

Allylic acetate 40. To a cooled (-20°C) solution of thiazole **28** (241 mg, 0.5 mmol) in 7 mL of THF was added 0.7 mL of buffered TBAF solution (1.0 M in THF with 20 mol % acetic acid added). After 1.5 h, the reaction was quenched with 3 mL of pH 7 buffer, diluted with 10 mL CH_2Cl_2 and warmed to 25°C . The layers were separated and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash chromatography to give 156 mg (98%) of the alcohol as a yellow oil: $R_f = 0.16$ (1:1-EtOAc:hexanes); IR (thin film) 3706-3047, 2977, 2955, 1725, 1225 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.10 (s, 1H), 5.97 (dq, $J = 1.2, 8.7$ Hz, 1H), 4.79 (dt, $J = 5.7, 8.7$ Hz, 1H), 4.14 (q, $J = 7.2$ Hz, 2H), 3.26 (d, $J = 5.7$ Hz, 2H), 2.90-3.20 (br s, 1H), 2.28 (d, $J = 1.2$ Hz, 3H), 1.40 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 167.2, 161.2, 146.7, 132.8, 127.6, 124.6, 68.0, 61.5, 40.1, 24.0, 14.3; HRMS (FAB) Calcd for $\text{C}_{11}\text{H}_{14}^{81}\text{BrNO}_3\text{S}[\text{M}+\text{H}]$: 321.9935. Found: 321.9926. To a solution of the alcohol (156 mg, 0.5 mmol) in 5 mL of CH_2Cl_2 was added acetic anhydride (0.070 mL, 0.74 mmol), pyridine (0.060 mL, 0.74 mmol) and DMAP (6 mg, 0.05 mmol) sequentially. After 2.5 h, the reaction was quenched with 1 mL pH 7 buffer and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 and the combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. Purification by flash chromatography on SiO_2 eluting with hexanes:EtOAc (4:1 $\rightarrow$ 1:1) gave 149.8 mg (83%) of acetate **40** as a clear colorless oil: $R_f = 0.67$ (1:1-EtOAc:hexanes); IR (thin film) 2986, 1739, 1229, 1028 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.11 (s, 1H), 5.85 (dq, $J = 1.2, 9.6$ Hz, 1H), 5.70 (ddd, $J = 5.7, 7.2, 9.6$ Hz, 1H), 4.43 (q, $J = 7.2$ Hz, 2H), 3.42 (dd, $J = 7.2, 14.9$ Hz, 1H), 3.35 (dd, $J = 5.7, 14.9$ Hz, 1H), 2.33 (d, $J = 1.2$ Hz, 3H), 2.06 (s, 3H), 1.41 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 169.7, 165.4, 161.2, 147.0, 128.6, 128.0, 127.8, 69.6, 61.5, 38.0, 24.4, 21.0, 14.4; HRMS (FAB) Calcd for $\text{C}_{13}\text{H}_{16}^{79}\text{BrNO}_4\text{S}[\text{M}+\text{H}]$: 362.0061. Found: 362.0063.

Triene 41. A solution of bromide **28** (9.8 mg, 0.02 mmol) in 0.1 mL of N-methyl pyrrolidinone (NMP) was degassed by several freeze/thaw cycles. Trifurylphosphine (0.6 mg, 0.002 mmol) and Pd_2dba_3 (0.9 mg, 0.0009 mmol) were added as solids resulting in a yellow homogenous solution. To this was added a solution of stannane **4** in 0.3 mL of NMP. After 18 h, the reaction was partitioned between Et_2O and 10% aqueous NH_4OH . The aqueous layer was extracted with Et_2O and the combined organic layers were dried over Na_2SO_4 . Concentration *in vacuo* and purification of the residue by flash chromatography on SiO_2 eluting with hexanes:EtOAc: Et_3N (89:10:1 $\rightarrow$ 79:20:1) to hexanes:acetone: Et_3N (79:20:1 $\rightarrow$ 77:20:3) gave 46 mg (46%) of triene **41** as a clear colorless oil: IR (thin film) 2943, 2863, 1719, 1066 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.09 (s, 1H), 6.24 (d, $J = 15.9$, 1H), 6.13 (d, $J = 15.9$ Hz, 1H), 5.60 (t, $J = 6.9$ Hz, 1H), 5.49 (d, $J = 8.7$ Hz, 1H), 5.02 (dt, $J = 5.7$, 8.4 Hz, 1H), 4.42 (q, $J = 6.9$ Hz, 2H), 3.35 (dd, $J = 6.3$, 14.4 Hz, 1H), 3.25 (dd, $J = 5.4$, 14.4 Hz, 1H), 3.06 (d, $J = 6.6$ Hz, 2H), 2.23 (s, 6H), 1.79 (s, 3H), 1.70 (s, 3H), 1.39 (t, $J = 6.9$ Hz, 3H), 1.01 (s, 21H); ^{13}C NMR (75 MHz, CDCl_3) δ 167.6, 161.8, 146.5, 134.4, 134.0, 133.0, 131.6, 128.3, 69.0, 61.5, 57.3, 45.4, 42.7, 18.2, 18.1, 14.6, 13.4, 13.0, 12.6.

Triene 42. A stirred solution of bromide **40** (21 mg, 0.057 mmol) in 0.3 mL of THF was degassed by several freeze/thaw cycles. Trifurylphosphine (3.3 mg, 0.014 mmol) and Pd_2dba_3 (2.7 mg, 0.003 mmol) were added as solids resulting in a yellow-green colored homogenous solution after 15 min. A solution of stannane **4** in 0.3 mL of THF was then added via cannula. After 46 h, the reaction was diluted with EtOAc, filtered through Celite and concentrated *in vacuo*. Purification of the residue by flash chromatography on SiO_2 eluting with hexanes:EtOAc (4:1 $\rightarrow$ 0:1) to hexanes:acetone: Et_3N (35:6:1 $\rightarrow$ 24:25:1) gave 12.3 mg (53%) of triene **42** as an orange-yellow oil: IR (CHCl_3) 3017, 2971, 2926, 1797, 1715, 1214 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.10 (s, 1H), 6.34 (d, $J = 15.9$ Hz, 1H), 6.17 (d, $J = 15.9$ Hz, 1H), 5.93 (ddd, $J = 5.9$, 7.4, 9.3 Hz, 1H), 5.63 (t, $J = 6.6$ Hz, 1H), 5.44 (d, $J = 9.3$ Hz, 1H), 4.43

(q, $J = 7.2$ Hz, 2H), 3.45 (dd, $J = 7.4, 14.9$ Hz, 1H), 3.38 (dd, $J = 5.9, 14.9$ Hz, 1H), 3.05 (d, $J = 6.9$ Hz, 2H), 2.34 (s, 6H), 2.05 (s, 3H), 1.87 (d, $J = 1.2$ Hz, 3H), 1.79 (s, 3H), 1.41 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 169.8, 166.2, 161.3, 146.8, 139.2, 135.8, 134.4, 130.7, 130.4, 127.9, 127.0, 69.9, 61.4, 57.2, 45.3, 38.7, 21.1, 14.4, 13.2, 12.6; HRMS (FAB) Calcd for $\text{C}_{21}\text{H}_{30}^{79}\text{BrN}_2\text{O}_4\text{S}[\text{M}+\text{Na}]$: 429.1836. Found: 429.1824.

β -Hydroxyester 44. To a cooled (0°C) solution of diisopropylamine (6.82 mL, 52 mmol) in 200 mL of THF was added 21.0 mL of an *n*-butyllithium solution (48 mmol, 2.3 M in hexanes). After 30 min, the solution was cooled to -78°C and methyl acetate (3.82 mL, 48 mmol) was added. Hydrocinnamaldehyde (5.27 mL, 40 mmol) was added to the flask after an additional 30 min. The reaction was partitioned between pH 7 buffer and EtOAc after 7 h and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with NH_4Cl , H_2O , then brine, and dried over Na_2SO_4 . Purification by flash chromatography on SiO_2 eluting with hexanes:EtOAc (7:3) gave 6.66 g (80%) of ester 44 as a clear colorless oil which correlated with data previously reported for this compound⁴¹: $R_f = 0.40$ (3:7-EtOAc:hexanes); IR (thin film) 3453, 3026, 2950, 2860, 1719, 1602, 1495 cm^{-1} ; ^1H NMR (300 MHz) δ 7.15-7.25 (m, 5H), 4.06 (m, 1H), 3.73 (s, 3H), 2.66-2.92 (m, 2H), 2.44-2.60 (m, 2H), 1.72-1.94 (m, 2H); ^{13}C NMR (300 MHz) δ 173.5, 141.9, 128.6, 128.6, 126.1, 67.4, 51.9, 41.3, 38.3, 31.9; HRMS (FAB) Calcd for $\text{C}_{12}\text{H}_{17}\text{O}_3[\text{M}+\text{H}]$: 209.1178. Found: 209.1184.

β -Hydroxyamide 45. To a cooled (0°C) suspension of *O*-benzylhydroxylamine hydrochloride (2.71 g, 17 mmol) in 20 mL of CH_2Cl_2 was added 8.5 mL of trimethylaluminum (17 mmol, 2.0 M in hexanes) and the solution was warmed to 25°C . After 1 h, the solution was recooled to 0°C and a solution of β -hydroxyester 44 (1.99 g, 9.6 mmol) in 10 mL of CH_2Cl_2 solution was added dropwise. After stirring for 12 h at 25°C , the reaction was cooled to 0°C and quenched with 50 mL of 10% aqueous citric acid. After 1 h, the layers were separated and the aqueous layer was extracted with CH_2Cl_2 . The combined organic layers were washed with 10%

aqueous citric acid, saturated NaHCO_3 and brine then dried over Na_2SO_4 . Concentration *in vacuo* gave white crystals that were washed with Et_2O to yield 1.79 g (63%) of amide **45** which was of sufficient purity to be used in the next step without further purification: $R_f = 0.41$ (1:1-EtOAc:hexanes); ^1H NMR (300 MHz, d_6 -DMSO) δ 7.15-7.45 (m, 10H), 4.81 (s, 2H), 3.80-3.95 (m, 1H), 2.66-2.80 (m, 1H), 2.50-2.66 (m, 1H), 2.13 (d, $J = 6.6$ Hz, 2H), 1.52-1.76 (m, 2H); ^{13}C NMR (75 MHz, d_6 -DMSO) δ 167.7, 142.2, 136.1, 128.7, 128.3, 128.24, 128.18, 125.6, 76.8, 66.4, 40.9, 40.3, 38.8; HRMS (FAB) Calcd for $\text{C}_{18}\text{H}_{22}\text{NO}_3[\text{M}+\text{H}]$: 300.1600. Found: 300.1612.

β -Lactam 46a. To a solution of β -hydroxyamide **45** (1.6 g, 5.4 mmol) in 37 mL of acetonitrile was added 1.35 mL of carbon tetrachloride (14.0 mmol), 0.82 mL of Et_3N (5.9 mmol), and triphenylphosphine (1.55 g, 5.9 mmol). After stirring 12 h, the solvent was removed *in vacuo*. The residue was recrystallized from EtOAc/hexanes to give a white crystalline solid that was discarded ($\text{Ph}_3\text{P}=\text{O}$). The mother liquor was purified by flash chromatography on SiO_2 eluting with hexanes:EtOAc (7:3) to give 1.03 g (68%) of lactam **46a** as a white crystalline solid: IR (thin film) 3231, 3063, 3027, 2951, 2851, 1773, 1495, 1453 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.00-7.60 (m, 5H), 4.95 (s, 2H), 3.43-3.57 (m, 1H), 2.67 (dd, $J = 5.1, 13.5$ Hz, 1 H), 2.50-2.64 (m, 2H), 2.24 (dd, $J = 2.7, 13.5$ Hz, 1H), 1.90-2.06 (m, 1H), 1.60-1.80 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 164.3, 140.8, 135.5, 129.4, 129.1, 128.8, 128.7, 128.5, 128.4, 126.4, 78.4, 57.7, 38.0, 34.2, 32.0; HRMS (FAB) Calcd for $\text{C}_{18}\text{H}_{20}\text{NO}_2[\text{M}+\text{H}]$: 282.1494. Found: 282.1506.

β -Lactam 46b. A solution of β -lactam (1.18 g, 4.2 mmol) in 2.4 mL of THF and 1.5 mL of water (83 mmol) was deoxygenated by several freeze/thaw cycles. To this solution was added 42 mL of SmI_2 solution (12.6 mmol, 0.3 M in Et_2O). After stirring for 10 h, the reaction was partitioned between EtOAc and saturated NaHCO_3 and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with $\text{Na}_2\text{S}_2\text{O}_3$, water, and brine and then dried

over Na_2SO_4 . Concentration *in vacuo* followed by purification by flash chromatography eluting with hexanes:EtOAc (2:3) gave 652 mg (88%) of a white amorphous solid: $R_f = 0.50$ (1:1-EtOAc:hexanes) IR (thin film) 3252, 3026, 2938, 1735 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.16-7.36 (m, 5H), 5.90-6.15 (br s, 1H), 3.53-3.69 (m, 1H), 3.03 (ddd, $J = 1.8, 5.1, 14.9$ Hz, 1H), 2.67 (td, $J = 1.8, 7.5$ Hz, 2H), 2.55 (dd, $J = 1.8, 14.9$ Hz, 1H), 1.95 (dd, $J = 7.8, 14.4$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.0, 168.2, 140.6, 129.1, 128.4, 128.4, 128.3, 128.2, 126.8, 126.1, 60.3, 47.6, 43.3, 36.8, 32.6, 20.9, 14.1; HRMS (FAB) Calcd for $\text{C}_{11}\text{H}_{14}\text{NO}[\text{M}+\text{H}]$: 176.1075. Found: 176.1073.

N-Boc- β -lactam 46c. To a solution of β -lactam **46b** (105 mg, 0.6 mmol) in 3.0 mL of CH_2Cl_2 was added a solution of di-*tert*-butyl dicarbonate (605 mg, 2.8 mmol) and dimethylaminopyridine (10 mg, 0.08 mmol) in 1 mL of CH_2Cl_2 at 25°C followed by 0.4 mL of Et_3N (2.73 mmol). After 2 h, the reaction was quenched with 1 mL pH 7 buffer and extracted with CH_2Cl_2 . The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash chromatography on SiO_2 eluting with hexanes:EtOAc (1:4) to give 124 mg (75%) of lactam **46c** as a clear colorless oil: $R_f = 0.50$ (3:7-EtOAc:hexanes); IR (thin film) 3409, 3112, 2975, 2949, 2932, 2869, 1813, 1715 cm^{-1} ; ^1H NMR (300 MHz, CD_2Cl_2) δ 7.16-7.34 (m, 5H), 3.92 (qt, $J = 3.3, 6.0$ Hz, 1H), 3.01 (dd, $J = 6.0, 16.1$ Hz, 1H), 2.67 (t, $J = 8.0$ Hz, 2H), 2.56 (dd, $J = 3.3, 16.1$ Hz, 1H), 2.39-2.50 (m, 1H), 1.77-1.90 (m, 1H), 1.49 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 164.6, 160.5, 148.0, 140.6, 128.7, 128.3, 126.3, 83.2, 51.2, 42.1, 34.4, 31.6, 28.1; HRMS (FAB) Calcd for $\text{C}_{16}\text{H}_{22}\text{NO}_3[\text{M}+\text{H}]$: 276.1600. Found: 276.1608.

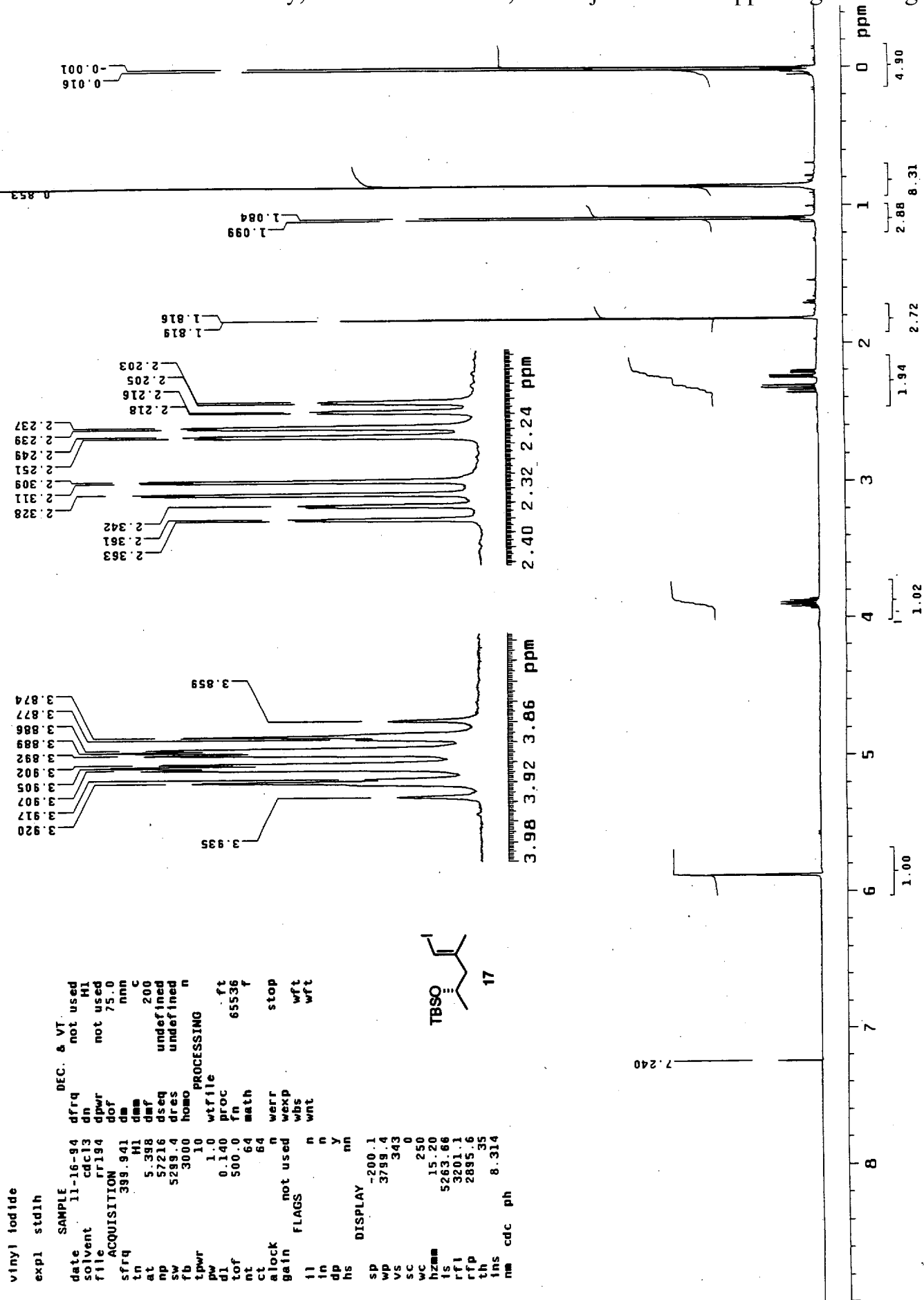
N-Trifluoroacetyl- β -lactam 46d. This compound was prepared using a similar procedure as for β -lactam **46c** using lactam **46b** (76 mg, 0.4 mmol), trifluoroacetic anhydride (0.094 mL, 0.7 mmol) and pyridine (0.057 mL, 0.7 mmol) in 4.5 mL of CH_2Cl_2 to give 55 mg

(46%) of lactam **46d**: $R_f = 0.48$ (3:7-EtOAc:hexanes); IR (thin film) 3424, 3084, 3064, 3026, 2952, 2652, 2860, 1818, 1724 cm^{-1} ; ^1H NMR (200 Mhz, CDCl_3) δ 7.2-7.5 (m, 5H), 4.15 (m, 1H), 3.23 (dd, $J = 6.6, 16.9$, 1H), 2.75 (dd, $J = 4.0, 16.9$, 1H), 2.5-2.8 (m, 2H), 1.8-2.0 (m, 2H); ^{13}C NMR (200 MHz, CDCl_3) δ 161.4, 140.0, 129.0, 128.5, 126.8, 51.4, 43.0, 33.7, 31.6.

β -Aminoester 47a. Performed as described below for β -aminoester **47c** using 48 mg (0.2 mmol) of β -lactam **46a**, 0.094 mL of sodium bis(trimethylsilyl)amide (0.19 mmol, 2.0 M in THF), and 0.013 mL of isopropyl alcohol (0.2 mmol) in 1 mL of THF. Purification by flash chromatography on SiO_2 eluting with hexanes/EtOAc (17:3) gave 39 mg (67%) of β -lactam **47a**. IR (thin film) 3027, 2979, 2932, 1719 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.15-7.35 (m, 10H), 5.00 (m, 1H), 4.71 (s, 2H), 3.35 (m, 2H), 2.45 (m, 1H), 2.45-2.47 (m, 2H), 1.60-2.00 (m, 2H), 1.20-1.22 (m, 6H); ^{13}C (75 MHz, CDCl_3) δ 171.8, 141.8, 137.9, 128.4, 128.3, 128.3, 127.8, 125.8, 76.4, 67.7, 57.1, 37.2, 33.6, 32.3, 21.8; HRMS (FAB) Calcd for $\text{C}_{21}\text{H}_{27}\text{NO}_3[\text{M}+\text{H}]$: 342.2069. Found 342.2086.

β -Aminoester 47c. To a cooled (-42°C) solution of 0.010 mL of isopropyl alcohol (0.15 mmol) in 0.5 mL of THF was added 0.080 mL of sodium bis(trimethylsilyl)amide (0.16 mmol, 2.0 M in THF). After 30 minutes, a solution of β -lactam **46c** (39 mg, 0.15 mmol) in 1 mL of THF was transferred via cannula. After 1.5 hours, the reaction was quenched with pH 7 buffer, warmed to 25°C and extracted with EtOAc. The combined organic layers were washed with water and brine, then dried over Na_2SO_4 . Purification by flash chromatography on SiO_2 eluting with hexanes:EtOAc (17:3) gave 39 mg (80%) of ester **47c** as solid: $R_f = 0.61$ (3:7-EtOAc:hexanes); IR (thin film) 3303, 2975, 1736, 1678, 1536 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.12-7.35 (m, 5H), 5.01 (app quin, $J = 6.0$ Hz, 1H), 3.90-4.10 (m, 1H), 2.60-2.77 (m, 2H), 2.42-2.58 (m, 2H), 1.70-1.90 (m, 2H), 1.55-1.70 (br s, 1H), 1.45 (s, 9H), 1.23 (d, $J = 6.0$ Hz,

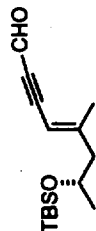
6H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.1, 155.3, 141.5, 128.4, 128.3, 125.9, 79.2, 68.0, 47.5, 39.6, 36.5, 32.5, 28.3, 21.8, 21.7, 16.1; HRMS (FAB) Calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_4$ [M+H]: 336.2135. Found: 336.2181.



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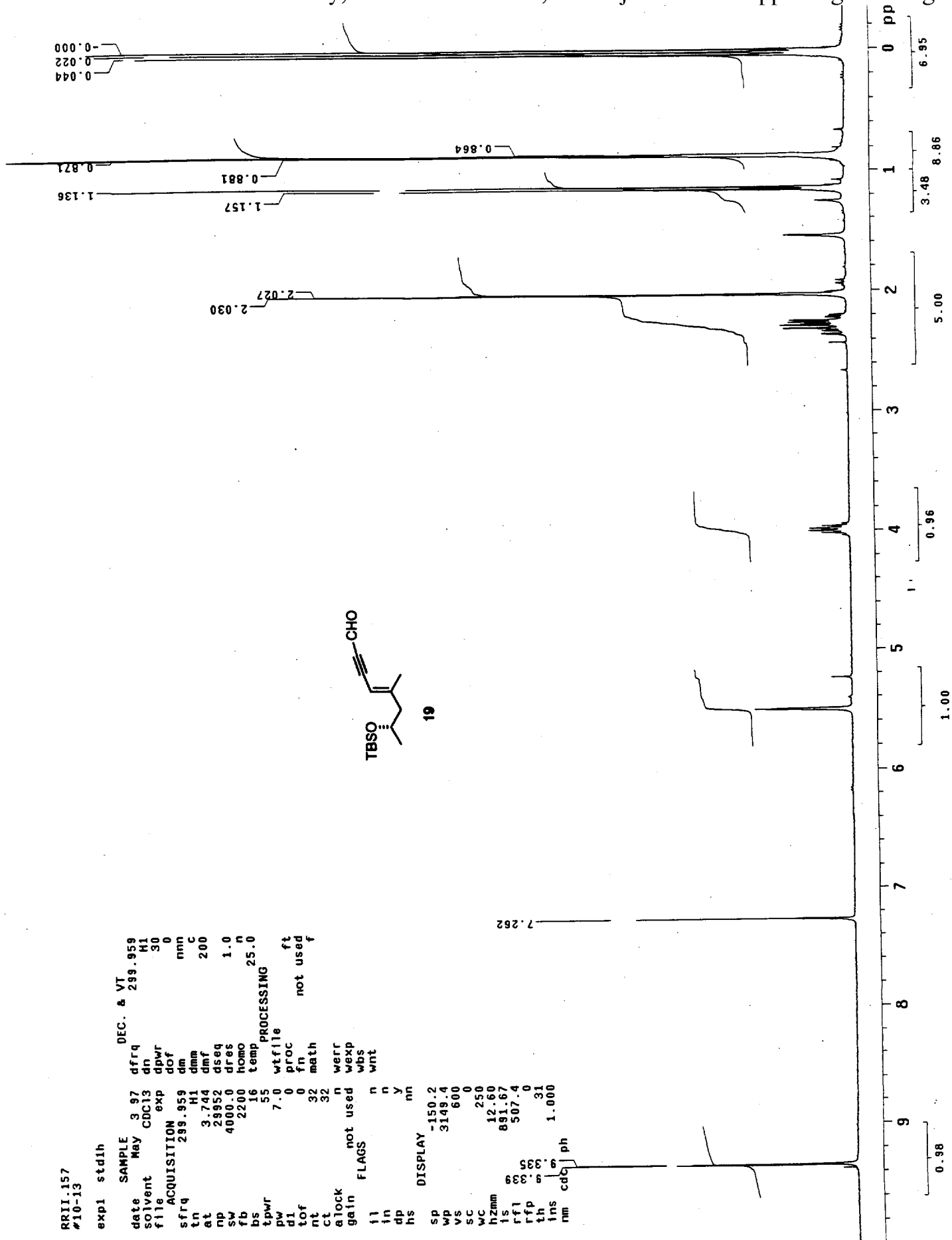
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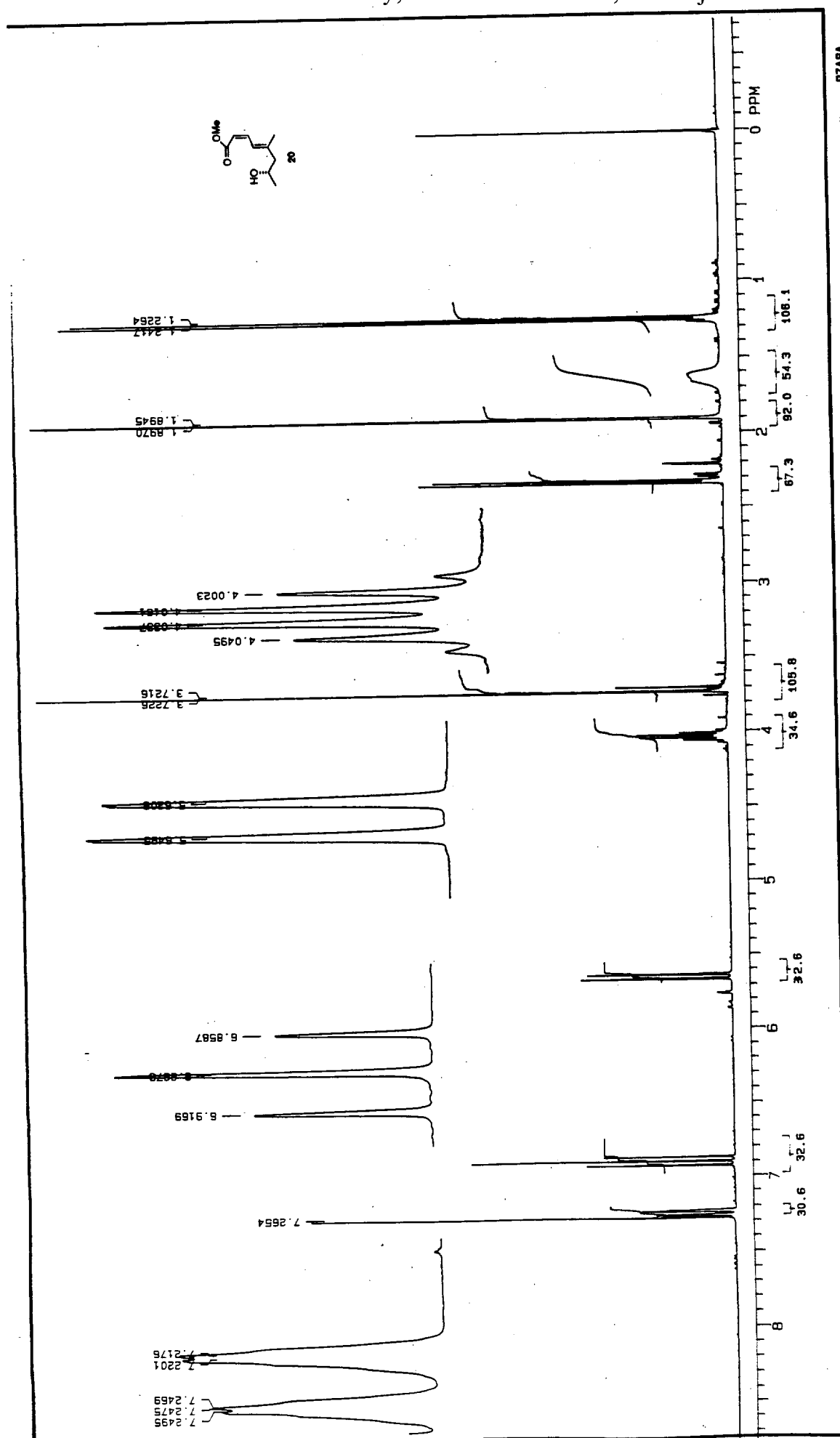
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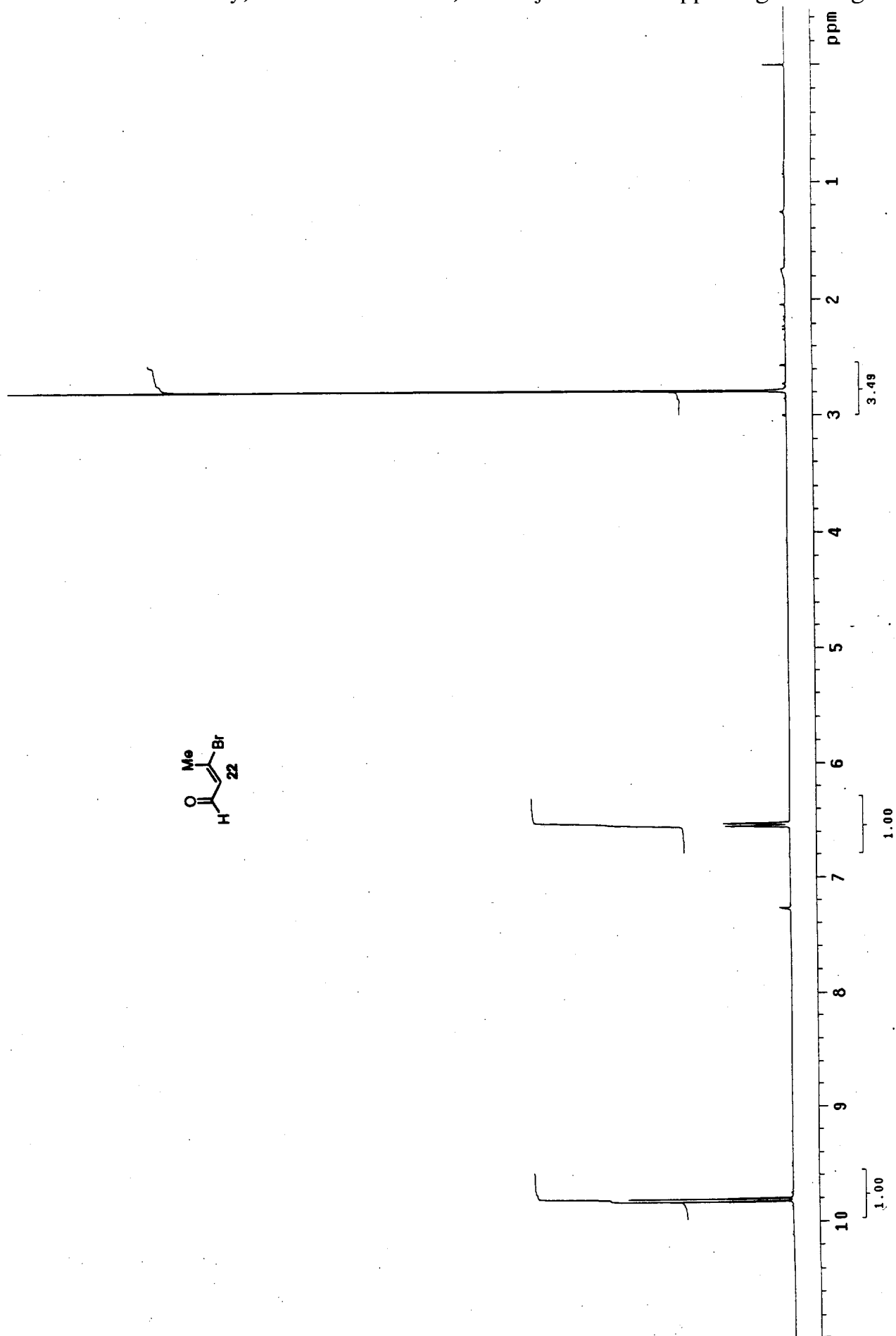
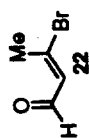
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RR1-218



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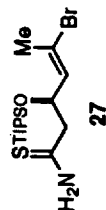
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5.030
5.028
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5.024
5.022
5.020
5.018
5.016
5.014
5.012
5.010
5.008
5.006
5.004
5.002
5.000



1.072

2.291
2.286

2.295 ppm

2.930
2.923
2.913

2.942

2.882
2.865

2.990
2.970

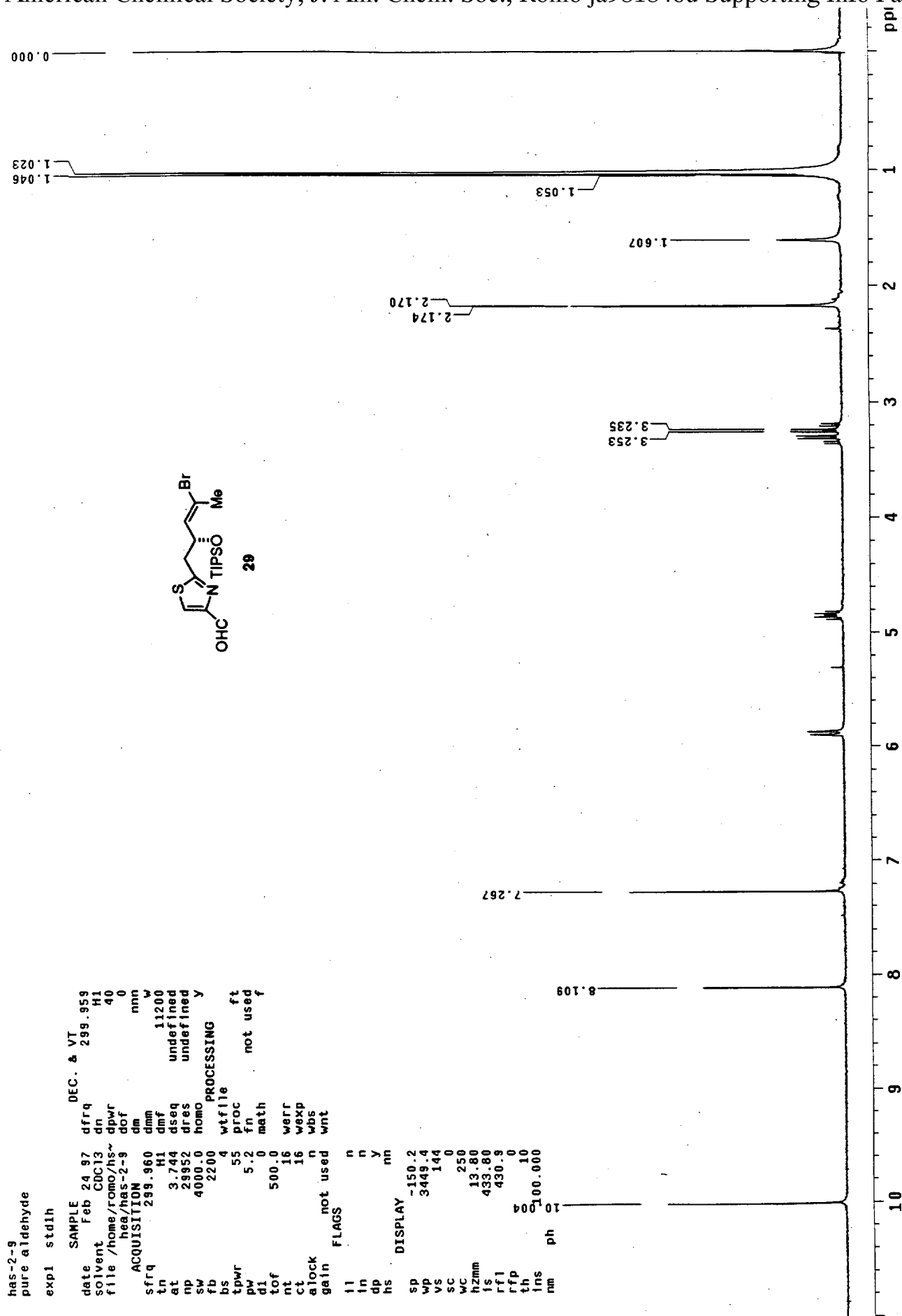
3.04 2.98 2.92 2.86 ppm

4.887
4.870
4.857
4.852
4.841
4.822

4.92 4.88 4.84 ppm

6.82 5.96 ppm

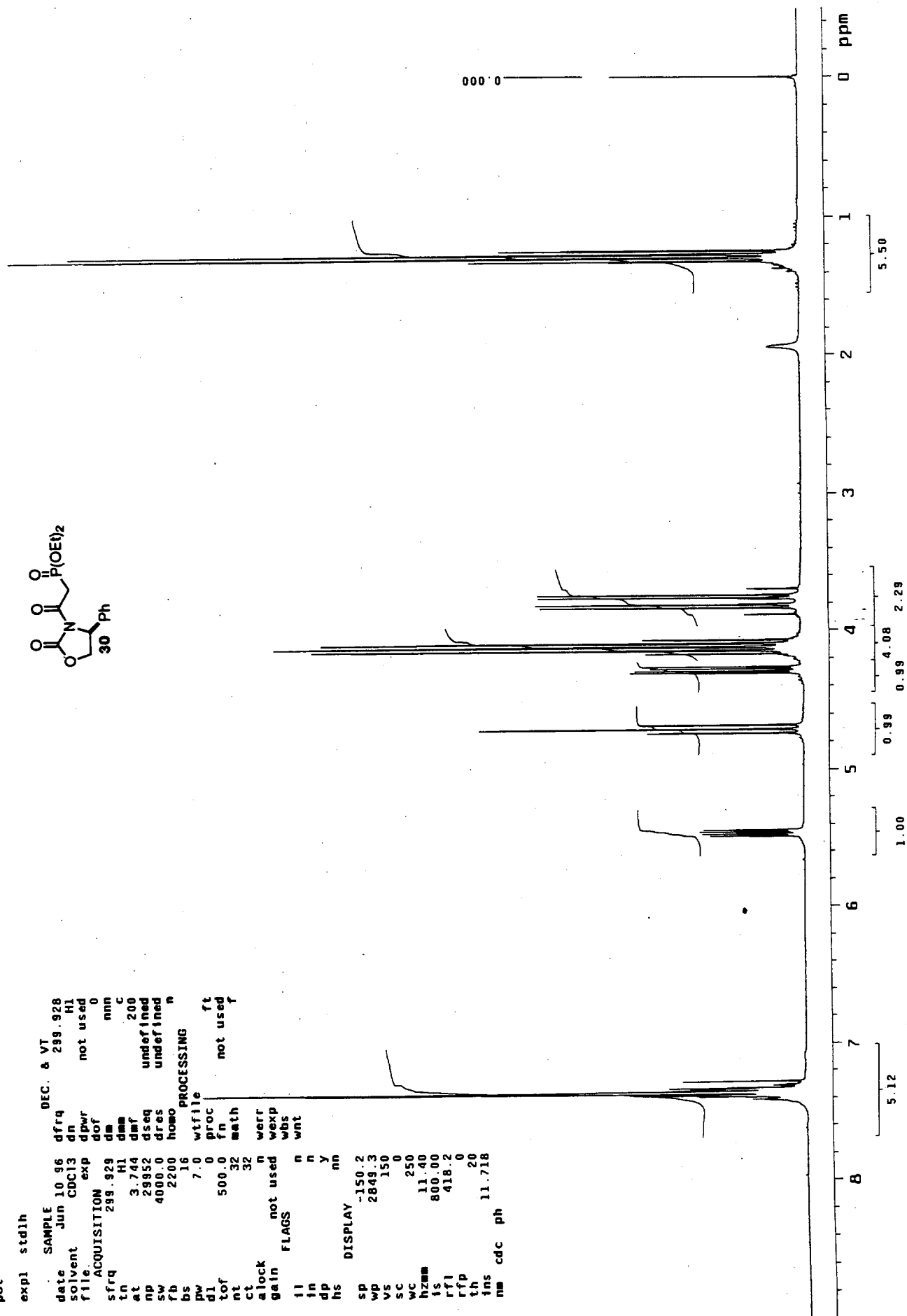
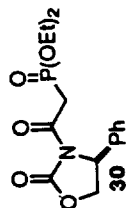
0 ppm
1 21.67
2 2.83
3 1.79
4 1.1
5 0.99
6 1.00
7
8
9



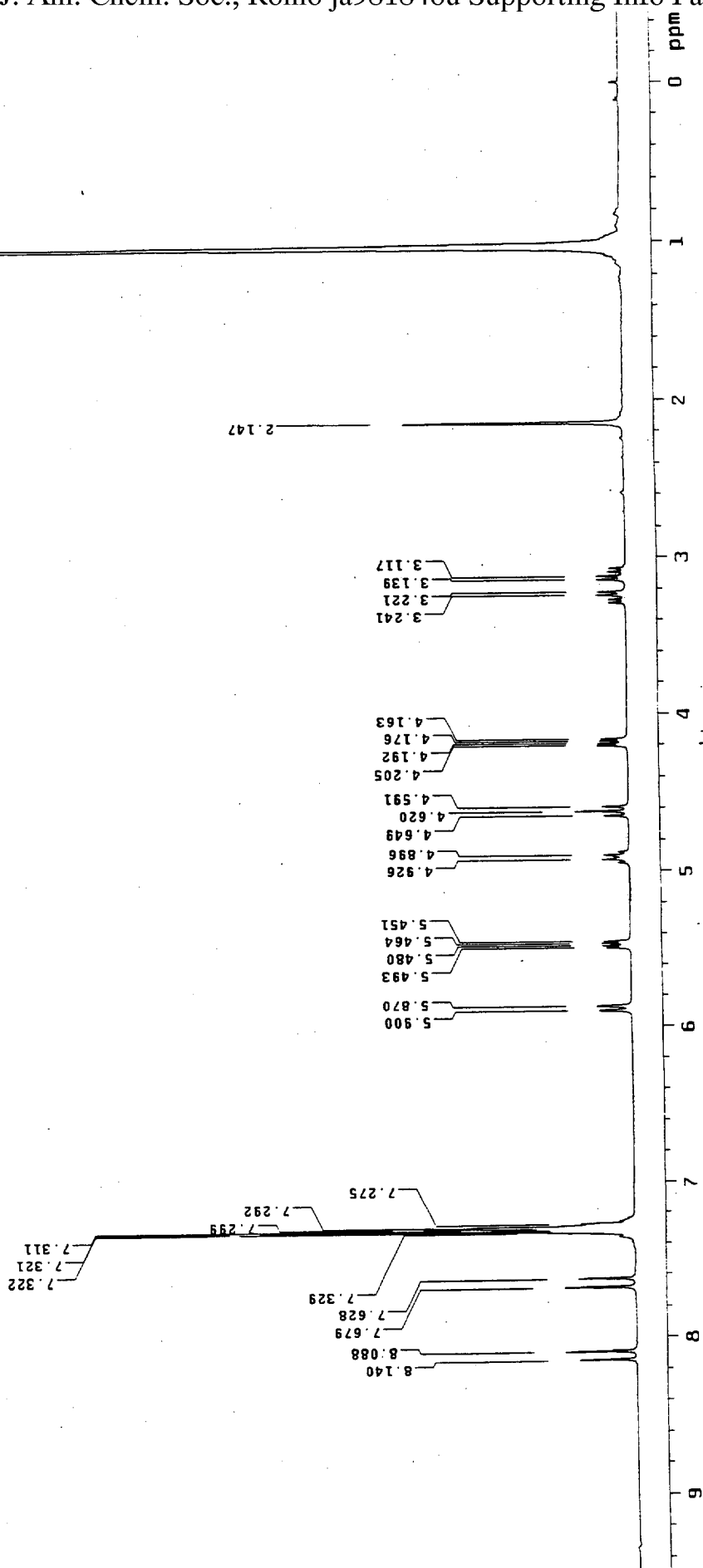
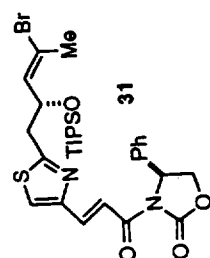
PRI 299
Kugelrohr dist. under vacuum at 90C
pot

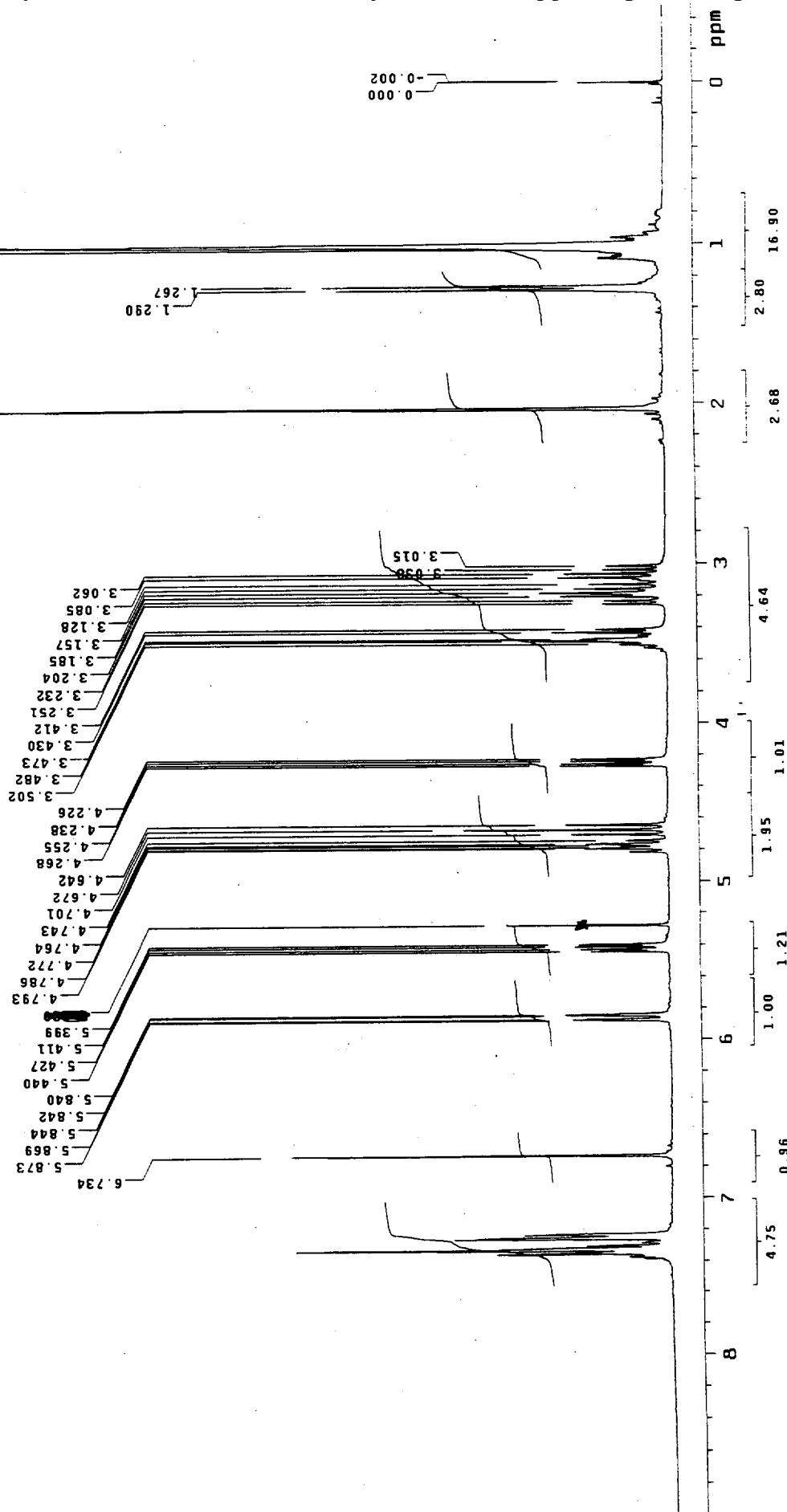
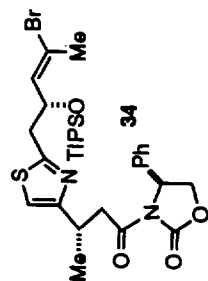
expl stdlh

date	SAMPLE	DEC. & VT
Jun 10 96	299.928	
solvent	Jun CDC13	H1
file	exp	not used
ACQUISITION	dof	0
sfrq	299.928	nm
tn	H1	C
at	3.744	200
np	29952	undefined
sw	4000.0	undefined
fb	2200	homo
bs	16	PROCESSING
pw	7.0	wtfile
dl	500.0	proc
nt	32	fn
ct	32	math
alock	not used	n
gain	FLAGS	werr
ll	n	wexp
in	n	wbs
dp	y	wnt
hs	nn	
sp	-150.2	
wp	2849.3	
vs	150	
sc	0	
wc	250	
hzmm	11.40	
ts	800.00	
rfl	418.2	
rfp	0	
th	20	
ins	11.718	
nm	cdc	ph



has-2-11
pure alkene product





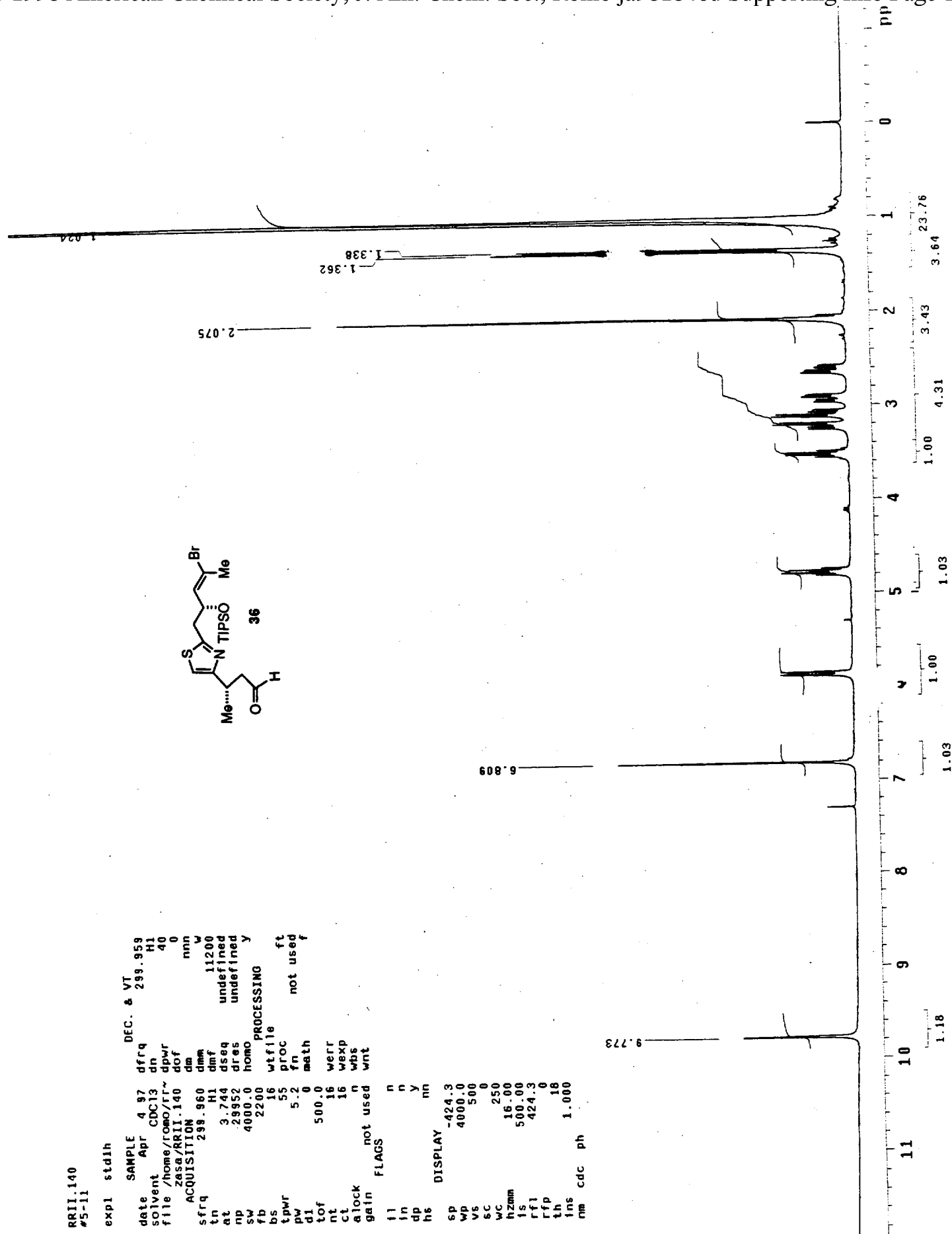
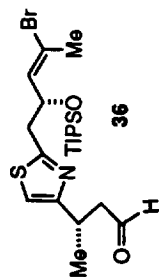
HAS-II.17
#36-41

tl5

RR11.140
#5-11

expl stdlh

SAMPLE 4 97 dfrq DEC. & VI
 date Apr 299.959
 solvent CDC13 dn H1
 file /home/romo/tr~ dpwr 40
 zasa/RR11.140 dof 0
 ACQUISITION dm nnn
 sfrq 299.960 dmf w
 tn 11200
 at 3.744 dseq undefined
 np 29952 dres undefined
 sw 4000.0 homo y
 fb 2200
 PROCESSING
 bs 16 wtfile
 tpwr 55 proc ft
 pw 5.2 fn not used
 dl 0 math
 tof 500.0
 nt 16 weff
 ct 16 wexp
 alock n wbs
 gain not used wnt
 FLAGS
 t1 n
 tn n
 dp y
 hs nm
 DISPLAY
 sp -424.3
 wp 4000.0
 vs 500
 sc 0
 wc 250
 hzmm 16.00
 fs 500.00
 rfl 424.3
 rfp 0
 th 18
 tns 1.000
 nm cdc ph

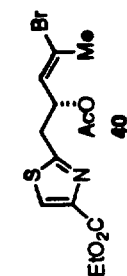


pe11.57
#15-23

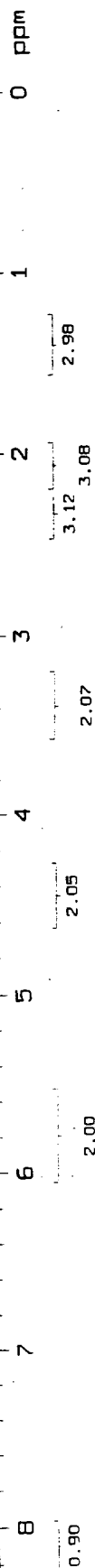
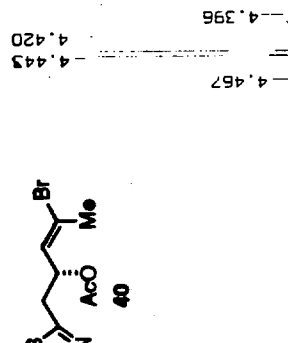
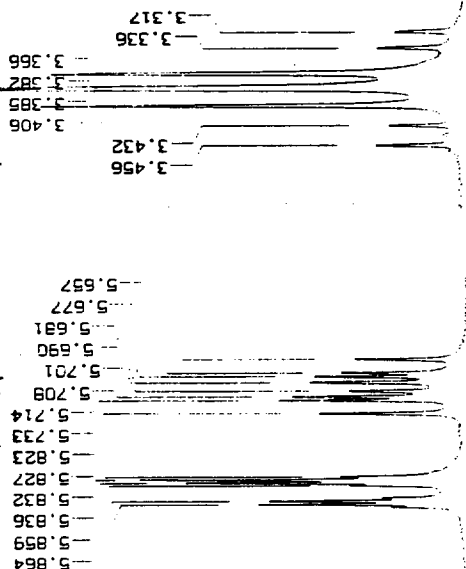
exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 14 96	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpr	40
ACQUISITION		dof	0
sfrq	299.960	dm	nm
tn	H1	dmm	nm
at	3.744	dms	11200
np	29952	dseq	undefined
sw	4000.0	dres	undefined
fb	2200	homo	y
bs	16	PROCESSING	
tpwr	55	wtfile	
pw	5.2	proc	ft
dl	0	fn	not used
tof	500.0	math	
nt	32	werr	
ct	32	wexp	
alock	n	wbs	
gain	not used	wnt	
ll	n	FLAGS	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-150.2		
wp	2849.5		
vs	170		
sc	0		
wc	250		
hzmm	11.40		
ls	1200.00		
rfl	429.0		
rfl	0		
th	106		
ins	12.603		
nm	cdc	ph	

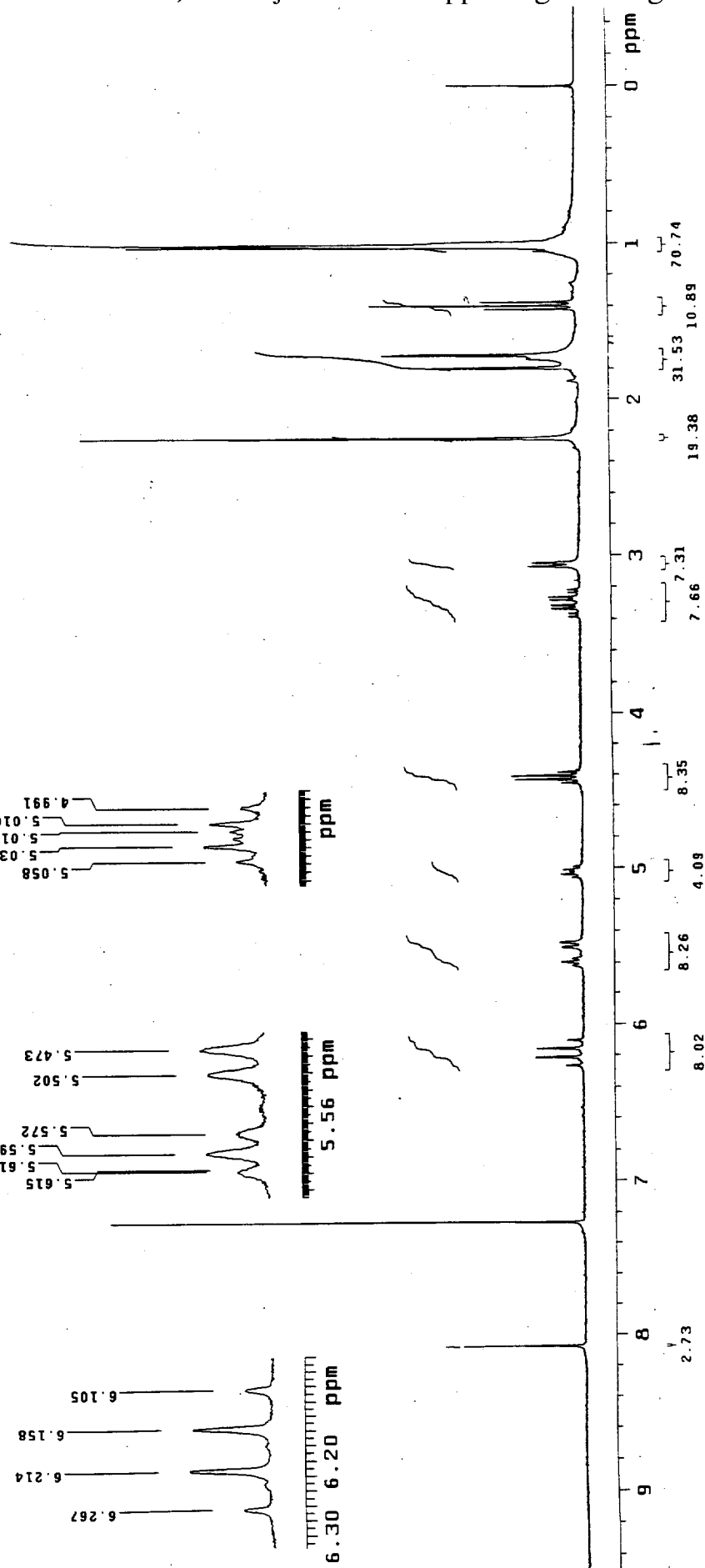
5.85 5.70 ppm



3.50 3.38 ppm



bis



RRII.60

13-20
Second Rack

expl stdlh

SAMPLE DEC. & VT
 date Oct 24 96 dfrq 299.928
 solvent CDCl3 dn H1
 file exp dpr 40
 ACQUISITION
 sfrq 299.929 nnn w
 tn H1 dm w
 at 3.744 dmf 11200
 np 29952 dseq undefined
 sw 4000.0 dres undefined
 fb 2200 homo y
 bs 16
 tpwr 55
 pw 5.2
 dl 0
 tof 500.0 math
 nt 32 werr
 ct 32 n wexp
 alock not used
 gain not used
 FLAGS
 fl n
 in n
 dp y
 hs nn
 DISPLAY
 sp 150.2
 wp 2849.3
 vs 400
 sc 0
 wc 250
 hzmm 11.40
 ls 463.14
 rfi 419.0
 rfp 0
 th 50
 ins 8.787
 nm cdc ph

PROCESSING

wtfile

proc

fn

math

werr

n wexp

whs

wnt

not used

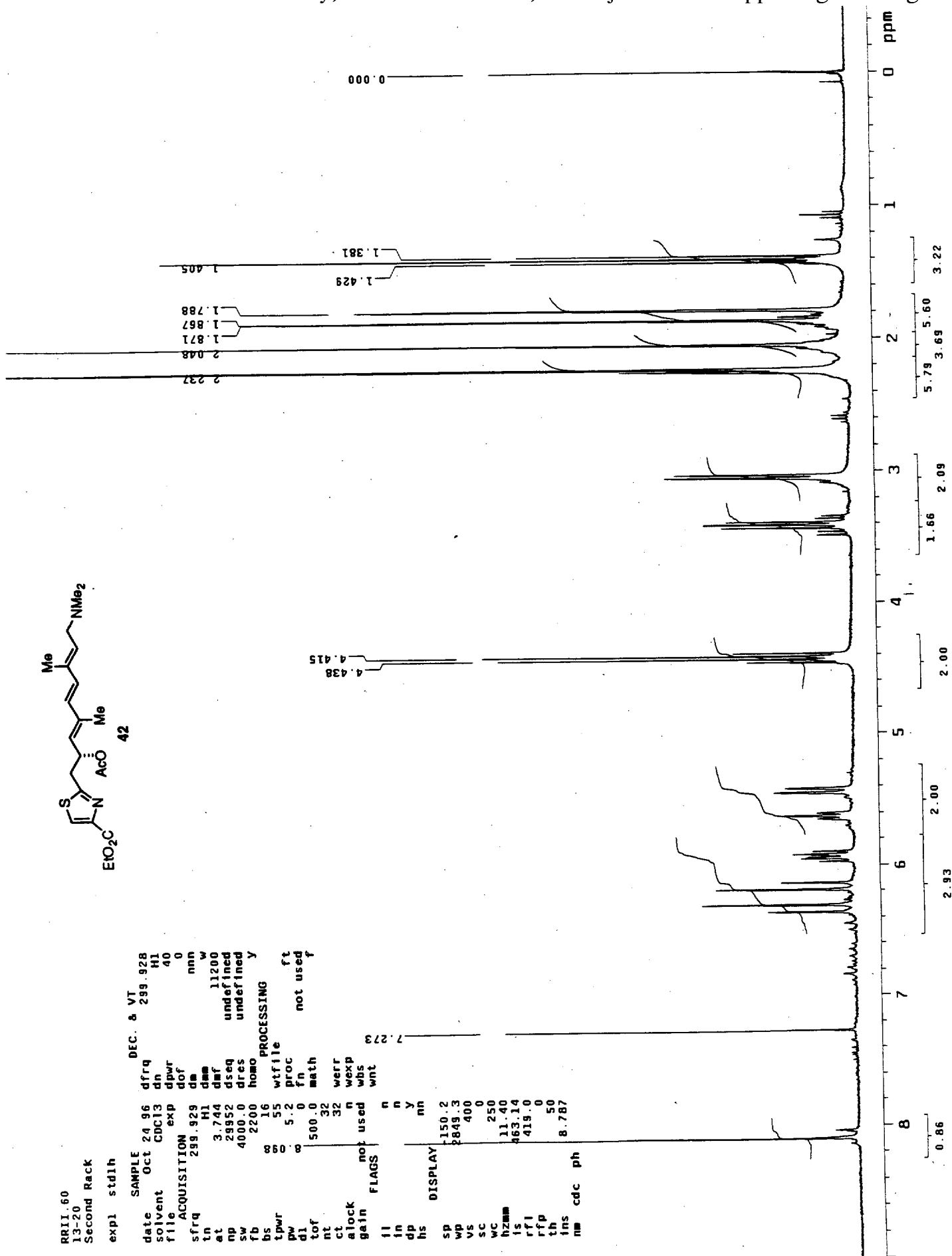
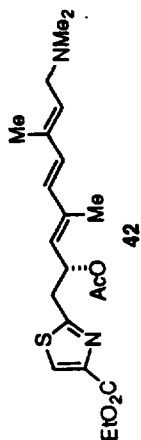
ft

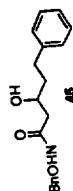
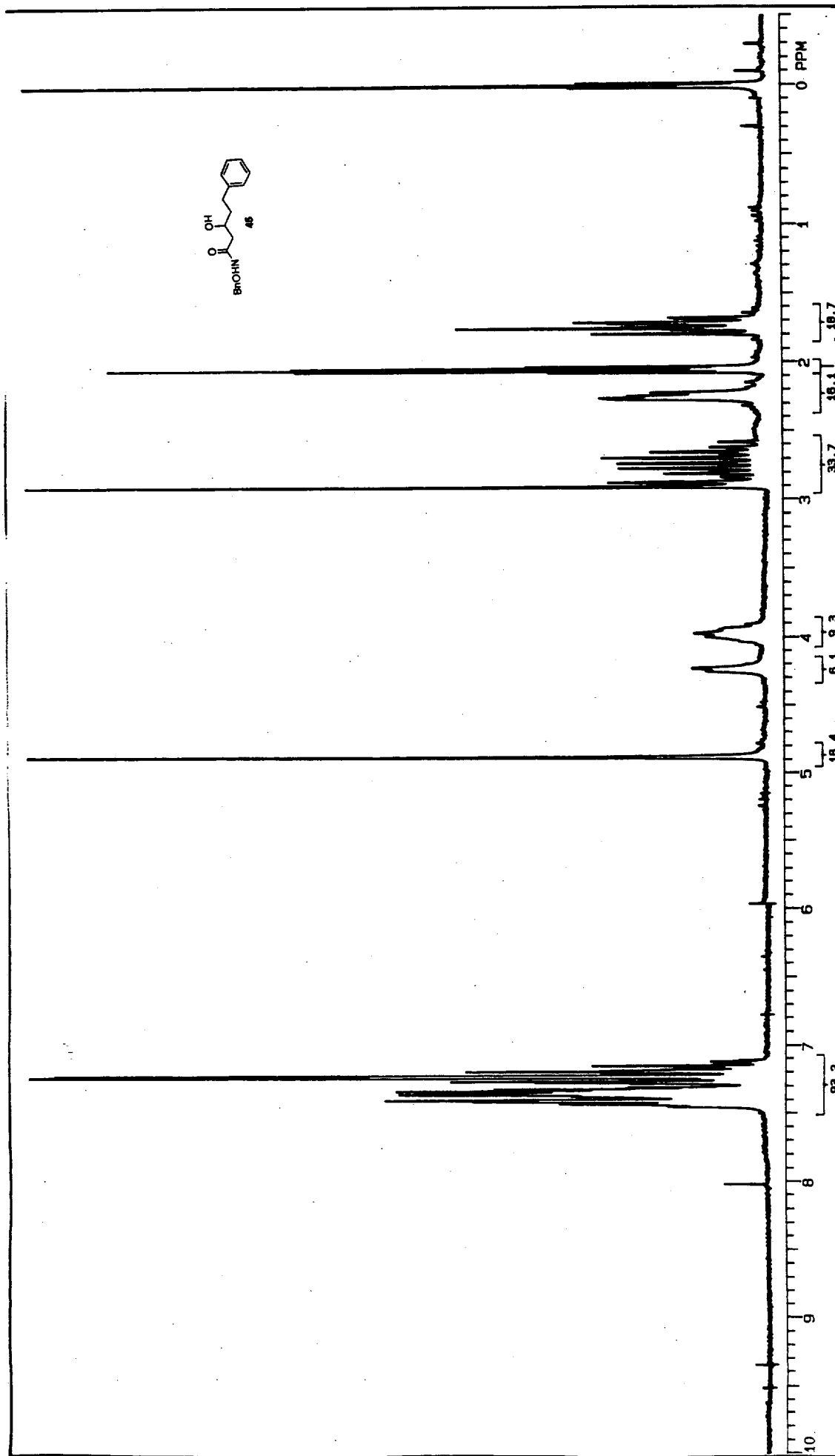
f

4.438

4.415

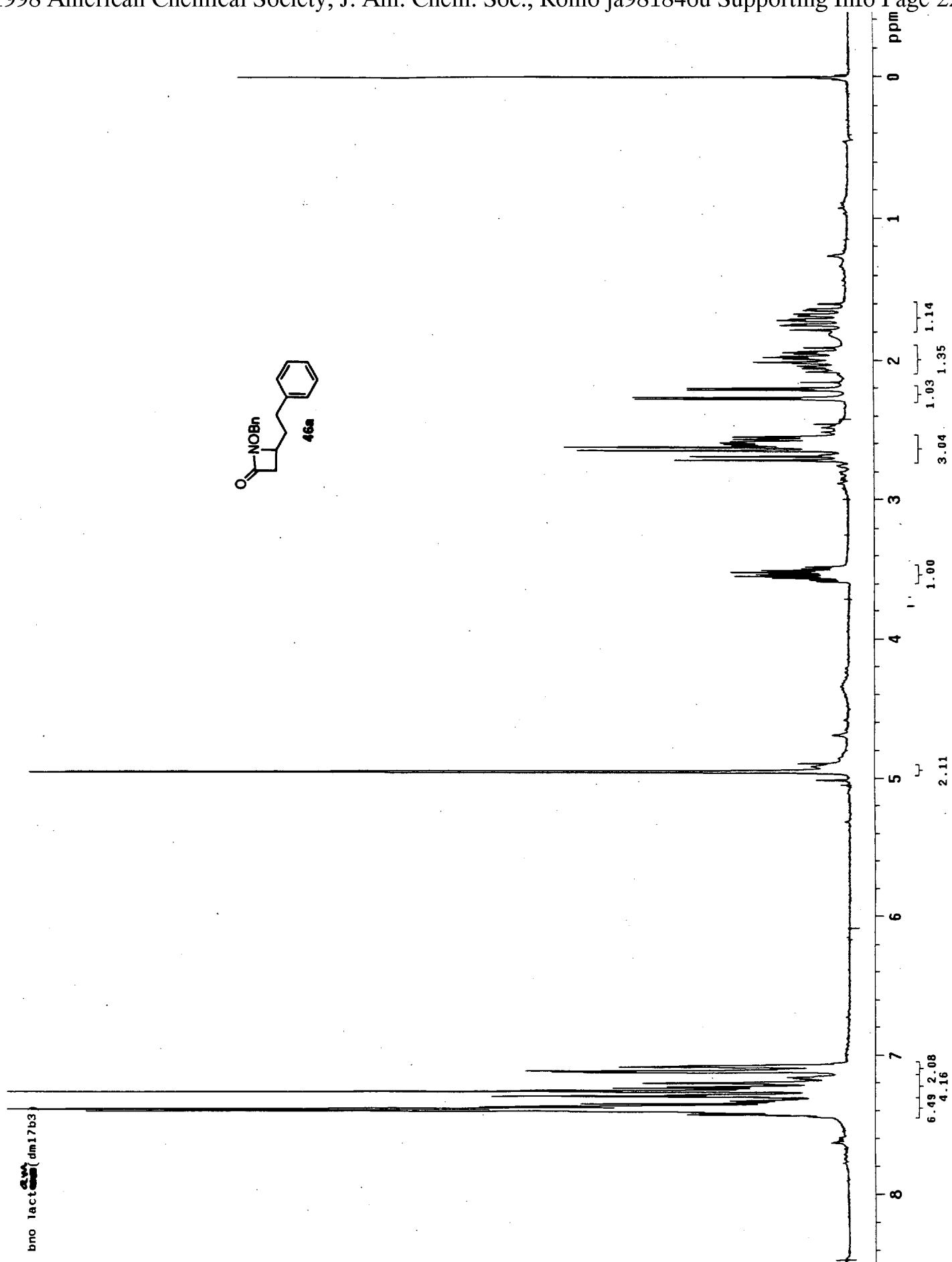
7.273



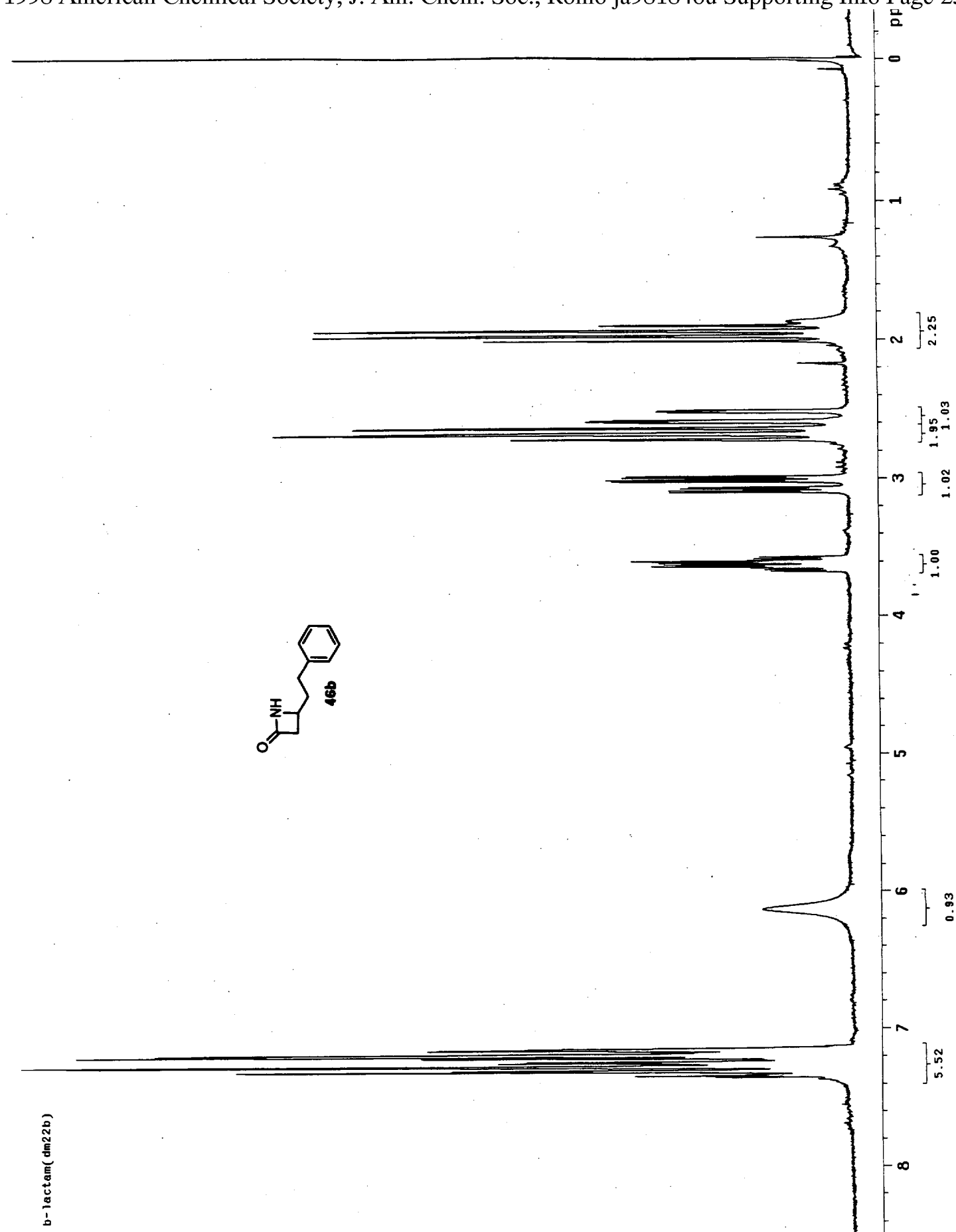
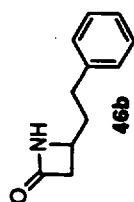


User: MILLER		File: H		Date: 07-13-98		XLA 200	
SAMPLE: DM158 RECRYSTALLIZED AMIDE				Pulse Sequence: STDH			
Probe: DUAL 5		Temp: °C		Solvent: ACETON			
EXPERIMENT				PLOT/PROCESSING			
FN: 32	K: K	RE: RE	sec: sec	CD: CD	sec: sec	Hz: Hz	sec: sec
LB: 2100.7	Hz: Hz	AF: AF	sec: sec	CD: CD	sec: sec	Hz: Hz	sec: sec
Width: 2100.7	Hz: Hz	Start: -100.0	Hz: Hz	Reference:			
Nucleus: 1.250		Offset: 209.3		Hz: Hz			
Mode: NNN		Power: 20		dB: dB			
Modulation Mode: C		Freq: 200		Hz: Hz			
Pulse Width: 5.8		µsec: µsec		Power Mode: ---			
Nucleus: 1.250		Freq: 200		MHz: MHz			
Spec. Width: 2901.1		Hz: Hz		Offset: 400			
Acq. Time: 5.173		sec: sec		Delay: 0.350			
Pulse Width: 5.8		µsec: µsec		Transients: 64			
OBSERVE							

522



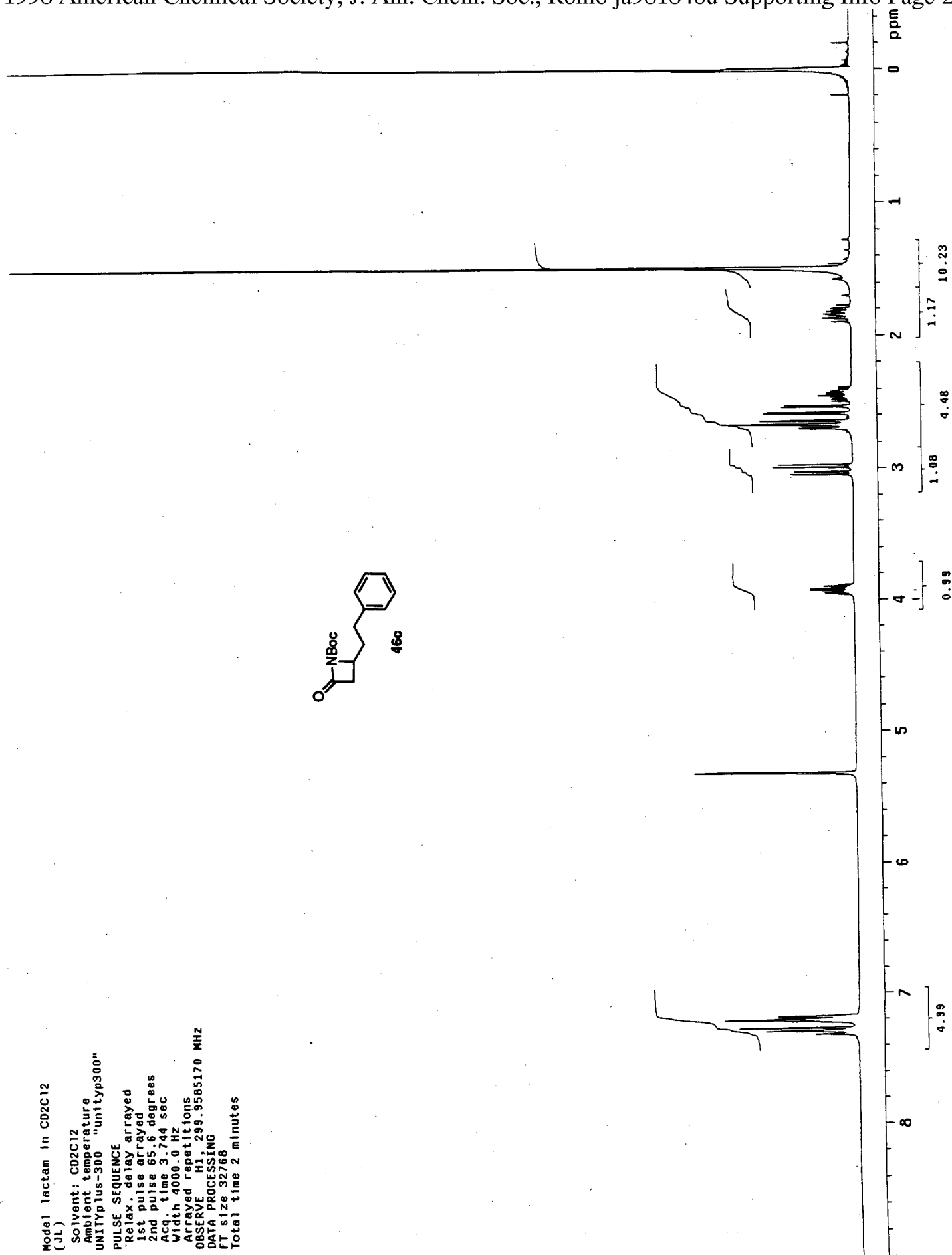
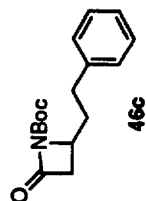
b-lactam(dm22b)

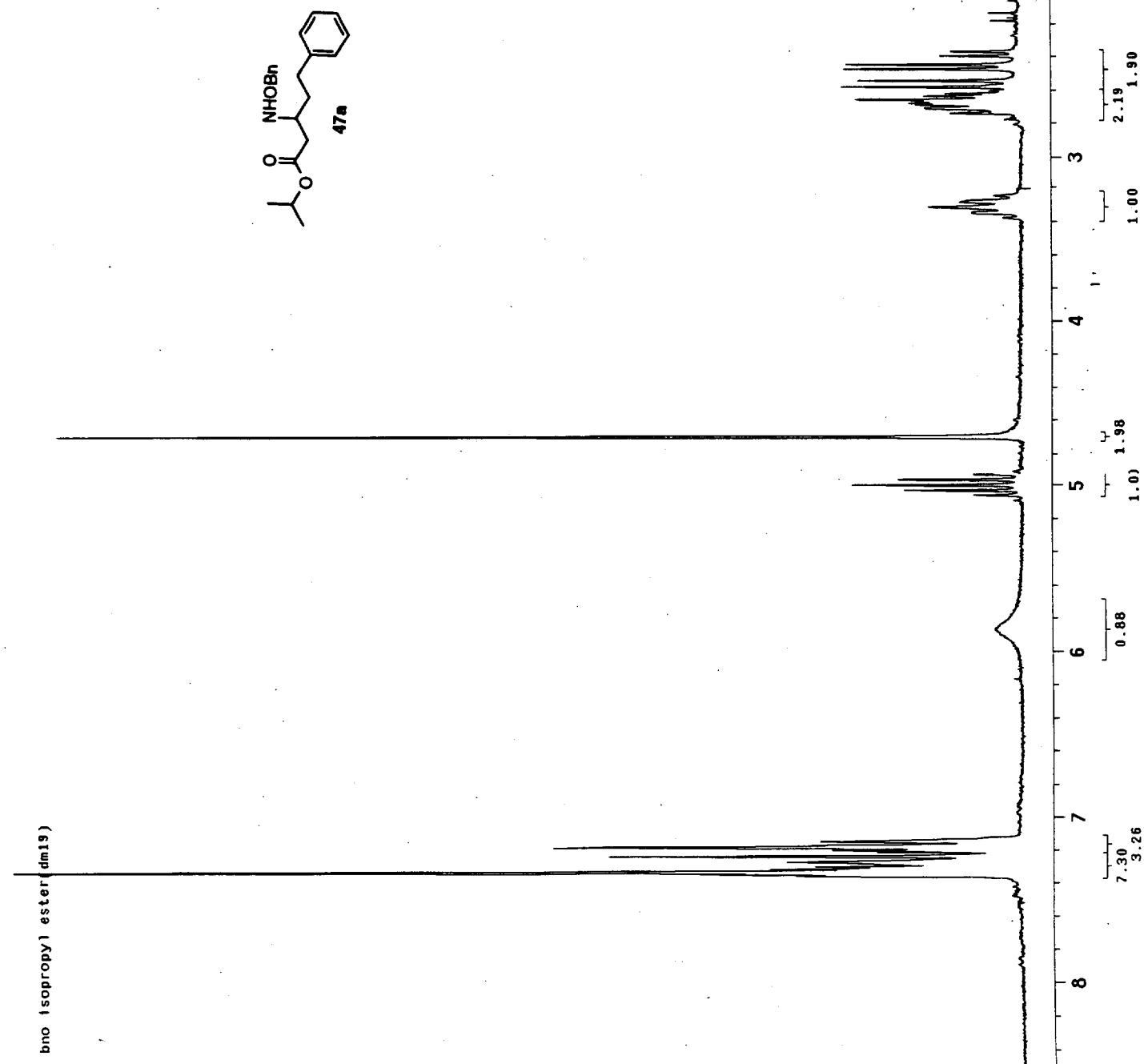


A

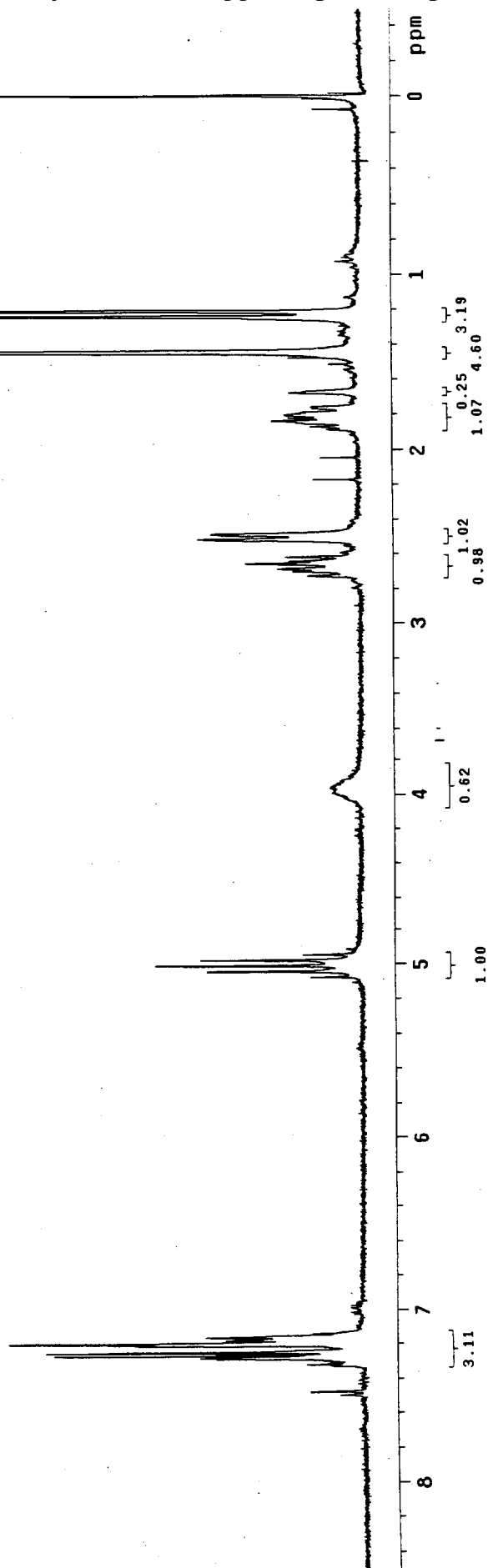
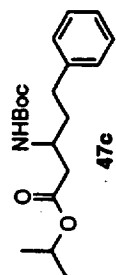
hes

Model lactam in CD2Cl2
(JL)
Solvent: CD2Cl2
Ambient temperature
UNITYplus-300 "unityp300"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 65.6 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
Arrayed repetitions
OBSERVE H1, 299.9585170 MHz
DATA PROCESSING
FT size 32768
Total time 2 minutes

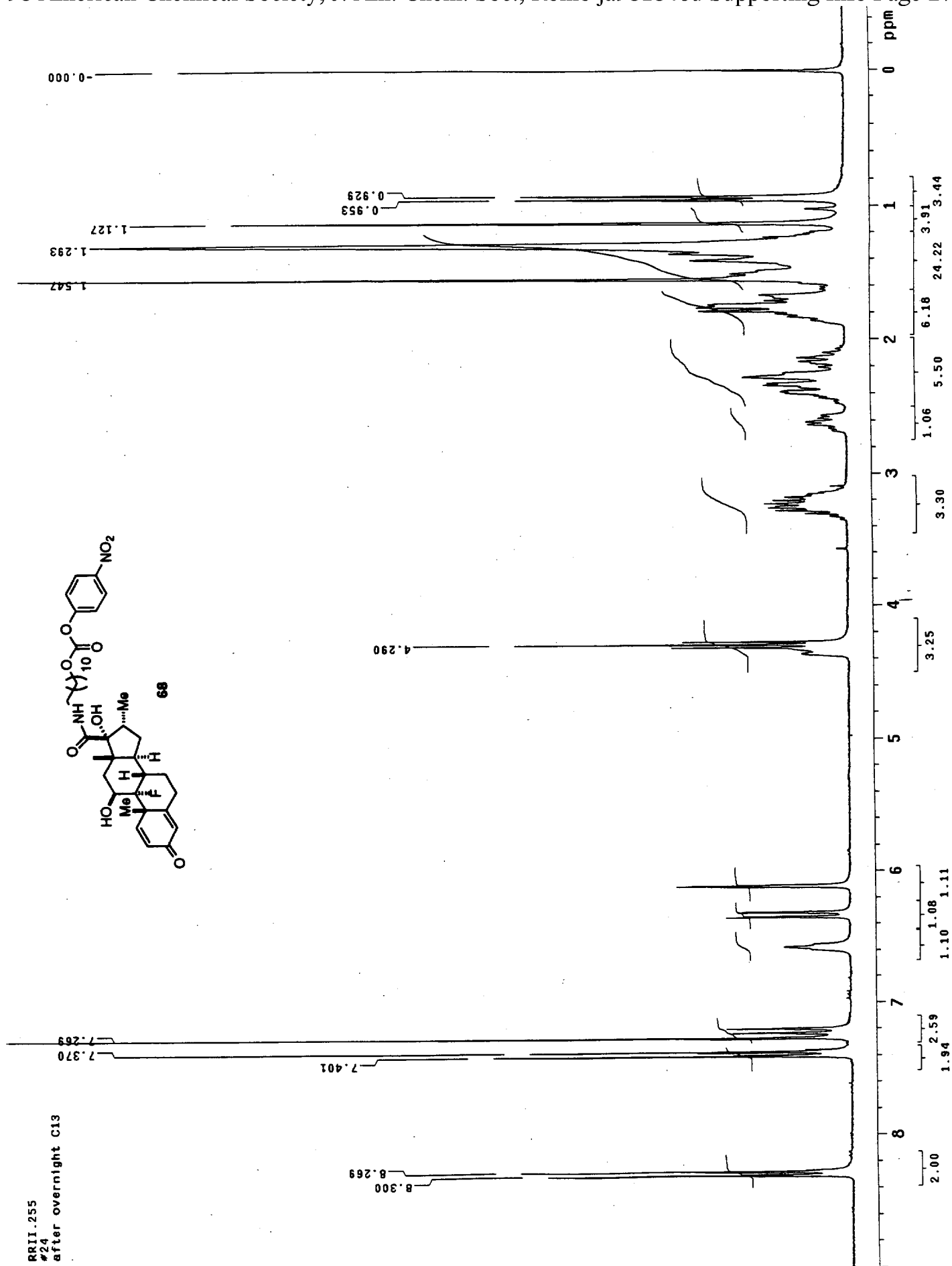




boc isopropyl ester (dm26)



tes



69

