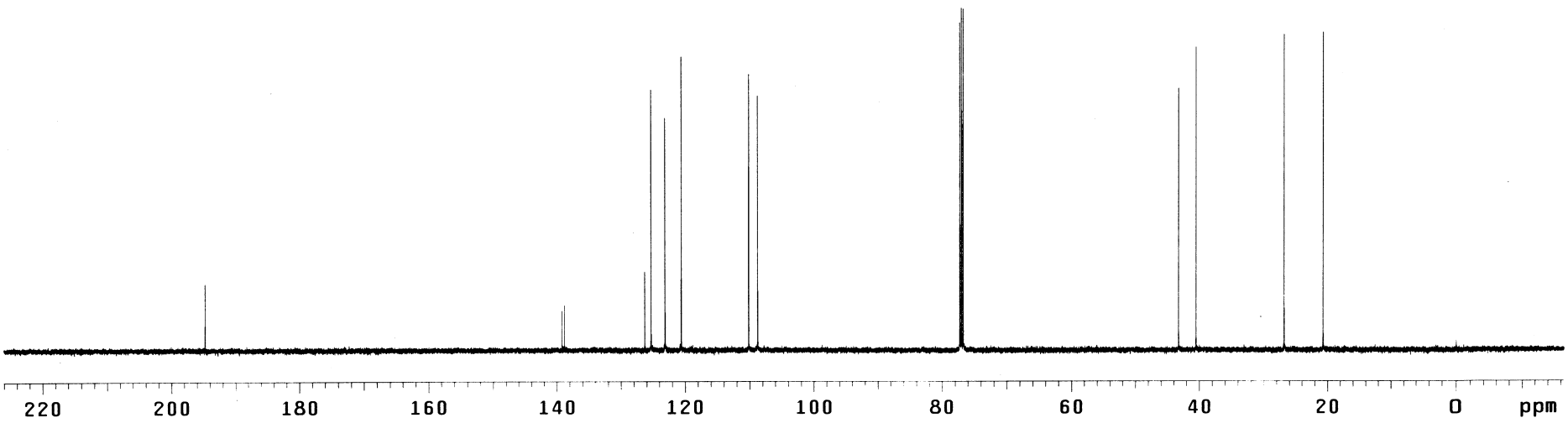
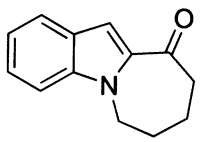




13

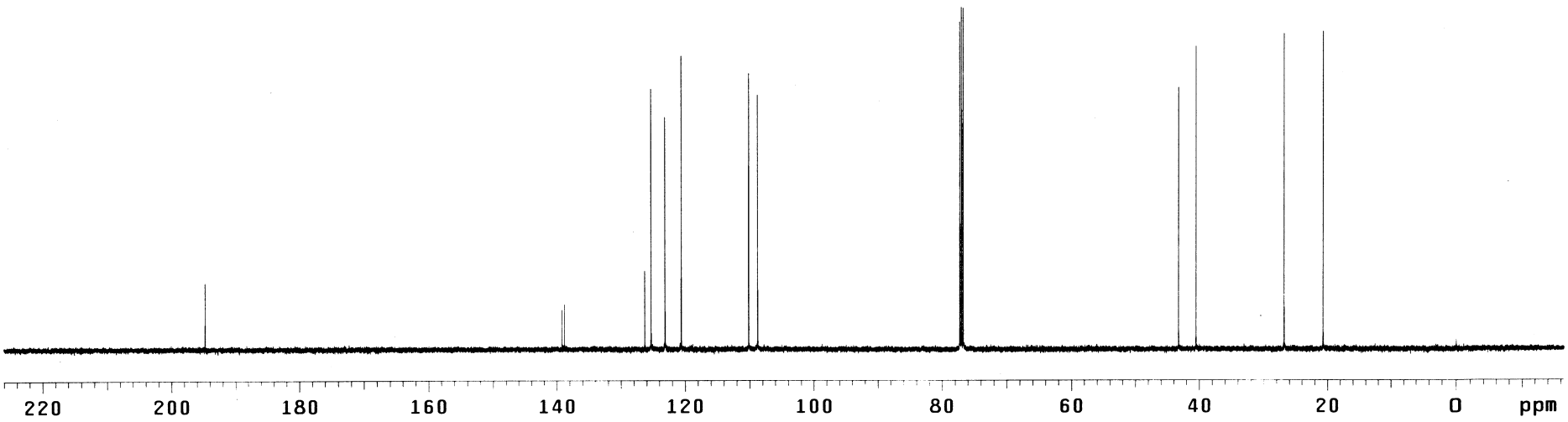
```
  Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_14
  File : exp
  Sample id : tmpstudy

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

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



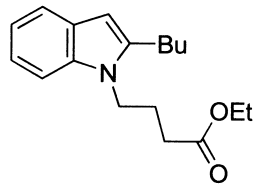
13

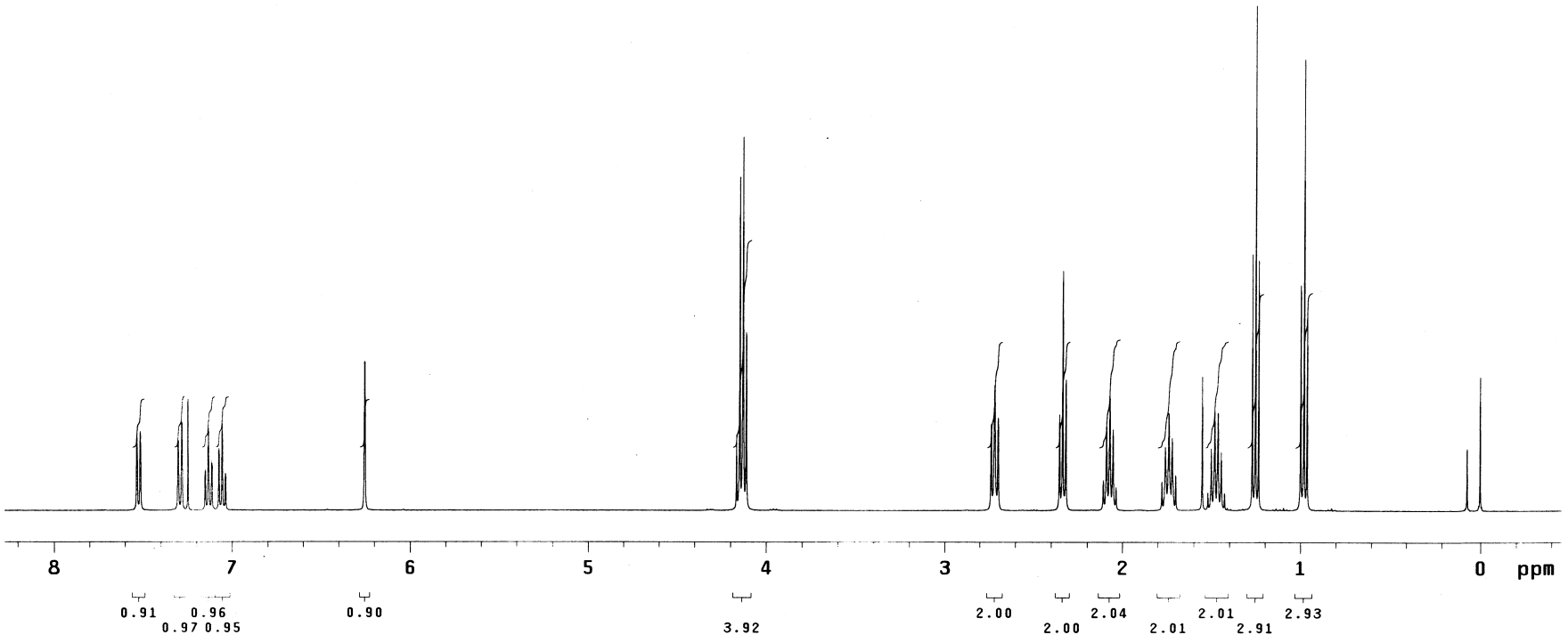
```
Automation directory: /home/walkup/vnmrsys/data/auto_2007.06.25_14
File : exp
Sample id : tmpstudy

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

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

exp1 Proton

SAMPLE		SPECIAL	
date	Aug 31 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	nmr400/walkup/data~	hst	0.008
	/Zhang/GuotaoLi/K3~	pw90	13.400
	77-1b.fid	alfa	6.600
ACQUISITION		FLAGS	
sw	6410.3	il	n
at	2.049	in	n
np	26264	dp	y
fb	4000	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.20
nt	8	fn	65536
ct	8	DISPLAY	
TRANSMITTER		sp	-182.9
tn	H1	wp	3490.9
sfrq	399.866	rfl	805.9
tof	399.6	rfp	0
tpwr	59	rp	27.7
pw	6.700	lp	-27.0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	328
dmm	c	th	12
dpwr	34	ai cdc ph	
dmf	29412		





exp1 Carbon

SAMPLE		SPECIAL	
date	Aug 31 2007	temp	25.0
solvent	cdcl3	gain	30
file	/home/walkup/~	spin	not used
	vnmrsys/data/auto_~	hst	0.008
	2007.08.31_02/Aug.~	pw90	15.300
	01/data/cdcl3_02.f~	alfa	10.000
	id	FLAGS	
ACQUISITION		il	n
sw	30487.8	in	n
at	1.300	dp	y
np	79298	hs	nn
fb	17000	PROCESSING	
bs	64	lb	0.50
d1	1.000	fn	not used
nt	3000	DISPLAY	
ct	64	sp	-2081.2
TRANSMITTER		wp	30487.3
tn	C13	rfl	11758.6
sfrq	125.687	rfp	9676.9
tof	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	ai cdc ph	
dpwr	39		
dmf	12200