JOC The Journal of Organic Chemistry

J. Org. Chem., 1997, 62(6), 1610-1616, DOI:10.1021/jo970042e

**Terms & Conditions**

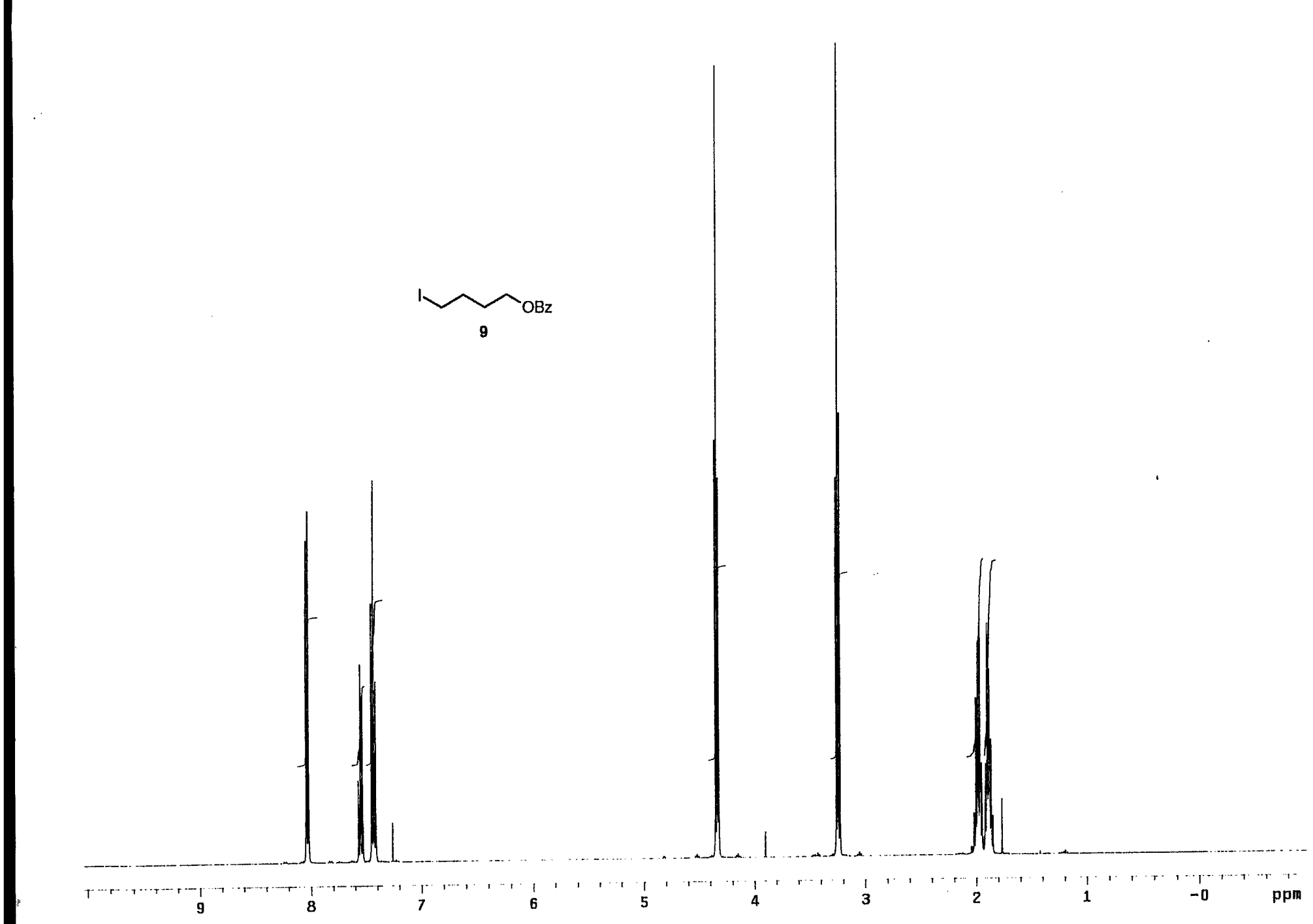
Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at http://pubs.acs.org/page/copyright/permissions.html



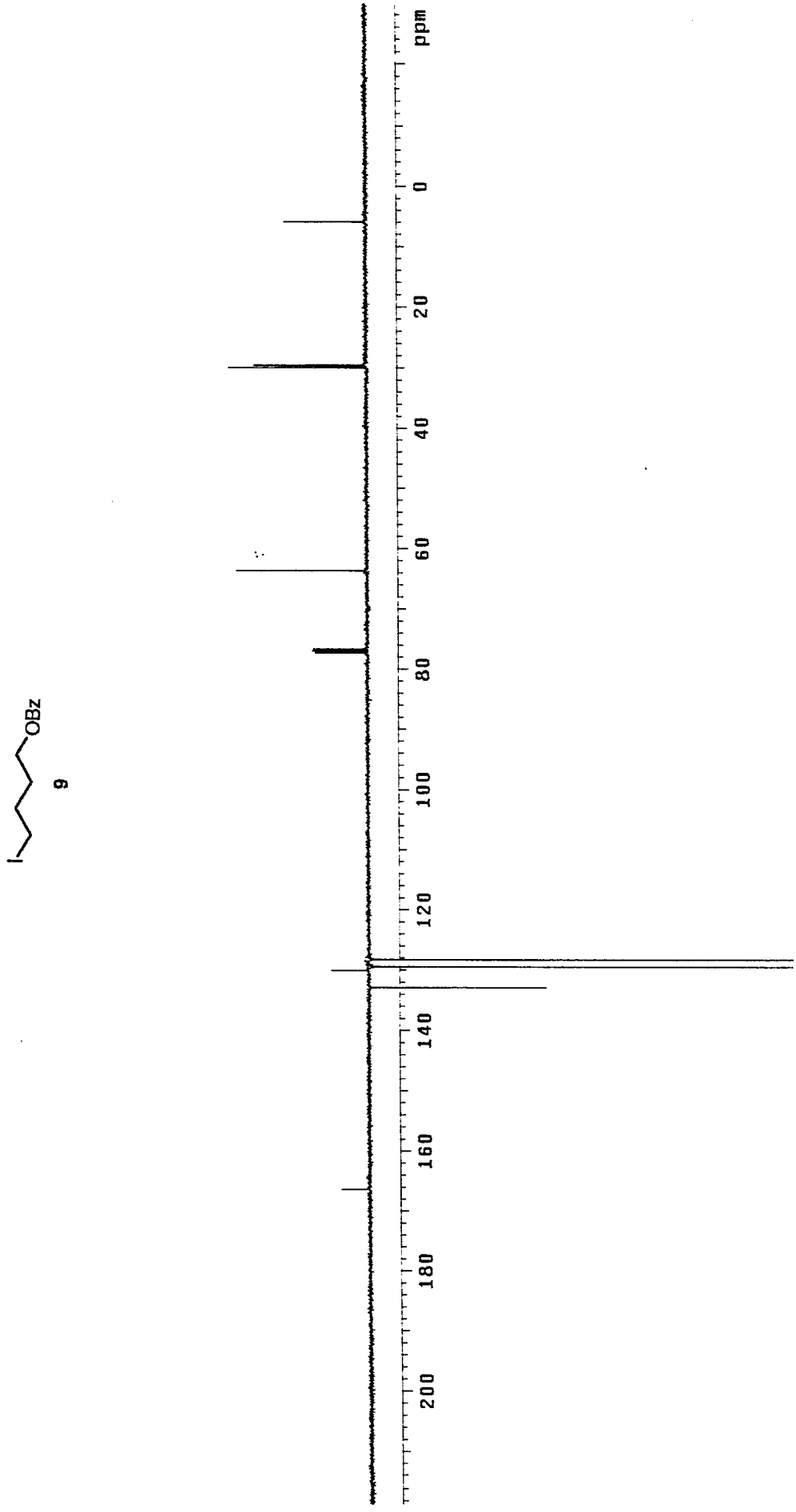
ACS Publications

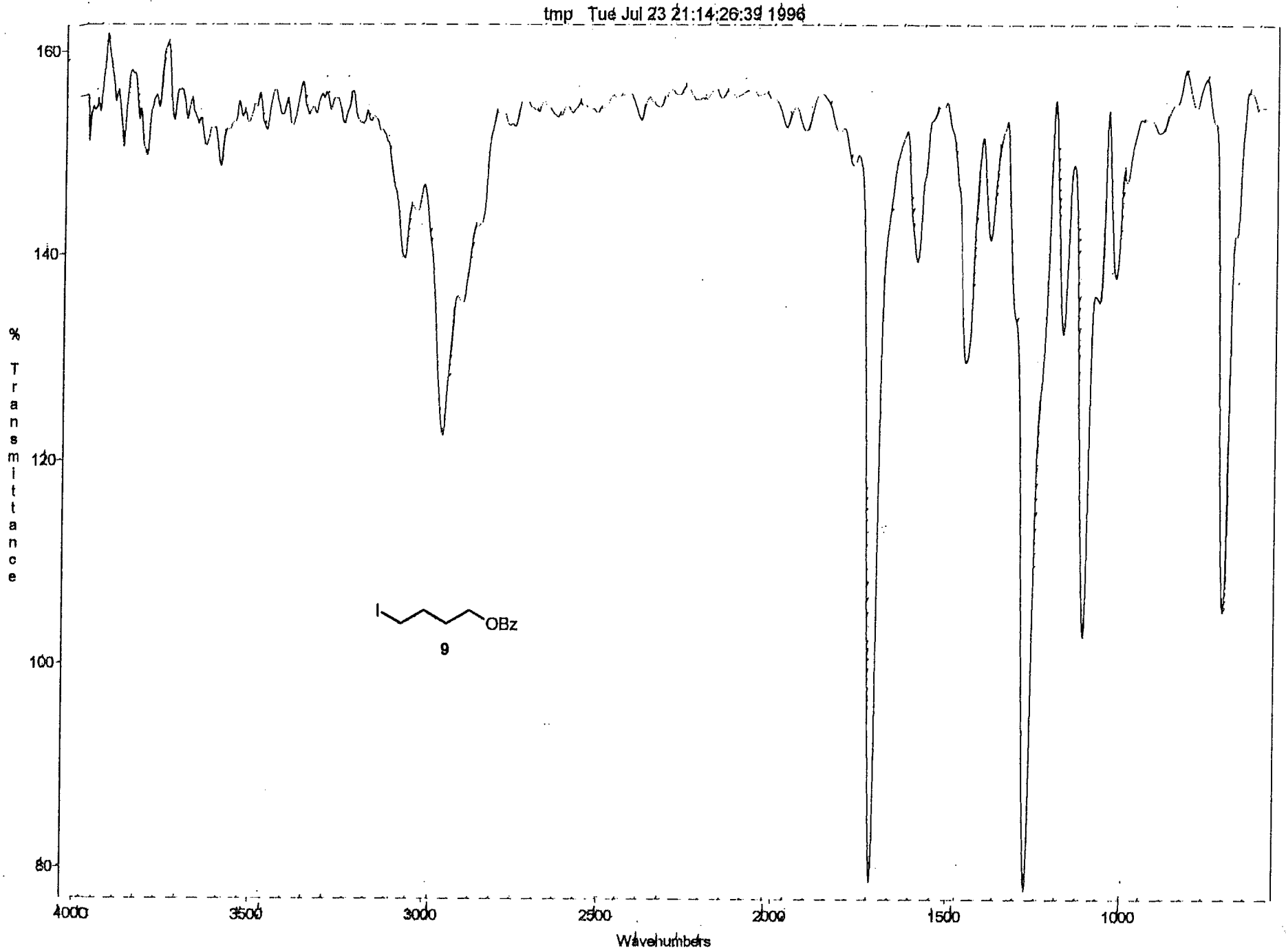
MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1997 American Chemical Society




©1997 American Chemical Society Journal of Organic Chemistry Vol. 1 age 1010 Out Supplemental Page 1


OBz
I
9
ppm
0
20
40
60
80
100
120
140
160
180
200




© 1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1610 Of Supplemental Page 3

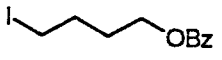
**Elemental Composition** **Date : 30-JUL-1996**

File:LIT0730C Ident:36_42 SMO(1,7) PKD(3,3,5,0.00%,0.0,0.00%,F,F)
70S EI+ Voltage BpI:1187456 TIC:23819400 Flags:NORM
File Text:OTT/LITTLE, MMO1, M/Z 305, LRCI/CH4
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

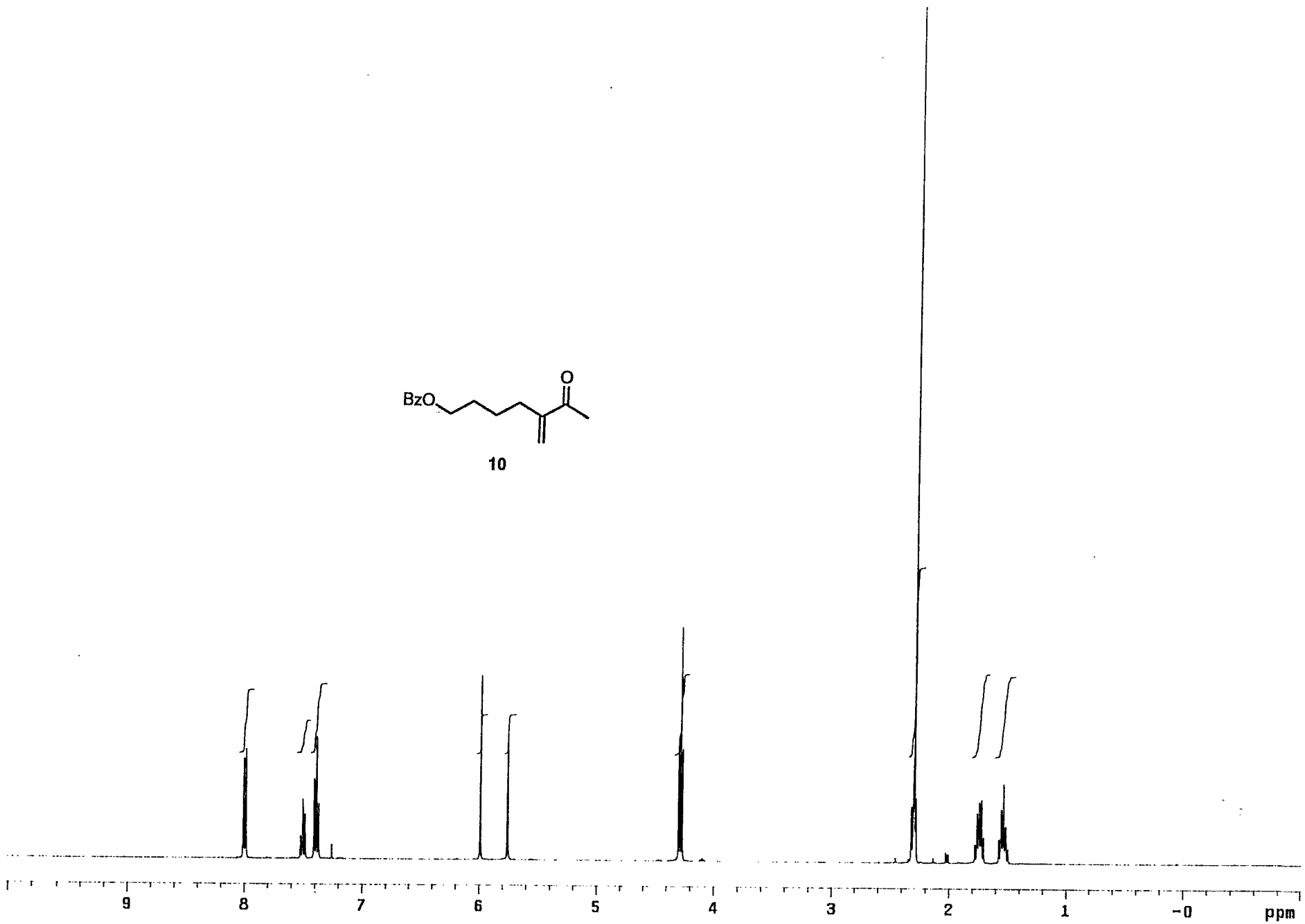
| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | -0.5 | 0 | 0 | 0 | 0 |
| 305.004001 | 10.0 | | | 20.0 | 200 | 400 | 20 | 4 |

| Mass | mDa | PPM | Calc. Mass | DBE | C | H | O | I |
|---|---|---|---|---|---|---|---|---|
| 305.004001 | -0.1 | -0.5 | 305.003857 | 4.5 | [illegible] | | | |
| | 4.6 | 15.1 | 305.008613 | 15.5 | 17 | 5 | 6 | |
| | -4.8 | -15.6 | 304.999230 | 2.5 | 6 | 9 | 14 | |

$(P+4)^+$

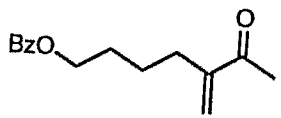



**9**

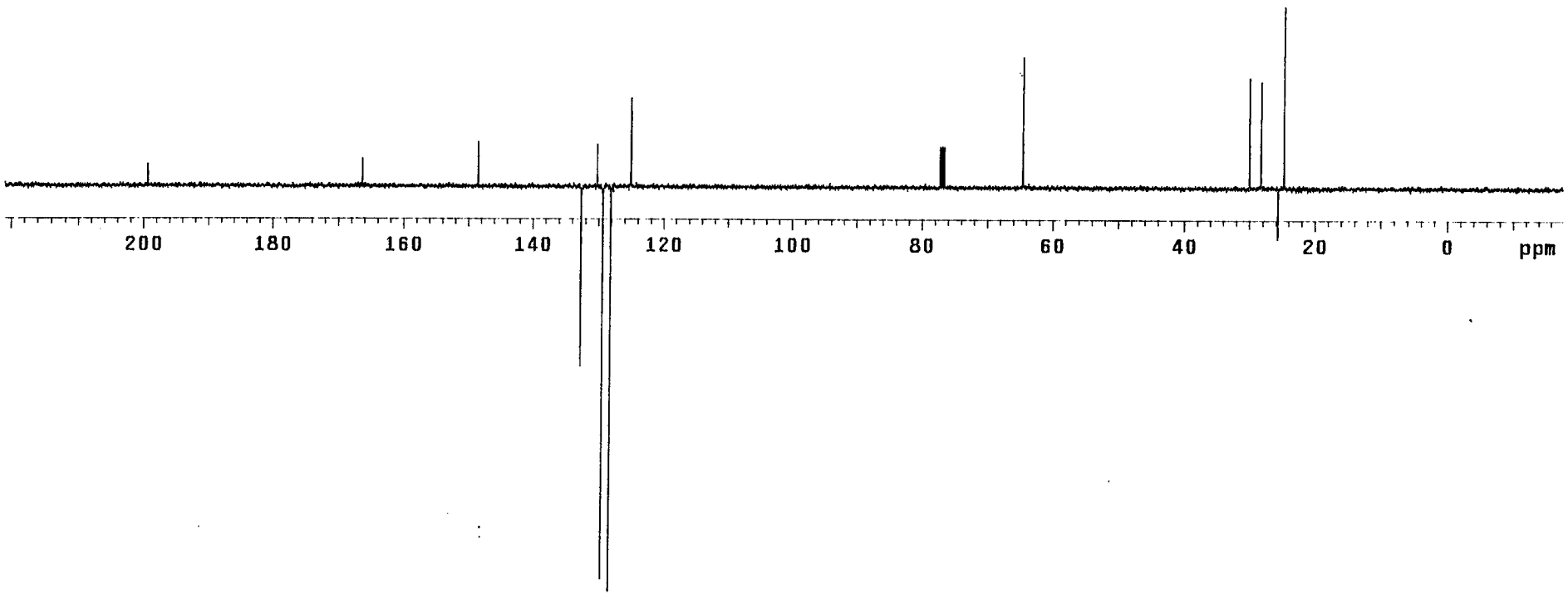



10

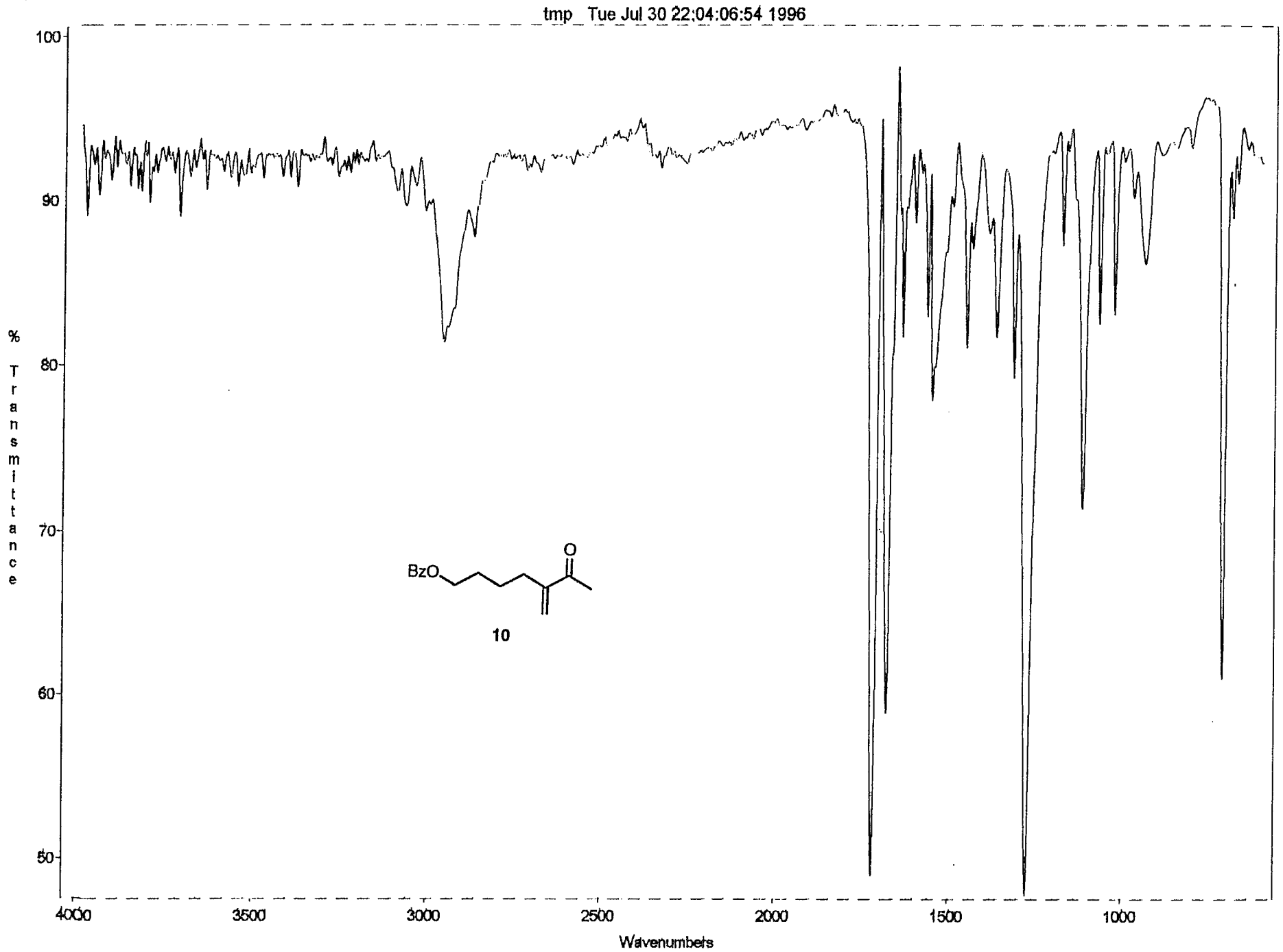
©1997 American Chemical Society 




10

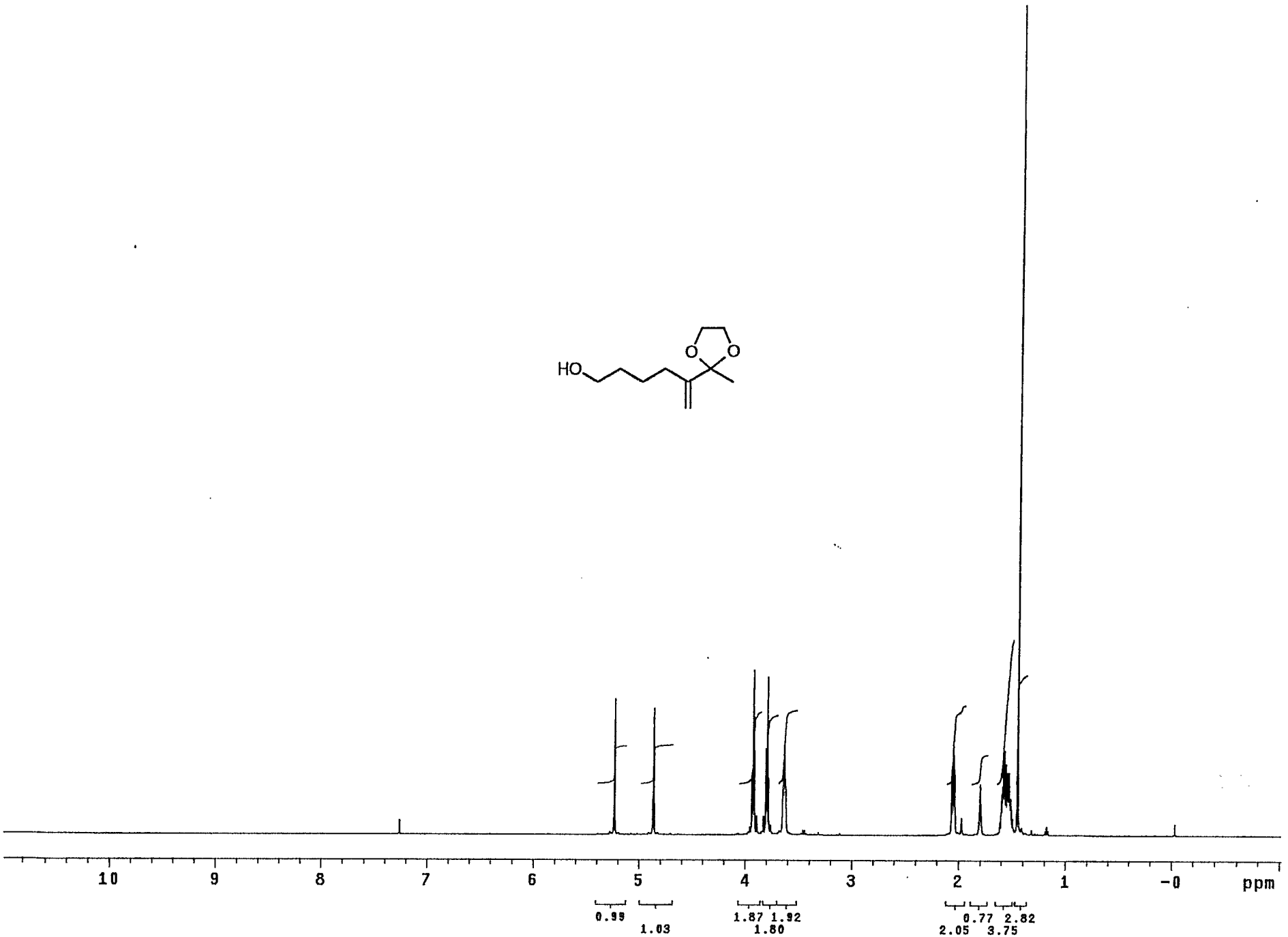



© 1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 6

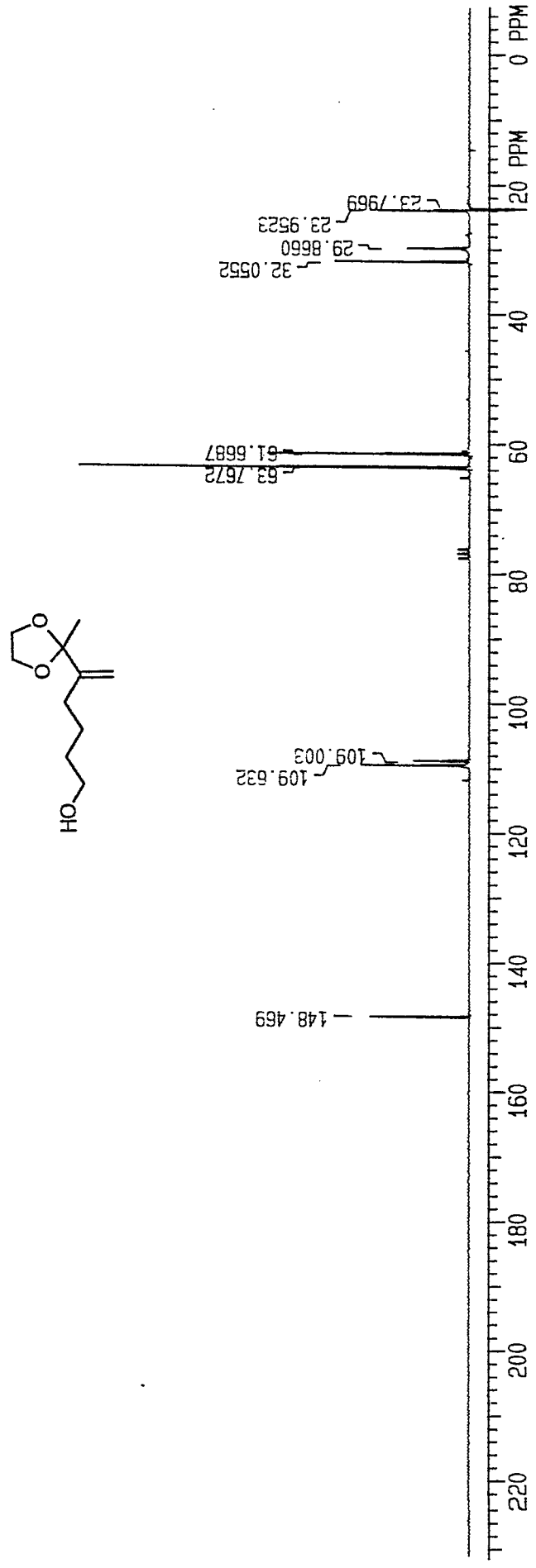


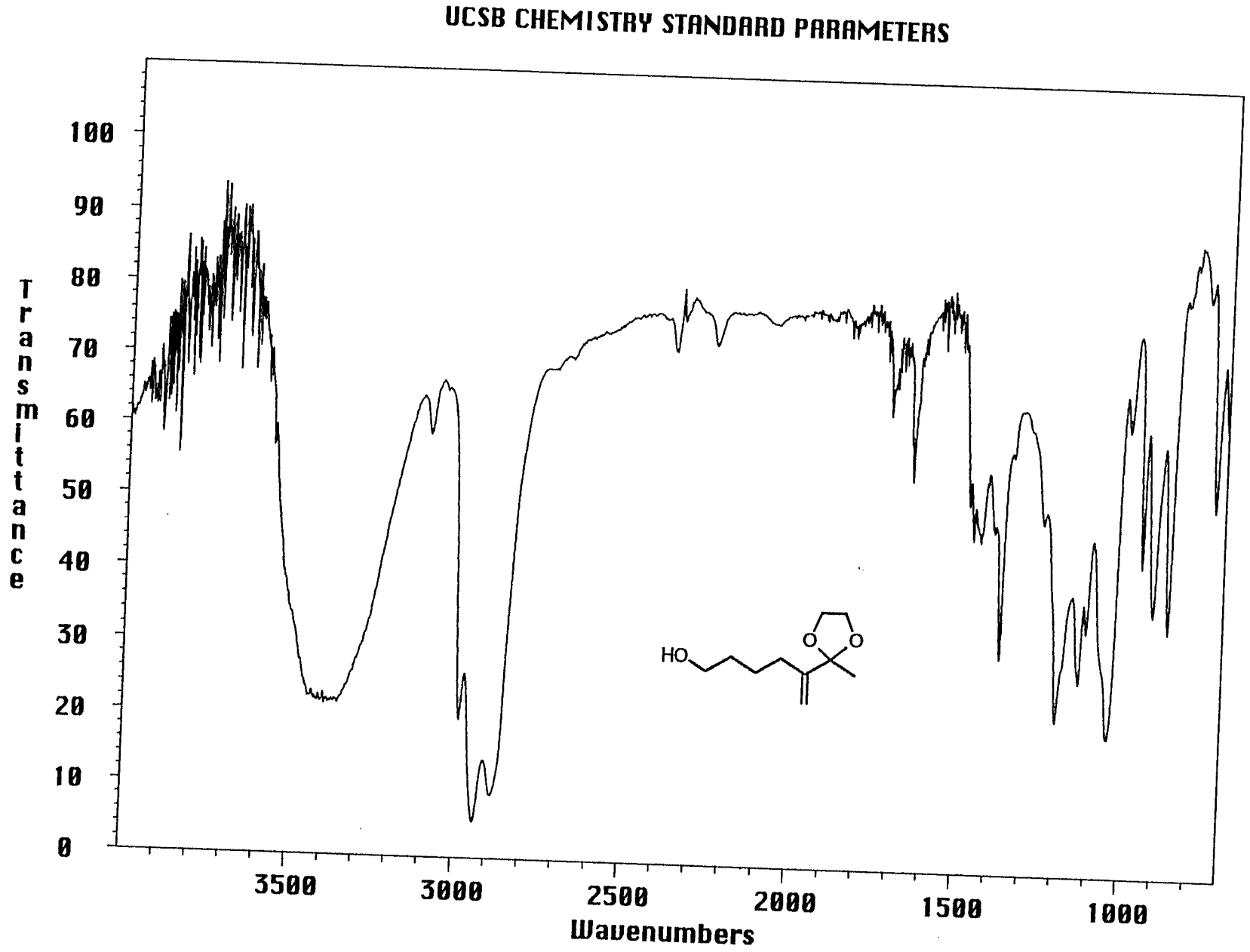

10

©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 7

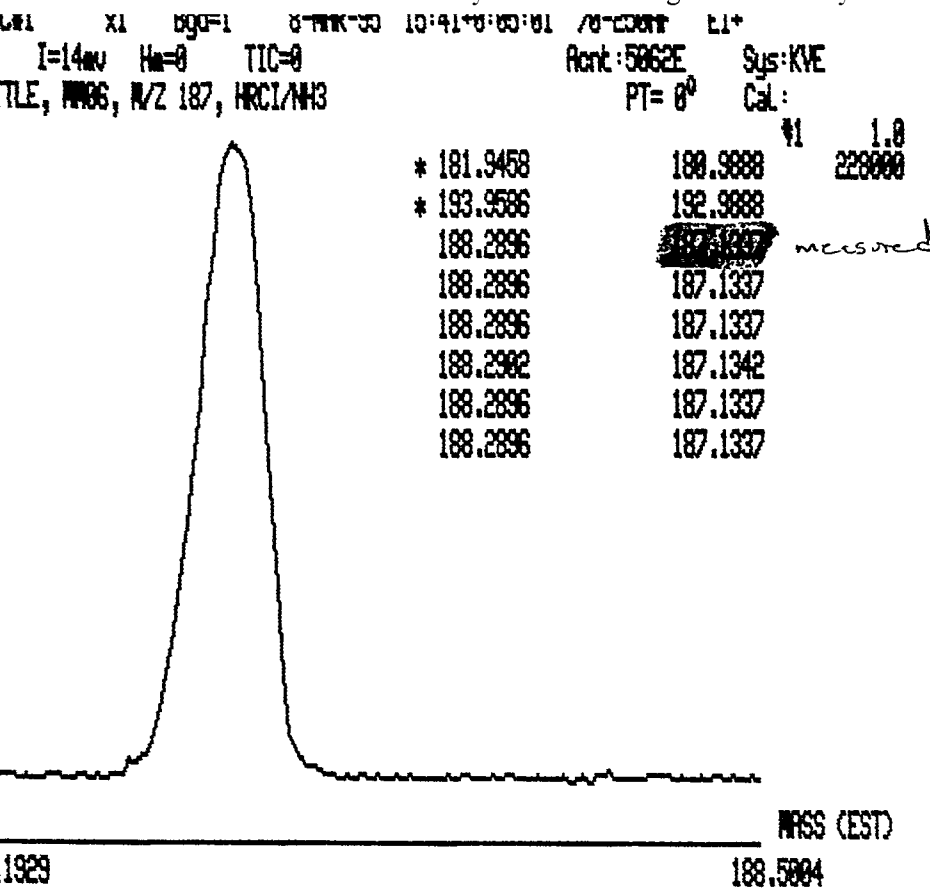


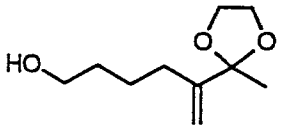

©2007 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page 1010 Of Supplemental Page 6


0 PPM
20 PPM
40
60
80
100
120
140
160
180
200
220
23.7969
23.9523
29.8660
32.0552
61.6687
63.7672
109.003
109.632
148.469
HO
O
O




©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 10



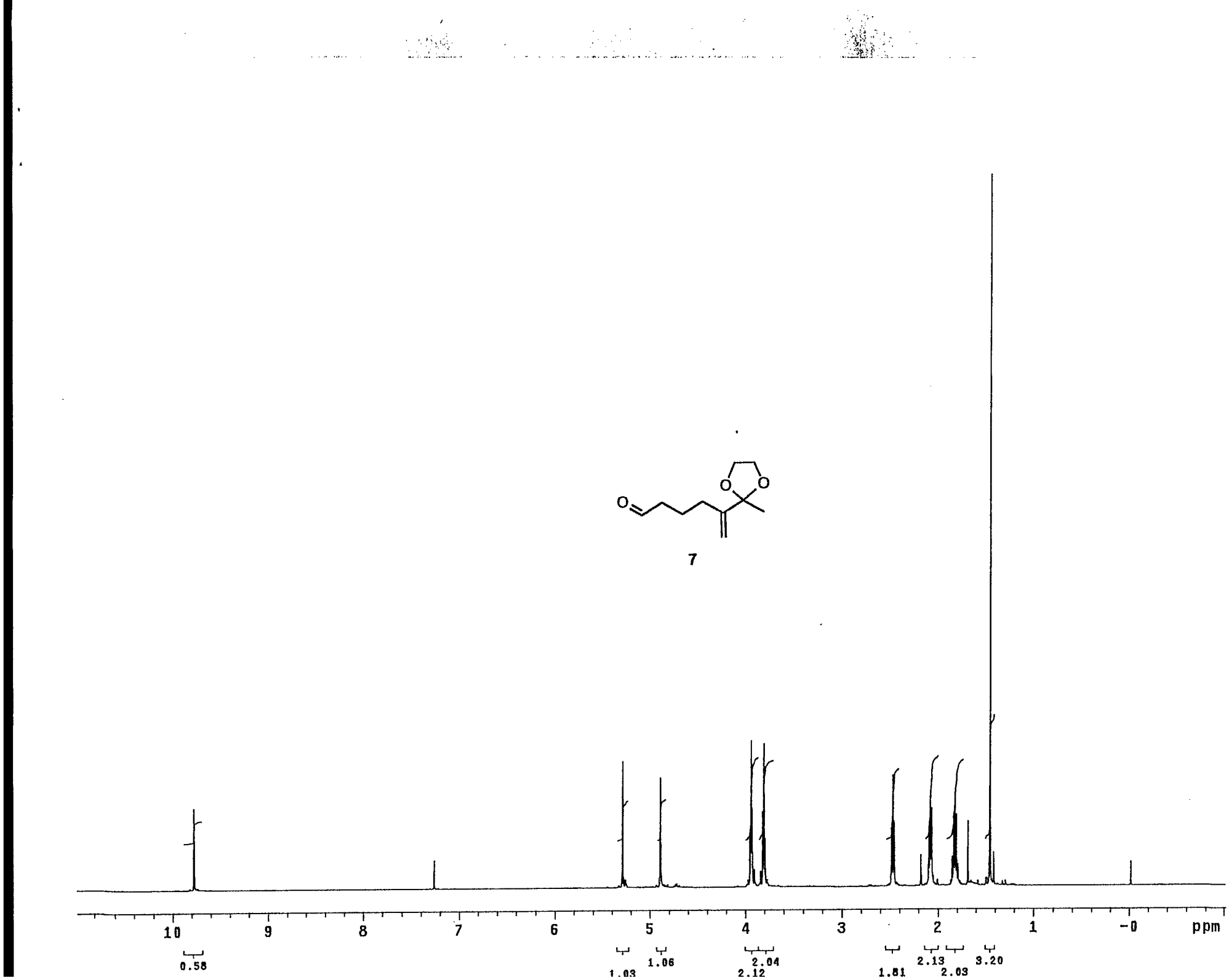




Single Mass Input Page: 1-1

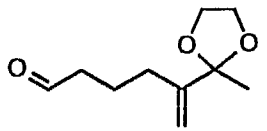
| M/E | C | H | O | PPM | DBE | ACC.MASS |
|---|---|---|---|---|---|---|
| | 12 | | | | | |
| 187 | 14 | 19 | 0 | -00.0 | 5.5 | 187.1486758 |
| | 13 | 15 | 1 | 114.4 | 6.5 | 187.1122982 |
| | 10 | 19 | 3 | 1.5 | 1.5 | 187.1334197 |

$(P+H)^+$ actual mass

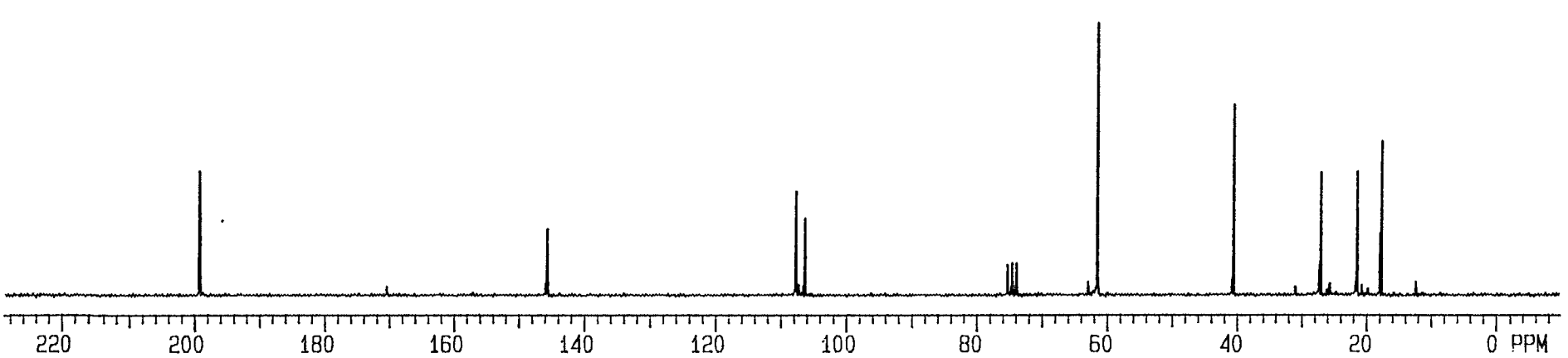
?=help RETURN=next page !S=skip !G=return to parameters !X=exit !E=editor



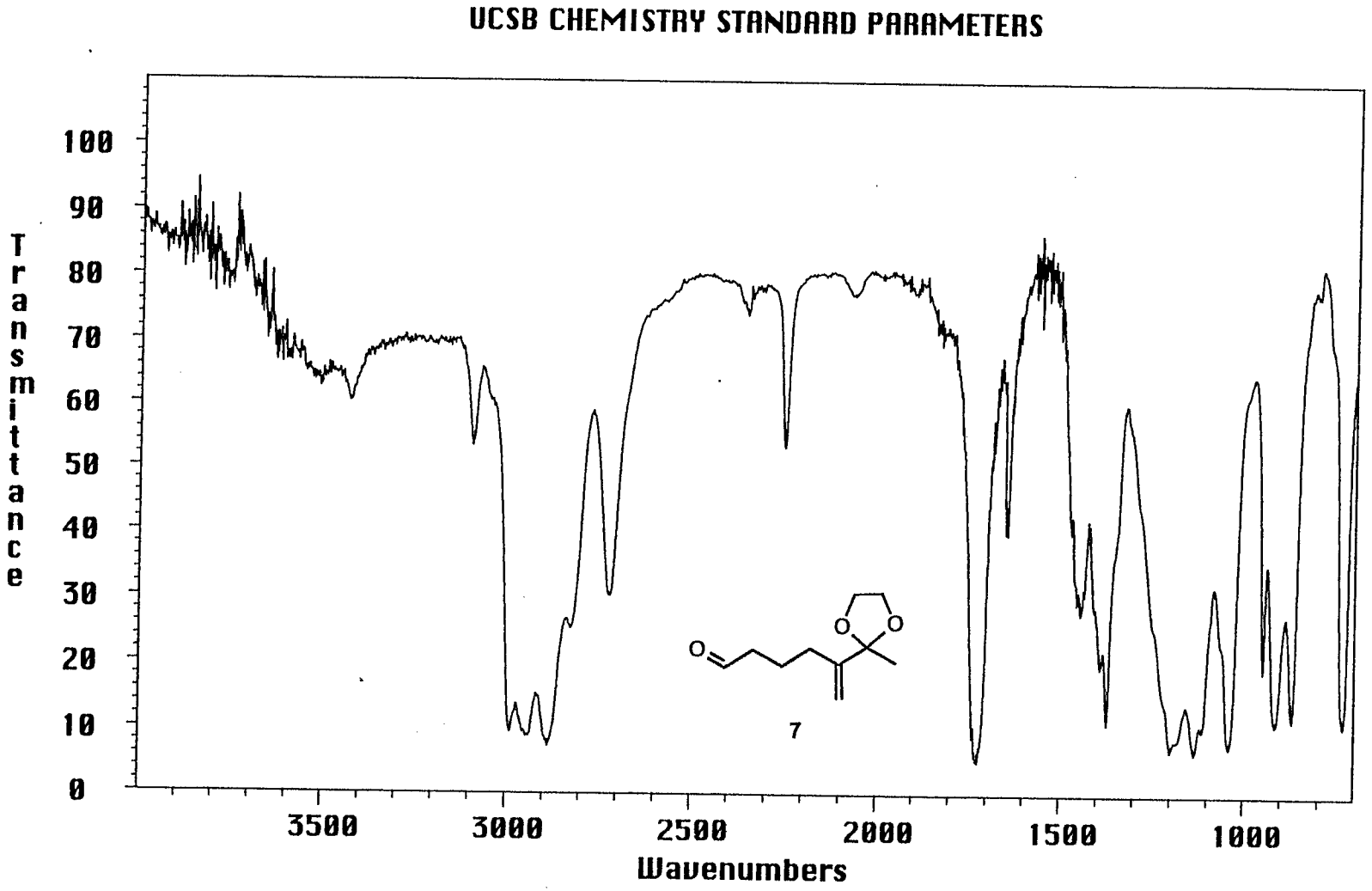

© 1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 12



7

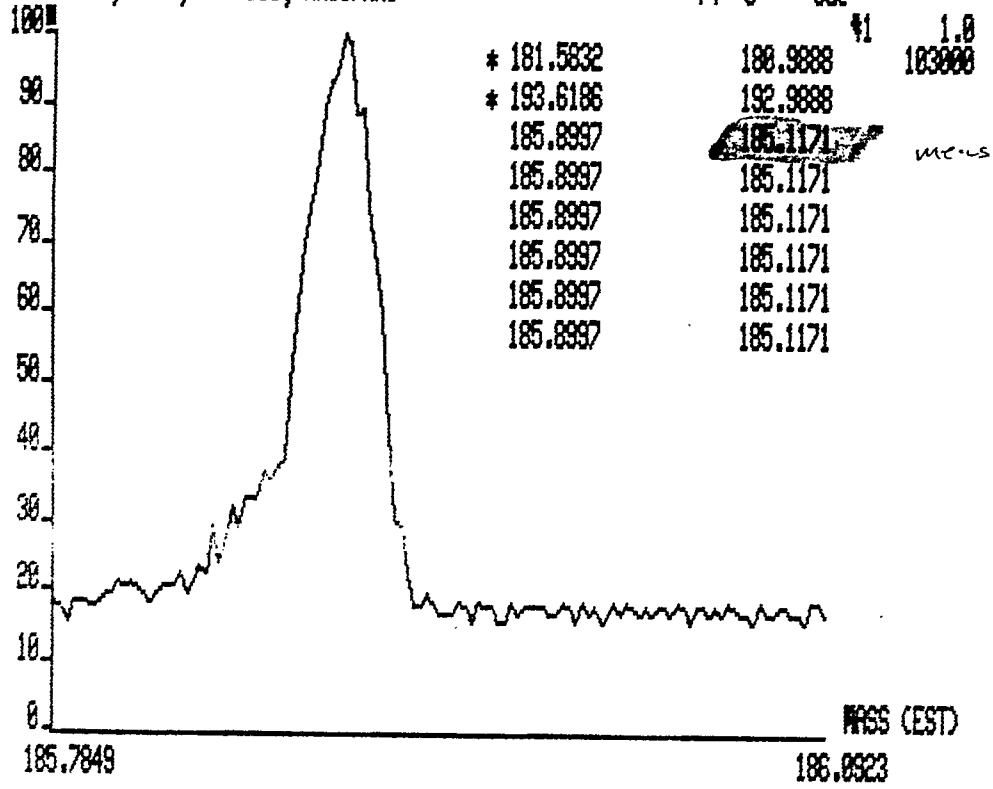



©1997 American Chemical Society Journal of Organic Chemistry Vol. 1 age 1010 Oct Supplemental Page 15



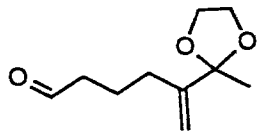

©1997 American Chemical Society Journal of Organic Chemistry Vol. 1 age 1010 Oct Supplemental Page 14

LIT03088#1 x1 Bgd=1 8-MAR-95 17:28+0:05:03 70-250MF EI+
BpM=0 I=18mv Ma=0 TIC=0 Acnt:5062E Sys:KVE
OTT/LITTLE, MW07, M/Z 185, HRCI/NH3 PT= 0° Cal:




| | | | %1 | 1.0 |
|---|---|---|---|---|
| * | 181.5832 | 186.9888 | | 103000 |
| * | 193.6186 | 192.9888 | | |
| | 185.8997 | 185.1171 | | |
| | 185.8997 | 185.1171 | | |
| | 185.8997 | 185.1171 | | |
| | 185.8997 | 185.1171 | | |
| | 185.8997 | 185.1171 | | |
| | 185.8997 | 185.1171 | | |

measured mass



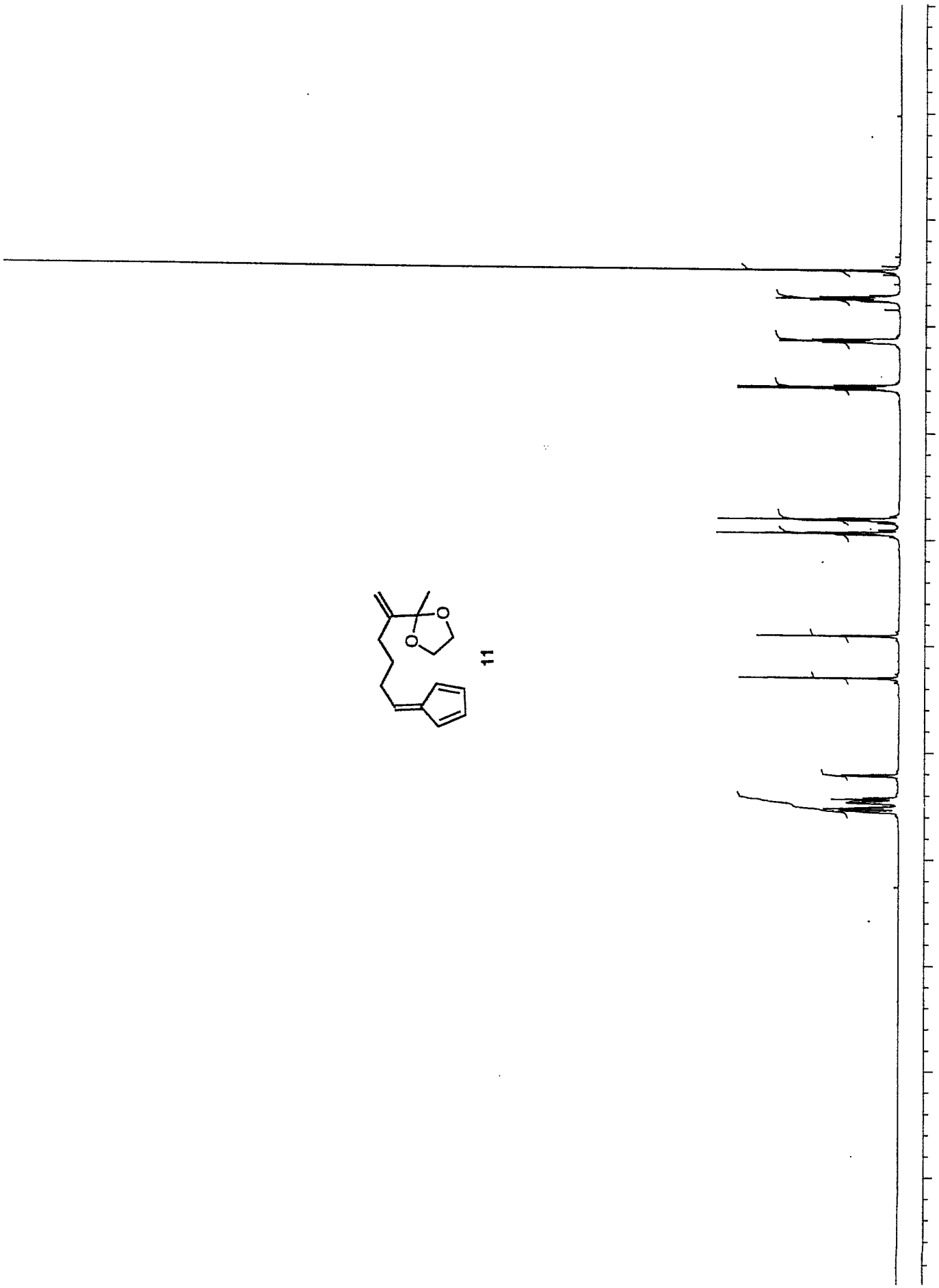
7

Single Mass Input Page: 1-1

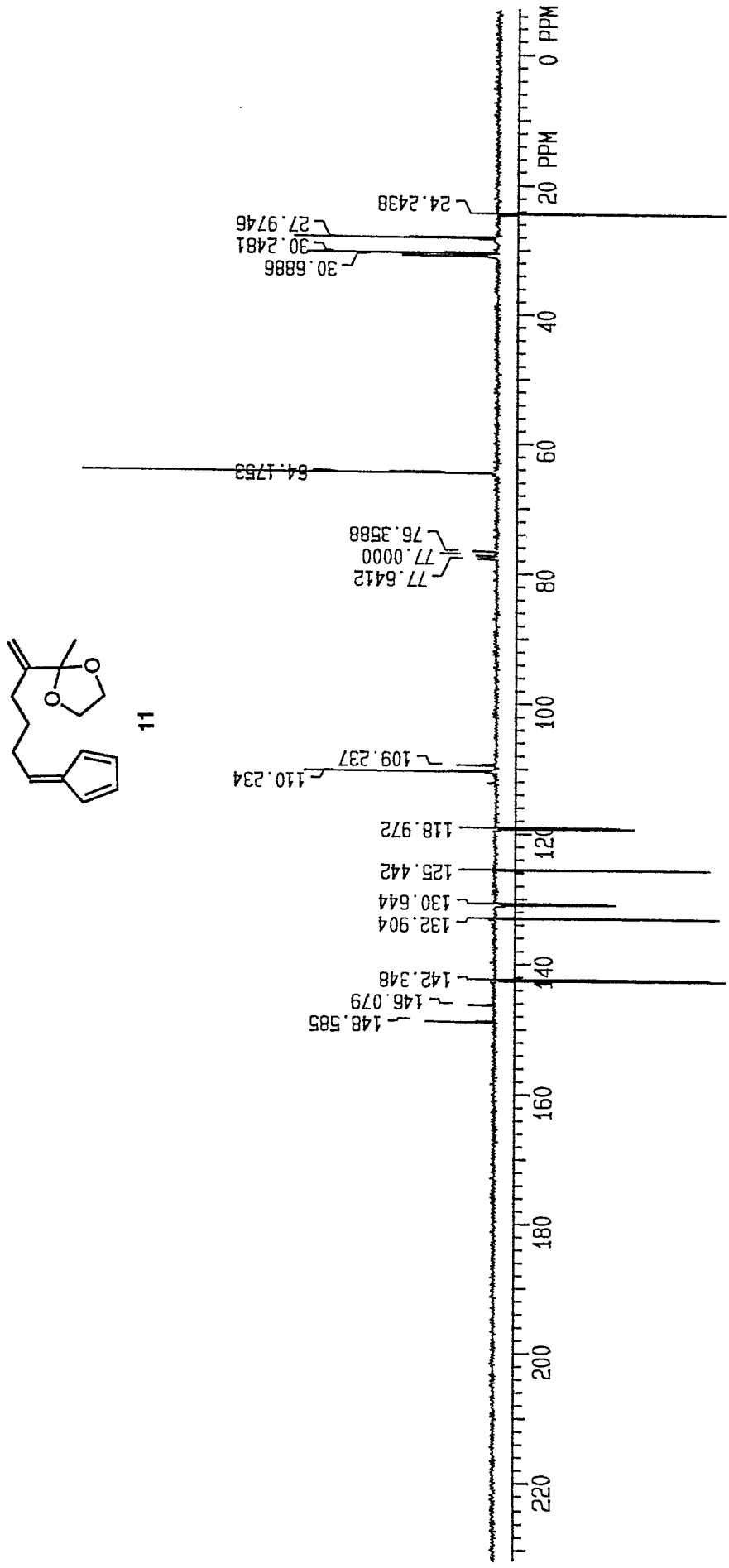
| M/E | C | H | O | PPM | DBE | RCC.MASS |
|---|---|---|---|---|---|---|
| | 12 | | | | | |
| 185 | 14 | 17 | 0 | -86.0 | 6.5 | 185.1338257 |
| | 13 | 13 | 1 | 116.5 | 7.5 | 185.0000000 |
| | 10 | 17 | 3 | -3.6 | 2.5 | 185.117696 |

$(P+H)^+$ actual mass

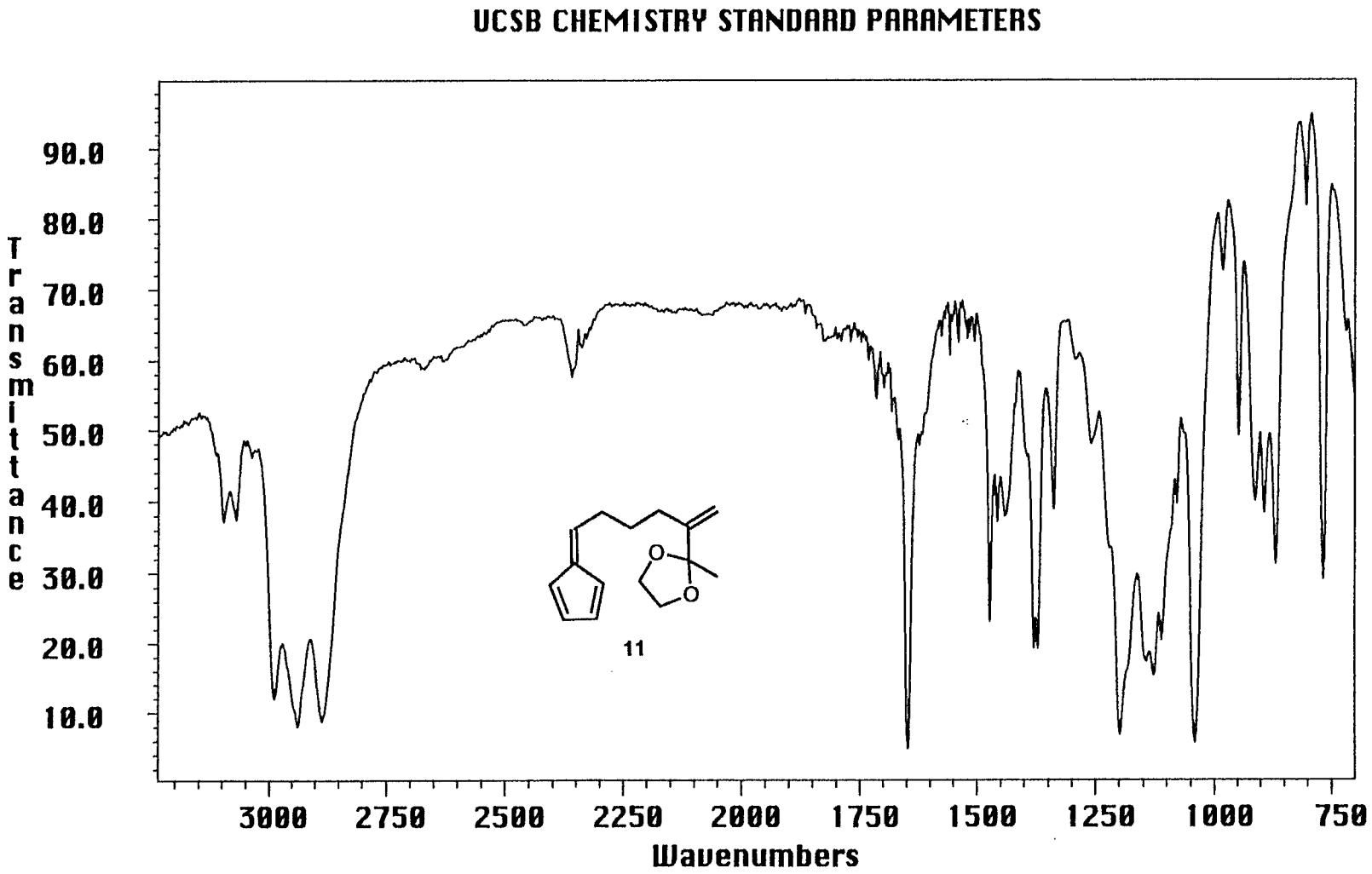
?=help RETURN=next page !S=skip !Q=return to parameters !X=exit !E=editor



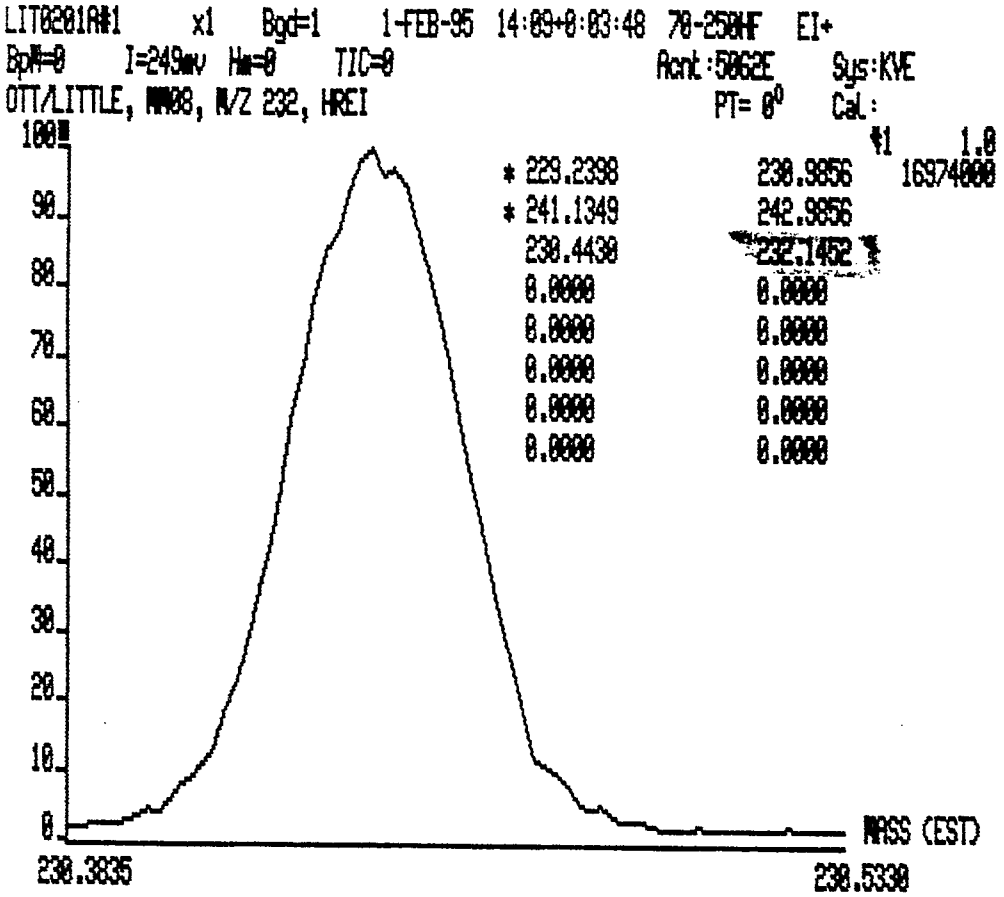
11

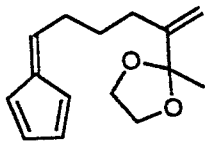

24.2438
27.9746
30.2481
30.6886
64.1753
76.3588
77.0000
77.6412
109.237
110.234
118.972
125.442
130.644
132.904
142.348
146.079
148.585
0 PPM
20 PPM
40
60
80
100
120
140
160
180
200
220

11




©1997 American Chemical Society Journal Of Organic Chemistry V62 Page 1610 Ott Supplemental Page 18



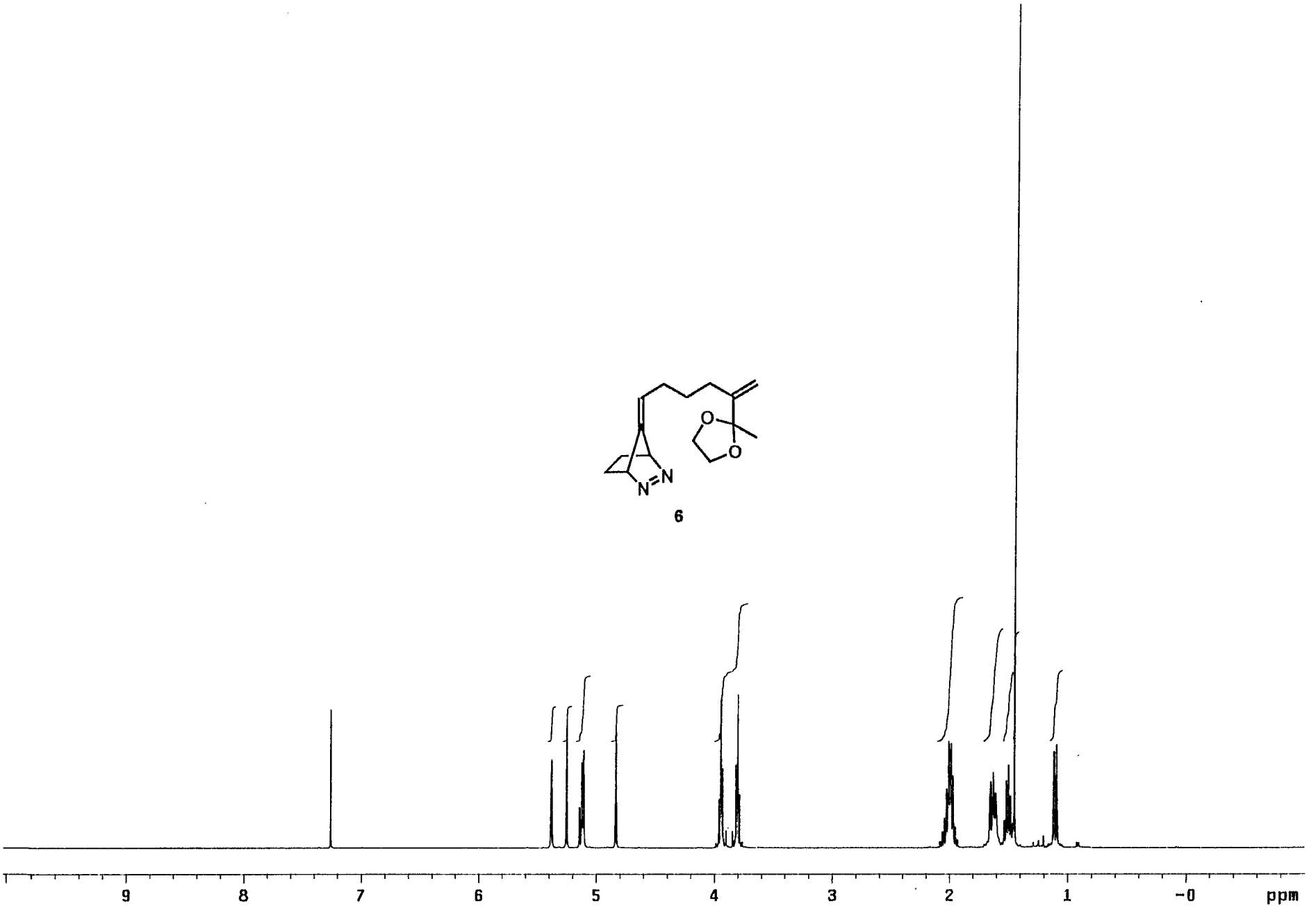



11

Single Mass Input Page: 1-1

| M/E | C | H | O | PPM | DBE | ACC.MASS |
|---|---|---|---|---|---|---|
| | 12 | | | | | |
| 232 | 18 | 16 | 0 | 86.2 | 11.0 | 232.1252006 |
| | 15 | 20 | 2 | -4.9 | 6.0 | 232.1463001 % |

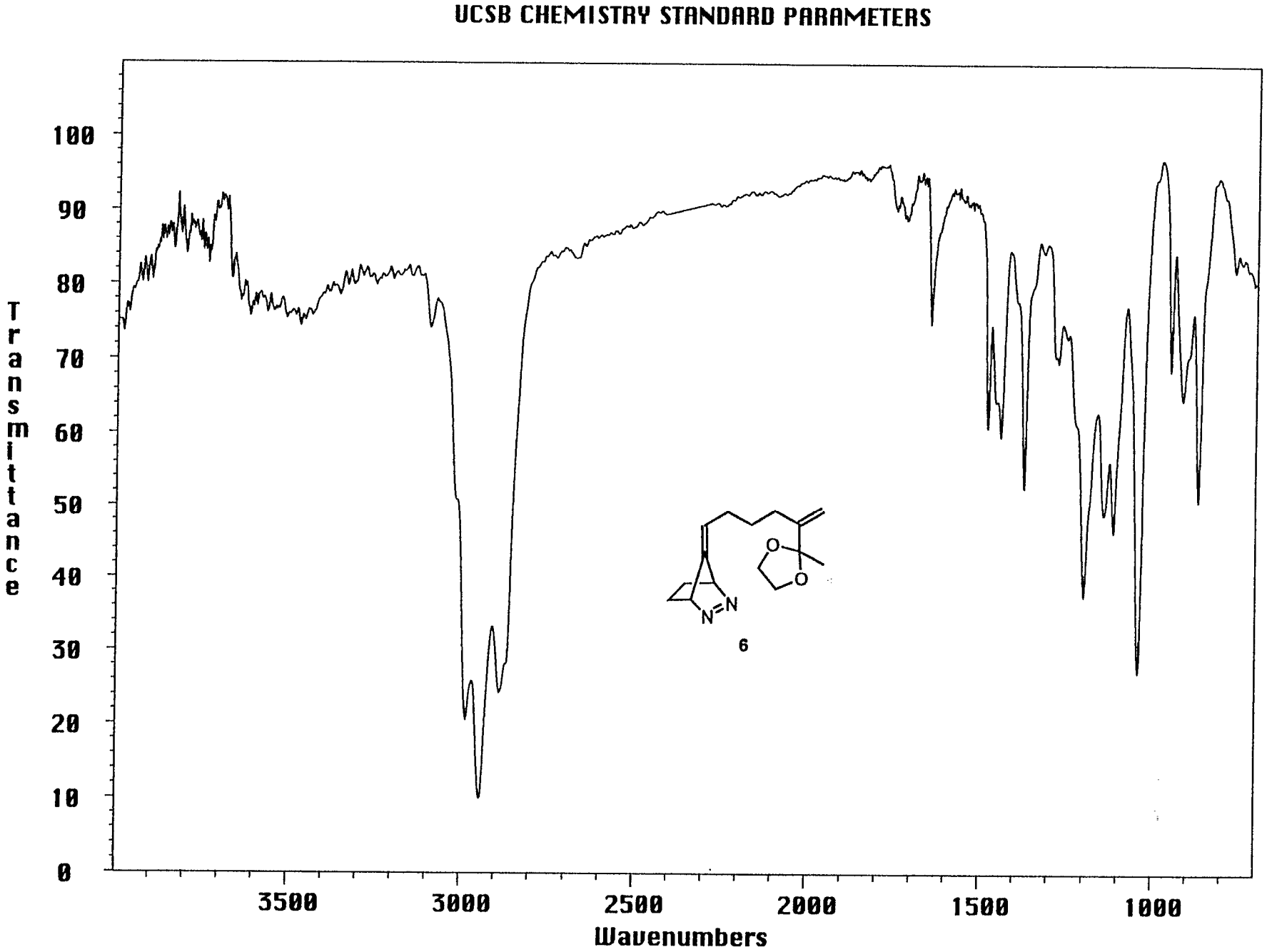
?=help RETURN=next page !S=skip !Q=return to parameters !X=exit !E=editor




©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 20

148.717
144.992
117.160
110.264
109.377
77.319
77.000
76.848
76.681
72.615
64.347
30.051
28.875
28.048
24.354
21.509
21.062

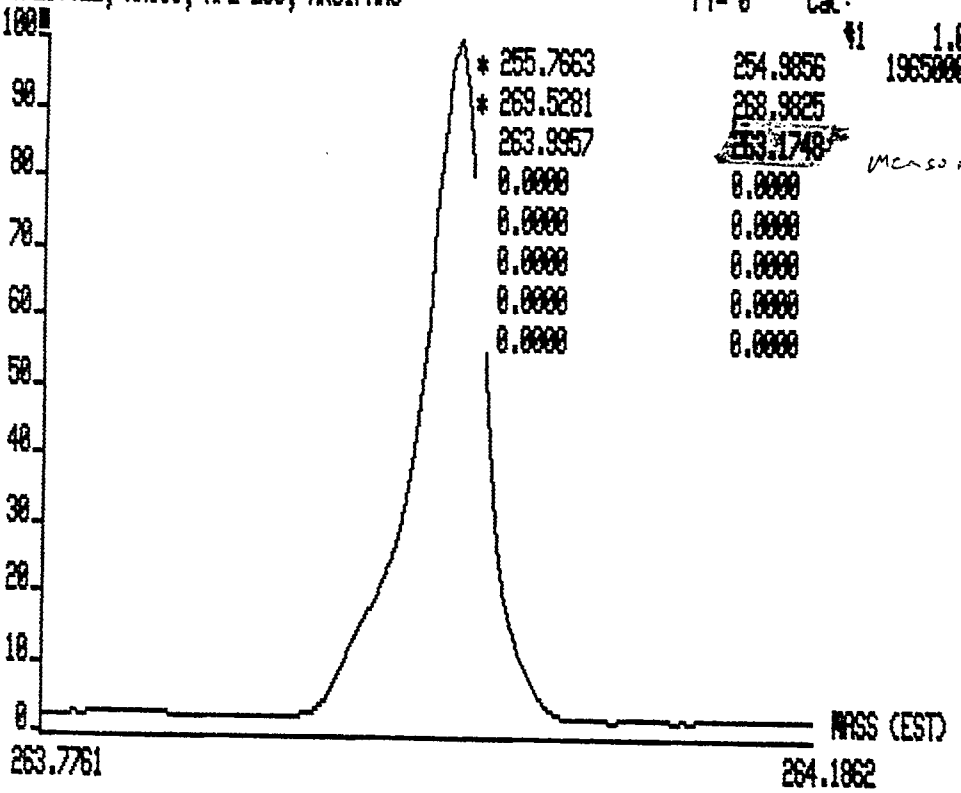
6

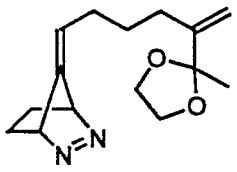



©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 22

LIT0368#1 x1 Bgd=1 8-MAR-95 16:54+0:05:14 70-250HF EI+
Cent=4 I=1.5av Hw=0 TIC=0 Mont:5862E Sys:KVE
OTT/LITTLE, MM#10, M/Z 263, HRCI/NH3 PT= 0° Cal:

| | | | |
|---|---|---|---|
| | | #1 | 1.0 |
| * | 255.7663 | 254.9856 | 1965000 |
| * | 269.5281 | 268.9825 | |
| | 263.9957 | 263.1748 | measured mass |
| | 0.0000 | 0.0000 | |
| | 0.0000 | 0.0000 | |
| | 0.0000 | 0.0000 | |
| | 0.0000 | 0.0000 | |
| | 0.0000 | 0.0000 | |



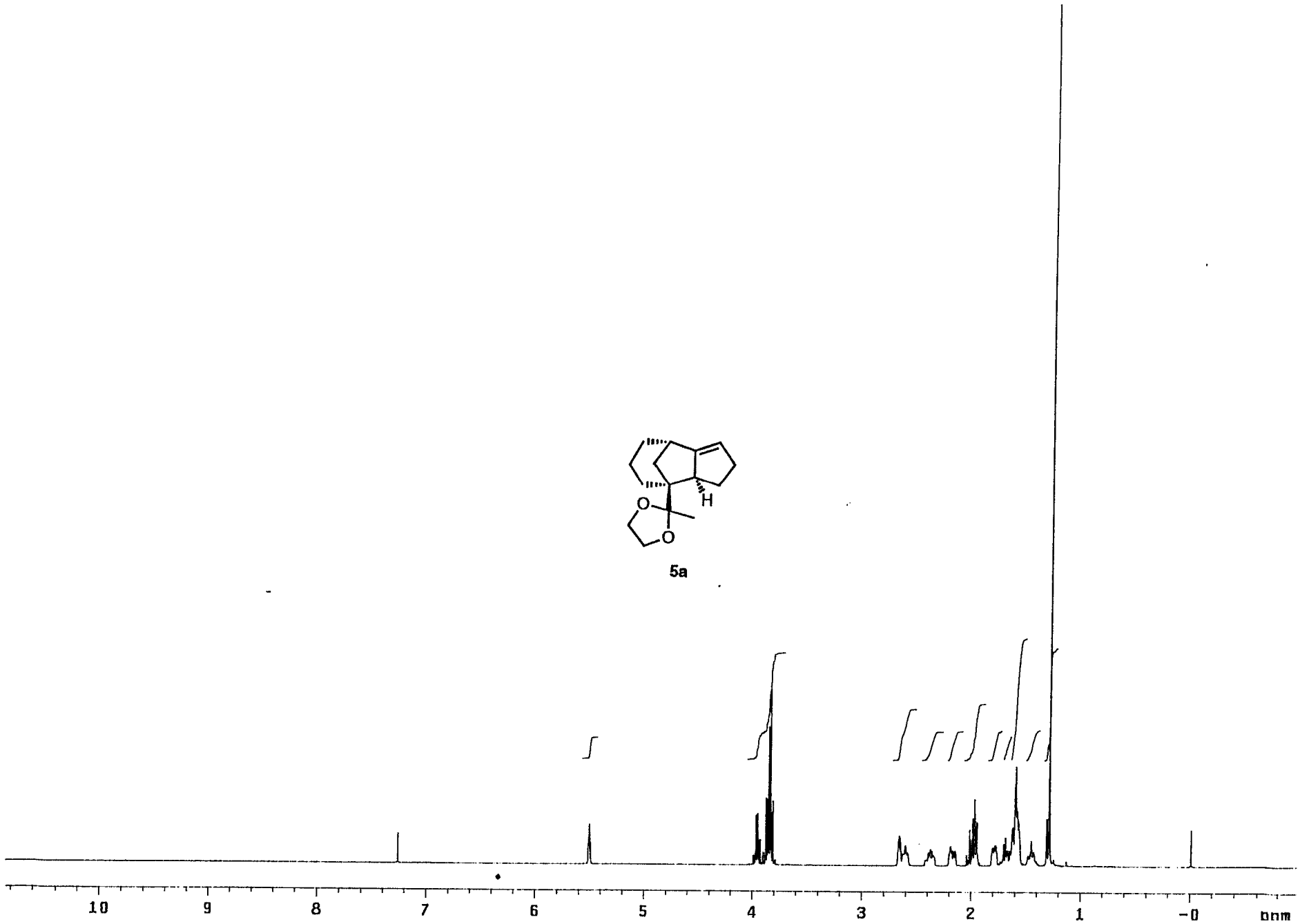

6

Single Mass Input Page: 1-1

| M/E | C | H | O | N | PPM | DBE | ACC.MASS |
|---|---|---|---|---|---|---|---|
| | 12 | | | | | | |
| 263 | 20 | 23 | 0 | 0 | -19.7 | 9.5 | 263.1799759 |
| | 19 | 21 | 0 | 1 | 28.1 | 10.0 | 263.1673998 |
| | 18 | 19 | 0 | 2 | 75.9 | 10.5 | 263.1548238 |
| | 15 | 25 | 1 | 3 | -94.8 | 5.0 | 263.1997627 |
| | 17 | 27 | 2 | 0 | -99.9 | 4.5 | 263.2011054 |
| | 16 | 25 | 2 | 1 | -52.2 | 5.0 | 263.1885293 |
| | 15 | 23 | 2 | 2 | -4.4 | 5.5 | 263.1759532 |
| | 14 | 21 | 2 | 3 | 43.4 | 6.0 | 263.1633771 |
| | 16 | 23 | 3 | 0 | 38.3 | 5.5 | 263.1647198 |
| | 15 | 21 | 3 | 1 | 86.1 | 6.0 | 263.1521438 |

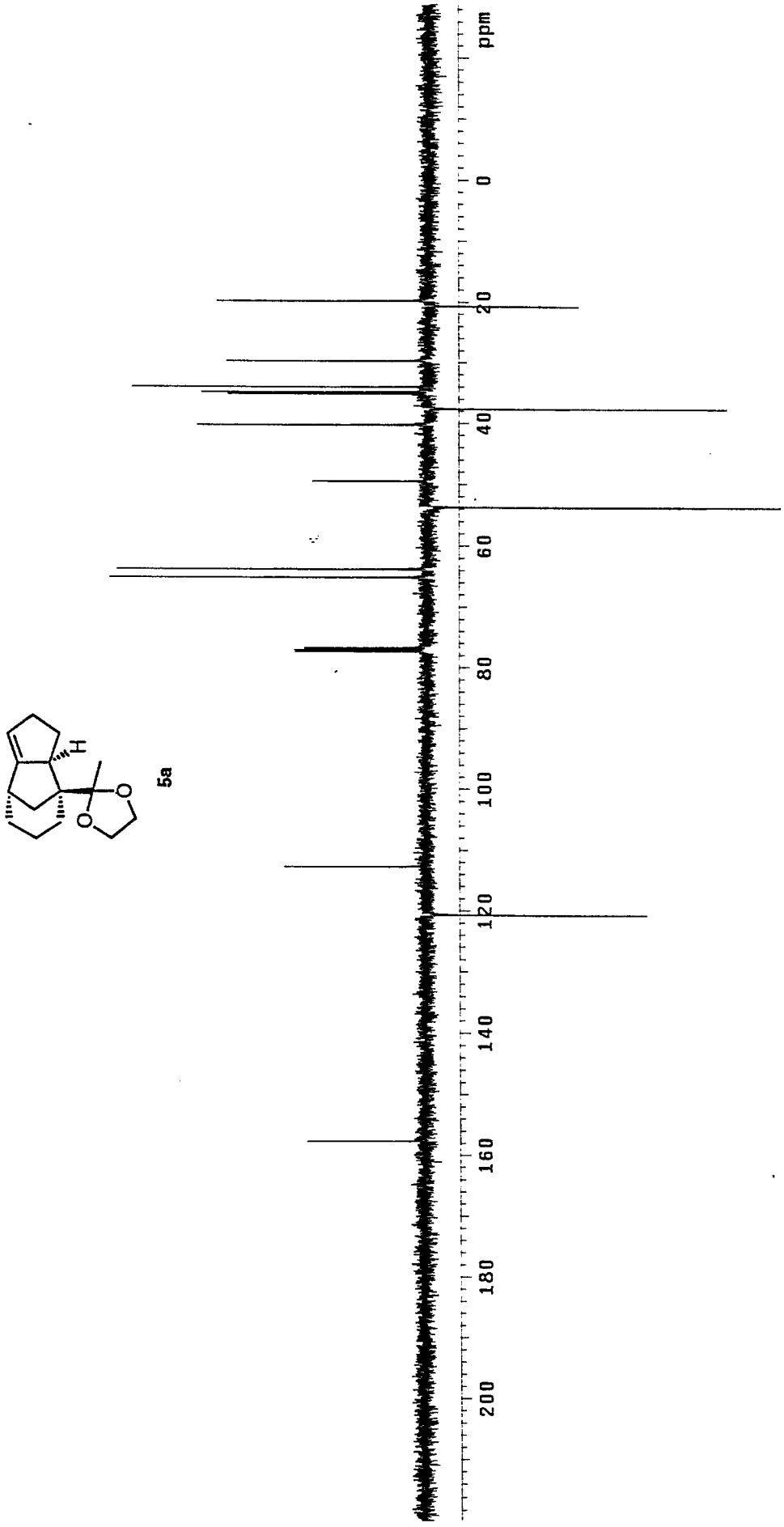
$(P+H)^+$ calcd. mass

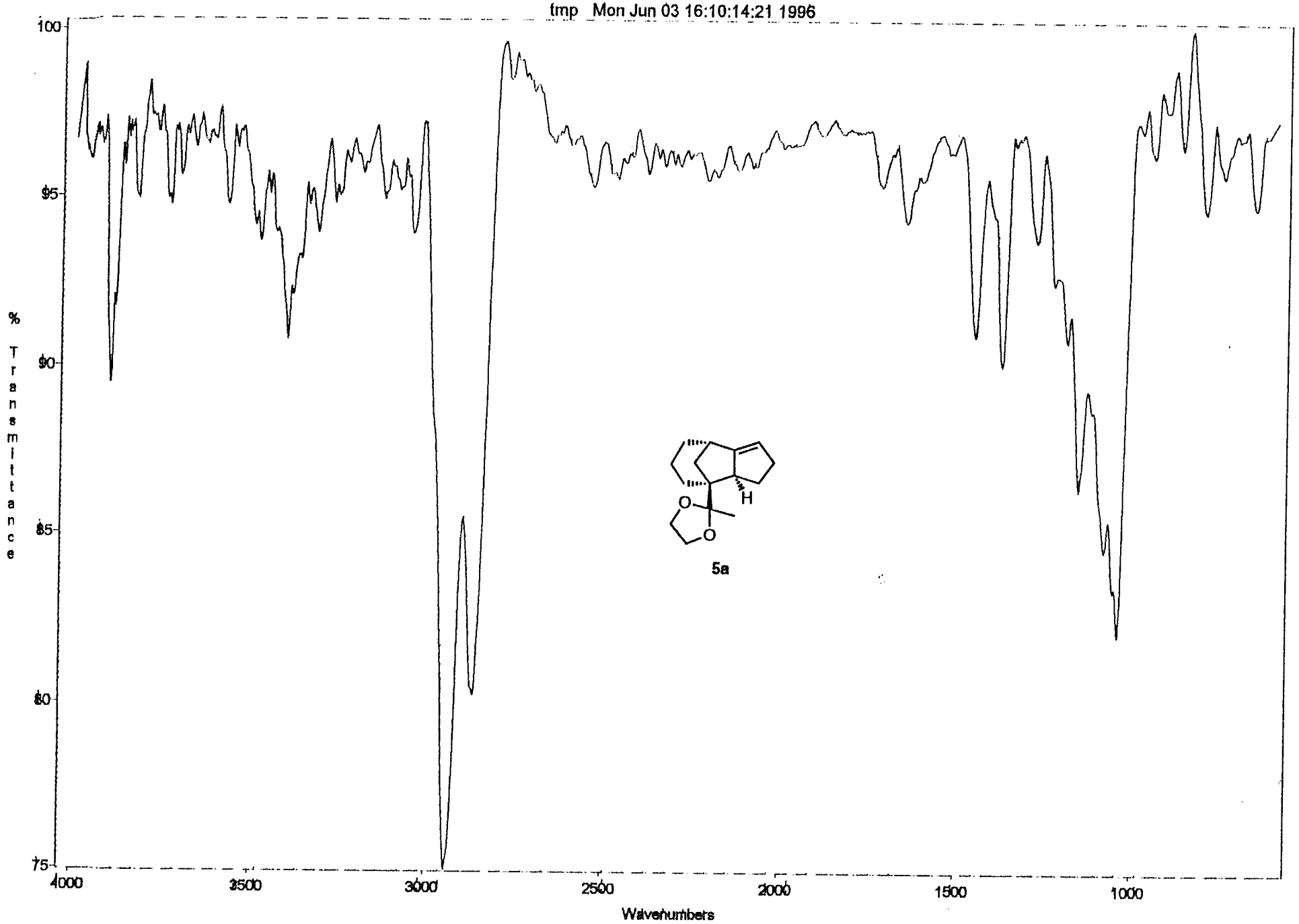
?=help RETURN=next page !S=skip !Q=return to parameters !X=exit !E=editor




©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 24

©1997 American Chemical Society Journal Of Organic Chemistry V62 Page 1010 Ott Supplemental Page 25





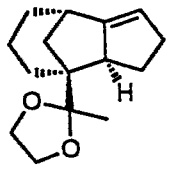

5a

©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1610 Off Supplemental Page 26

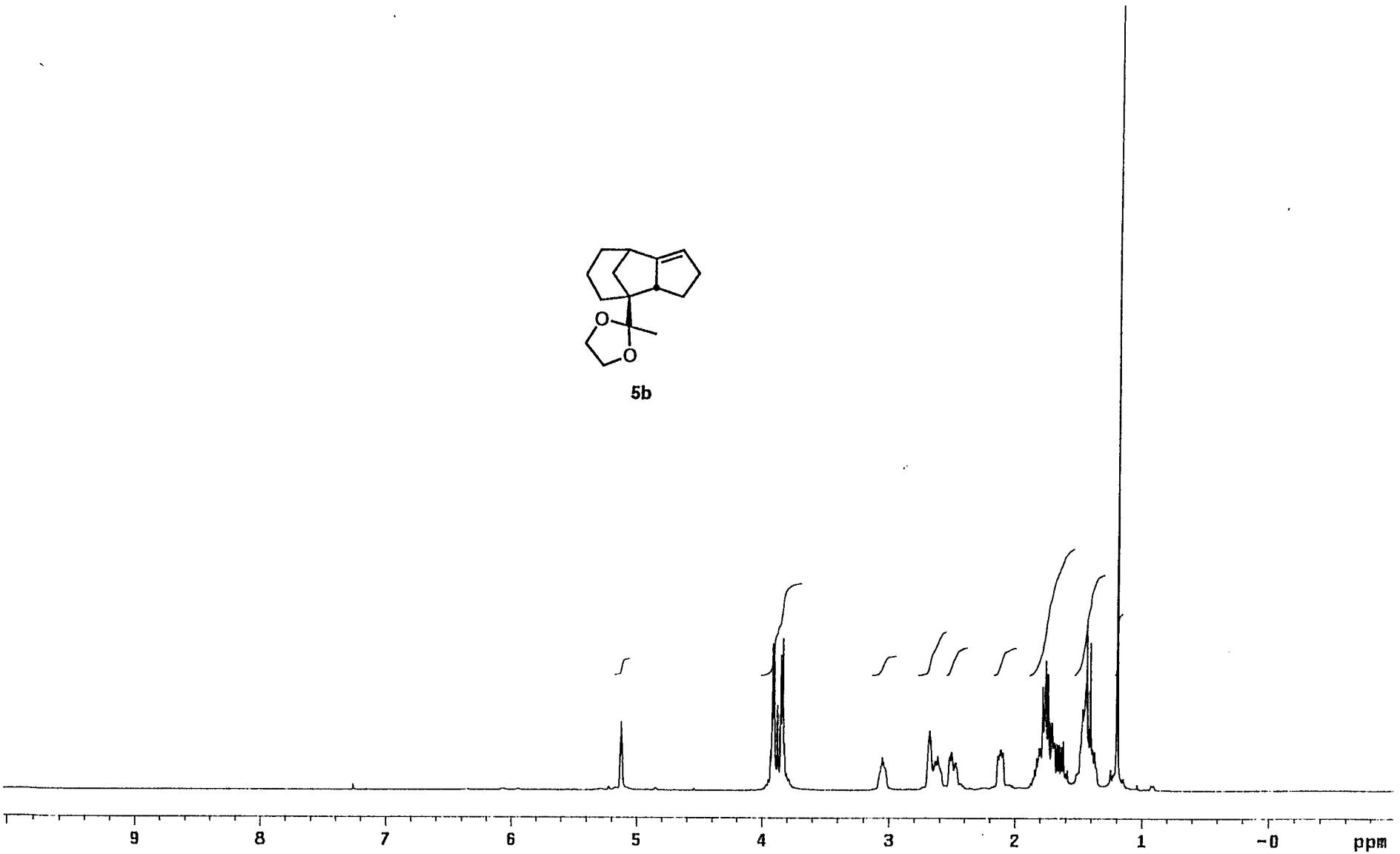
**Elemental Composition** **Date : 7-JUN-1996**

File:LIT0607B Ident:35_37 SMO(1,7) PKD(7,3,7,0.01%,0.0,0.00%,F,F)
70S EI+ Voltage BpI:388304 TIC:13077025 Flags:NORM
File Text:OTT/LITTLE, MM09, M/Z 234, HREI
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 234.161107 | | 20.0 | | -0.5<br>20.0 | 0<br>200 | 0<br>400 | 0<br>20 |
| **Mass** | **mDa** | **PPM** | **Calc. Mass** | **DBE** | **C** | **H** | **O** |
| 234.161107 | 0.9 | 3.7 | 234.161980 | 5.0 | 15 | 22 | 2 |

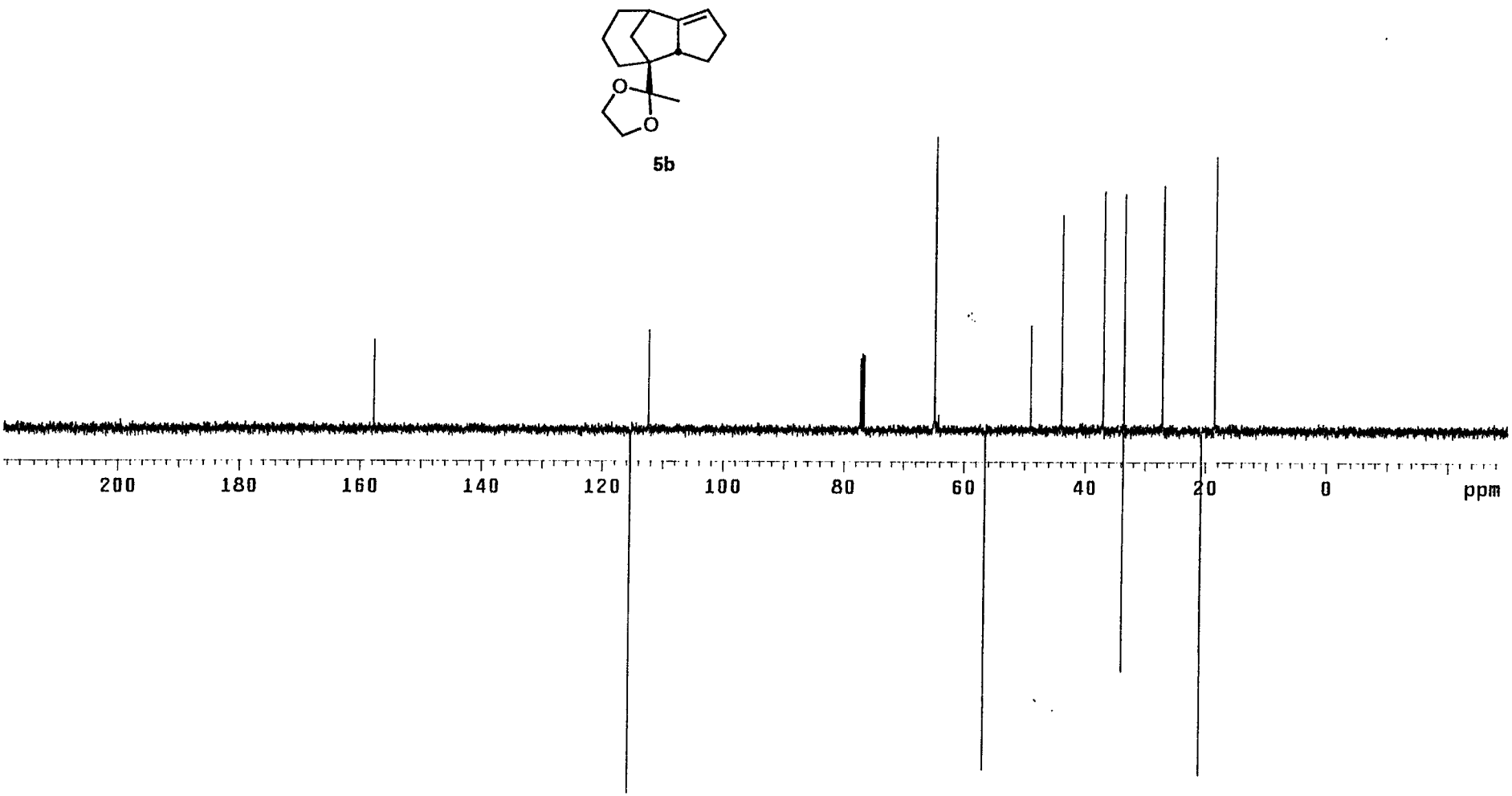



**5a**



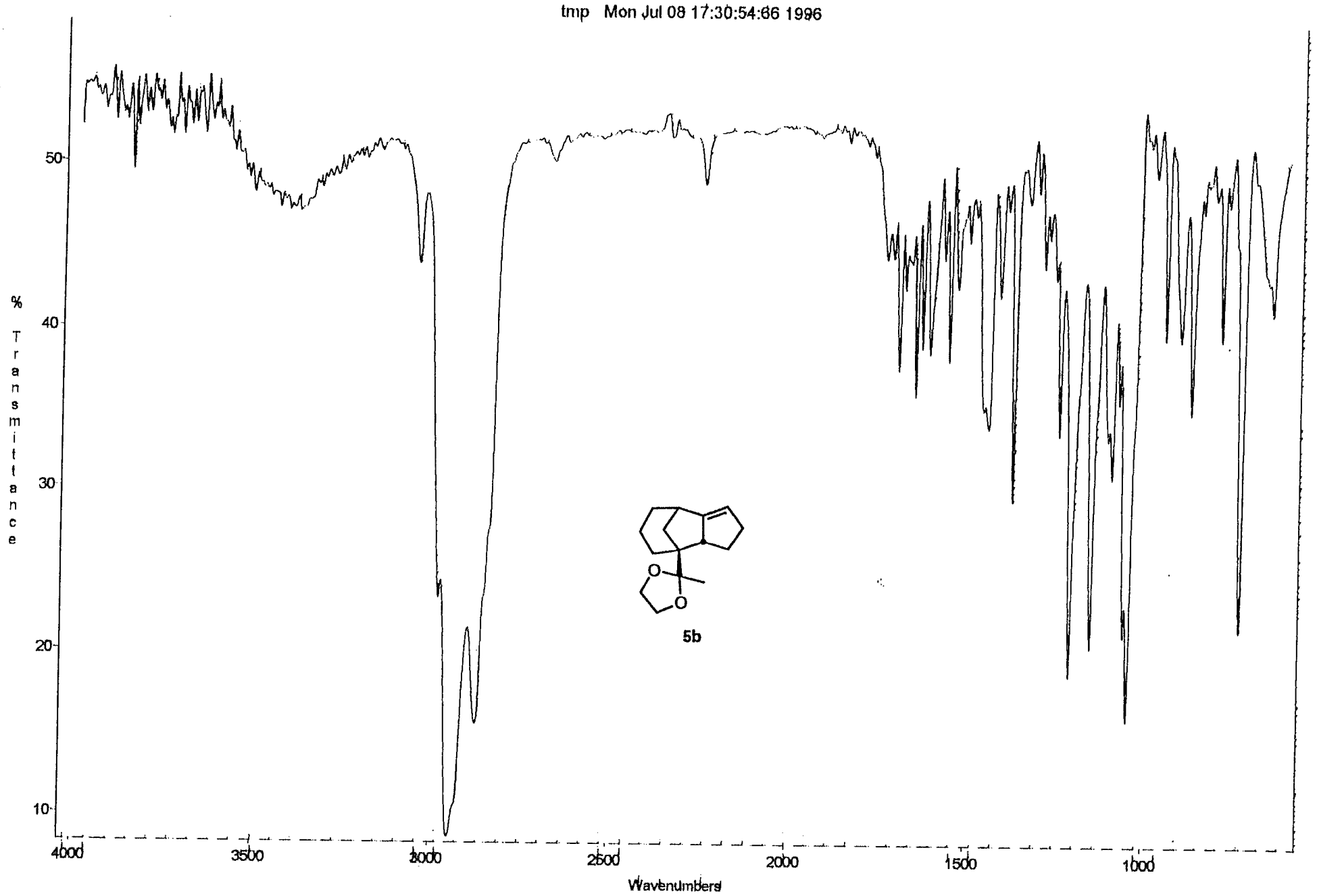

5b

©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 26



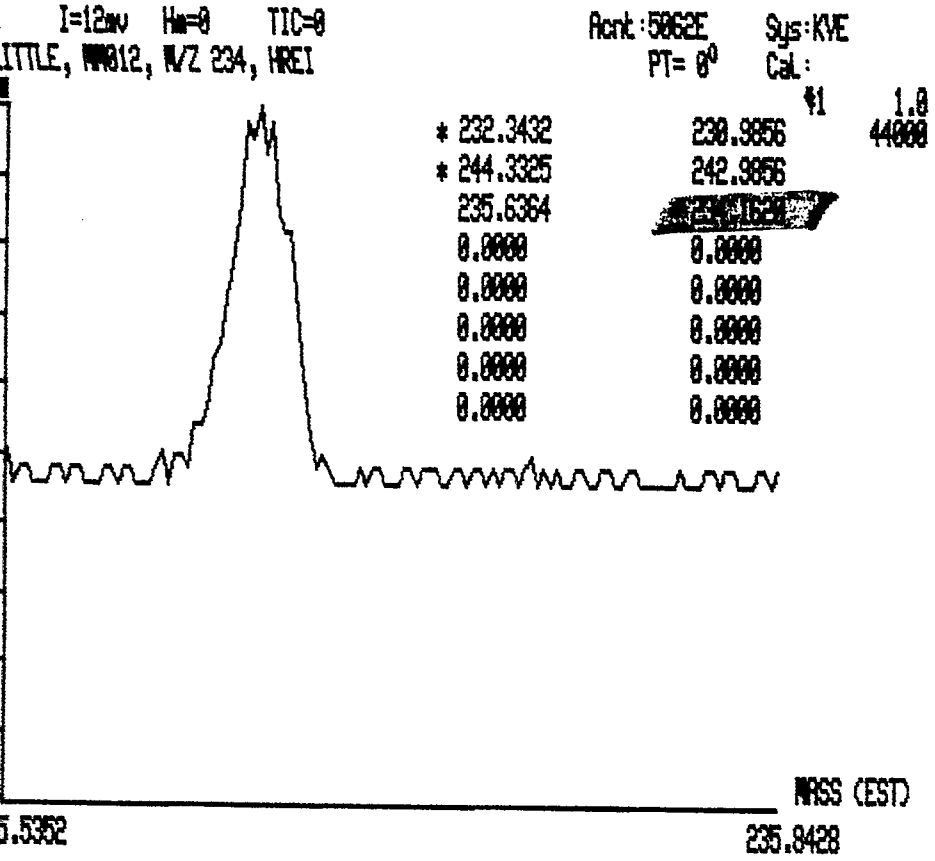

5b

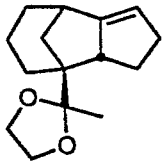
©1997 American Chemical Society 




5b

©1997 American Chemical Society Journal Of Organic Chemistry Vol. Page 1010 Of Supplemental Page 50



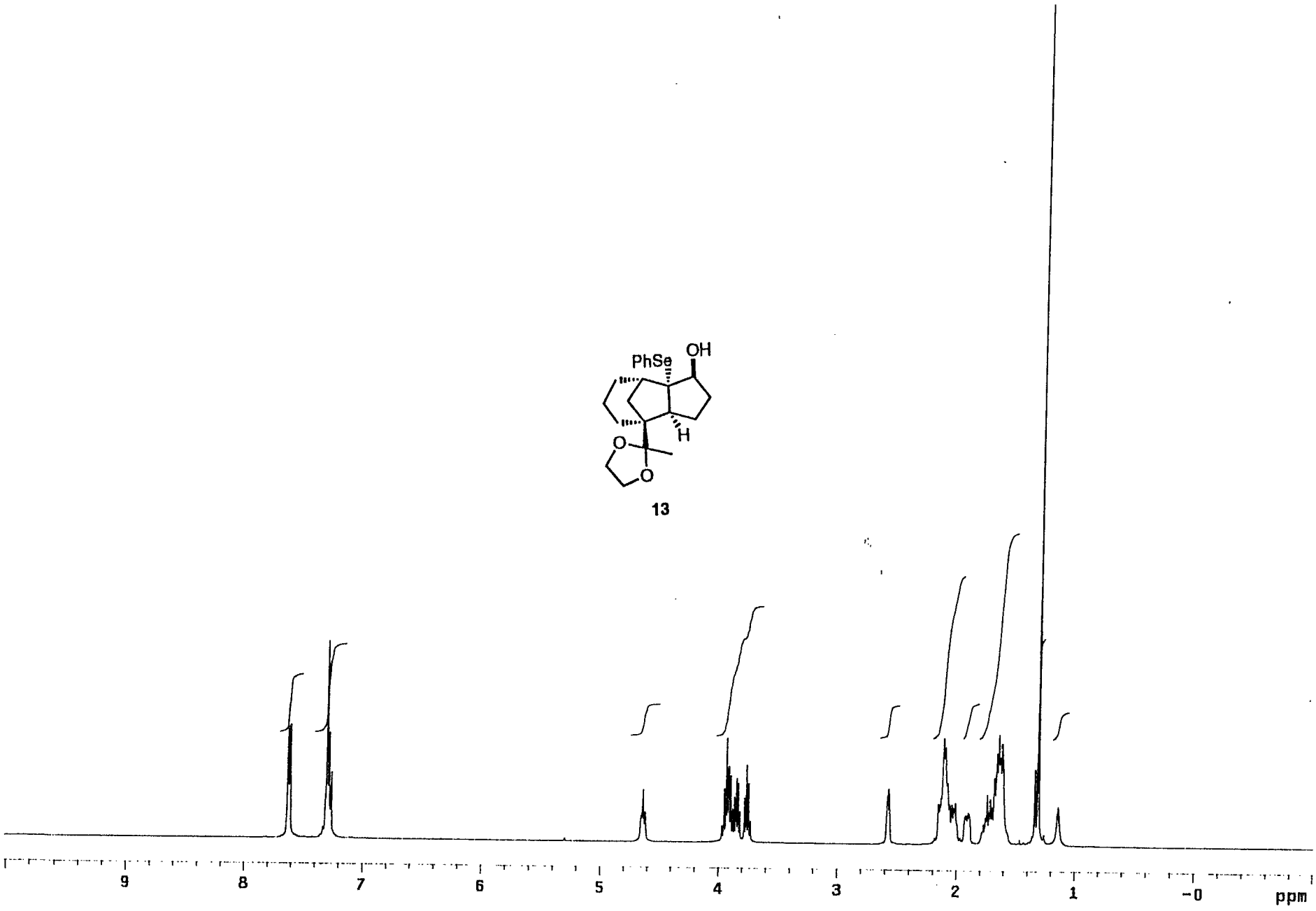



5b

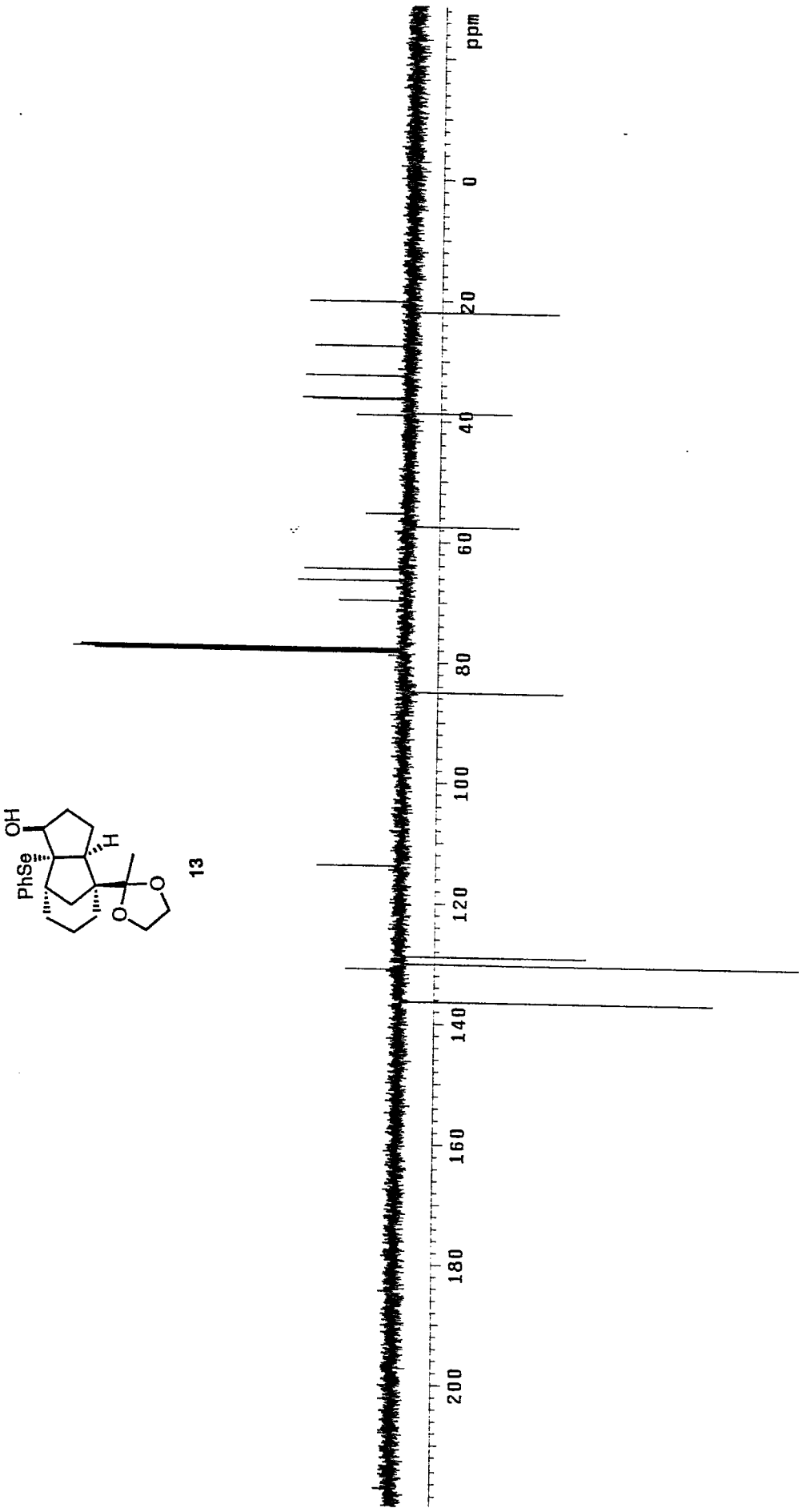
Single Mass Input Page: 1-1

| MVE | C | H | O | PPM | DBE | ACC.MASS |
|---|---|---|---|---|---|---|
| | 12 | | | | | |
| 234 | 18 | 18 | 0 | 90.3 | 10.0 | 234.1408587 |
| | 15 | 22 | 2 | 90.1 | 5.0 | 234.1619882 |
| | 12 | 26 | 4 | -90.1 | 0.0 | 234.1831096 |

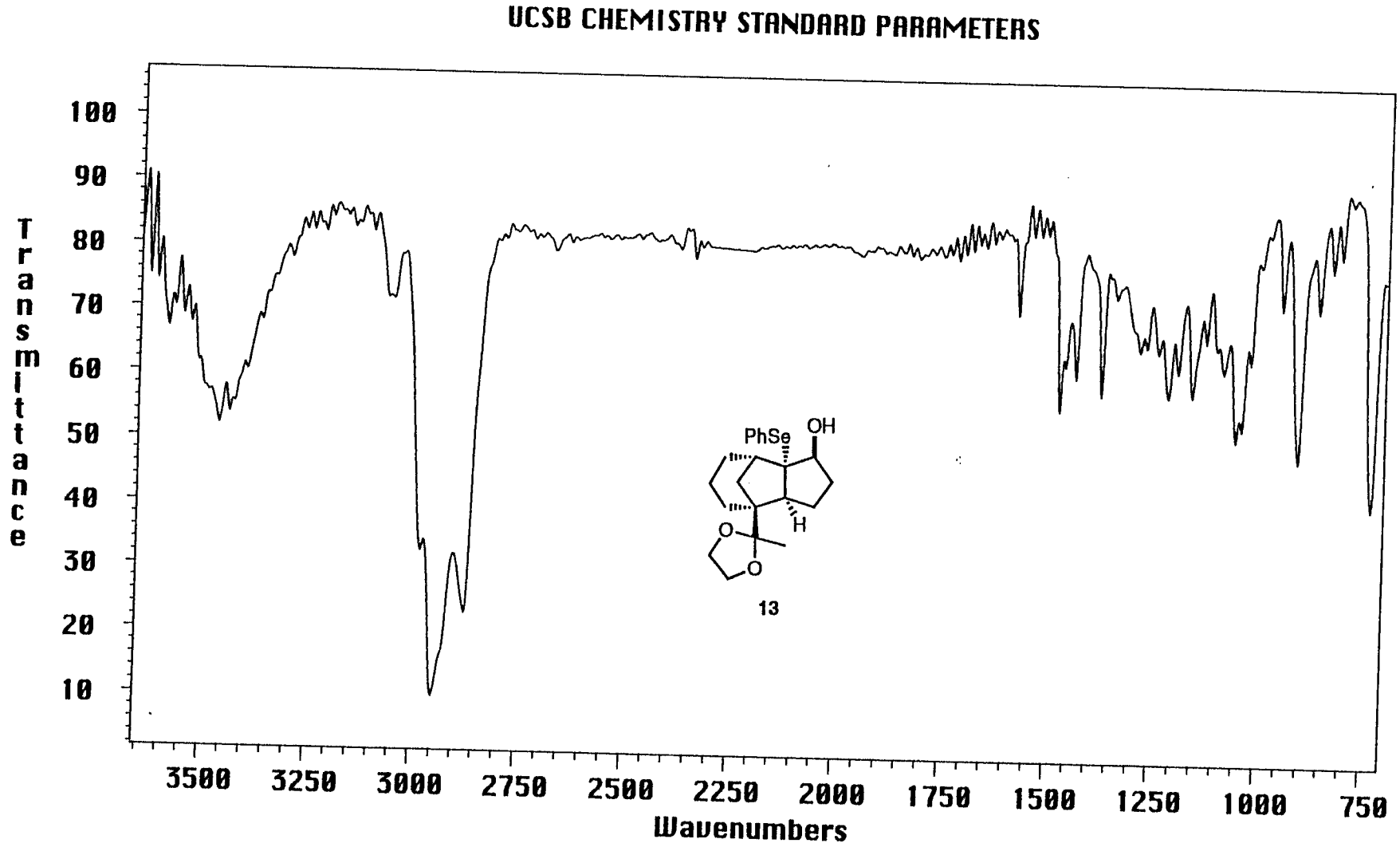
?=help RETURN=next page !S=skip !G=return to parameters !X=exit !E=editor



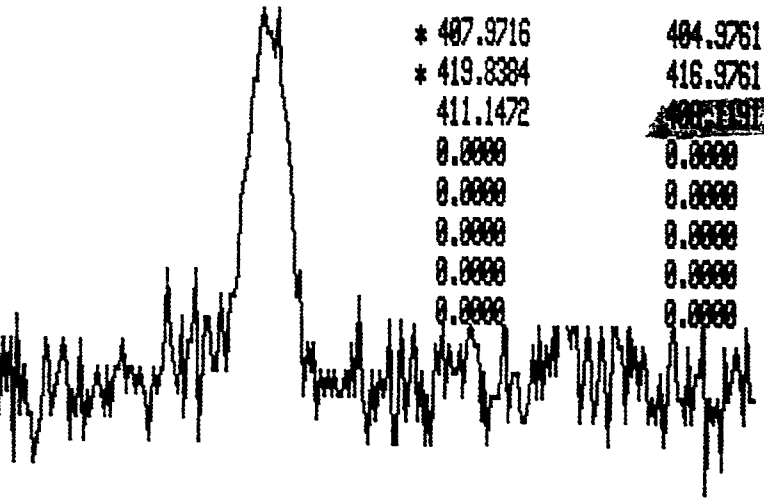

©1997 American Chemical Society Journal Of Organic Chemistry V62 Page 1610 Ott Supplemental Page 32


OH
H
PhSe
O
O
13
ppm
0
20
40
60
80
100
120
140
160
180
200

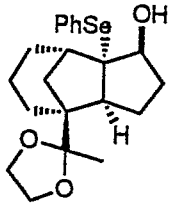
©1997 American Chemical Society Journal Of Organic Chemistry V62 Page 1610 Ott Supplemental Page 34



I=1.4mv Hm=0 TIC=0 Rcnt:5862E Sys:KVE
TLE, MM813, M/Z 408, HREI PT= 0° Cal:




| | %1 | 1.0 |
|---|---|---|
| 404.9761 | | 29000 |
| 416.9761 | | |



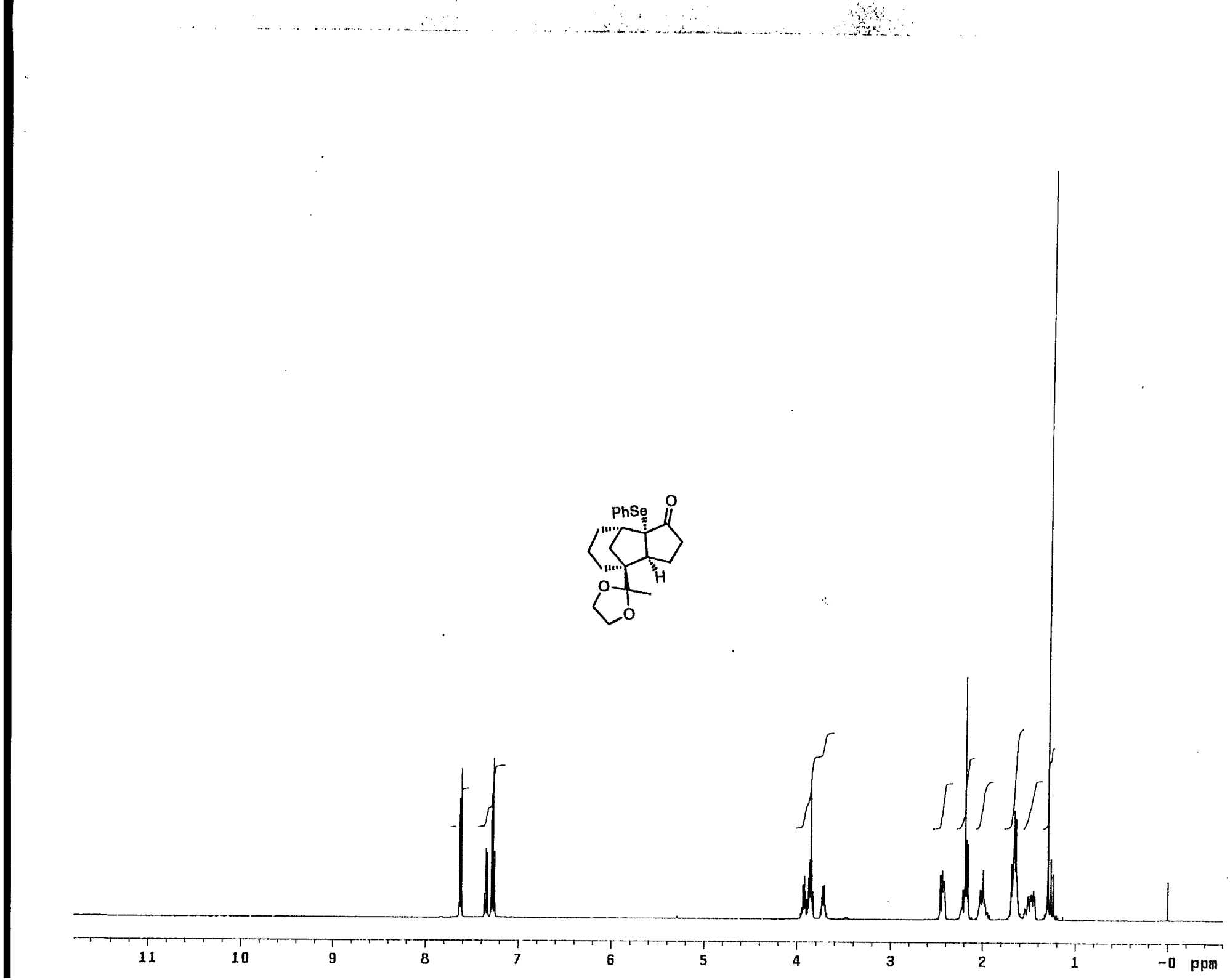

13

MASS (EST)
3174 411.7189

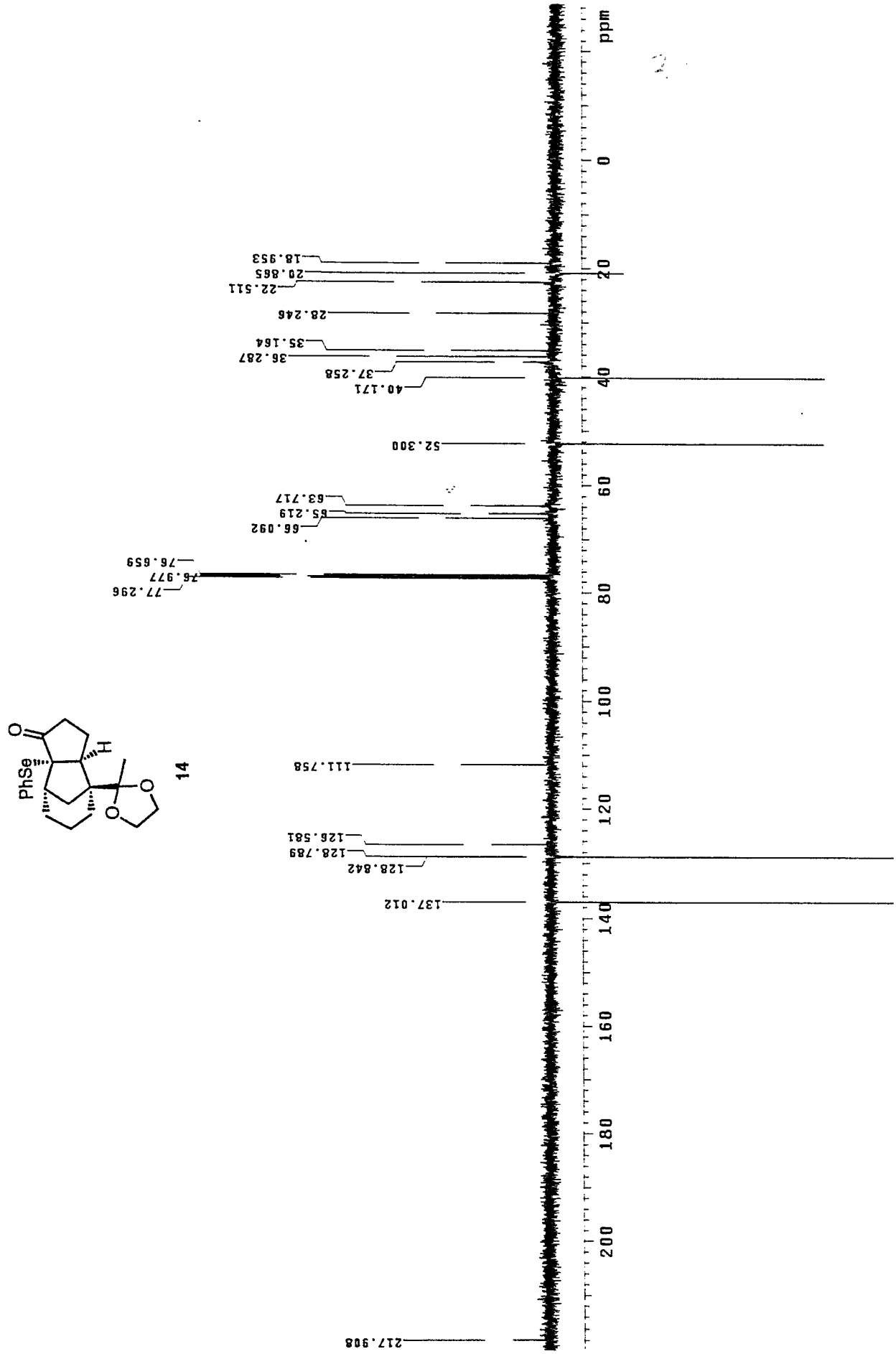
Single Mass Input Page: 1-1

| M/E | C | H | O | SE | PPM | DBE | ACC.MASS |
|---|---|---|---|---|---|---|---|
| | 12 | | | | | | |
| 408 | 25 | 28 | 0 | 1 | -49.5 | 12.0 | 408.1356216 |
| | 24 | 24 | 1 | 1 | 48.7 | 13.0 | 408.0992361 |
| | 15 | 36 | 2 | 2 | 35.6 | -2.0 | 408.1045717 |
| | 21 | 28 | 3 | 1 | -3.1 | 8.0 | 408.1283655 |
| | 27 | 20 | 4 | 0 | -41.8 | 18.0 | 408.1361594 |
| | 18 | 32 | 5 | 1 | -54.9 | 3.0 | 408.1414950 |

?=help RETURN=next page !S=skip !Q=return to parameters !X=exit !E=editor



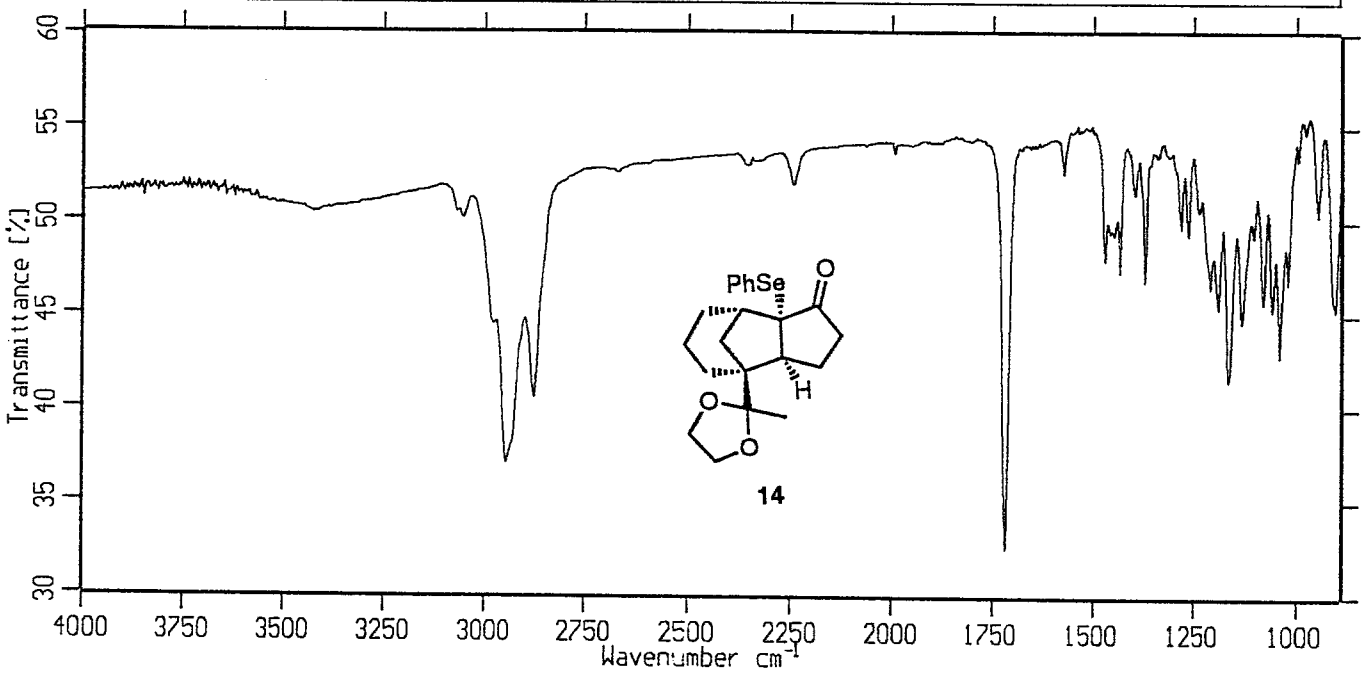

©1997 American Chemical Society


ppm
0
20
40
60
80
100
120
140
160
180
200
18.953
20.865
22.511
28.246
35.164
36.287
37.258
40.171
52.300
63.717
65.219
66.092
76.659
76.977
77.296
111.758
126.581
128.789
128.842
137.012
217.908
PhSe
H
O
O
O
14

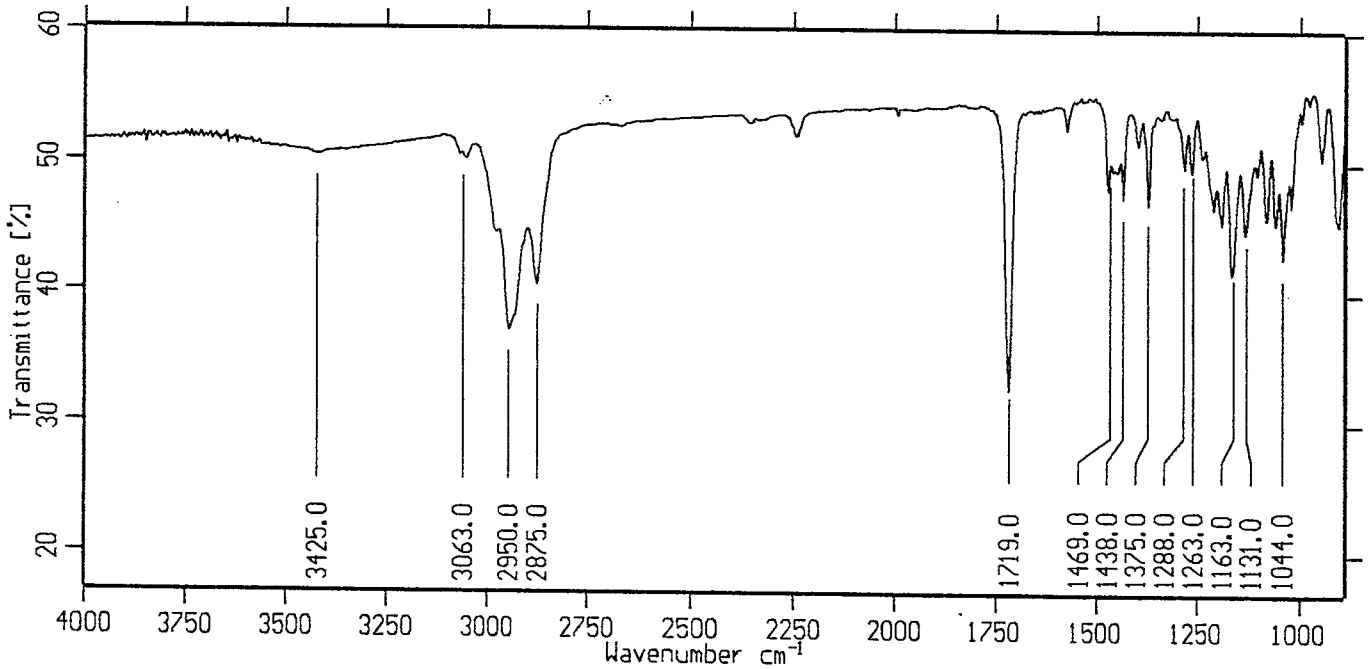
SAMPLE NAME :
OPERATOR :
SAMPLE FORM :
FILENAME :BACKGROUND.14

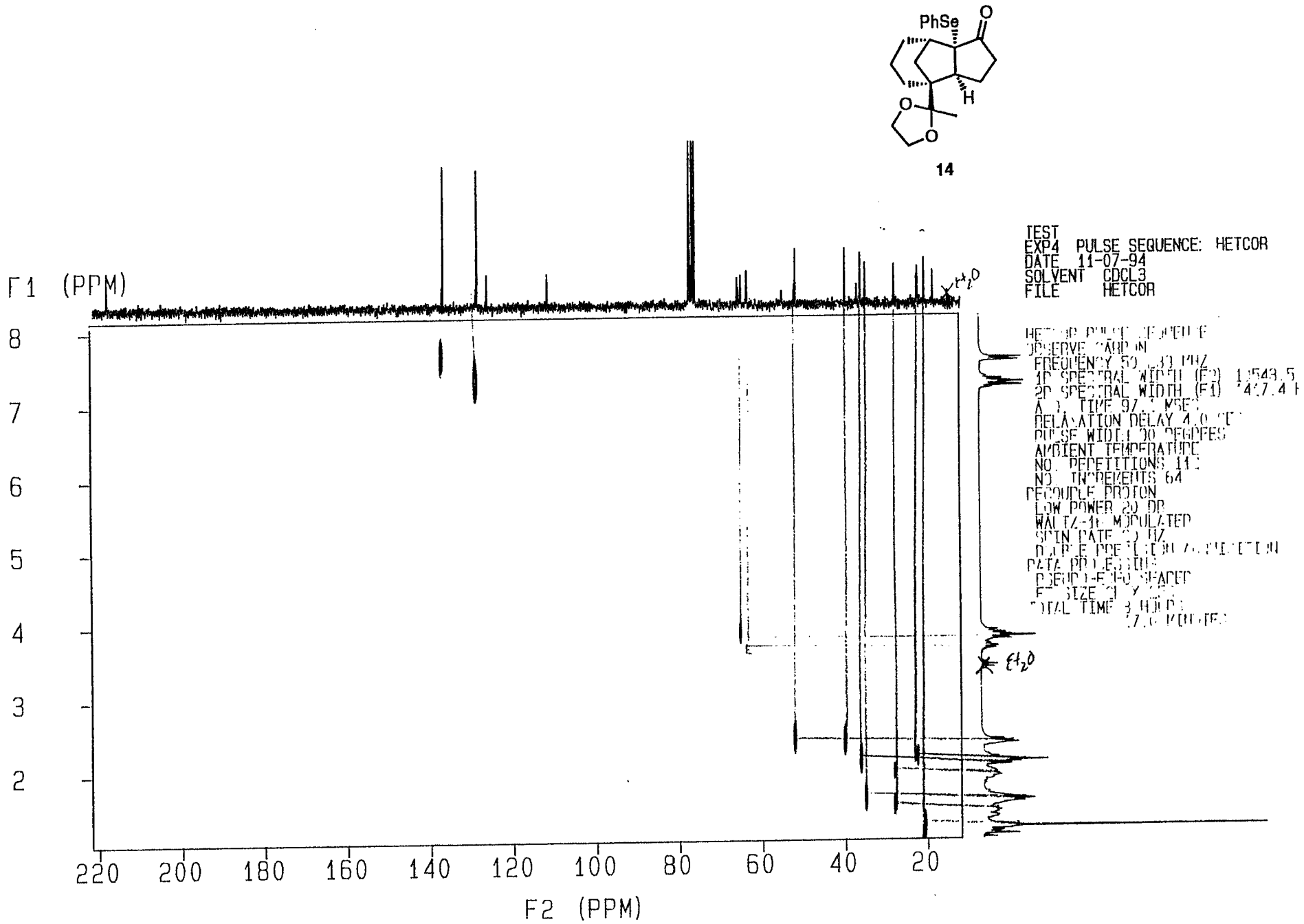
Source : Globar (MIR)
Optical Filter :
Aperture : 3 mm
Beamsplitter:
Sample Channel : Front
Detector : DTGS
Gain Switch Gain / Window : ON / 300
Sample/Reference Gain :Automatic / Automatic
Velocity : 6 ; 10.0 KHz
Resolution : 4.0
Sample Scans : 16

Acquisition Mode : Double Sided,Forward-Backward
Low / High Folding Limit : 0.0 / 7899.5070
Low / High Pass Filter : Open /
HeNe/Raman Laser Wave Number :15799.0140/
Phase Correction Mode : Mertz
Apodization Function : Blackman-Harris 3-Term
Zero Filling Factor : 2
Peak Amplitude / Location : -8107 / 66034
Actual Signal Gain : 2
Experiment File : BRIAN
Instrument type : VECTOR22
Date / Time : 12/ 6/1996 / 8:1:52

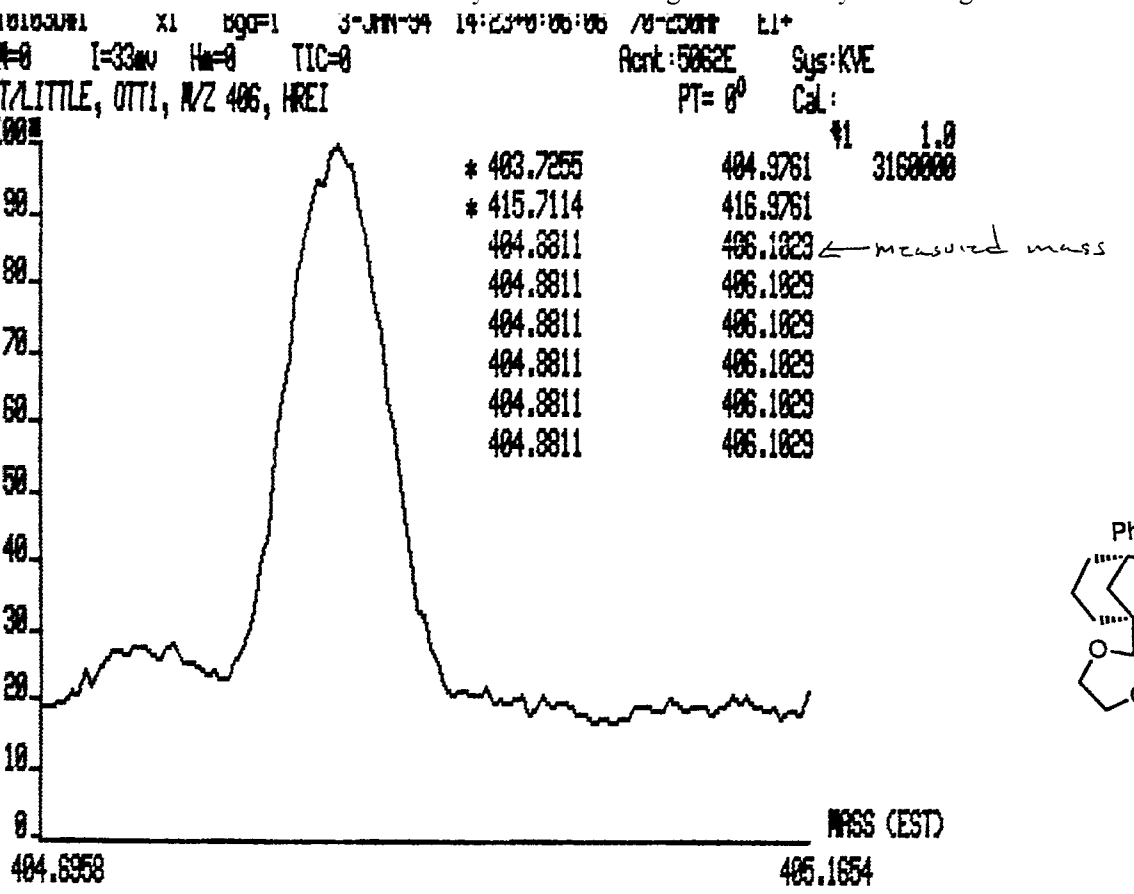



14

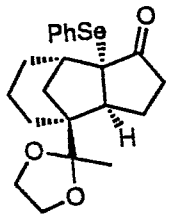





©1997 American Chemical Society Journal Of Organic Chemistry Vol. 1 age 1010 Oct Supplemental Page 39




measured mass




Single Mass Input Page: 1-1

| | C | H | O | DE | PPM | DBE | exact mass |
|---|---|---|---|---|---|---|---|
| | 12 | | | | | | |
| 406 | 25 | 26 | 0 | 1 | -42.0 | 13.0 | 406.1199715 |
| | 24 | 22 | 1 | 1 | 47.6 | 14.0 | 406.0835860 |
| | 16 | 38 | 1 | 2 | -55.2 | -2.0 | 406.1253072 |
| | 15 | 34 | 2 | 2 | 34.4 | -1.0 | 406.0889216 |
| | 21 | 26 | 3 | 1 | -4.5 | 9.0 | 406.1047155 |
| | 27 | 18 | 4 | 0 | -43.4 | 19.0 | 406.1205093 |
| | 18 | 30 | 5 | 1 | -56.5 | 4.0 | 406.1258449 |

theoretical mass Pt

?=help RETURN=next page !S=skip !Q=return to parameters !X=exit !E=editor