A Stereocomplementary Approach to β-Lactones: Highly Diastereoselective Synthesis of *cis*-β-Lactones, a β-Chloroacid, and a Tetrahydrofuran

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Supporting Information

Experimental procedures and characterization data for all new compounds; ^{1}H and ^{13}C NMR spectra for compounds **3a**, **d**, **e**, **h**, **5b**, **c**, **7b** (22 pages).

General

All reactions were carried out under N_2 in oven-dried glassware. CH_2Cl_2 was distilled from CaH_2 immediately prior to use. All aldehydes were distilled (Kugelrohr Distillation) or purified by flash chromatography immediately prior to use. Preparation of ketene acetals **2a-c** has been described previously[1,2] After purification by distillation or flash chromatography, the ketene acetals **2a-c** were used as mixtures of Z/E isomers: **2b** (Z/E = 1:7), **2c** (Z/E = 1:19).

General Procedure for β-Lactone Synthesis as Described for β-Lactone 3a. Hydrocinnamaldehyde (731 μL, 90% purity, 5.0 mmol, 1.0 equiv) and ketene acetal **2b** (2.11 g, 80% purity, 6.0 mmol, 1.2 equiv) were dissolved in 25 mL CH_2Cl_2 and cooled to –78 °C. With stirring, 6 mL of a 1.0 M $SnCl_4$ solution (6.0 mmol, 1.2 equiv) in CH_2Cl_2 was added slowly (~0.10 mL/min via syringe pump) over 1 h. After stirring for another 2 h, the reaction was quenched with pH 7 buffer (~10 mL) at –78 °C. The reaction mixture was warmed to 25 °C with vigorous stirring, and then filtered through a small pad of Celite. The CH_2Cl_2 solution was separated, dried over Na_2SO_4, filtered (if necessary, the volume of $CH_2Cl_2$was adjusted to make the final concentration of theoretical β-lactones ~0.15 M), and then directly treated with $CuBr_2$ (1.3 equiv relative to ketene acetal). The resulting suspension was stirred for 1.5 h, filtered through Celite, and washed with 10% aqueous K_2CO_3 solution and then brine. The CH_2Cl_2 solution was dried over Na_2SO_4, filtered, and concentrated in vacuo to afford the crude product. Purification by flash chromatography (gradient elution; 1:19 → 1:4 ethyl acetate/hexanes) gave 592 mg (62%)

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(1) a) Yang, H. W.; Romo, D. *J. Org. Chem.* **1997**, *62*, 4-5. b) Yang, H. W.; Zhao, C.; Romo, D. *Tetrahedron* **1997**, *53*, 16471-16488.

(2) a) Hattori, K.; Yamamoto, H. *J. Org. Chem.* **1993**, 58, 5301-5303. b) Hirai, K.; Iwano, Y.; Mikoshiba, I.; Koyama, H.; Nishi, T. *Heterocycles* **1994**, *38*, 277-280.

of cis-(+/-)-3-methyl-4-(2-phenylethyl)oxetan-2-one (**3a**) as a colorless oil: R_f 0.33 (1:4 ethyl acetate/hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18-7.34 (m, 5 H), 4.56 (ddd, *J*=4.1, 6.3, 9.6 Hz, 1 H), 3.74 (dq, *J*=6.3, 7.8 Hz, 1 H), 2.83-2.93 (m, 1 H), 2.65-2.75 (m, 1 H), 1.90-2.15 (m, 2 H) 1.27 (d, *J*=7.8 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5, 140.3, 128.5, 128.4, 126.3, 74.6, 47.1, 31.9, 31.5, 8.0; IR (thin film) 3027, 1820 cm^{-1}; FAB HRMS Calcd for $C_{12}H_{14}O_2$ [M+Na]: 213.0892. Found: 213.0896. Anal. Calcd for $C_{12}H_{14}O_2$: C, 75.76; H, 7.42. Found: C, 75.57; H, 7.33.

(+/-)-4-(2-phenylethyl)oxetan-2-one (3b): This β-lactone was prepared according to the general procedure using hydrocinnamaldehyde (117 μL, 90% purity, 0.8 mmol, 1.0 equiv), ketene acetal **2a** (314 mg, 75% purity, 0.88 mmol, 1.1 equiv) and $SnCl_4$ (0.96 mL of 1.0 M solution in CH_2Cl_2, 1.2 equiv). Workup followed by flash chromatography (gradient elution; 1:19 → 1:6 ethyl acetate/hexanes) gave 45 mg (32%) of β-lactone **3b** as a colorless oil. Spectral data matched that previously reported.[1b]

Cis-(+/-)-3-methyl-4-*p*-nitrophenyloxetan-2-one (3c): This β-lactone was prepared according to the general procedure using *p*-nitrobenzaldehyde (121 mg, 0.8 mmol, 1.0 equiv), ketene acetal **2b** (270 mg, 0.96 mmol, 1.2 equiv) and $SnCl_4$ (0.96 mL of 1.0 M solution in CH_2Cl_2, 1.2 equiv). Workup followed by flash chromatography (1:3 ethyl acetate/hexanes) gave 109 mg (66%) of β-lactone **3c** as a crystalline solid. Spectral data matched that previously reported.[1b]

Cis-(+/-)-4-heptyl-3-methyloxetan-2-one (3d): This β-lactone was prepared according to the general procedure using octanal (102 mg, 0.8 mmol, 1.0 equiv), ketene acetal **2b** (270 mg, 0.96 mmol, 1.2 equiv) and $SnCl_4$ (0.96 mL of 1.0 M solution in CH_2Cl_2, 1.2

equiv). Workup followed by flash chromatography (1:12 ethyl acetate/hexanes) gave 95 mg (64%) of β-lactone **3d** as a colorless oil. Spectral data for this compound matched that reported previously.[3] Data not reported previously follows: ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 75.7, 47.2, 31.7, 30.0, 29.2, 29.0, 25.4, 22.6, 14.0, 8.0; IR (thin film) 2924, 1826 cm^{-1}; FAB HRMS Calcd for $C_{11}H_{20}O_2$ [M+H]: 185.1542. Found: 185.1540.

Cis-(+/-)-4-Cyclohexyl-3-methyloxetan-2-one (3e): This β-lactone was prepared according to the general procedure using cyclohexanecarboxaldehyde (99 μL, 0.8 mmol, 1.0 equiv), ketene acetal **2b** (270 mg, 0.96 mmol, 1.2 equiv) and $SnCl_4$ (0.96 mL of 1.0 M solution in CH_2Cl_2, 1.2 equiv). ^{1}H NMR (300 MHz, $CDCl_3$) of the crude reaction showed a cis/trans ratio of 2.3:1. Flash chromatography (gradient elution; 1:39 → 1:9 ethyl acetate/hexanes) gave 12 mg (9%) of cis β-lactone **3e** as a colorless oil: R_f 0.49 (1:4 ethyl acetate/hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.17 (dd, *J*=6.2, 7.7 Hz, 1 H), 3.72 (dd, *J*=6.2, 7.8 Hz, 1 H), 0.9-2.0 (br, 11 H) 1.34 (d, *J*=7.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 79.1, 46.9, 37.8, 29.0, 28.2, 26.1, 25.16, 25.14, 8.5; IR (thin film) 2932, 1826 cm^{-1}; FAB LRMS Calcd for $C_{10}H_{16}O_2$ [M+H]: 169. Found: 169. Satisfactory HRMS could not be obtained.

(+/-)-4-Cyclohexyloxetan-2-one (3f): This β-lactone was prepared according to the general procedure using cyclohexanecarboxaldehyde (99 μL, 0.8 mmol, 1.0 equiv), ketene acetal **2a** (314 mg, 75% purity, 0.88 mmol, 1.1 equiv) and $SnCl_4$ (0.96 mL of 1.0 M solution in CH_2Cl_2, 1.2 equiv). Workup followed by flash chromatography (gradient elution; 1:39 → 1:9 ethyl acetate/hexanes) gave 49 mg (40%) of β-lactone **3f** as a

(3) Danheiser, R. L.; Nowick, J. S. *J. Org. Chem.* **1991**, *56*, 1176-1185.

colorless oil which exhibited physical and spectral data that matched those reported previously.[1b]

β-lactone 3h. This β-lactone was prepared according to the general procedure using β-siloxy aldehyde **1h**[4] (1.01 g, 4.7 mmol, 1.0 equiv), ketene acetal **2a** (1.98 g, 80% purity, 5.6 mmol, 1.2 equiv) and $SnCl_4$ (5.6 mL of 1.0 M solution in CH_2Cl_2, 1.2 equiv). GC analysis of the crude reaction showed a anti/syn ratio of 3.3:1. Workup followed by flash chromatography (gradient elution; 1:99 → 1:19 ethyl acetate/hexanes) gave 993 mg (81%) of β-lactone **3h** as a colorless oil: R_f 0.51 (1:9 ethyl acetate/hexanes); $[\alpha]^{23}_D$ 63.8 (*c* 1.05, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.77 (ddd, *J*=3.8, 6.6, 9.0 Hz, 1 H), 3.96-4.06 (m, 1 H), 3.77 (dq, *J*=6.6, 7.8 Hz, 1 H), 1.65-1.81 (m, 2 H), 1.27 (d, *J*=7.8 Hz, 3 H), 1.21 (d, *J*=6.0 Hz, 3 H), 0.89 (s, 9 H), 0.078 (s, 3 H), 0.074 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 72.7, 64.7, 47.1, 39.8, 25.8, 24.6, 18.0, 8.4, -4.4, -4.9; IR (thin film) 2955, 1829 cm^{-1}; FAB HRMS Calcd for $C_{13}H_{26}O_3Si$ [M+Na]: 281.1549. Found: 281.1549. Anal. Calcd for $C_{13}H_{26}O_3Si$: C, 60.42; H, 10.14. Found: C, 60.95; H, 10.22.

Preparation of β-Chloro Acid 5b and β-Chloro Ester 5c: Hydrocinnamaldehyde (0.73 mL, 90% purity, 5.0 mmol, 1 equiv) and ketene acetal **2c** (1.69 g, 6.0 mmol, 1.2 equiv) were dissolved in 25 mL CH_2Cl_2 and cooled to –78 °C. With stirring, 6 mL of a 1.0 M $SnCl_4$ solution (6.0 mmol, 1.2 equiv) in CH_2Cl_2 was added very slowly (~0.10 mL/min via syringe pump) over 1 h. After stirring for another 2 h at –78 °C, the reaction

(4) Aldehyde **1h** (94% ee by chiral GC analysis; 94% purity (^{1}H NMR), used directly after half-reduction of the corresponding ester with DIBAl-H) was prepared as previously described: Romo, D.; Rzasa, R. M.; Shea, H. A.; Park, K.; Langenhan, J. M.; Sun, L.; Akhiezer, A.; Liu, J. O. *J. Am. Chem. Soc.* **1998**, *120*, 12237-12254.

mixture was allowed to warm to ~0 °C slowly over several hours. The reaction was quenched with pH 7 buffer at 0 °C, stirred vigorously for 20 min, and then filtered through a small pad of Celite. The CH_2Cl_2 solution was separated, dried over Na_2SO_4, filtered (if necessary, the CH_2Cl_2 volume was adjusted to make the final concentration of theoretical chloroester ~0.15 M), and then directly treated with $CuBr_2$ (1.3 equiv relative to ketene acetal). The resulting suspension was stirred for 1.5 h, filtered through Celite, and washed with 10% aqueous K_2CO_3 solution and then brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo to afford 1.84 g of crude β-chloro silyl ester **5a**. The silyl ester **5a** partially desilylates on silica gel and thus was directly converted to the corresponding carboxylic acid **5b** or ester **5c** as described below.

The silyl ester **5a** (1/5 of the crude reaction mixture from above, 1 mmol) was dissolved in 12 mL MeOH and 4 mL THF, then treated with 4 mL 10% aqueous K_2CO_3 and stirred at 25 °C for 1 h. The reaction mixture was extracted with Et_2O (4 X 20 mL) to remove organic soluble compounds, and the combined organic extracts were washed twice with 10% aqueous K_2CO_3 (40 mL). The aqueous solutions were combined and acidified to pH~2 with 2N HCl, and extracted with EtOAc (4 X 20 mL). The combined EtOAc extractions were dried over Na_2SO_4, filtered, and concentrated in vacuo to give 194 mg (86%, based on 1.0 mmol starting aldehyde) of β-chloro acid **5b** as a crystalline solid. ^{1}H NMR indicated a purity of >95% for this material. This acid could be recrystallized from chloroform/pentane by the layering method and a crystal for x-ray analysis was prepared in this way. Physical and spectral data for acid **5b** follows: R_f 0.07 (1:4 ethyl acetate/hexanes); m.p. 103.5-104.5 °C (chloroform/pentane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18-7.33 (m, 5 H), 4.12 (ddd, *J*=3.0, 6.9,10.0 Hz, 1 H), 2.86-2.99 (m, 2

H), 2.69-2.79 (m, 1 H), 1.94-2.18 (m, 2 H), 1.28 (d, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.9, 140.7, 128.52, 128.50, 126.2, 62.3, 46.8, 36.2, 32.5, 13.6; IR (thin film) 2500-3300 (br), 3030, 1709 cm^{-1}; FAB HRMS Calcd for $C_{12}H_{15}ClO_2$ [M+Na]: 249.0658. Found: 249.0650. Anal. Calcd for $C_{12}H_{15}ClO_2$: C, 63.58; H, 6.67; Cl, 15.64. Found: C, 63.72; H, 6.70; Cl, 15.75.

The silyl ester **5a** (1/5 of the crude reaction mixture from above, 1 mmol) was dissolved in 3 mL MeOH, 0.05 mL concentrated H_2SO_4 was added, and the mixture was heated to reflux for 3 h. The acid was neutralized by addition of 80 mg solid $NaHCO_3$. The MeOH solution was dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash chromatography (1:9 ethyl acetate/hexanes) gave 202 mg (84%, based on 1.0 mmol starting aldehyde) of methyl ester **5c** as a colorless oil: R_f 0.62 (1:4 ethyl acetate/hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17-7.32 (m, 5 H), 4.11 (ddd, *J*=3.2, 6.8, 9.9 Hz, 1 H), 3.68 (s, 3 H), 2.65-2.98 (m, 3 H), 1.88-2.14 (m, 2 H), 1.23 (d, *J*=7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.6, 140.7, 128.5, 128.4, 126.1, 62.7, 51.8, 46.9, 36.2, 32.5, 13.5; IR (thin film) 3024, 1742 cm^{-1}; FAB HRMS Calcd for $C_{13}H_{17}ClO_2$ [M+Na]: 263.0815. Found: 263.0823.

Preparation of 2-(tetrahydro-furan-2-yl)-propionic acid (7b): Freshly prepared 4-*tert*-butyldimethylsiloxybutanal[5] (162 mg, 0.8 mmol, 1.0 equiv) and ketene acetal **2c** (270 mg, 0.96 mmol, 1.2 equiv) were dissolved in 4.4 mL CH_2Cl_2 and cooled to –78 °C. With stirring, 0.96 mL of 1.0 M $SnCl_4$ solution (0.96 mmol, 1.2 equiv) in CH_2Cl_2 was added very slowly (~0.015 mL/min via syringe pump) over 1 h. After stirring for another 2 h at –78 °C, the reaction mixture was allowed to warm to -45 °C slowly over several hours.

(5) Ikeda, Y.; Ukai, J.; Ikeda, N.; Yamamoto, H. *Tetrahedron Lett.* **1984**, 25, 5177-5180

The reaction was quenched with pH 7 buffer at -45 °C, stirred vigorously, warmed to 25 °C, and then filtered through a small pad of Celite. The CH_2Cl_2 solution was separated, dried over Na_2SO_4, filtered (if necessary, the CH_2Cl_2 volume was adjusted to make the final concentration of theoretical acid ~0.15 M), and then directly treated with $CuBr_2$ (1.3 equiv relative to ketene acetal). The resulting suspension was stirred for 1.5 h, filtered through Celite, and washed with 10% aqueous K_2CO_3 solution and then brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo to afford the crude 2-(tetrahydro-furan-2-yl)-propionic acid TBS ester **7a**. The TBS ester **7a** partially desilylates on silica gel and thus was directly converted to the corresponding carboxylic acid **7b** as described below.

The TBS ester **7a** was dissolved in 12 mL MeOH and 4 mL THF, then treated with 4 mL 10% aqueous K_2CO_3 and stirred at 25 °C for 1 h. The reaction mixture was extracted with Et_2O (4 X 20 mL) to remove organic soluble compounds, and the combined organic extracts were washed twice with 10% aqueous K_2CO_3 (40 mL). The aqueous solutions were combined and acidified to pH 2 with 2N HCl, and extracted with EtOAc (4 X 20 mL). The combined EtOAc extractions were dried over Na_2SO_4, filtered, and concentrated in vacuo to give 61 mg (53%) of acid **7b** as a crystalline solid. Spectral data for this compound matched that reported previously.[6] Higher resolution data is provided: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.01 (ddd, *J*=6.4, 8.3, 8.3 Hz, 1 H), 3.91 (dt, *J*=6.9, 8.4 Hz, 1 H), 3.82 (dt, *J*=6.0, 7.8 Hz, 1 H), 2.53 (dq, *J*=7.2, 8.6 Hz, 1 H), 2.01-2.12 (m, 1 H), 1.87-1.98 (m, 2 H), 1.53-1.65 (m, 1 H), 1.18 (d, *J*=7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.9, 80.3, 68.3, 44.7, 29.6, 25.6, 13.4.

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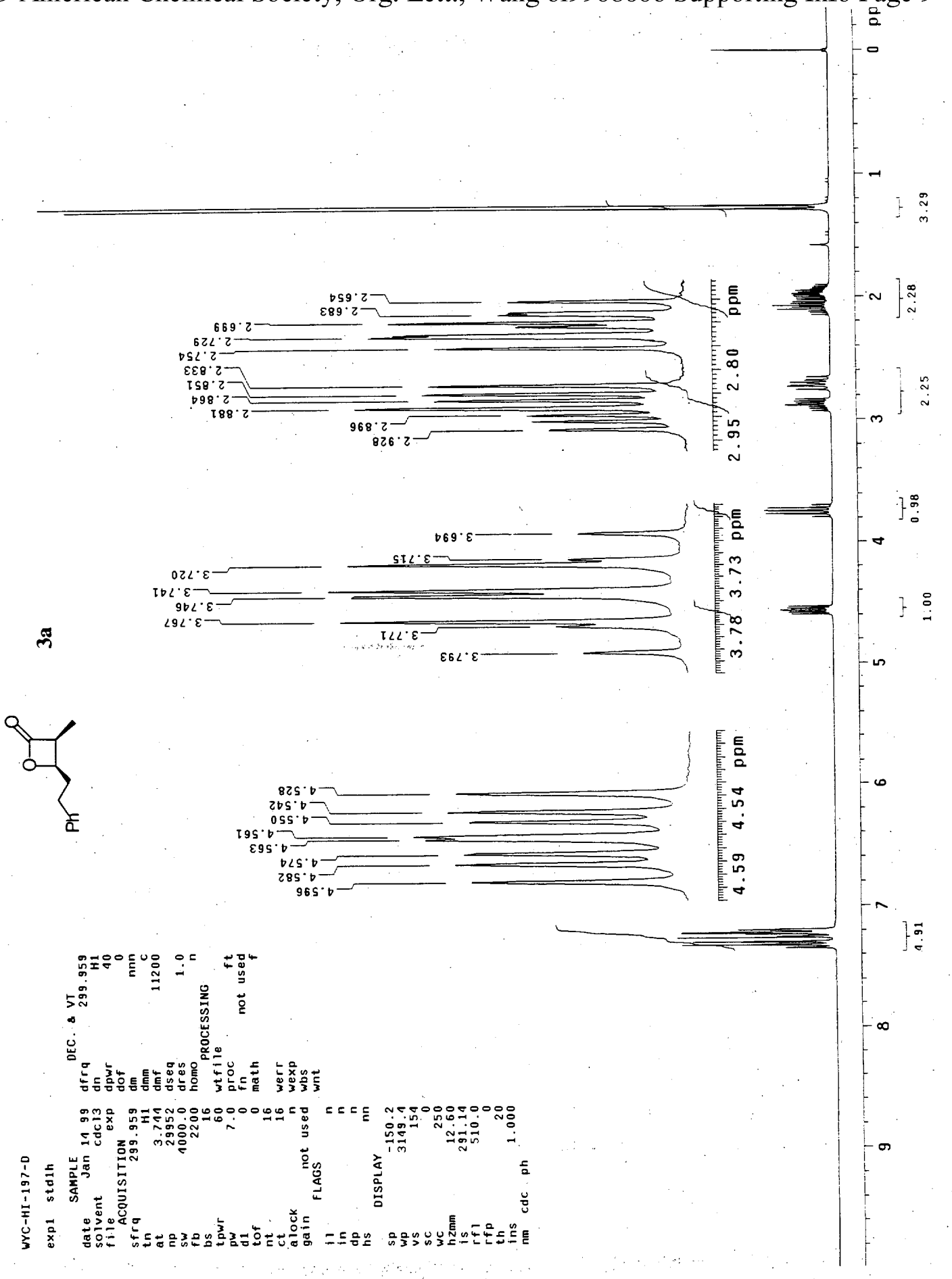
(6) Mead, K. T.; Yang, H. -L. *Tetrahedron Lett.* **1989**, 30, 6829-6832.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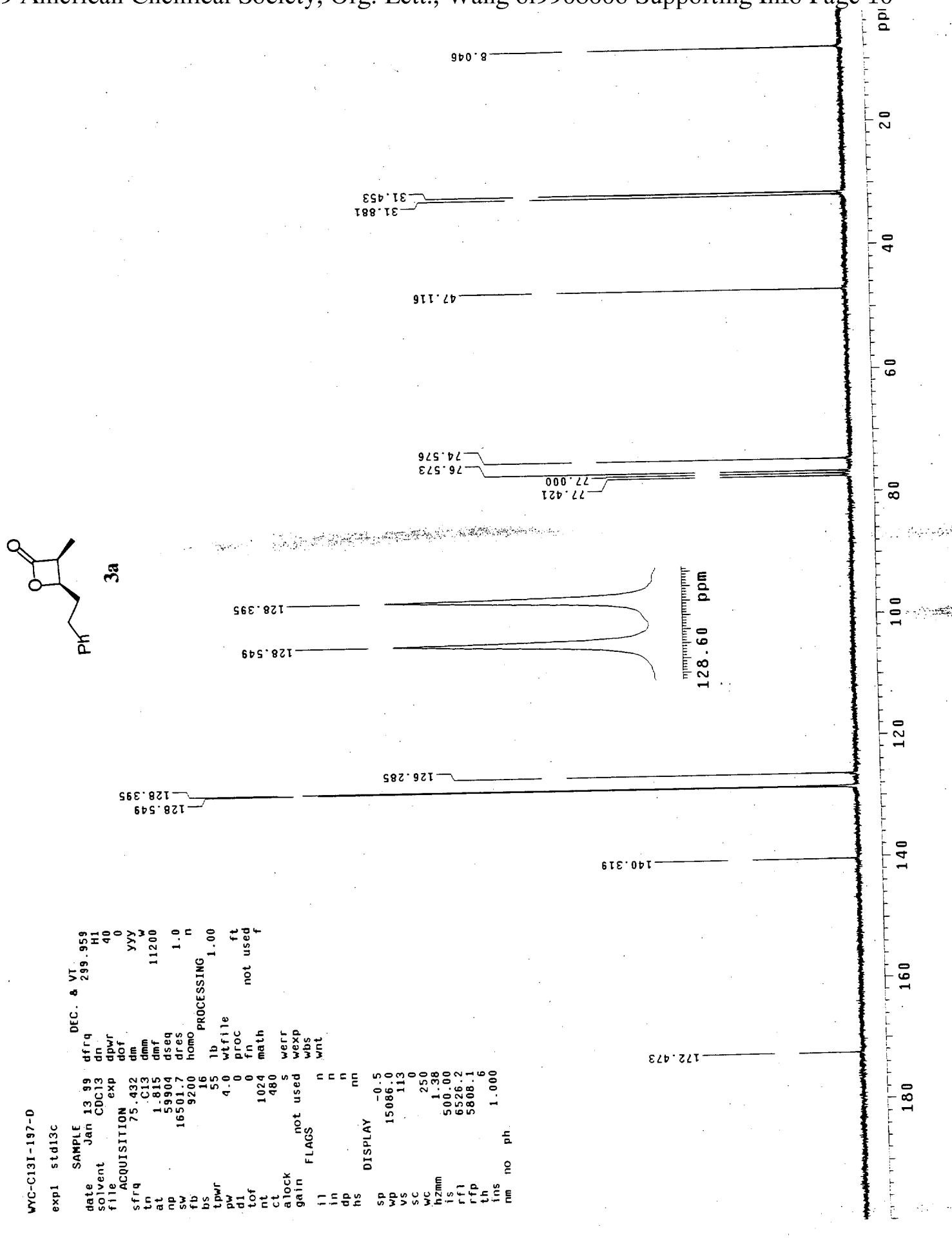

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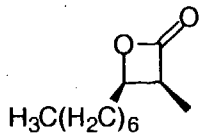

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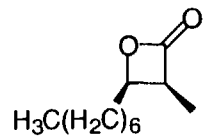

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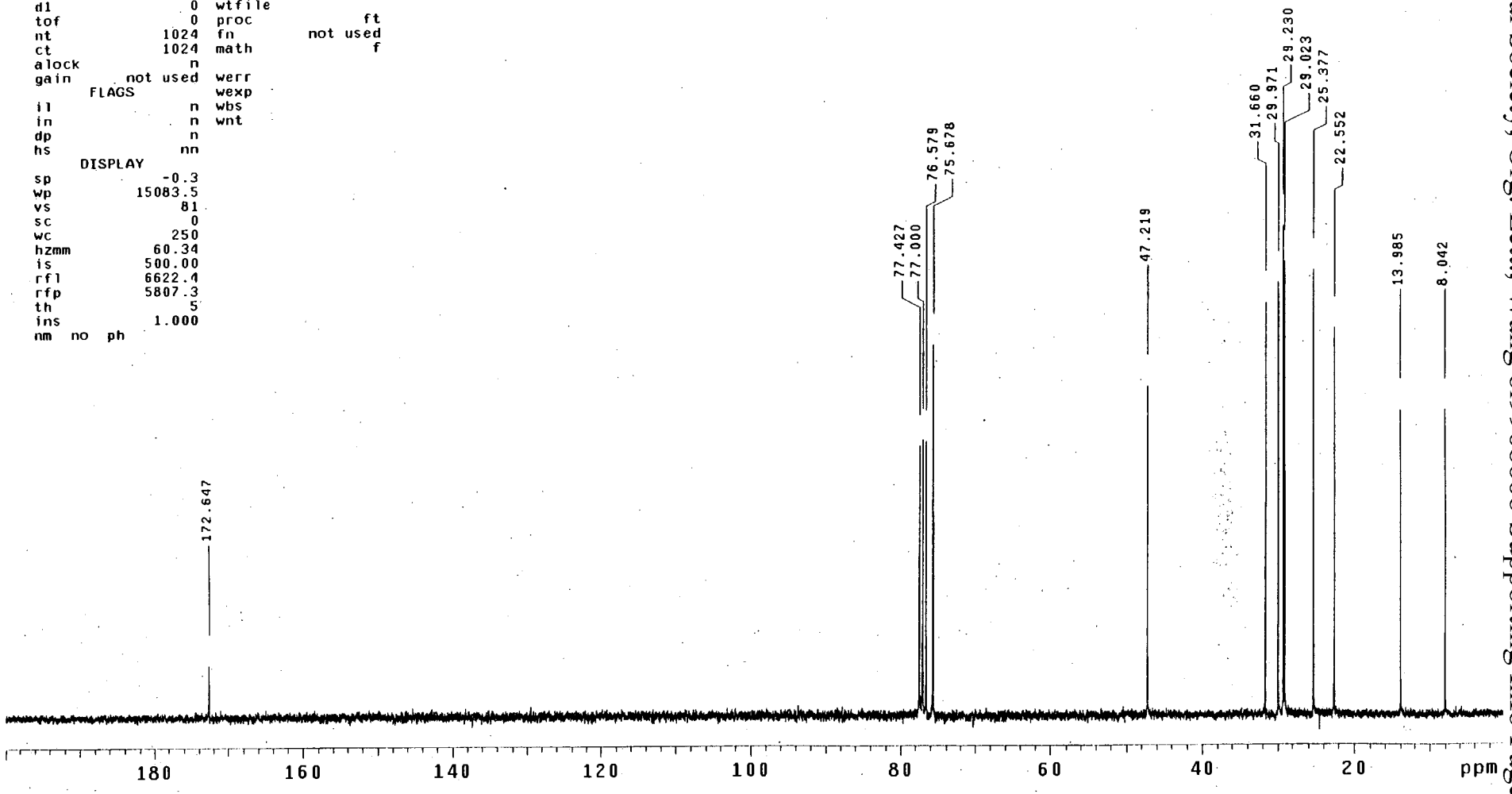

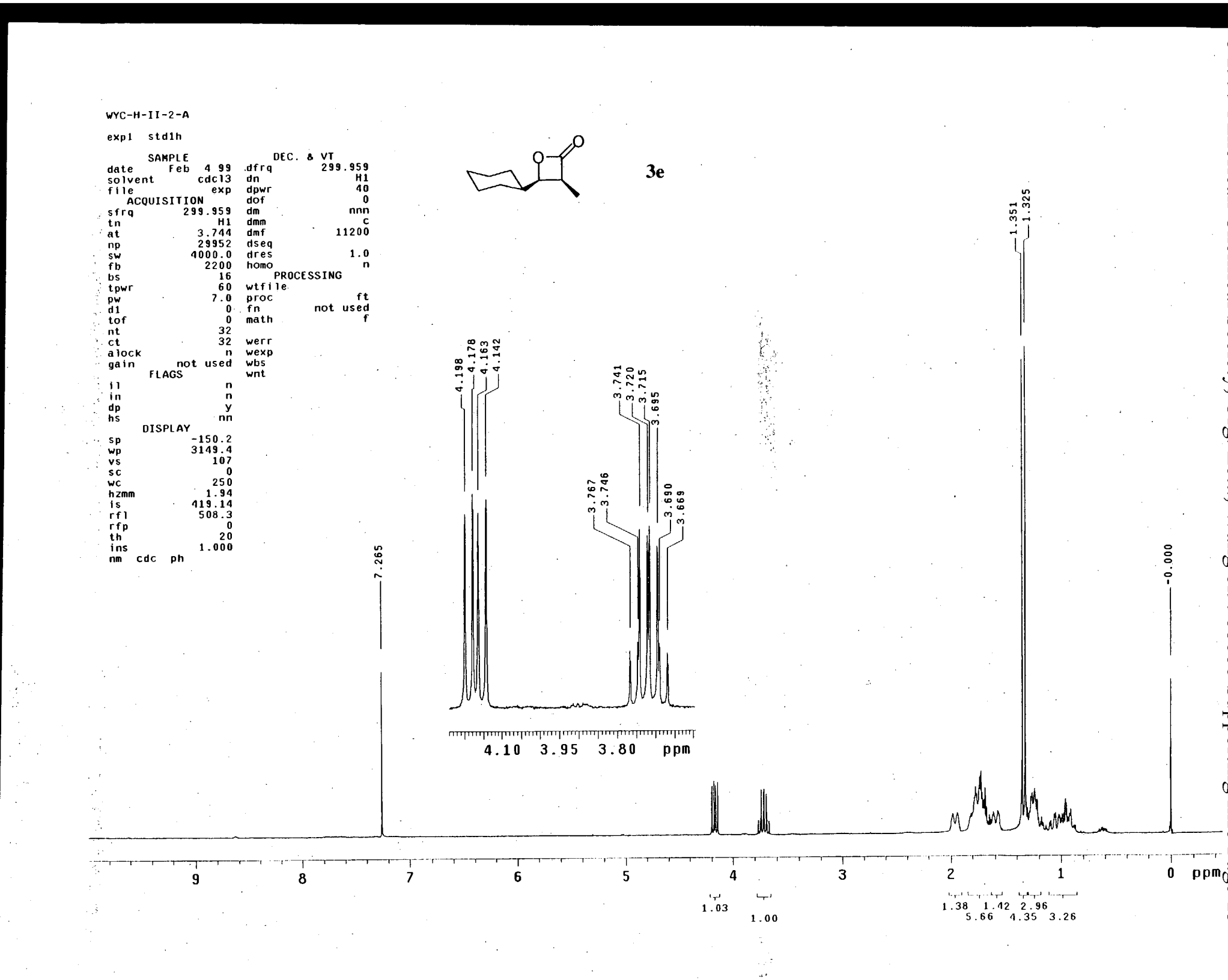

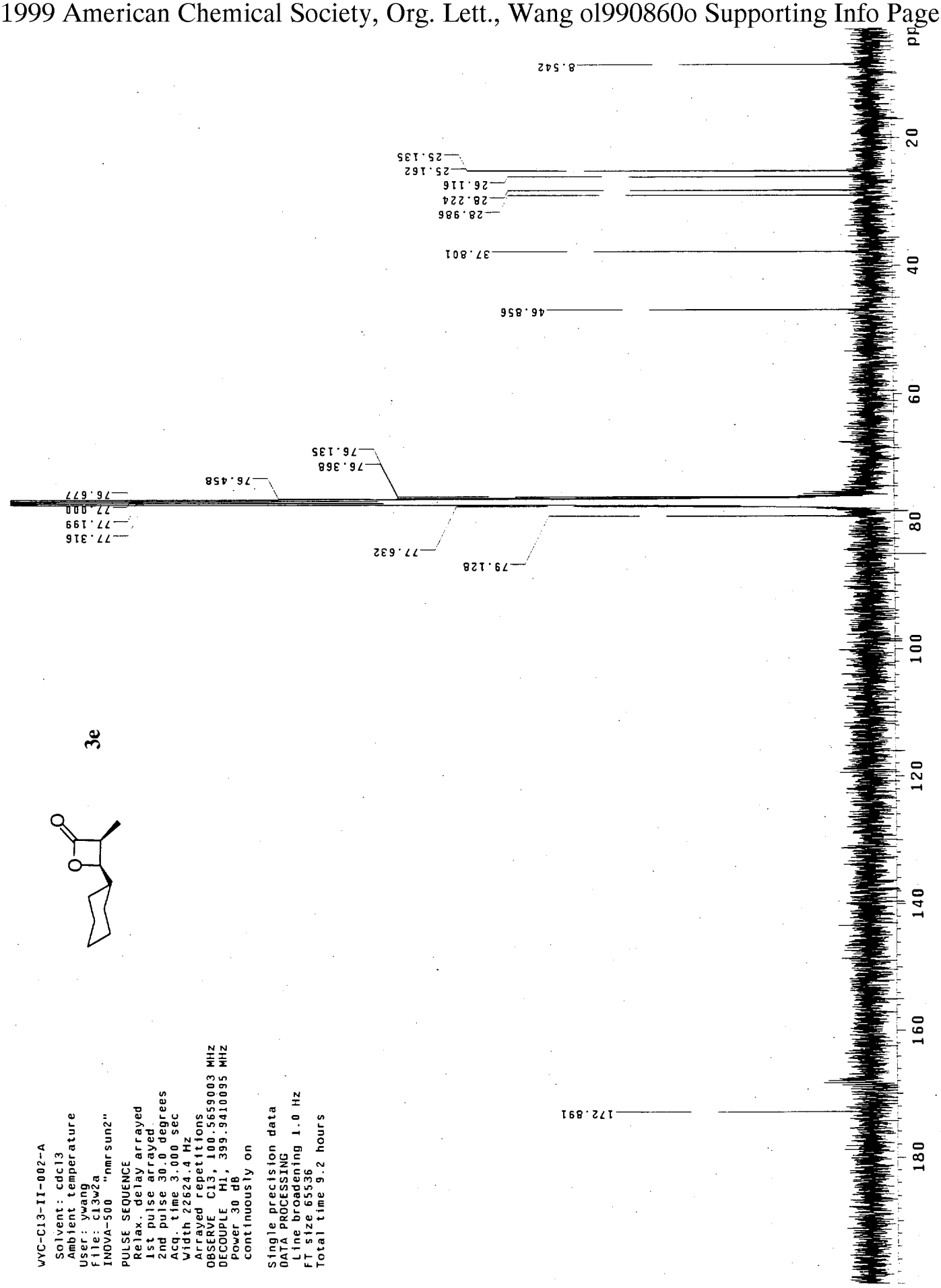
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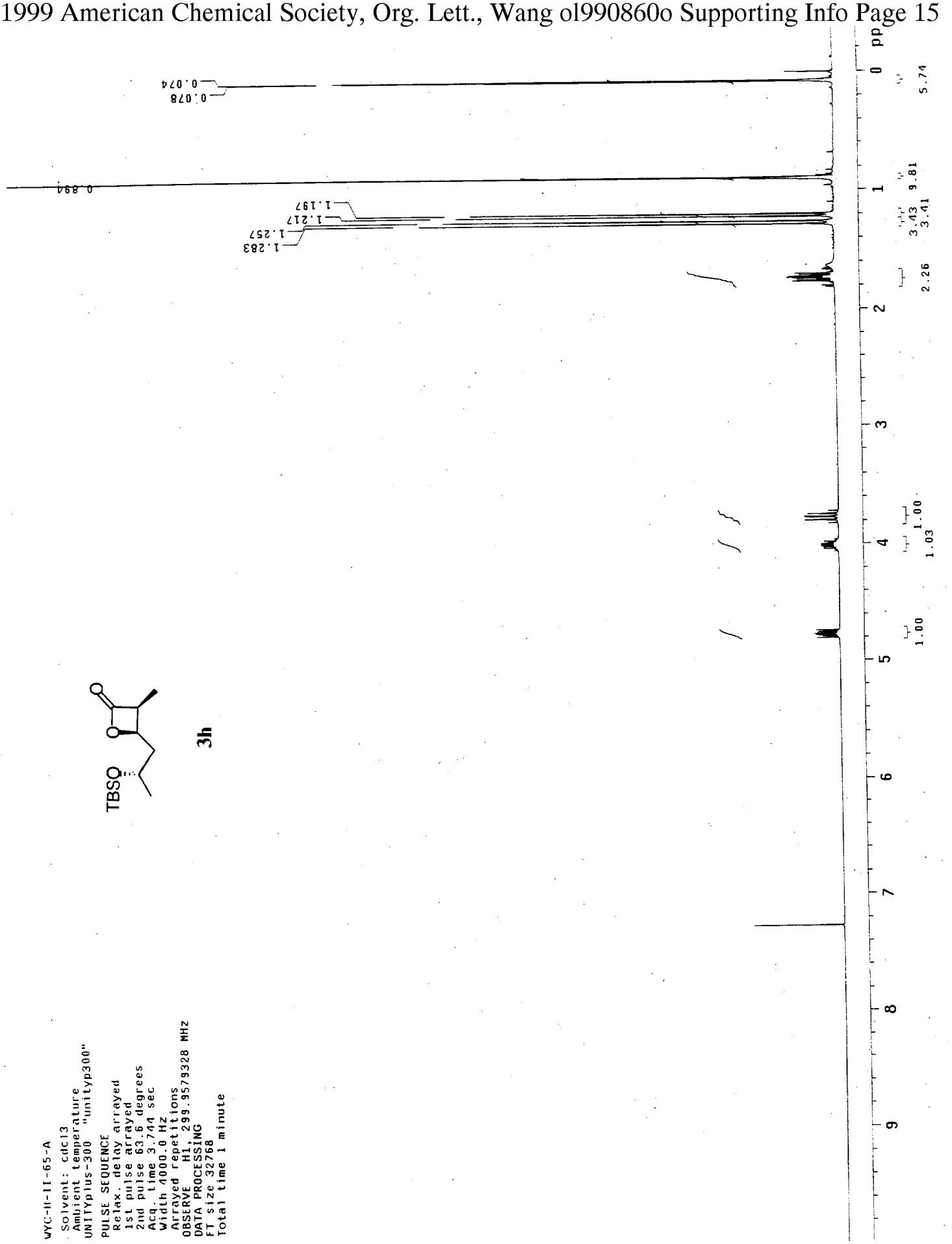

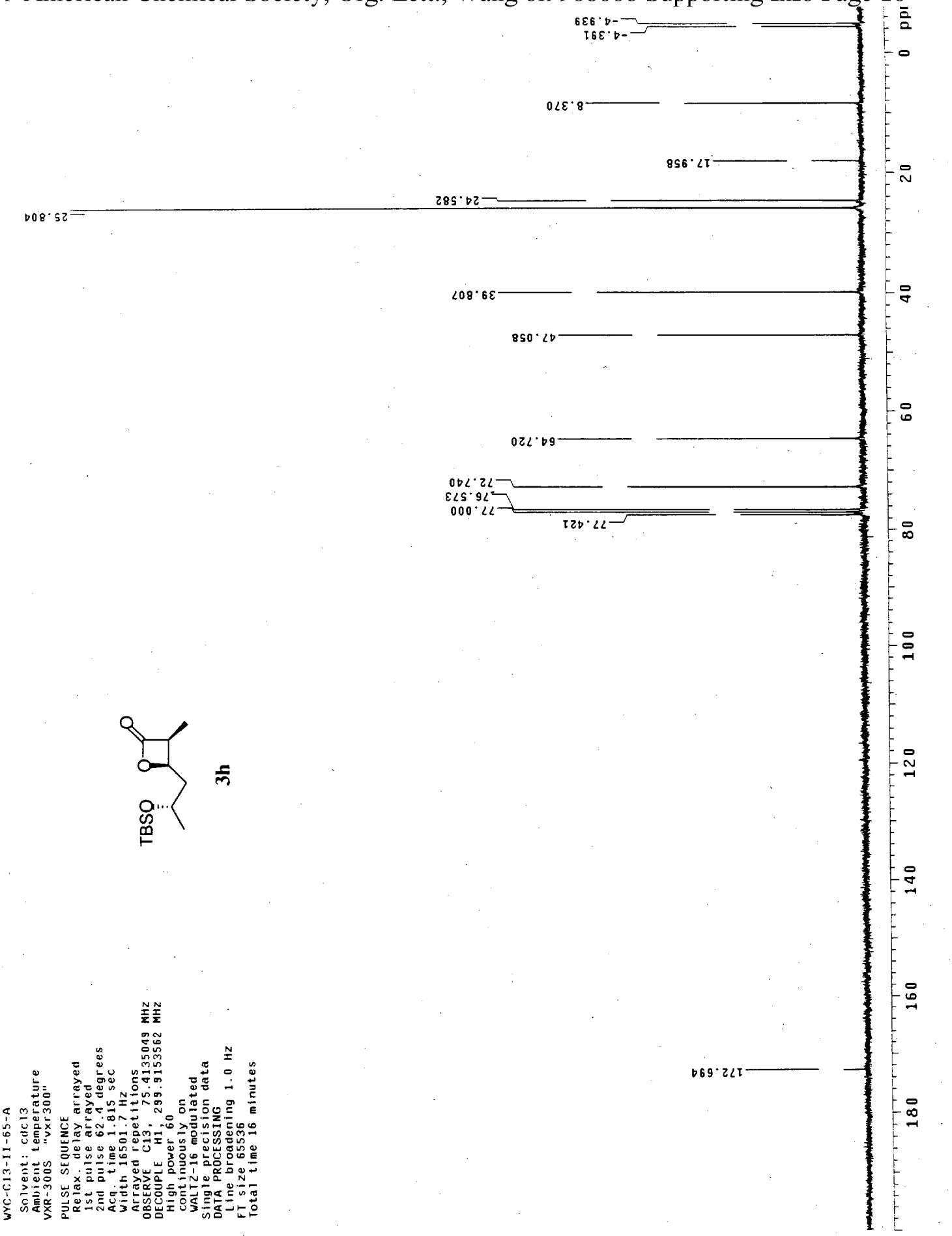

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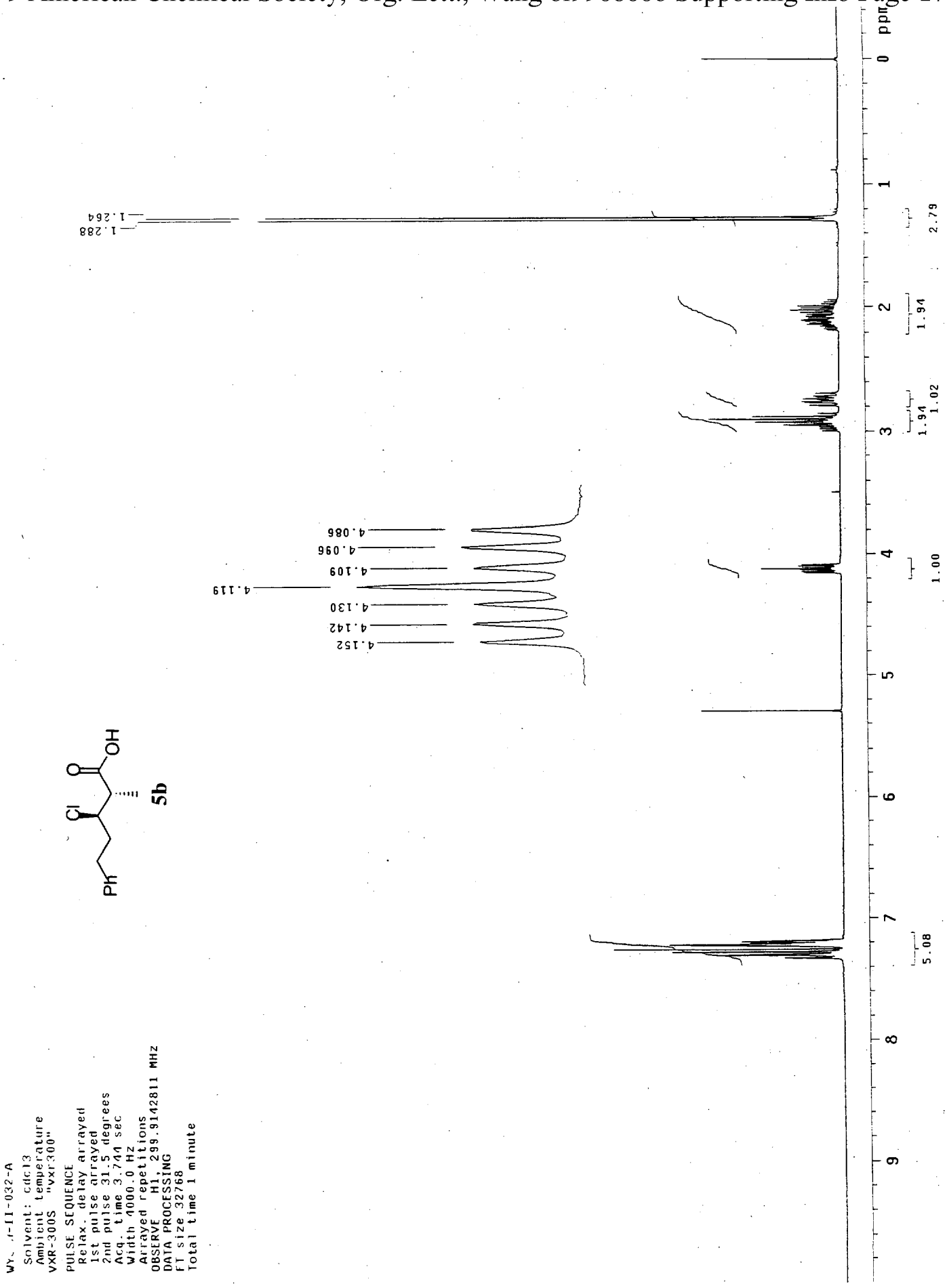
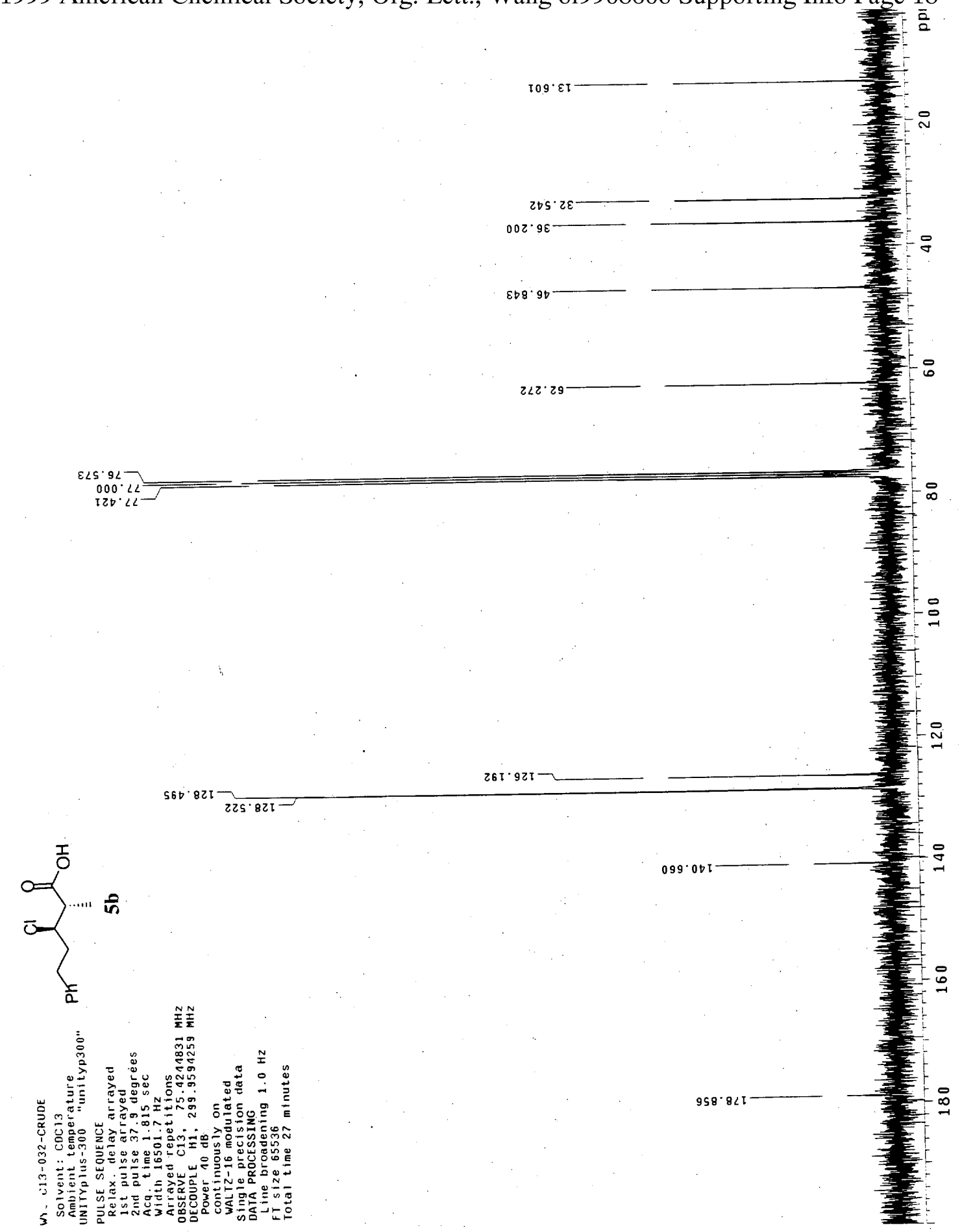
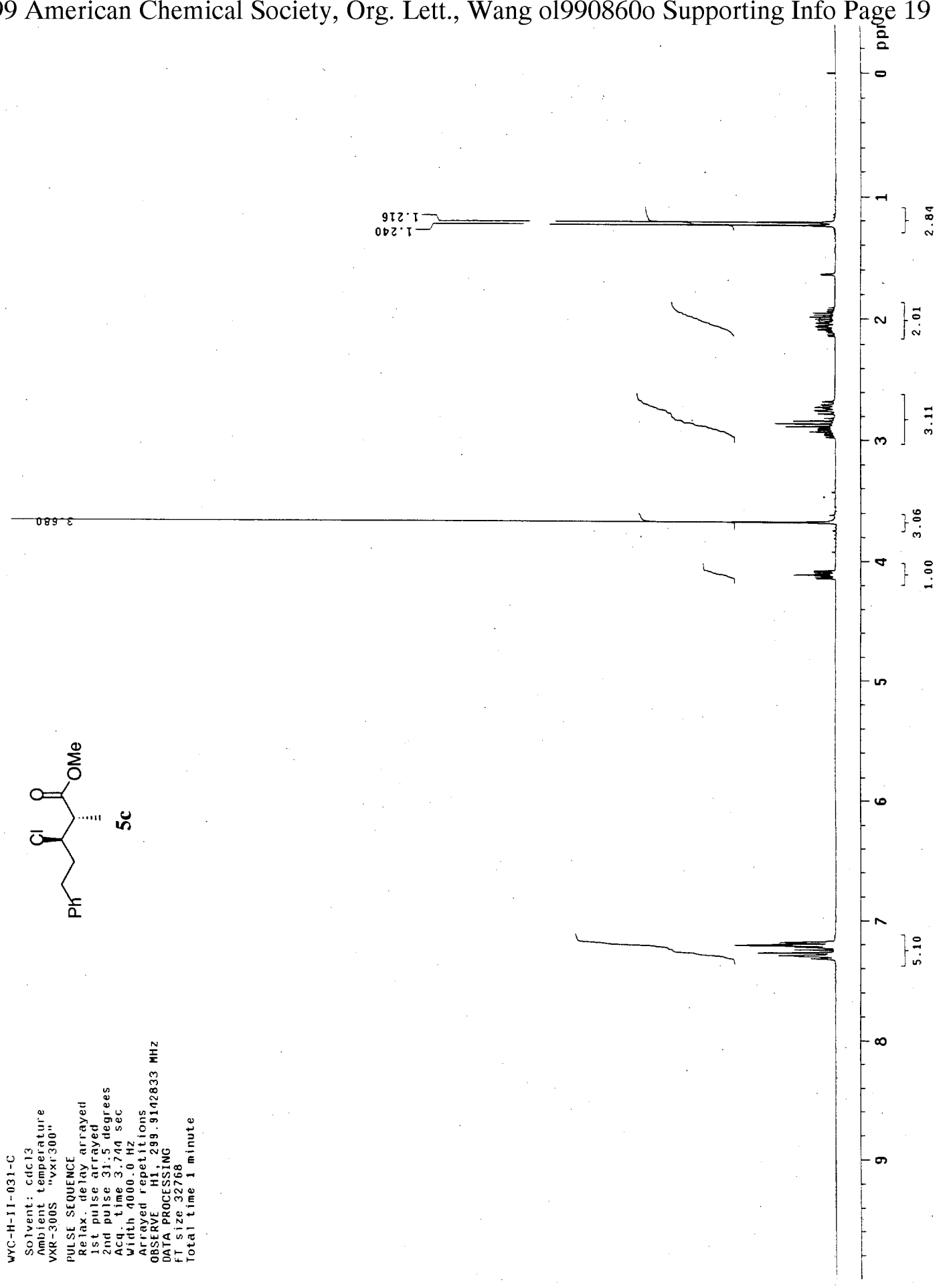
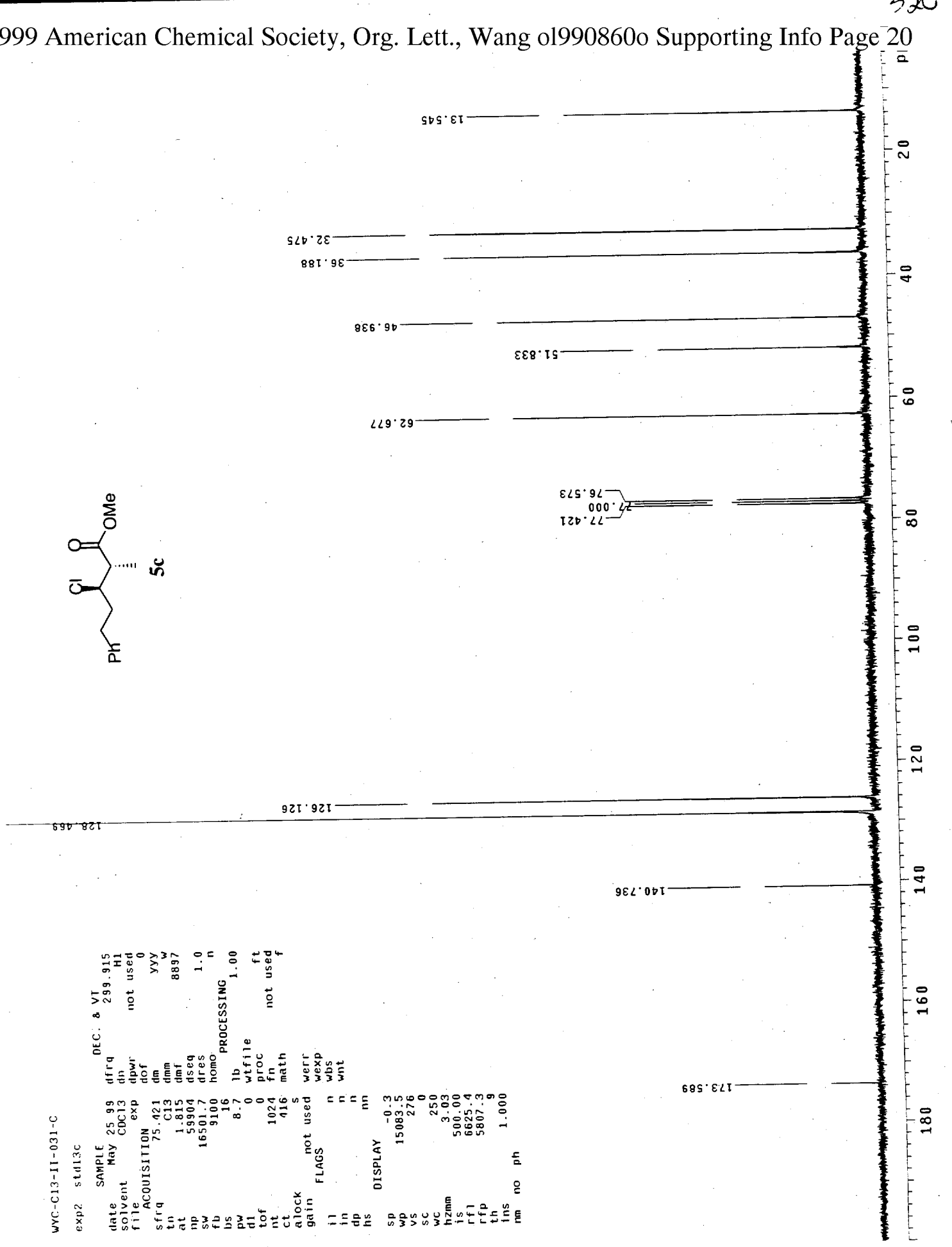
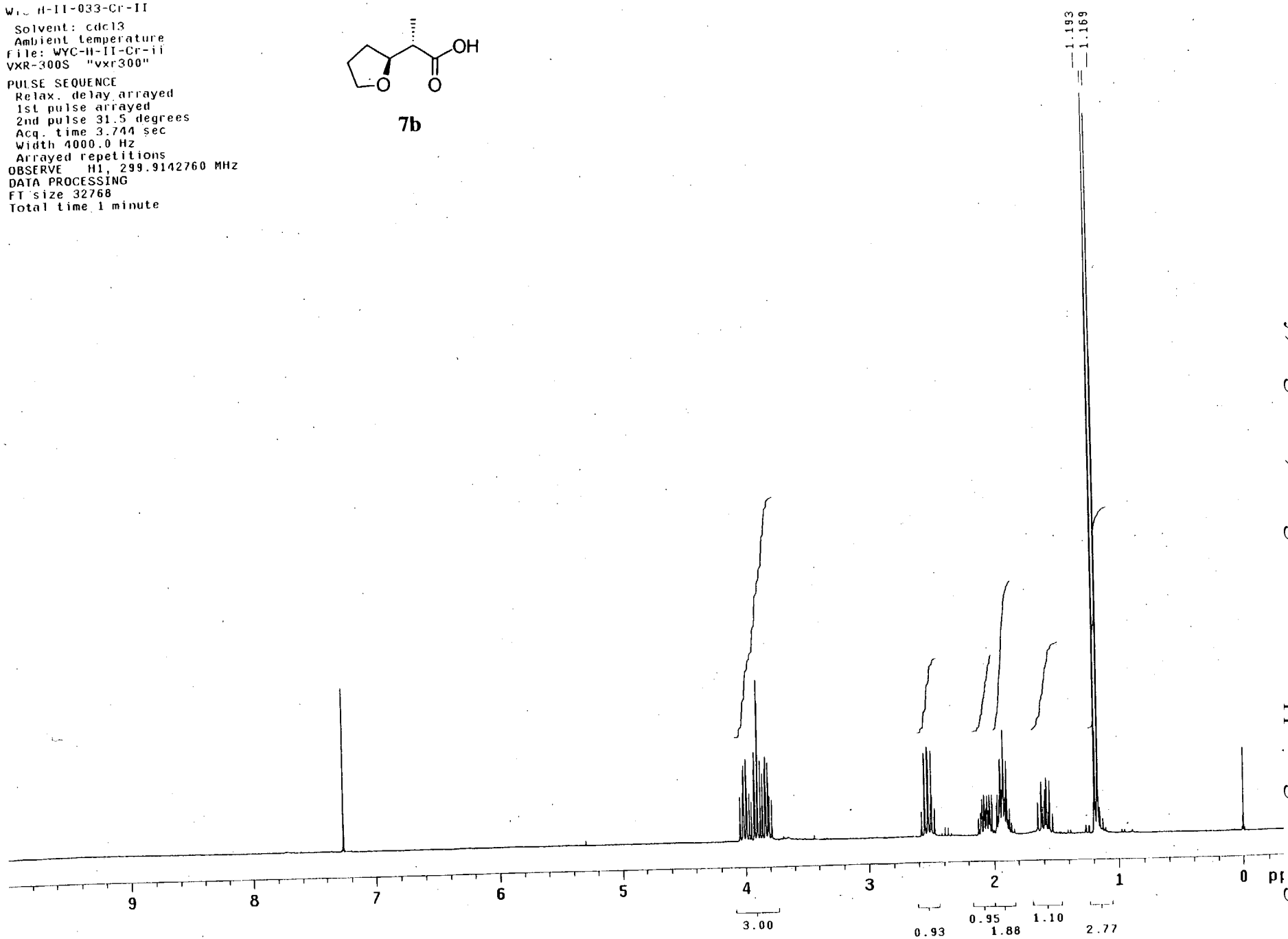
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 16 minutes


WY-II-032-A
Solvent: cdcl3
Ambient temperature
VXR-300S "vxr300"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 31.5 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
Arrayed repetitions
OBSERVE H1, 299.9142811 MHz
DATA PROCESSING
FT size 32768
Total time 1 minute
1.288
1.264
4.152
4.142
4.130
4.119
4.109
4.096
4.086
5.08
1.00
1.02
1.94
1.94
2.79
ppm
OH
O
Cl
Ph
5b




WYC-H-II-031-C
Solvent: cdcl3
Ambient temperature
VXR-300S "vxr300"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 31.5 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
Arrayed repetitions
OBSERVE H1, 299.9142833 MHz
DATA PROCESSING
FT size 32768
Total time 1 minute
Cl
O
OMe
Ph
5c
1.240
1.216
3.680
ppm
0
1
2
3
4
5
6
7
8
9
5.10
1.00
3.06
3.11
2.01
2.84



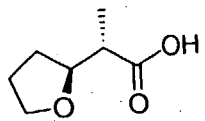


WYC-C13-II-033-Cr-II

13C OBSERVE

Solvent: CDCl3
Ambient temperature
VXR-300S "vxr300"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 62.4 degrees
Acq. time 1.815 sec
Width 16501.7 Hz
Arrayed repetitions
OBSERVE C13, 75.4135039 MHz
DECOUPLE H1, 299.9153562 MHz
High power 60
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 16 minutes



7b