The First Total Synthesis of (±)-Eremopetasidione

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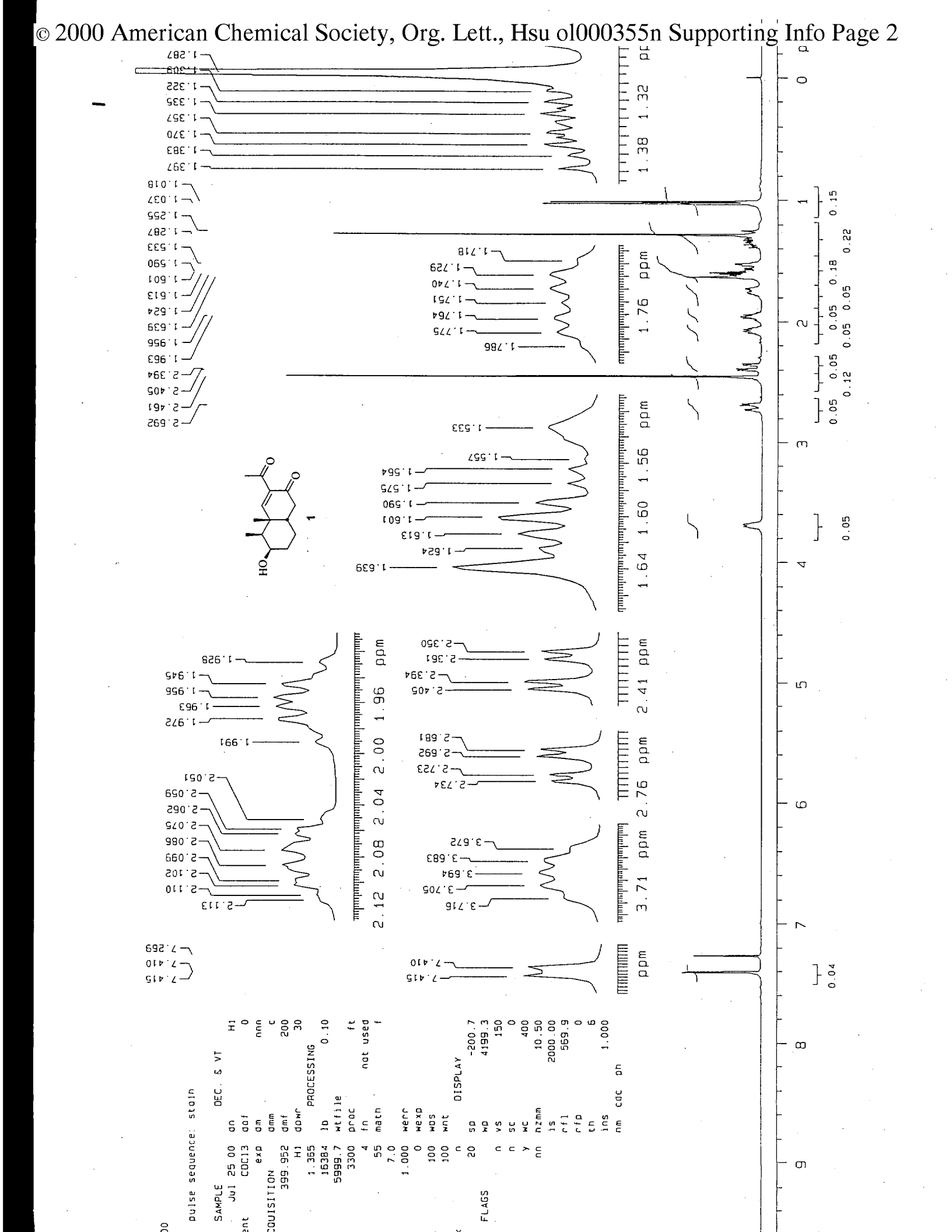
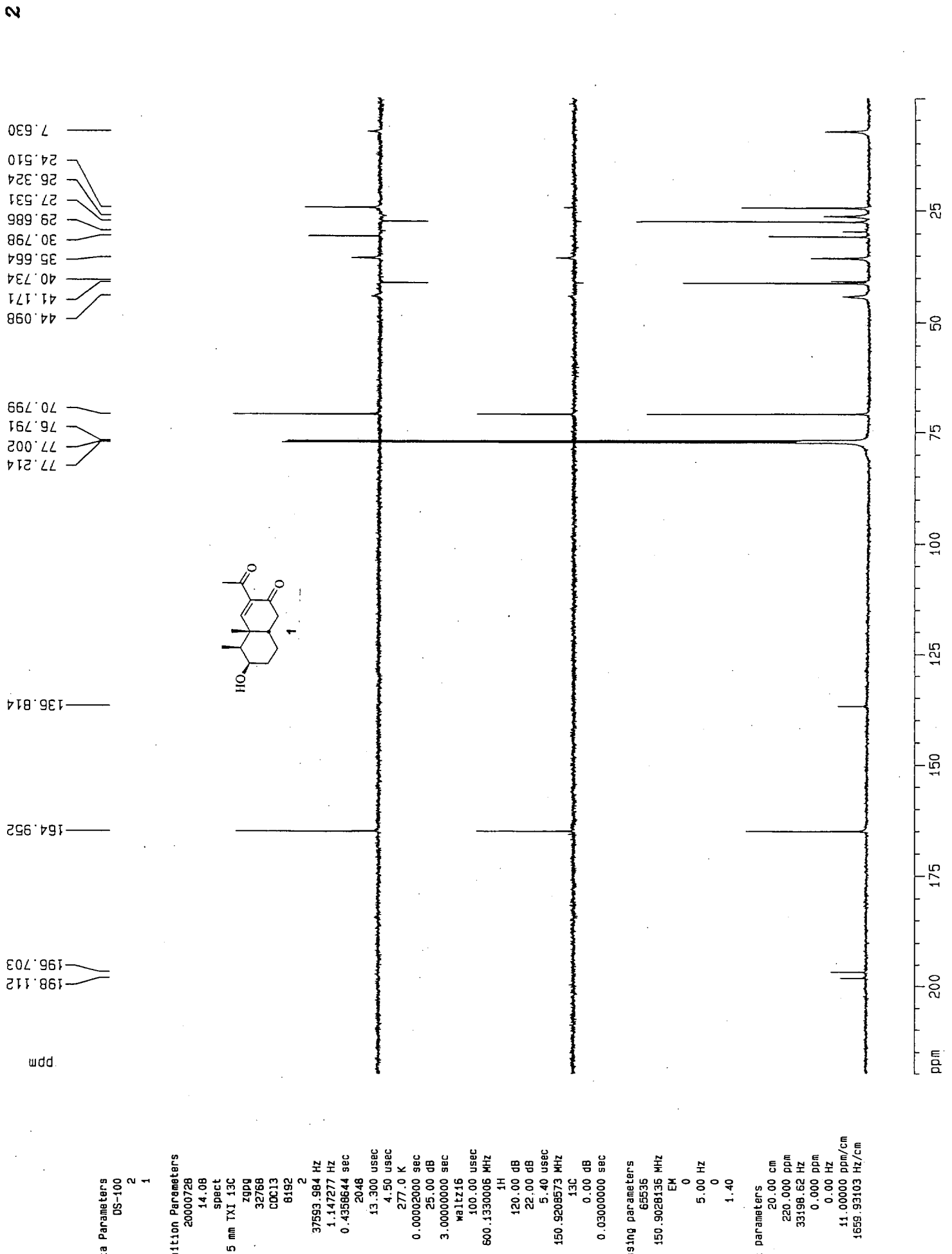
Supporting Information

^{1}H (400 MHz) and ^{13}C (100 MHz) NMR and DEPT Spectra for Compounds **1-4** and **7-11**, ^{1}H-^{1}H and ^{1}H-^{13}C COSY spectra for compound **1** and X-ray crystallographic data for compound **8**

23 pages (excl. this page)

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7.630
24.510
26.324
27.531
29.686
30.798
35.664
40.734
41.171
44.098
70.799
76.791
77.002
77.214
136.814
164.952
196.703
198.112
ppm
25
50
75
100
125
150
175
200
ppm
O
O
OH
1
Data Parameters
DS-100
2
1
uisition Parameters
20000728
14.08
spect
5 mm TXI 13C
zgpg
32768
CDCl3
8192
2
37593.984 Hz
1.147277 Hz
0.4356644 sec
2048
13.300 usec
4.50 usec
277.0 K
0.00002000 sec
25.00 dB
3.00000000 sec
waltz16
100.00 usec
600.1330006 MHz
1H
120.00 dB
22.00 dB
5.40 usec
150.9208573 MHz
13C
0.00 dB
0.03000000 sec
cessing parameters
65536
150.9028135 MHz
EM
0
5.00 Hz
0
1.40
lot parameters
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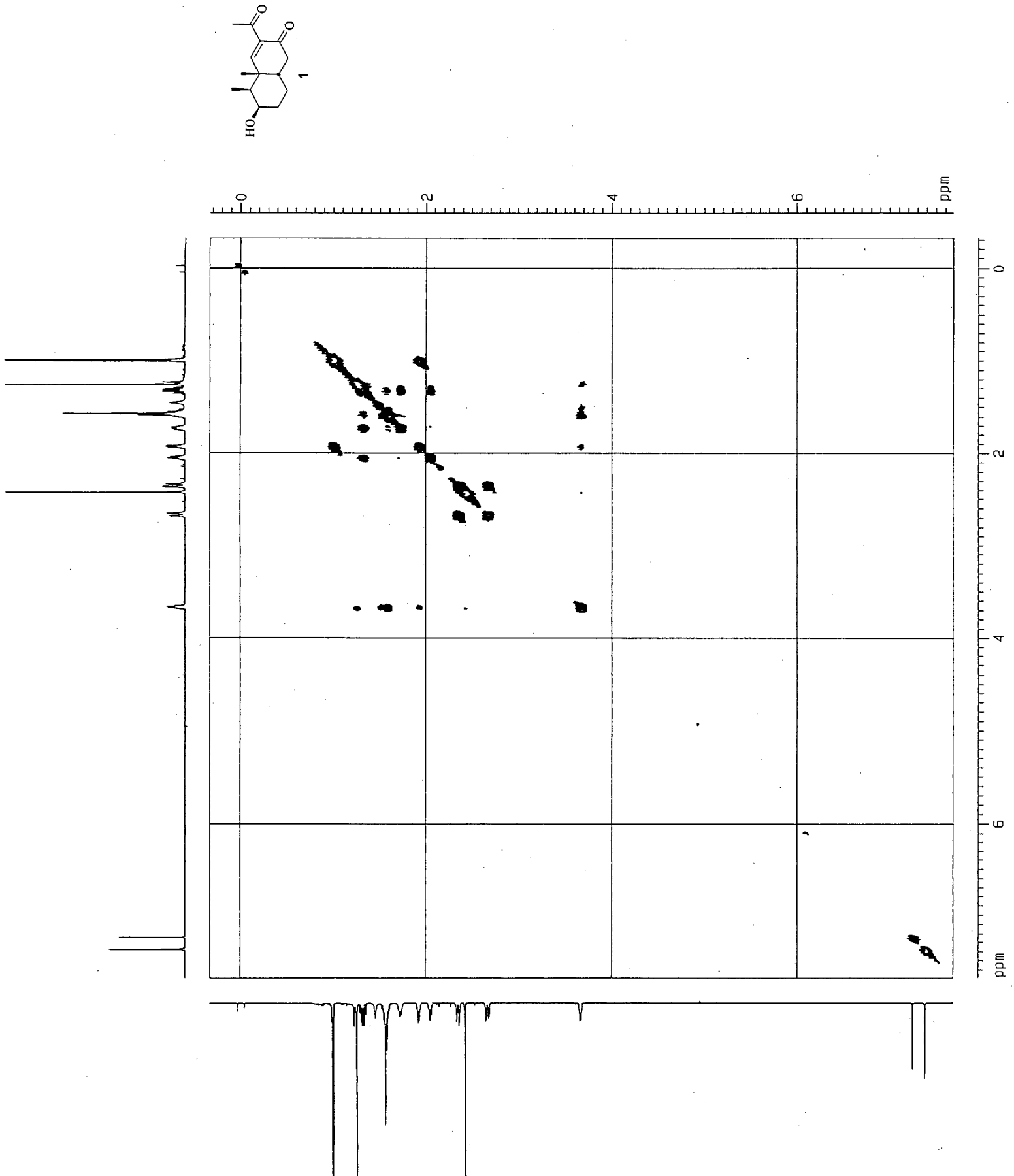
220.000 ppm
33198.62 Hz
0.000 ppm
0.00 Hz
11.00000 ppm/cm
1659.93103 Hz/cm

3



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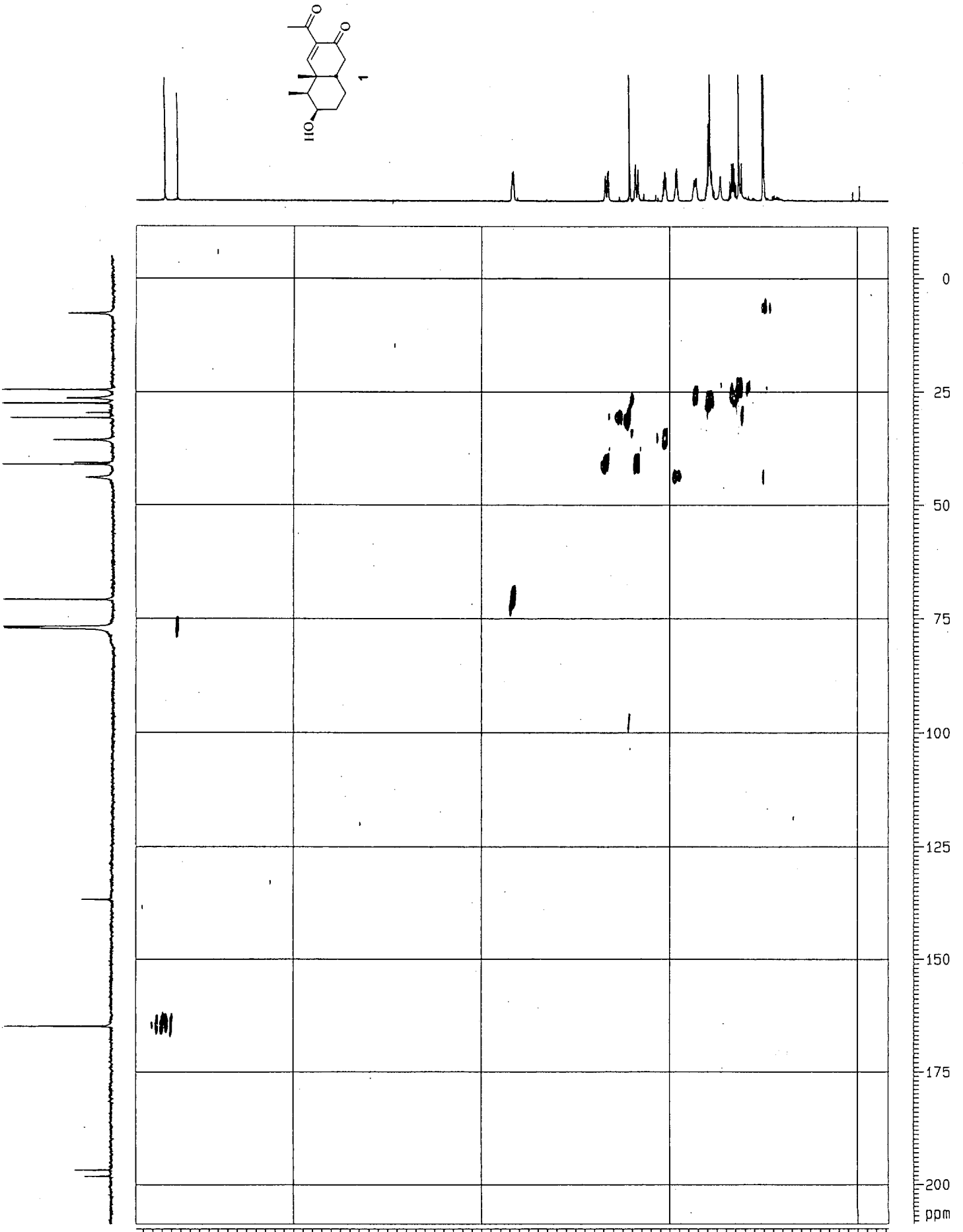

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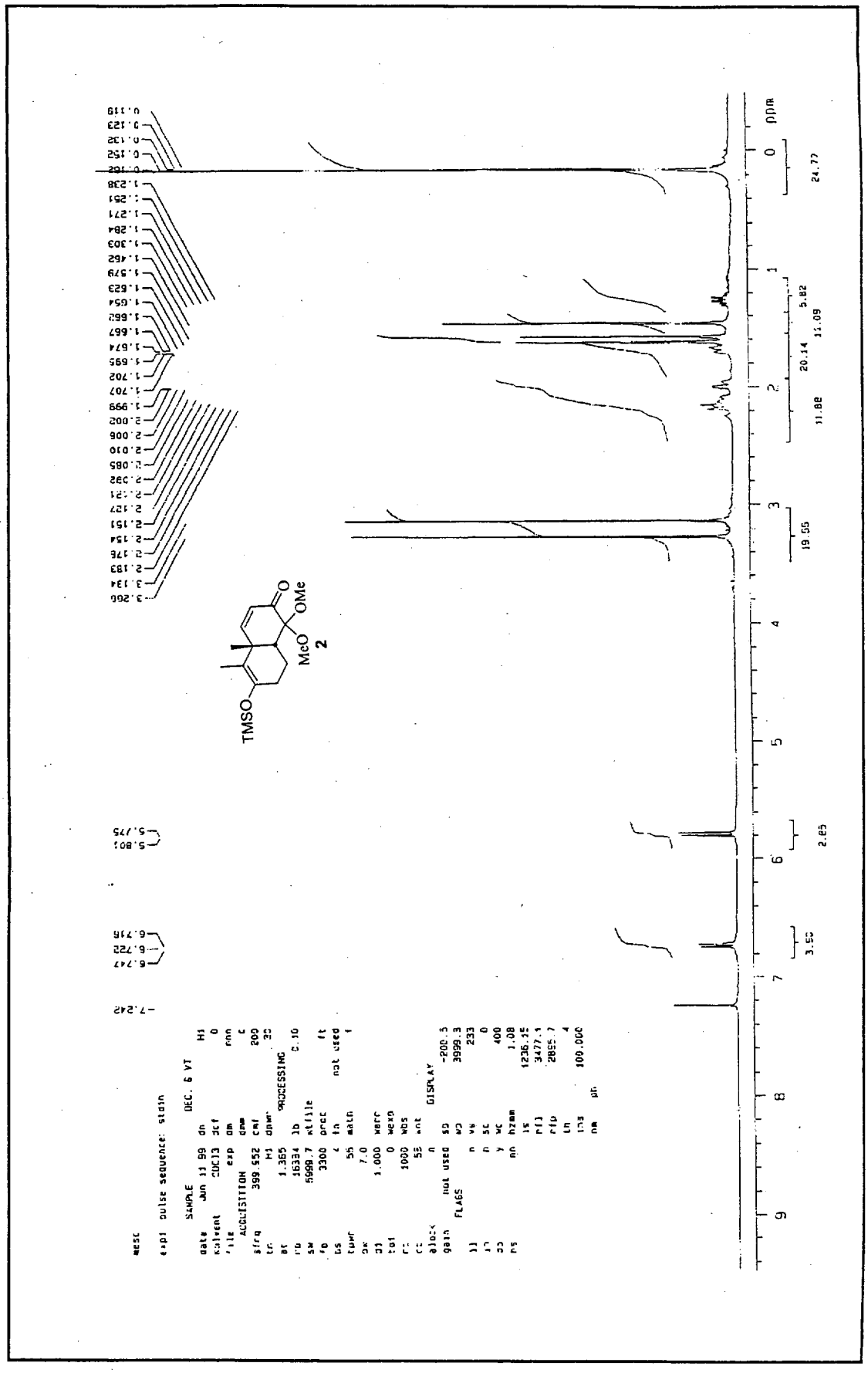

b
0.573
10.617
20.972
26.729
30.067
42.697
45.975
47.241
50.154
76.675
76.993
77.312
99.758
116.280
122.493
145.328
153.368
192.981
TMSO
MeO
OMe
O
2
pulse sequence: dept
SAMPLE
Mar 25 97
COC13
exp
UISITION
100.577
C13
1.199
59958
13800
8
1
21.0
55
9.0
55
2.000
0
151.00
arrayed
0
500
120
DEC. & VT
H1
-999.9
nny
ccs
9900
39
PROCESSING
1.00
proc
ft
fn
not used
math
f
werr
wexp
deptp
wbs
wnt
DISPLAY
-1005.9
22126.7
27804
0
400
55.32
ACQUISITION ARRAYS
array
arraydim
mult
2
1
1.5
dn
dof
dm
dmm
dmf
dpwr
lb
wtfile
sp
wp
vs
sc
wc
hzmm
FLAGS
rfl
rfp
th
ins
ai
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140
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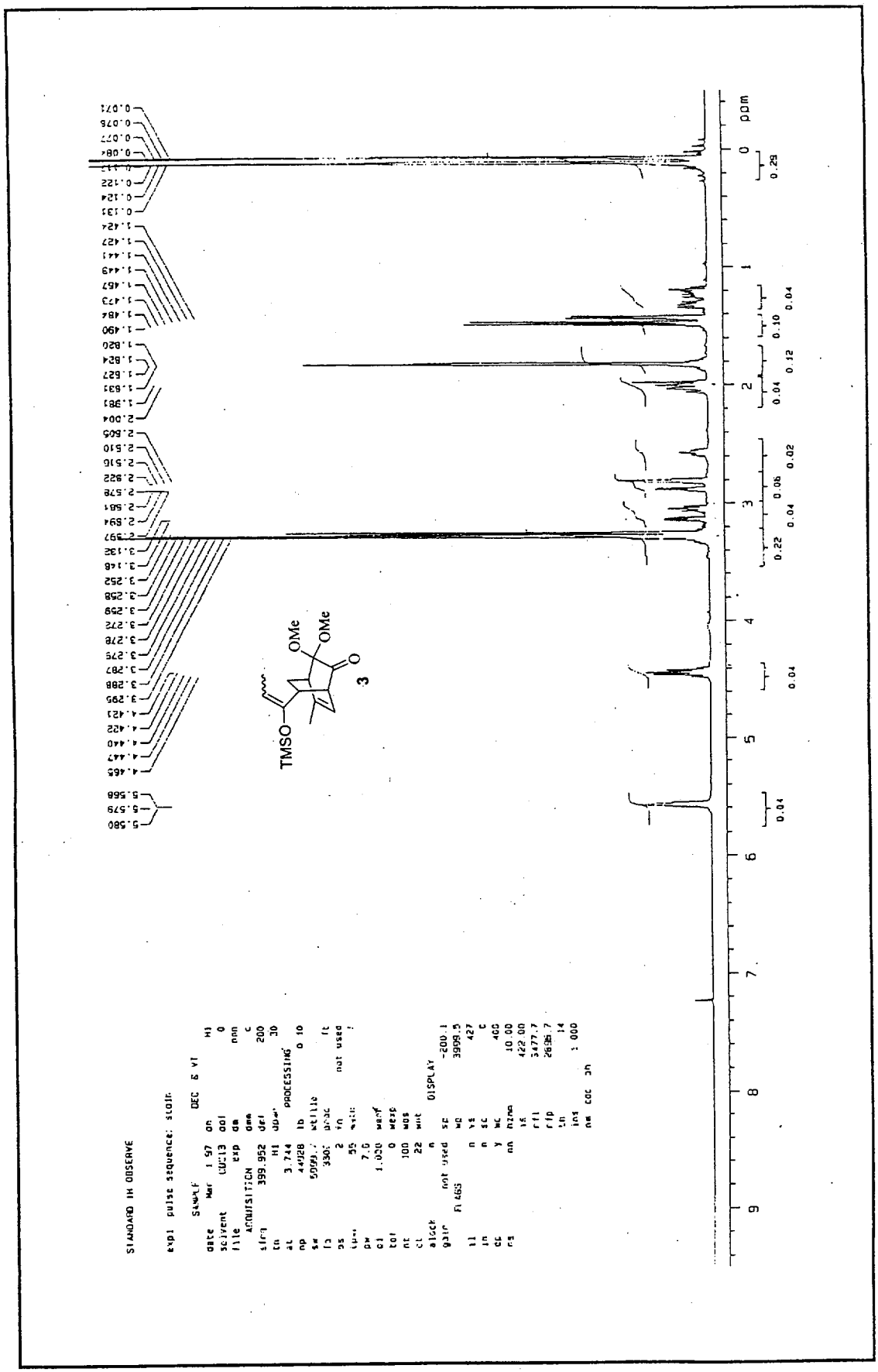
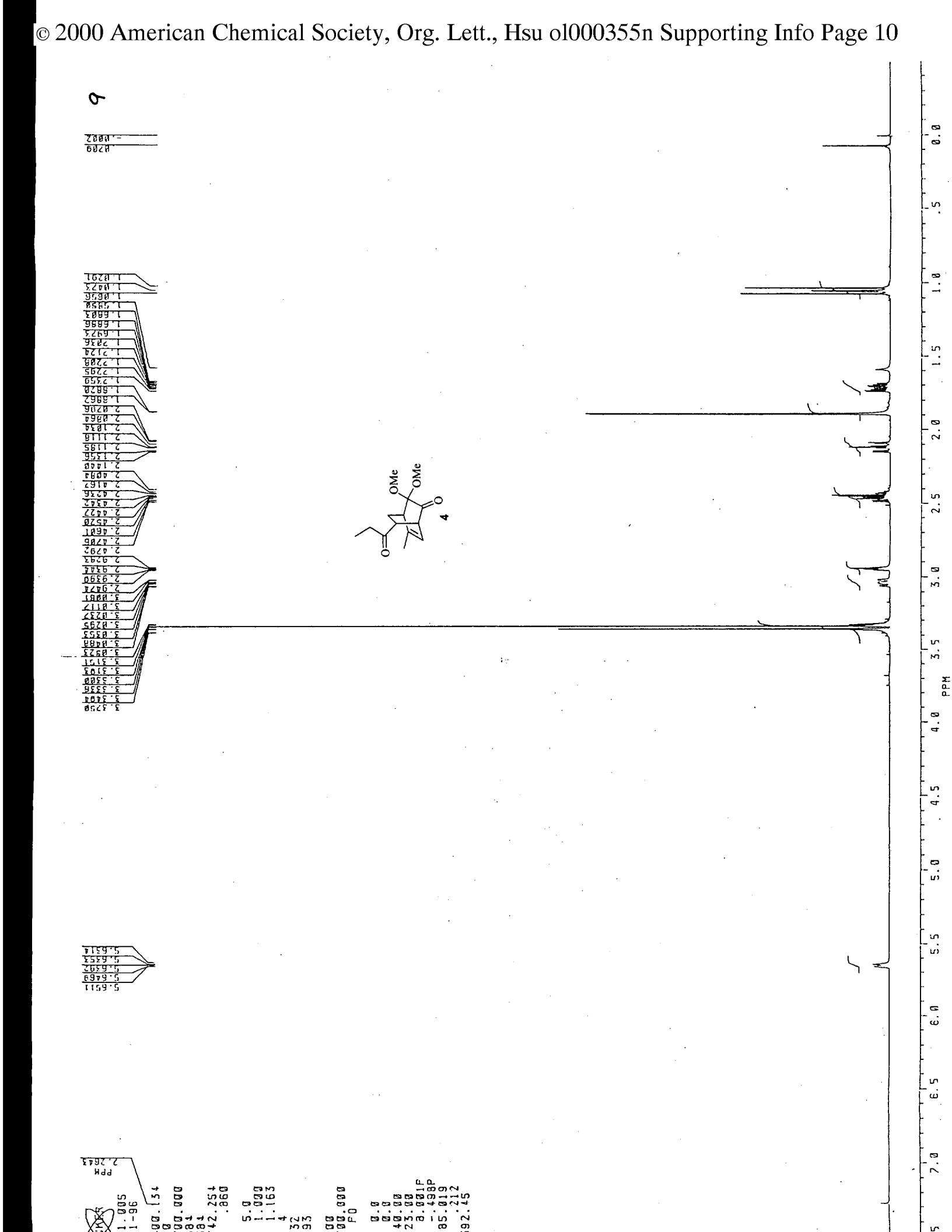
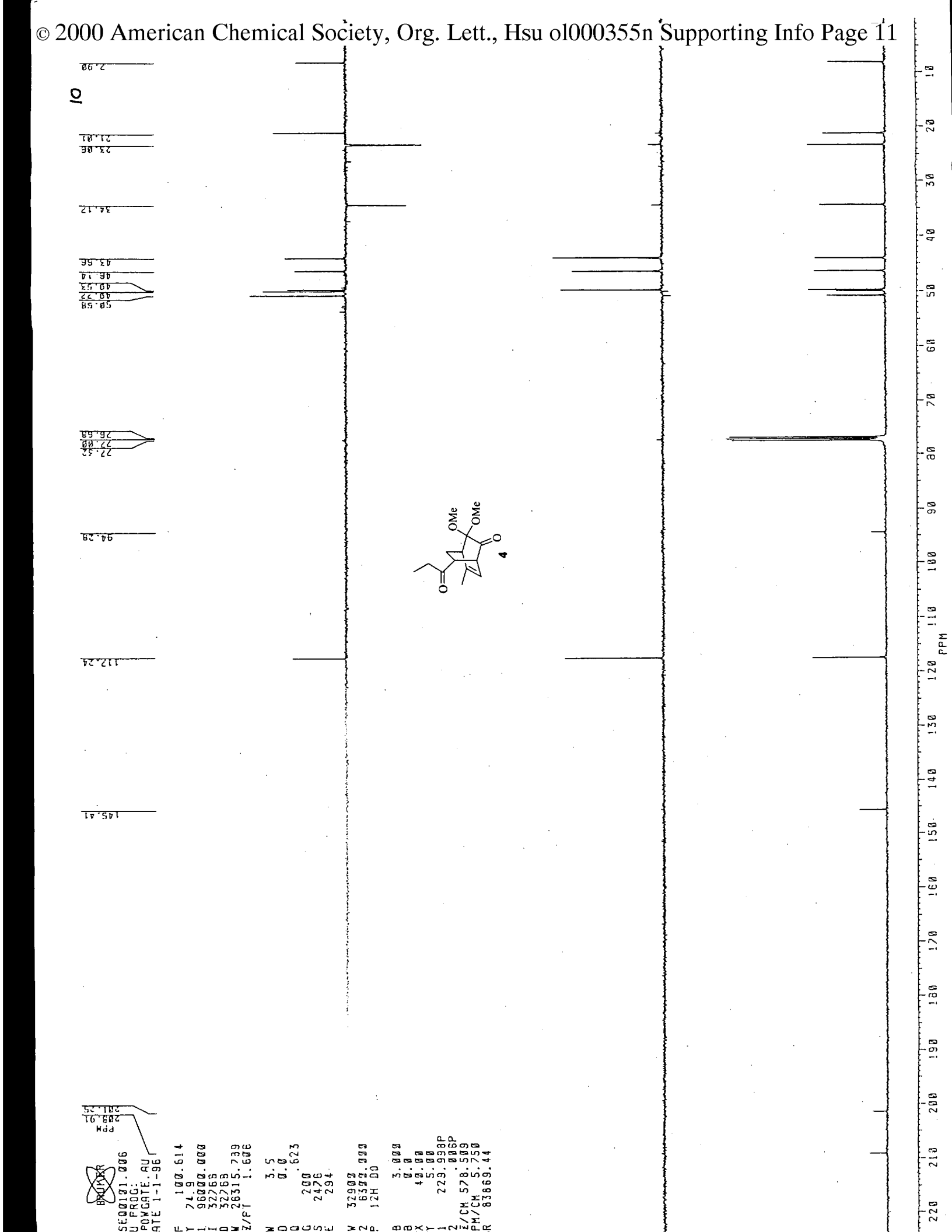
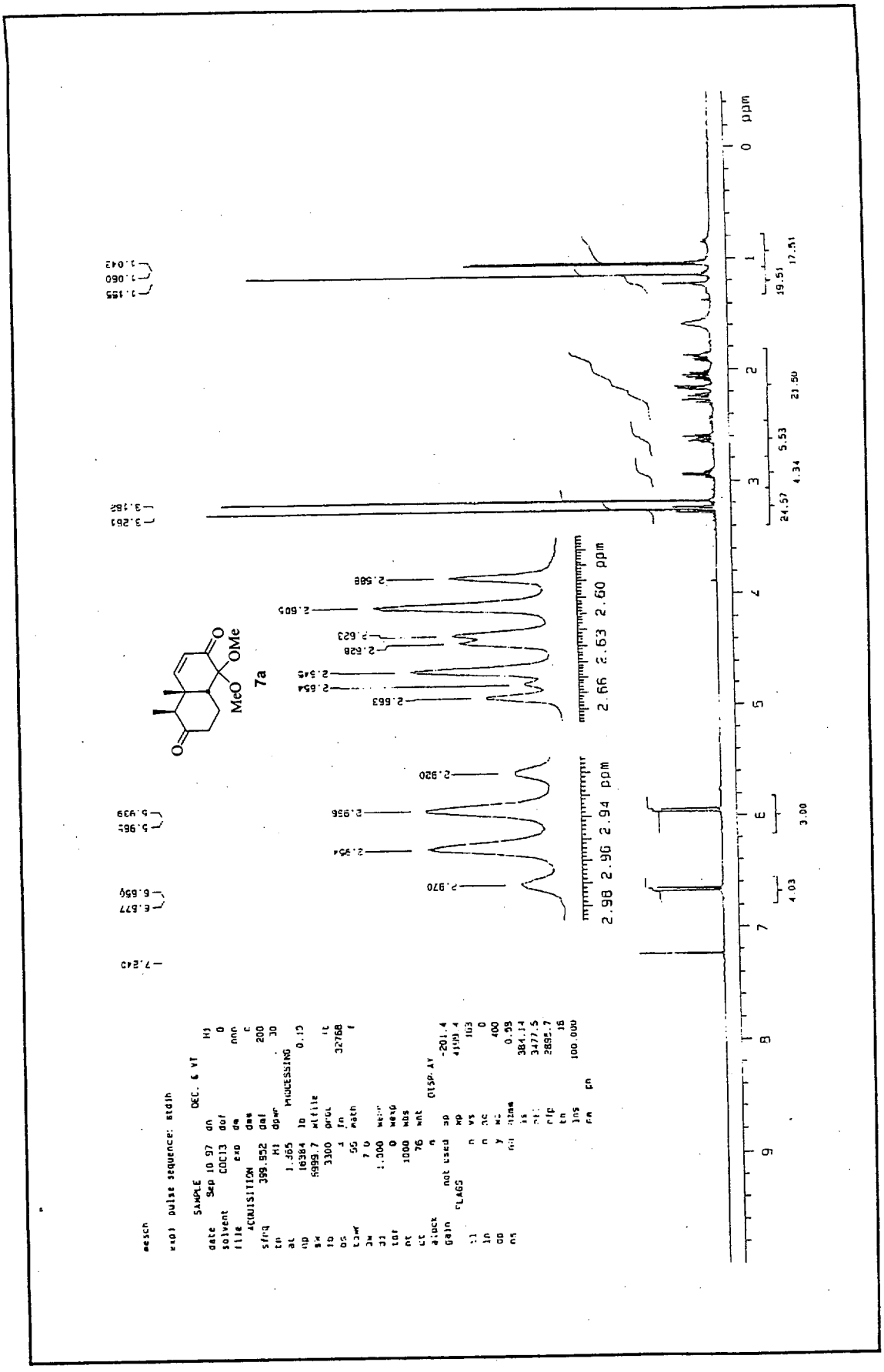
180
pp

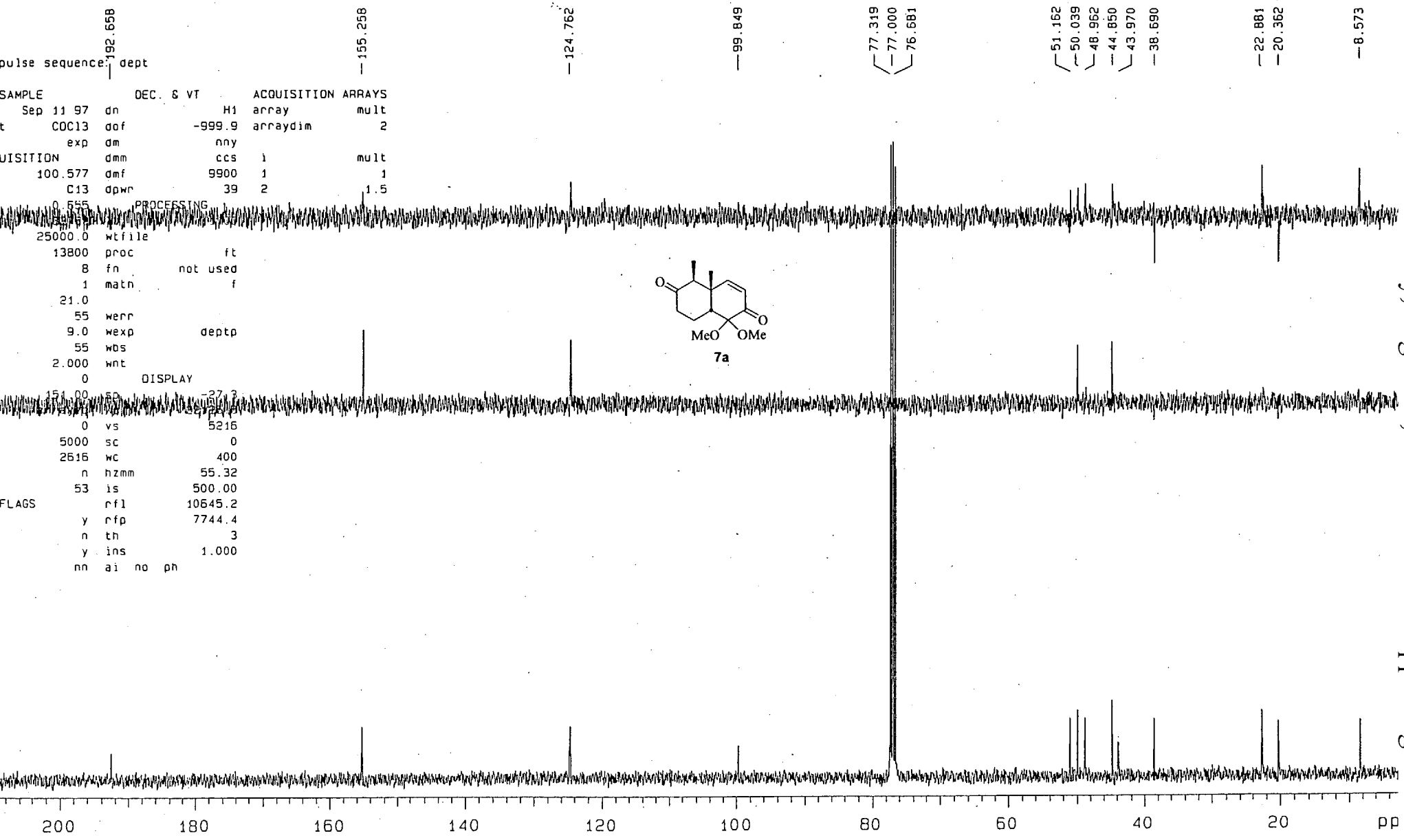
7

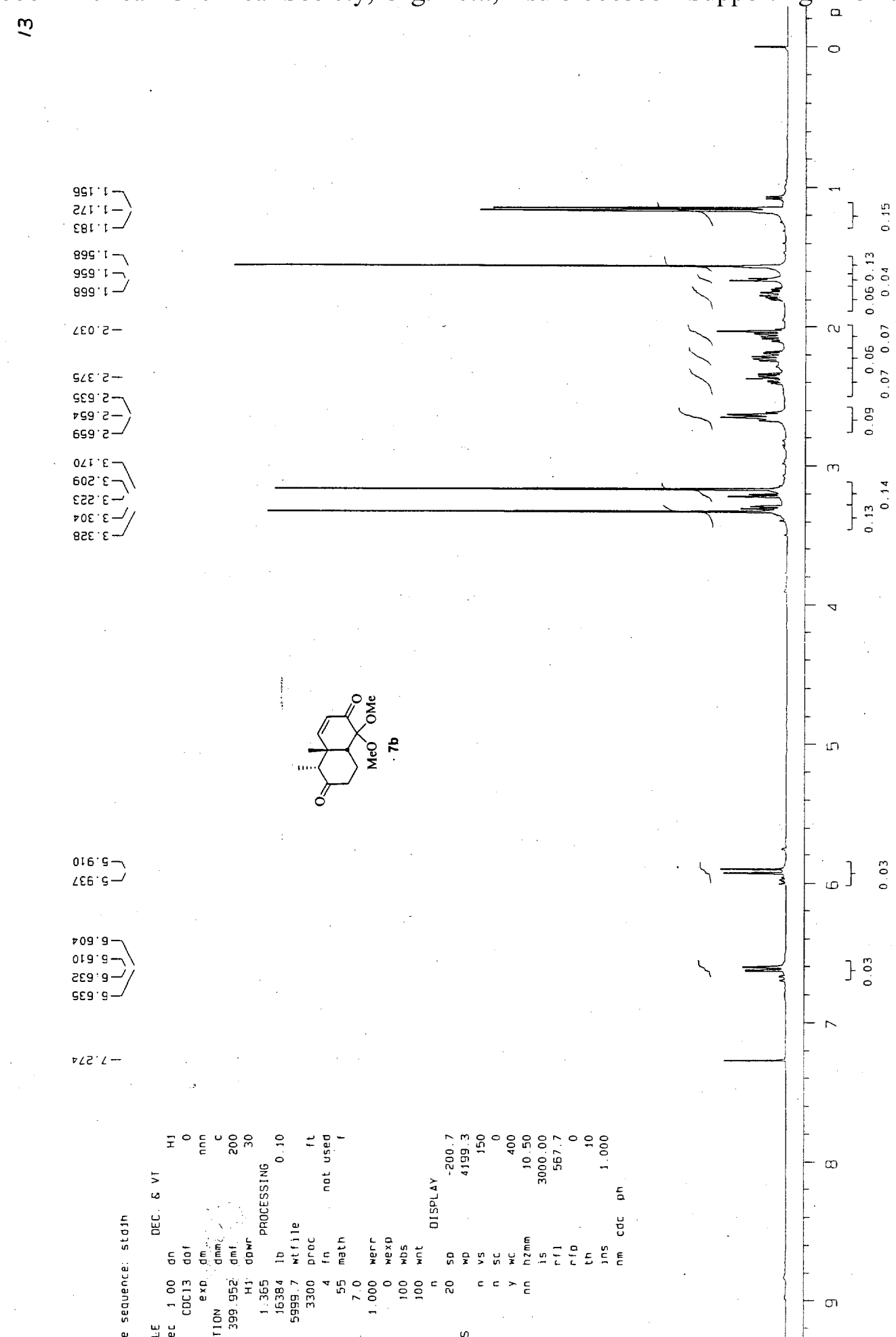





OMe
OMe
O
O
4
PPM







14
7b
MeO
OMe
O
O
77.319
77.000
76.854
76.681
53.381
50.222
47.527
45.920
45.537
38.103
28.123
22.551
99.390
126.241
150.811
192.435
pulse sequence: std13c
SAMPLE
DEC. & VT
ACQUISITION
PROCESSING
DISPLAY
FLAGS
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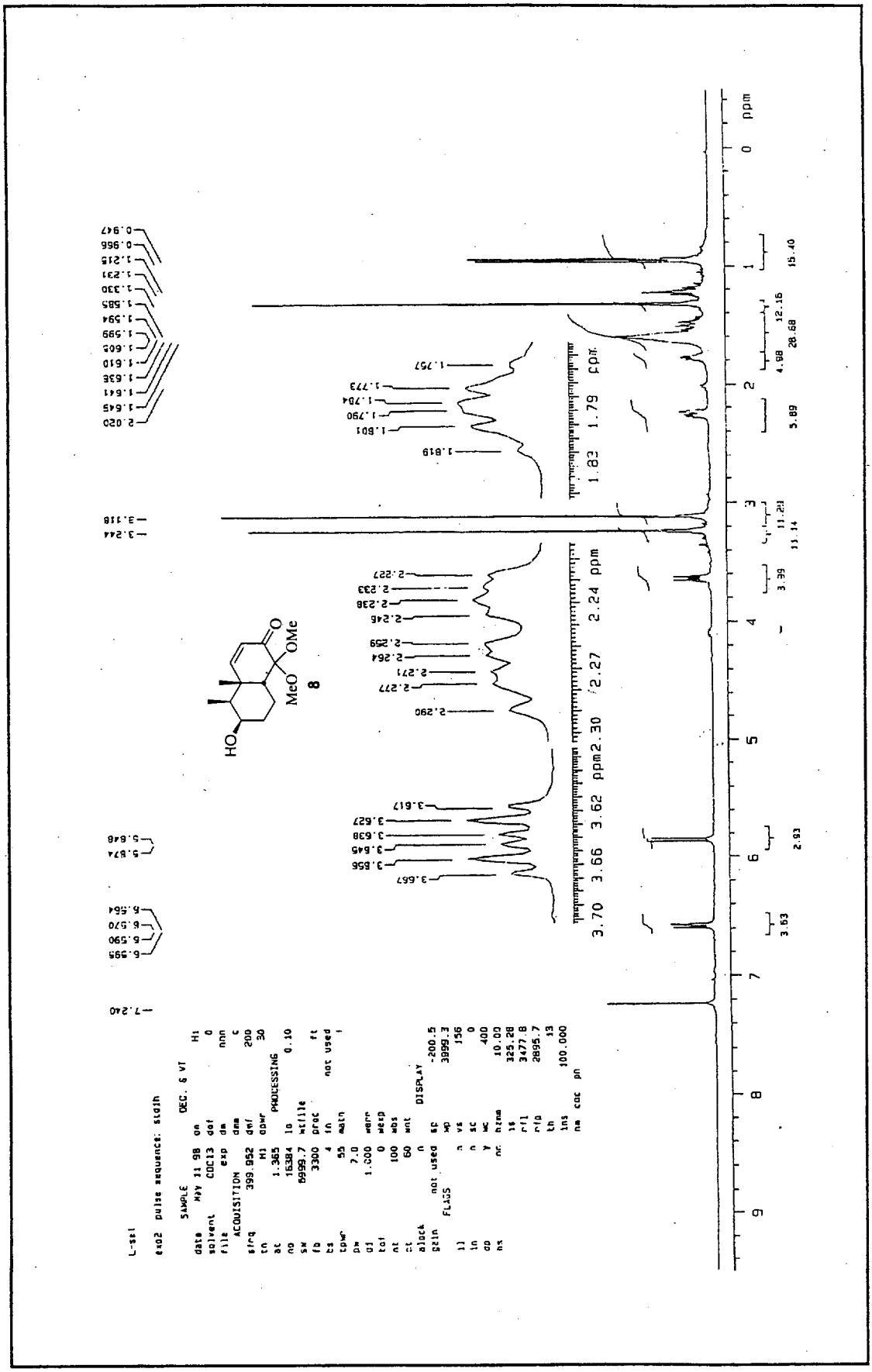
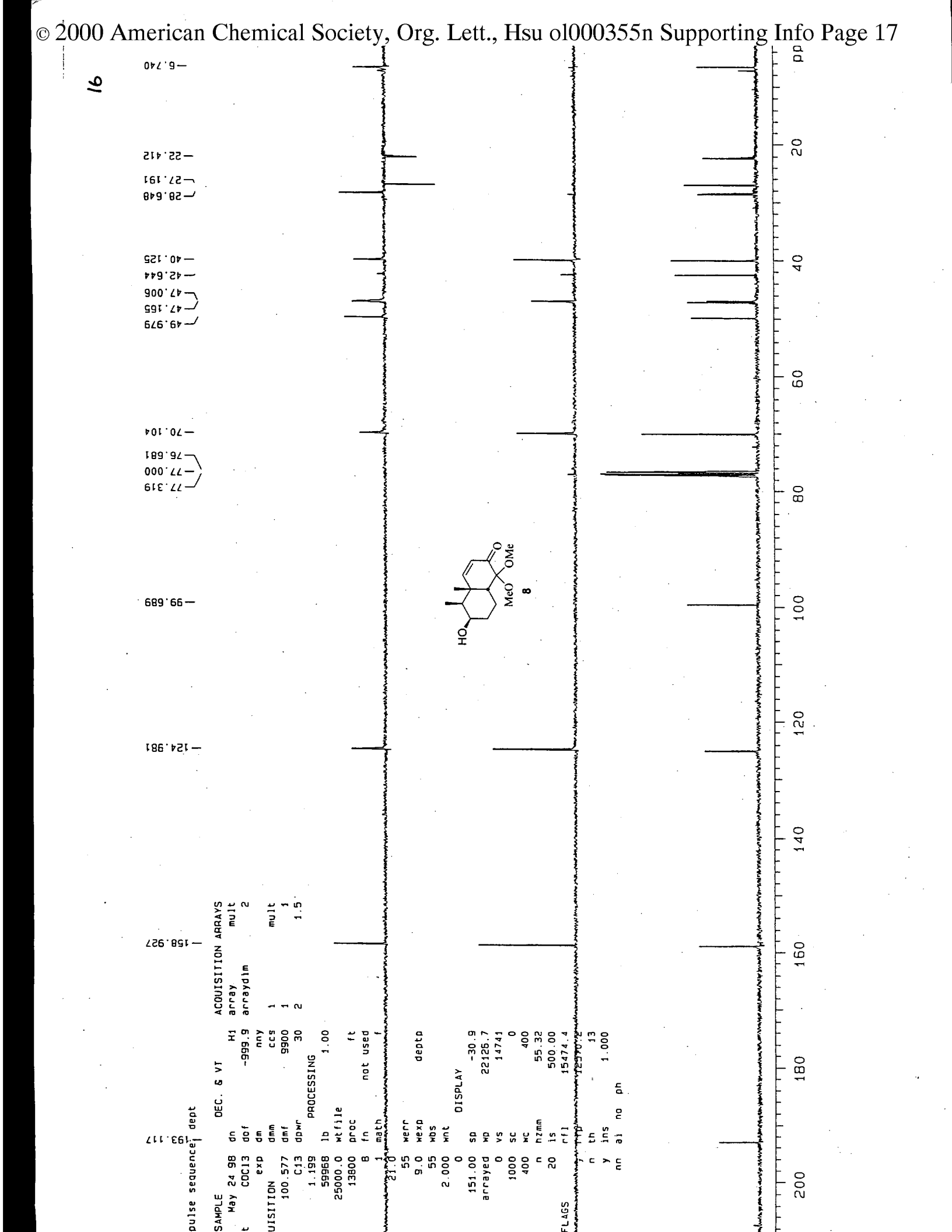
180
160
140
120
100
80
60
40
20
0

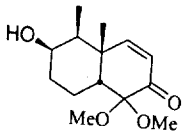
15





Summary of Crystal Data and Intensity Collection 98spl3 (980195)

Empirical Formula	$C_{14}H_{22}O_4$
Color; Habit	Colorless ; Bladed
Crystal Size (mm)	0.70 x 0.40 x 0.25
Space Group	$P2_1/n$; Monoclinic
Unit Cell Dimensions	a = 8.4285(2) Å b = 13.9535(1) Å; β = 90.956(1)° c = 11.5719(3) Å
Volume	1360.7(4) $Å^3$
Z	4
Formula Weight	254.3
Density(calc.)	1.241 Mg/m^3
Absorption Coefficient	0.090 mm^{-1}
F(000)	552
Diffractometer Used	Siemens Smart CCD
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	296
2θ Range	2.5 to 55.6°
Scan Speed	10.00 seconds/ frame
Scan Range (ω)	0.30°/ frame
Index Ranges	$-10 \le h \le 10, -17 \le k \le 15, -12 \le \ell \le 15$
Reflections Collected	7486(5576≥3.0σ(I))
Independent Reflections	2891(1527≥3.0Gs(I))(R_{int}=4.06%)
System Used	Siemens SHELXTL PLUS (VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Extinction Correction	χ = 0.0010(4), where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Located on difference map
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0005F^2$
Number of Parameters Refined	164
Final R Indices (obs. data)	R = 0.0455, Rw = 0.0517
Goodness-of-Fit	1.54
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	9.3:1
Largest Difference Peak/hole	0.16/-0.14 $eÅ^{-3}$



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