

## Supporting Information for:

# Efficient Synthesis of 2-Deoxy L-Ribose from L-Arabinose: Mechanistic Information on the 1,2-Acyloxy Shift in Alkyl Radicals

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**1,2-O-[Ethylthio(4-methylphenyl)methylidene]-3,5-di-O-p-toluoyl- $\alpha$ -L-arabinofuranose (7).** The arabinofuranosyl bromide **2a** (1.14 g, 2.0 mmol) was dissolved in 8 mL of dry nitromethane. Collidine (352  $\mu$ L, 2.4 mmol) and ethanethiol (237  $\mu$ L, 2.4 mmol) were added, and the mixture was stirred at 25 °C. After it stirred for 2 h, the reaction was diluted in 150 mL of dichloromethane and was extracted with ice water (3 x 60 mL). The organic layer was then washed with brine, dried over magnesium sulfate and rotoevaporated to give a white solid residue (1.1 g, 100%). Recrystallization in ether and hexanes then gave the pure product **7** (0.95 g, 86%). mp 74 – 76 °C.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 200 MHz)  $\delta$ : 7.93 (d, 2H,  $J = 8.2$  Hz), 7.91 (d, 2H,  $J = 8.2$  Hz), 7.57 (d, 2H,  $J = 8.1$  Hz), 7.15 – 7.30 (m, 6H), 6.35 (d, 1H,  $J = 4.1$  Hz,  $\text{H}_1$ ), 5.56 (s, 1H,  $\text{H}_3$ ), 5.17 (d, 1H,  $J = 4.2$  Hz,  $\text{H}_2$ ), 4.61 (t, 1H,  $J = 7.1$  Hz,  $\text{H}_4$ ), 4.22 (d, 2H,  $J = 7.2$  Hz,  $\text{H}_5$ ), 2.42 (s, 3H, Me), 2.39 (s, 3H, Me), 2.37 (q, 2H,  $J = 7.4$  Hz), 2.36 (s, 3H, Me), 1.08 (t, 3H,  $J = 7.4$  Hz).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 50 MHz)  $\delta$ : 165.89, 165.35, 144.49, 143.70, 138.97, 136.14, 129.92, 129.84, 129.26, 129.06, 128.79, 127.06, 126.34, 125.81, 119.95, 107.28, 85.22, 84.70, 77.61, 63.60, 24.69, 21.74, 21.67, 21.29, 14.47. IR (KBr): 2967 (w), 1725 (s), 1613 (m), 1271 (s), 1103 (s), 1071 (m), 752 (m)  $\text{cm}^{-1}$ . High-resolution MS

(CI,  $m/z$ ): 487.1761, calcd for  $C_{29}H_{27}O_7$  487.1757 (M – EtS)<sup>+</sup>.  $[\alpha]_D^{20} = +24.1$  (c = 1.3,  $CHCl_3$ ).

**2-Deoxy-1,3,5-tri-*O*-*p*-toluoyl- $\beta$ -L-erythro-pentofuranose (4).** A solution of 1,2-*O*-[ethylthio(4-methylphenyl)methylidene]-3,5-di-*O*-toluoyl- $\beta$ -L-arabinofuranose **7** (0.11 g, 0.2 mmol) and 2,4,6-collidine (50  $\mu$ L, to make a 0.5% solution) in 10 mL of dry toluene was vigorously stirred at 105 °C. AIBN (8.2 mg, 0.05 mmol) and tributylstannane (108  $\mu$ L, 0.4 mmol) in 10 mL of dry toluene was added via a syringe pump over 1 h. The solution was heated for another hour after the addition was complete. The toluene was rotoevaporated. The residue was dissolved in acetonitrile (30 mL), and the solution was extracted with pentane (3 x 20 mL) to remove the tin residue. The acetonitrile was then evaporated *in vacuo*. The residue was co-rotoevaporated with xylene and then chloroform to remove collidine. Recrystallization in ether and pentane furnished the product as thin needles (83 mg, 85%). mp 105 – 107 °C, (lit. 107 °C for D-isomer). <sup>1</sup>H NMR ( $CDCl_3$ , 200 MHz)  $\delta$ : 7.94 (d, 2H,  $J = 8.4$  Hz), 7.89 (d, 2H,  $J = 8.4$  Hz), 7.87 (d, 2H,  $J = 8.1$  Hz), 7.24 (d, 2H,  $J = 8.0$  Hz), 7.17 (d, 2H,  $J = 8.1$  Hz), 7.11 (d, 2H,  $J = 8.1$  Hz), 6.74 (dd, 1H,  $J = 5.6, 2.4$  Hz,  $H_1$ ), 5.73 (ddd, 1H,  $J = 7.1, 4.8, 2.8$  Hz,  $H_3$ ), 4.47 – 4.77 (m, 3H,  $H_4, H_5$ ), 2.85 (ddd, 1H,  $J = 14.4, 7.0, 2.4$  Hz,  $H_2$ ), 2.62 (ddd, 1H,  $J = 14.4, 5.4, 5.4$  Hz,  $H_2$ ), 2.41 (s, 3H,  $CH_3$ ), 2.39 (s, 3H,  $CH_3$ ), 2.36 (s, 3H,  $CH_3$ ). <sup>13</sup>C ( $CDCl_3$ ): 166.21, 166.09, 165.60, 144.29, 144.06, 143.74, 129.85, 129.82 (2 C's), 129.24, 129.16, 129.06, 126.96, 126.92, 126.67, 99.04, 82.94, 74.58, 64.43, 38.81, 21.72 (2 C's), 21.67. IR (KBr): 2965 (w), 1732 (s), 1721 (s), 1613 (m), 1269 (s), 1173 (m), 1100 (m), 961 (m), 750 (m)  $cm^{-1}$ . High-resolution MS (CI,  $m/z$ ): 353.1382, calcd for  $C_{21}H_{21}O_5$  353.1389 (M – MePhCO<sub>2</sub>)<sup>+</sup>.  $[\alpha]_D^{20} = +12.1$  (c = 1.0,  $CHCl_3$ ).





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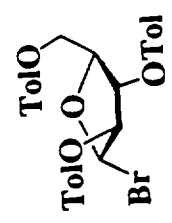
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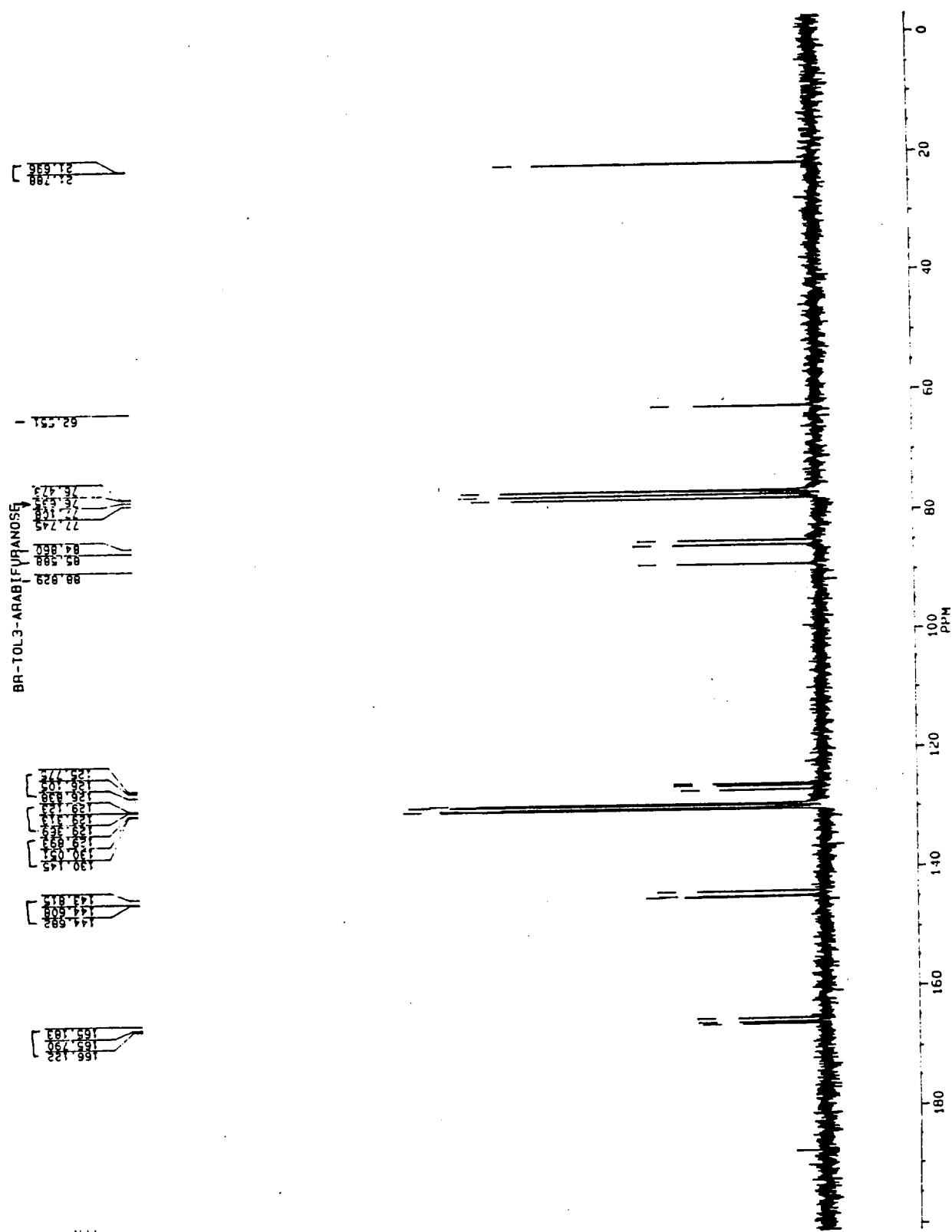
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2a





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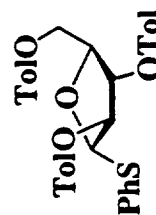
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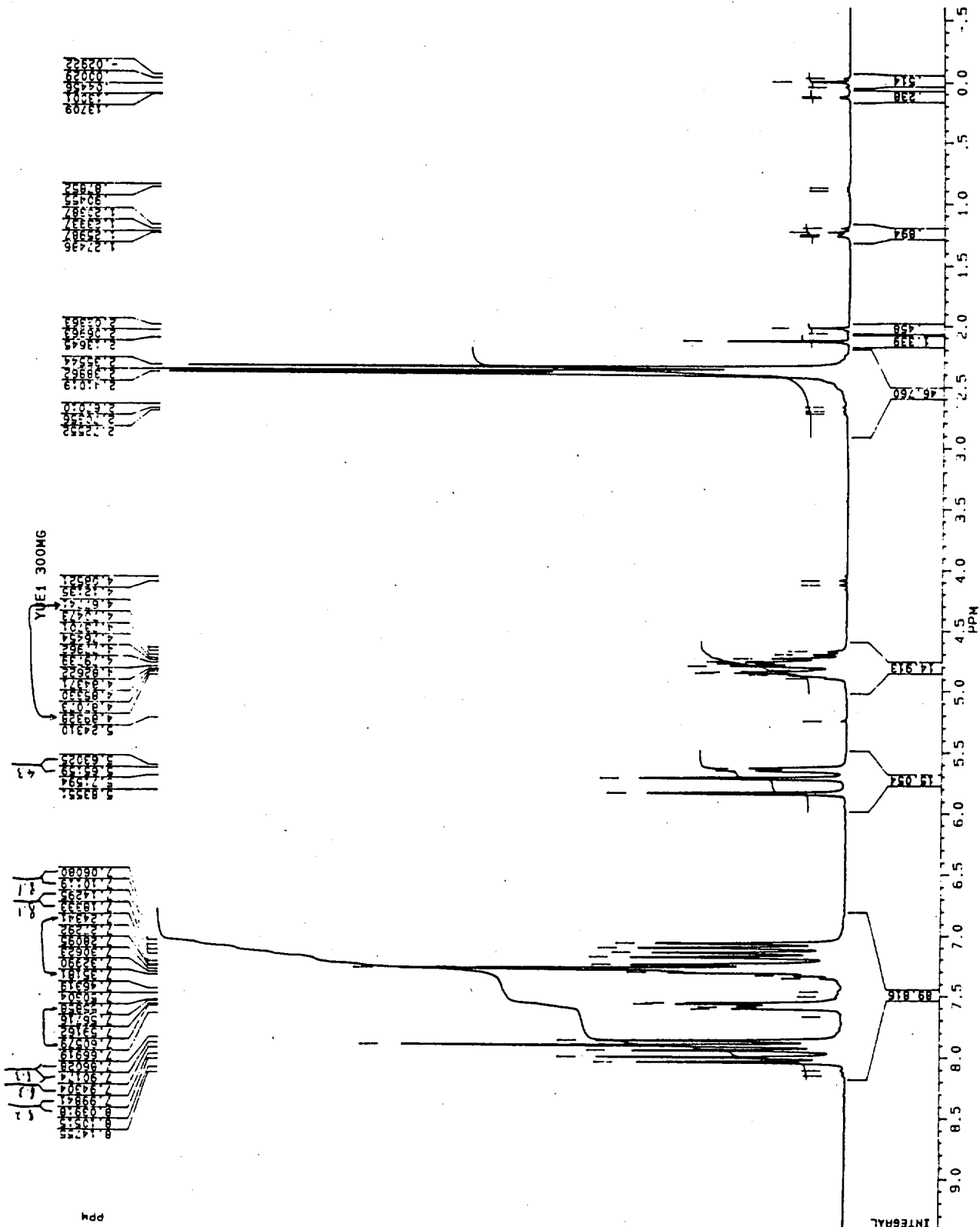
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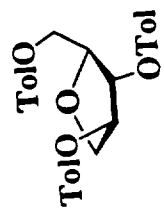
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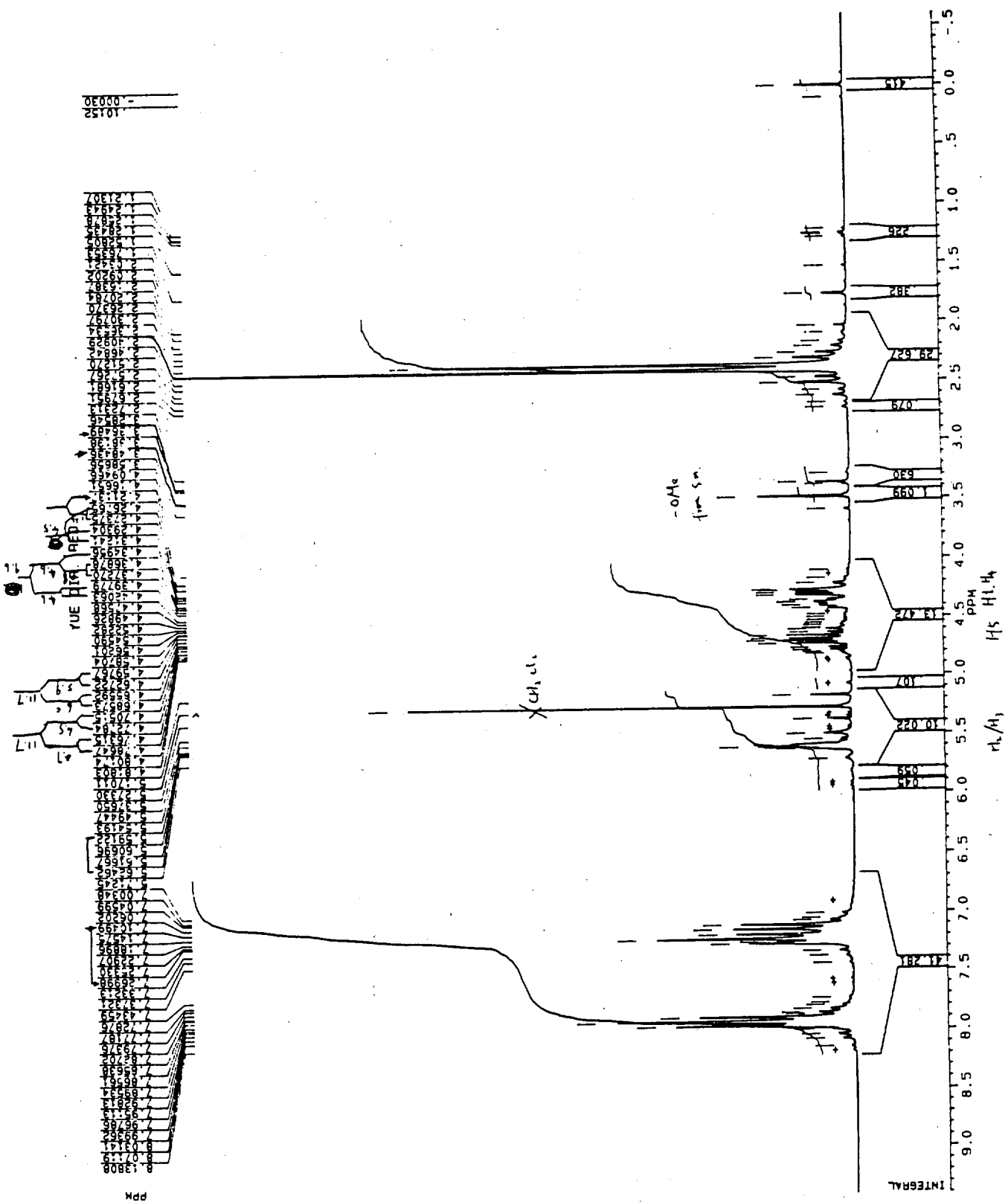
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 12766









J2420F.150  
 AU PR06:  
 X00-AU  
 DATE 25-7-98  
 TIME 23:05

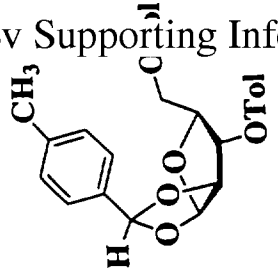
SF 200.132  
 O1 3545.000  
 SI 16384  
 TD 16384  
 SM 4032.258  
 HZ/PT .492

PM 0.0  
 RD 0.0  
 AO 2.032  
 RG 200  
 NS 128

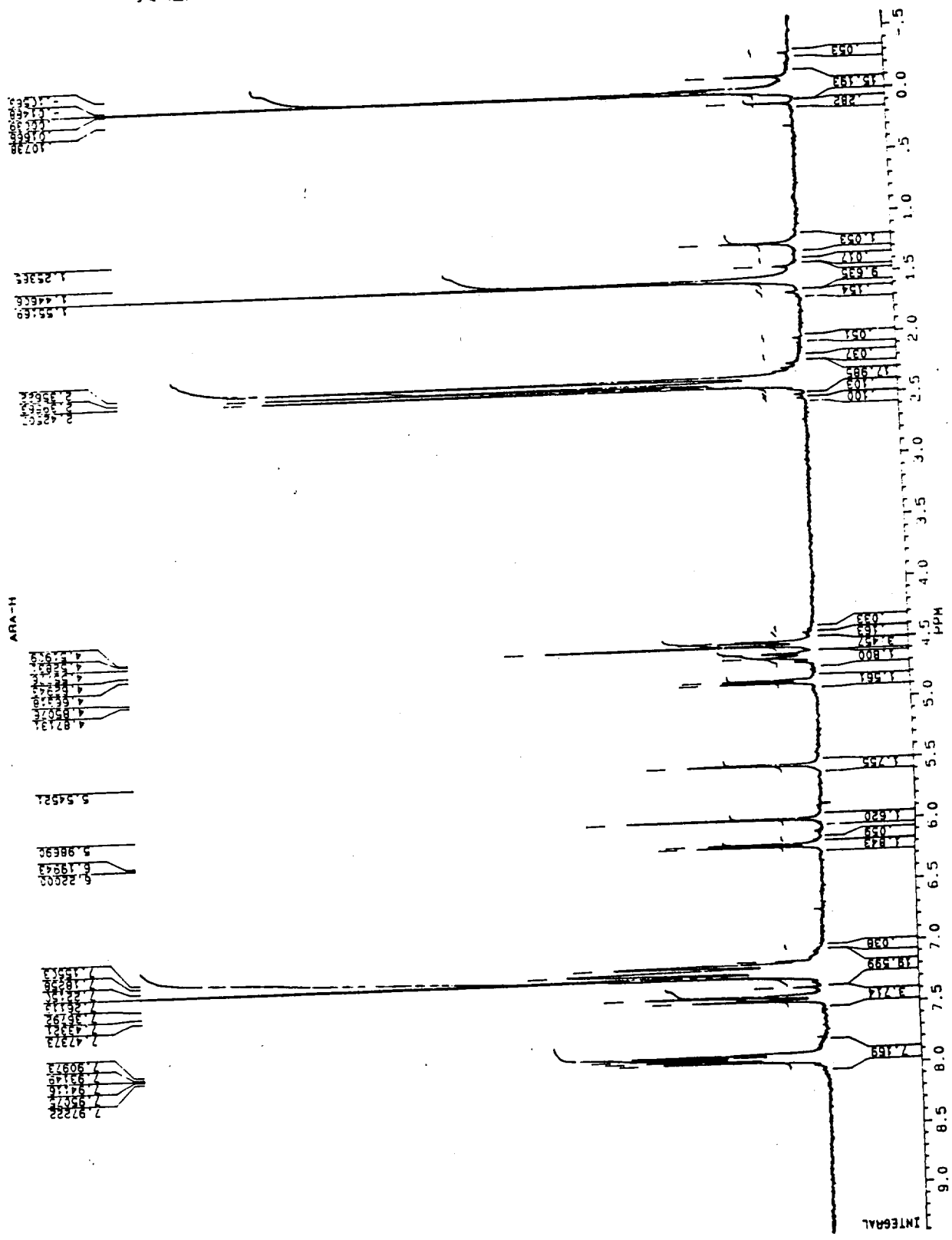
DE 155.0  
 DR 12  
 DM 124  
 FM 5100  
 O2 0.0  
 DP 63L P0

LB .300  
 NC 4  
 CX 32.00  
 CY 18.00  
 F1 9.403P  
 F2 -.598P  
 MI 0.0  
 HZ/CM 62.543  
 PPM/CM 2.313  
 IS 2340.54  
 SR

D1 1.0000000  
 P0 2.4  
 RGA 0.0  
 RD 0.0  
 PM 155.0  
 DE 128  
 DS 0



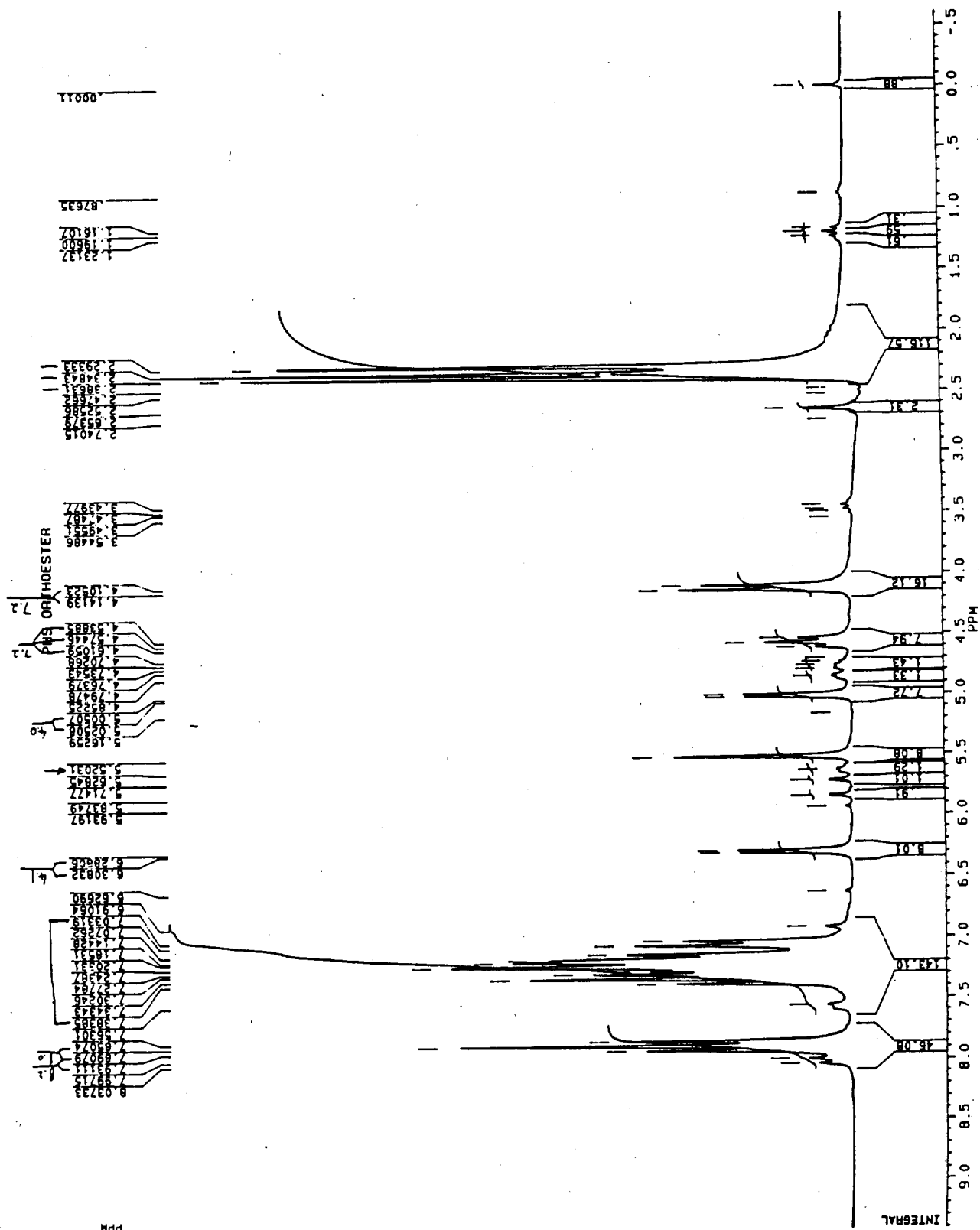
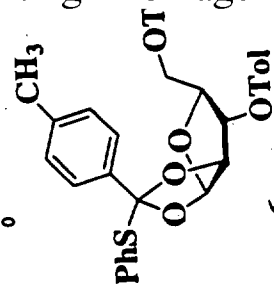
5







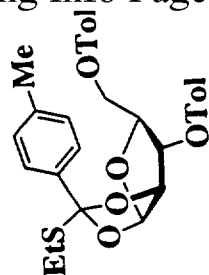
F2320F.141  
 AU PROG:  
 X00 AU  
 DATE 23-2-98  
 TIME 21:30  
 SF 200.132  
 O1 3545.000  
 SI 16384  
 TD 16384  
 SM 4032.258  
 HZ/PT .492  
 PM 0.0  
 RD 0.0  
 AG 2.032  
 RG 4  
 NS 16  
 DE 155.0  
 DR 12  
 DM 124  
 FW 5100  
 O2 0.0  
 DP 63L P0  
 LB .300  
 NC -1  
 CX 32.00  
 CY 18.00  
 F1 9.403P  
 F2 -.598P  
 MI 0.0  
 HZ/CH 62.543  
 PPM/CH .313  
 IS 5  
 SR 2347.43  
 D1 1.0000000  
 P0 2.4  
 RBA 0.0  
 RD 0.0  
 PW 155.0  
 NS 16  
 DS 0



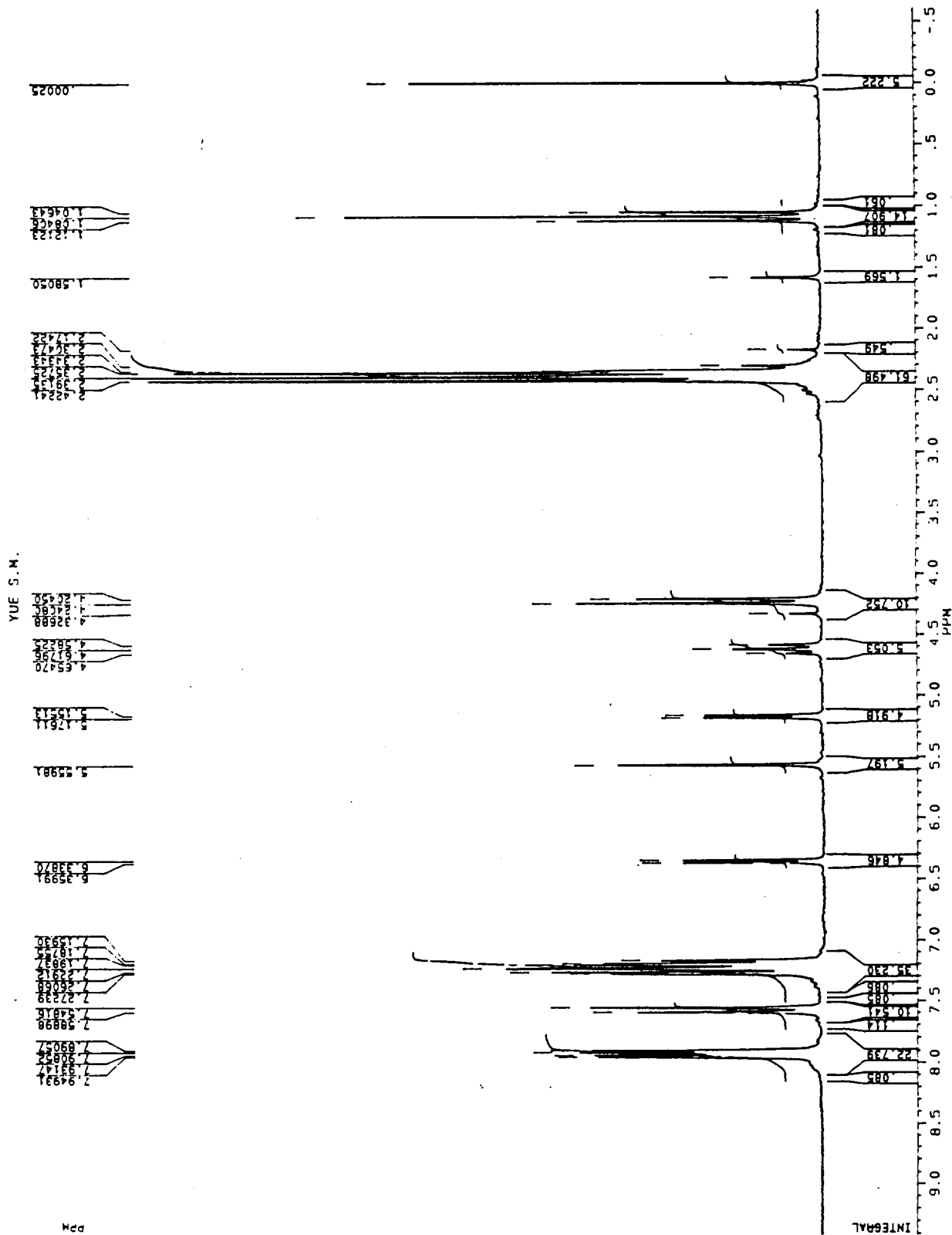
ppm



M1820F.134  
 AU PROG:  
 X00 AU  
 DATE 18-3-98  
 TIME 15:39  
 SF 200.132  
 O1 3545.000  
 SI 16384  
 TD 16384  
 SM 4032.258  
 HZ/PT .432  
 PW 0.0  
 RD 0.0  
 AQ 2.032  
 RG 40  
 NS 16  
 DE 155.0  
 DA 12  
 DM 124  
 FM 5100  
 O2 0.0  
 DP 63L P0  
 LB .300  
 NC 0  
 CX 32.00  
 CY 18.00  
 F1 9.403P  
 F2 .598P  
 M1 0.0  
 HZ/CH 62.543  
 PPM/CM .313  
 IS 3  
 SR 2341.03  
 D1 1.0000000  
 P0 2.4  
 RGA 0.0  
 RD 0.0  
 PW 0.0  
 DE 155.0  
 NS 16  
 DS 0



7





F1921F.106  
 AU PROG:  
 X02.AU  
 DATE 19-2-98  
 TIME 11:02

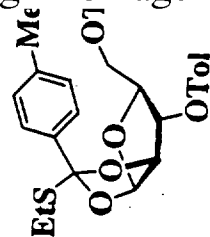
SF 50.323  
 O1 8349.000  
 SI 32768  
 TD 32768  
 SM 12500.000  
 HZ/PT .763

PW 0.0  
 RD 0.0  
 AO 1.311  
 RG 200  
 NS 512

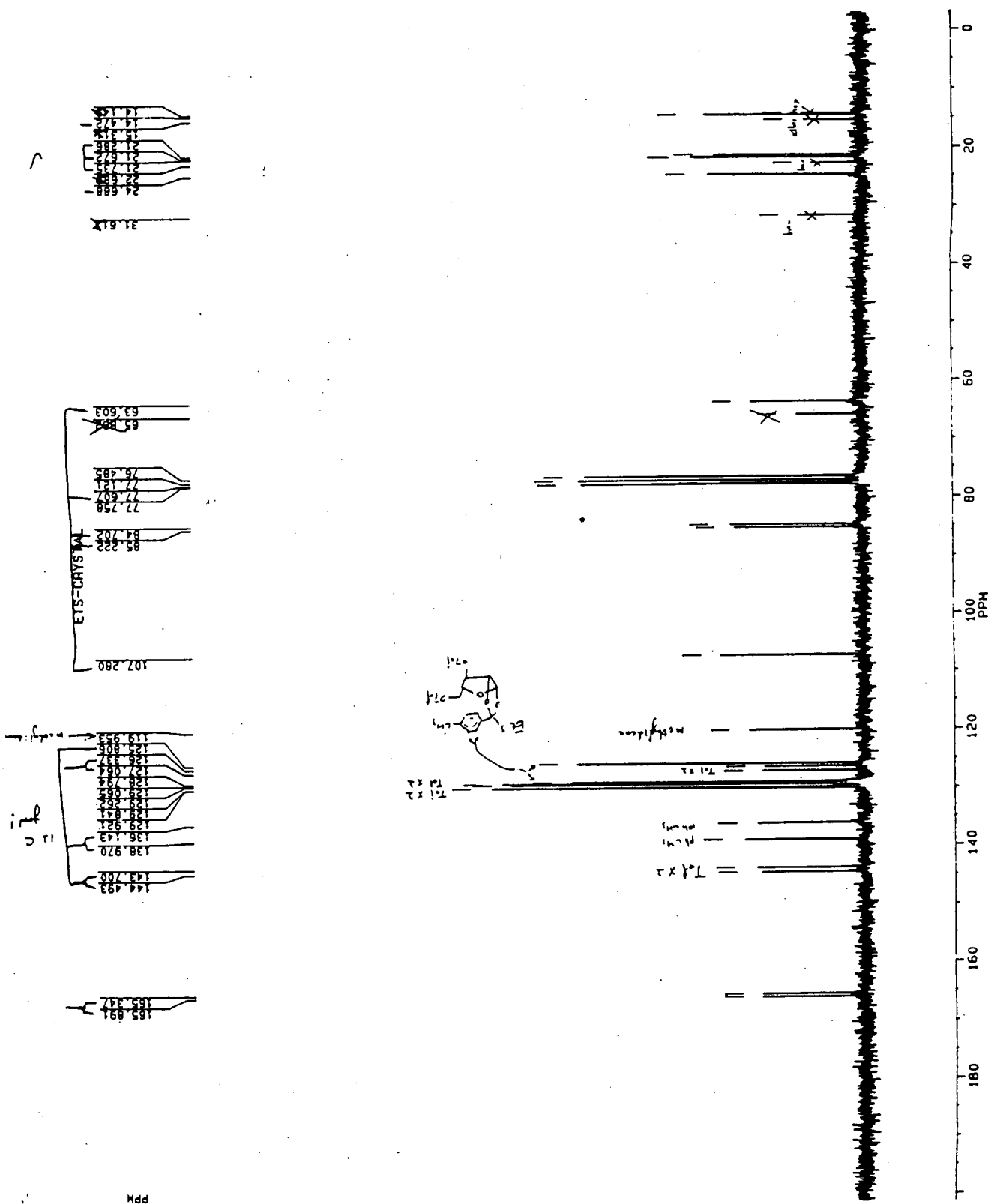
DE 50.0  
 DR 12  
 DW 40  
 FWH 15700  
 O2 3545.000  
 DP 17H 88

LB 1.000  
 NC 4  
 CX 32.00  
 CY 10.00  
 F1 201.034P  
 F2 -2.969P  
 W1 0.0  
 W2 0.0  
 HZ/CM 320.815  
 PPM/CM 6.375  
 IS 1  
 SR 3315.00

D1 2.0000000  
 S1 17H  
 D5 .0010000  
 P0 2.0  
 RGA 0.0  
 RD 0.0  
 PM 50.0  
 DE 512  
 NS 0  
 DS 0



7













A312IF.167  
 AU PROG:  
 X02.AU  
 DATE 31-8-98  
 TIME 23:52

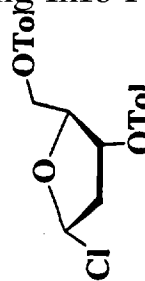
SF 50.323  
 O1 8349.000  
 SI 32768  
 TD 32768  
 SM 12500.000  
 HZ/PT .763

PM 0.0  
 RD 0.0  
 AG 1.311  
 R6 200  
 NS 512

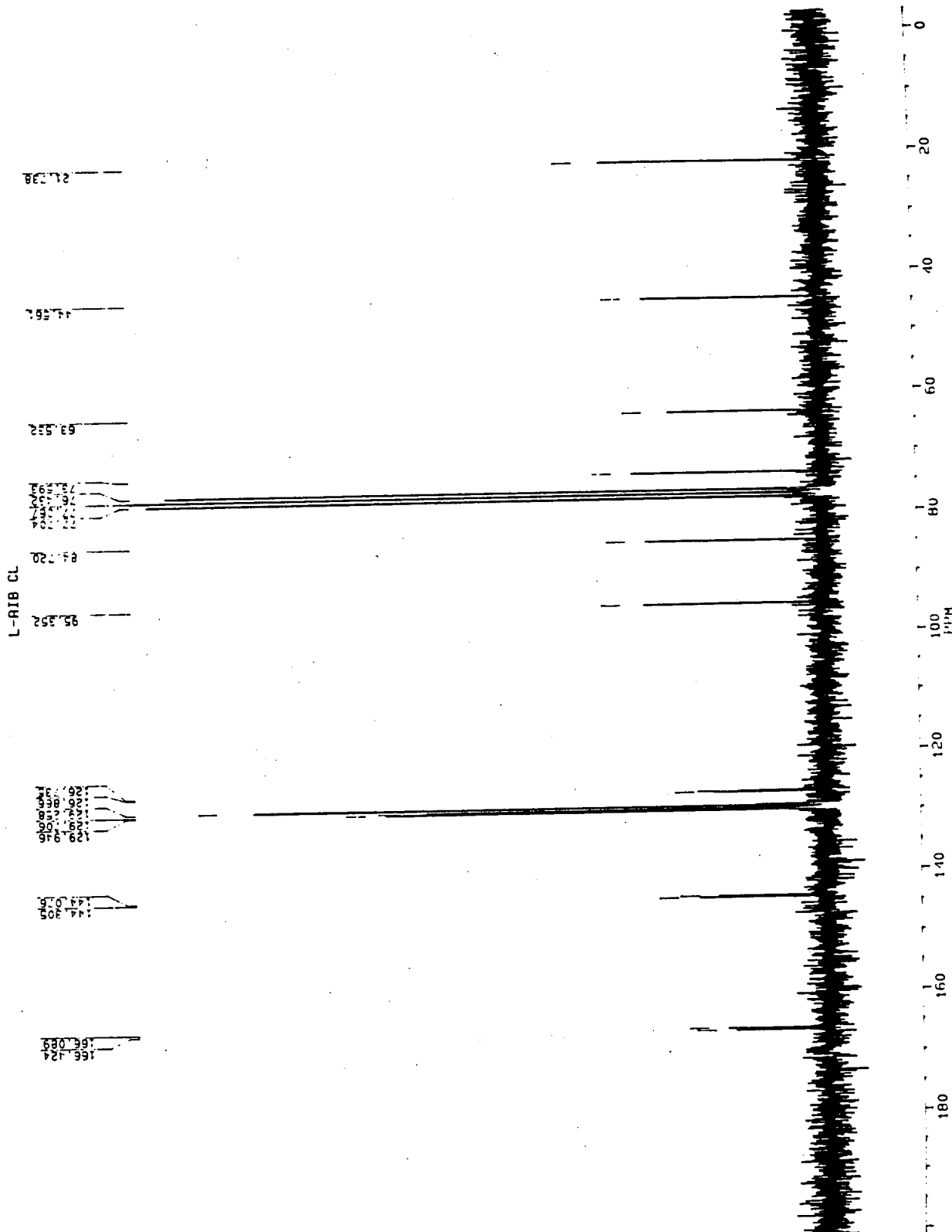
DE 50.0  
 DR 12  
 DM 40  
 FM 15700  
 O2 3545.000  
 DP 17H BB

LB 1.000  
 NC 3  
 CX 32.00  
 CY 18.00  
 F1 201.034P  
 F2 -2.969P  
 MI 0.0  
 HZ/CN 320.816  
 PPM/CM 6.375  
 IS 1  
 SR 3315.00

D1 2.0000000  
 S1 17H  
 D5 .0010000  
 P0 2.0  
 RGA 0.0  
 RD 0.0  
 PM 50.0  
 DE 512  
 DS

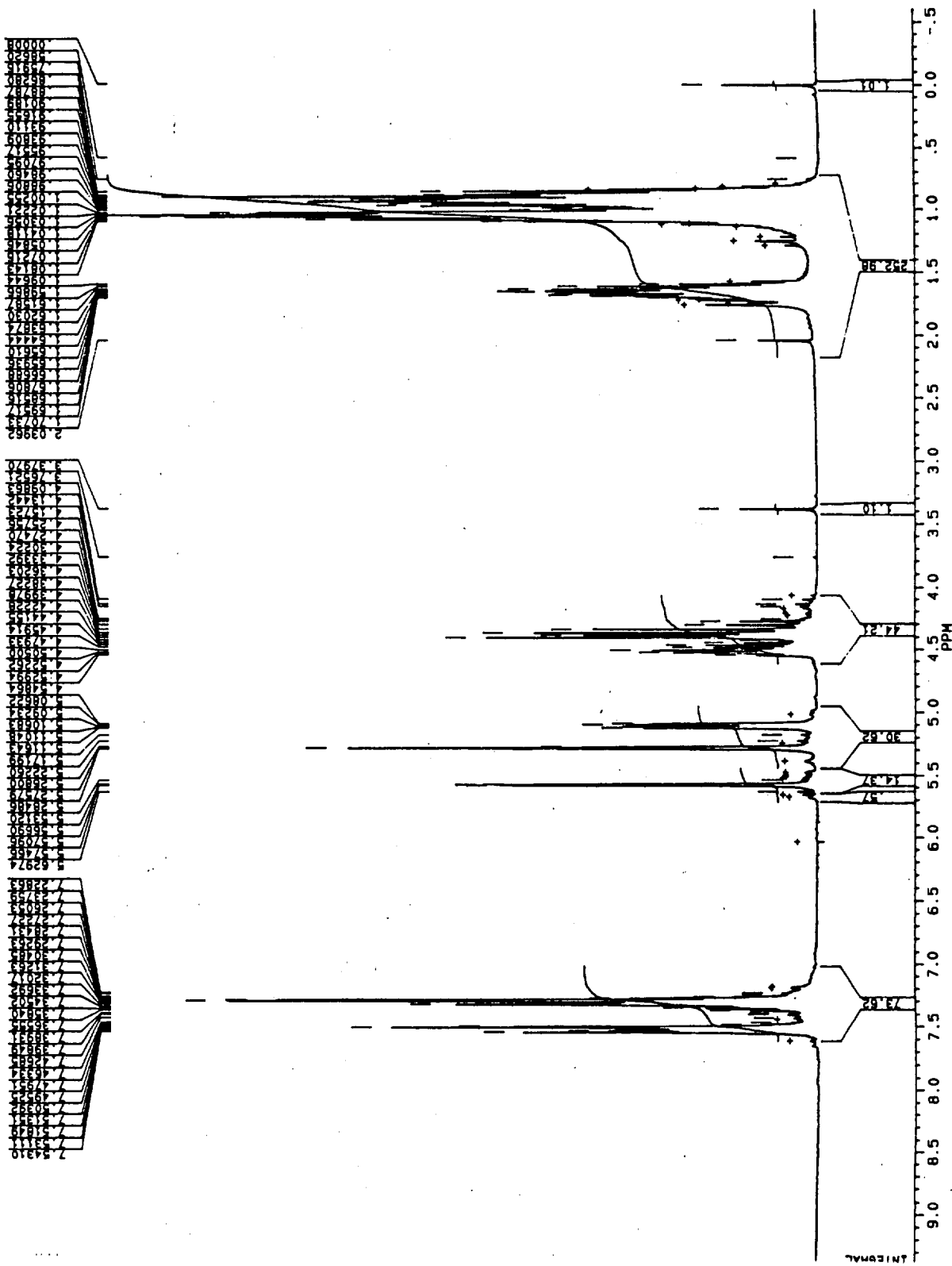


10

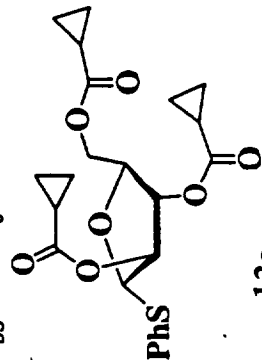


hcc

ARA C-PRO3 PHS



A0620F.146  
 AU PROG:  
 X00.AU  
 DATE 6-8-98  
 TIME 23:19  
 SF 200.132  
 O1 3545.000  
 SI 16384  
 TD 16384  
 SM 4032.258  
 HZ/PT .492  
 PM 0.0  
 RD 0.0  
 AD 2.032  
 RG 4  
 NS 16  
 DE 155.0  
 DR 12  
 DW 124  
 FV 5100  
 O2 0.0  
 DP 63L P0  
 LB .300  
 NC -2  
 CX 32.00  
 CY 18.00  
 F1 19.403P  
 F2 -.598P  
 MI 0.0  
 HZ/CM 62.543  
 PPM/CM 5.313  
 IS 2338.08  
 SR 2338.08  
 D1 1.0000000  
 P0 2.4  
 RGA 0.0  
 RD 0.0  
 PM 0.0  
 DE 155.0  
 NS 16  
 DS 0



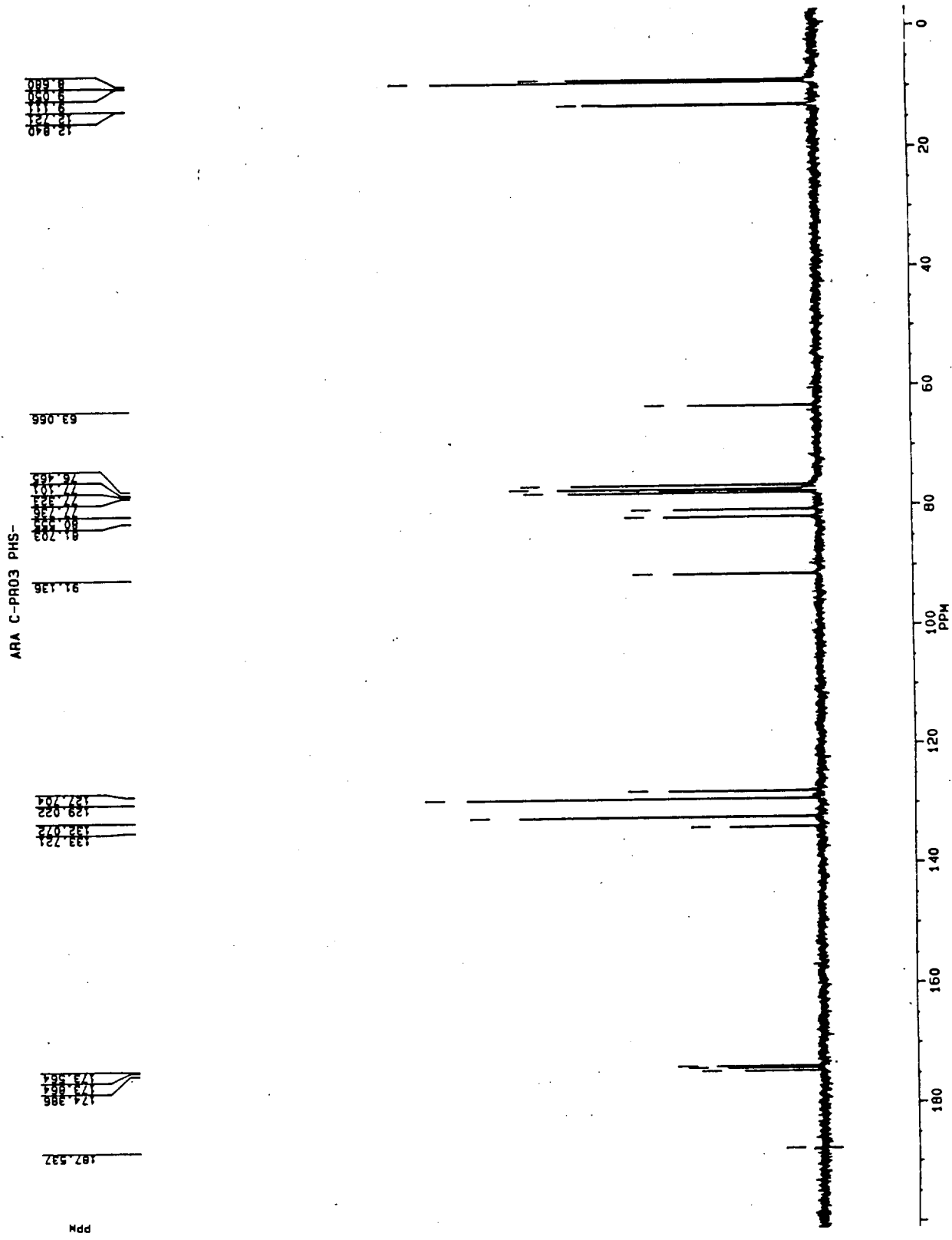
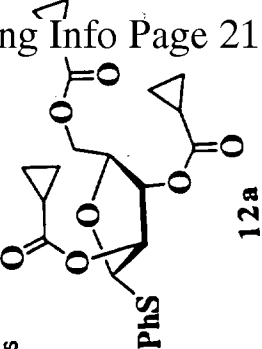
12a

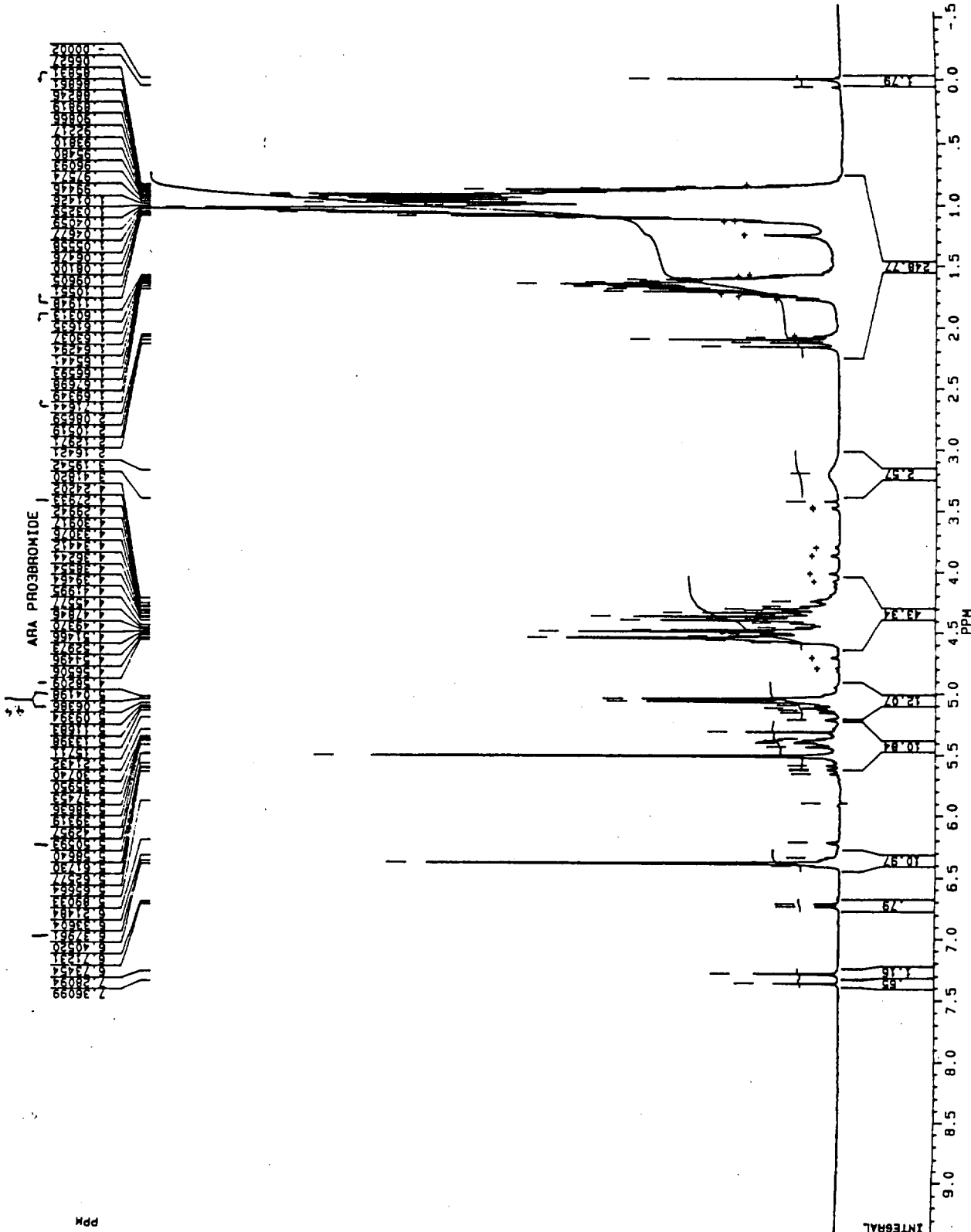


A0621F.146  
 AU PROG:  
 X02.AU  
 DATE 7-B-98  
 TIME 0:19  
 SF 50.323  
 O1 8349.000  
 S1 32768  
 TD 32768  
 SM 12500.000  
 HZ/PT .763

PM 0.0  
 RD 0.0  
 AD 1.311  
 RG 200  
 NS 1000  
 DE 50.0  
 DR 12  
 DW 40  
 FM 15700  
 O2 3545.000  
 DP 17H BB  
 LB 1.000  
 MC 5  
 CX 32.00  
 CY 10.00  
 F1 201.034P  
 F2 -2.969P  
 MI 0.0  
 HZ/CH 320.815  
 PPM/CH 6.375  
 IS 1  
 SR 3315.00

D1 2.0000000  
 S1 17H  
 D5 .0010000  
 P0 2.0  
 RGA 0.0  
 RD 0.0  
 PM 50.0  
 DE 1000  
 NS  
 DS





A1420F.143  
 AU PROG:  
 X00.AU  
 DATE 15-8-98  
 TIME 23:01

SF 200.132  
 D1 3545.000  
 S1 16384  
 TD 16384  
 SW 4032.258  
 HZ/PT .492

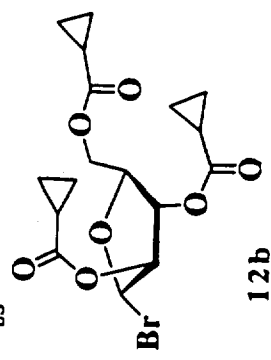
PW 0.0  
 RD 0.0  
 AG 2.032  
 RE 10  
 NS 16

DE 155.0  
 DR 12  
 DW 124  
 FV 5100  
 O2 0.0  
 DP 63L P0

LB .300  
 NC -1  
 CX 32.00  
 CY 18.00  
 F1 9.403P  
 F2 -.598P  
 MI 0.0

HZ/CM 62.543  
 PPM/CM 5.313  
 IS 2336.60  
 SR

D1 1.0000000  
 P0 2.4  
 RGA 0.0  
 RD 0.0  
 PW 155.0  
 DE 16  
 DS A



12b

ARA CPO3 BR

77.782  
77.656  
76.450  
76.238  
62.332

88.552  
84.992  
84.027

174.300  
174.015  
173.408



A142IF.143  
AU PROG:  
X02.AU  
DATE 16-8-98  
TIME 0:01

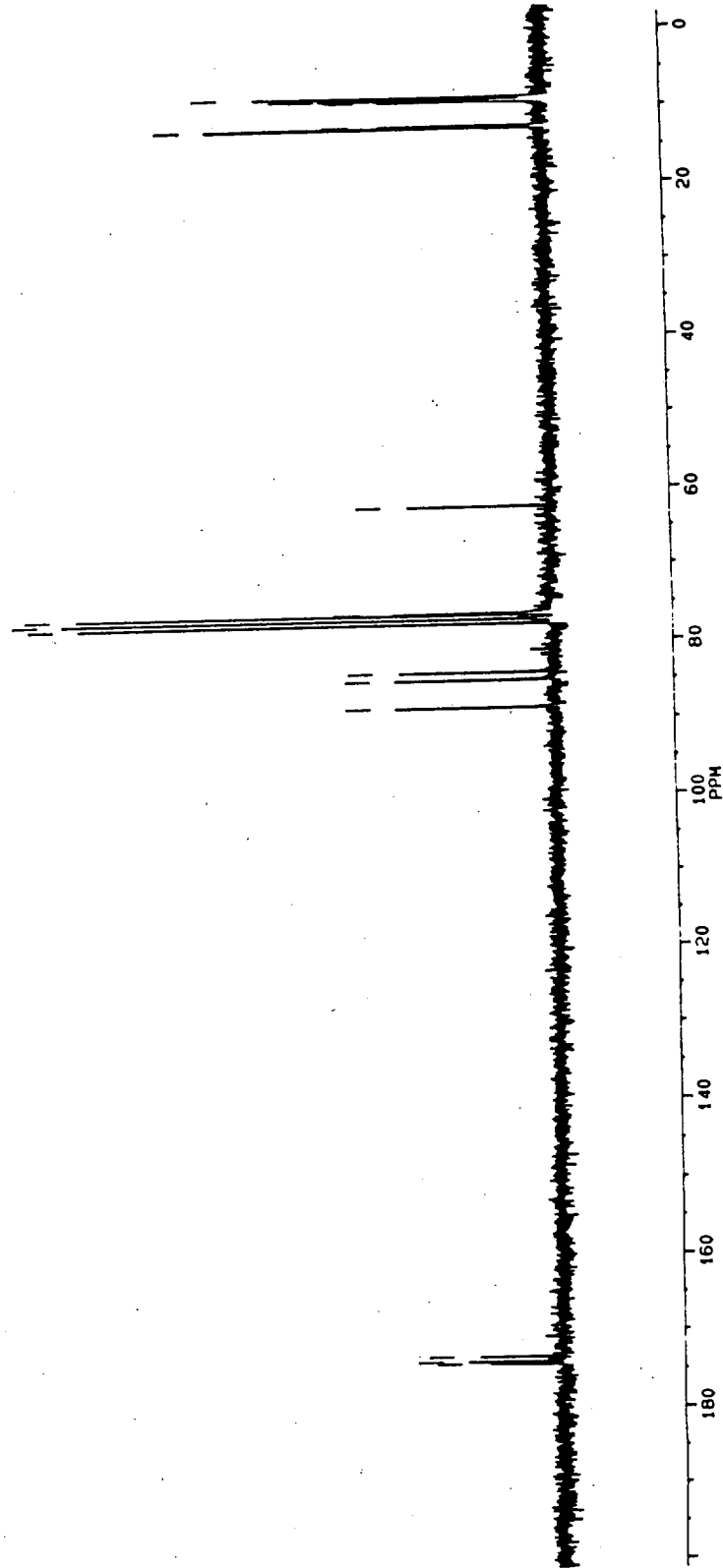
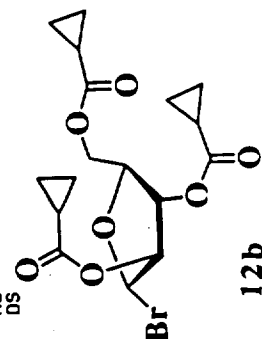
SF 50.323  
F1 6349.000  
SI 32768  
TO 32768  
SW 12500.000  
HZ/PI .763

PM 0.0  
RD 0.0  
AQ 1.311  
RG 200  
NS 1024

DE 50.0  
DM 12  
DH 40  
FM 15700  
O2 3545.000  
DP 17H.88

LB 1.000  
LC 4  
NC 32.00  
CX 10.00  
CY 201.034P  
F2 -2.969P  
MI 0.0  
HZ/CM 320.816  
PPM/CH 6.375  
IS 1  
SR 3315.00

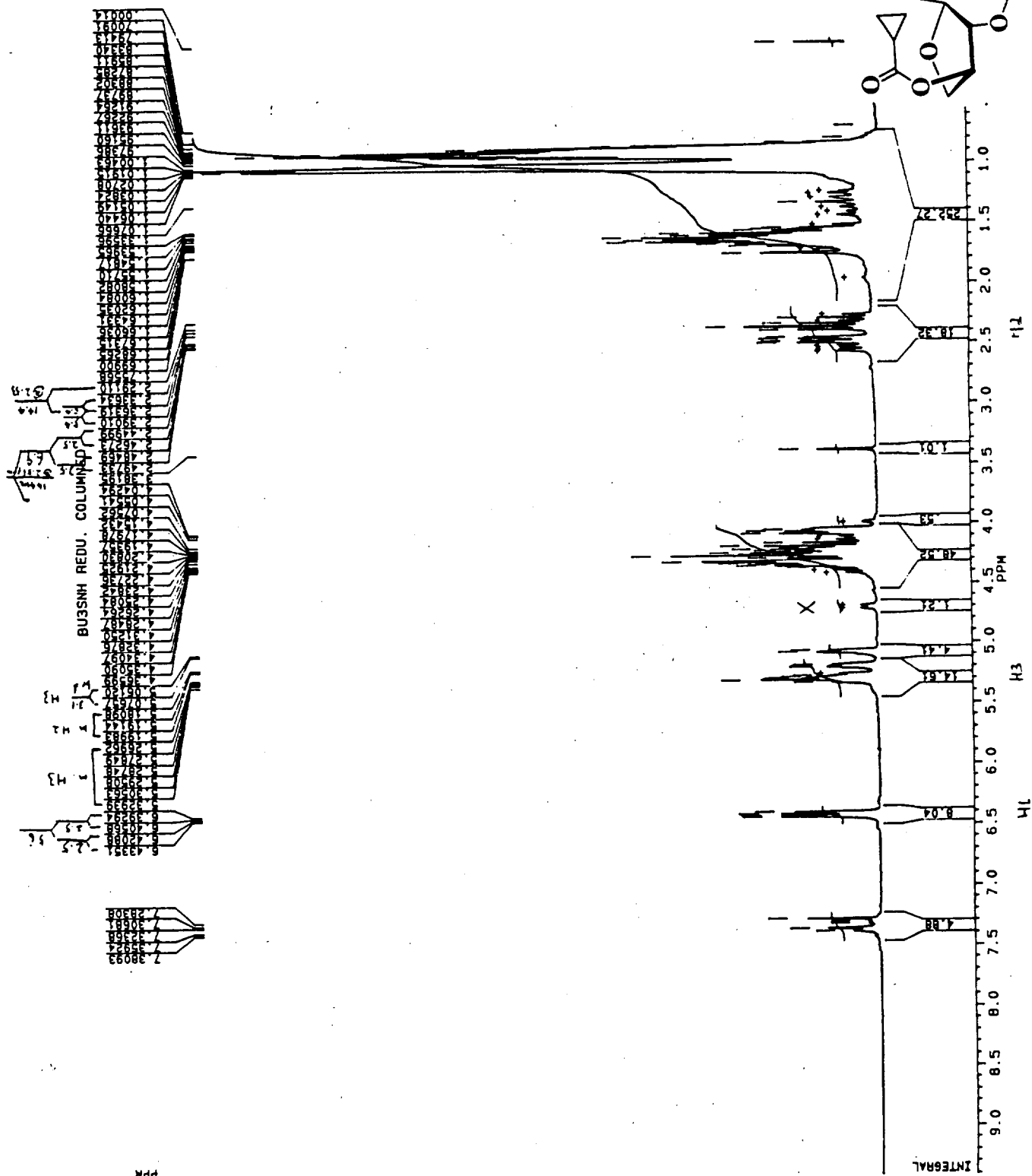
D1 2.0000000  
S1 17H  
D5 .0010000  
P0 2.0  
RGA 0.0  
RD 0.0  
PH 50.0  
DE 102.4  
DS



ppm



A0820F .113  
 AU PROG: X00 .AU  
 DATE: 8-8-98  
 TIME: 21:23  
 SF: 200.132  
 O1: 3545.000  
 SI: 16384  
 TO: 16384  
 SM: 4032.258  
 HZ/PT: 492  
 PW: 0.0  
 RD: 0.0  
 AQ: 2.032  
 RG: 10  
 NS: 16  
 DE: 155.0  
 DR: 12  
 DM: 124  
 FM: 5100  
 O2: 0.0  
 DP: 63L P0  
 LB: .300  
 NC: -1  
 CX: 32.00  
 CY: 18.00  
 F1: 9.403P  
 F2: -.598P  
 M1: 0.0  
 HZ/CM: 62.543  
 PPM/CM: .313  
 IS: 5  
 SR: 2336.11  
 D1: 1.0000000  
 R0: 2.4  
 RGA: 0.0  
 RD: 0.0  
 PM: 155.0  
 DE: 16  
 NS: 0  
 DS: 0



13/14

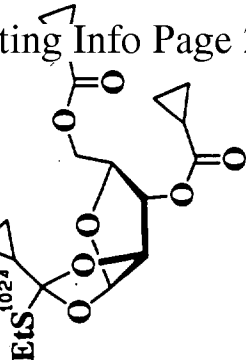




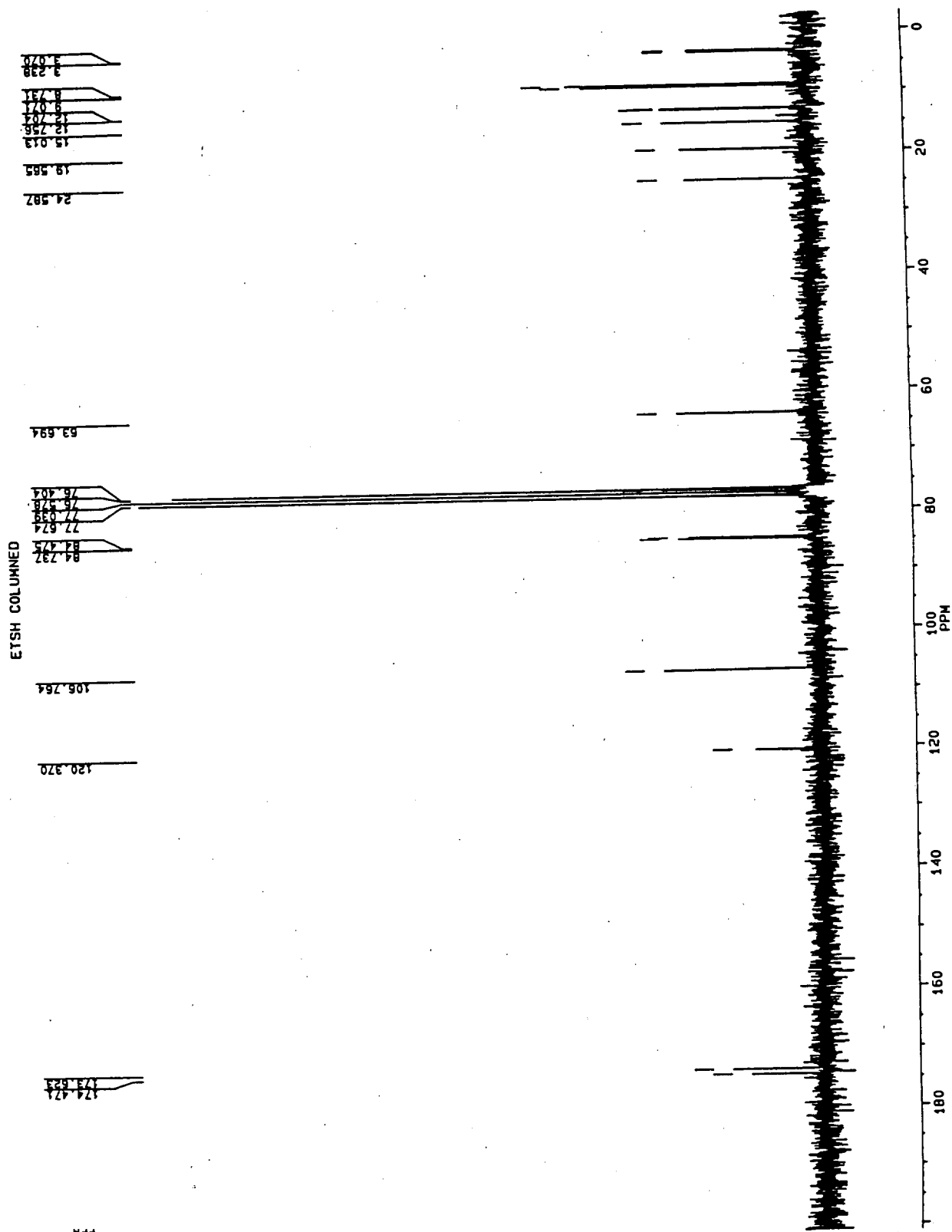




A172IF.147  
 AU PROG:  
 X02.AU  
 DATE 17-8-98  
 TIME 21:22  
 SF 50.323  
 O1 8349.000  
 SI 32768  
 TD 32768  
 SM 12500.000  
 HZ/PT .763  
 PW 0.0  
 RD 0.0  
 AQ 1.311  
 RG 200  
 NS 1024  
 DE 50.0  
 DR 12  
 DM 40  
 FM 15700.000  
 D2 3545.000  
 DP 17H BB  
 LB 1.000  
 NC 4  
 CY 32.00  
 CY 18.00  
 F1 201.034P  
 F2 -2.969P  
 MI 0.0  
 HZ/CH 320.816  
 PPM/CM 6.375  
 IS 1  
 SR 3315.00  
 D1 2.0000000  
 S1 17H  
 D5 .0010000  
 P0 2.0  
 RGA 0.0 0.0  
 RD 50 0.0  
 PM 102.4  
 DE 50  
 NS 102.4  
 D'



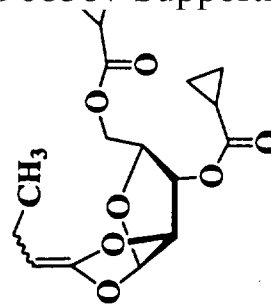
15



ppm



TL:001  
 DATE 19-8-98  
 TIME 17:03  
 SF 90.556  
 O1 6147.000  
 S1 65536  
 TD 65536  
 SW 22727.273  
 HZ/PT .694  
 PM 2.5  
 RD 2.000  
 AQ 1.442  
 RG 1600  
 NS 256  
 DE 30.0  
 OR 16  
 OW 22  
 FM 28500  
 OZ 6148.100  
 DP 16H CPD  
 LB 1.000  
 NC 8  
 CX 32.00  
 CY 8.00  
 F1 199.998P  
 F2 .002P  
 M1 .01  
 HZ/CH 565.962  
 PPM/CH 6.250  
 IS 3  
 SR -4239.60

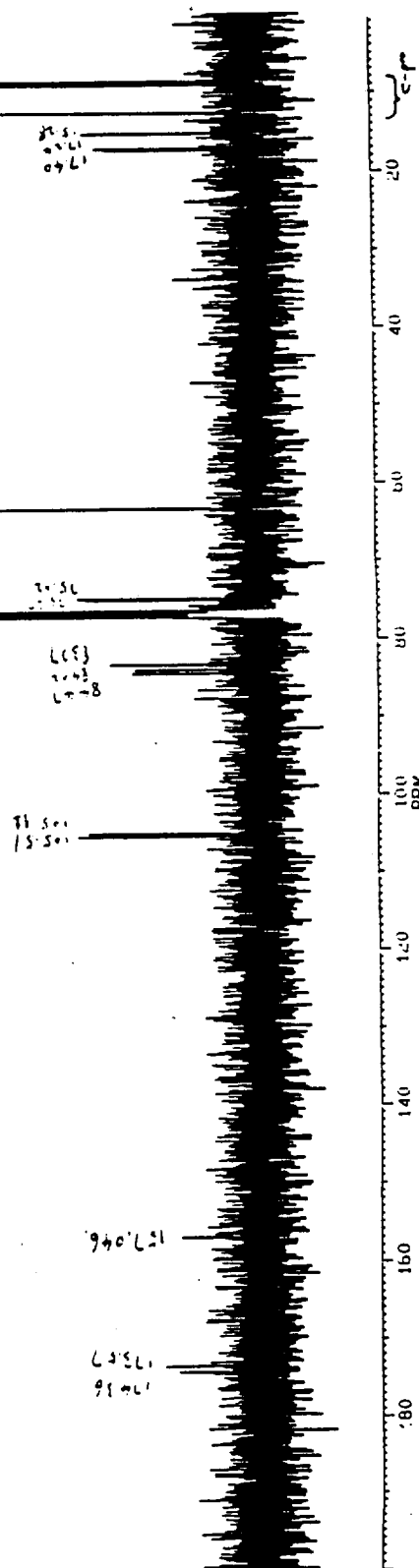


16ab

77.453  
 77.000  
 76.547  
 61.4515

61.4515

77.453  
 77.000  
 76.547



proton default parameters

Current Data Parameters  
 NAME x  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 980819  
 Time 17.27

INSTRUM arx400  
 PROBHD 5 mm DNP 1H  
 PULPROG zg30

TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0

SMH 8064.516 Hz  
 FIDRES 0.123055 Hz

AQ 4.0632820 sec  
 RG 1430

DM 62.000 usec  
 DE 88.57 usec

TE 300.0 K  
 D1 2.0000000 sec

P1 8.25 usec  
 DE 88.57 usec

SFO1 400.1324008 MHz  
 NUCLEUS 1H

F2 - Processing parameters  
 SI 65536  
 SF 400.1300136 MHz

WDW EM  
 SSB 0  
 LB 0.30 Hz

GB 0  
 PC 1.00

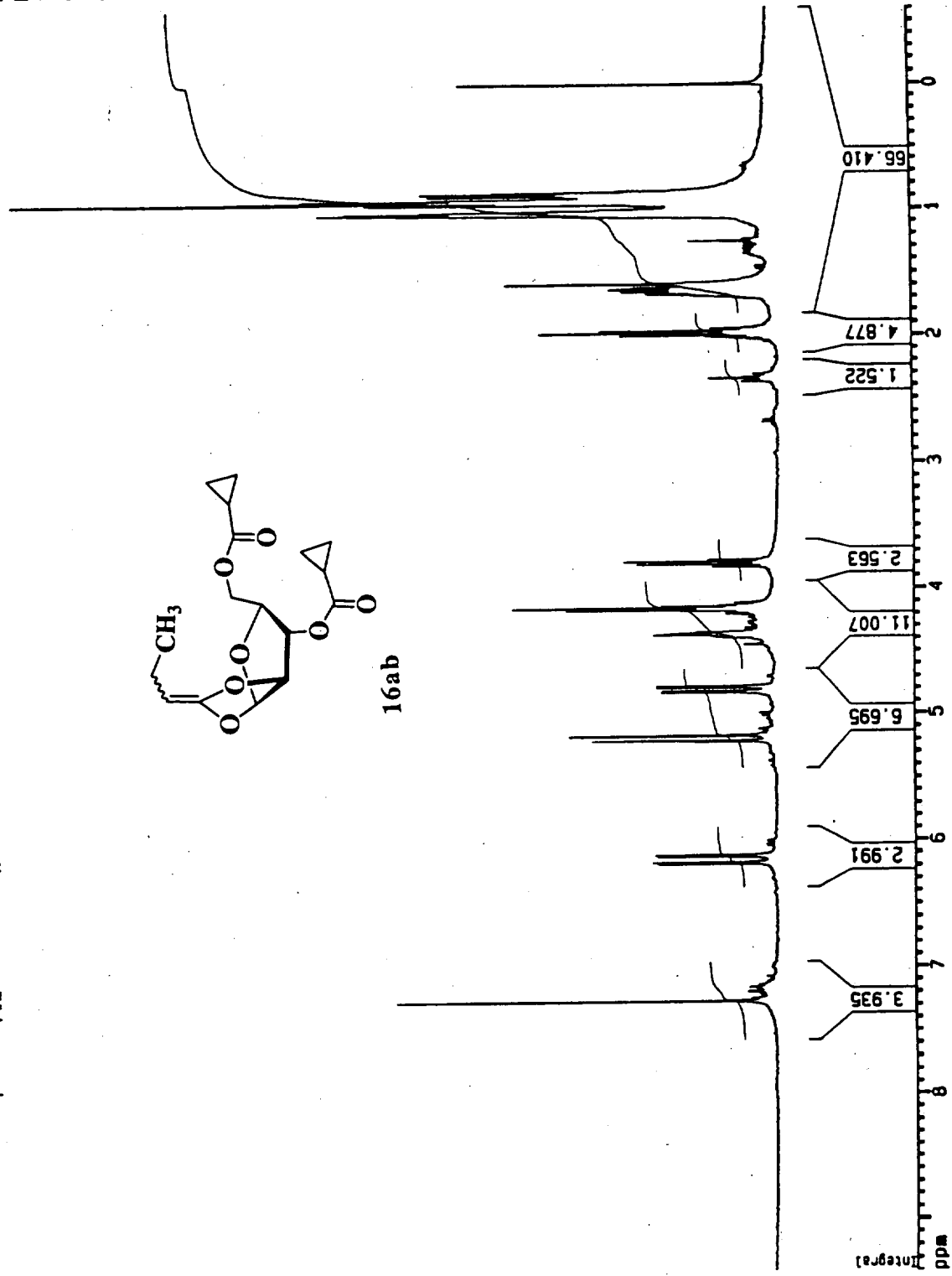
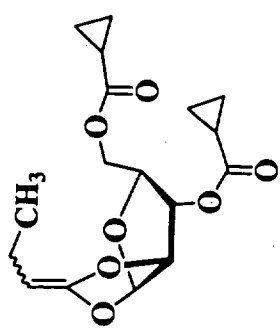
1D NMR plot parameters  
 CX 20.00 cm  
 F1P 9.400 ppm

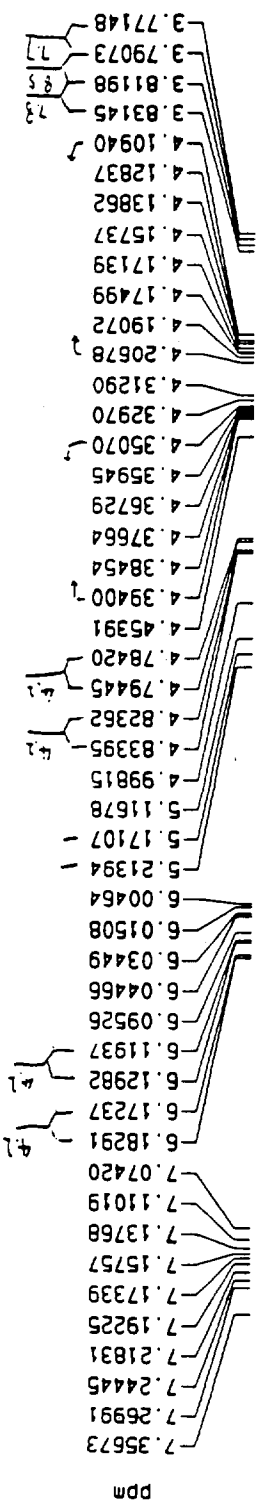
F1 3761.22 Hz  
 F2P -0.600 ppm

F2 -240.08 Hz  
 PPMCH 0.50000 ppm/cm

HZCM 200.06500 Hz/cm

7.9537  
7.9567  
7.2699  
7.2445  
7.1923  
7.1734  
7.1576  
7.1377  
6.1829  
6.1724  
6.1298  
6.1194  
5.2139  
5.1711  
4.8340  
4.8236  
4.7944  
4.7842  
4.3766  
4.3673  
4.3594  
4.3507  
4.1750  
4.1714  
4.1574  
4.1386  
3.8120  
3.7907  
1.9811  
1.9625  
1.6396  
1.6312  
1.5940  
1.0499  
1.0465  
1.0386  
1.0281  
1.0206  
0.9663  
0.9629  
0.9601  
0.9473  
0.9443  
0.9295  
0.9256  
0.8967  
0.8879  
0.8767





16ab

F2 - Acquisition Parameters  
 Date\_ 980819  
 Time 17.27  
 INSTRUM arx400  
 PROBHD 5 mm QNP 1H  
 PULPROG zg30  
 TO 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 8064.516 Hz  
 FIDRES 0.123055 Hz  
 AQ 4.0632820 sec  
 RG 1430  
 DW 62.000 usec  
 DE 88.57 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 P1 8.25 usec  
 DE 88.57 usec  
 SF01 400.1324008 MHz  
 NUCLEUS 1H

F2 - Processing parameters  
 SI 65536  
 SF 400.1300136 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 8.000 ppm  
 F1 3201.04 Hz  
 F2P 3.000 ppm  
 F2 1200.39 Hz  
 PPMCH 0.25000 ppm/cm  
 HZCH 100.03250 Hz/cm

