JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(15), 5173-5178, DOI:10.1021/jo980429x

Terms & Conditions

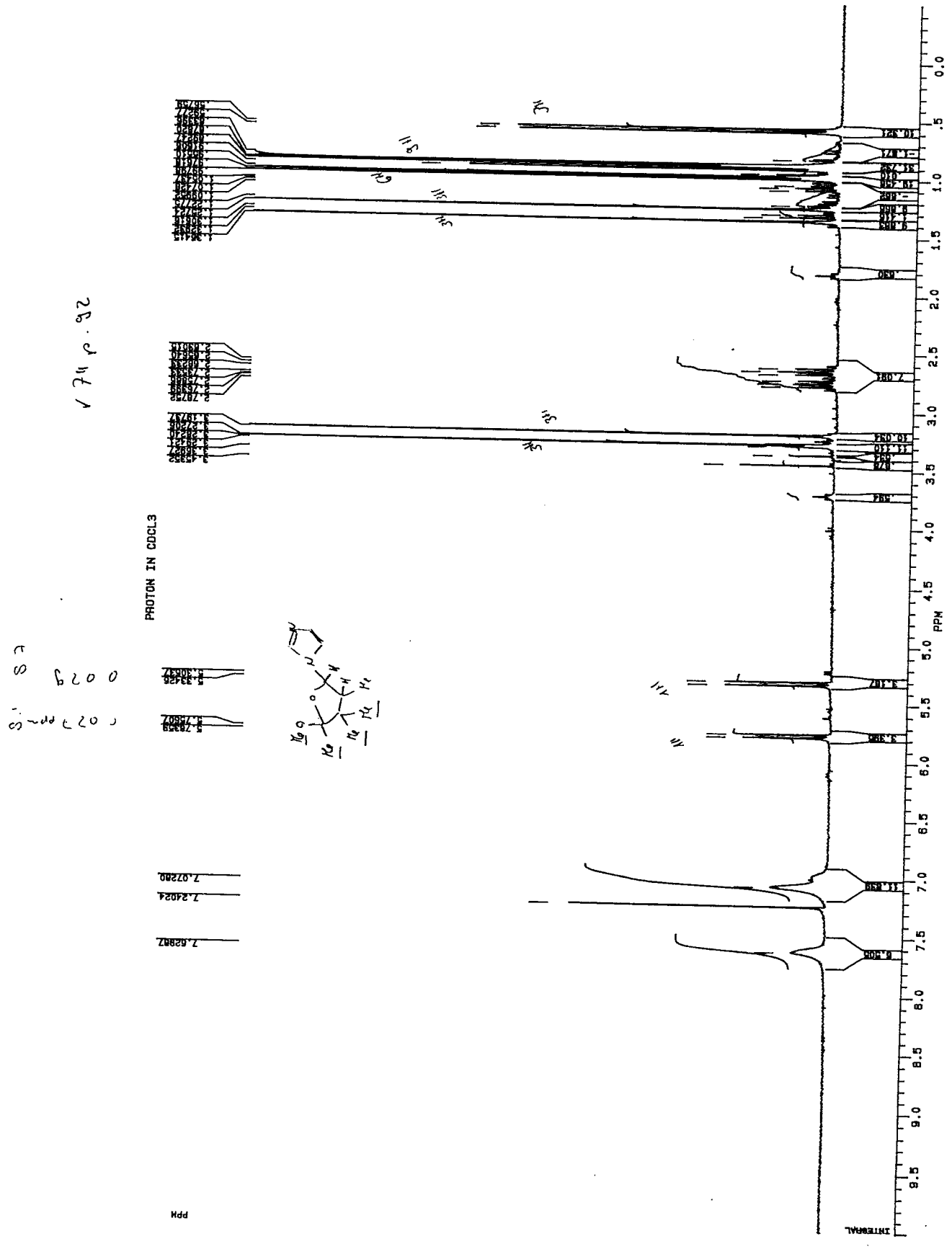
Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not 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

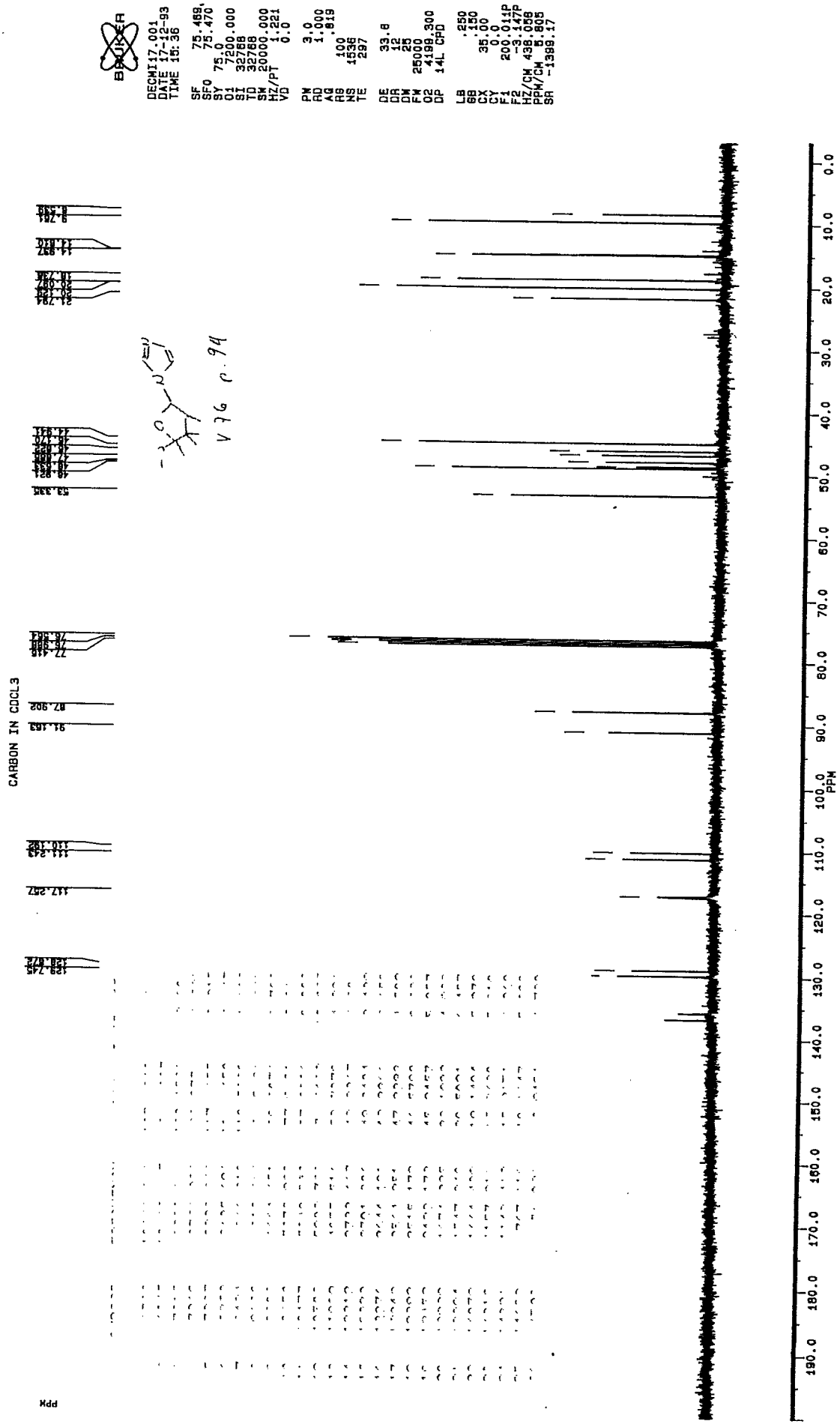
MOST TRUSTED. MOST CITED. MOST READ.

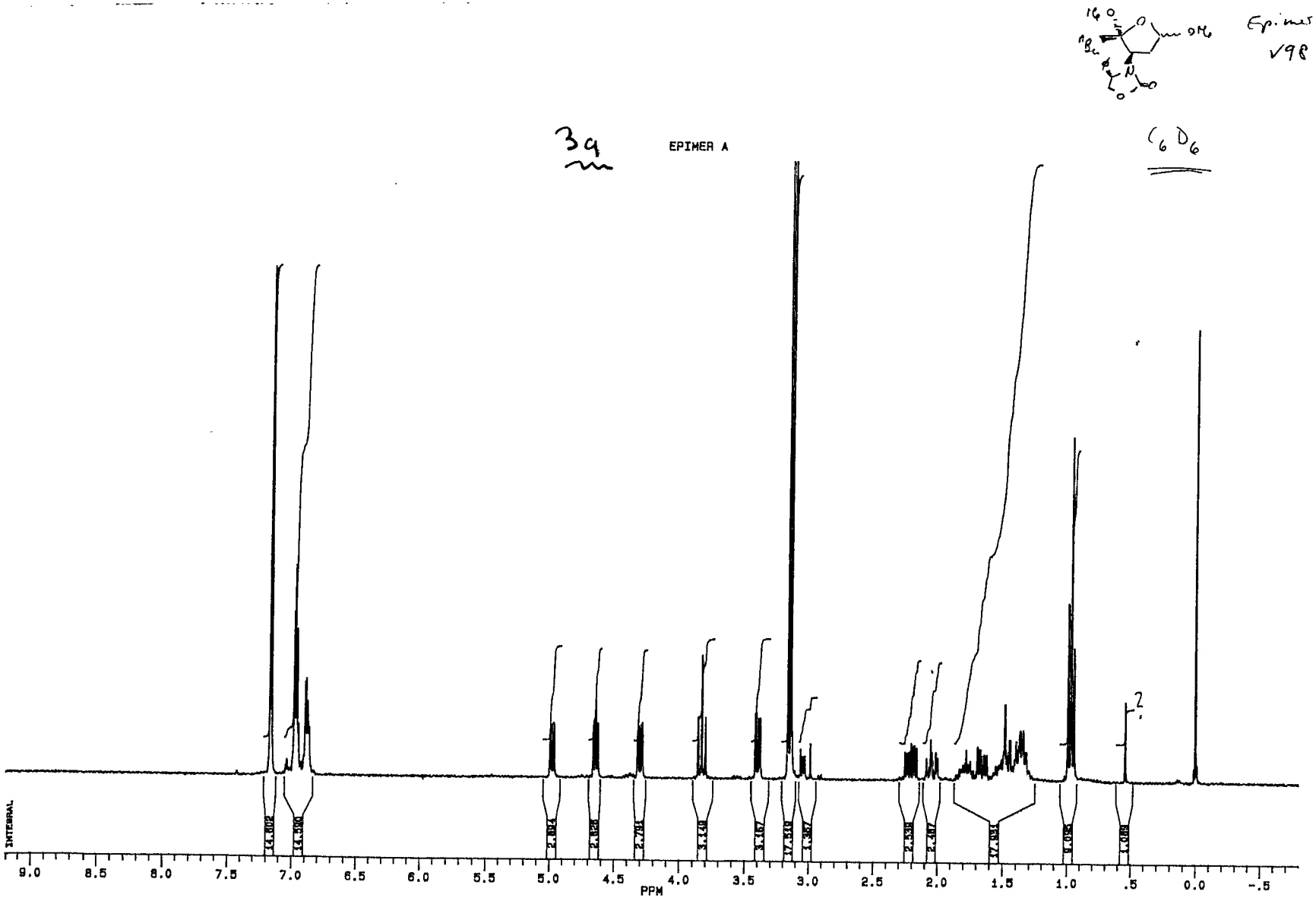
Copyright © 1998 American Chemical Society


PROTON IN CDCL3
PPM
INTEGRAL

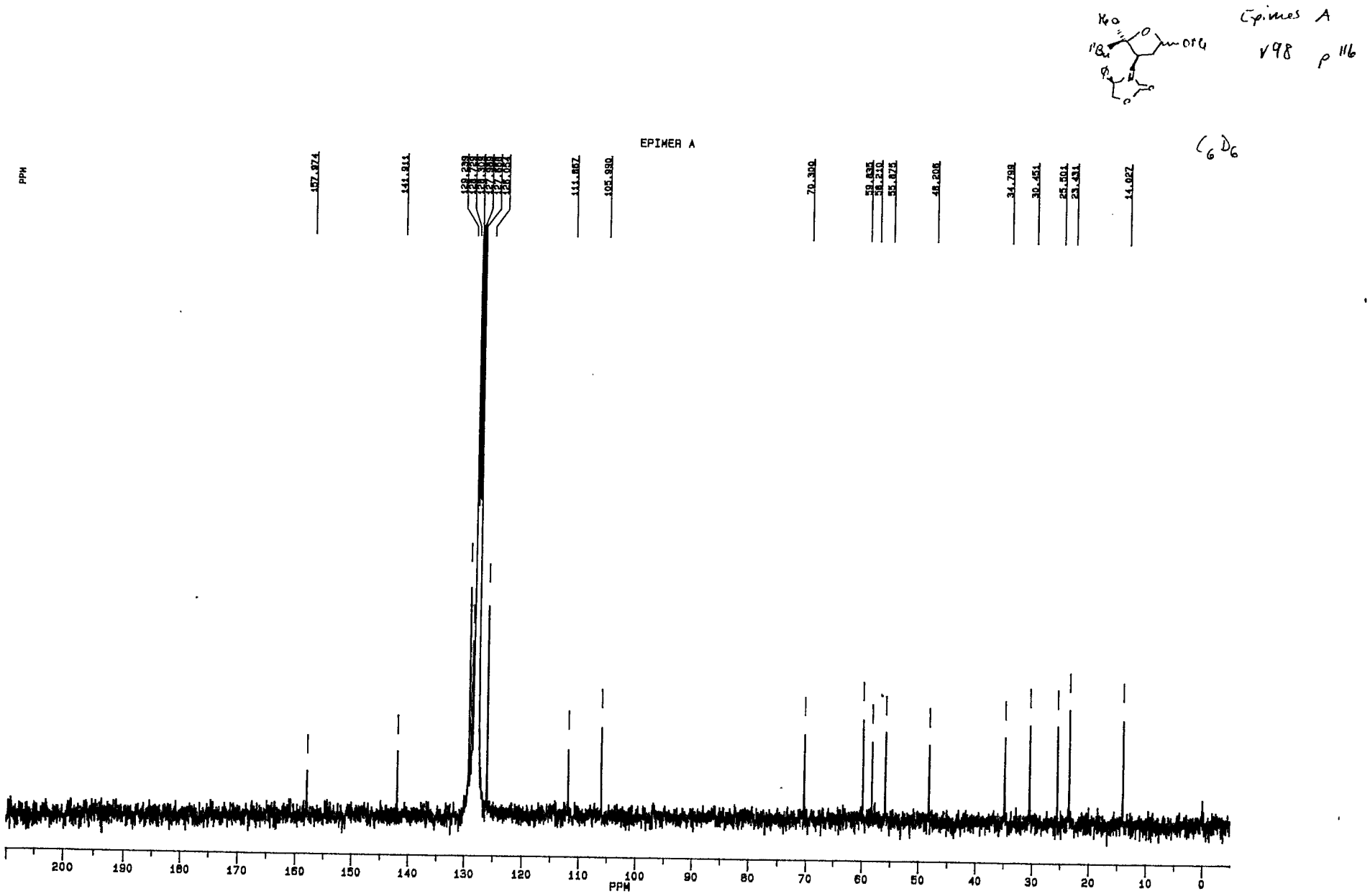
©1998 American Chemical Society Journal Of Organic Chemistry V63 Page 5173 Uniform Supplemental Page 2

POOR QUALITY ORIGINAL





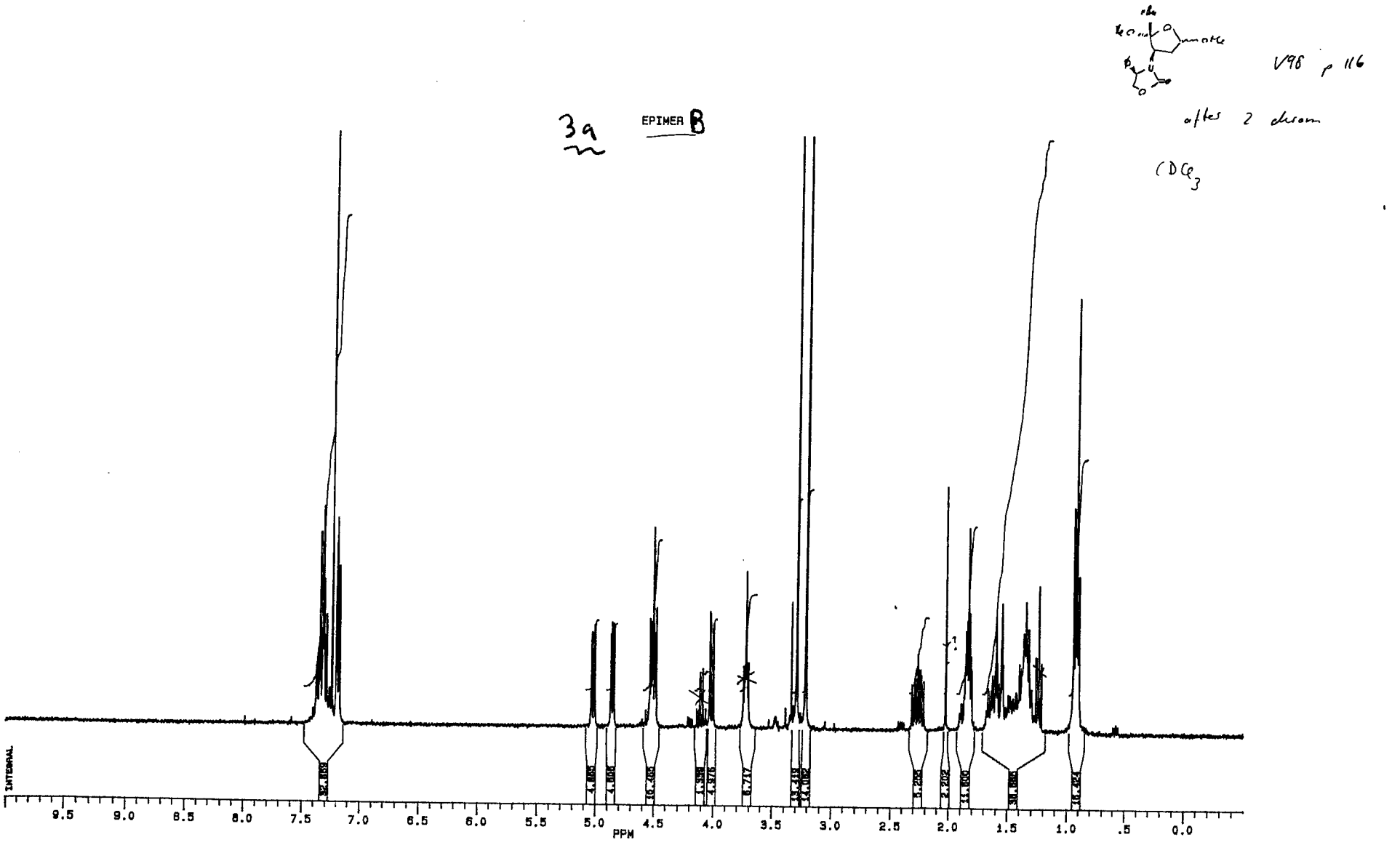
©1998 American Chemical Society Journal Of Organic Chemistry V63 Page 5173 Umbricht Supplemental Page 3



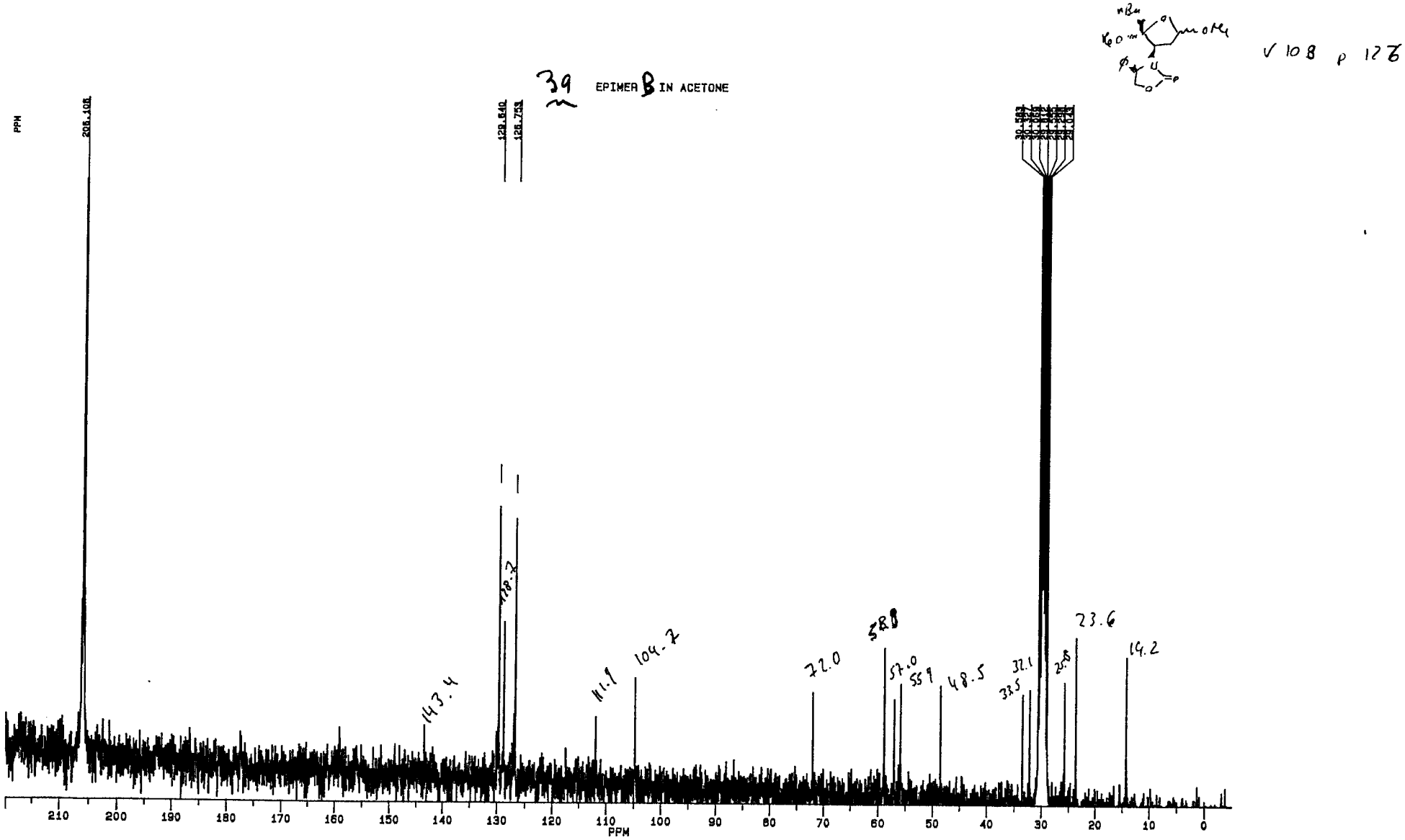
Epimer A
V98 p 116

C_6D_6

© 1996 American Chemical Society Journal Of Organic Chemistry V65 Page 5175 Supplemental Page 4



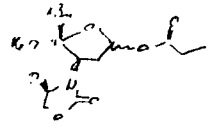
©1998 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page 5175 Chemical Supplemental Page 5



V 108 p 126

©1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5173 Supplemental Page 6

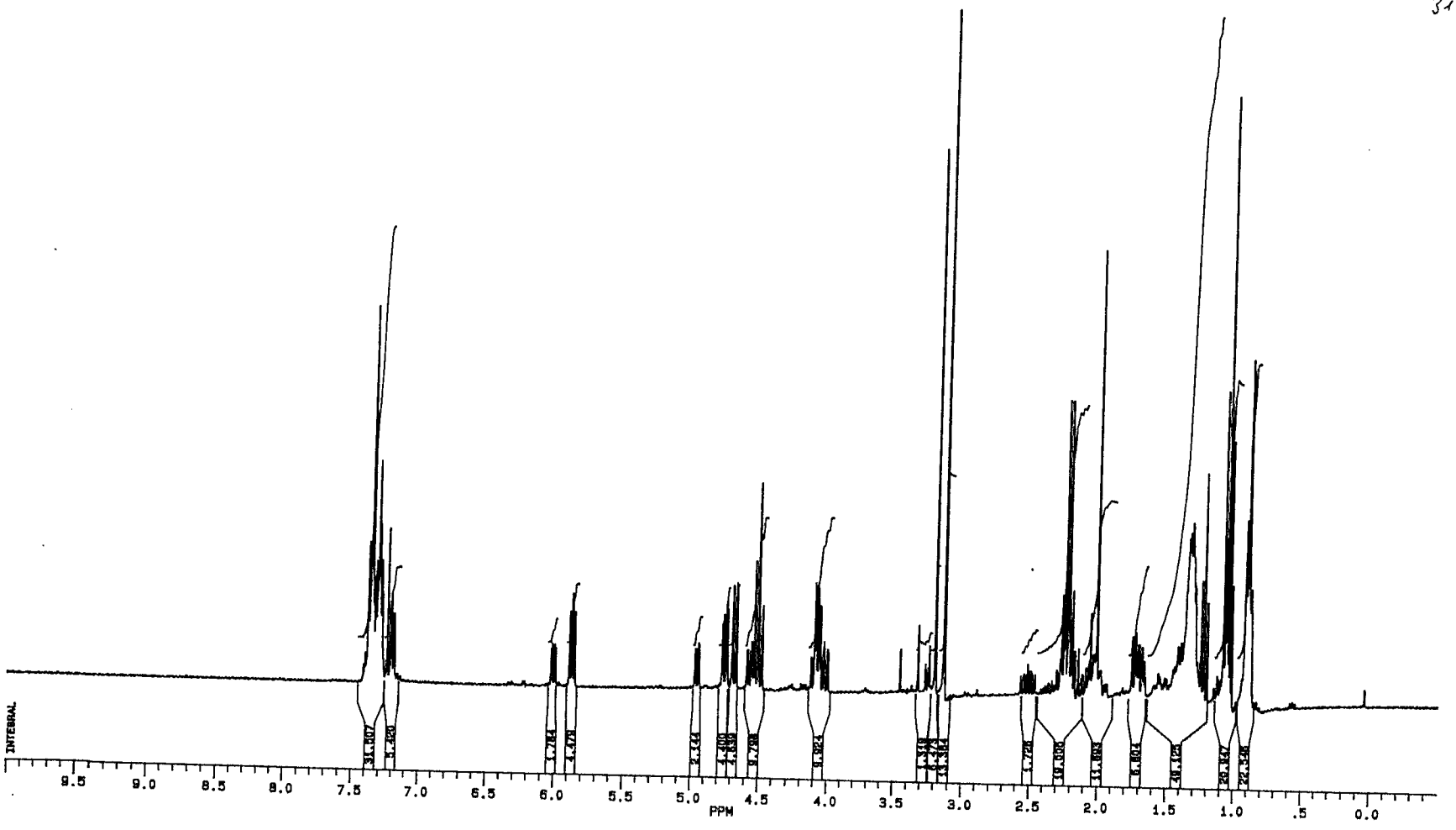
3b



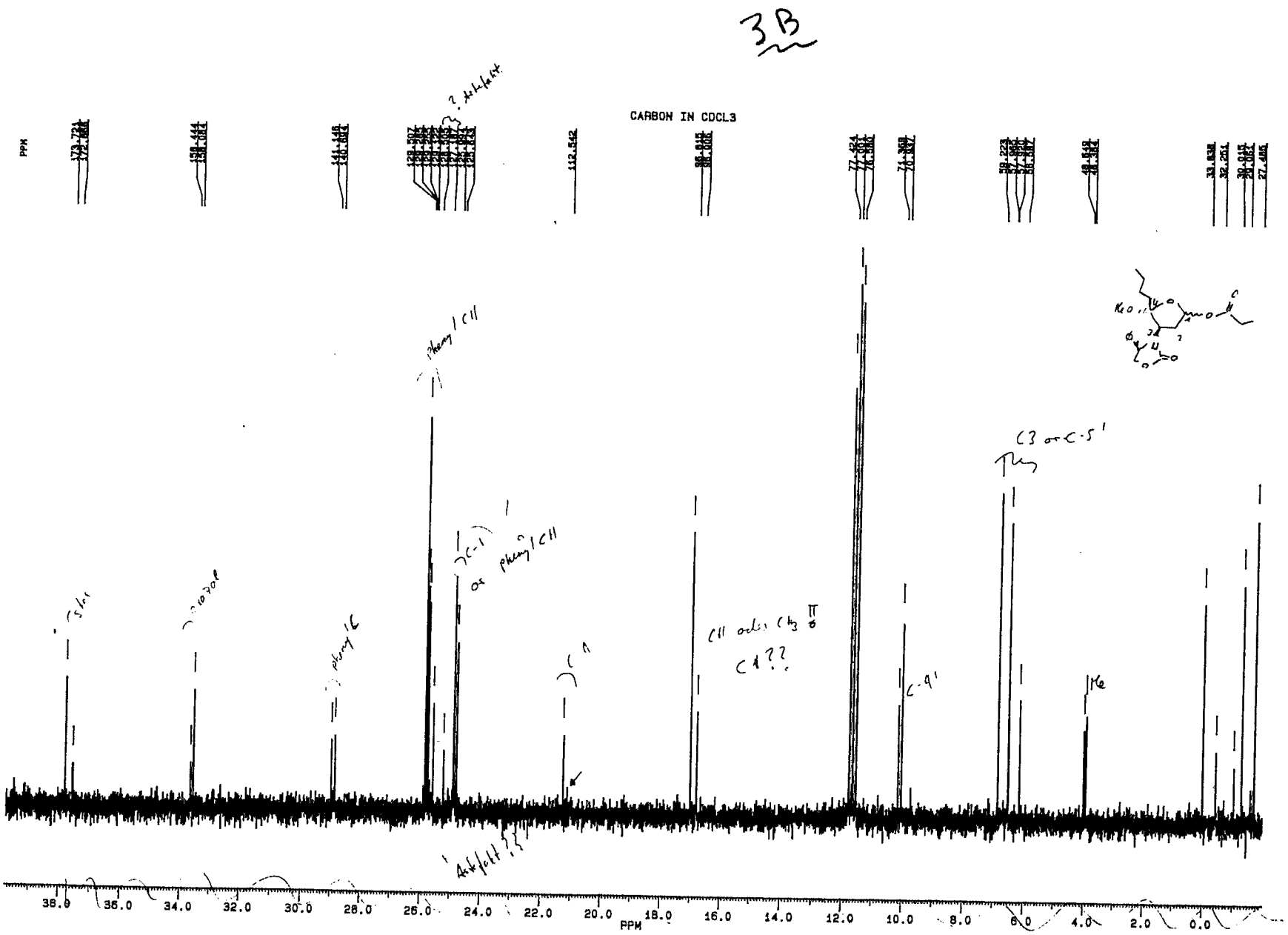
V136 p. 155

31-d

INSERTION PROPIONIC ACID CHROM.



© 1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 7

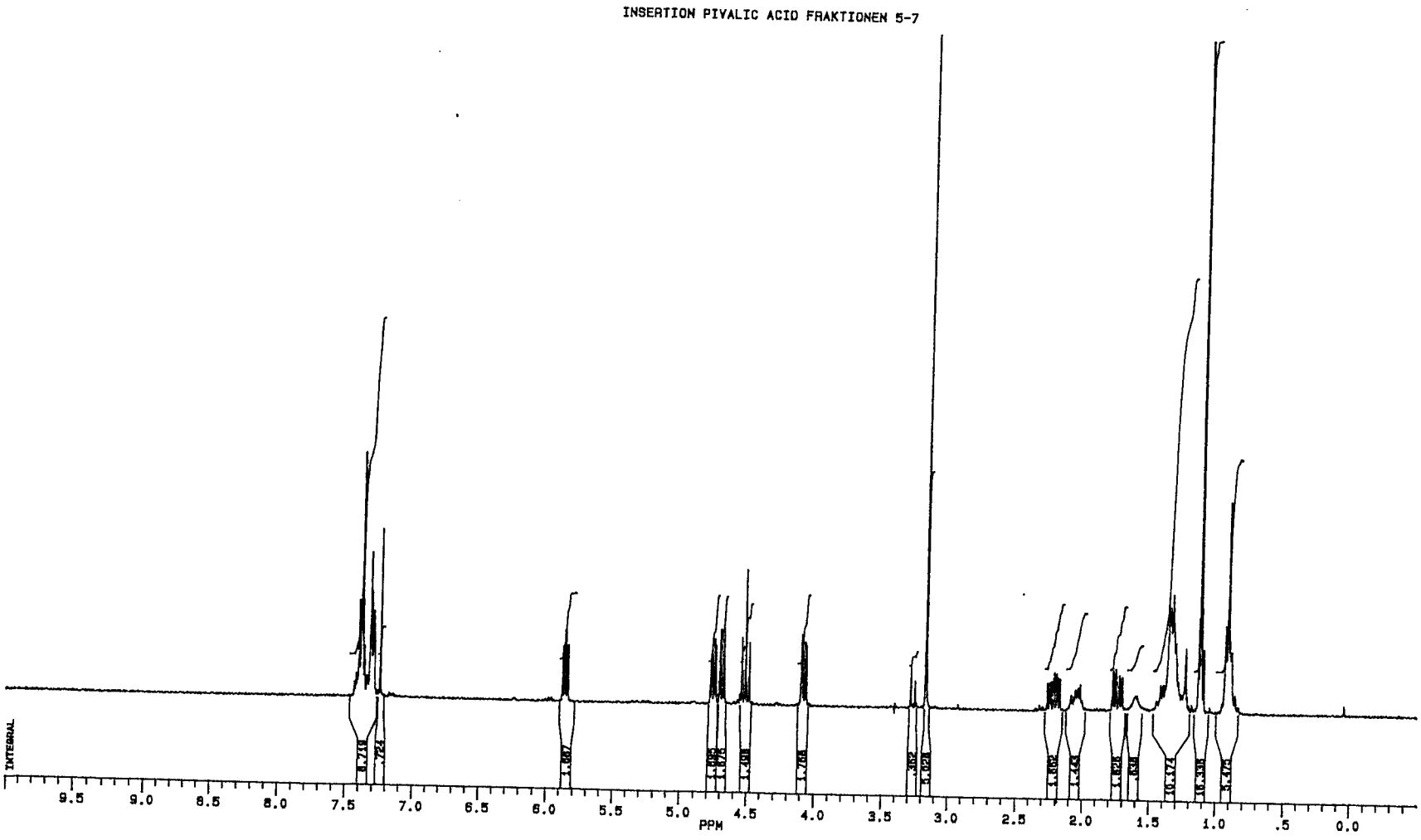


©1996 American Chemical Society Journal Of Organic Chemistry V03 Page 3175 Supplemental Page 6

©1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5173 Supplemental Page 9

3c

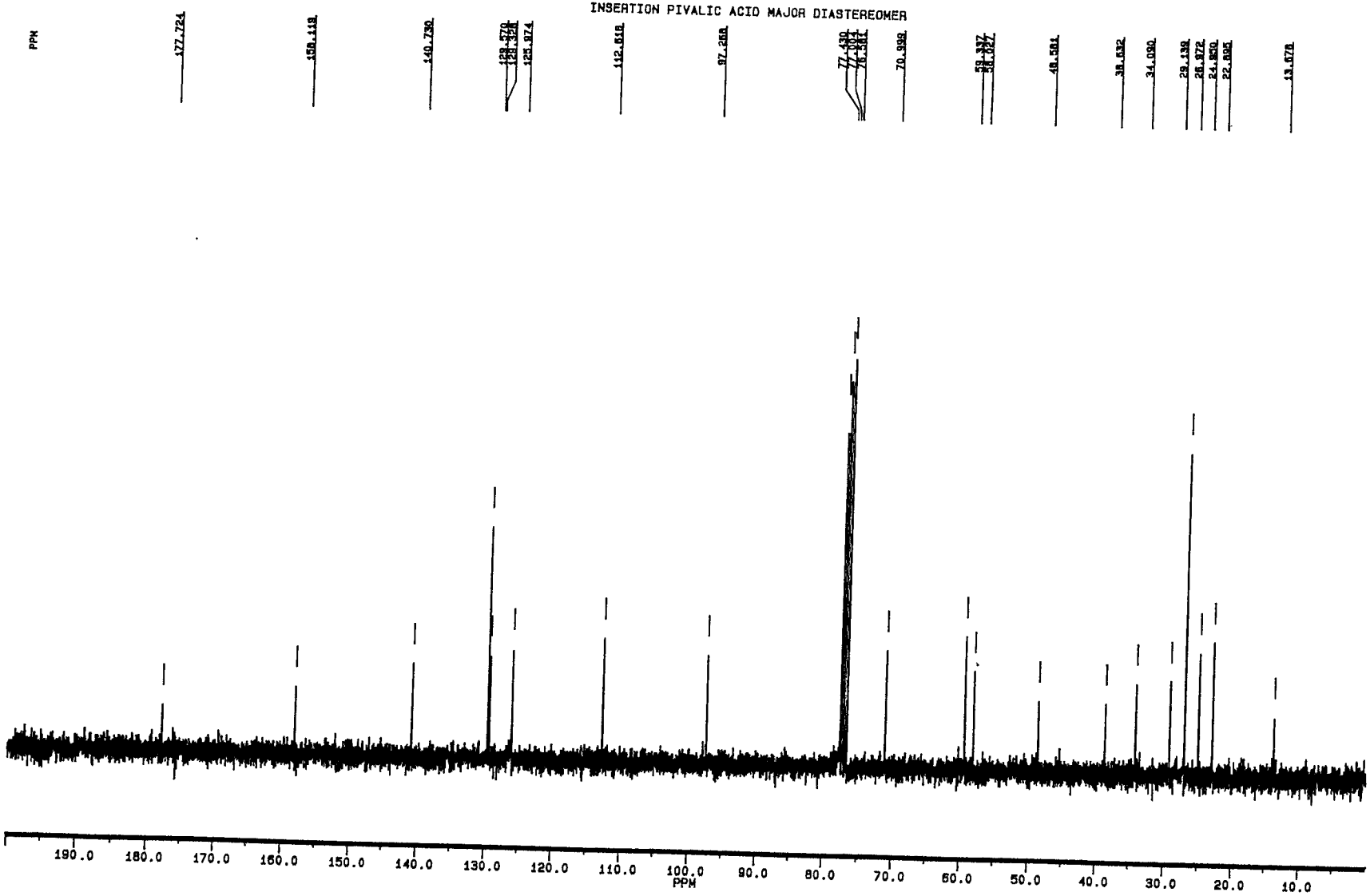
V.37 p. 156
Spinus A



3c

V137 p 156
Spinal A

INSERTION PIVALIC ACID MAJOR DIASTEREOMER

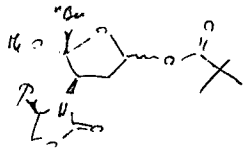


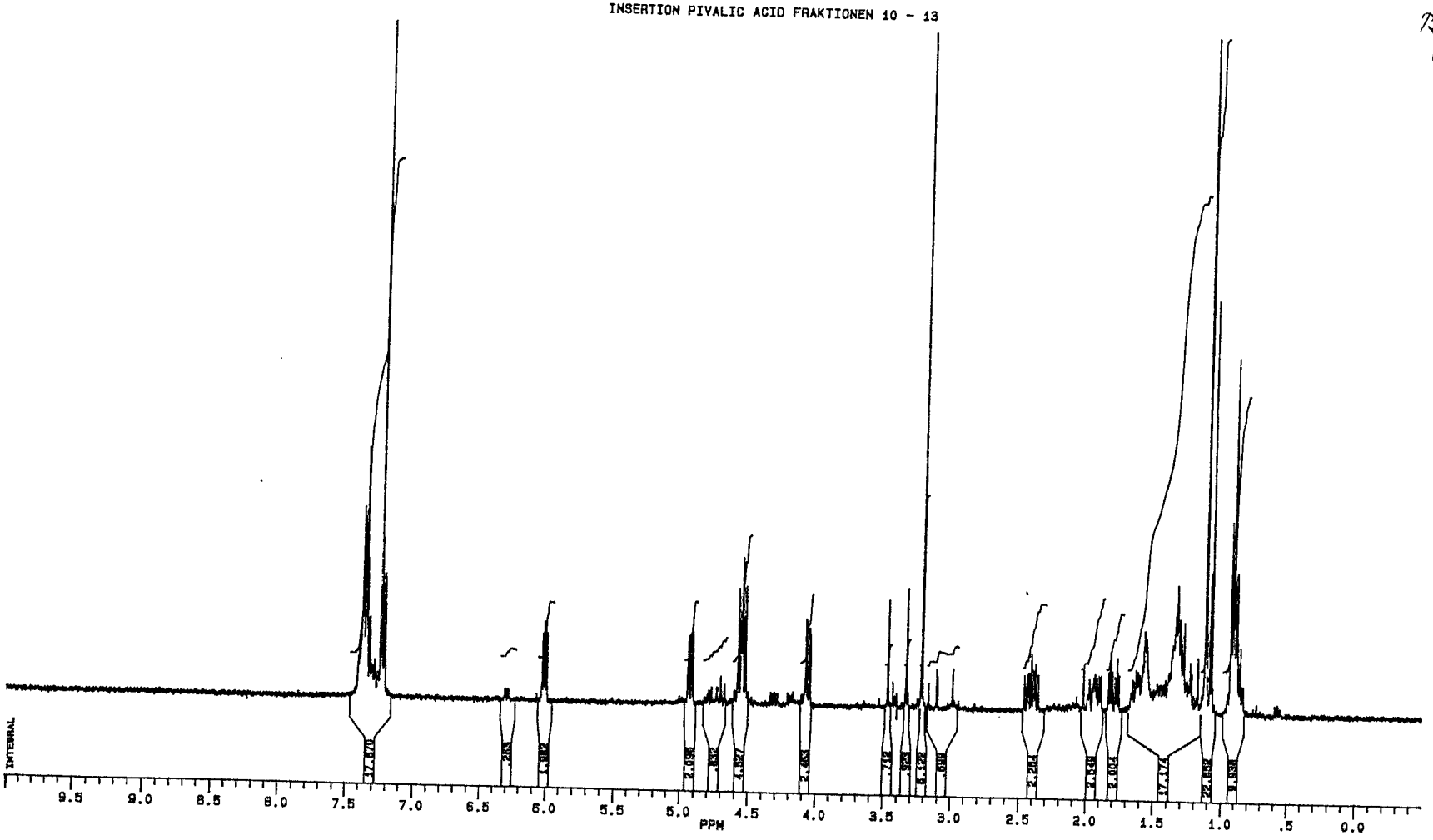
©1998 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page 0175 Online Supplemental Page 10

3c

V 137 p 156

Epimer B

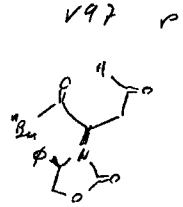


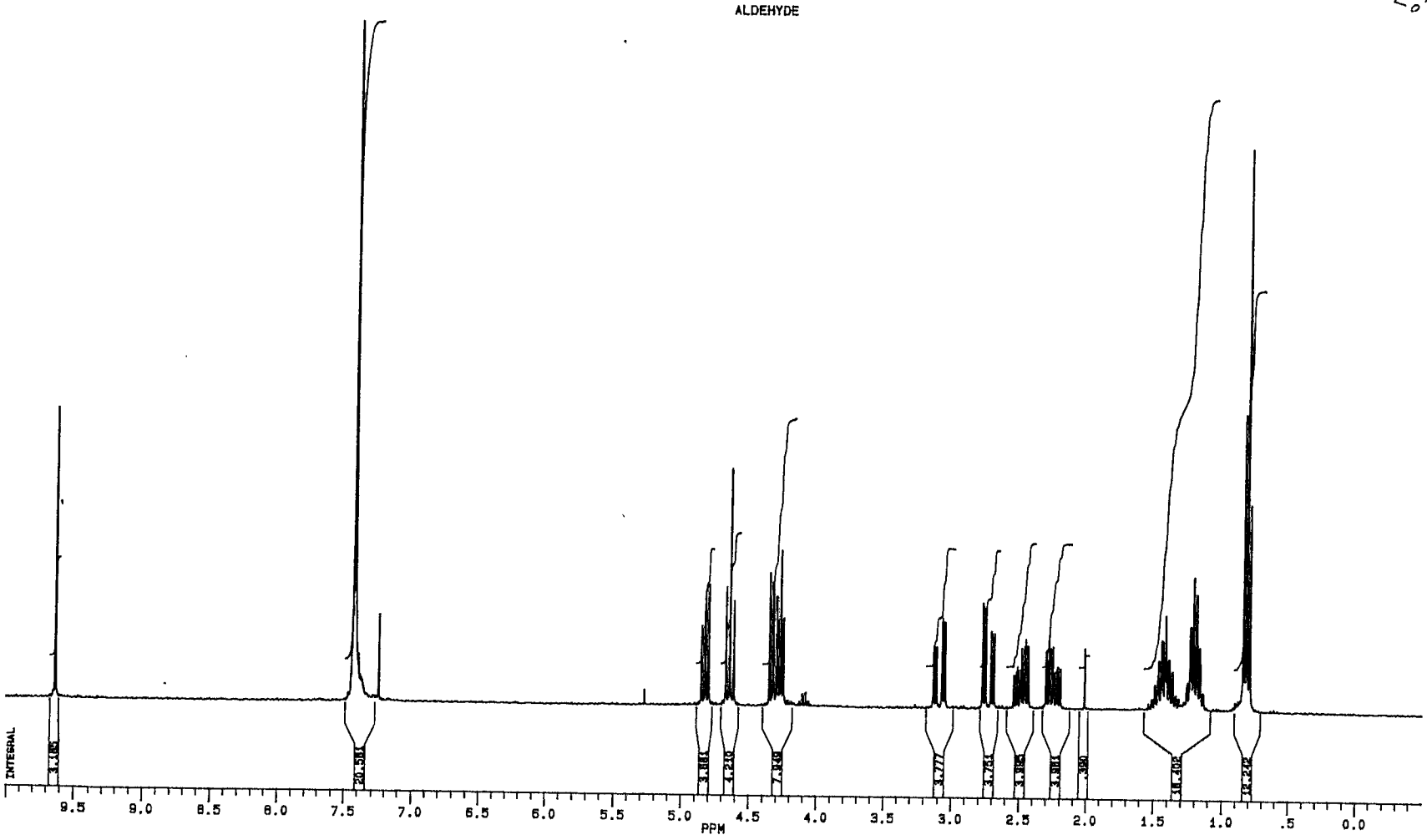


© 1996 American Chemical Society Journal Of Organic Chemistry V03 Page 3175 Supplemental Page 11

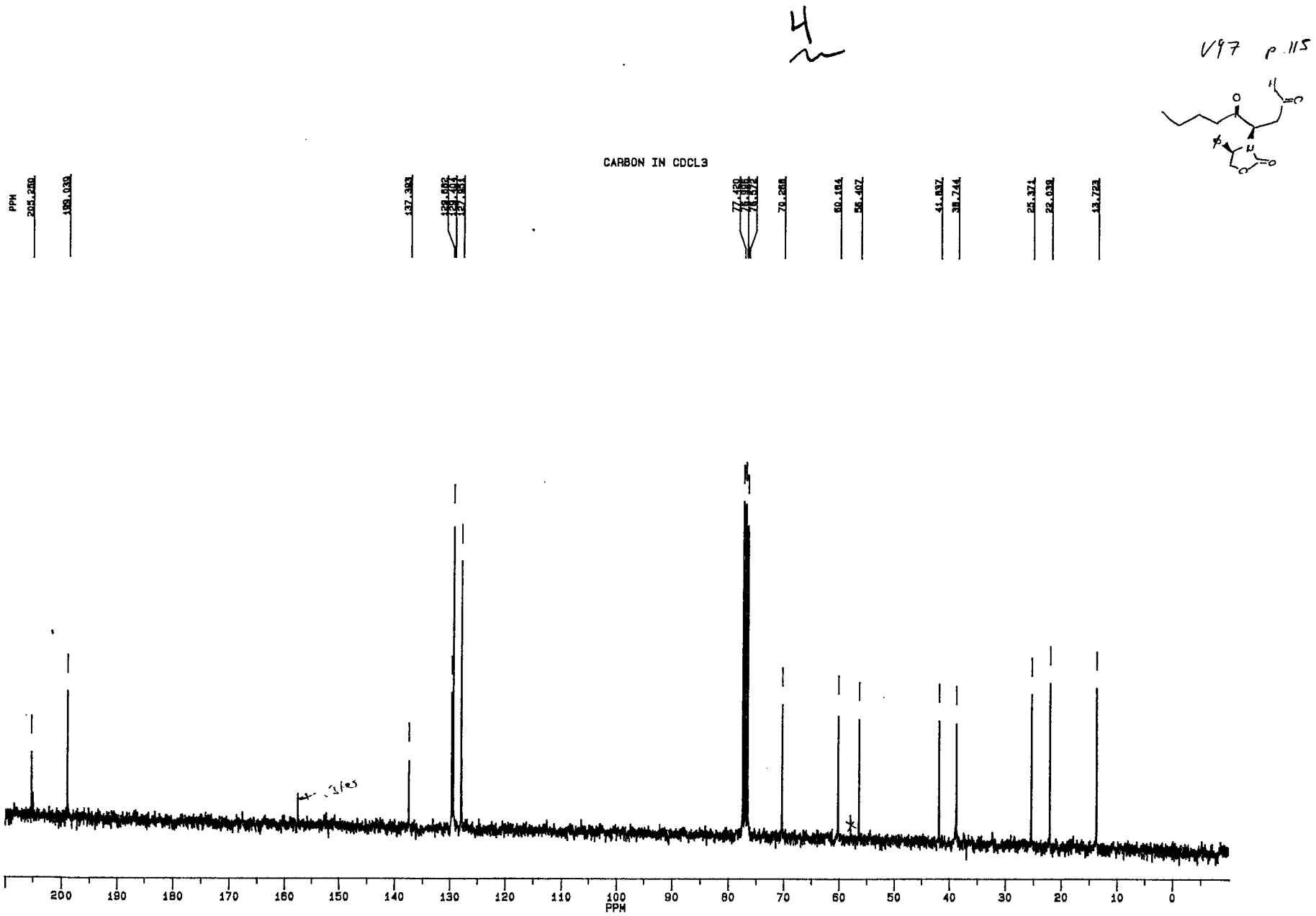
4

V97 p 115





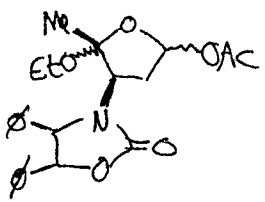
©1998 American Chemical Society Journal Of Organic Chemistry V63 Page S175 Chiron Supplement Page 12



©1998 American Chemical Society Journal of Organic Chemistry Vol. 1 Page S175 Supplement Page 15

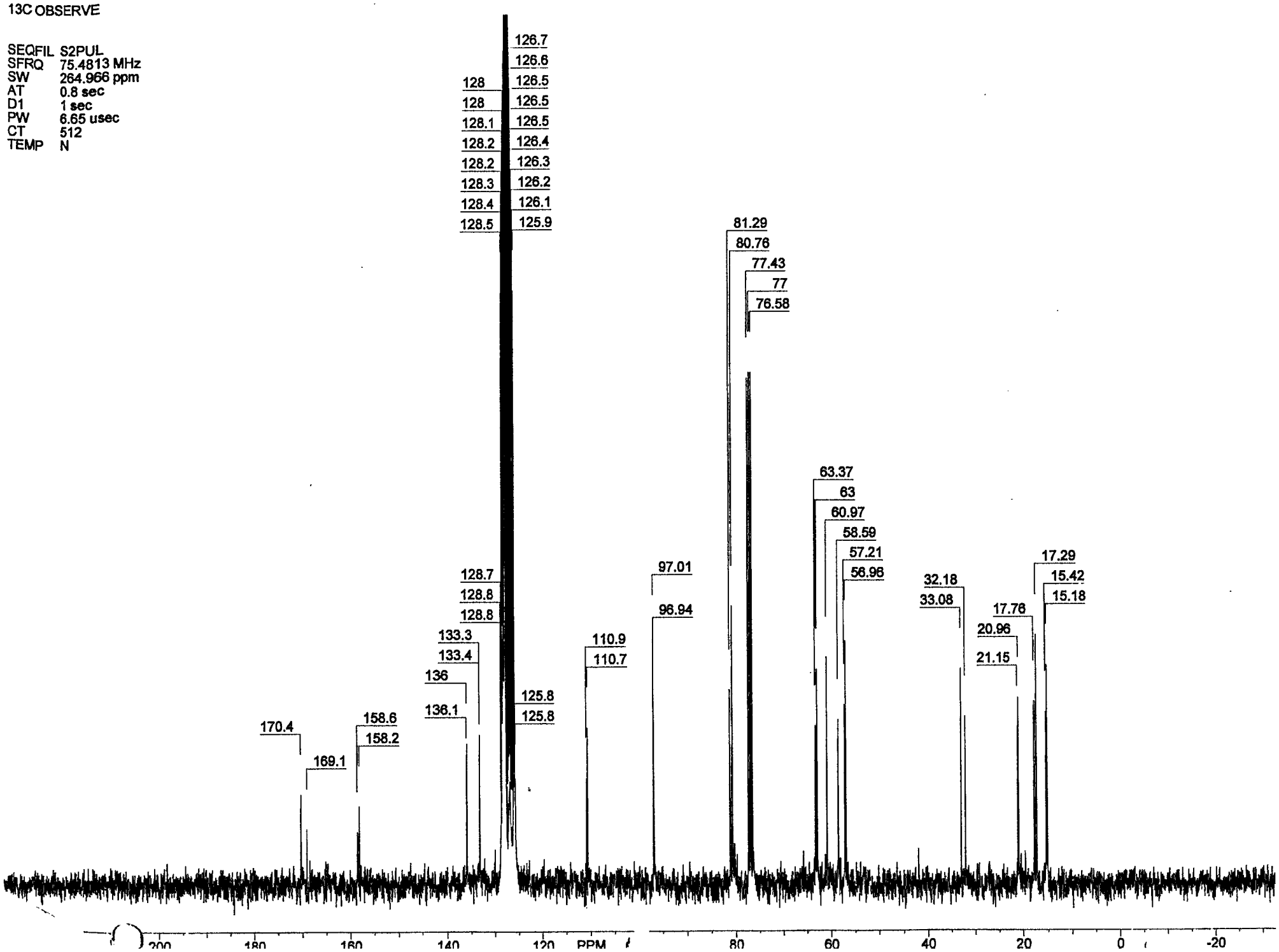
(6b)

Filename: MDH1388B.H01



6b

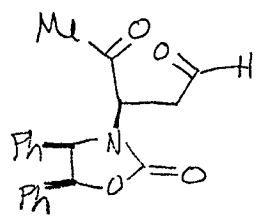
©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 14



©1998 American Chemical Society Journal Of Organic Chemistry Vol. Page S173 Electronic Supplementary Page 15

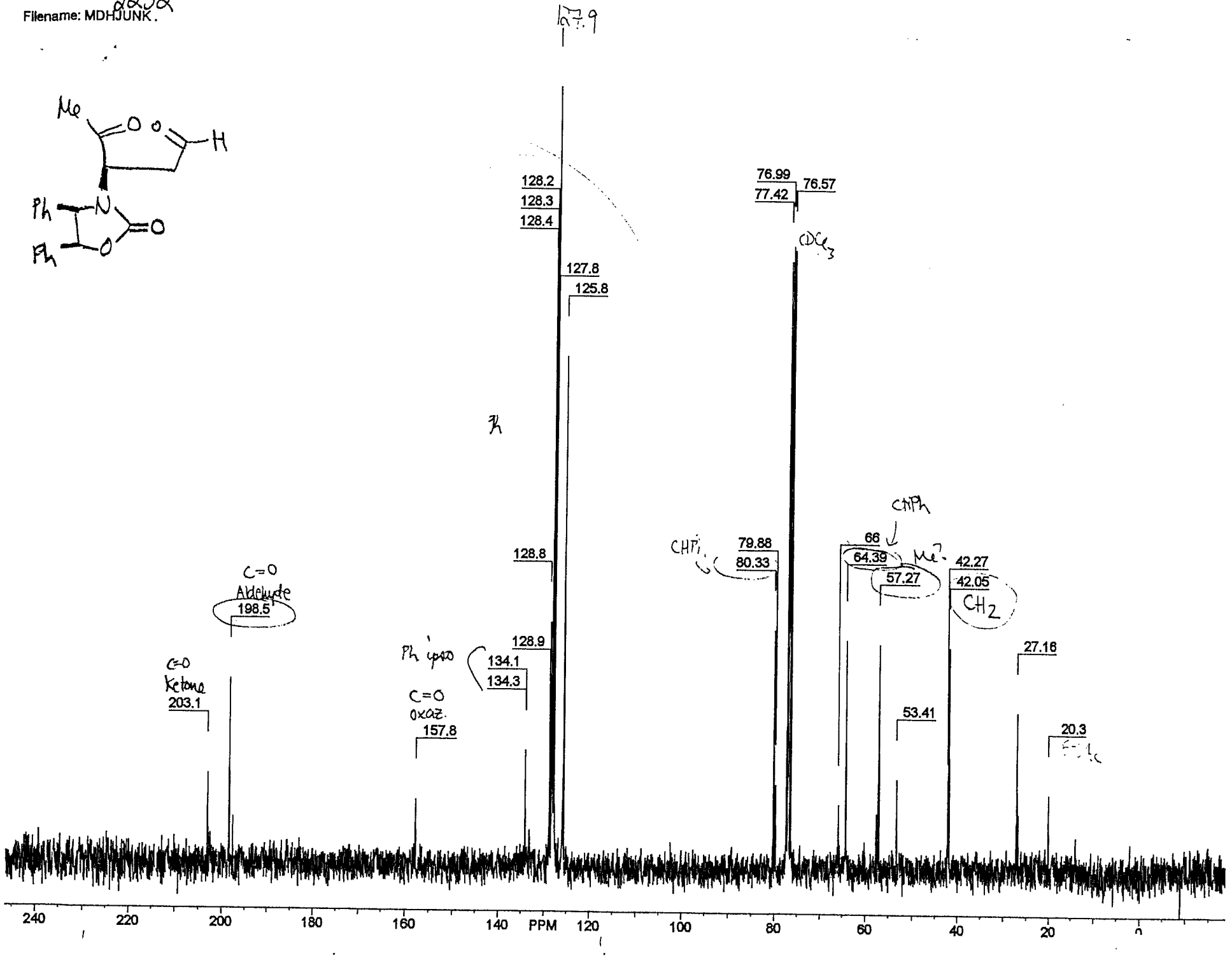
Numbering compound 2232 (Me analog of (4) derivatized form (5)

Filename: MDHOPEN .H01



©2020 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page S175 Omitted Supplemental Page 10

Filename: MDHJUNK.2232

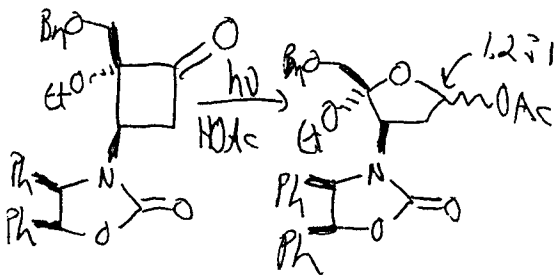


© 1996 American Chemical Society Journal Of Organic Chemistry V03 Page S175 Supplemental Page 17

8)

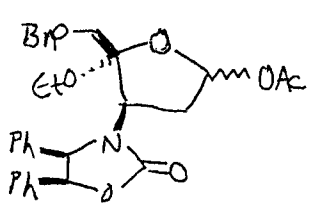
8

Filename: MDH1532A.H01



©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 16

Filename: MDH1532E.C13



128.1
128.2
128.3
128.4
128.5
128.6
128.7
136.2
110.1
96.84
96.26
80.84
80.45
74.59
74.49
69.17
68.43
63.4
63.34
57.49
57.39
57.32
56.89
32.1
33.99
20.99
21.11
15.54
15.33

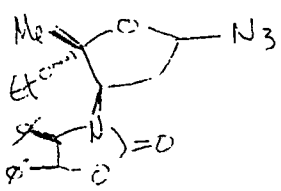
220 200 180 160 140 120 100 PPM 80 60 40 20 0

©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 19

Filename: MDH1456B.H01

9b

Epimer A

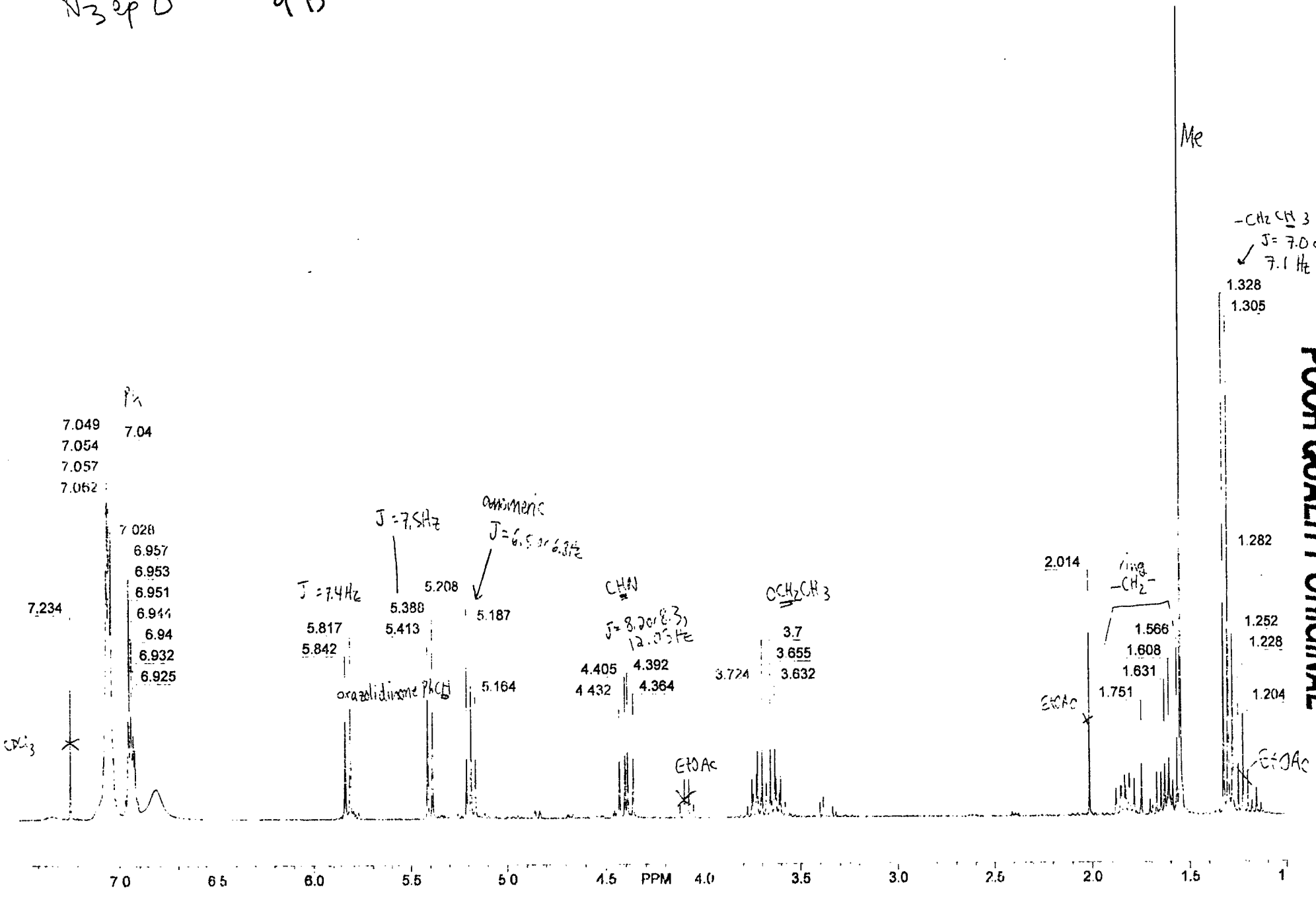


POOR QUALITY ORIGINAL

©1998 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page S175 Chiroptical Supplemental Page 20

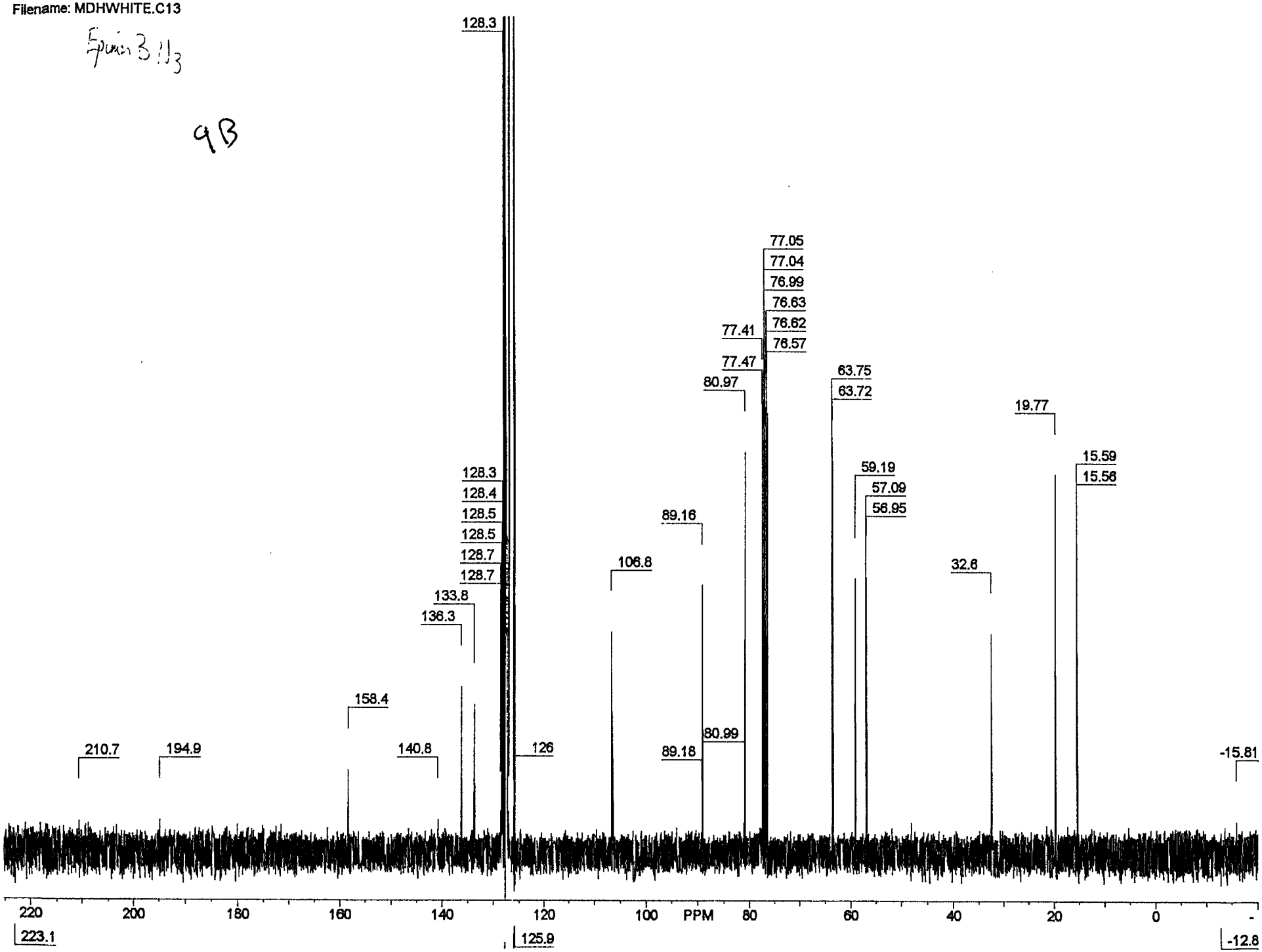
Filename: MDH1456D.H01

N3 epB 9B



POOR QUALITY ORIGINAL

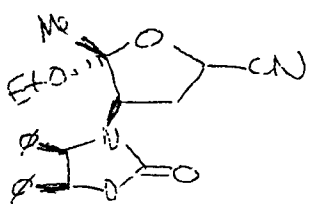
©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 3175 Chemical Supplemental Page 21



© 1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 3175 Supplemental Page 22

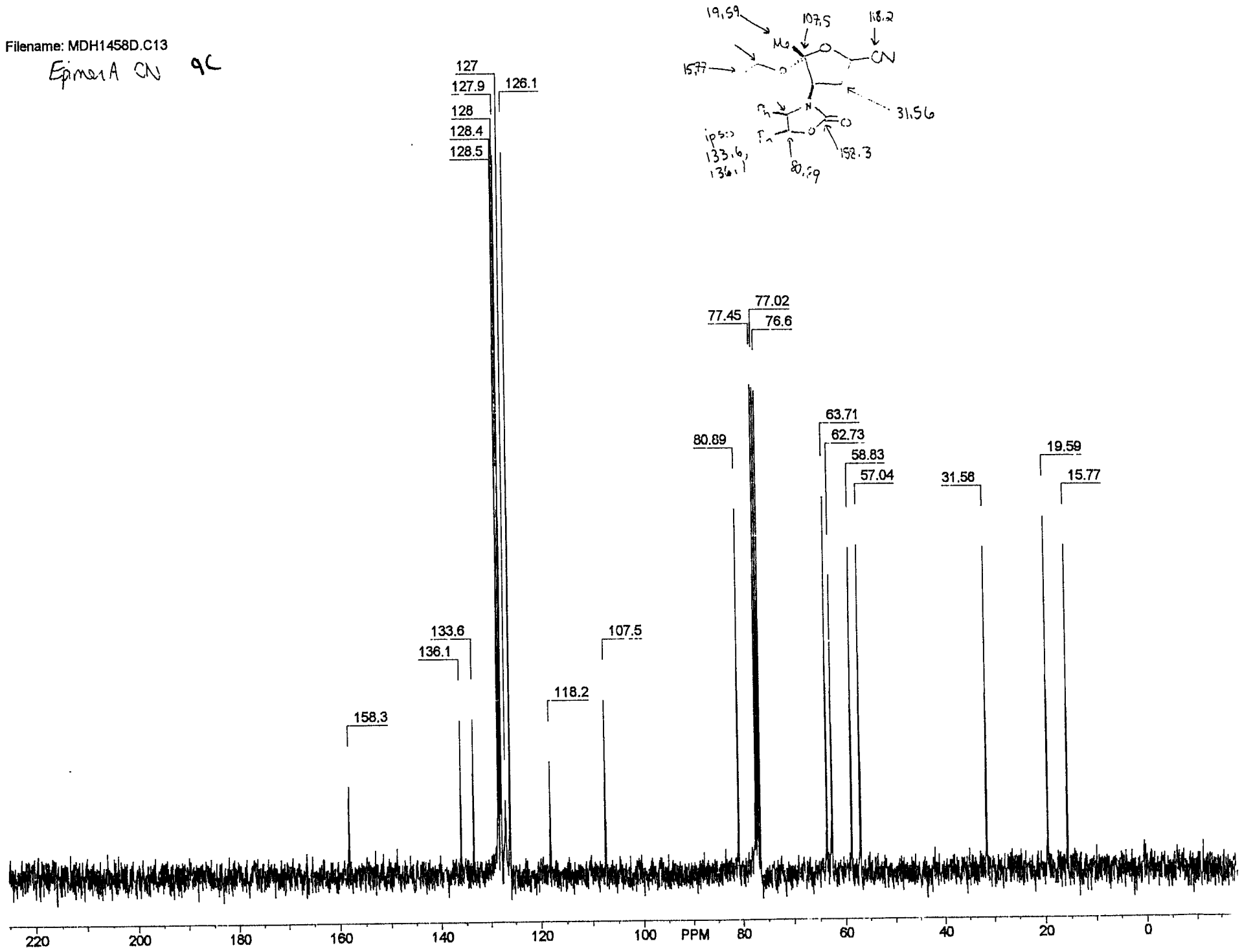
Filename: MDH1458B.H01

- Epimer A



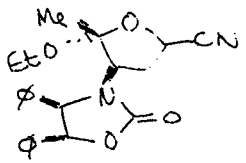
9C

©2020 American Chemical Society Journal Of Organic Chemistry



©1998 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page S175 Supplemental Page 24

Filename: MDH1458C.H01



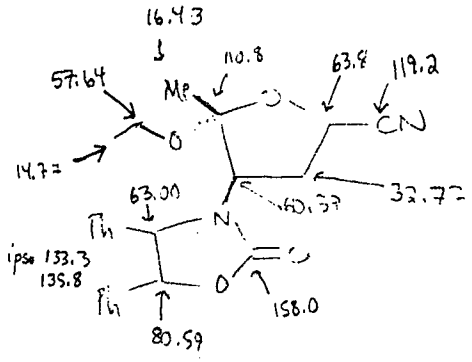
Epimer B

9C

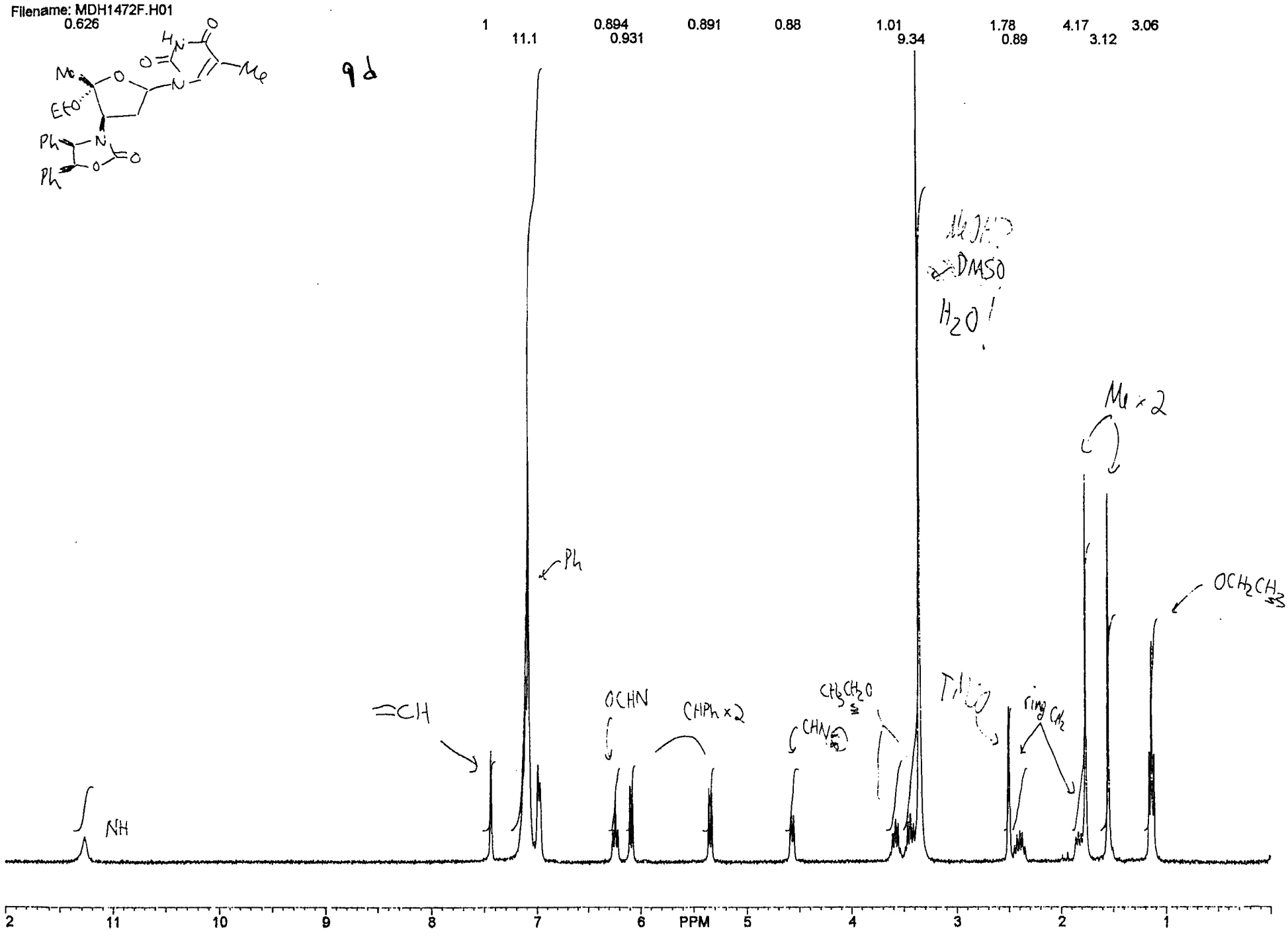
©1998 American Chemical Society Journal of Organic Chemistry Vol. 1 Page S175 Electronic Supplementary Page 25

Filename: MDH1458E.C13

Epimer B CN QC

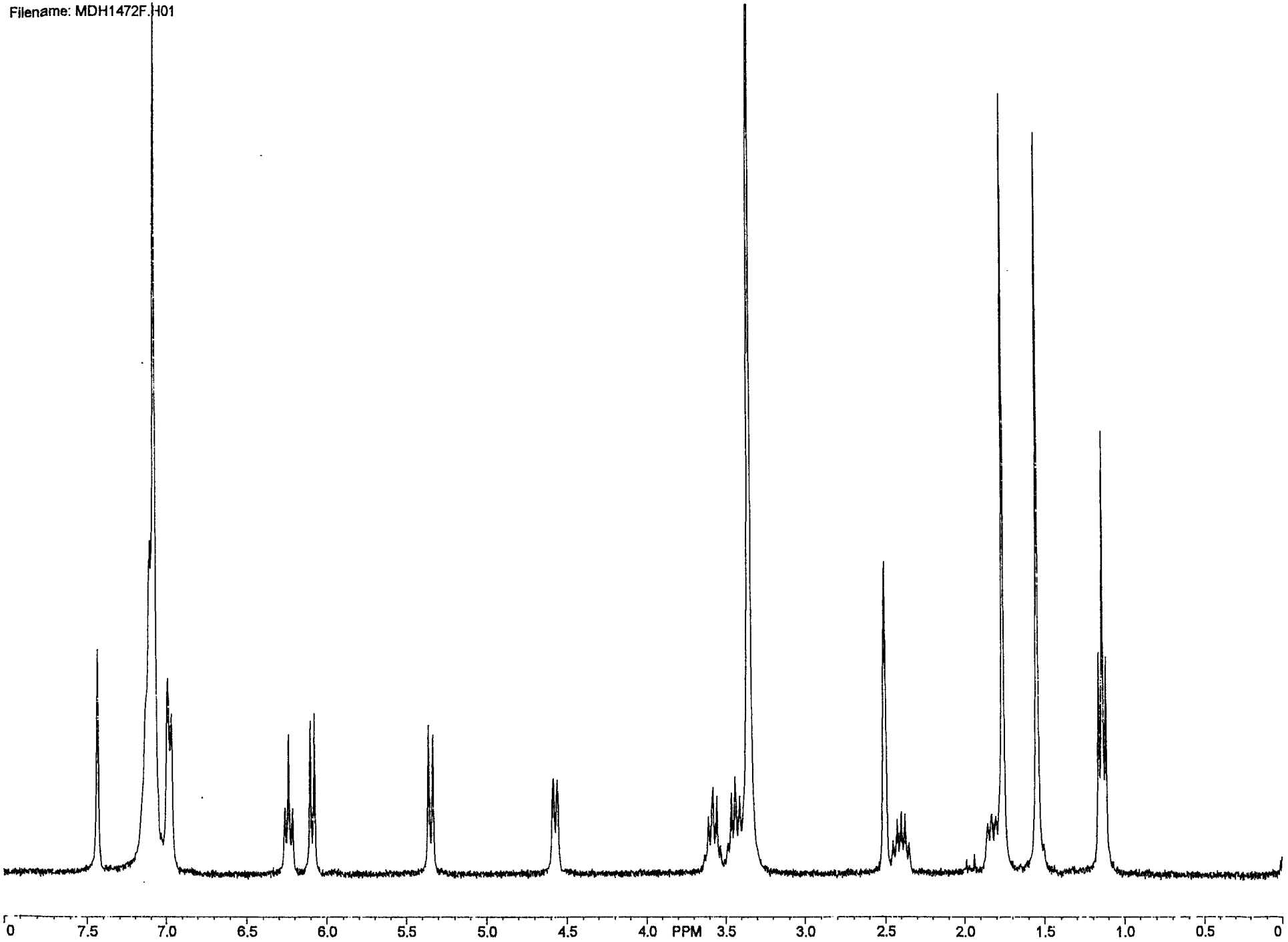


©2008 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 20

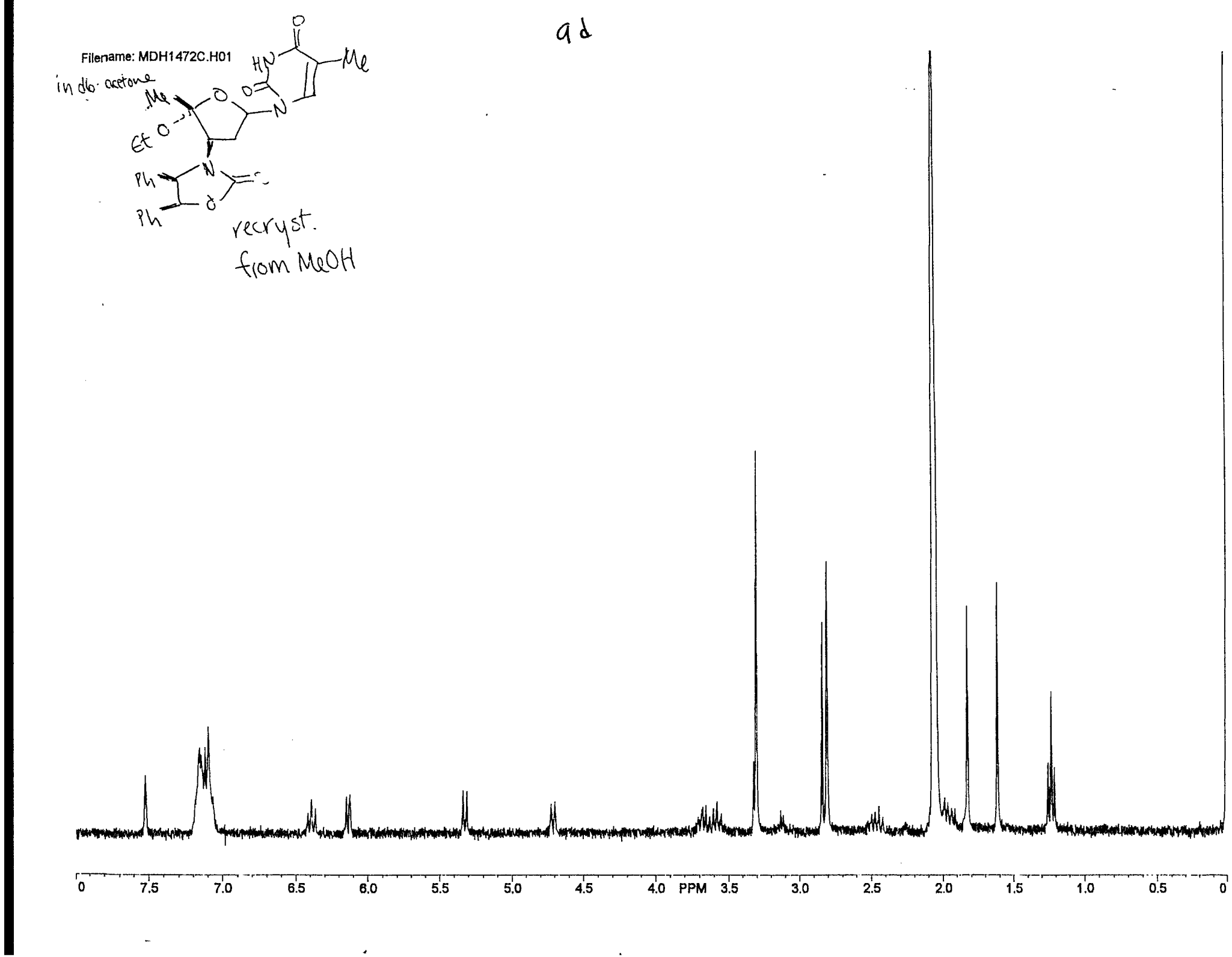


© 1996 American Chemical Society Journal Of Organic Chemistry Vol. Page S175 Supplemental Page 27

9d

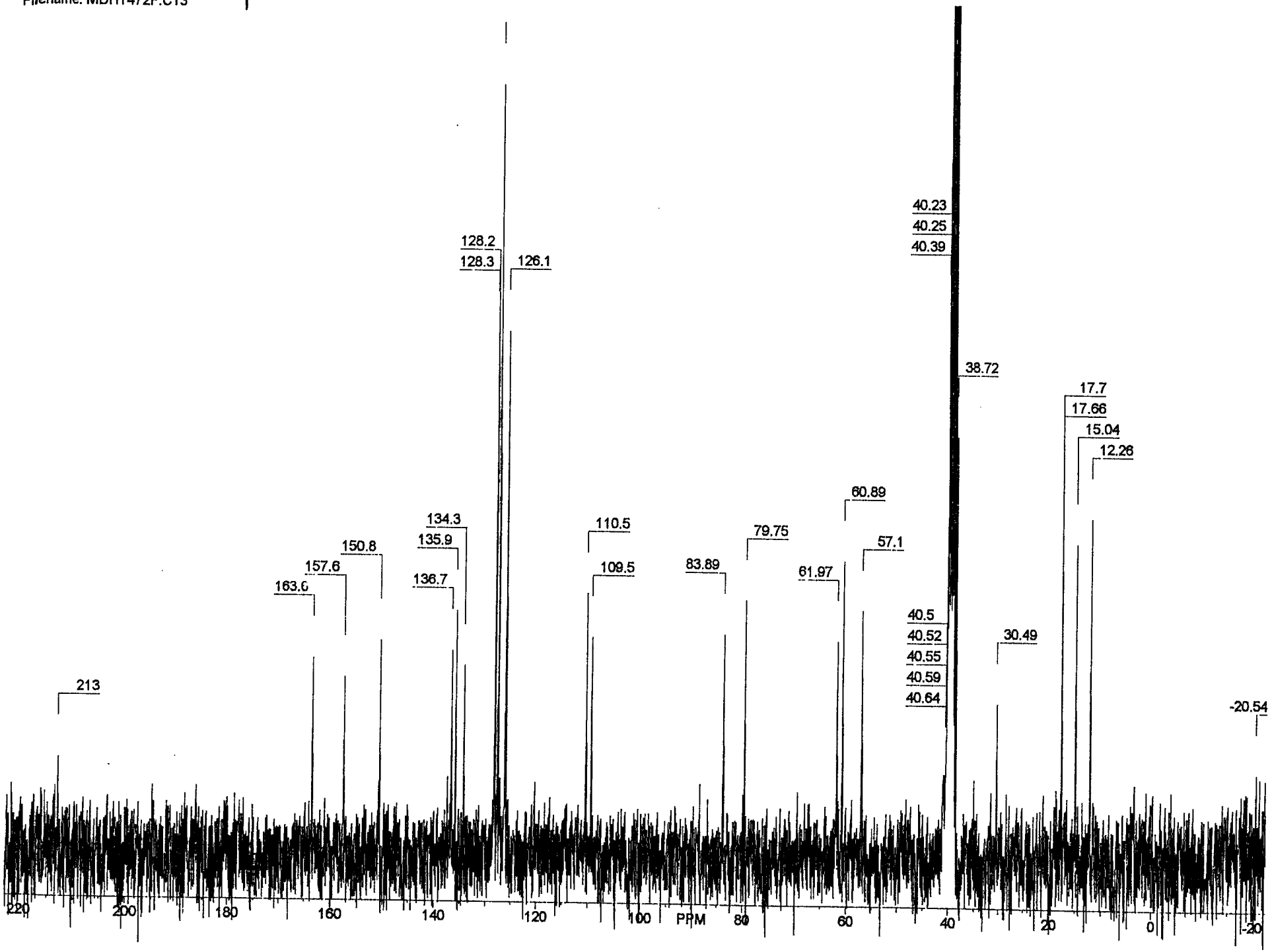


©2006 American Chemical Society Journal Of Organic Chemistry Vol. Page S175 Supplemental Page 26



©2008 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 27

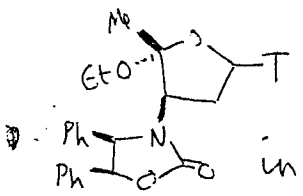
Filename: MDH1472F.C13 9d



© 1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5173 Umbricht Supplemental Page 30

9d

Filename: MDH1472F.C13



in DMSO

Should see 23 C signals

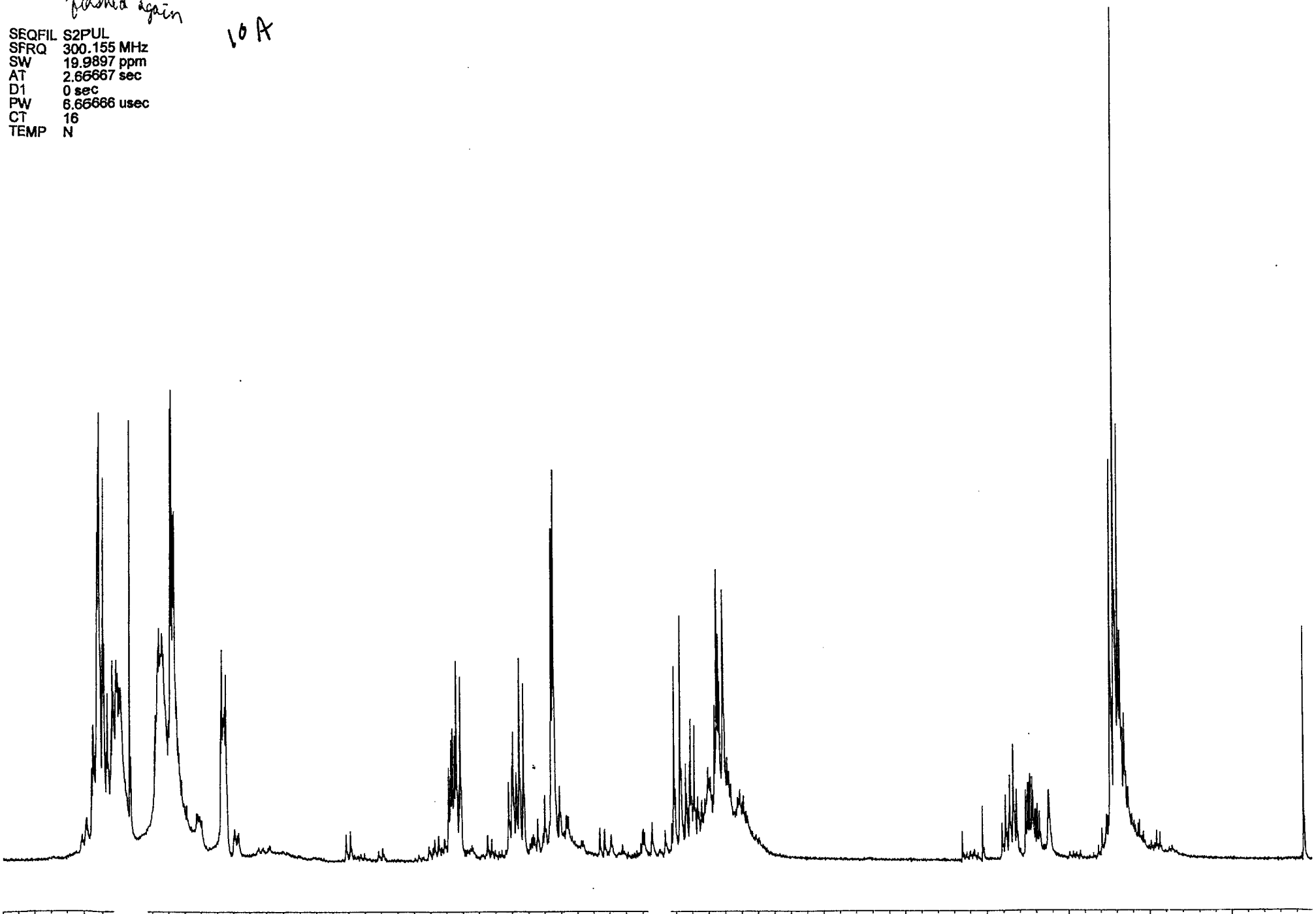
(

©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 51

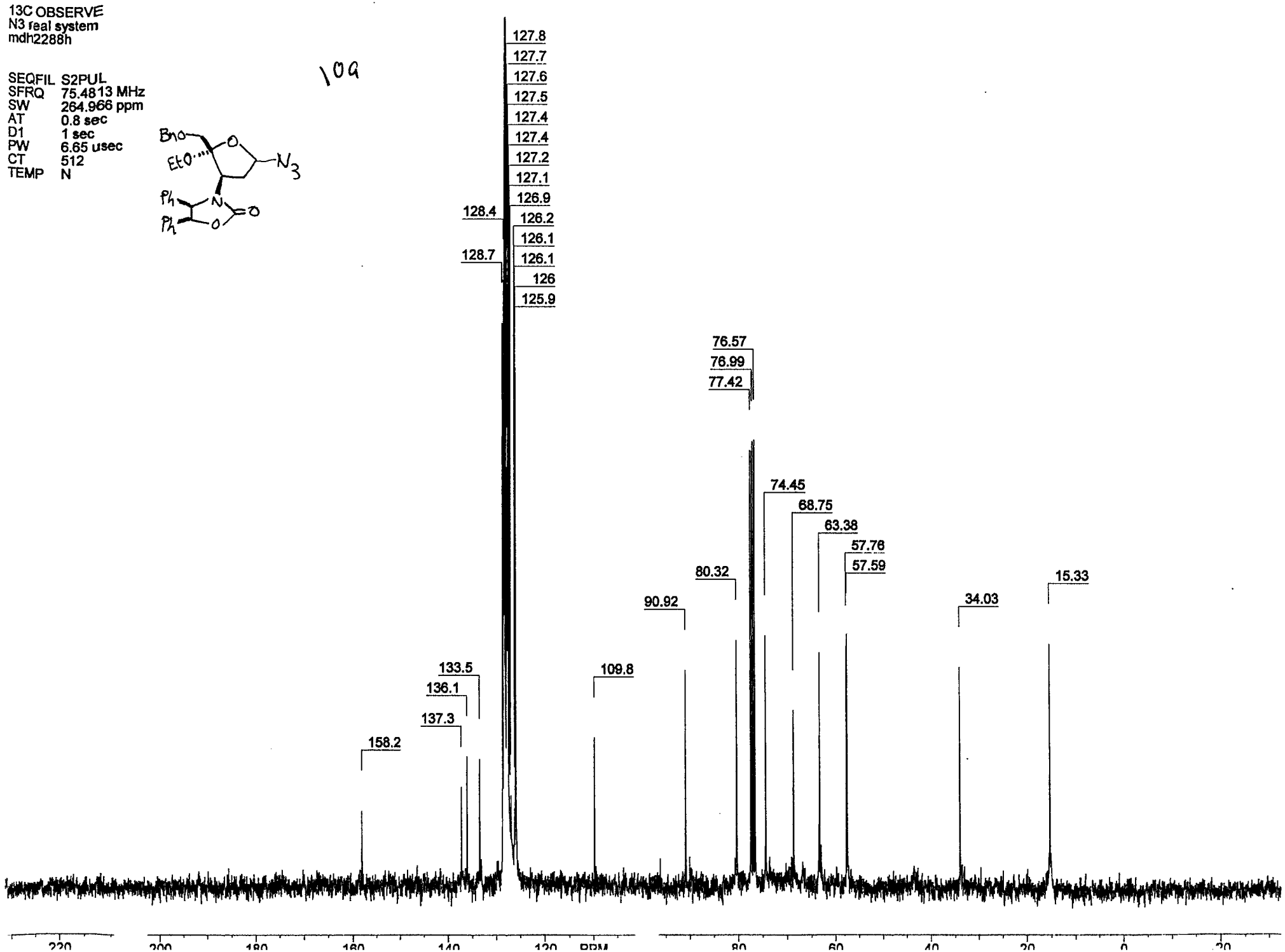
STANDARD 1H OBSERVE
mdh2288g=mdh2288e N_3 real
fr 4-6 → flashed again

10 A

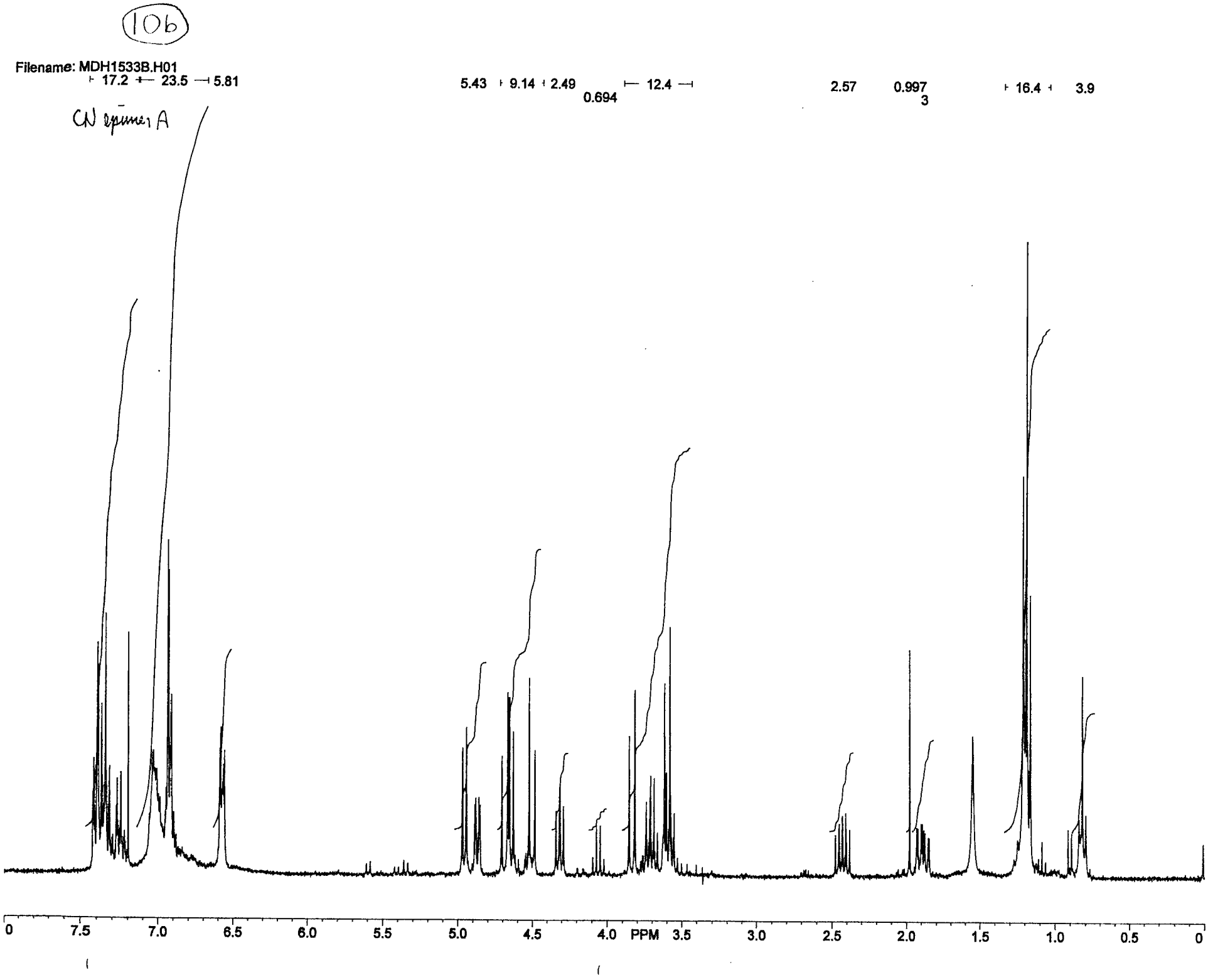
SEQFIL S2PUL
SFRQ 300.155 MHz
SW 19.9897 ppm
AT 2.66667 sec
D1 0 sec
PW 6.66666 usec
CT 16
TEMP N



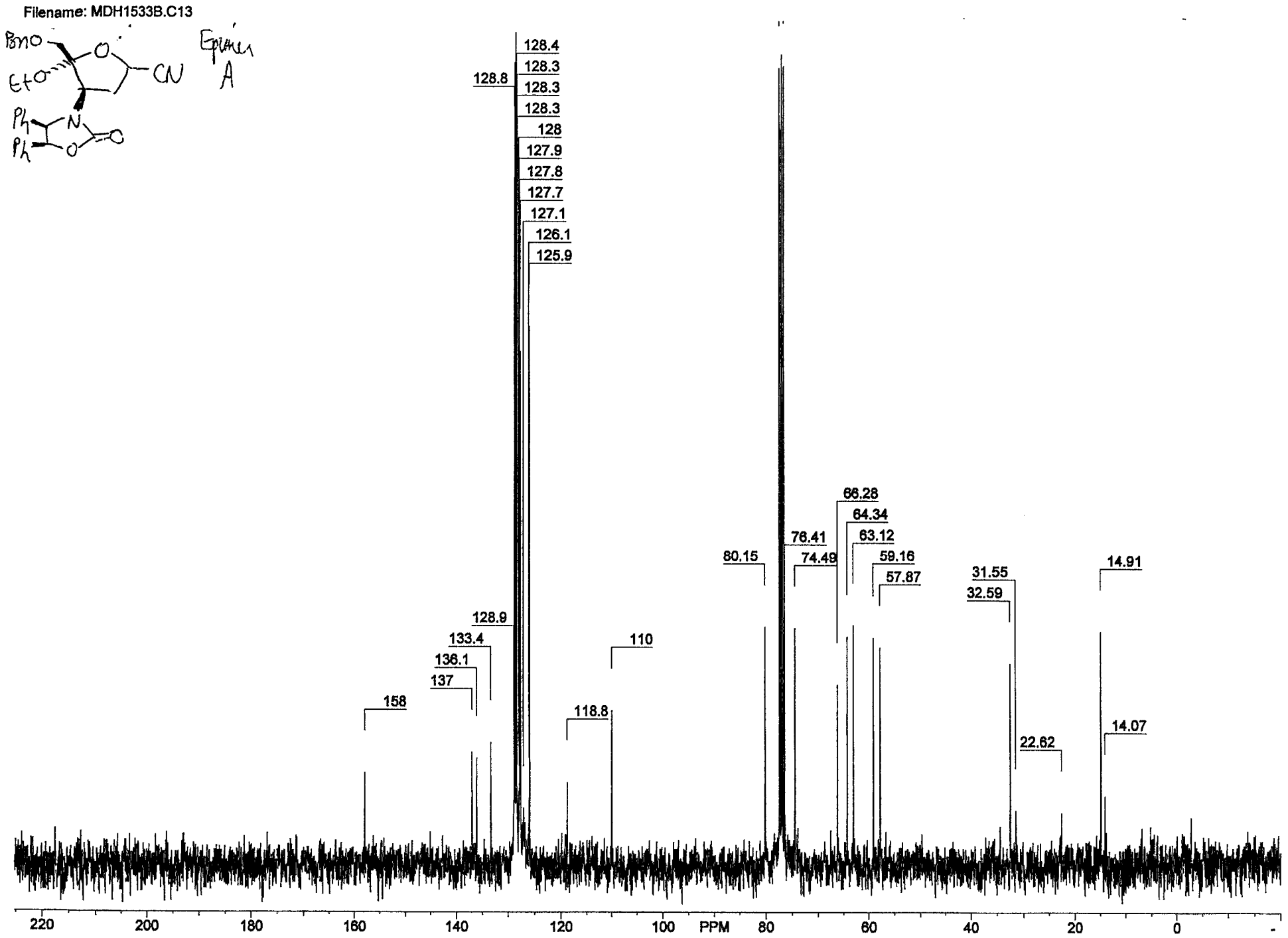
©1998 American Chemical Society Journal Of Organic Chemistry Vol. 1 Page S175 Electronic Supplementary Page 52



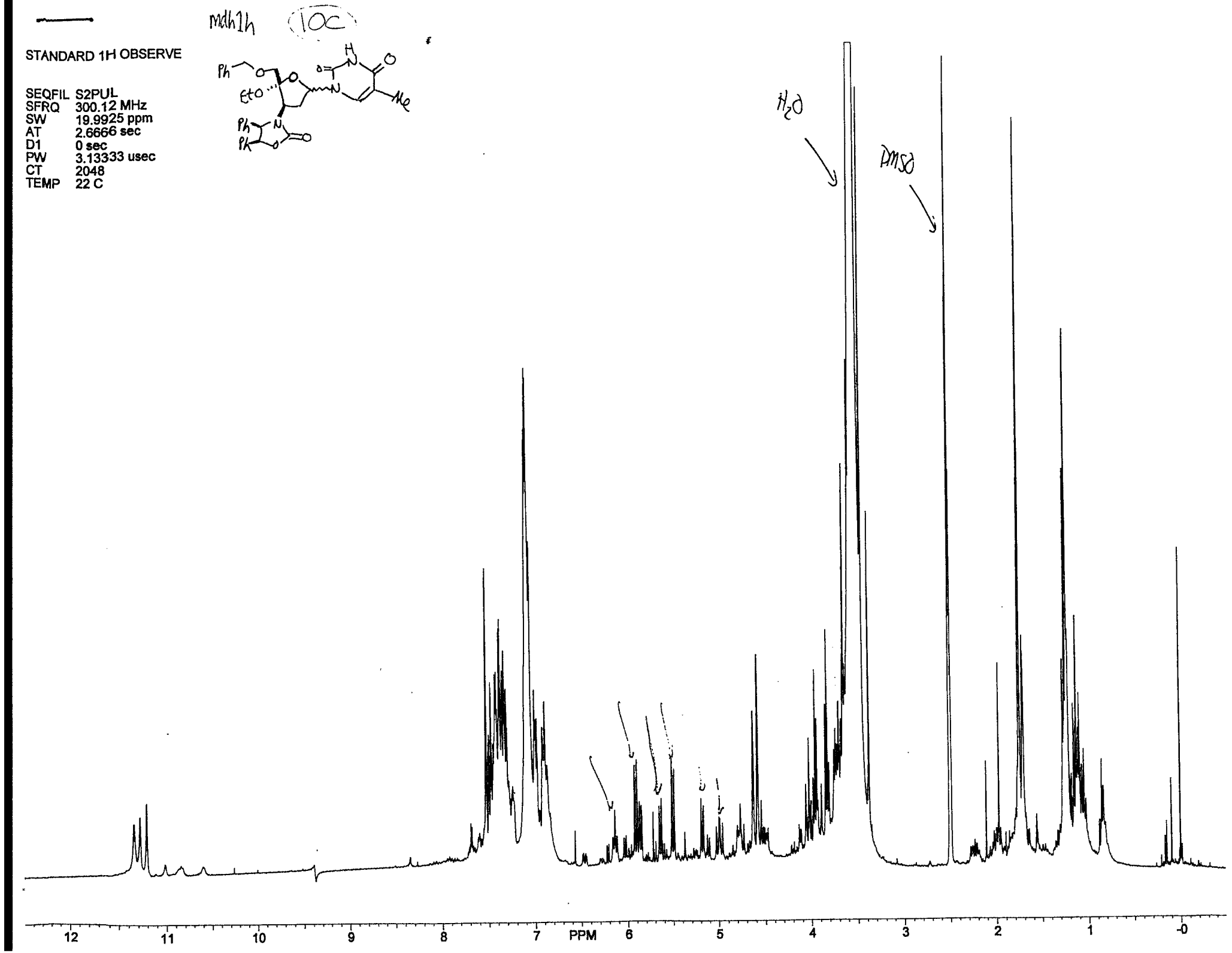
©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page S175 Supplemental Page 55



© 1998 American Chemical Society Journal Of Organic Chemistry Vol. Page S173 Omnient Supplemental Page 54



©1996 American Chemical Society Journal Of Organic Chemistry Vol. Page S175 Supplemental Page 55

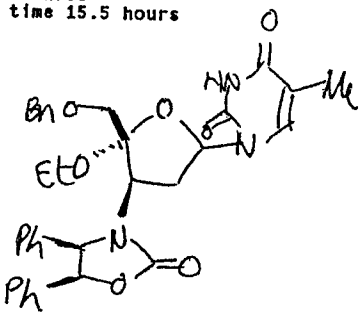


©2020 American Chemical Society Journal Of Organic Chemistry

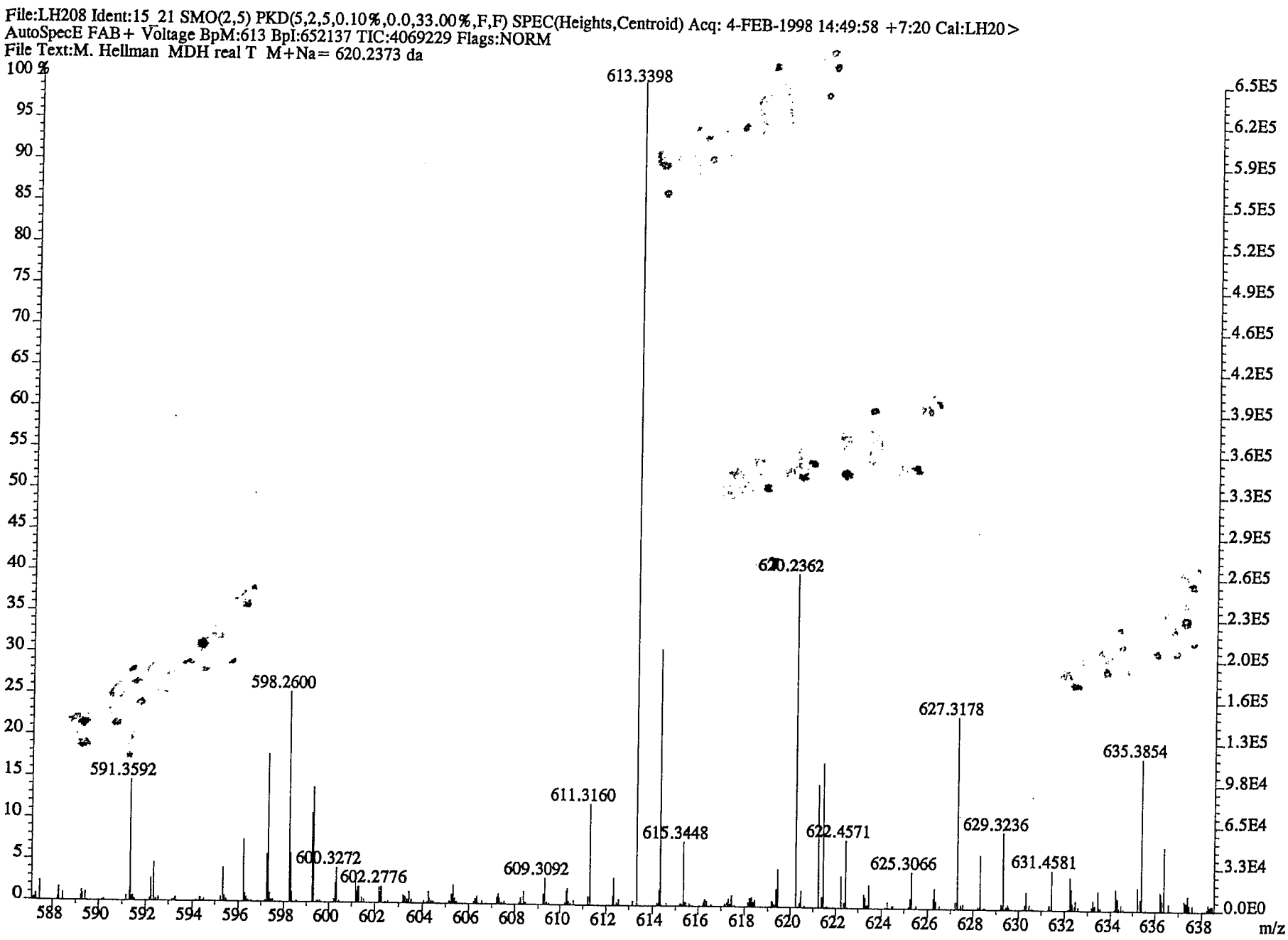
13C OBSERVE

Solvent: DMSO
Ambient temperature
INOVA-300 "elfin"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 35.0 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
30944 repetitions
OBSERVE C13, 75.4650065 MHz
DECOUPLE H1, 300.1204509 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 15.5 hours



©1998 American Chemical Society Journal of Organic Chemistry Vol. 1 Page 5175 Supplementary Page 37



© 1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 36

Elemental Composition Date : 4-FEB-1998

File:LH208 Ident:15_21 SMO(2,5) PKD(5,2,5,0.10%,0.0,33.00%,F,F)
AutoSpecE FAB+ Voltage BpM:613 BpI:652137 TIC:4069229 Flags:NORM
File Text:M. Hellman MDH real T M+Na= 620.2373 da
Heteroatom Max: 16 Ion: Both Even and Odd
Limits:

619.438	1.8					-0.5	32	0	34	0	5	0
622.242	100.0			4.0		20.0	34	1	38	3	7	1
Mass	%RA	Pks	Std	PPM	mDa	Calc. Mass	DBE	C	13C	H	N	O	Na
622.242477	3.8												
621.453002	17.4												
621.238207	14.8			3.9	2.4	621.240625	18.5	33	1	35	3	7	1
620.441160	2.0												
620.236207	40.3			1.7	1.1	620.237271	18.5	34		35	3	7	1*
				-5.5	-3.4	620.232800	19.0	33	1	34	3	7	1

©1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5173 Omonent Supplemental Page 39

Elemental Composition

Date : 4-FEB-1998

Formula	Monoisotopic Mass	Average Mass	Integer Mass	DBE
C29.H44.N4.O6.Na	567.315055	567.683789	567	9.5
C34.H35.N3.O7.Na	620.237271	620.659752	620	18.5

© 1998 American Chemical Society Journal Of Organic Chemistry Vol. Page 5175 Supplemental Page 40