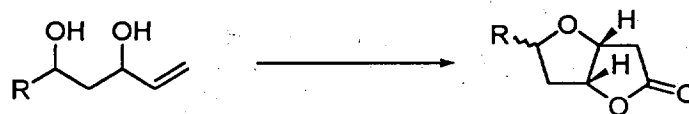


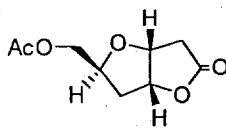
**Illustrative Pd(II)-Mediated Hydroxycyclisation-carbonylation-lactonisation**

The following procedure is representative of the palladium catalysed hydroxycyclisation-carbonylation-lactonisation sequence.

NaOAc (3eq) and  $\text{CuCl}_2$  (3eq) were added to a dry 3-necked flask containing ~10mL glacial acetic acid. The solution was stirred until the solids dissolved, and then the unsaturated diol (1eq) was added as an acetic acid solution. The flask was purged several times with nitrogen after which carbon monoxide was added from an attached balloon, and the system flushed several times with this gas. A catalytic amount of  $\text{PdCl}_2$  (0.1eq) was then added, and stirring continued under these conditions. After refilling the balloon with CO, the reaction mixture was stirred overnight at room temperature. Generally this resulted in a change in colour from bright green to a dull brown. The CO atmosphere was removed, and ~50mL  $\text{H}_2\text{O}$  was added which caused the solution to turn black. This solution was then neutralized with solid  $\text{NaHCO}_3$  and extracted with EtOAc. Removal of the solvent provided the crude bicyclic lactones, which were purified and separated by flash chromatography ( $\text{SiO}_2$ , 20% ethylacetate/hexane) sometimes followed by further chromatography ( $\text{SiO}_2$ , 10% ethylacetate/hexane) if necessary for diastereomer separation. Yields for the conversion of enediols to bicyclic lactones were generally very good (> 80% isolated yields).

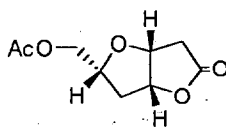
Characterisation of Compounds **8**, **11**, **12**, **16**, **17**, **20** and **23**

Compound **8** : mp 66-67° (*trans*). Calc. for C<sub>9</sub>H<sub>12</sub>O<sub>5</sub> : C, 54.00 ; H, 6.04. Found C, 54.12 ; H, 6.23. HREIMS (M-HOAc) : C<sub>7</sub>H<sub>8</sub>O<sub>3</sub> requires 140.0473. Found : 140.0474.



$\delta$ (ppm)	multiplicity	J (Hz)
5.1	app.t	4.7
4.81	dddd	5.2, 4.7, 1.8, 0.4
4.36	dddd	9.7, 6.0, 5.6, 3.2, 0.4
4.22	dd	11.9, 3.2
4.04	dd	11.9, 6.0
2.73	dd	18.7, 5.2
2.68	dd	18.7, 1.8
2.40	ddd	14.7, 5.6, 0.4
2.07	s	
1.91	ddd	14.7, 9.7, 5.1

<sup>13</sup> C
175.3
170.7
84.2
78.5
76.4
65.2
36.6
35.1
20.8



$\delta$ (ppm)	multiplicity	J (Hz)
5.01	ddd	6.7, 4.2, 1.6
4.61	ddd	4.5, 4.2, 1.8
4.27	m	
4.22	dd	12.0, 3.2
4.05	dd	12.0, 5.9
2.76	dd	18.2, 1.8
2.70	dd	18.2, 4.5
2.43	ddd	14.4, 8.2, 6.7
2.09	ddd	14.4, 6.4, 1.6
2.07	s	

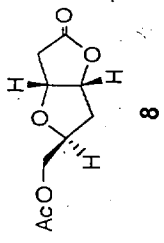
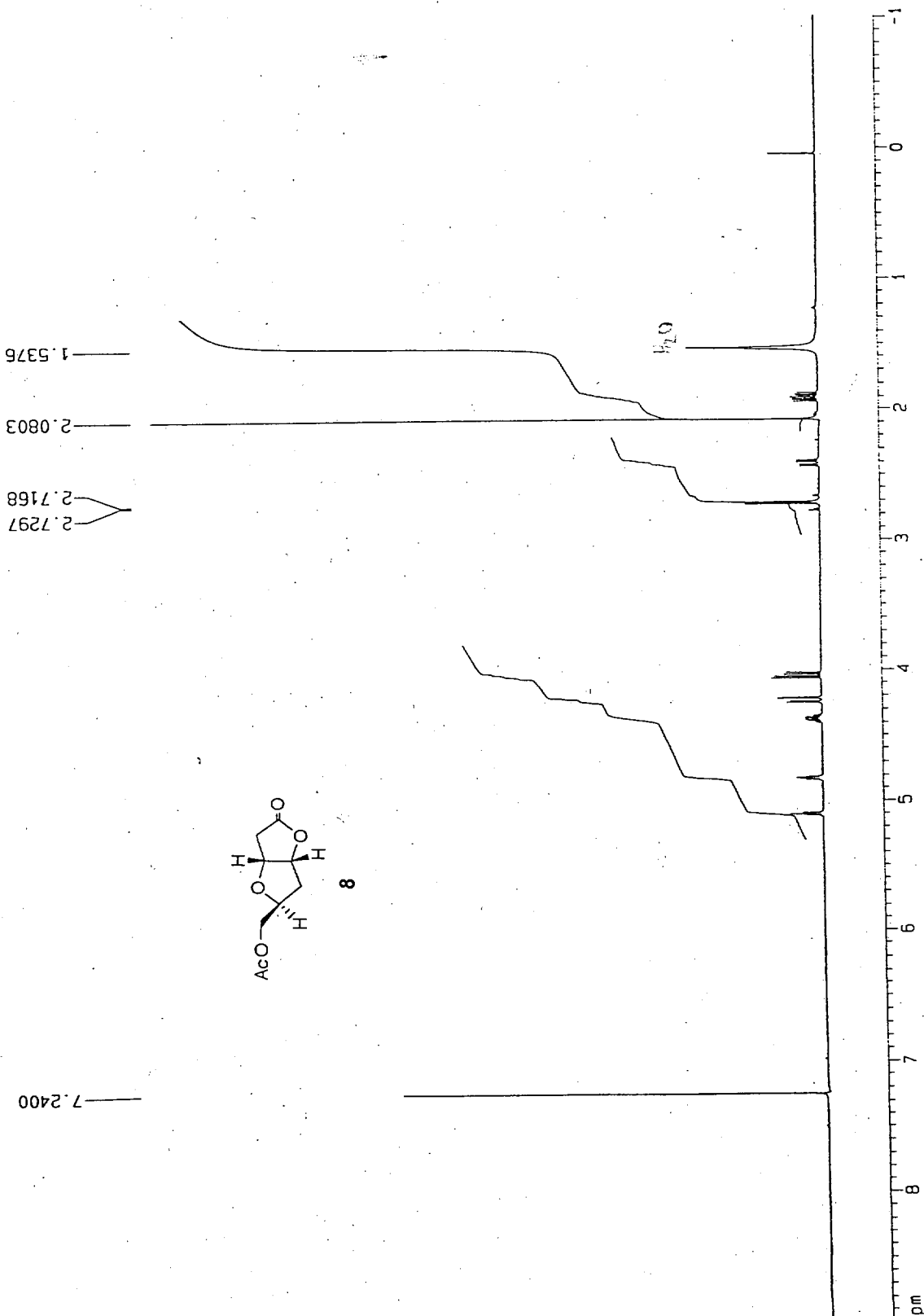
<sup>13</sup> C
174.9
170.9
83.9
79.4
78.0
65.5
36.5
34.7
20.7

Current Data Parameters  
 NAME alcoola  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 990915  
 Time 10.40  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 32  
 DS 0  
 SWH 4807.692 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4079220 sec  
 RG 2048  
 DW 104.000 usec  
 DE 148.57 usec  
 TE 305.0 K  
 HL1 0 dB  
 D1 0.6000002 sec  
 P1 3.70 usec  
 DE 148.57 usec  
 SF01 400.1364000 MHz  
 NUCLEUS 1H

F2 - Processing parameters  
 SI 16384  
 SF 400.1344024 MHz  
 XDM EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

10 NMR plot parameters  
 CX 22.00 cm  
 F1P 9.043 ppm  
 F1 3618.39 Hz  
 F2P -1.400 ppm  
 F2 -560.19 Hz  
 PPMCM 0.47468 ppm  
 HZCM 189.93513 Hz



Current Data Parameters  
 NAME gregacetate  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

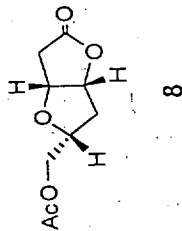
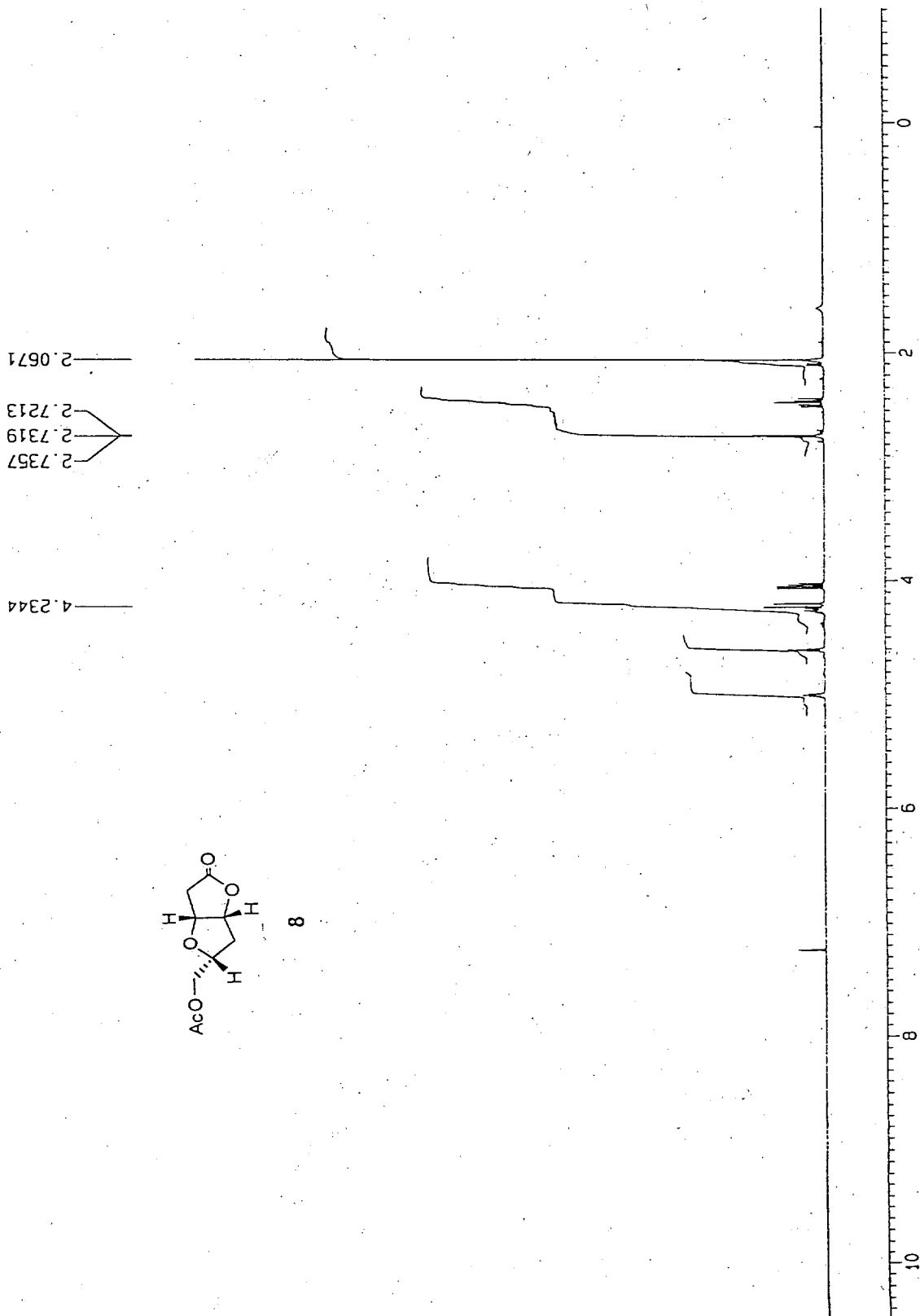
Date\_ 990907  
 Time 12.50  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TD 32768  
 SOLVENT CDC13  
 NS 32  
 DS 0  
 SWH 4807.692 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4079220 sec  
 RG 256  
 OW 104.000 usec  
 DE 148.57 usec  
 TE 305.0 K  
 HL1 0 dB  
 D1 0.6000002 sec  
 P1 3.70 usec  
 DE 148.57 usec  
 SFO1 400.1364000 MHz  
 NUCLEUS 1H

F2 - Processing parameters

SI 16384  
 SF 400.1344021 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

10 NMR plot parameters

CX 22.00 cm  
 F1P 11.001 ppm  
 F1 4401.73 Hz  
 F2P -1.015 ppm  
 F2 -405.97 Hz  
 PPMCH 0.54615 ppm/ci  
 HZCH 218.53148 Hz/cm



6

Current Data Parameters  
 NAME gregacetate  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 990907  
 Time 12.58  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TO 65536  
 SOLVENT CHCl3  
 NS 463  
 DS 1  
 SWH 21739.131 Hz  
 FIDRES 0.331713 Hz  
 AQ 1.5073780 sec  
 RG 32768  
 OW 23.000 usec  
 DE 32.86 usec  
 TE 302.0 K  
 HL1 48 dB  
 O1 1.5000000 sec  
 CPOPRG waltz16  
 P31 100.00 usec  
 S4 44 dB  
 O11 0.0300000 sec  
 S2 27 dB  
 P1 2.40 usec  
 DE 32.86 usec  
 SF01 100.6236000 MHz  
 NUCLEUS 13C

F2 - Processing parameters

SF 32768  
 SF 100.6138744 MHz  
 WDM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters

CX 22.00 cm  
 F1P 185.631 ppm  
 F1 18677.01 Hz  
 F2P -10.840 ppm  
 F2 -1090.68 Hz  
 PPKCM 8.93049 ppm/cm  
 HZCM 898.53131 Hz/cm

20.72

34.71

36.47

65.48

76.68

77.00

77.32

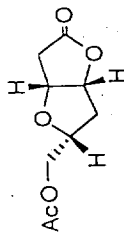
77.97

79.36

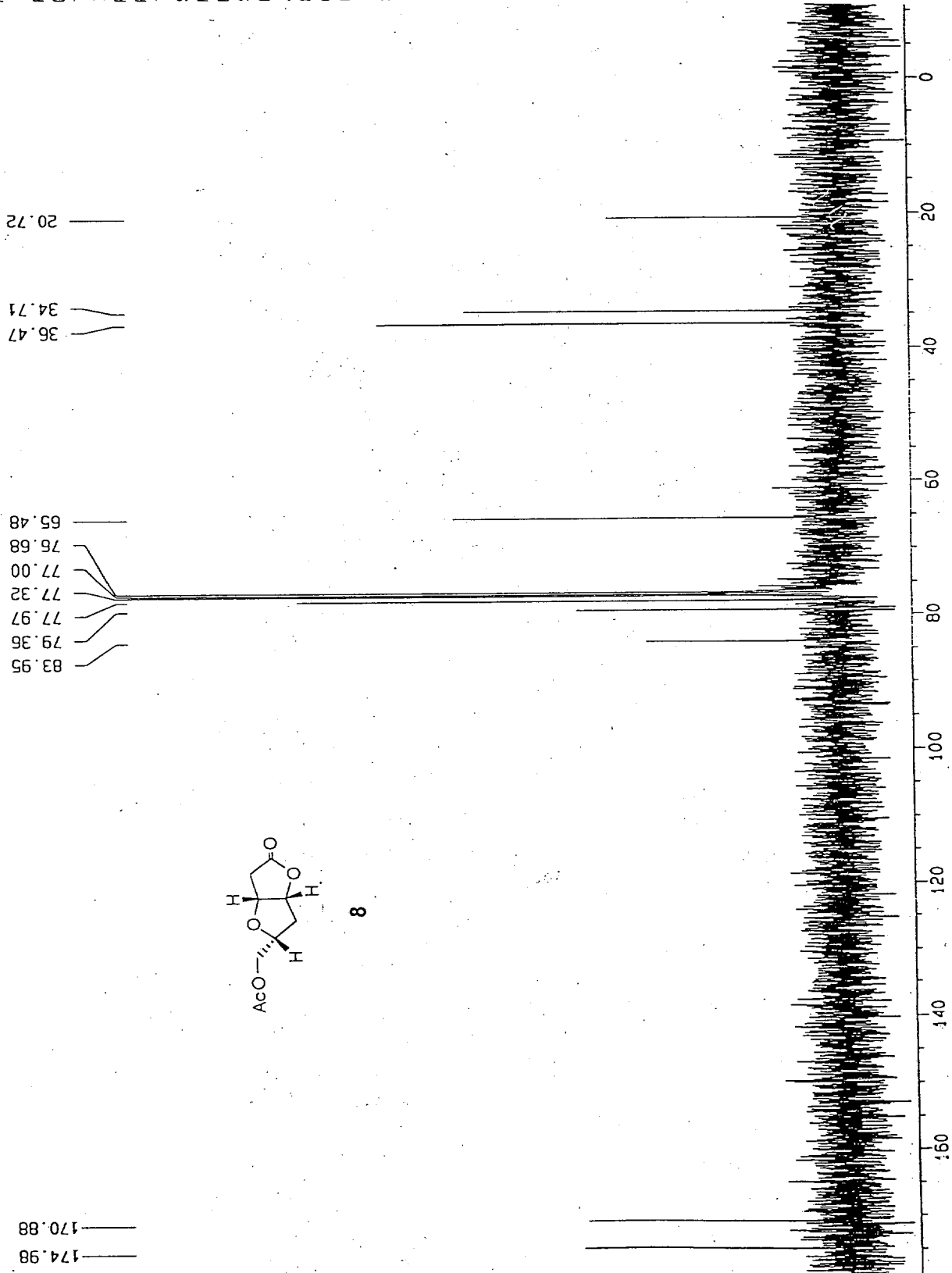
83.95

170.88

174.98

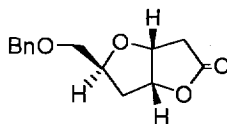


8



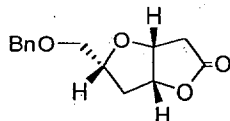
7

Compound 11 : Calc. for  $C_{14}H_{16}O_4$  : C, 67.73 ; H, 6.50. Found C, 67.95 ; H, 6.48.  
 HREIMS :  $C_{14}H_{16}O_4$  requires 248.1049. Found : 248.1048.



$\delta$ (ppm)	multiplicity	J (Hz)
7.35/7.38	m (5H)	
5.09	5t	4.7
4.82	ddd	5.3, 4.7, 2.2
4.51	s (2H)	
4.32	m	
3.60	dd	10.5, 3.2
3.46	dd	10.5, 5.0
2.72	dd	18.6, 4.7
2.67	dd	18.6, 1.5
2.33	dd	14.0, 5.8
2.04	dd	14.3, 9.2, 5.3

$^{13}C$
176.1
138.2
128.5
127.8
127.7
84.7
78.5
77.9
73.5
71.5
36.7
34.9



$\delta$ (ppm)	multiplicity	J (Hz)
7.34/7.28	m (5H)	
5.00	ddd	6.8, 4.6, 1.8
4.59	ddd	6.6, 4.6, 1.6
4.57	d	11.6
4.51	d	11.6
4.22	m	
3.50	dd	10.2, 6.2
3.49	dd	10.2, 4.6
2.76	dd	18.4, 1.6
2.70	dd	18.4, 4.9
2.39	ddd	14.6, 7.9, 6.8
2.04	ddd	14.6, 6.6, 1.8

$^{13}C$
175.2
137.8
128.4
127.9
127.8
84.1
79.3
79.1
73.5
72.2
36.5
35.2

Current Data Parameters  
 NAME benzy1  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 990907  
 Time 13.25  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TD 32768  
 SOLVENT COCl3  
 NS 32  
 DS 0  
 SWH 4807.692 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4079220 sec  
 RG 256  
 DW 104.000 usec  
 DE 148.57 usec  
 TE 305.0 K  
 HL1 0 dB  
 O1 0.6000002 sec  
 P1 3.70 usec  
 DE 148.57 usec  
 SFO1 400.1354000 MHz  
 NUCLEUS 1H

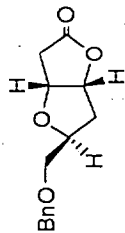
F2 - Processing parameters

SI 16384  
 SF 400.1344018 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

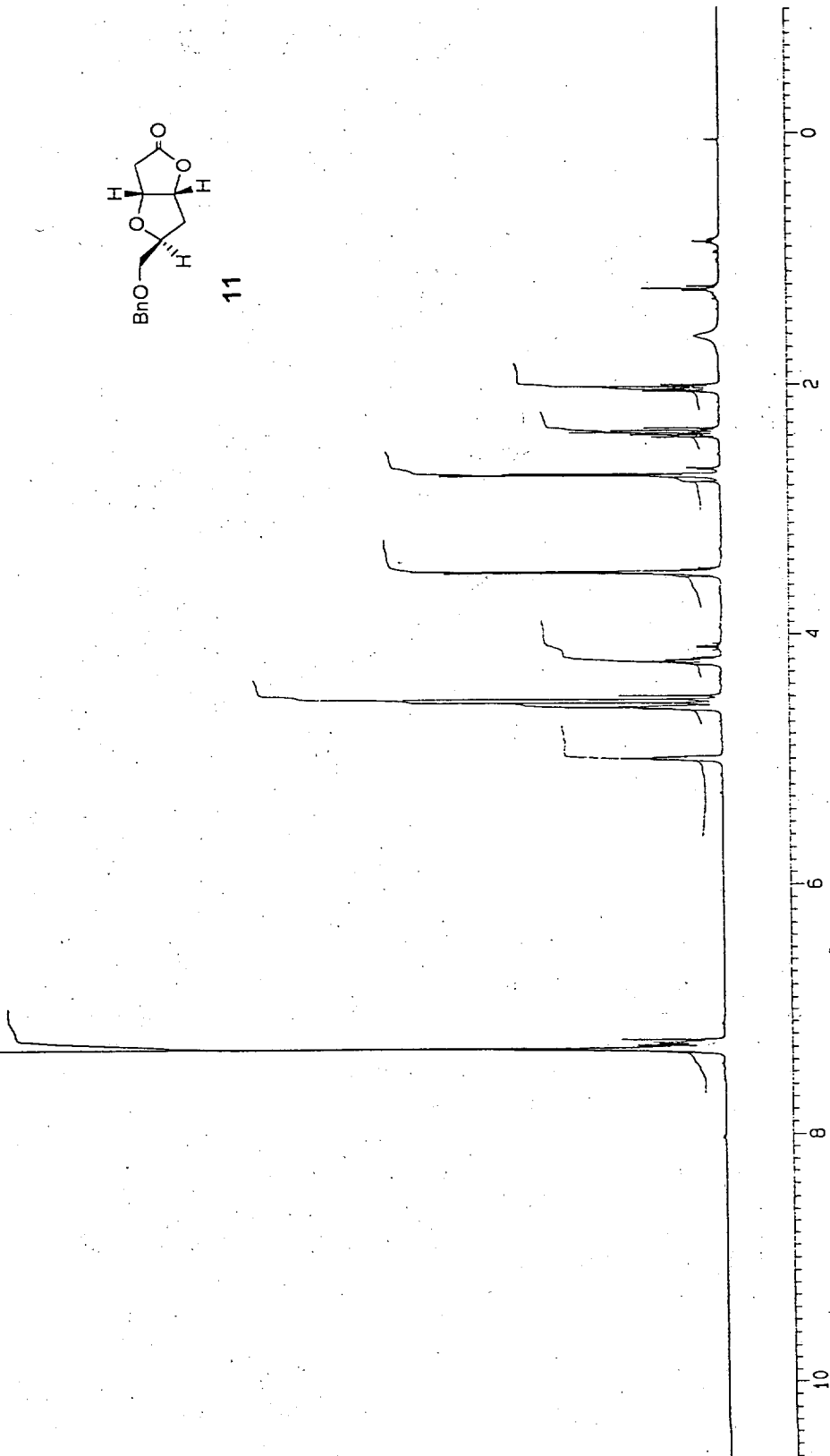
1D NMR plot parameters

CX 22.00 cm  
 F1P 11.001 ppm  
 F1 4402.02 Hz  
 F2P -1.014 ppm  
 F2 -405.67 Hz  
 PPMCH 0.54615 ppm/ci  
 HZCM 218.53148 Hz/cm

7.3246  
 7.3220  
 7.3086  
 7.2975  
 7.2934  
 7.2827  
 7.2781  
 7.2615  
 7.2401  
 5.0047  
 4.9999  
 4.6023  
 4.5983  
 4.5910  
 4.5869  
 4.5838  
 4.5800  
 4.5335  
 4.5247  
 4.4944  
 3.5051  
 3.4966  
 3.4896  
 3.4849  
 2.7308  
 2.7268  
 2.7234  
 2.7115  
 2.3983  
 2.3788  
 2.3619  
 2.3592  
 2.3422  
 2.0582  
 2.0407  
 2.0216  
 2.0186  
 1.2347



11



8

19

Current Data Parameters  
 NAME benzyl  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 990907  
 Time 13.34  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CHCl3  
 NS 949  
 DS 1  
 SWH 21739.131 Hz  
 FIDRES 0.331713 Hz  
 AQ 1.5073780 sec  
 RG 32768  
 DM 23.000 usec  
 DE 32.85 usec  
 TE 302.0 K  
 HL1 48 dB  
 D1 1.50000000 sec  
 CPOPRG waltz16  
 P31 100.00 usec  
 S4 44 dB  
 D11 0.0300000 sec  
 S2 27 dB  
 P1 2.40 usec  
 DE 32.85 usec  
 SF01 100.6236000 MHz  
 NUCLEUS 13C

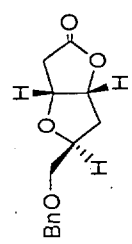
F2 - Processing parameters

S1 32768  
 SF 100.6138757 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters

CX 22.00 cm  
 F1P 176.321 ppm  
 F1 17740.36 Hz  
 F2P 123.717 ppm  
 F2 12447.68 Hz  
 PPMCM 2.39109 ppm/cm  
 HZCM 240.57674 Hz/cm

35.17  
 36.50

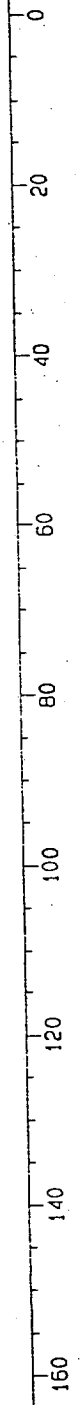


11

72.21  
 73.46  
 76.68  
 77.00  
 77.32  
 79.08  
 79.22  
 84.09

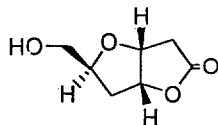
127.74  
 127.82  
 128.39  
 137.77

175.25



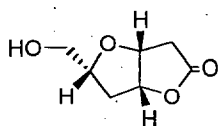


10

Compound 12 : HREIMS (M-H<sub>2</sub>O) : C<sub>7</sub>H<sub>8</sub>O<sub>3</sub> requires 140.0473. Found : 140.0473.

$\delta$ (ppm)	multiplicity	J (Hz)
5.10	br.t	4.6
4.80	br.t	~5
4.26	m	
3.80	dd	12.0, 3.0
3.69	dd	12.0, 3.4
2.72	dd	18.6, 4.6
2.67	dd	18.6, 1.5

<sup>13</sup> C
175.3
84.8
79.0
78.4
63.3
36.7
33.8



$\delta$ (ppm)	multiplicity	J (Hz)
5.02	ddd	6.4
4.58	m	
4.14	m	
3.56	dd	11.9, 6.4
3.52	dd	11.9, 7.6
2.70	d (2H)	3.6
2.30	dd	14.1, 5.2
2.04	dd	14.1, 5.0

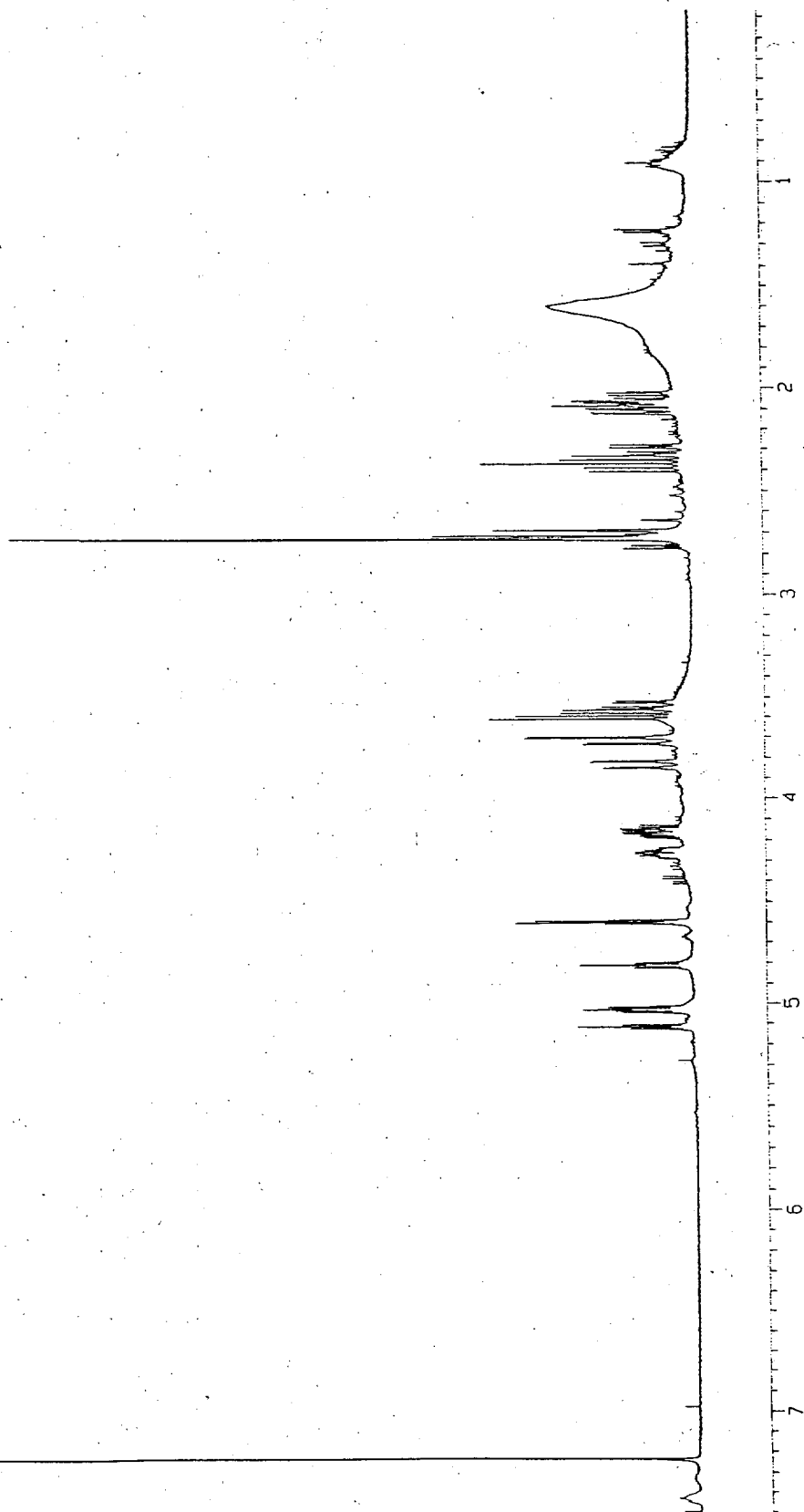
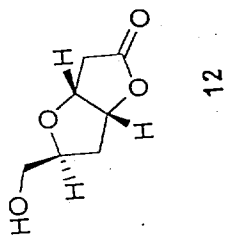
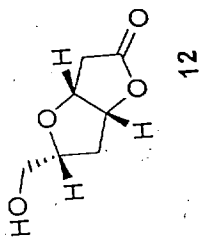
<sup>13</sup> C
175.6
84.4
80.72
79.0
64.5
36.4
34.0

Current Data Parameters  
 NAME gpj222  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 970831  
 Time 9.42  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TO 32768  
 SOLVENT CDCl3  
 NS 98  
 DS 0  
 SWH 4807.692 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4079220 sec  
 RG 512  
 DW 104.000 usec  
 DE 148.57 usec  
 TE 305.0 K  
 HL1 0 dB  
 O1 0.60000002 sec  
 P1 3.70 usec  
 DE 148.57 usec  
 SF01 400.1364000 MHz  
 NUCLEUS 1H

F2 - Processing parameters  
 SI 16384  
 SF 400.1344018 MHz  
 WDM no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 7.674 ppm  
 F1 3070.48 Hz  
 F2P 0.164 ppm  
 F2 65.67 Hz  
 PPMCM 0.34134 ppm/  
 HZCM 136.58217 Hz/c



10

Current Data Parameters  
 NAME gp1216  
 EXPNO 2  
 PROCNO 1

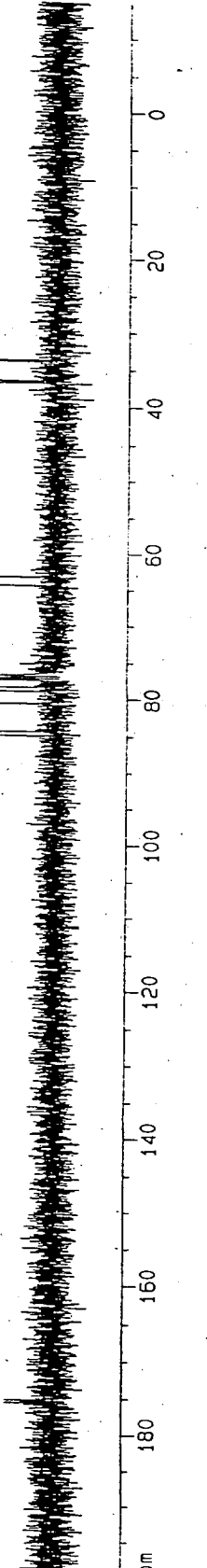
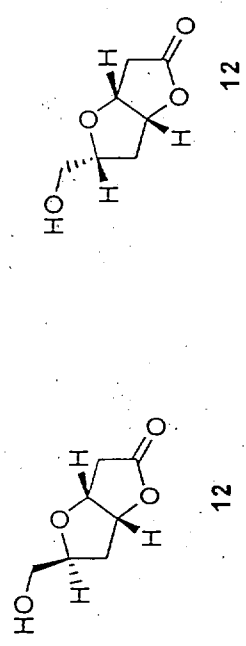
F2 - Acquisition Parameters  
 Date\_ 970729  
 Time 19.05  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CHCl3  
 NS 206  
 DS 1  
 SWH 21739.131 Hz  
 FIDRES 0.331713 Hz  
 AQ 1.5073780 sec  
 RG 32768  
 DW 23.000 usec  
 DE 28.75 usec  
 TE 302.0 K  
 HL1 48 dB  
 D1 1.50000000 sec  
 CPOPRG waltz16  
 P31 100.00 usec  
 S4 43 dB  
 D11 30.0000000 sec  
 S2 28 dB  
 P1 2.30 usec  
 DE 28.75 usec  
 SF01 100.6232500 MHz  
 NUCLEUS 13C

F2 - Processing parameters  
 SI 32768  
 SF 100.6138760 MHz  
 MDW EM  
 SSB 0  
 LB 0.50 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 201.200 ppm  
 F1 20243.55 Hz  
 F2 -14.865 ppm  
 PPMCM 9.82113 ppm/cm  
 HZCM 988.14233 Hz/cm

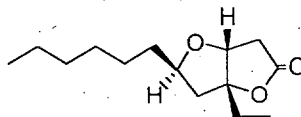
84.98  
84.44  
80.72  
79.04  
79.01  
78.38  
77.32  
77.00  
76.68  
64.48  
63.30  
36.72  
36.42  
34.02  
33.82

175.64  
175.26



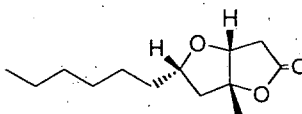
(12)

13

Compound 14 : HREIMS : C<sub>14</sub>H<sub>24</sub>O<sub>3</sub> requires 240.1725. Found : 240.1737.

$\delta$ (ppm)	multiplicity	J (Hz)
4.42	d	6.5
4.05	dddd	10.5, 8.2, 4.5, 4.4
2.76	dd	18.8, 6.5
2.61	dt	18.8, 1
2.32	dd	13.4, 4.5
1.54	dd	13.4, 10.5
1.71/1.86	m (2H)	
1.26/1.64	m (10H)	
1.00	t	7.4
0.86	t	7.1

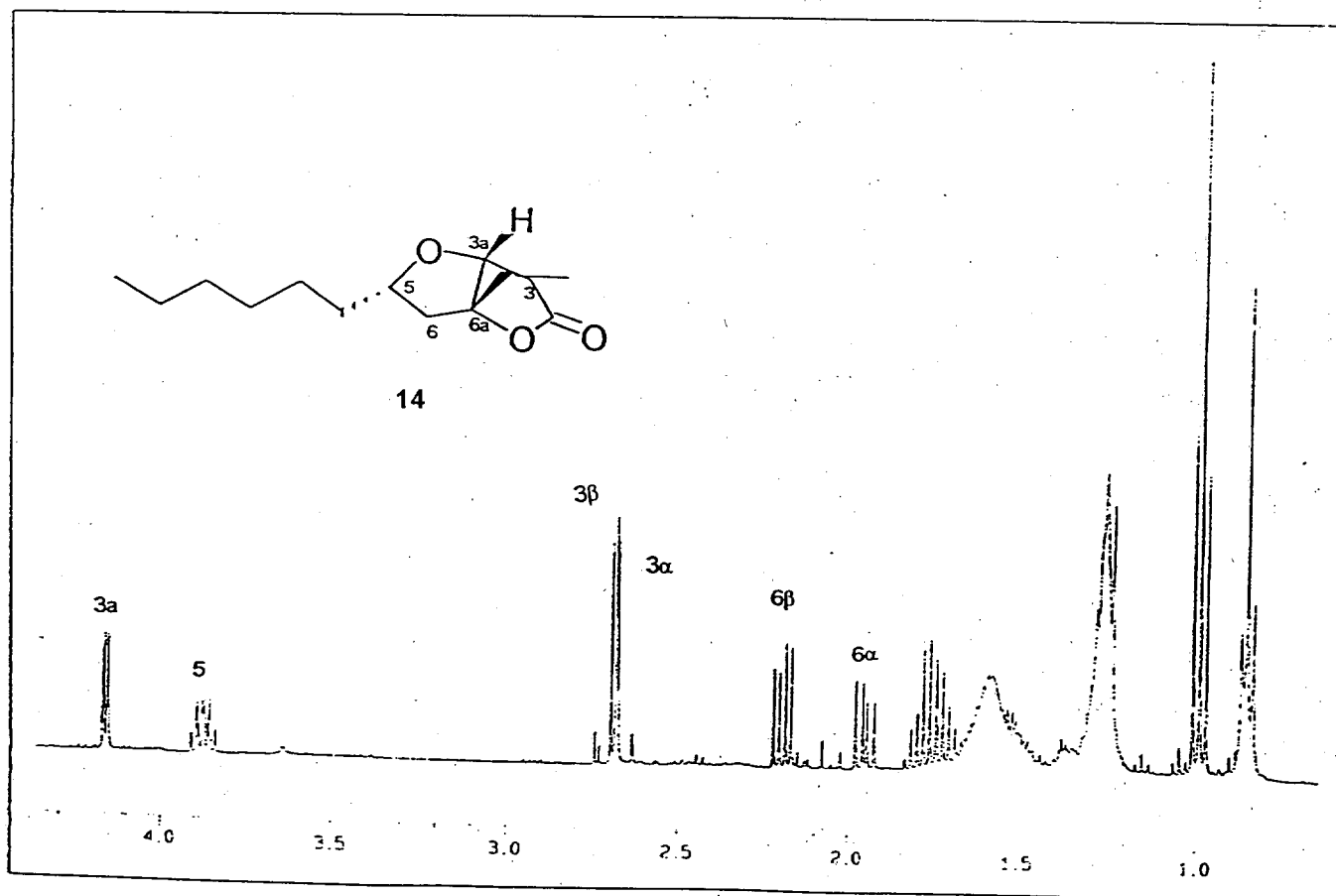
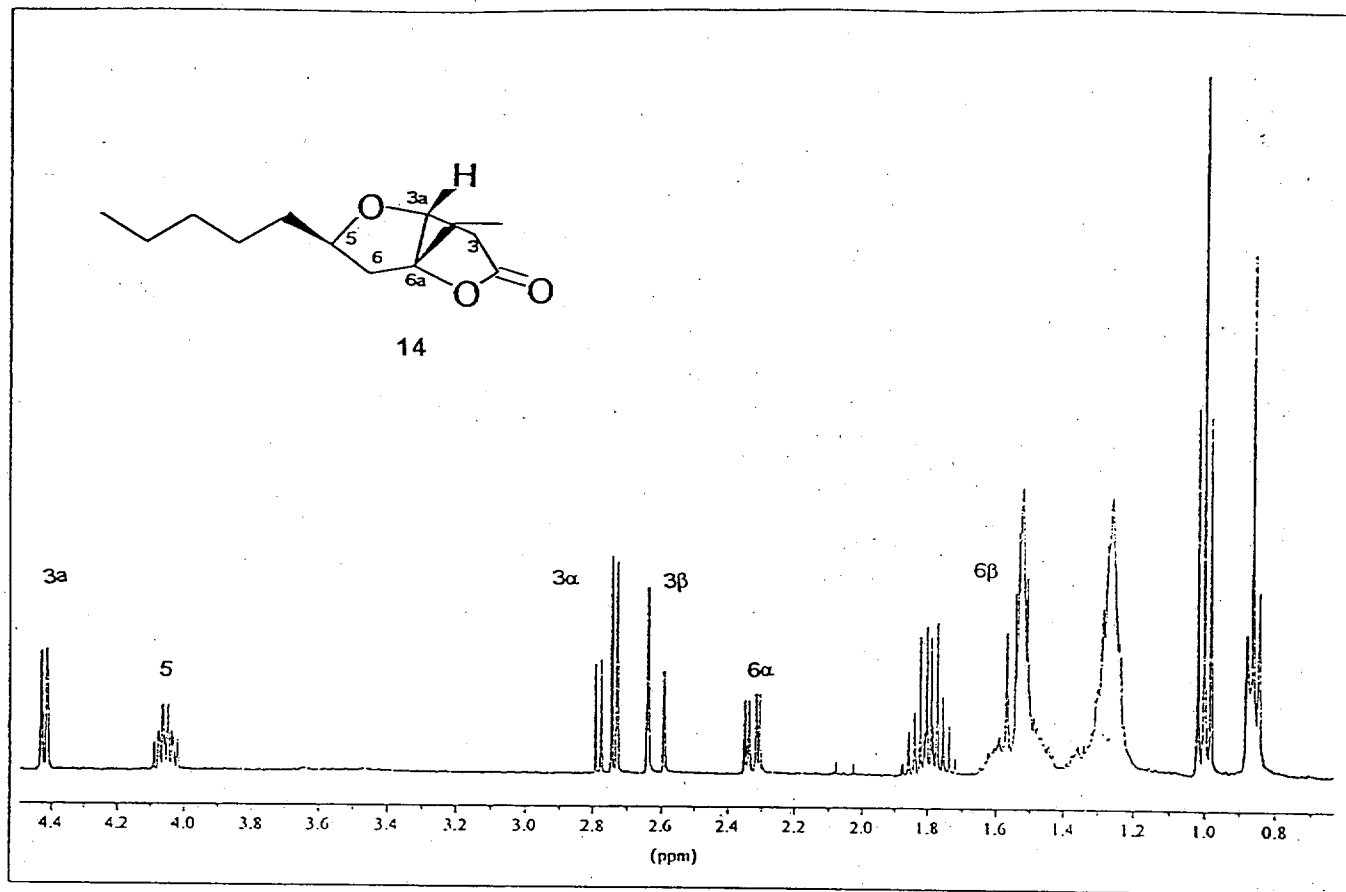
<sup>13</sup> C
175.8
97.3
80.4
78.6
42.6
37.3
34.7
31.7
29.7
29.3
25.9
22.6
10.0
8.7



$\delta$ (ppm)	multiplicity	J (Hz)
4.16	dd	4.8, 1.8
3.89	m	
2.69	dd	18.6, 5.0
2.68	dd	18.6, 1.9
1.95	dd	13.8, 8.7
2.19	dd	13.8, 6.7
1.75	ddd (2H)	7.5
1.25/1.53	m (10H)	
0.99	t	7.4
0.86	t	7.1

<sup>13</sup> C
175.1
98.6
80.9
79.52
42.8
36.6
35.1
31.5
29.0
28.3
25.3
22.4
13.9
8.1

14



Current Data Parameters  
 NAME gpj214  
 EXPNO 2  
 PROCNO 1

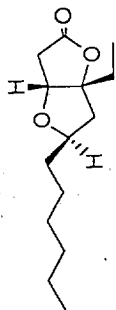
F2 - Acquisition Parameters  
 Date\_ 970725  
 Time 8.25  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TO 65536  
 SOLVENT CHCl3  
 NS 3566

DS 1  
 SWH 21739.131 Hz  
 FIDRES 0.331713 Hz  
 AQ 1.5073780 sec  
 RG 32768  
 DW 23.000 usec  
 DE 28.75 usec  
 TE 302.0 K  
 HL1 48 dB  
 D1 1.50000000 sec  
 CPROG waltz16  
 P31 100.00 usec  
 S4 43 dB  
 D11 30.0000000 sec  
 S2 28 dB  
 P1 2.30 usec  
 DE 28.75 usec  
 SF01 100.6232500 MHz  
 NUCLEUS 13C

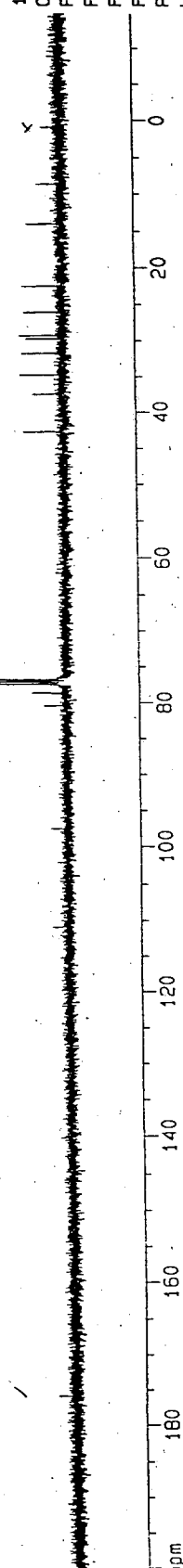
F2 - Processing parameters  
 SI 32768  
 SF 100.6138720 MHz  
 WDM EM  
 SSB 0  
 LB 0.50 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00 cm  
 FJP 201.240 ppm  
 F1 20247.53 Hz  
 F2P -14.825 ppm  
 F2 -1491.60 Hz  
 PPMCM 9.82113 ppm/cm  
 HZCM 988.14227 Hz/cm

175.84  
 111.00  
 102.12  
 97.34  
 80.45  
 78.75  
 78.63  
 77.32  
 77.20  
 77.00  
 76.68  
 42.65  
 37.30  
 34.68  
 31.71  
 29.70  
 29.23  
 25.93  
 22.53  
 14.03  
 8.67  
 1.00  
 -8.86  
 -9.55



14



(45)

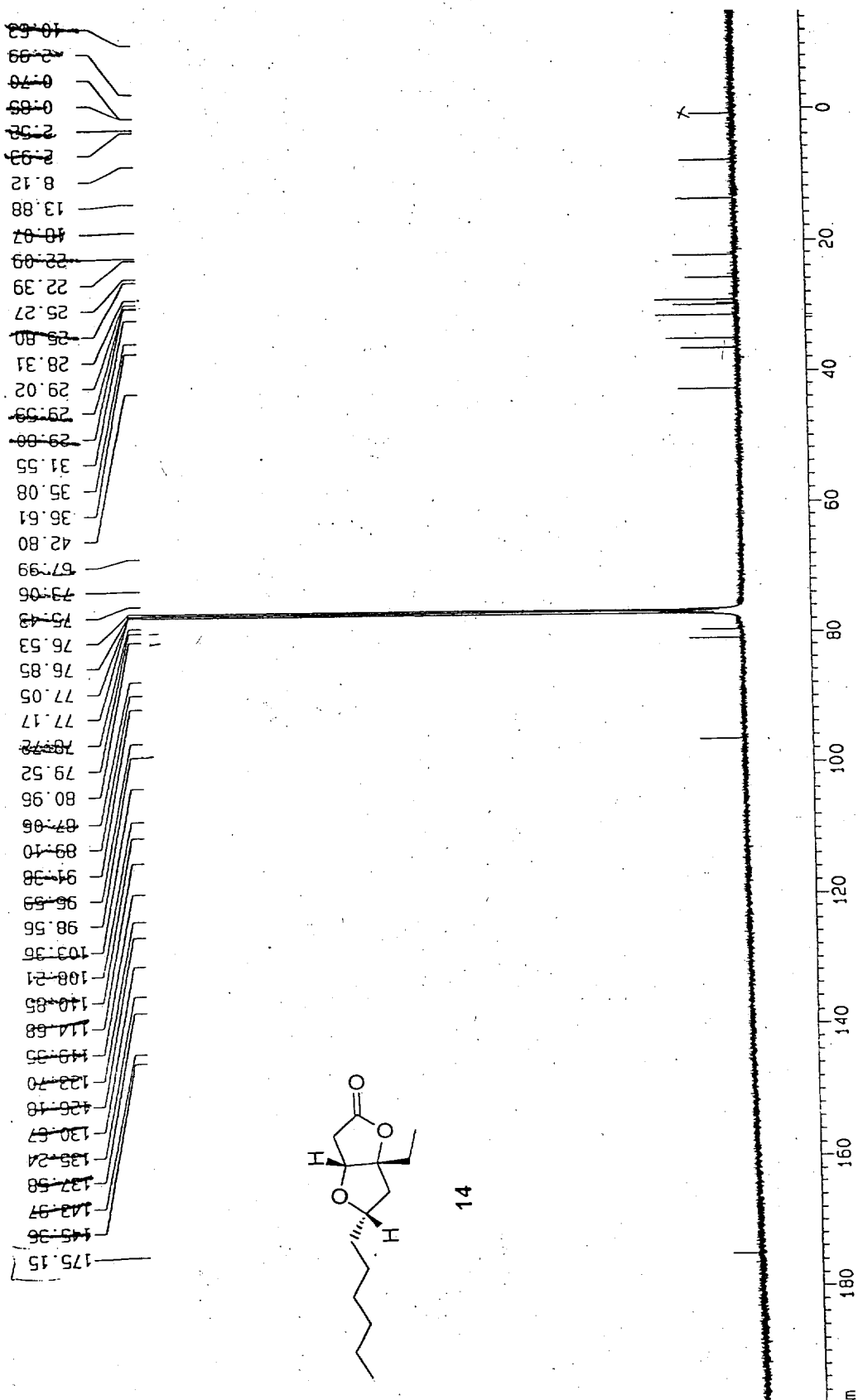
Current Data Parameters  
 NAME gpj218  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 970801  
 Time 19.52  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CHCl3  
 NS 899353  
 DS 1  
 SWH 21739.131  
 FIDRES 0.331713  
 AQ 1.5073780  
 RG 32768  
 DW 23.000  
 DE 28.75  
 TE 302.0  
 HL1 48  
 1.50000000  
 CPDPRG waltz16  
 P31 100.00  
 S4 43  
 D11 30.0000000  
 S2 28  
 P1 2.30  
 DE 28.75  
 SF01 100.6232500  
 NUCLEUS 13C

F2 - Processing parameters

SI 32768  
 SF 100.6138872  
 WDW EM  
 SSB 0  
 LB 0.50  
 GB 0  
 PC 1.00  
 1D NMR plot parameters  
 CX 22.16  
 F1P 190.538  
 F1 19170.79  
 F2P 77.134  
 F2 7760.77  
 PPMCM 5.11739  
 HZCM 514.88074

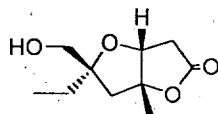


14

(46)

(17)

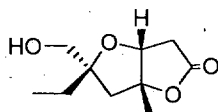
Compounds **16** and **17** : Calc. for  $C_{13}H_{20}O_5$  (for acetate) : C, 60.92 ; H, 7.87. Found C, 60.70 ; H, 8.14. HREIMS (M- $CH_2OH$ ):  $C_{10}H_{15}O_3$  requires 183.1021. Found : 183.1016.



16

$\delta$ (ppm)	multiplicity	J (Hz)
4.44	dd	4.1, 1.5
3.52	d	11.4
3.43	d	11.4
2.70	dd	18.2, 4.1
2.65	dd	18.2, 2.0
2.27	d	14.4
2.09	d	14.4
1.70/1.80	br.s	
1.78	quintet	7.6
1.71	quintet	7.6
1.59	dq (2H)	7.4, 1.2
1.00	t	7.4
0.85	t	7.6

$^{13}C$
175.2
98.0
87.9
82.0
67.1
40.8
37.6
29.8
29.4
8.6
8.3

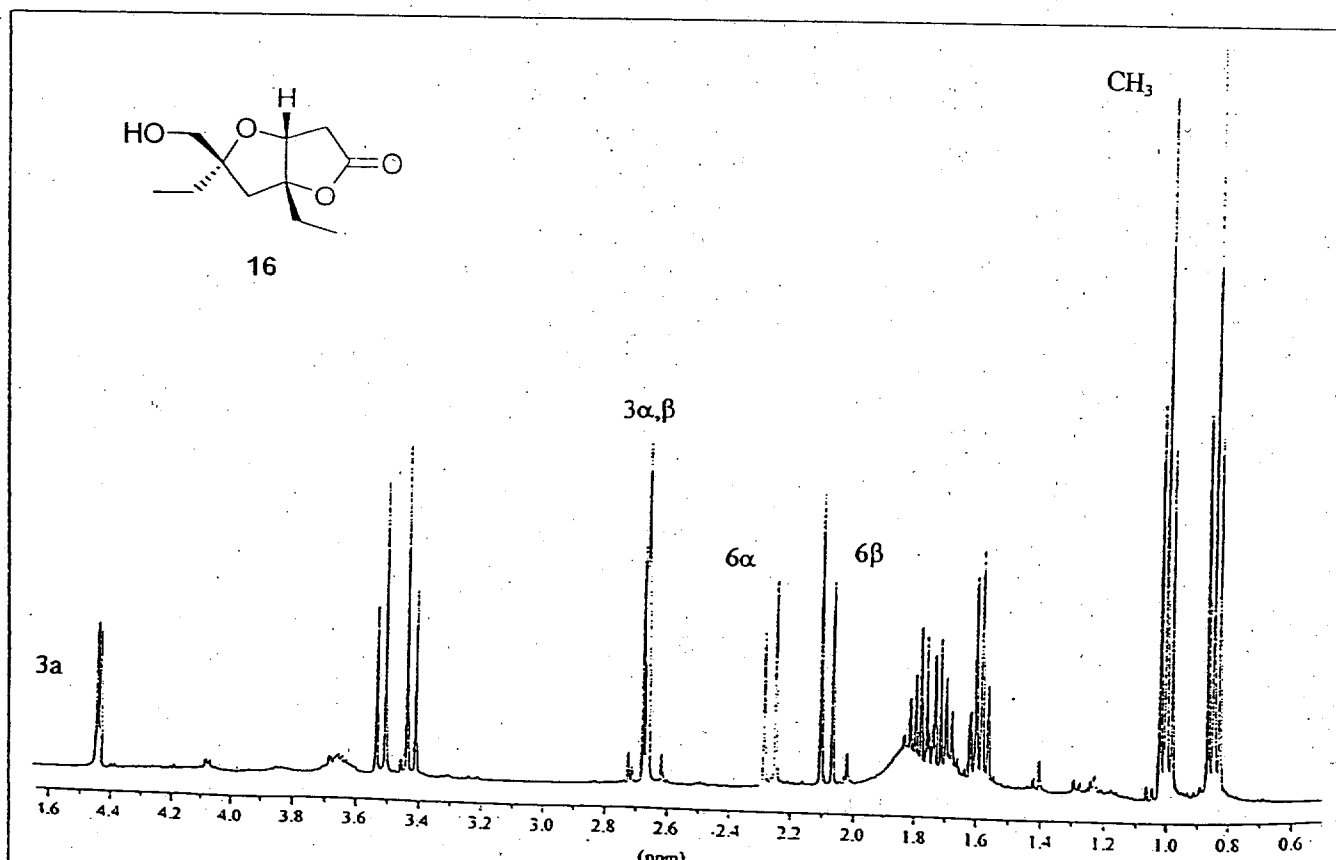
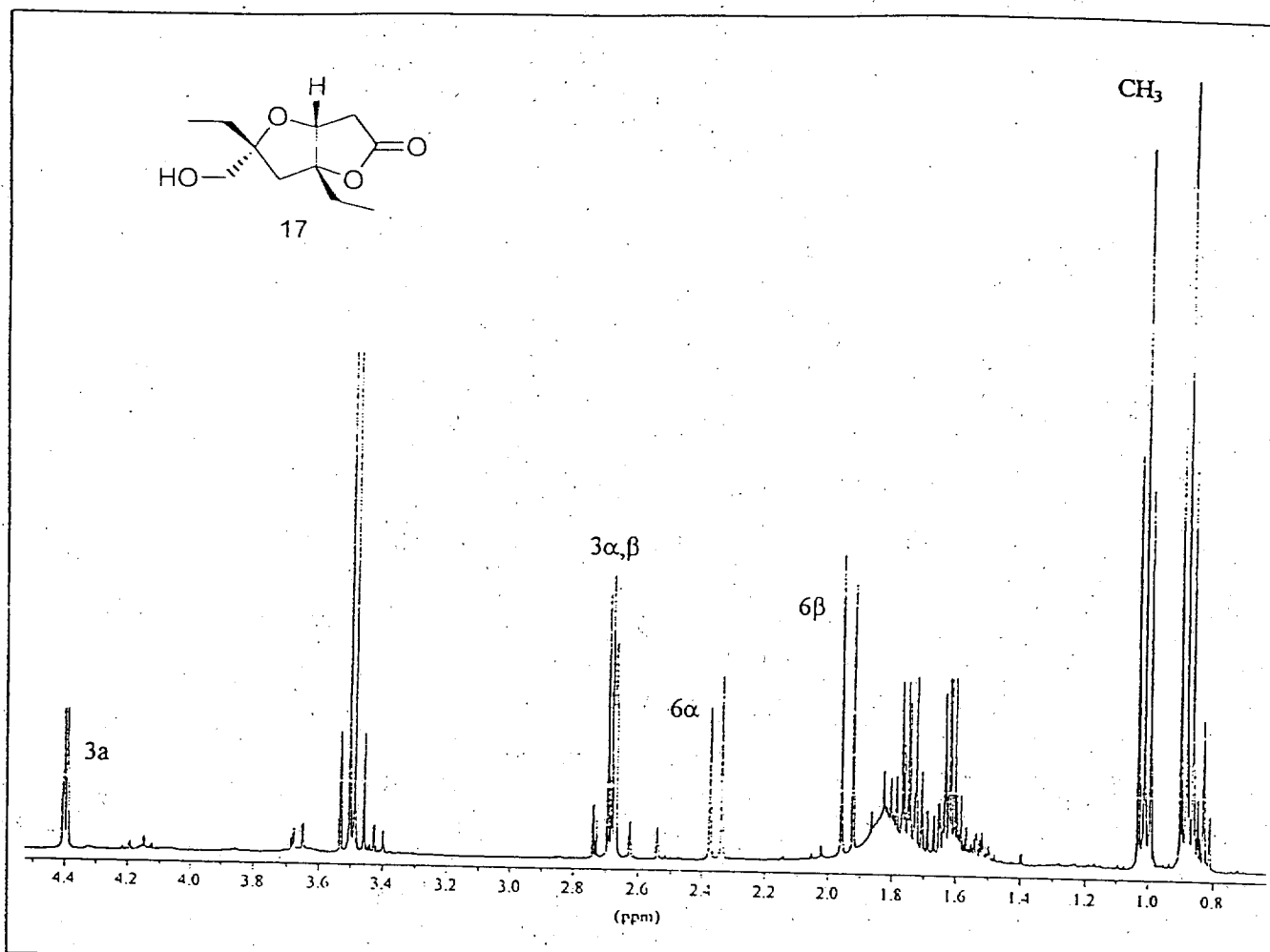


17

$\delta$ (ppm)	multiplicity	J (Hz)
4.40	dd	4.8, 1.2
3.52	d	12.3
3.47	d	11.6
2.71	dd	18.3, 4.8
2.64	dd	18.3, 1.2
2.35	d	14.6
1.94	d	14.6
1.50/1.85	m (5H)	
1.01	t	7.4
0.88	t	7.4

$^{13}C$
175.1
97.6
87.1
81.3
67.0
41.7
37.4
30.1
29.0
8.5
8.2





\*\*\* Current Data Parameters \*\*\*

NAME : gpj226  
 EXPNO : 2  
 PROCNO : 0

\*\*\* Acquisition Parameters \*\*\*

AQ\_mod : qsim  
 CNST[2] : 160.000000  
 D[1] : 1.500000 sec  
 DATE\_d : Sep 04 1997

DS : 1  
 HL1 : 48 dB  
 HL2 : 48 dB

NS : 165701  
 NUCLEUS : <sup>13</sup>C  
 O1 : 13250.00 Hz  
 O2 : 6400.00 Hz

P[1] : 2.4 usec  
 PULPROG : zgpg

RG : 32768.000000  
 SFO1 : 100.6232500 MHz  
 SFO2 : 400.1364000 MHz

SOLVENT : CHCl<sub>3</sub>  
 SW : 216.0448 ppm  
 SW\_h : 21739.130 Hz  
 TD : 65536

\*\*\* Processing Parameters \*\*\*

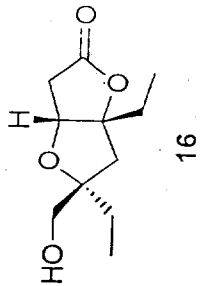
LB : 0.50 Hz  
 SI : 32768  
 TI :

\*\*\* ID NMR Plot Parameters \*\*\*

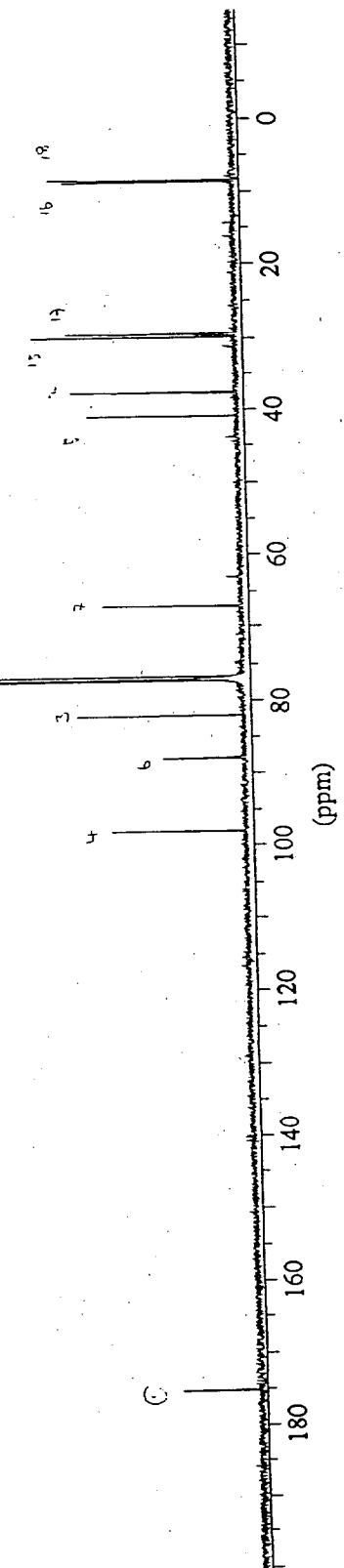
Start : 201.23 ppm  
 Stop : -14.83 ppm  
 SR : 3872.70 Hz  
 ppm\_cm : 10.29  
 Hz\_cm : 1035.20  
 AQ\_time : 1.5073280 sec  
 NUCLEUS : <sup>13</sup>C

51

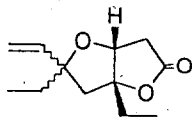
8.2599  
 8.5632  
 29.3665  
 29.7885  
 37.6351  
 40.7936  
 67.0764  
 76.6835  
 77.2044  
 77.3165  
 81.9981  
 87.9589  
 97.9814



175.2275



20

Compound **20** : HREIMS : C<sub>12</sub>H<sub>18</sub>O<sub>3</sub> requires 210.1255. Found : 210.1263.

$\delta$ (ppm)	multiplicity	J (Hz)
5.66/5.74	m (1H)	
5.10/5.19	m (2H)	
4.28	t	3.2
2.69	d	20
2.68	d	20
2.26	d	14
2.16	d	14
1.58/1.76	m (4H)	
0.96	t	7.2
0.85	t	7.4

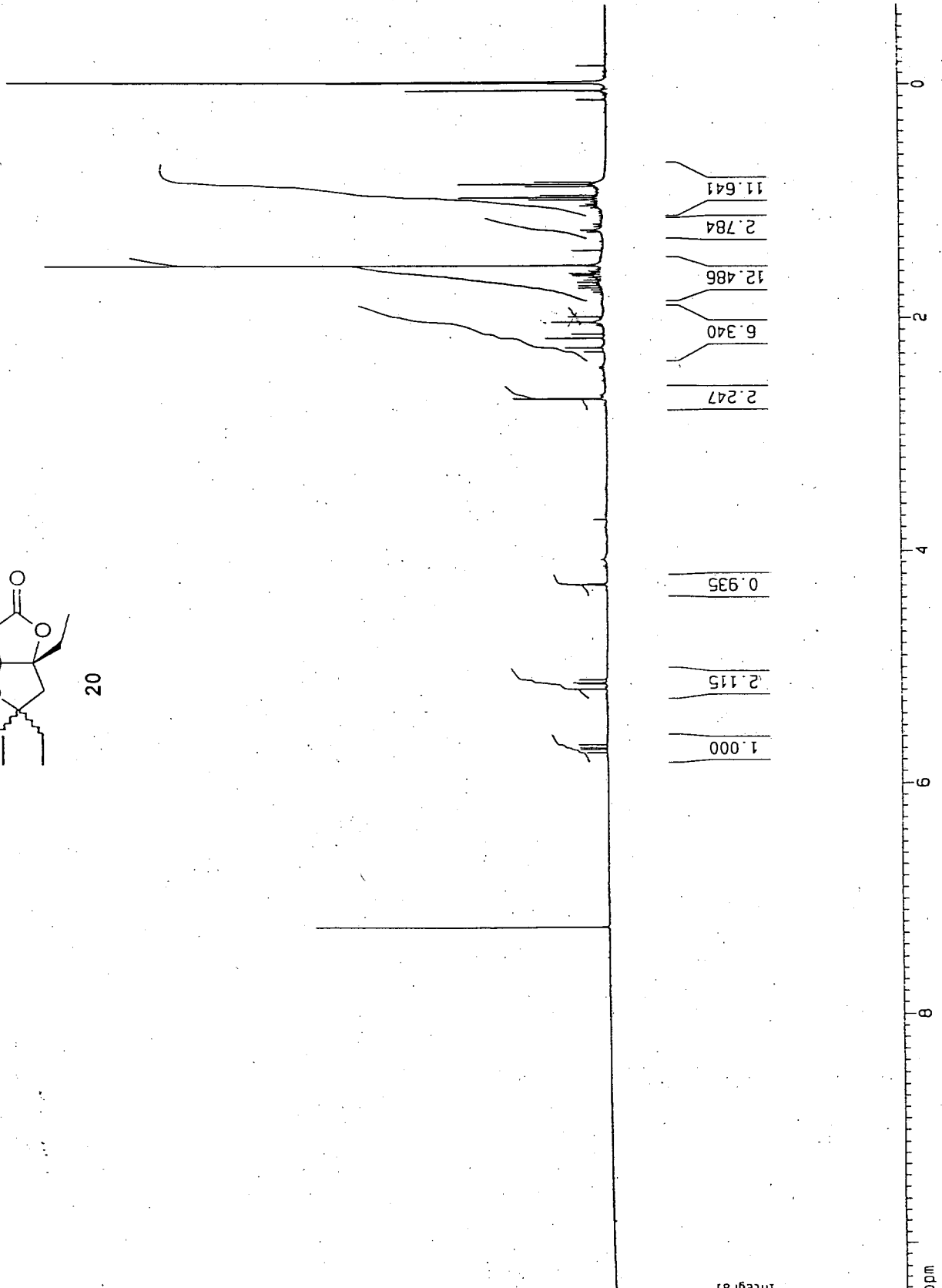
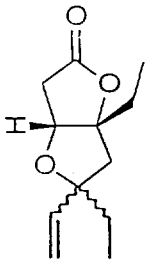
<sup>13</sup> C
175.4
141.4
113.4
97.3
86.9
80.2
45.8
36.8
33.4
30.2
8.4
8.3

PROCNO 1

F2 - Acquisition Parame  
 Date\_ 980210  
 Time 18.29  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TO 32768  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWH 4464.286  
 FIDRES 0.136239  
 AQ 3.6700661  
 RG 256  
 DW 112.000  
 DE 140.00  
 TE 305.0  
 HL1 0  
 D1 0.60000002  
 P1 3.60  
 DE 140.00  
 SFO1 400.1363559  
 NUCLEUS 1H

F2 - Processing paramet  
 SI 16384  
 SF 400.1344023  
 WDW no  
 SSB 0  
 LB 0.00  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00  
 F1P 10.461  
 F1 4185.79  
 F2P -0.696  
 F2 -278.49  
 PPMCM 0.50713  
 HZCM 202.92207



12

Current Data Parameters  
 NAME mh09fr8  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

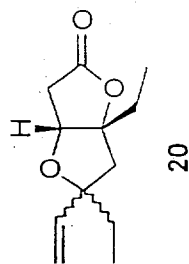
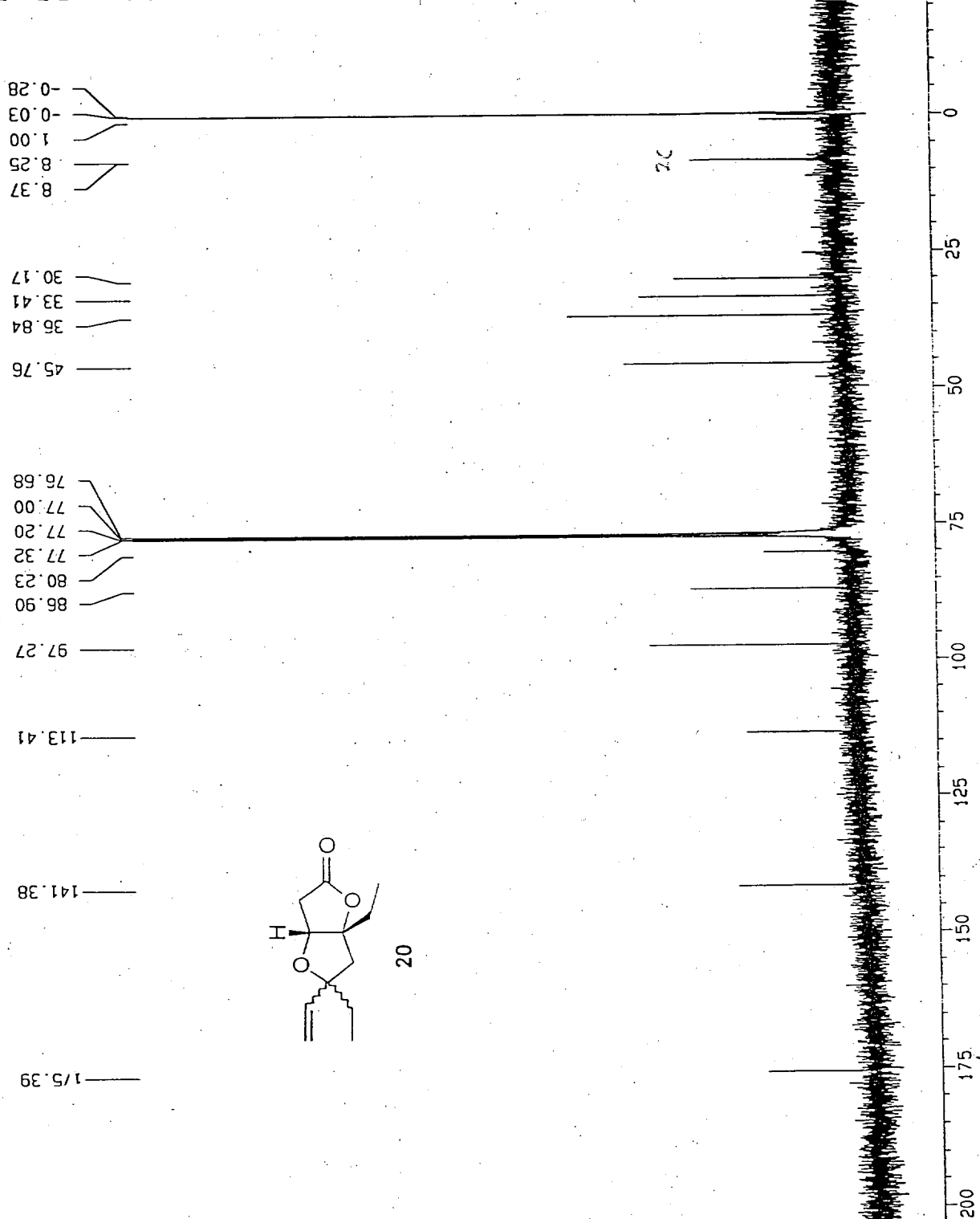
Date\_ 980210  
 Time 18.32  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CHCl3  
 NS 386585  
 DS 1  
 SMH 25000.000 Hz  
 FIDRES 0.381470 Hz  
 AQ 1.3107700 sec  
 RG 32768  
 DW 20.000 usec  
 DE 28.57 usec  
 TE 302.0 K  
 HL1 48 dB  
 D1 1.50000000 sec  
 CPOPRG waltz16  
 P31 100.00 usec  
 S4 44 dB  
 D11 0.0300000 sec  
 S2 31 dB  
 P1 2.40 usec  
 DE 28.57 usec  
 SFO1 100.6243000 MHz  
 NUCLEUS 13C

F2 - Processing parameters

SI 32768  
 SF 100.6138718 MHz  
 MDW EM  
 SSB 0  
 LB 0.50 Hz  
 GB 0  
 PC 1.00

ID NMR plot parameters

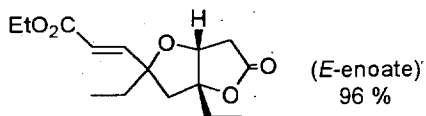
CX 22.00 cm  
 F1P 227.883 ppm  
 F1 22928.24 Hz  
 F2P -20.591 ppm  
 F2 -2071.76 Hz  
 PPMCM 11.29430 ppm/cm  
 HZCM 1136.36353 Hz/cm



22

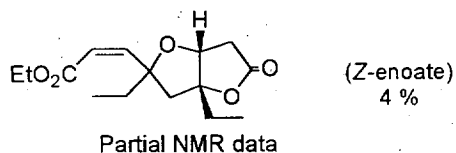
Compound **23** : Calc. for C<sub>15</sub>H<sub>22</sub>O<sub>5</sub> : C, 63.81 ; H, 7.85. Found C, 63.69 ; H, 8.31.

23



$\delta$ (ppm)	multiplicity	J (Hz)
6.77	d	15.7
5.97	d	15.7
4.32	t	3.0
4.18	q	7.0
2.70	d	3.0
2.39	d	14.4
2.15	d	14.4
1.75/1.79	m (4H)	
1.28	t	7.1
0.95	t	7.4
0.85	d	7.4

<sup>13</sup> C
175.0
166.3
150.6
119.7
96.7
86.3
80.0
60.6
46.1
39.6
33.1
30.1
14.2
8.4
8.2



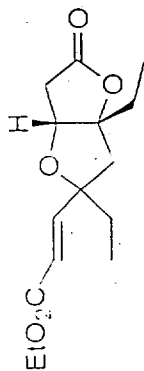
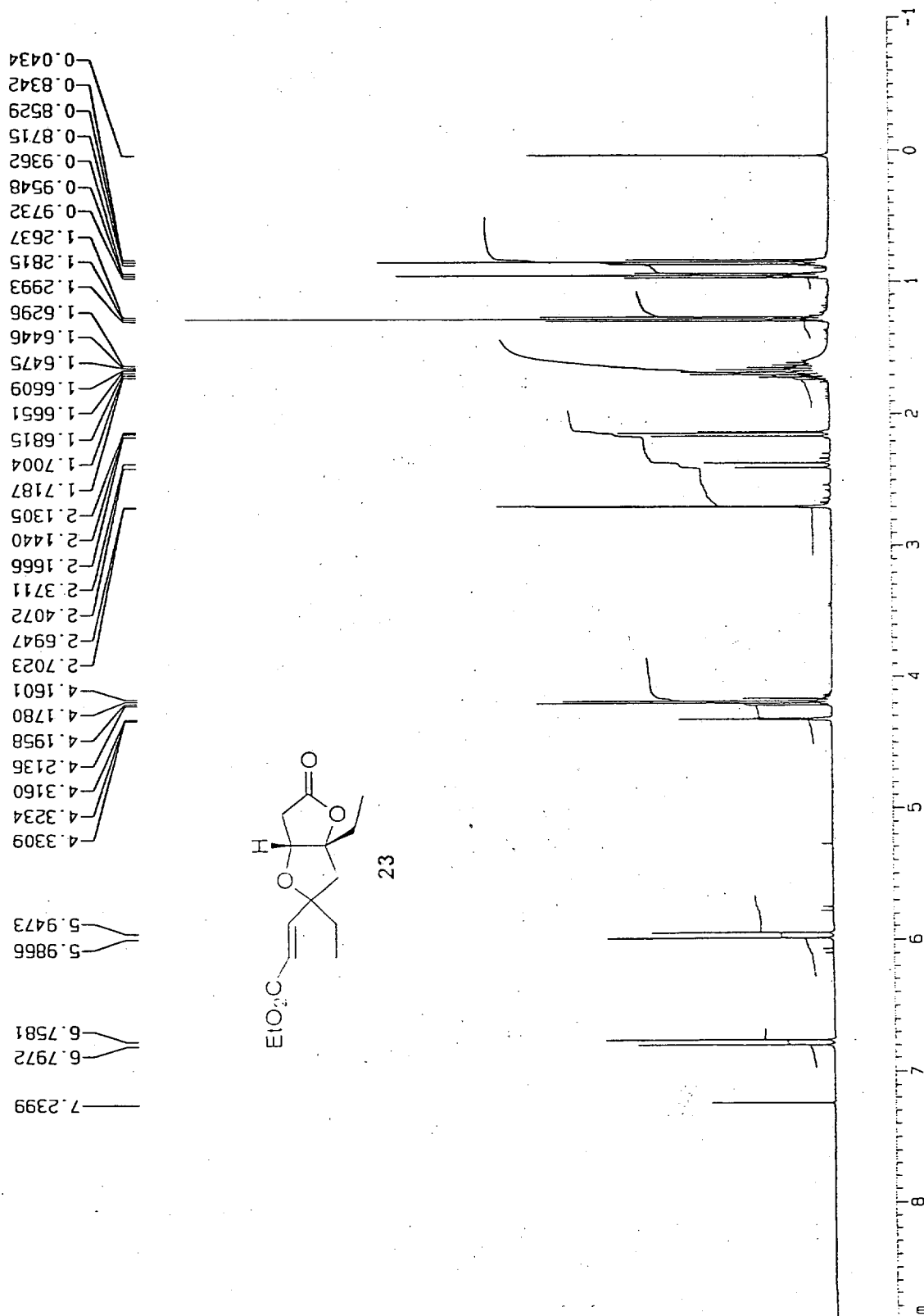
$\delta$ (ppm)	multiplicity	J (Hz)
6.07	d	12.6
5.76	d	12.6
2.67	d	4.9
2.55	d	14.7
2.31	d	14.7
0.96	t	7.6
0.88	t	7.4

Current Data Parameters  
 NAME esterp127  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 990903  
 Time 13.35  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zg  
 TD 32768  
 SOLVENT CDCl3  
 NS 27  
 OS 0  
 SMH 4807.692 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4079220 sec  
 RG 512  
 \*OM 104.000 usec  
 DE 148.57 usec  
 TE 305.0 K  
 HL1 0 dB  
 D1 0.6000002 sec  
 P1 3.70 usec  
 DE 148.57 usec  
 SFO1 400.1364000 MHz  
 NUCLEUS 1H

F2 - Processing parameters  
 SI 16384  
 SF 400.1344024 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 9.043 ppm  
 F1 3618.39 Hz  
 F2P -1.400 ppm  
 F2 -560.19 Hz  
 PPMCH 0.47458 ppm/1  
 HZCM 189.93513 Hz/1



27

Current Data Parameters  
 NAME esterp127  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 990903  
 Time 13.42  
 INSTRUM amx400  
 PROBHD 5 mm Multinu  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CHCl3  
 NS 1323  
 DS 1  
 SMH 26315.789 Hz  
 FIDRES 0.401547 Hz  
 AQ 1.2452340 sec  
 RG 32768  
 DW 19.000 usec  
 DE 27.14 usec  
 TE 302.0 K  
 HL1 48 dB  
 D1 1.7999995 sec  
 CPOPRG waltz16  
 P31 100.00 usec  
 S4 44 dB  
 O11 0.0300000 sec  
 S2 27 dB  
 P1 2.40 usec  
 DE 27.14 usec  
 SF01 100.6254000 MHz  
 NUCLEUS 13C

F2 - Processing parameters  
 SI 32768  
 SF 100.6138734 MHz  
 MDW EM  
 SSB 0  
 LB 0.40 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.00 cm  
 FJP 189.567 ppm  
 F1 19073.07 Hz  
 F2P -15.572 ppm  
 F2 -1566.76 Hz  
 PPHCM 9.32450 ppm/cm  
 HZCM 938.17432 Hz/cm

