

Supporting Information For

Nitrogen Heterocycles via Palladium-Catalyzed Carbocyclization.

Formal Synthesis of (+)- α -Allokainic Acid

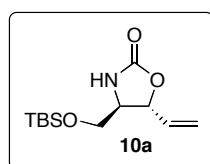
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Experimental Section

Thin layer chromatography (TLC) was performed on silica gel Whatman-60F glass plates, and components were visualized by illumination with UV light or by staining with potassium permanganate solution. Chromatography was performed using silica E. Merck silica gel 60 (230-400 mesh). NMR spectra were recorded in CDCl₃ on a Varian Inova 500 MHz, 400 MHz, or Varian Mercury 300 MHz spectrometer. ¹³C NMR was recorded using broad band proton decoupling. Chemical shifts are reported in δ relative to TMS and coupling constants in Hz. Optical rotations were recorded on a JASCO-CIP-370 instrument. Elemental analyses were performed on a Perkin Elmer Series II CHNS/O Analyzer 2400. High-resolution mass spectra (HRMS) were obtained at the Mass Spectrometry Laboratory, Department of Chemistry, The Ohio State University, Columbus, OH. Melting points were determined without correction. Reactions were carried out under an inert atmosphere of nitrogen or argon. Solvents were dried using a nitrogen pressurized alumina column system from Solvtek.

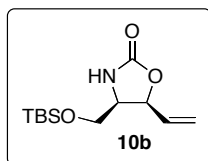
Isomerization of 10a and b: A 1.5:1 mixture of **10a,b** (1.27 g, 4.93 mmol) were placed in a schlenk flask along with Pd₂dba₃•CHCl₃ (102 mg, 0.10 mmol) and dppp (163 mg, 0.40 mmol). The flask was evacuated and purged with nitrogen three times. Degassed toluene (75 mL) was added. The reaction was allowed to stir at ambient temperature. After 16 hours, the reaction mixture was filtered through celite and concentrated under vacuum. The residue was purified by silica gel column chromatography (hexane:EtOAc, 4:1) to afford **10a** (1.13 g, 4.39 mmol) in 89% yield and **10b** (0.143 g, 0.56 mmol) in 11% yield.



trans-(4*R*,5*R*)-4-(*tert*-Butyl-dimethyl-silanyloxymethyl)-5-vinyl-oxazolidin-2-one

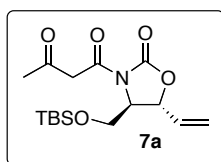
10a: [α]_D = 48.7° (c = 1.07, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 5.89 (ddd, *J* = 16.8, 10.4, 6.4 Hz, 1H), 5.37 (dd, *J* = 16.8, 1.2 Hz, 1H), 5.27 (d, *J* = 10.4 Hz, 1H), 3.63-3.58 (m, 3H), 0.86 (s, 9H), 0.04 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 147.8, 123.4, 107.0, 68.2,

53.1, 48.1, 14.5, 7.0, -16.7; IR (film): 3277, 2929, 2857, 1755, 1255, 1120 cm^{-1} ; HRMS calcd for $\text{C}_{12}\text{H}_{23}\text{SiNO}_3$ ($\text{M}+\text{Na}$) 280.1339, found 280.1346.



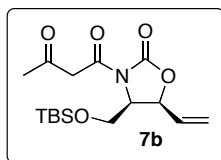
***cis*-(4*R*,5*S*)-4-(*tert*-Butyl-dimethyl-silanyloxymethyl)-5-vinyl-oxazolidin-2-one 10b:**

$[\alpha]_{\text{D}} = 43.4^\circ$ ($c = 1.19$, CH_2Cl_2); ^1H NMR (400 MHz, CDCl_3): δ 5.89 (ddd, $J = 17.2$, 10.4, 7.2 Hz, 1H), 5.44 (ddd, $J = 17.2$, 1.2, 1.2 Hz, 1H), 5.33 (ddd, $J = 10.4$, 1.2, 1.2 Hz, 1H), 5.07-5.03(m, 1H), 3.86-3.83(m, 1H), 3.60-3.49(m, 2H), 0.85(s, 9H), 0.02(s, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 148.0, 119.4, 108.9, 67.9, 51.0, 45.8, 14.5, 6.9, -16.8; IR (film): 3277, 2953, 2929, 2885, 2857, 1759, 1254, 1132, 1099 cm^{-1} ; HRMS calcd for $\text{C}_{12}\text{H}_{23}\text{SiNO}_3$ ($\text{M}+\text{Na}$) 280.1339, found 280.1344.



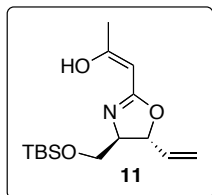
Preparation of (4*R*,5*R*)-1-[4-(*tert*-Butyl-dimethyl-silanyloxymethyl)-2-oxo-5-vinyl-oxazolidin-3-yl]-butane-1,3-dione 7a:

To a solution of **10a** (1.09 g, 4.23 mmol) in toluene (22 mL) was added dioxinone (0.84 mL, 6.34 mmol) dropwise. After 15 min, the mixture was heated at reflux for 2 hrs. The solvent was distilled under vacuum, and the residue was purified by silica gel column chromatography (hexane:EtOAc, 3:1) to afford **7a** (1.38 g, 4.04 mmol) in 96% yield. $[\alpha]_{\text{D}} = -15.8^\circ$ ($c = 1.34$, CH_2Cl_2); ^1H NMR (500 MHz, CDCl_3): δ 0.05 (d, $J = 4.8$ Hz, 6H), 0.87 (s, 9H), 2.26 (s, 3H), 3.79 (dd, $J = 3.0$, 10.5 Hz, 1H), 3.89 (d, $J = 15.9$ Hz, 1H), 3.93 (dd, $J = 4.8$, 10.5 Hz, 1H), 4.18 (d, $J = 15.9$ Hz, 1H), 4.24 (dt, $J = 3.0$, 4.8 Hz, 1H), 4.92-4.95 (m, 1H), 5.35 (d, $J = 10.5$ Hz, 1H), 5.43 (d, $J = 16.8$ Hz, 1H), 5.90 (ddd, $J = 16.8$, 10.5, 6.6 Hz, 1H). ^{13}C NMR (75.5 MHz, CDCl_3): δ 201.0, 166.94, 133.9, 119.1, 76.8, 61.1, 60.6, 51.6, 30.4, 25.9, 18.3, -5.4; IR (film): 2954, 2939, 2885, 2857, 1785, 1728, 1703, 1377, 1255, 1124, 1049 cm^{-1} ; HRMS calcd for $\text{C}_{16}\text{H}_{27}\text{SiNO}_5$ ($\text{M}+\text{Na}$) 364.1551, found 364.1557.



Preparation of (4*R*,5*S*)-1-[4-(*tert*-Butyl-dimethyl-silanyloxymethyl)-2-oxo-5-vinyl-oxazolidin-3-yl]-butane-1,3-dione 7b:

The procedure was identical to the preparation of **7a**. **7b** was obtained (560 mg, 1.64 mmol) in 63% yield. $[\alpha]_{\text{D}} = -61.7^\circ$ ($c = 1.24$, CH_2Cl_2); ^1H NMR (500 MHz, CDCl_3): δ 0.00 (s, 3H), 0.02 (s, 3H), 0.85 (s, 9H), 2.25 (s, 3H), 3.68 (dd, $J = 9.5$, 1.5 Hz, 1H), 3.79 (d, $J = 16.5$ Hz, 1H), 3.99 (dd, $J = 11.5$, 3.5 Hz, 1H), 4.26 (d, $J = 16.5$ Hz, 1H), 4.47-4.44 (m, 1H), 5.01 (dd, $J = 8.8$ Hz, 1H), 5.46 (d, $J = 11.5$ Hz, 1H), 5.49 (d, $J = 17.5$ Hz, 1H), 6.11 (ddd, $J = 17.5$, 11.5, 8.0 Hz, 1H); ^{13}C NMR (125.5 MHz, CDCl_3): δ 201.0, 166.5, 153.8, 130.5, 123.0, 58.8, 58.7, 51.5, 30.4, 25.8, 18.0, -5.6; IR (film): 2954, 2930, 2884, 2857, 1781, 1366, 1193, 1066 cm^{-1} ; HRMS calcd for $\text{C}_{16}\text{H}_{27}\text{SiNO}_5$ ($\text{M}+\text{Na}$) 364.1551, found 364.1548.

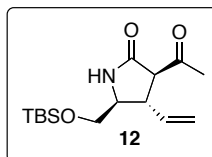


Pd-Catalyzed isomerization of 7a,b to (4*R*,5*R*)-1-[4-(*tert*-Butyl-dimethyl-silanyloxymethyl)-5-vinyl-4,5-dihydro-oxazol-2-yl]-propen-2-ol **11:** To a solution of **7** (120 mg, 0.35 mmol) in THF (4 mL) was added *t*-BuLi dropwise at 0° C. After 15 min, a solution of AllylPdCl₂ (3.2 mg, 0.009 mmol) and dppp (14.4 mg, 0.035 mmol) in

THF (0.6 mL) was added to the mixture and it mixture was allowed to stir overnight at room temperature. Aqueous sat. NH₄Cl was added and the organics were extracted with EtOAc, washed with Brine, dried over MgSO₄, and concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 3:1) to give **11** (86 mg, 0.29 mmol) in 83% yield. **Alternative method:** A solution of **7** (100 mg, 0.29 mmol), Pd₂(dba)₃.CHCl₃ (9.1 mg, 0.009 mmol), dppp (14.5 mg, 0.035 mmol) and BSA (206 mg, 1.0 mmol) in THF (5 mL) was heated to reflux for 19 hrs. The solvent was evaporated under vacuum and the residue was purified by silica gel column chromatography (Hexane:EtOAc, 3:1) to give **11** (62 mg, 0.21 mmol) in 72% yield. [α]_D = -6.8° (c = 0.71, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃): δ 0.05 (s, 3H), 0.06 (s, 3H), 0.88 (s, 9H), 2.01(d, *J* = 2.4 Hz, 3H), 3.80-3.61 (m, 4H), 4.90-4.80 (m, 1H), 4.91 (s, 1H), 5.32 (d, *J* = 10.5 Hz, 1H), 5.40 (d, *J* = 16.8 Hz, 1H), 5.95-5.83 (m, 1H); ¹³C NMR (75.5 MHz, CDCl₃): δ 194.5, 168.8, 134.3, 119.3, 117.2, 82.5, 77.7, 64.0, 62.3, 29.0, 26.0, 184, -5.2, -5.3; IR (film): 3272, 2954, 2929, 2896, 2857, 1637, 1558 cm⁻¹; HRMS calcd for C₁₅H₂₇SiNO₃ (M+Na) 320.1659, found 320.1657.

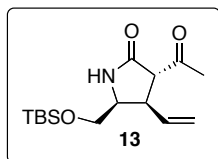
Typical procedure for the Pd-catalyzed cyclization of 7a:

To a mixture of **7a** (50 mg, 0.15 mmol), Pd₂(dba)₃.CHCl₃ (4.5 mg, 0.004 mmol) and dppp (7.2 mg, 0.017 mmol) was added a freshly prepared solution of EtOLi in EtOH (0.12 M, 3 mL) dropwise at room temperature. The mixture was stirred at room overnight. It was then quenched by saturated aqueous NH₄Cl solution, extracted with EtOAc, washed with Brine, dried over MgSO₄, and concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 4:1) to afford a mixture of **12** and **13** (40 mg, 0.13 mmol) in 92% combined yield. Further purification was carried if necessary by silica gel column chromatography (CH₂Cl₂:MeOH, 60:1) to afford pure **12** and **13**.

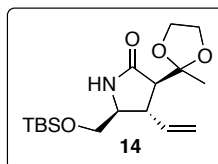


(3*R*,4*S*,5*S*)-3-Acetyl-5-(*tert*-butyl-dimethyl-silanyloxymethyl)-4-vinyl-pyrrolidin-2-one **12:** [α]_D = 35.6° (c = 1.24, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃): δ 0.04 (m, 6H), 0.86 (s, 9H), 2.41 (s, 3H), 3.17 (dd, *J* = 8.0, 16.5 Hz, 1H), 3.50-3.42 (m, 3H), 3.75 (dd, *J* = 2.5, 9.5 Hz, 1H), 5.06 (dt, *J* = 1.0, 10.0 Hz, 1H), 5.10 (dt, *J* = 1.0, 17.0 Hz, 1H), 5.68 (ddd, *J* = 8.0, 10.0, 16.5 Hz, 1H); ¹³C NMR (75.5 MHz, CDCl₃): δ 202.5, 171.4, 136.7, 118.1, 65.8, 60.2, 59.0, 42.0,

30.9, 26.0, 18.4, -5.2; IR (film): 3220, 3085, 2953, 2929, 2885, 2857, 1701, 1360, 1256, 1119 cm^{-1} ; HRMS calcd for $\text{C}_{15}\text{H}_{27}\text{SiNO}_3$ ($\text{M}+\text{Na}$) 320.1652, found 320.1661.

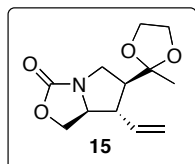


(3*S*,4*R*,5*S*)-3-Acetyl-5-(*tert*-butyl-dimethyl-silanyloxymethyl)-4-vinyl-pyrrolidin-2-one **13:** $[\alpha]_{\text{D}} = 79.4^\circ$ ($c = 1.20$, CH_2Cl_2); ^1H NMR (500 MHz, CDCl_3): δ 0.04 (s, 3H), 0.05 (s, 3H), 0.09 (s, 9H), 2.36 (s, 3H), 3.54 (d, $J = 9.5$ Hz, 1H), 3.66-3.55 (m, 4 H), 5.12 (dd, $J = 10$, 1.5 Hz, 1H), 5.18 (d, $J = 17.5$ Hz, 1H), 5.78 (ddd, $J = 17.5$, 10, 8.5 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ -5.4, -5.3, 18.3, 26.0, 31.0, 43.2, 56.8, 60.0, 63.8, 119.3, 134.5, 172.9, 203.2; IR (film): 3227, 3082, 2953, 2928, 2857, 1702, 1359, 1253, 1126 cm^{-1} ; HRMS calcd for $\text{C}_{15}\text{H}_{27}\text{SiNO}_3$ ($\text{M}+\text{Na}$) 320.1652, found 320.1654.



Preparation of (3*R*,4*S*,5*S*)-5-(*tert*-Butyl-dimethyl-silanyloxymethyl)-3-(2-methyl-[1,3]dioxolan-2-yl)-4-vinyl-pyrrolidin-2-one **14:** A stirred solution of **13** (306 mg, 1.0 mmol), ethylene glycol (280 mg, 4.11 mmol) and PPTS (50 mg, 0.20 mmol) in toluene (12 mL) were refluxed for 3h with azeotropic removal of water. Additional

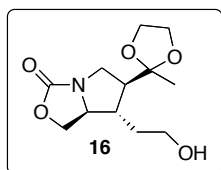
ethylene glycol (280 mg, 4.11 mmol) and PPTS (50 mg, 0.20 mmol) were added to the mixture, and refluxed for another 3h. The solution was cooled to room temperature and Et_3N (1 mL) was added. The mixture was concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 1:1) to give **14** (150 mg, 0.44 mmol) in 43% yield. $[\alpha]_{\text{D}} = 35.8^\circ$ ($c = 0.97$, CH_2Cl_2); ^1H NMR (500 MHz, CDCl_3): δ 0.02 (s, 6H), 0.85 (s, 9H), 1.48 (s, 3H), 2.59 (dd, $J = 15.2$, 8.0 Hz, 1H), 2.63 (d, $J = 8.8$ Hz, 1H), 3.34 (ddd, $J = 7.6$, 7.6, 3.2 Hz, 1H), 3.40 (dd, $J = 9.2$, 8.0 Hz, 1H), 3.66 (dd, $J = 9.6$, 3.2 Hz, 1H), 3.97-3.87 (m, 4H), 5.04 (d, $J = 17.2$ Hz, 1H), 5.04 (dd, $J = 11.2$ Hz, 1H), 5.81 (ddd, $J = 17.2$, 11.2, 10.0 Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ -5.2, 18.4, 22.9, 26.0, 43.0, 55.1, 58.4, 65.0, 65.1, 65.6, 109.8, 116.1, 139.4, 174.4; IR (film): 3214, 3082, 2953, 2929, 2885, 2857, 1699, 1253, 1122, 1044 cm^{-1} ; HRMS calcd for $\text{C}_{17}\text{H}_{31}\text{SiNO}_4$ ($\text{M}+\text{Na}$) 364.1914, found 364.1932.



Preparation of (3*S*,4*S*,5*S*)-6-(2-Methyl-[1,3]dioxolan-2-yl)-7-vinyl-tetrahydro-pyrrolo[1,2-*c*]oxazol-3-one **15:** A solution of **14** (302 mg, 0.88 mmol) in THF (3 mL) was added dropwise to a stirred suspension of LiAlH_4 (84 mg, 2.21 mmol) in THF (7

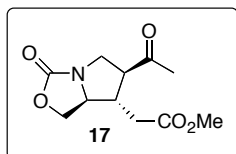
mL) at 0°C . The mixture was refluxed for 11 h and then cooled to 0°C . The reaction was quenched by sequential addition of water (0.08 mL), 15% NaOH solution (0.08 mL), and water (0.08 mL) and the mixture was stirred at rt until the gray color had completely disappeared. The suspension was filtered through a celite pad and the residue was washed with CH_2Cl_2 . The combined filtrates were concentrated in

vacuo and the residue was mixed with 10% NaOH solution (2.3 mL). A solution of methyl chloroformate (0.14 mL, 1.76 mmol) in CH₂Cl₂ (1.1 mL) was added dropwise to the mixture at 0°C. After 4 h, the mixture was extracted with EtOAc, washed with brine, dried over MaSO₄, filtered, concentrated under vacuum. The residue was dissolved in THF (5.6 mL), 60% NaH (70 mg, 1.76 mmol) was added to the solution at 0°C. The mixture was stirred at rt for 12h. Then it was quenched by saturated aqueous NH₄Cl solution, extracted with EtOAc, washed with brine, dried over MaSO₄, filtered, concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 5:4) to give **15** (135 mg, 0.56 mmol) in 64% overall yield. $[\alpha]_D = -3.1^\circ$ (c = 0.94, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃): δ 1.26 (s, 3H), 2.46 (dt, *J* = 9.0, 9.0 Hz, 1H), 2.53 (ddd, *J* = 15.0, 9.0, 6.0 Hz, 1H), 3.30 (dd, *J* = 12.0, 9.0 Hz, 1H), 3.70-3.64 (m, 2H), 4.00-3.91 (m, 4H), 4.16 (dd, *J* = 9.0, 2.5 Hz, 1H), 4.38 (dd, *J* = 8.5, 7.5 Hz, 1H), 5.11 (ddd, *J* = 10.5, 0.5, 0.5 Hz, 1H), 5.14 (d, *J* = 17.5 Hz, 1H), 5.71 (ddd, *J* = 17.5, 10.5, 8.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 161.5, 137.4, 117.8, 110.1, 66.2, 65.2, 65.1, 64.7, 54.5, 50.9, 47.7, 23.5; IR (film): 2980, 2896, 1755, 1393, 1208, 1039 cm⁻¹; HRMS calcd for C₁₂H₁₇NO₄ (M+Na) 262.1050, found 262.1061.



Preparation of (2S,6S,7S)-7-(2-Hydroxy-ethyl)-6-(2-methyl-[1,3]dioxolan-2-yl)-tetrahydro-pyrrolo[1,2-c]oxazol-3-one **16:**

To a solution of **15** (103 mg, 0.43 mmol) and RhCl(PPh₃)₃ in THF (4.5 mL) was added a solution of catecholborane (2.0M, 1.08 mL) dropwise at -15°C. After the mixture was stirred at for 30 minutes, it was allowed slowly warmed up to rt and stirred overnight. The mixture was cooled to 0°C, EtOH (0.44 mL) was added to quench the reaction. Then a solution of NaOH (3N, 0.6 mL) was added to the mixture followed by the addition of 30% H₂O₂ (0.86 mL). The mixture was stirred at 0°C for 1 h and rt for 1 h. The mixture was extracted with CHCl₃, washed with Brine, dried over MgSO₄, and concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 1:1) to give **16** (100 mg, 0.39 mmol) in 90% yield. $[\alpha]_D = 9.2^\circ$ (c = 1.08, CH₂Cl₂); ¹H NMR (400 MHz, CDCl₃): δ 1.29 (s, 3H), 1.67-1.58 (m, 1H), 2.07-1.90 (m, 3H), 2.32-2.27 (m, 1H), 3.20 (dd, *J* = 12.0, 8.8 Hz, 1H), 3.77-3.62 (m, 4H), 4.01-3.90 (m, 1H), 4.25 (dd, *J* = 9.2, 4.0 Hz, 1H), 4.48 (dd, *J* = 9.2, 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 161.2, 110.9, 68.8, 65.8, 65.2, 64.8, 61.4, 54.3, 47.1, 44.6, 36.6, 22.3; IR (film): 3437, 2894, 1732, 1404, 1219, 1073, 1041 cm⁻¹; HRMS calcd for C₁₂H₁₉NO₄ (M+Na) 280.1155, found 280.1154.



Preparation of (2S,6S,7R)-6-Acetyl-3-oxo-tetrahydro-pyrrolo[1,2-c]oxazol-7-yl-acetic acid methyl ester **17:**

To a solution of **16** (45 mg, 0.17 mmol) in Acetone (1.6 mL) Jones reagent (2.7M, 0.51 mL) was added dropwise at 0°C. The mixture was

stirred at for 1 h, then isopropanol (1.6 mL) was added to the mixture to quench the reaction. The mixture was extracted with CHCl_3 , washed with Brine, dried over MgSO_4 , concentrated under vacuum. The residue was dissolved in MeOH (1.6 mL), a solution of trimethylsilyldiazomethane (2.0M, 0.52 mL) in THF was added to the mixture. After 4 h, the mixture was concentrated under vacuum. The residue was dissolved in acetone (4 mL), and a catalytic amount of PTSA was added to the mixture. The mixture was stirred at rt for 36 h. The mixture was diluted with CHCl_3 , washed with brine, dried over MgSO_4 , and concentrated under vacuum. The residue was purified by silica gel column chromatography (Hexane:EtOAc, 9:7) to give **17** (23 mg, 0.095 mmol) 57% yield. $[\alpha]_{\text{D}} = -17.3^\circ$ ($c = 0.99$, CHCl_3); $[\alpha]_{\text{D}}^{\text{lit. 1}} = -17.9^\circ$ ($c = 1.1$, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 2.22 (s, 3H), 2.38 (dd, $J = 17.0, 10.5$ Hz, 1H), 2.63-2.56 (m, 2H), 3.08-3.03 (m, 1H), 3.55 (dd, $J = 11.5, 9.5$ Hz, 1H), 3.67 (s, 3H), 3.72 (dd, $J = 12.0, 6.5$ Hz, 1H), 3.80 (ddd, $J = 7.0, 7.0, 4.0$ Hz, 1H), 4.41 (dd, $J = 9.5, 4.0$ Hz, 1H), 4.53 (dd, $J = 9.5, 8.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 29.2, 35.8, 42.1, 47.5, 52.2, 58.2, 64.5, 68.1, 160.8, 172.0, 205.7.

¹ Hanessian, S.; Ninkovic, S. *J. Org. Chem.* **1996**, *61*, 5418.

sun-I-188H1

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Solvent: C6Cl3

Temp. 25.0 C / 298.1 K

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16 repetitions

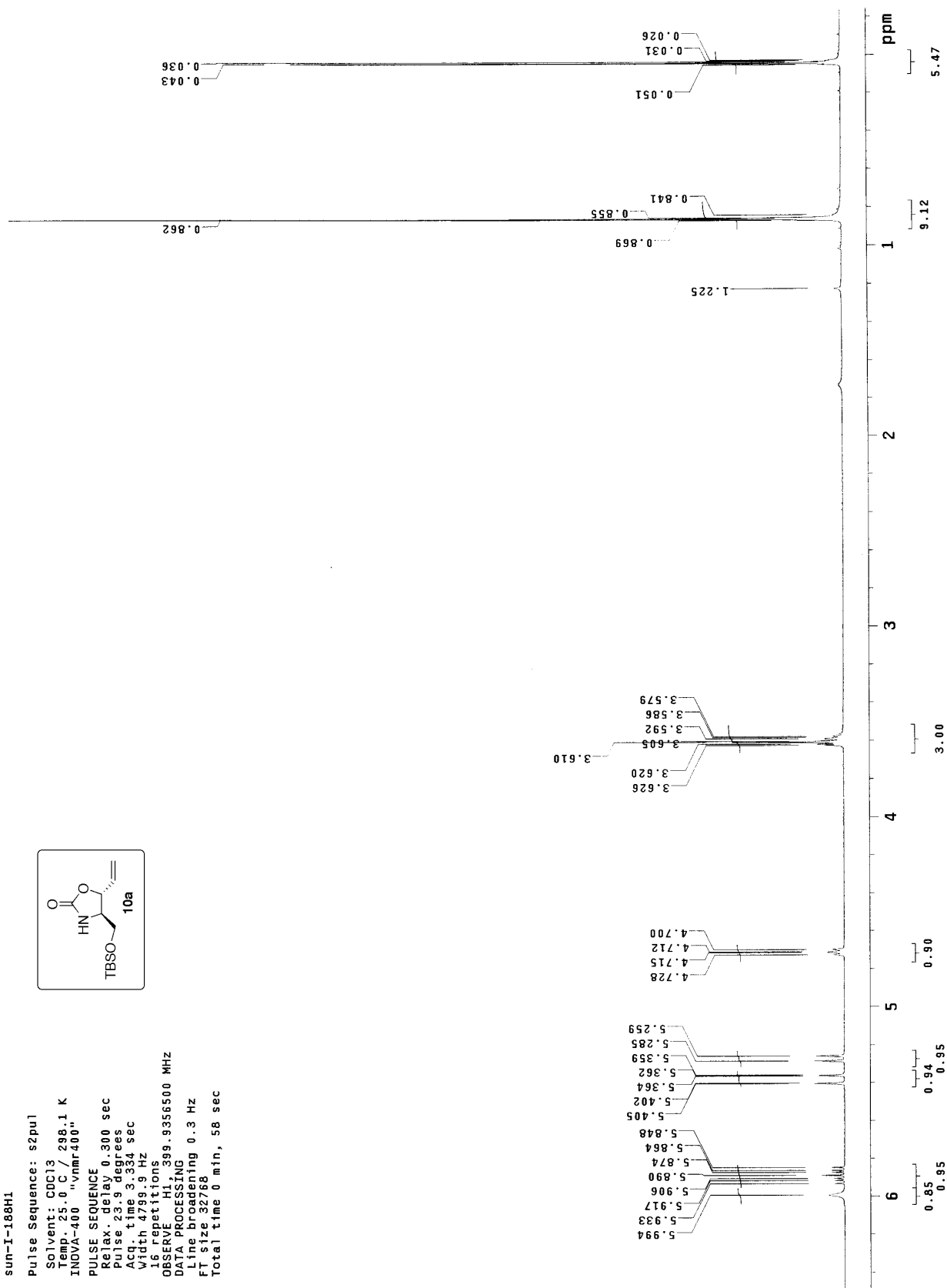
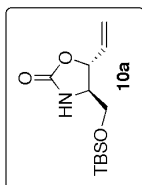
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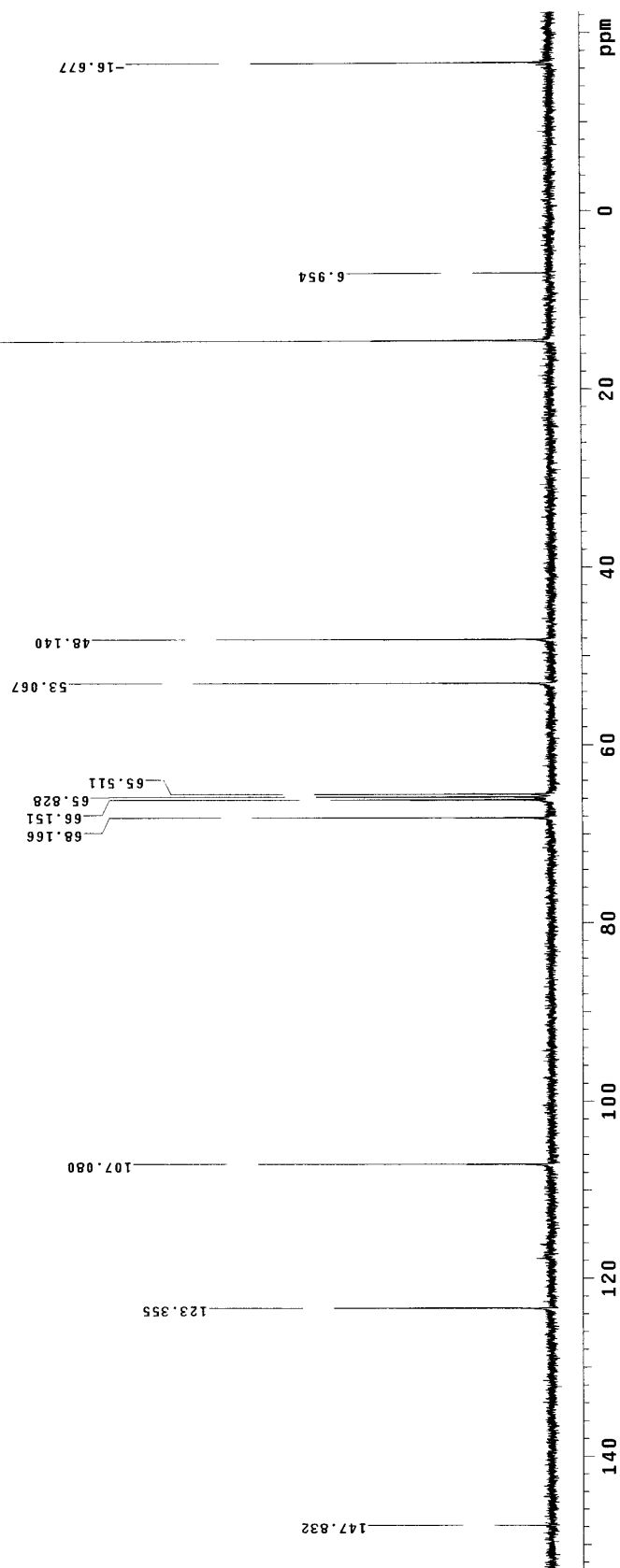
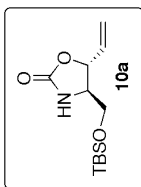
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Pulse Sequence: s2pul

Solvent: CDCl₃
Temp: 25.0 C / 298.1 K
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Width 24235.1 Hz
512 repetitions

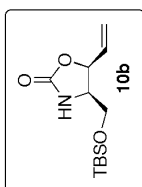
OBSERVE C13, 100.5650218 MHz
DECOUPLE H1, 399.9376496 MHz
Power 41 dB

on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 min, 48 sec



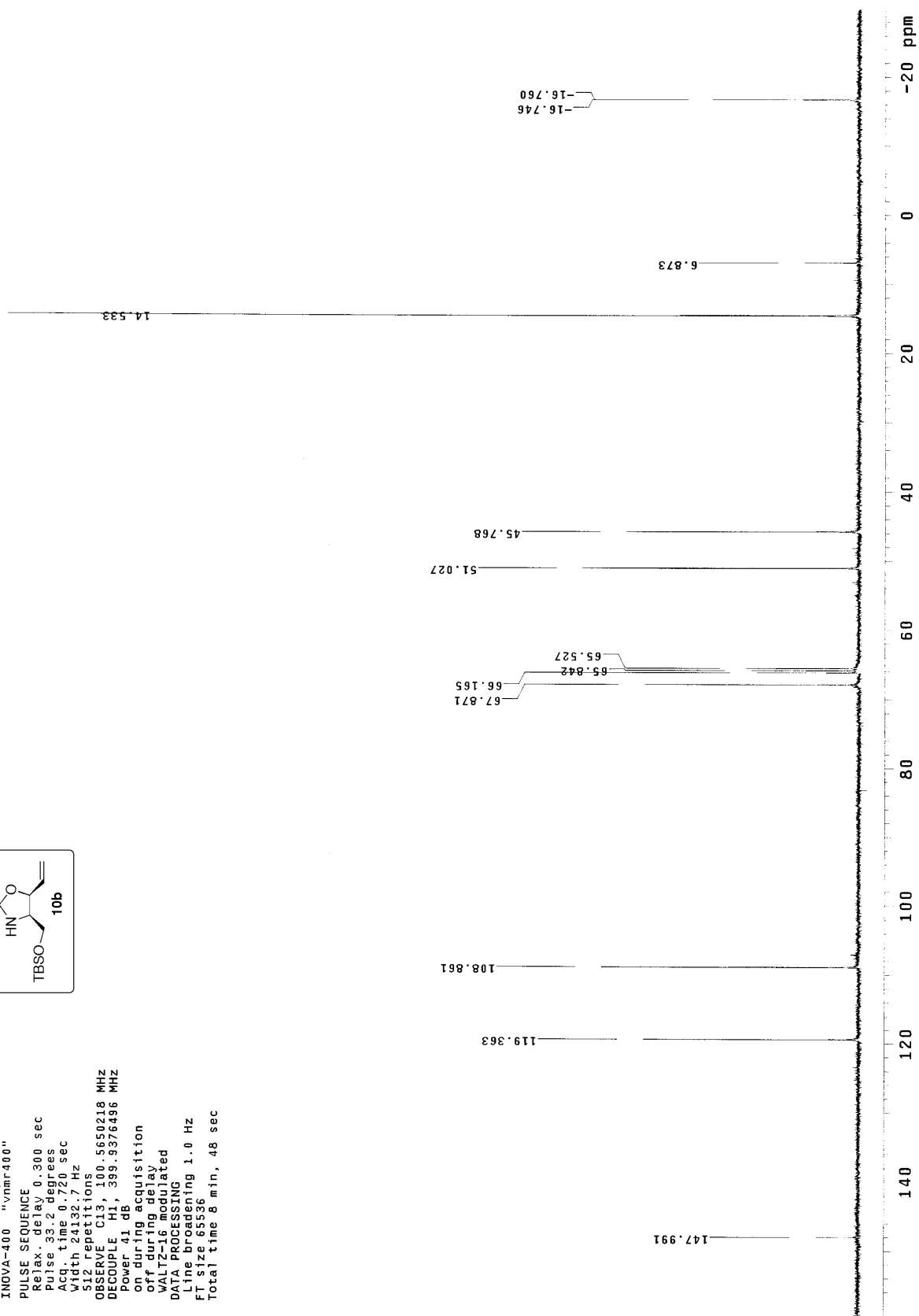
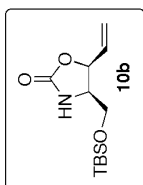
sun-I-64B

Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
INOVA-400 "vnmr400"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 23.9 degrees
Acq. time 3.334 sec
Width 4799.9 Hz
16 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 58 sec



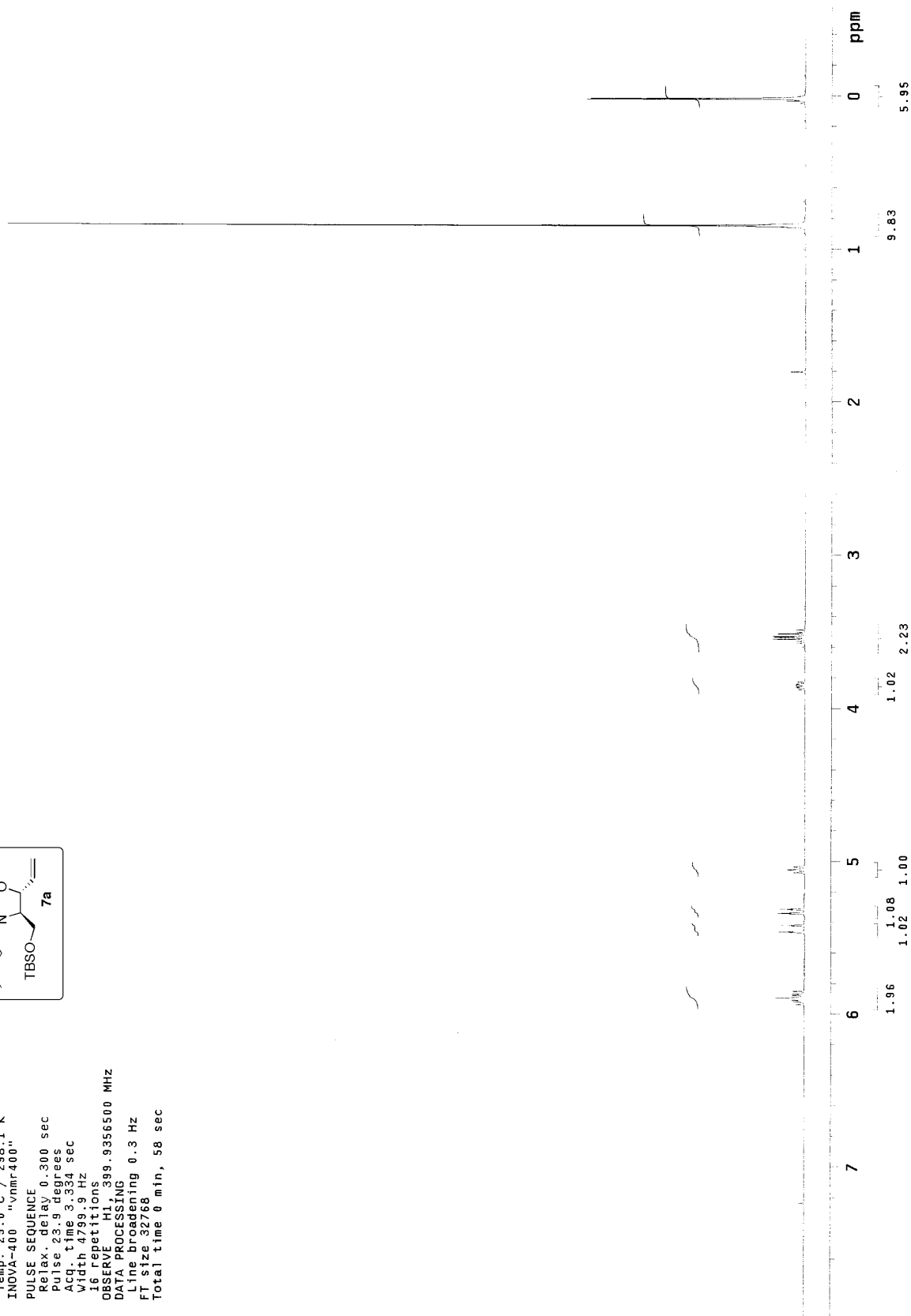
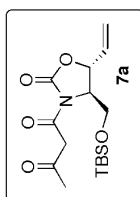
sun-i-648

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 25.0 C / 298.1 K
INOVA-400 "vnmr400"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 33.2 degrees
Acq. time 0.720 sec
Width 24132.7 Hz
512 repetitions
OBSERVE C13, 100.5650218 MHz
DECOUPLE H1, 399.9376496 MHz
Power 41 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 min, 48 sec



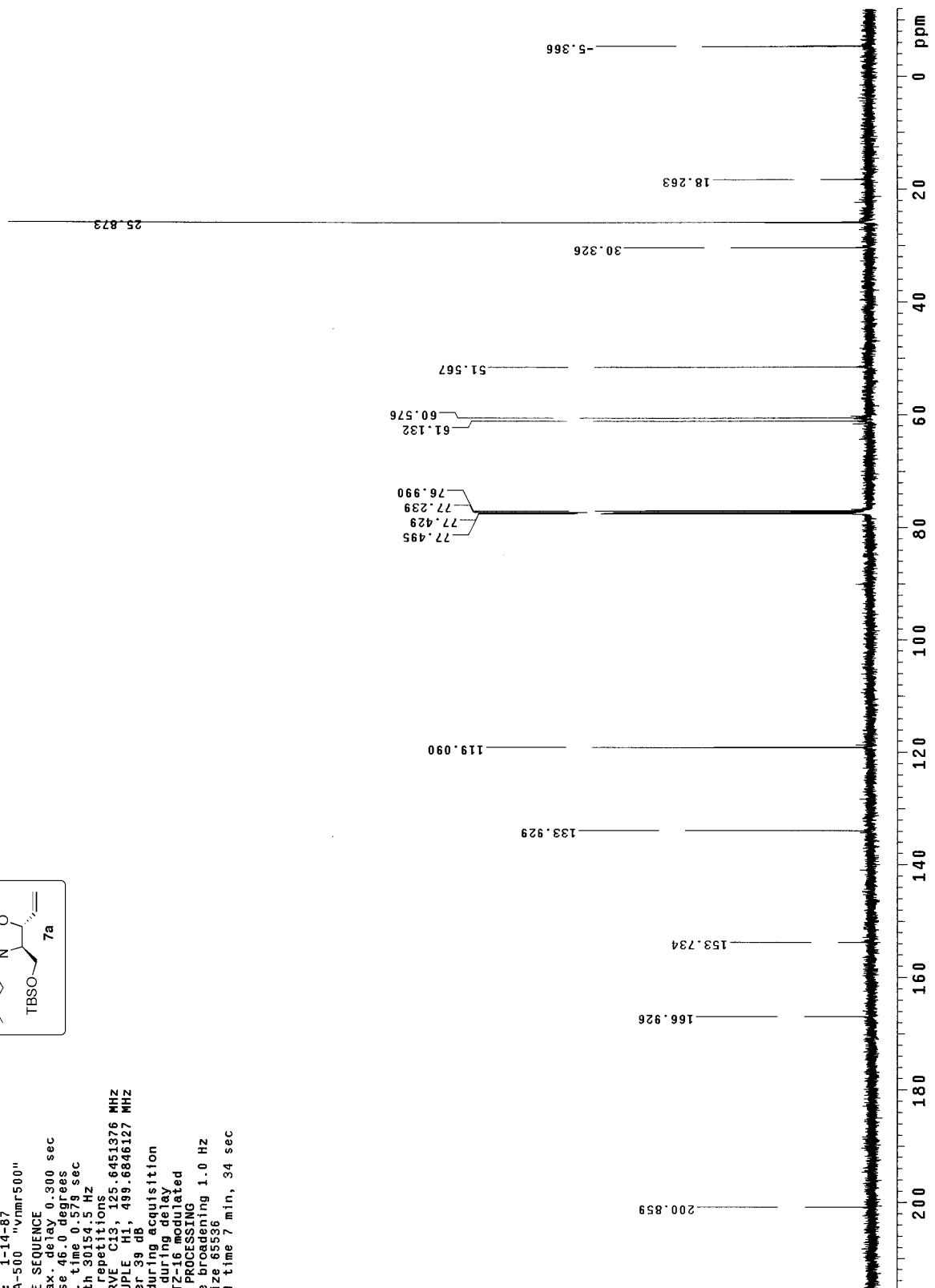
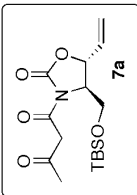
sun-I-64B

Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
INOVA-400 "vnmr400"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 23.9 degrees
Acq. time 3.334 sec
Width 4799.9 Hz
16 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 58 sec



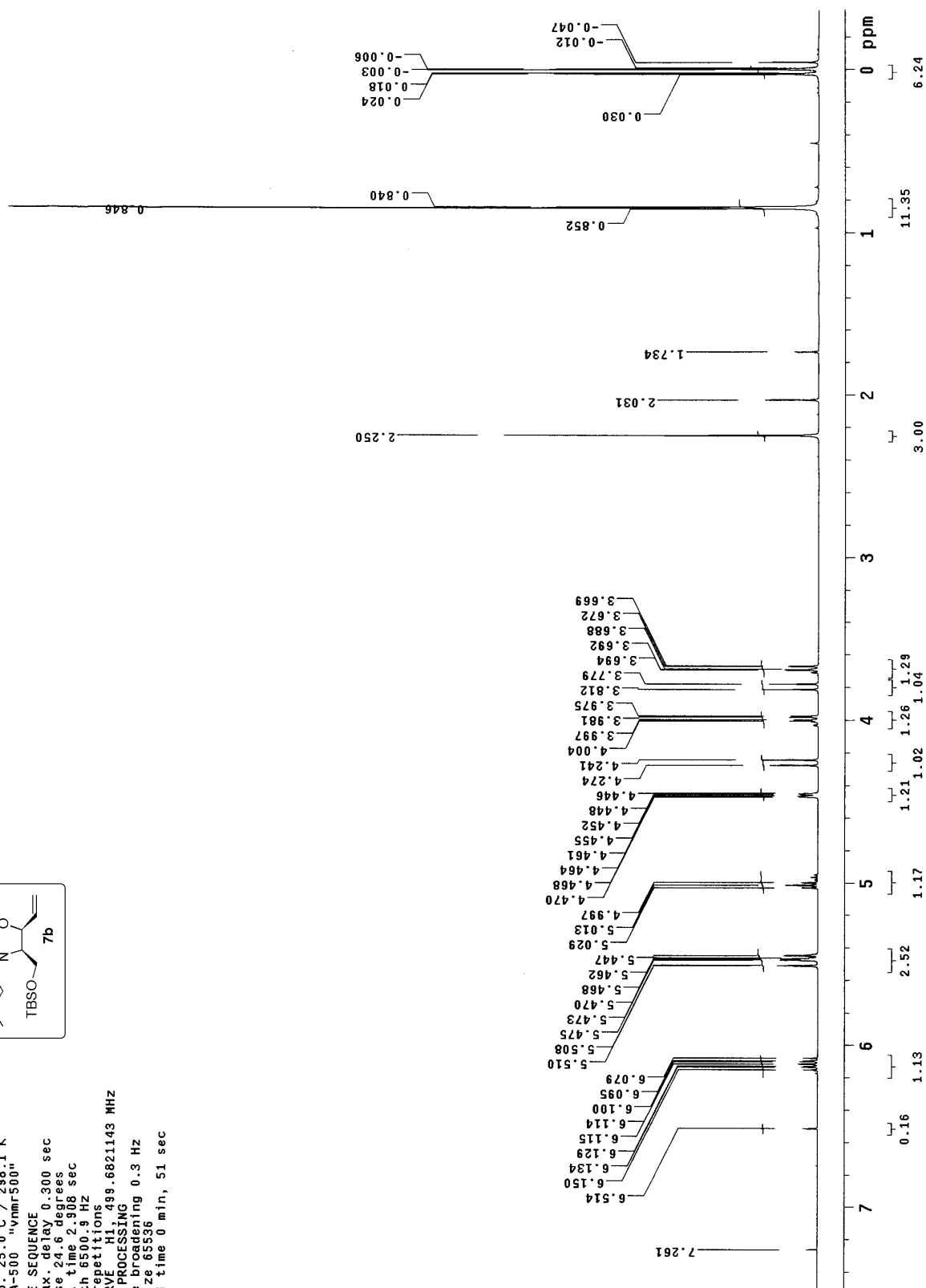
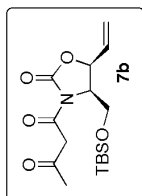
sun-I-176.S.M

Pulse Sequence: s2pu1
Solvent: CDCl₃
Temp: 25.0 C / 288.1 K
User: 1-14-87
INOVA-500 "vnmr500"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 46.0 degrees
Acq. time 0.579 sec
Width 30154.5 Hz
512 repetitions
OBSERVE C13, 125.6451376 MHz
DECUPLE H1, 499.6846127 MHz
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 min, 34 sec



sun-I-65B

Pulse Sequence: s2pu1
 Solvent: cdcl3
 Temp: 25.0 C / 298.1 K
 INOVA-500 "vnmr500"
 PULSE SEQUENCE
 Relax. delay 0.300 sec
 Pulse 24.6 degrees
 Acq. time 2.908 sec
 Width 6500.9 Hz
 16 repetitions
 OBSERVE H1, 499.6821143 MHz
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 65536
 Total time 0 min, 51 sec



sun-I-65B

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 25.0 C / 288.1 K

User: 1-14-87

INOVA-500 "vnmr500"

PULSE SEQUENCE

Relax. delay 0.300 sec

Pulse 46.0 degrees

Acq. time 0.579 sec

Width 30154.5 Hz

Siz 2 repetitions

OBSERVE C13, 125.6451376 MHz

DECOUPLE H1, 499.6846127 MHz

Power 39 dB

on during acquisition

off during delay

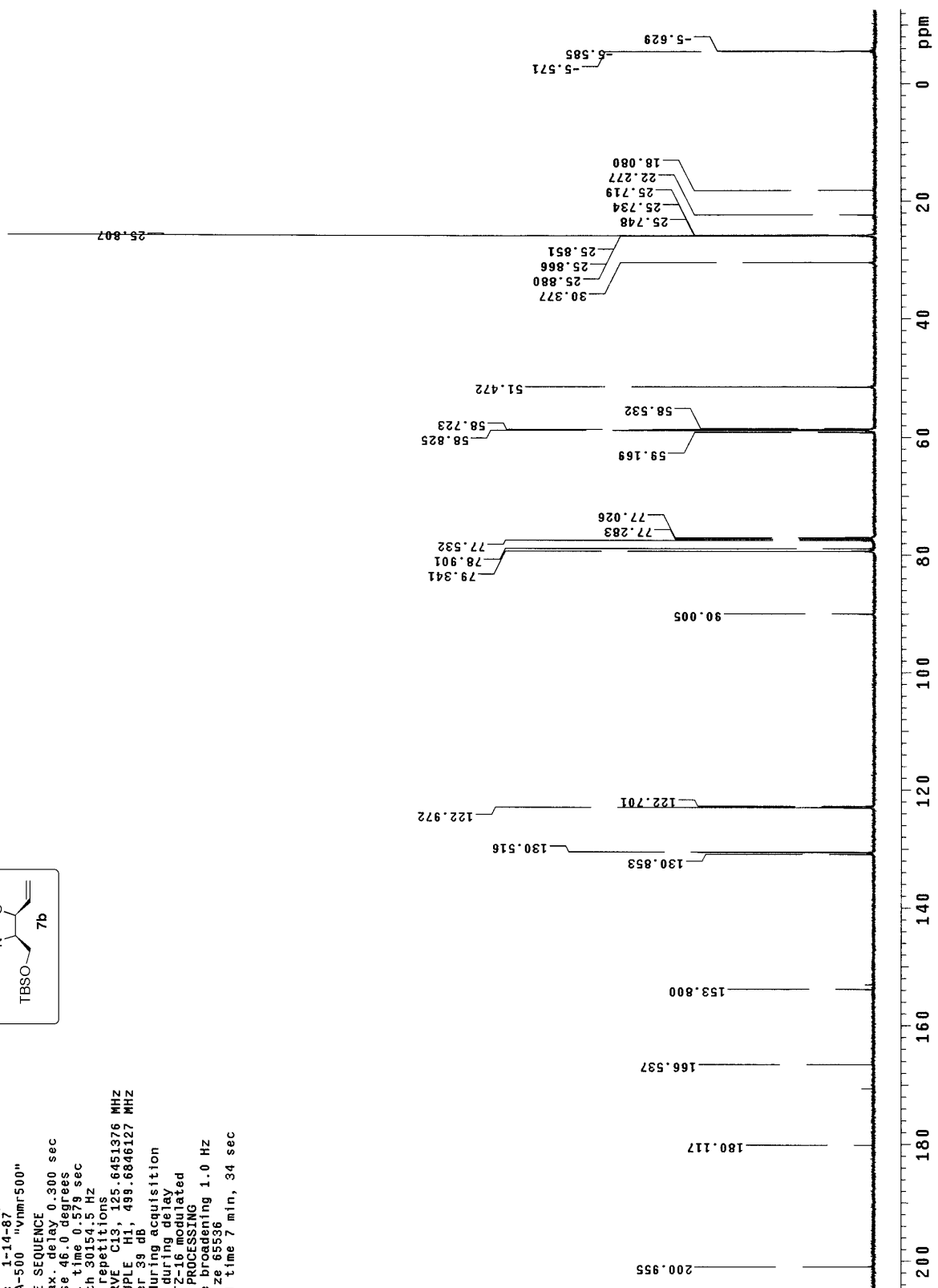
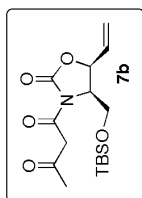
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

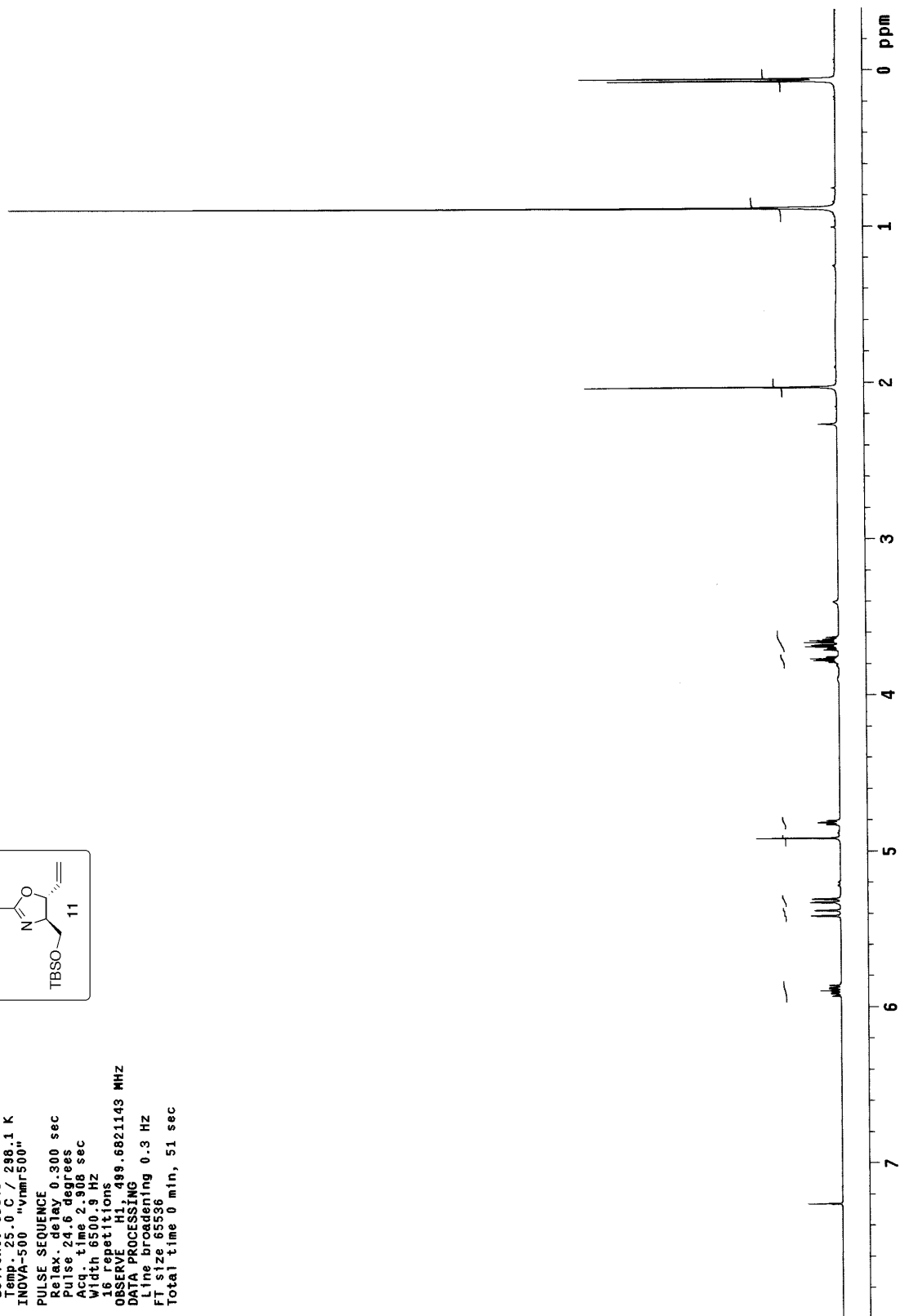
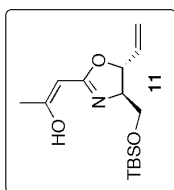
FT size 65536

Total time 7 min, 34 sec



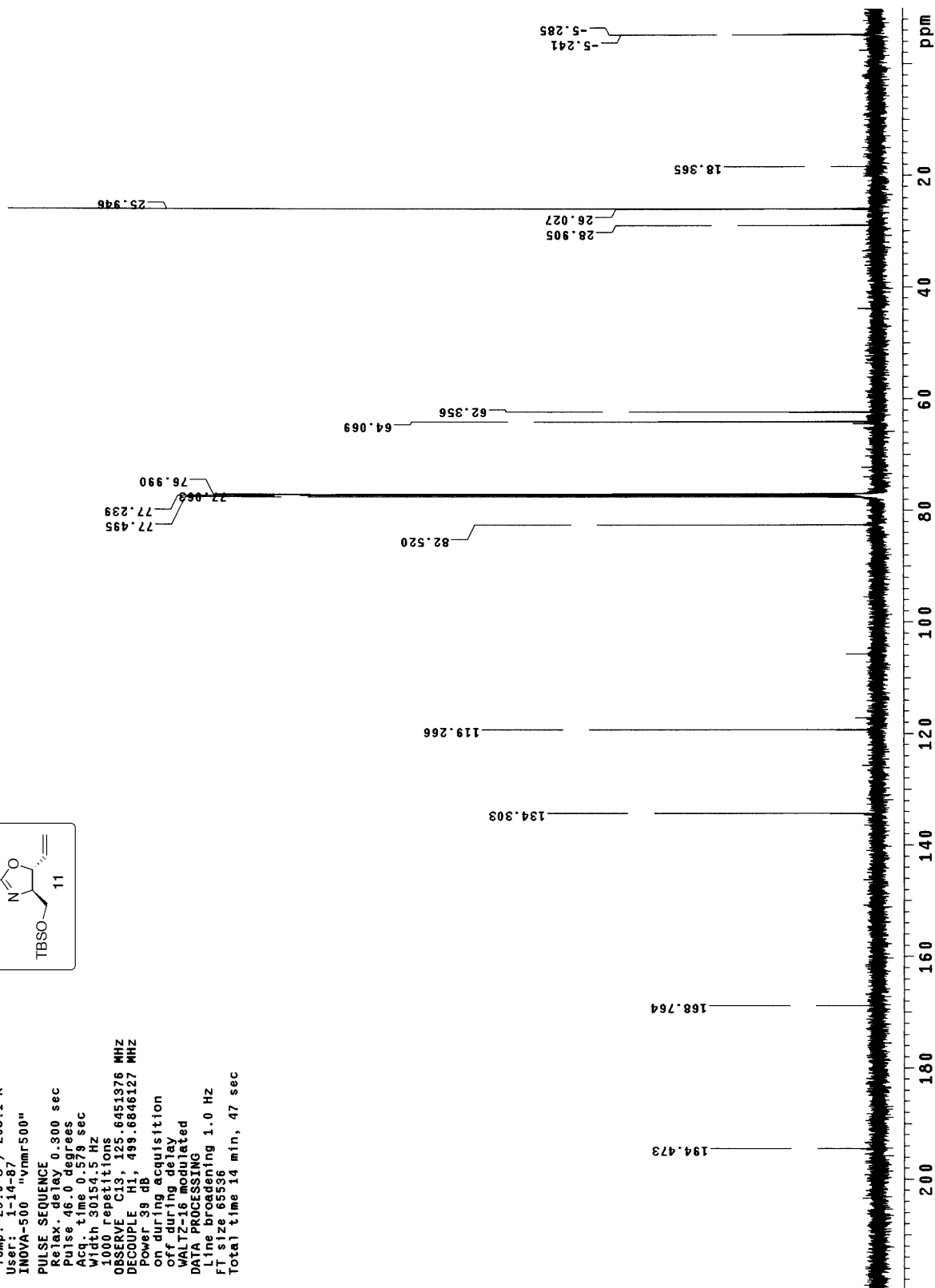
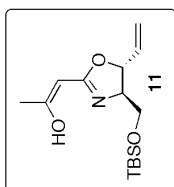
sun-I-243

Pulse Sequence: s2pul
Solvent: CDCl3
Temp: 25.0 C / 298.1 K
INOVA-500 "vnmr500"
PULSE SEQUENCE
Relax delay 0.300 sec
Pulse 24.6 degrees
Acq time 2.988 sec
Width 6500.9 Hz
16 repetitions
OBSERVE H1 499.6821143 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 51 sec



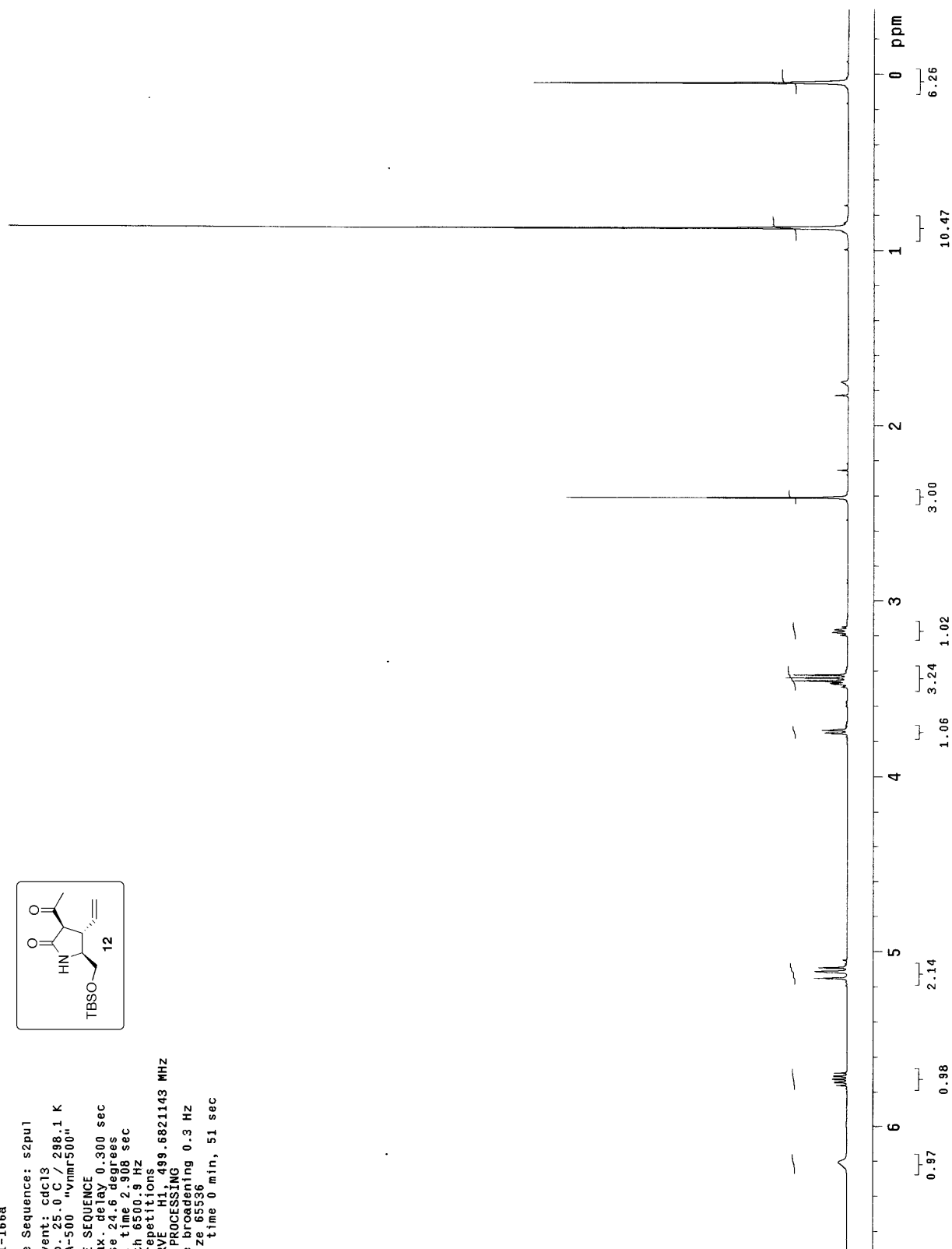
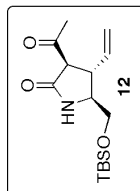
sun-I-243

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 25.0 C / 298.1 K
User: j-l-87
INOVA-500 "vnmf500"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 46.0 degrees
Acq. time 0.579 sec
Width 30154.5 Hz
1000 Repetitions
OBSERVE C13, 125.6451376 MHz
DECOUPLE H1, 499.6846127 MHz
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 14 min, 47 sec

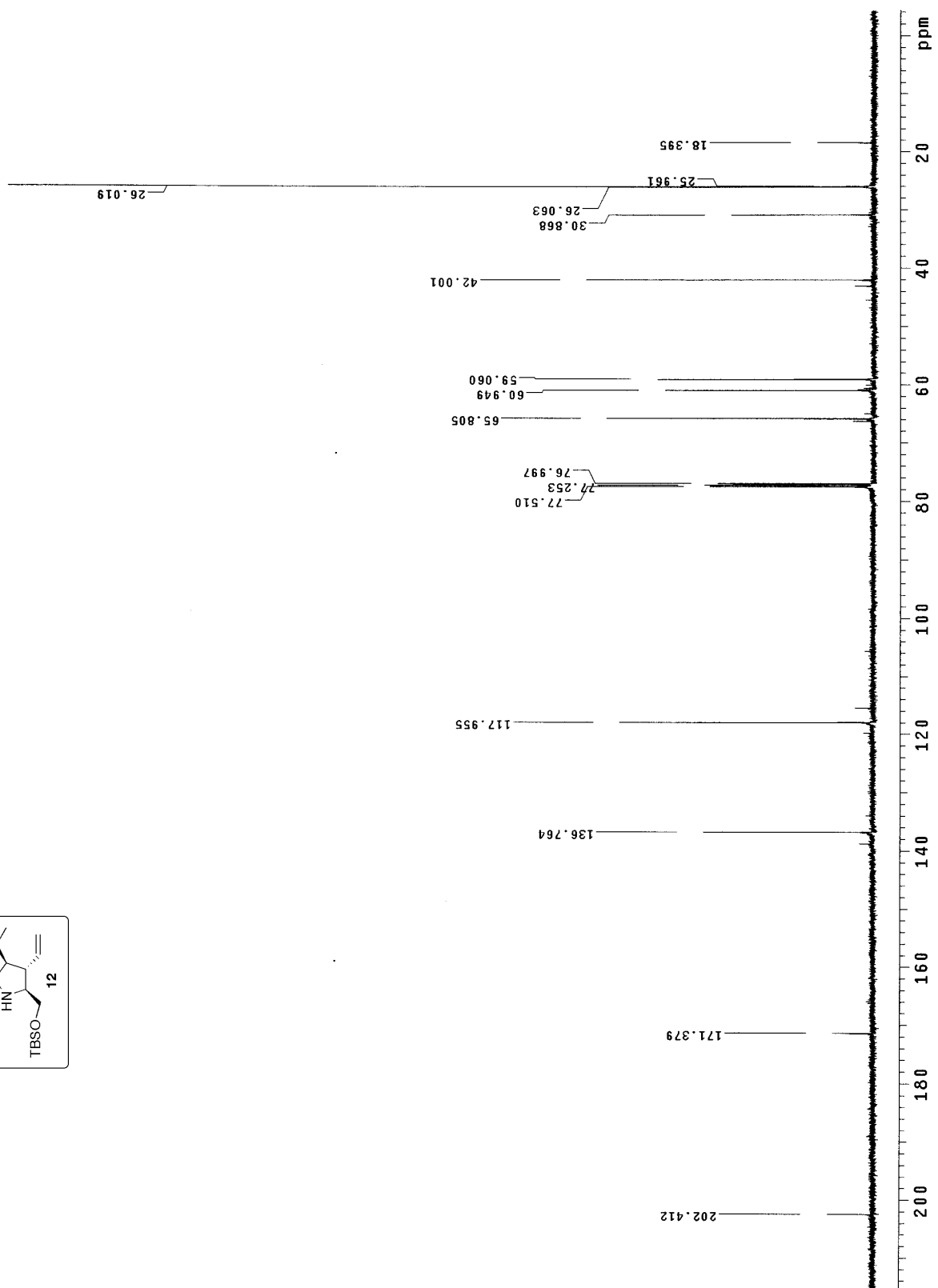
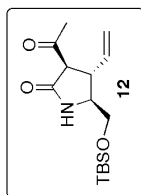


sun-I-186a

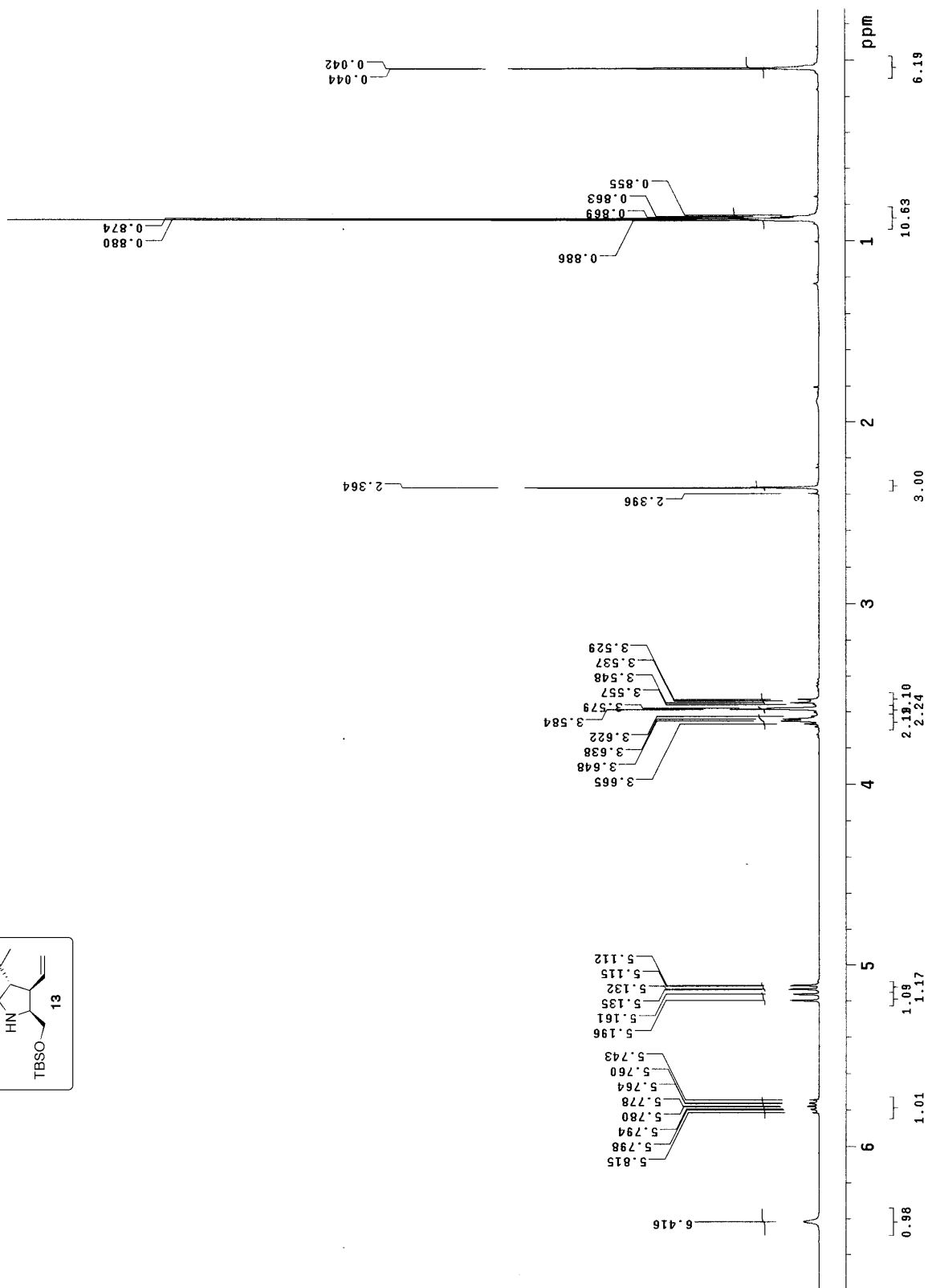
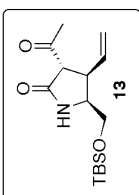
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
INOVA-500 "vnmr500"
PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 24.6 degrees
Acq. time 2.908 sec
Width 6500.9 Hz
16 repetitions
OBSERVE H1, 499.6821143 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 51 sec



sun-I-166a
Pulse Sequence: s2pu1



sun-I-166B
Pulse Sequence: s2pu1



sun-I-166BC13

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 25.0 C / 298.1 K

User: 1-14-87

INNOVA-500 "vnmr500"

PULSE SEQUENCE

Relax. delay 0.300 sec

Pulse 46.0 degrees

Acq. time 0.373 sec

Width 50134.5 Hz

600 repetitions

OBSERVE C13, 125.6451376 MHz

DECOUPLE H1, 499.6846127 MHz

Power 59 dB

on during acquisition

off during delay

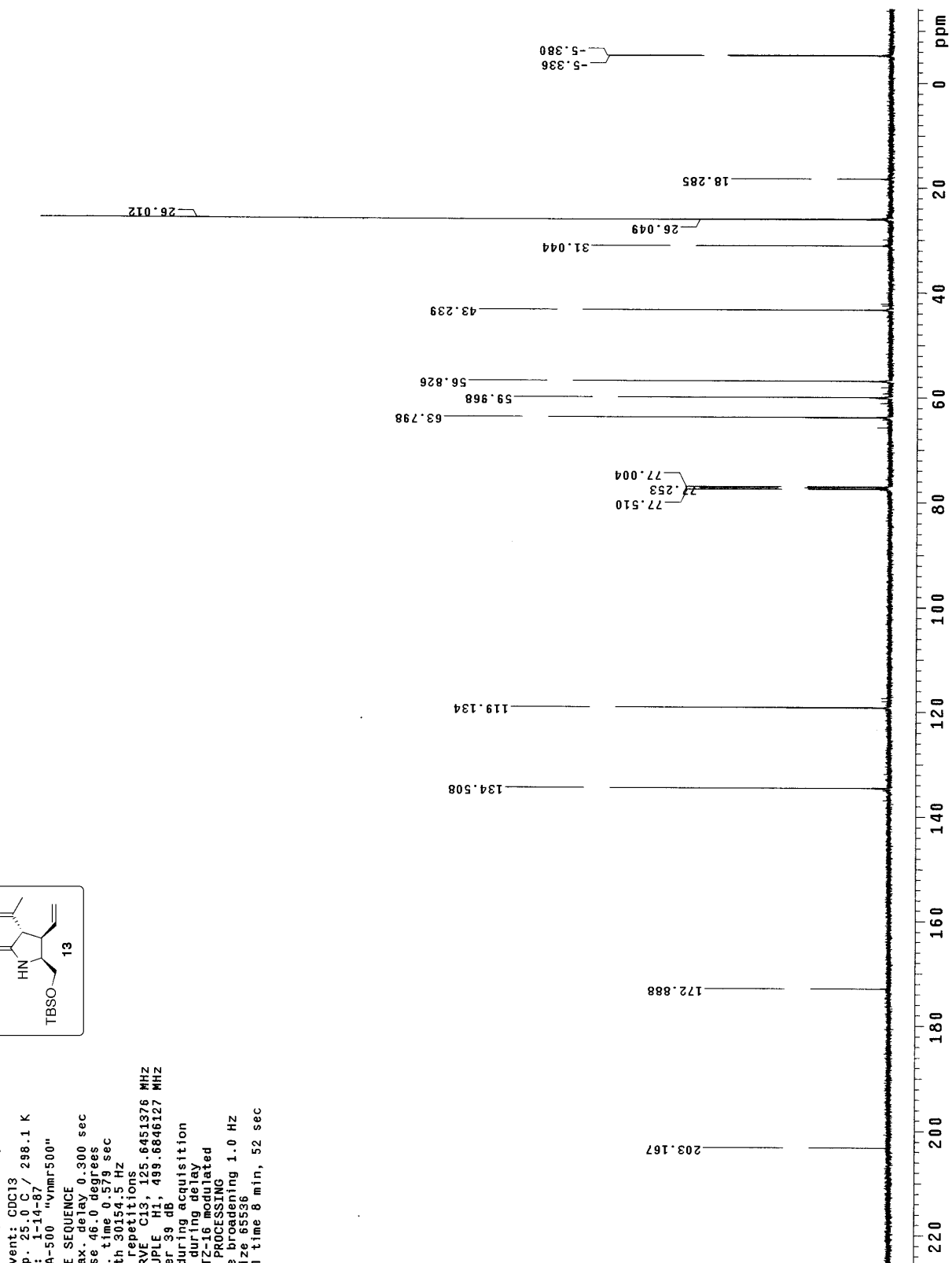
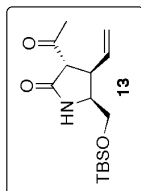
WALTZ16 modulated

DATA PROCESSING

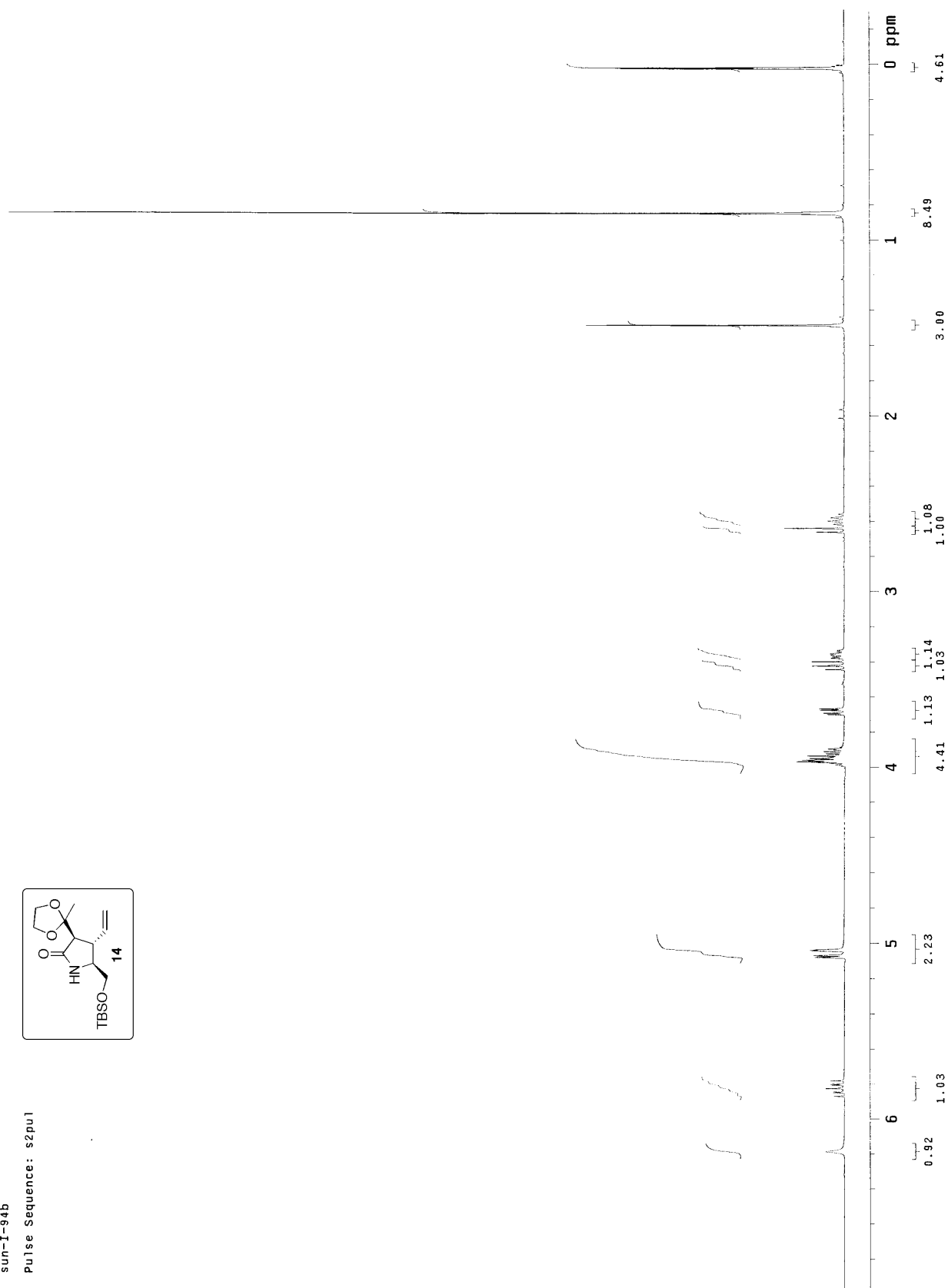
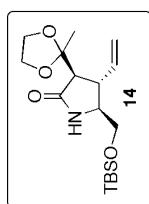
Line broadening 1.0 Hz

FI size 85536

Total time 8 min, 52 sec

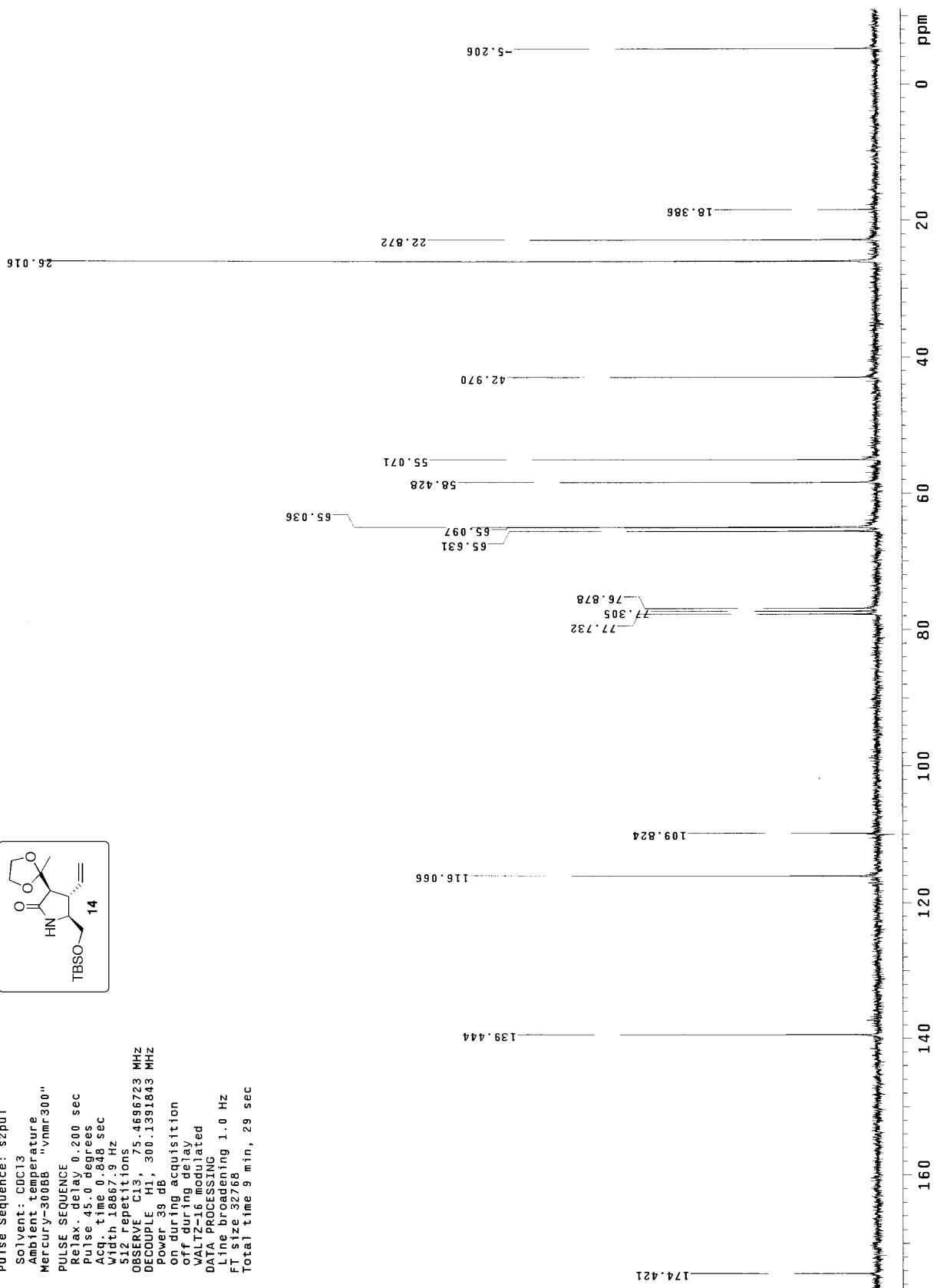
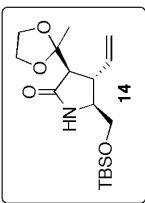


sun-I-94b
Pulse Sequence: s2pu1



sun-I-94b-C13

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-300BB "vnmr300"
PULSE SEQUENCE
Relax. delay 0.200 sec
Pulse 45.0 degrees
Acq. time 0.848 sec
Width 18867.9 Hz
512 repetitions
OBSERVE C13, 75.4686723 MHz
DECOUPLE H1, 300.1391843 MHz
Power 39 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 9 min, 29 sec



sun-I-160

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp: 25.0 C / 288.1 K

INNOVA-500 "vnmr500"

PULSE SEQUENCE

Relax. delay 0.300 sec

Pulse 24.6 degrees

Acq. time 2.908 sec

Width 6500.9 Hz

16 repetitions

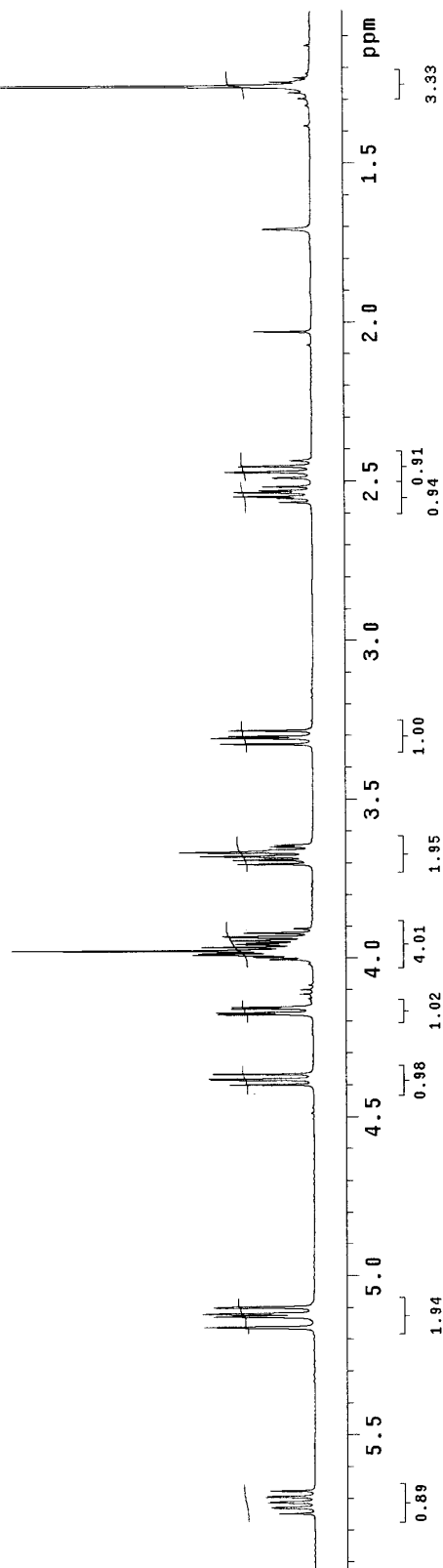
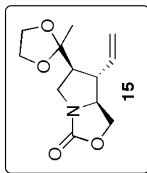
OBSERVE H1, 499.6821143 MHz

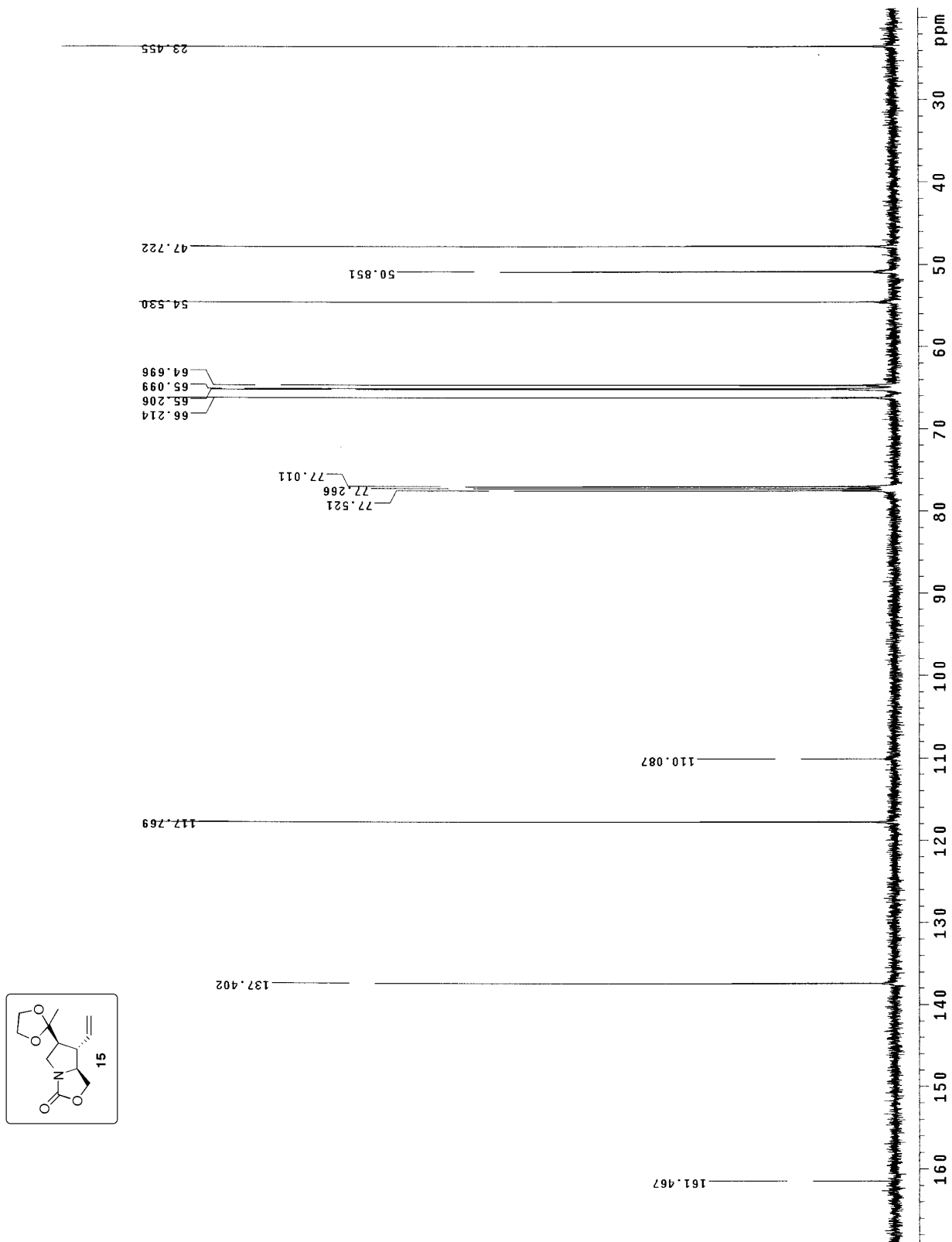
DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 0 min, 51 sec





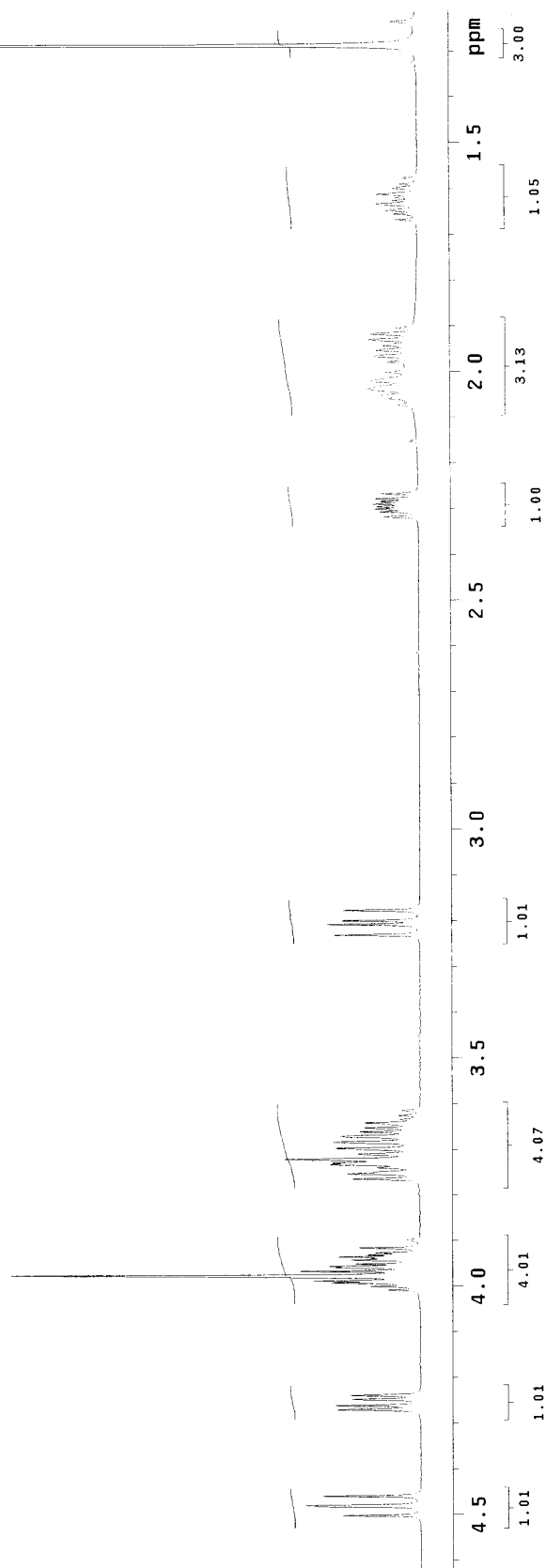
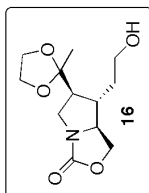
sun-I-161H1

Pulse Sequence: s2pu1

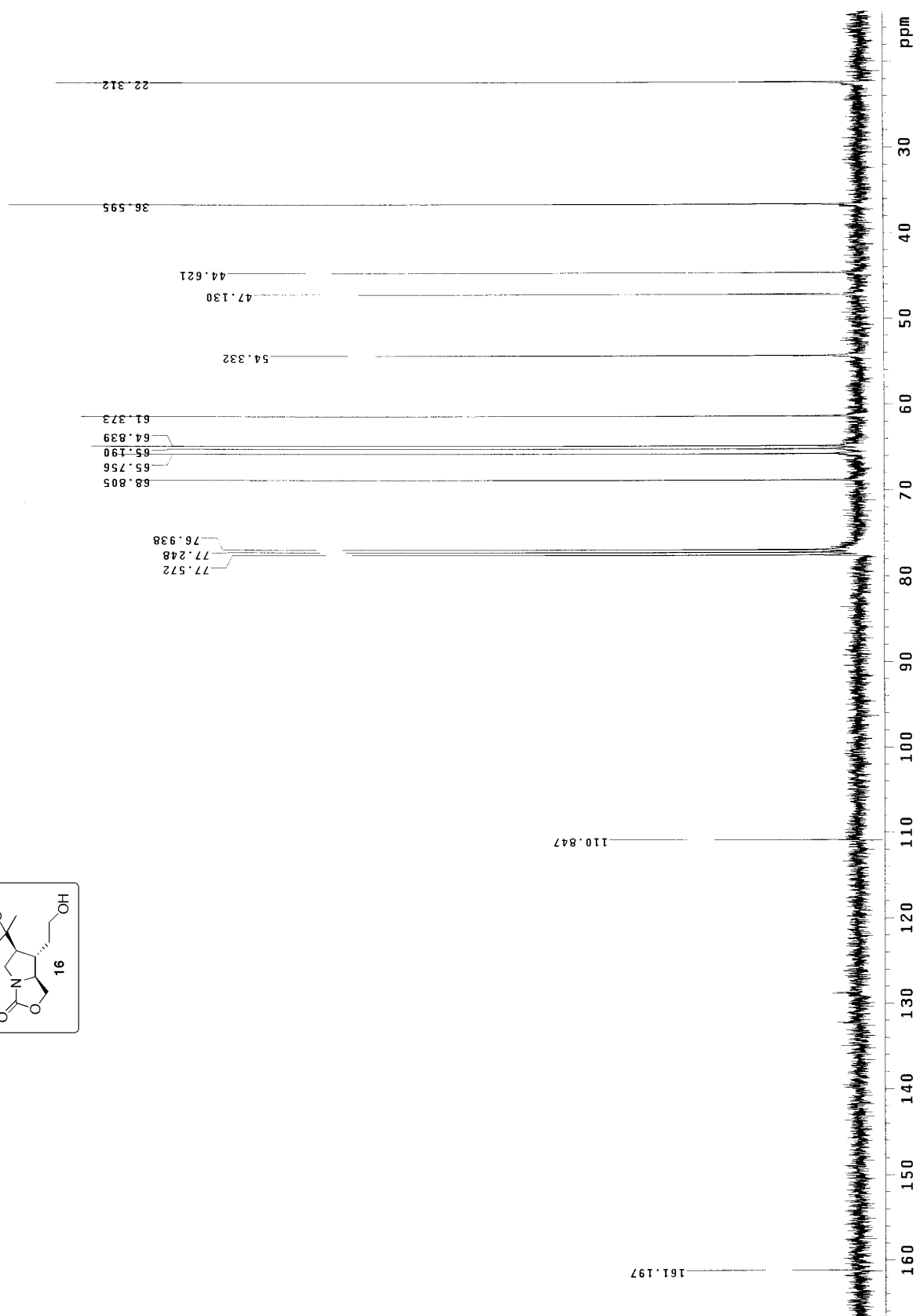
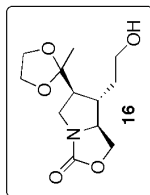
Solvent: cdcl3
Temp: 25.0 C / 288.1 K
INOVA-400 "vnmr400"

PULSE SEQUENCE
Relax. delay 0.300 sec
Pulse 23.9 degrees
Acq. time 3.334 sec
Width 4799.9 Hz
16 repetitions

OBSERVE H1, 399.9402100 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 58 sec



sun-I-161C13
Pulse Sequence: s2pu1



sun-I-220

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 25.0 C / 298.1 K

US60: 1:14.87

INOVA-500 "vnmr500"

PULSE SEQUENCE

Relax. delay 0.300 sec

Pulse 46.0 degrees

Acq. time 0.579 sec

Width 27643.4 Hz

800 repetitions

OBSERVE C13, 125.6451376 MHz

DECOUPLE H1, 499.6846127 MHz

Power 39 dB

on during acquisition

off during delay

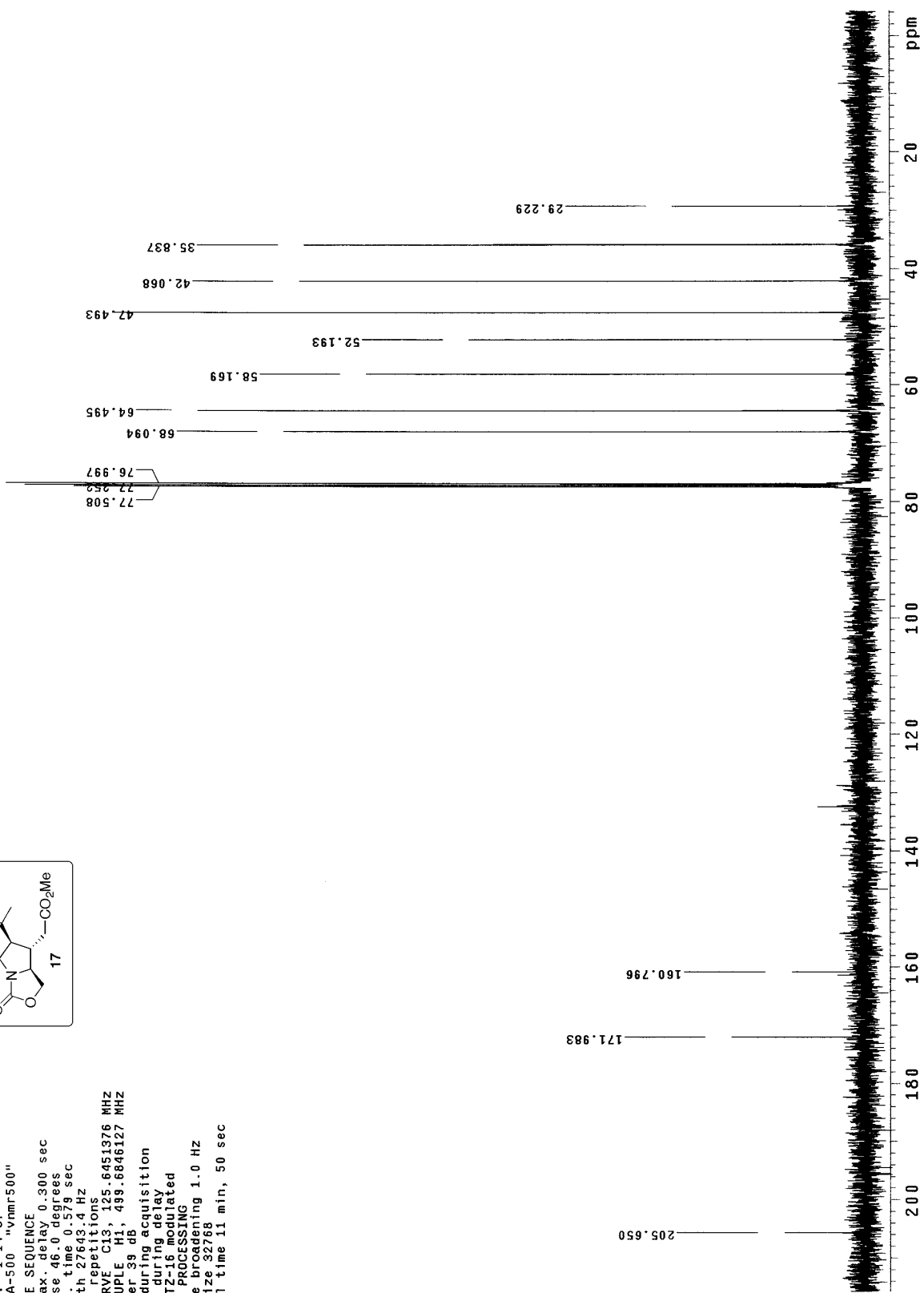
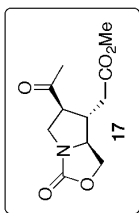
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 11 min, 50 sec



sun-I-220

Pulse Sequence: s2pu1

Solvent: CDC13

Temp: 25.0 C / 298.1 K

INOVA-500 "vnmr500"

PULSE SEQUENCE

Relax: delay 0.300 sec

Pulse: 24.6 degrees

Acq. time 2.908 sec

Width 6500.9 Hz

16 repetitions

OBSERVE H1, 499.6821143 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 0 min, 51 sec

