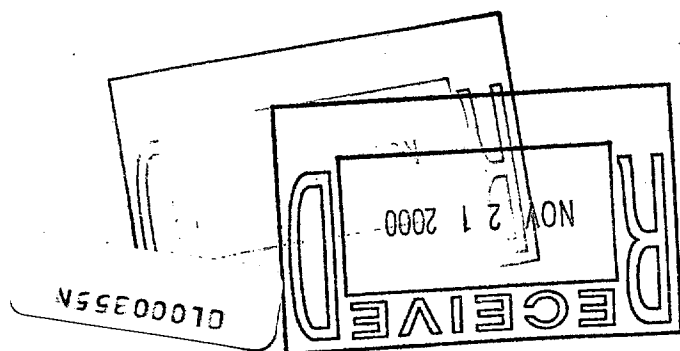
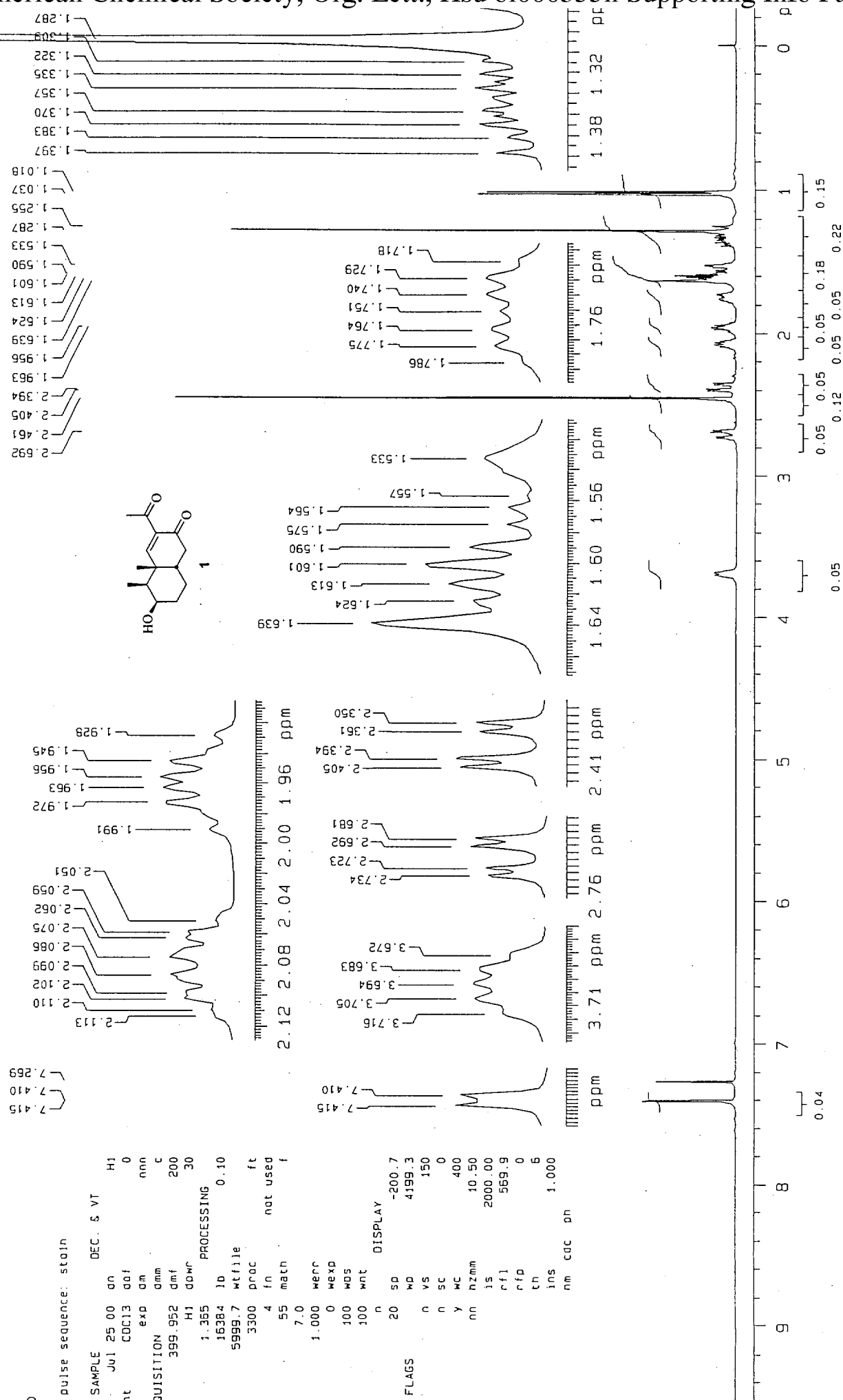


The First Total Synthesis of ($\pm$)-Eremopetasidione
Day-Shin Hsu, Po-Yi Hsu, and Chun-Chen Liao*

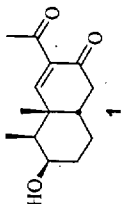
Supporting Information

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

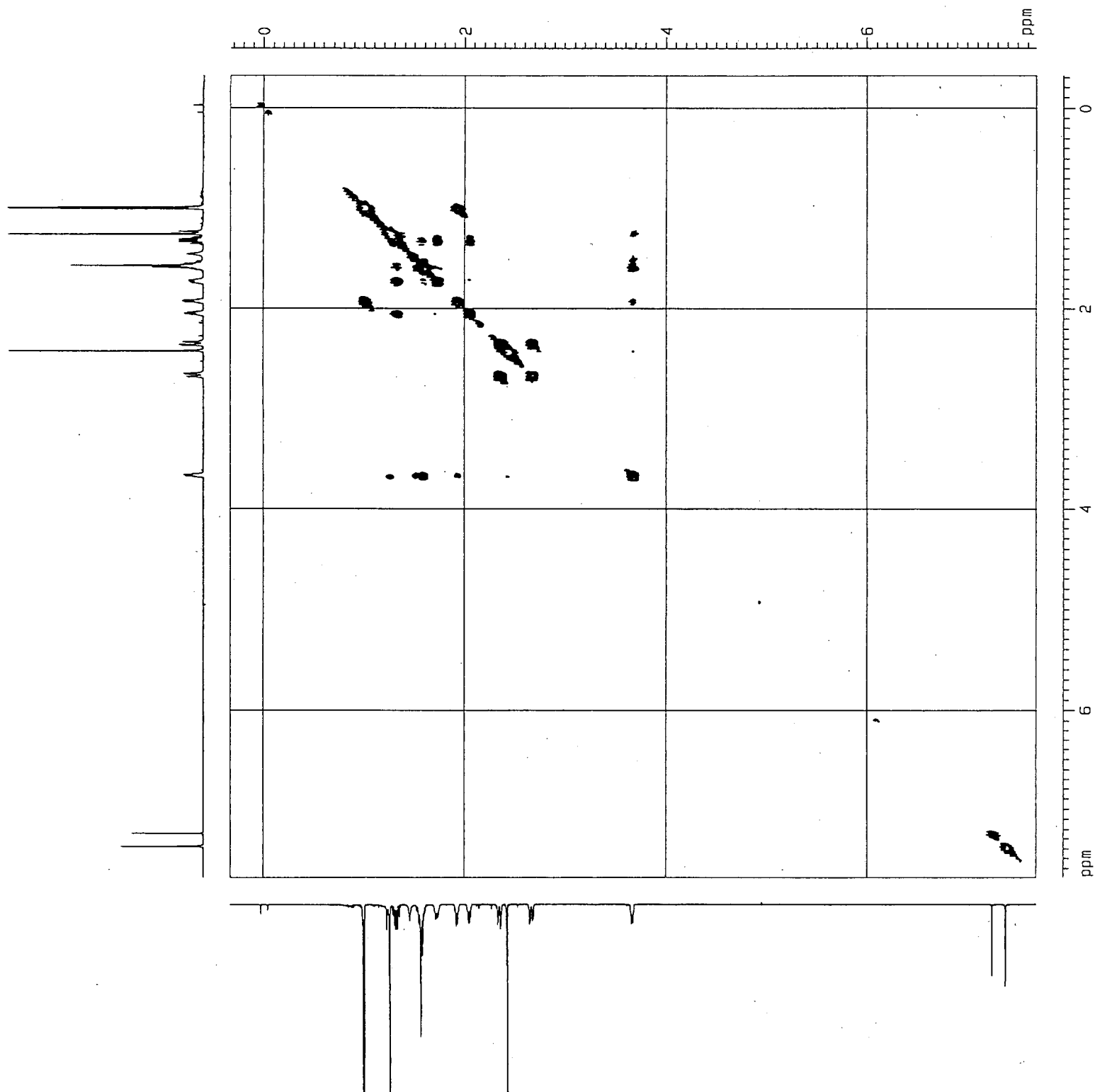
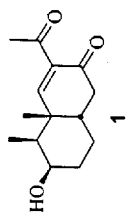




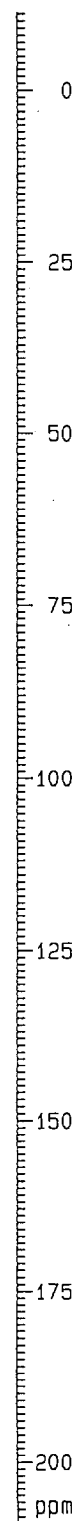
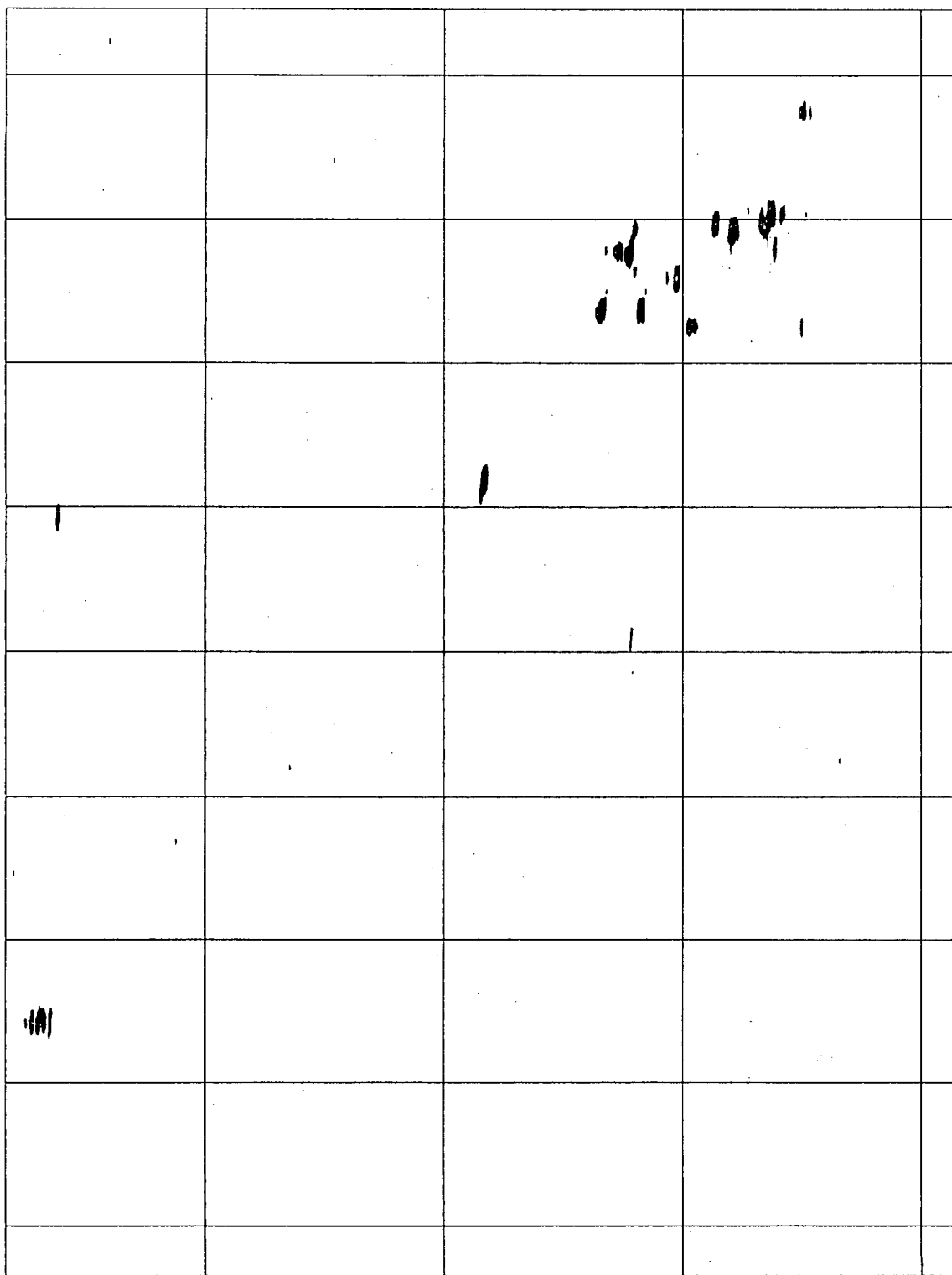
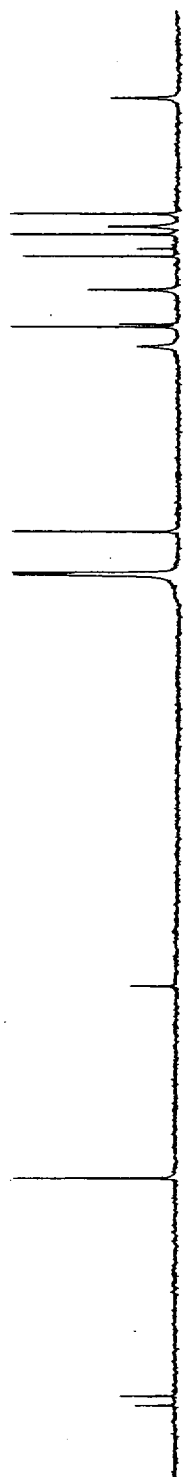
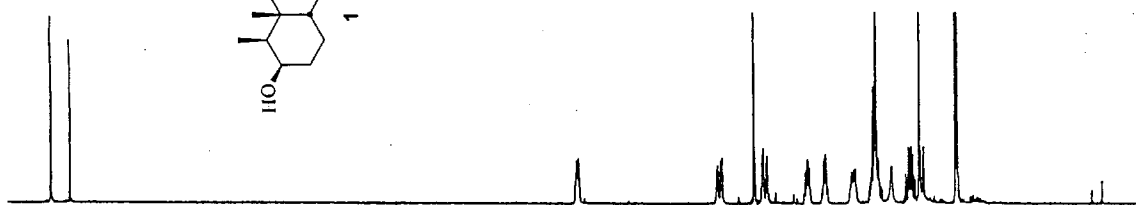
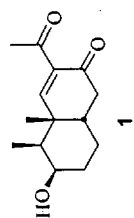
pulse sequence: stoin
 SAMPLE Jul 25 00 dn H1
 Cocent COC13 daf 0
 exp dm nnn
 ACQUISITION dmm c
 399.952 dmf 200
 H1 dmr 30
 PROCESSING
 1.355 1b 0.10
 16384
 5999.7 wfile
 3300 proc ft
 4 fn not used f
 55 math
 7.0
 1.000 werr
 0 wexp
 100 wos
 100 wnt
 DISPLAY
 20 sp -200.7
 4199.3
 150
 0
 400
 10.50
 2000.00
 569.9
 0
 6
 1.000
 nm coc pn



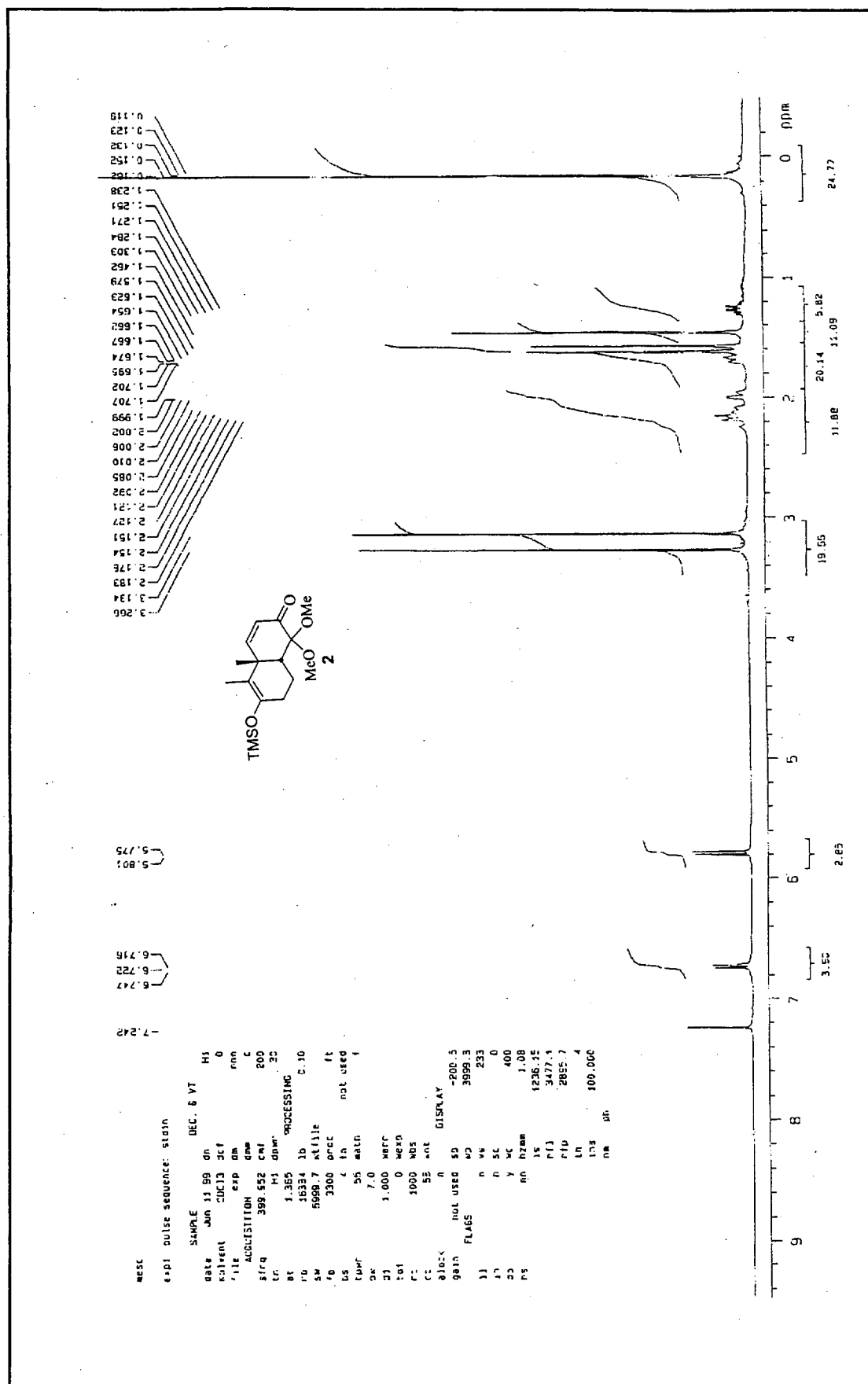
3

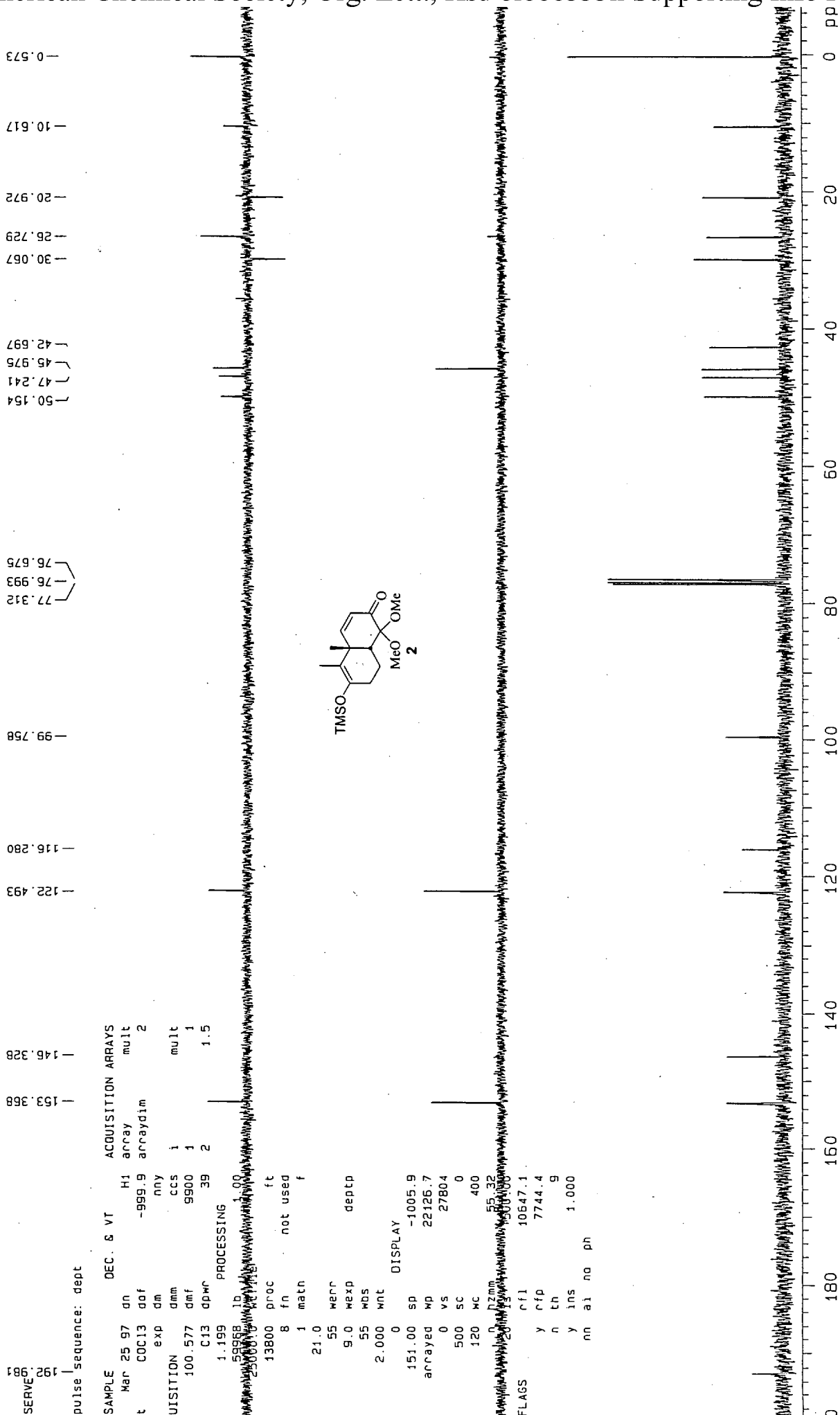


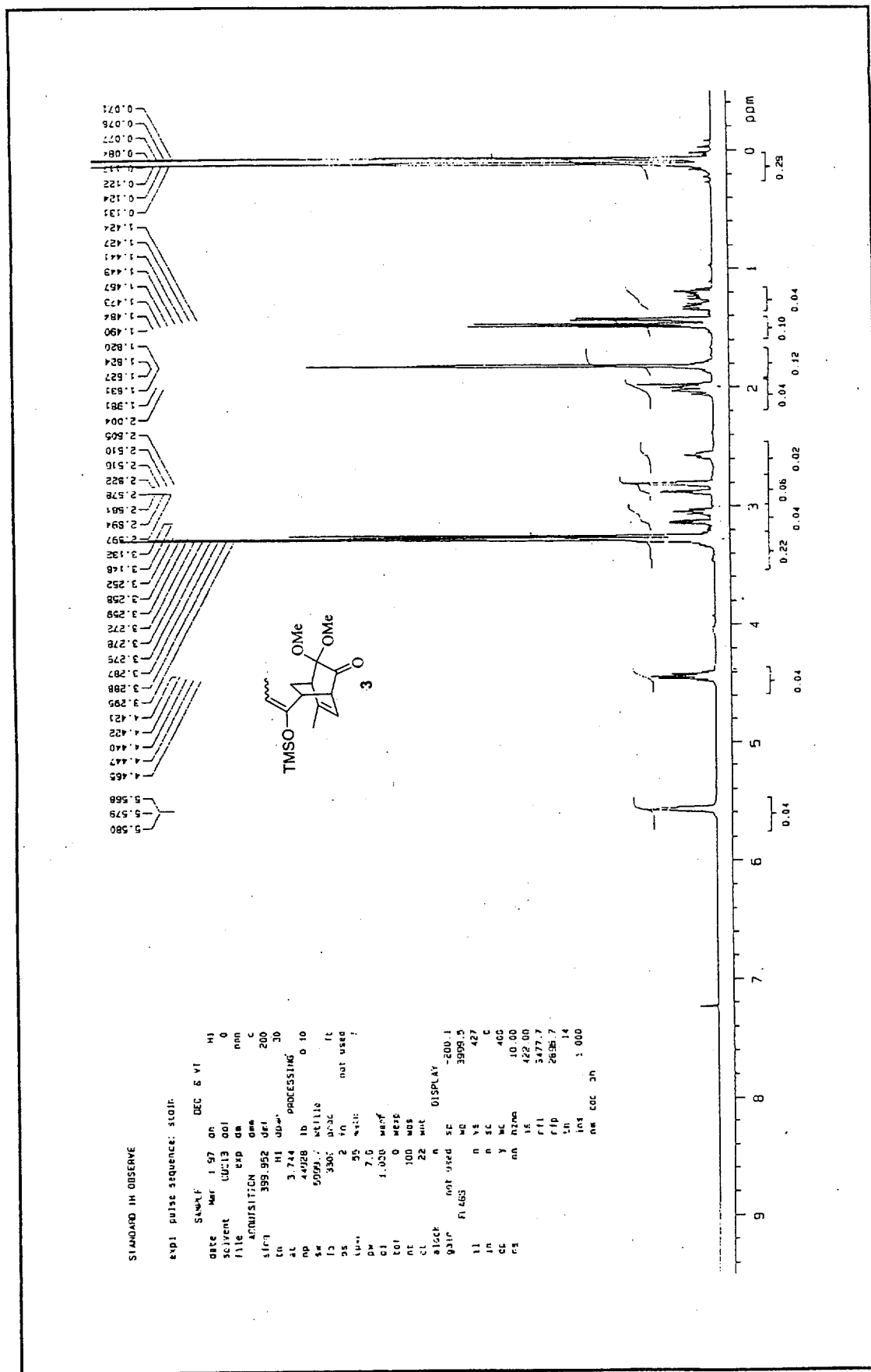
4



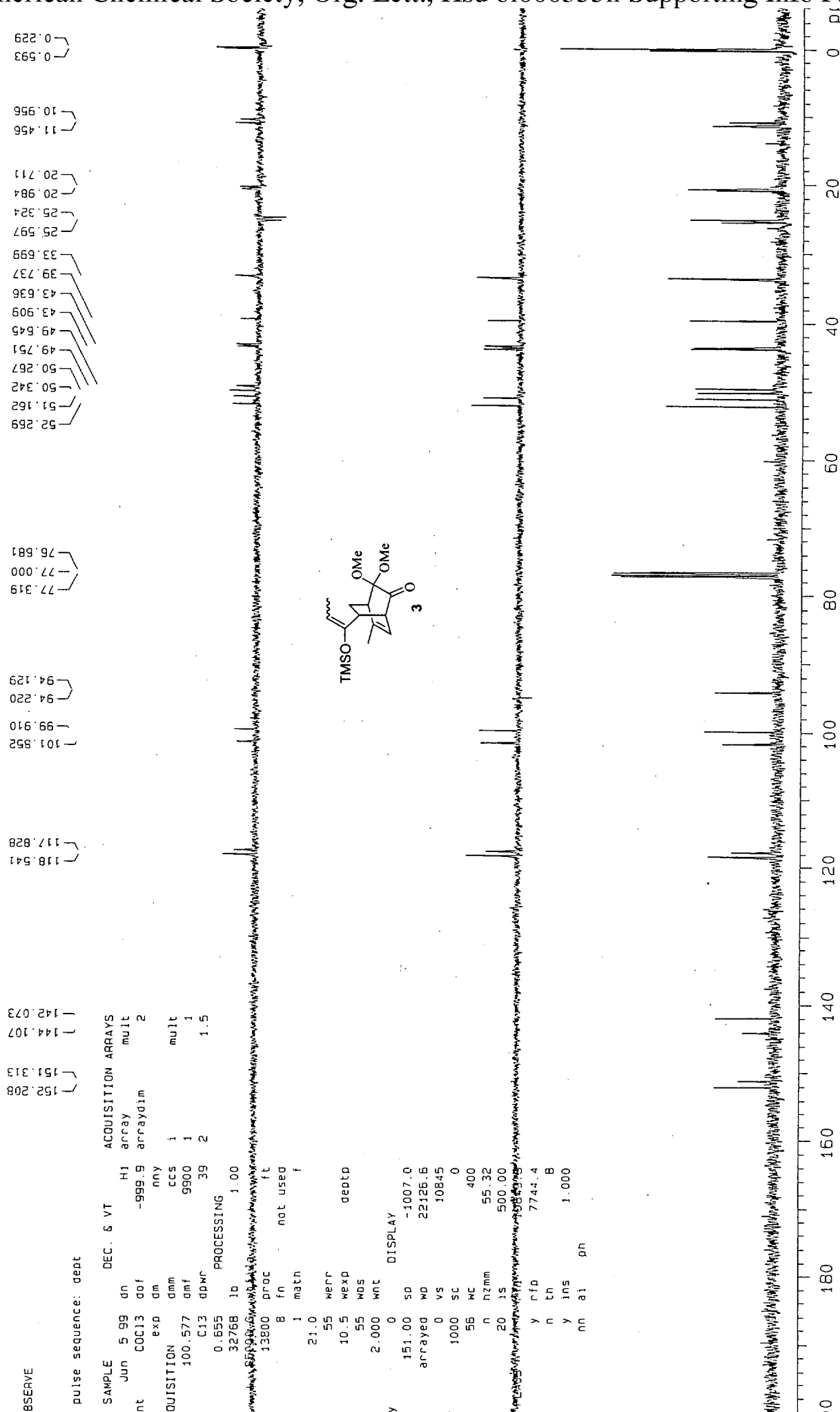
5







8



BSERVE

pulse sequence: dept

DEC. & VT

SAMPLE

Jun 5.99 dn H1 array mult

nt COC13 dof -999.9 arraydim 2

exp dm nny

QUISITION

100.577 dm ccs 1 mult

dmf 9900 1 1

C13 dpwr 39 2 1.5

0.655

PROCESSING

32768 lb 1.00

13800 proc ft

8 fn not used

1 math f

21.0

55 werr

10.5 wexp

55 wds

2.000 wnt

0 DISPLAY

151.00 so -1007.0

arrayed wd 22126.5

0 vs 10845

1000 sc 0

56 wc 400

n h2mm 55.32

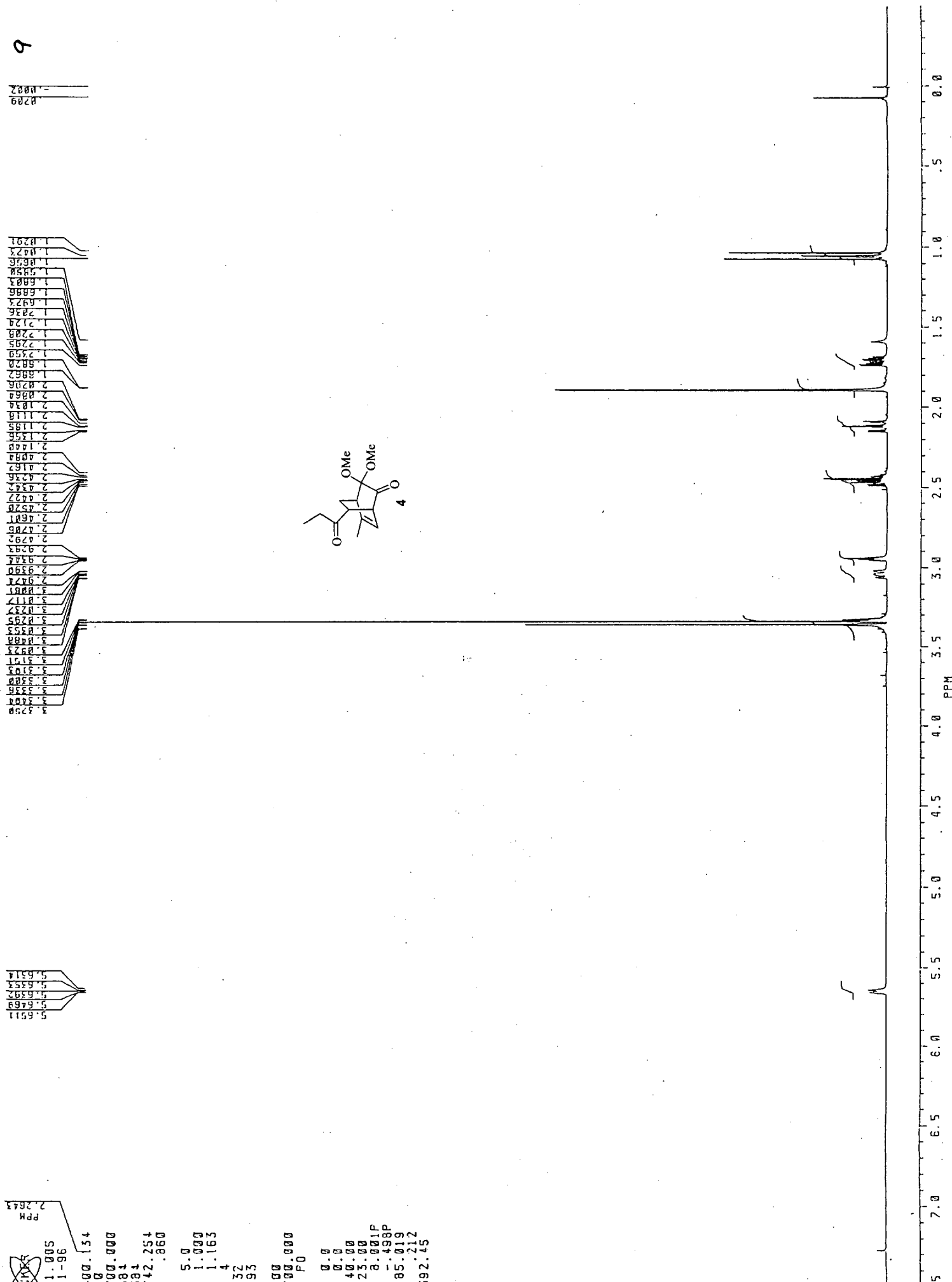
20 is 500.00

y rfp 7744.4

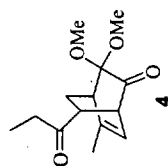
n th B

y ins 1.000

nn al ph



145.41
112.24
94.29
72.32
72.00
76.69
58.58
49.72
49.53
46.14
43.56
34.12
23.86
21.81
2.98



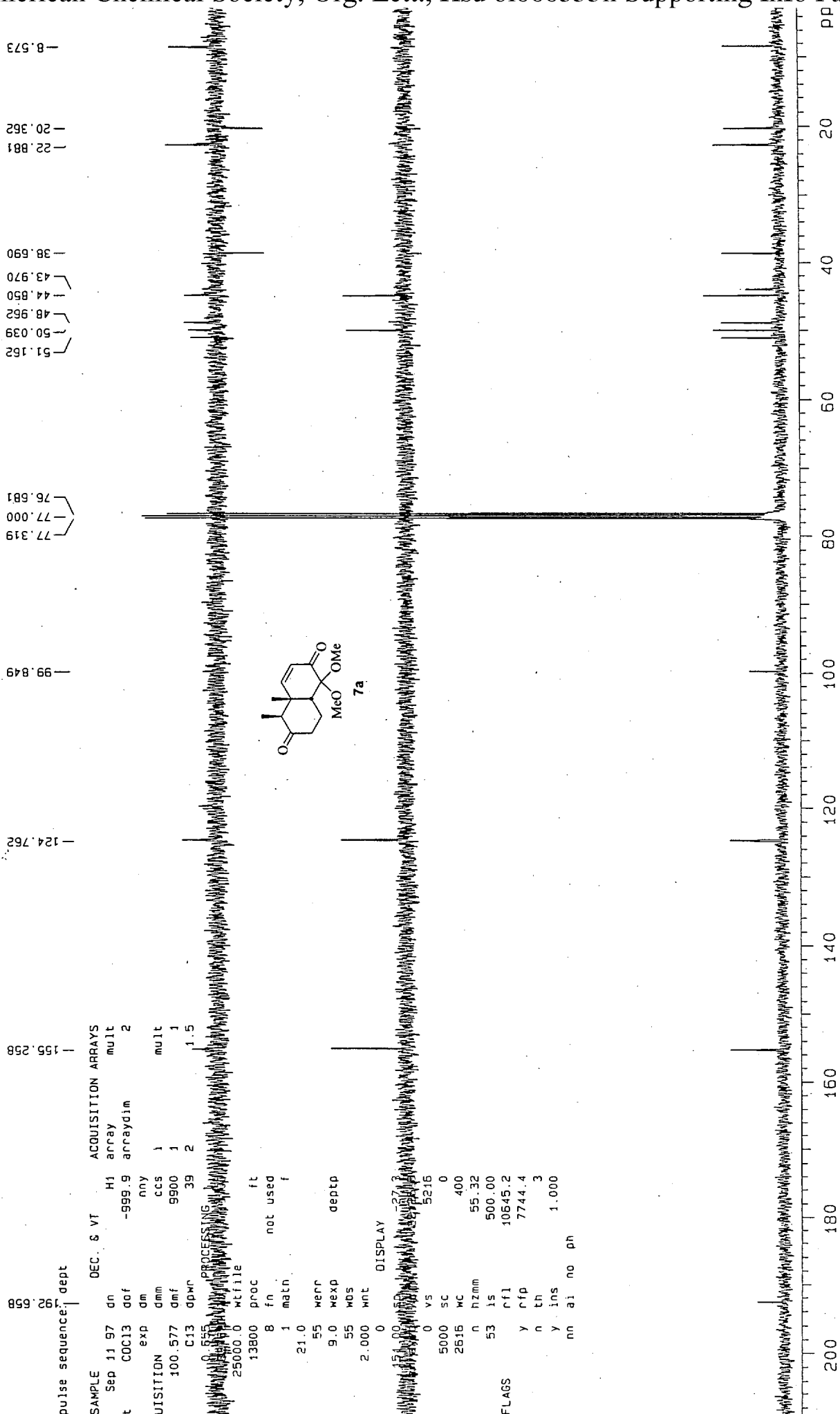
201.75
203.91
PPM

SEQUENCE: 006
U-PROG: 1-1-96
DATE: 1-1-96
F 100.614
Y 74.9
I 96000.000
I 32769
D 32769
W 26315.739
Z/F 1.606
W 3.5
D 0.0
Q 0.623
G 200
S 2476
E 294
W 32903
P 6399.999
P 12H 00
B 3.000
X 0.0
X 48.00
Y 5.00
Z 229.998P
Z/CM 578.509
PM/CM 5.750
R 83869.44

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10



12



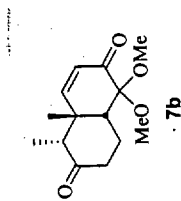
pulse sequence: dept
 SAMPLE Sep 11 97 dn
 t COC13 dof
 exp dm
 ACQUISITION dmm ccs 1 mult
 100.577 dmf 9900 1 1
 C13 dpmr 39 2 1.5
 PROCESSING
 25000.0 wfile
 13800 proc ft
 8 fn not used
 1 math f
 21.0
 55 werr
 9.0 wexp deptp
 55 wds
 2.000 wnt
 0
 151.00
 27.2
 5215
 0 vs
 5000 sc 0
 2616 wc 400
 n hzmm 55.32
 53 ls 500.00
 rfl 10645.2
 y rfp 7744.4
 n th 3
 y ins 1.000
 nn ai no ph

13

1.156
1.172
1.183
1.568
1.556
1.568
2.037
2.375
2.535
2.554
2.559
3.170
3.209
3.223
3.304
3.328

5.910
5.937
6.504
6.510
6.532
6.535

7.274



pulse sequence: stath

SAMPLE DEC. & VT

Dec 1.00 dn H1

0 0

ent CDC13 dof

nnn

ACQUISITION

399.952 dmf 200

30

H1 odmr

1.365 PROCESSING

16384 lb 0.10

5999.7 wfile

3300 proc ft

4 fn not used

55 math f

7.0

1.000 werr

0 wexp

100 nbs

100 wnt

DISPLAY

20 SD -200.7

WD 4199.3

VS 150

SC 0

WC 400

nn hzmm 10.50

ls 3000.00

rfl 567.7

rld 0

th 10

ins 1.000

nm cdc ph

FLAGS

9 8 7 6 5 4 3 2 1 0

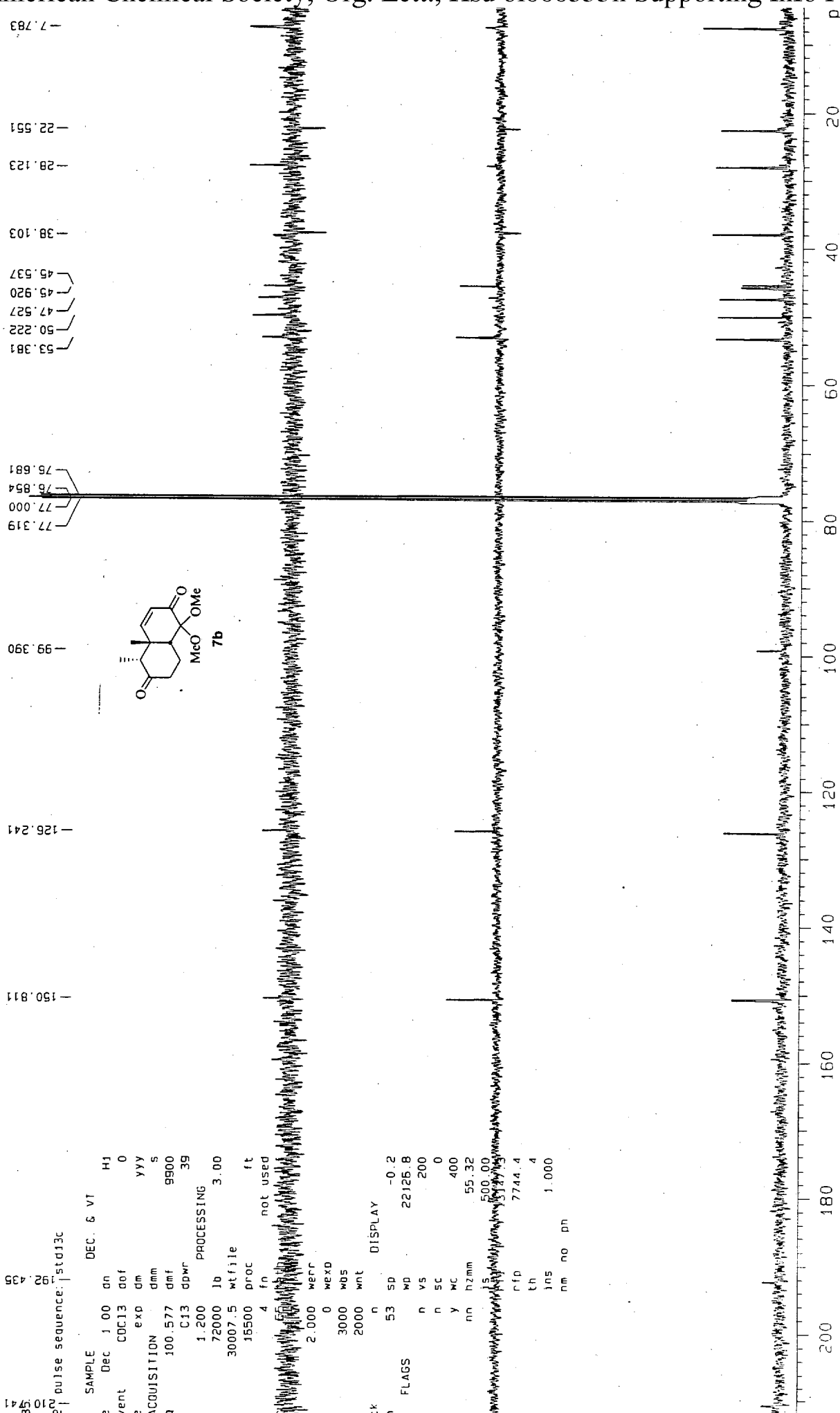
0.13
0.14

0.03

0.03

0.03

14



2 pulse sequence: std13c

SAMPLE DEC. & VI

2	Dec	1.00	dn	H1
3	Dec	0.00	dn	0
4	Dec	0.00	dn	0
5	Dec	0.00	dn	0
6	Dec	0.00	dn	0
7	Dec	0.00	dn	0
8	Dec	0.00	dn	0
9	Dec	0.00	dn	0
10	Dec	0.00	dn	0
11	Dec	0.00	dn	0
12	Dec	0.00	dn	0
13	Dec	0.00	dn	0
14	Dec	0.00	dn	0
15	Dec	0.00	dn	0
16	Dec	0.00	dn	0
17	Dec	0.00	dn	0
18	Dec	0.00	dn	0
19	Dec	0.00	dn	0
20	Dec	0.00	dn	0
21	Dec	0.00	dn	0
22	Dec	0.00	dn	0
23	Dec	0.00	dn	0
24	Dec	0.00	dn	0
25	Dec	0.00	dn	0
26	Dec	0.00	dn	0
27	Dec	0.00	dn	0
28	Dec	0.00	dn	0
29	Dec	0.00	dn	0
30	Dec	0.00	dn	0
31	Dec	0.00	dn	0
32	Dec	0.00	dn	0
33	Dec	0.00	dn	0
34	Dec	0.00	dn	0
35	Dec	0.00	dn	0
36	Dec	0.00	dn	0
37	Dec	0.00	dn	0
38	Dec	0.00	dn	0
39	Dec	0.00	dn	0
40	Dec	0.00	dn	0
41	Dec	0.00	dn	0
42	Dec	0.00	dn	0
43	Dec	0.00	dn	0
44	Dec	0.00	dn	0
45	Dec	0.00	dn	0
46	Dec	0.00	dn	0
47	Dec	0.00	dn	0
48	Dec	0.00	dn	0
49	Dec	0.00	dn	0
50	Dec	0.00	dn	0
51	Dec	0.00	dn	0
52	Dec	0.00	dn	0
53	Dec	0.00	dn	0
54	Dec	0.00	dn	0
55	Dec	0.00	dn	0
56	Dec	0.00	dn	0
57	Dec	0.00	dn	0
58	Dec	0.00	dn	0
59	Dec	0.00	dn	0
60	Dec	0.00	dn	0
61	Dec	0.00	dn	0
62	Dec	0.00	dn	0
63	Dec	0.00	dn	0
64	Dec	0.00	dn	0
65	Dec	0.00	dn	0
66	Dec	0.00	dn	0
67	Dec	0.00	dn	0
68	Dec	0.00	dn	0
69	Dec	0.00	dn	0
70	Dec	0.00	dn	0
71	Dec	0.00	dn	0
72	Dec	0.00	dn	0
73	Dec	0.00	dn	0
74	Dec	0.00	dn	0
75	Dec	0.00	dn	0
76	Dec	0.00	dn	0
77	Dec	0.00	dn	0
78	Dec	0.00	dn	0
79	Dec	0.00	dn	0
80	Dec	0.00	dn	0
81	Dec	0.00	dn	0
82	Dec	0.00	dn	0
83	Dec	0.00	dn	0
84	Dec	0.00	dn	0
85	Dec	0.00	dn	0
86	Dec	0.00	dn	0
87	Dec	0.00	dn	0
88	Dec	0.00	dn	0
89	Dec	0.00	dn	0
90	Dec	0.00	dn	0
91	Dec	0.00	dn	0
92	Dec	0.00	dn	0
93	Dec	0.00	dn	0
94	Dec	0.00	dn	0
95	Dec	0.00	dn	0
96	Dec	0.00	dn	0
97	Dec	0.00	dn	0
98	Dec	0.00	dn	0
99	Dec	0.00	dn	0
100	Dec	0.00	dn	0

ACQUISITION

101	100.577	dmf	9900
102	1.200	dmf	39
103	1.200	dmf	39
104	1.200	dmf	39
105	1.200	dmf	39
106	1.200	dmf	39
107	1.200	dmf	39
108	1.200	dmf	39
109	1.200	dmf	39
110	1.200	dmf	39
111	1.200	dmf	39
112	1.200	dmf	39
113	1.200	dmf	39
114	1.200	dmf	39
115	1.200	dmf	39
116	1.200	dmf	39
117	1.200	dmf	39
118	1.200	dmf	39
119	1.200	dmf	39
120	1.200	dmf	39
121	1.200	dmf	39
122	1.200	dmf	39
123	1.200	dmf	39
124	1.200	dmf	39
125	1.200	dmf	39
126	1.200	dmf	39
127	1.200	dmf	39
128	1.200	dmf	39
129	1.200	dmf	39
130	1.200	dmf	39
131	1.200	dmf	39
132	1.200	dmf	39
133	1.200	dmf	39
134	1.200	dmf	39
135	1.200	dmf	39
136	1.200	dmf	39
137	1.200	dmf	39
138	1.200	dmf	39
139	1.200	dmf	39
140	1.200	dmf	39
141	1.200	dmf	39
142	1.200	dmf	39
143	1.200	dmf	39
144	1.200	dmf	39
145	1.200	dmf	39
146	1.200	dmf	39
147	1.200	dmf	39
148	1.200	dmf	39
149	1.200	dmf	39
150	1.200	dmf	39
151	1.200	dmf	39
152	1.200	dmf	39
153	1.200	dmf	39
154	1.200	dmf	39
155	1.200	dmf	39
156	1.200	dmf	39
157	1.200	dmf	39
158	1.200	dmf	39
159	1.200	dmf	39
160	1.200	dmf	39
161	1.200	dmf	39
162	1.200	dmf	39
163	1.200	dmf	39
164	1.200	dmf	39
165	1.200	dmf	39
166	1.200	dmf	39
167	1.200	dmf	39
168	1.200	dmf	39
169	1.200	dmf	39
170	1.200	dmf	39
171	1.200	dmf	39
172	1.200	dmf	39
173	1.200	dmf	39
174	1.200	dmf	39
175	1.200	dmf	39
176	1.200	dmf	39
177	1.200	dmf	39
178	1.200	dmf	39
179	1.200	dmf	39
180	1.200	dmf	39
181	1.200	dmf	39
182	1.200	dmf	39
183	1.200	dmf	39
184	1.200	dmf	39
185	1.200	dmf	39
186	1.200	dmf	39
187	1.200	dmf	39
188	1.200	dmf	39
189	1.200	dmf	39
190	1.200	dmf	39
191	1.200	dmf	39
192	1.200	dmf	39
193	1.200	dmf	39
194	1.200	dmf	39
195	1.200	dmf	39
196	1.200	dmf	39
197	1.200	dmf	39
198	1.200	dmf	39
199	1.200	dmf	39
200	1.200	dmf	39

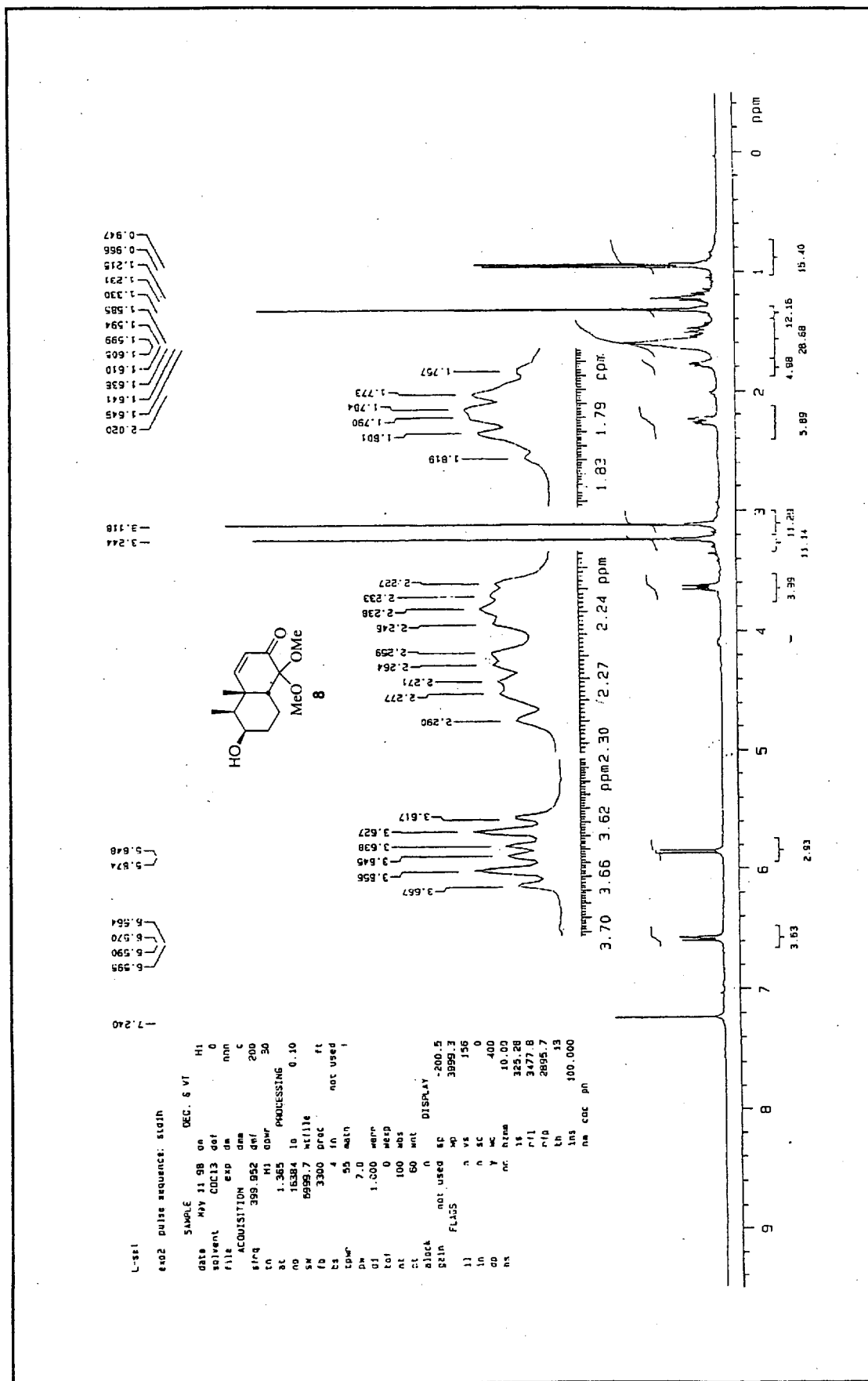
PROCESSING

201	72000	lb	3.00
202	30007.5	wtfile	
203	15500	proc	ft
204	4	fn	not used
205	2.000	werr	
206	0	wexd	
207	3000	wos	
208	2000	wnt	
209	53	sp	-0.2
210	22125.8	wp	
211	200	vs	
212	0	sc	
213	400	yc	
214	55.32	nn	
215	500.00	ss	
216	7744.4	rip	
217	4	th	
218	1.000	ins	
219	nm	no	pn

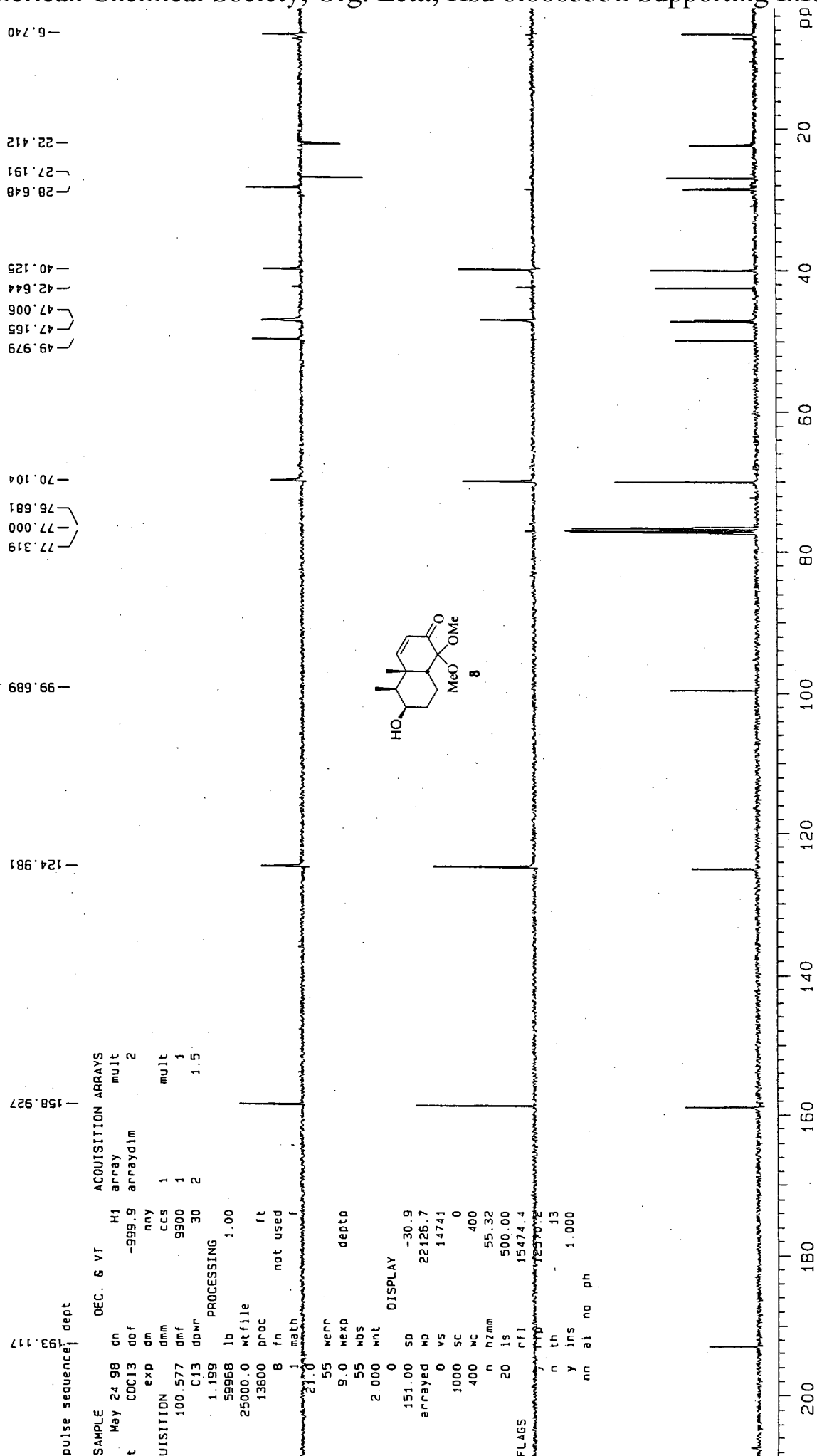
DISPLAY

220	53	sp	-0.2
221	22125.8	wp	
222	200	vs	
223	0	sc	
224	400	yc	
225	55.32	nn	
226	500.00	ss	
227	7744.4	rip	
228	4	th	
229	1.000	ins	
230	nm	no	pn

15

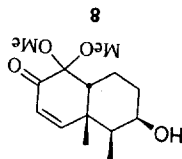


16



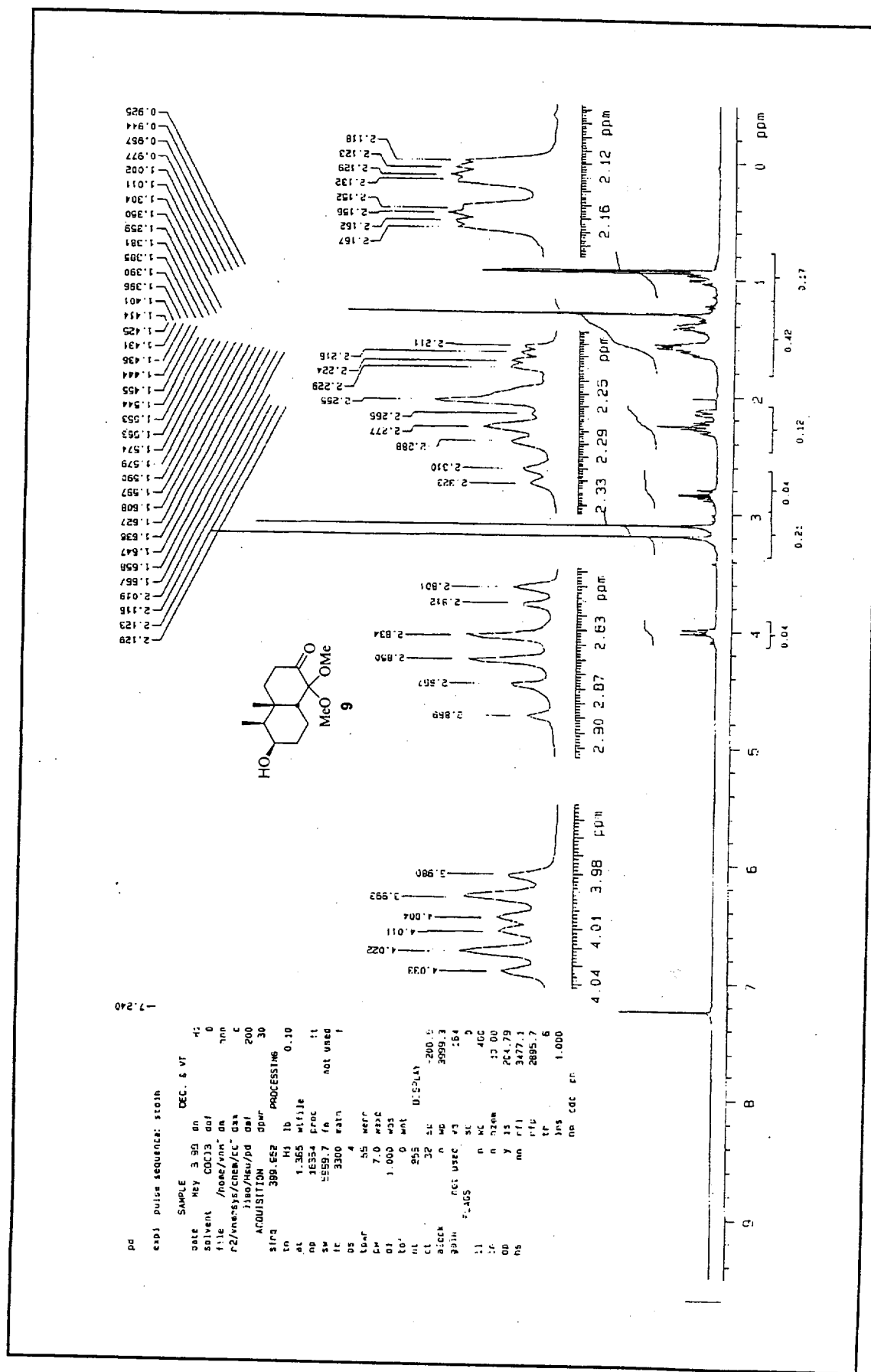
pulse sequence: dept
 163.117
 SAMPLE
 May 24 98 dn
 t COC13 dof
 exp dm
 USSION dmm
 100.577 dmf
 C13 dpmr
 1.199
 59968 lb
 25000.0 wfile
 13800 proc
 B fn
 1 math
 21.0
 DEC. & VT
 H1 array
 -999.9 arraydim
 nny
 ccs 1
 9900 1
 30 2
 1.5
 ACQUISITION ARRAYS
 mult 2
 mult
 1
 1.5
 PROCESSING
 1.00
 ft
 not used
 f
 55 werr
 9.0 wexp
 55 wbs
 2.000 wnt
 0
 151.00 sd
 arrayed wp
 0 vs
 1000 sc
 400 wc
 n hzmm
 55.32
 20 ls
 500.00
 15474.4
 12570.2
 13
 1.000
 nn ai no ph

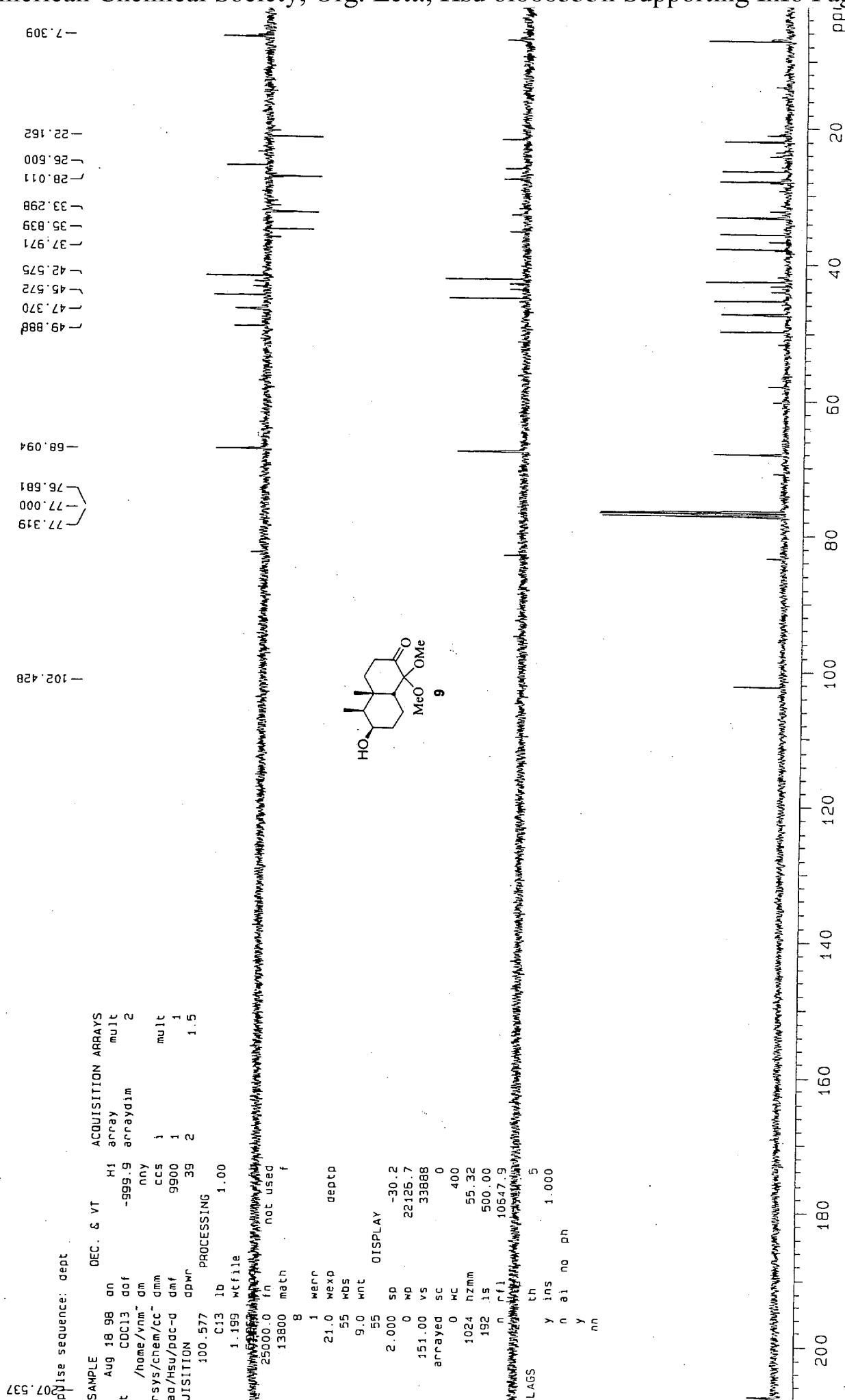
17



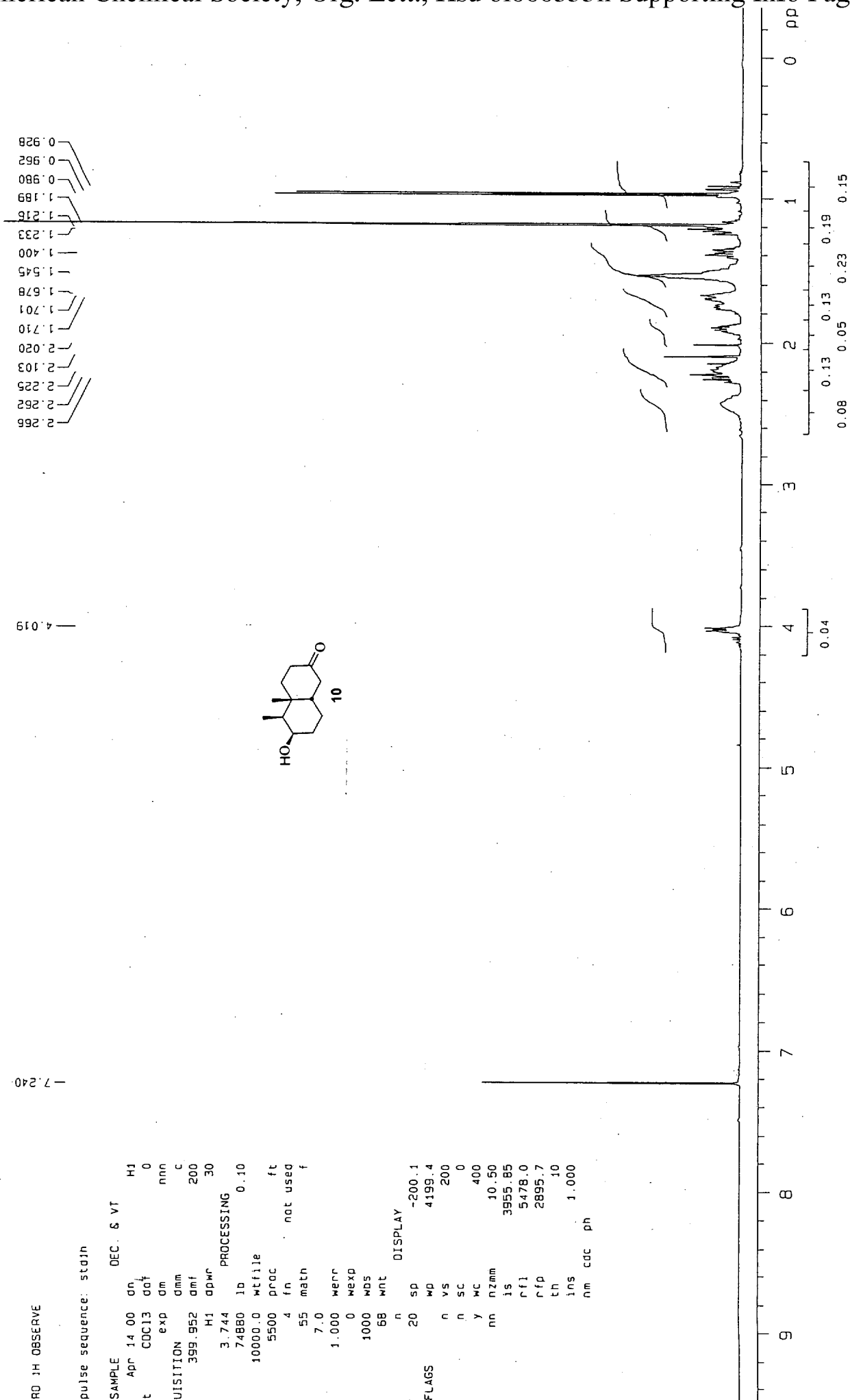
Summary of Crystal Data and Intensity Collection 98sp13 (980195)

Empirical Formula	C ₁₆ 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 V = 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 ≤ h ≤ 10, -17 ≤ k ≤ 15, -12 ≤ l ≤ 15
Reflections Collected	7486(557623.00(1))
Independent Reflections	2891(152723.00(1))(R _{int} = 4.06%)
System Used	Siemens SHELXTL PLUS (VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Extinction Correction	X = 0.0010(4), where F* = F [1 + 0.002X ² /sin(2θ)] ^{-1/4}
Hydrogen Atoms	Located on difference map
Weighting Scheme	w ⁻¹ = σ ² (F) + 0.0005F ²
Number of Parameters Refined	164
Final R Indices (obs. data)	R = 0.0455, R _w = 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





20



PRO 1H OBSERVE

pulse sequence: std1h

SAMPLE DEC. & VT

Apr 14 00 dn H1

t COC13 do 0

exp dm nnn

QUISITION c

399.952 dm 200

H1 dm 30

3.744 PROCESSING

74880 lb 0.10

10000.0 wfile

5500 proc ft

4 fn not used

55 math f

7.0

1.000 werr

0 wexp

1000 wds

58 wnt

DISPLAY

20 sp -200.1

wp 4195.4

n vs 200

n sc 0

y wc 400

nn nzmm 10.50

is 3955.85

rfl 5478.0

rfp 2895.7

th 10

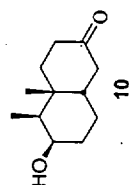
ins 1.000

nm cdc ph

21

77.349
77.243
77.212
77.030
76.712
69.353
69.308

-43.090
-37.613
-35.823
-28.752
-28.130
-23.958



pulse sequence: dept

SAMPLE DEC. & VT ACQUISITION ARRAYS
 May 4 99 on H1 array mult 2
 nt CDC13 of -999.9 arraydim
 exp om nny
 ACQUISITION omn ccs 1 mult
 100.577 of 9900 1 1
 C13 QPWR 39 2 1.5
 PROCESSING
 0.555
 32768 lb 3.00
 1 meth f
 21.0
 55 werr
 10.5 wexp
 55 wos
 2.000 wnt
 DISPLAY
 151.00 sp -1029.9
 arrayed wd 23132.2
 0 vs 289578
 1024 sc 0
 0 wc 400
 n n2mm 57.83
 50.100
 y c13 77.44 7
 n tn
 y tos 1.000
 nn al on

200

180

160

140

120

100

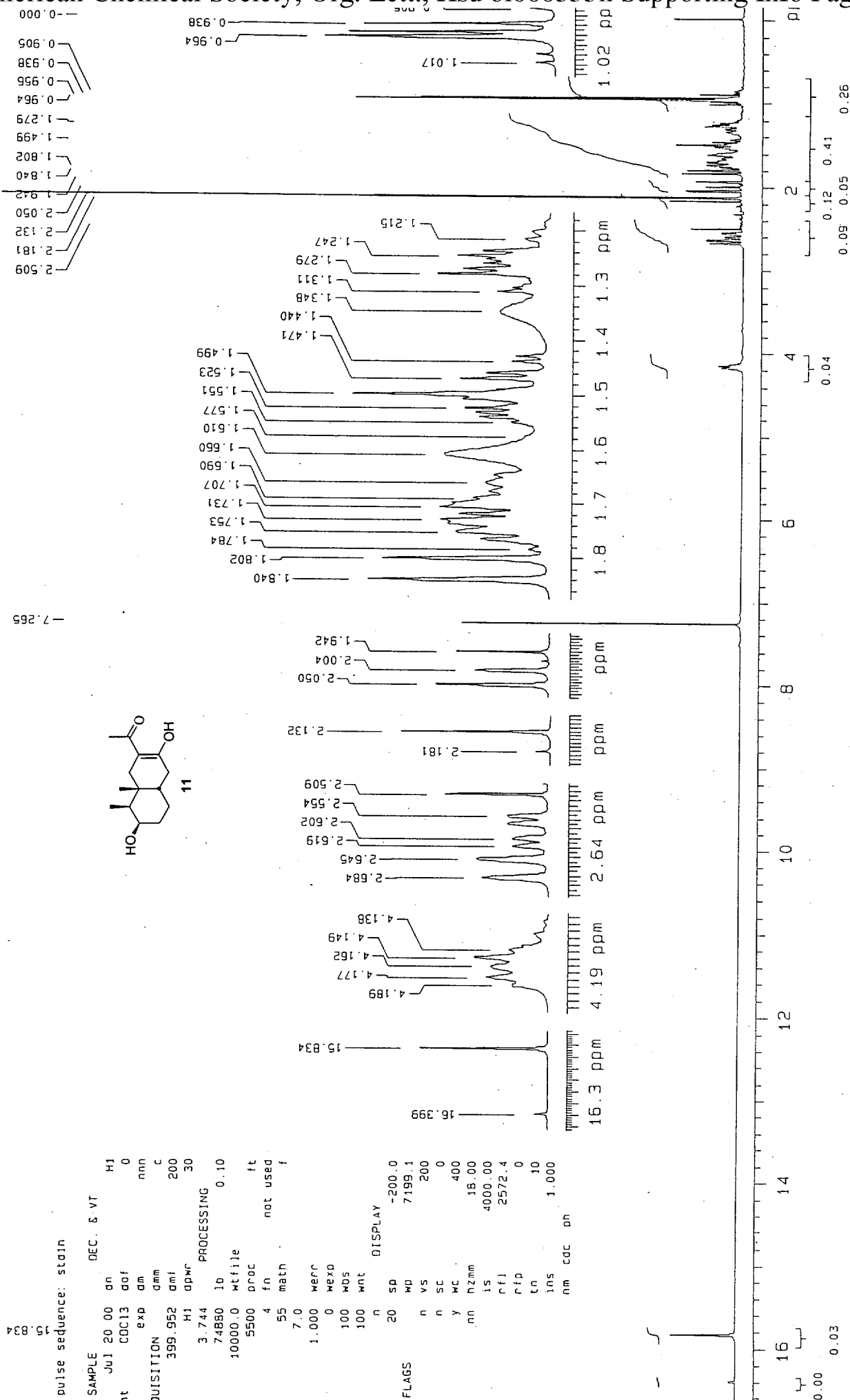
80

60

40

20

0



23

pulse sequence: std13c

SAMPLE DEC. & VT
 Jul 20 00 dn H1
 Coc13 cof 0
 exp dm yyy
 COUQUISITION dmm s
 100.577 dmf 9900
 C13 dmr 39
 1.200 PROCESSING
 72000 lb 3.00
 30007.5 wfile
 16500 proc ft

55 math
 8.7
 3.000 werr
 0 wexp
 2000 wos
 1500 wnt
 n DISPLAY
 53 sp -0.2
 22126.8
 n vs 60
 n SC 0
 y MC 400
 nn, hzmm 55.32
 500.00
 13150.5
 rfp 7744.4
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