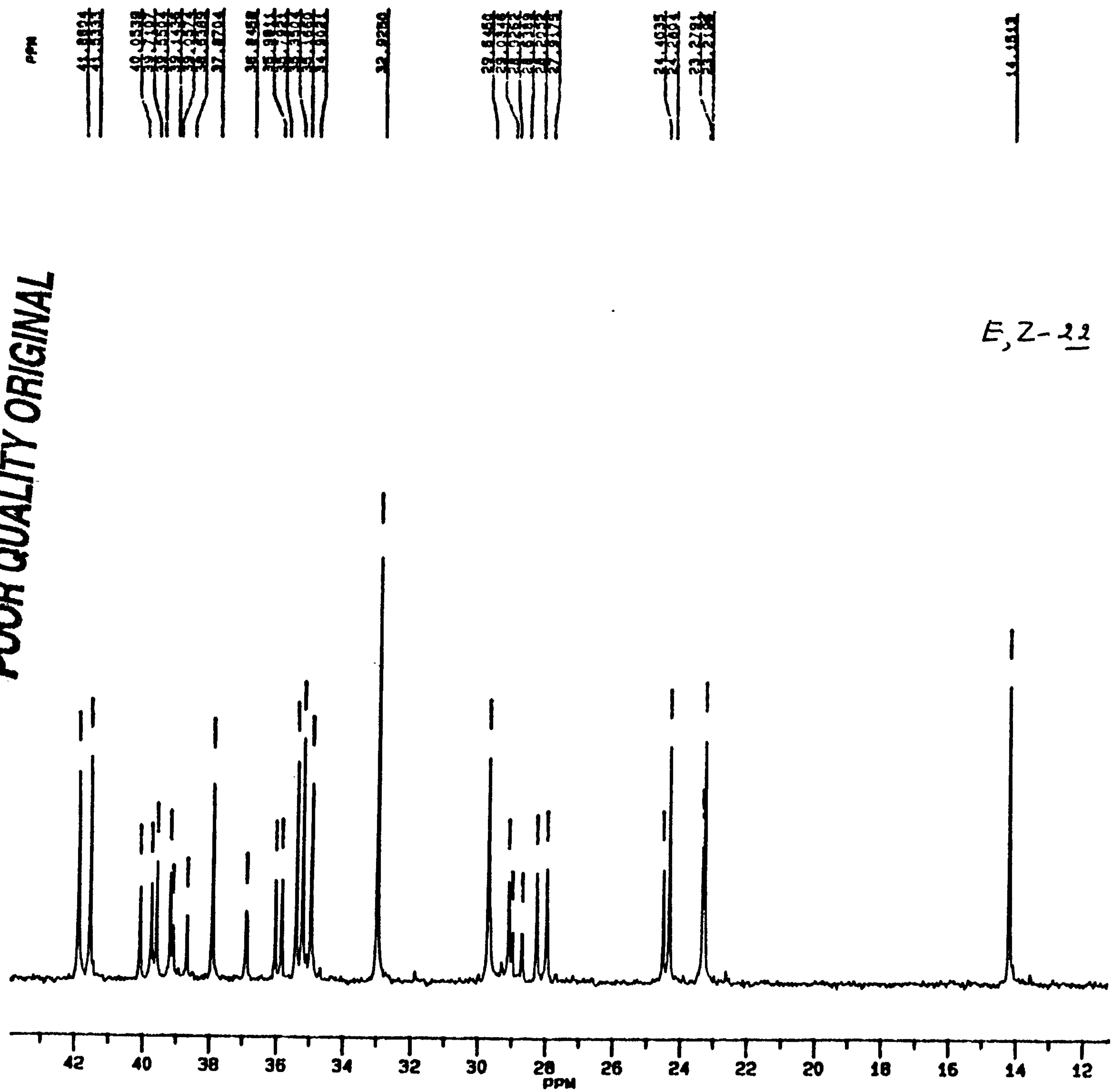
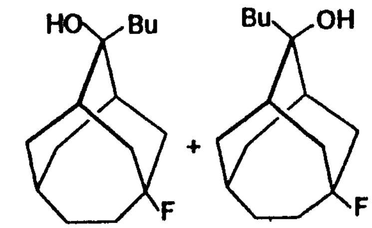
JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(10), 3218-3223, DOI:10.1021/jo9718197

81

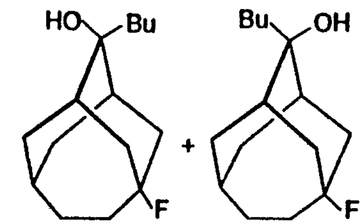


E,Z-22



POOR QUALITY ORIGINAL

E,Z-22



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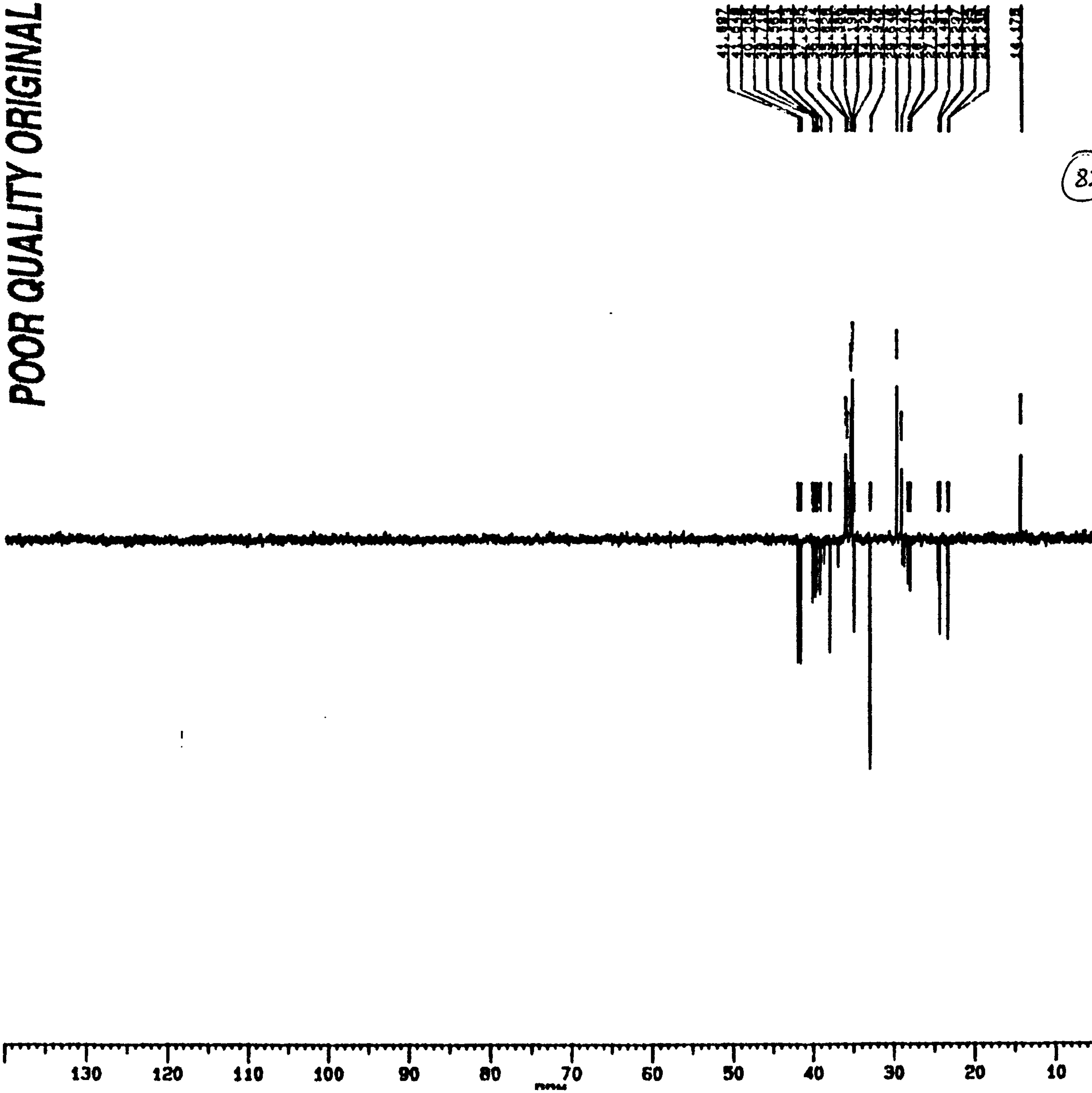

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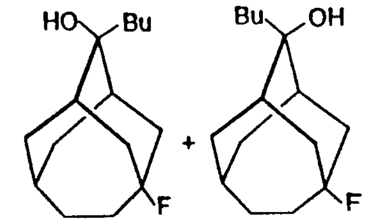
SY 62.0
O1 3218.018
SI 32768
TD 32768
SW 15625.000
HZ/PT .954

PW 0.0
RD 0.0
AQ 1.049
RG 200
NS 122
TE 297

FW 19600
O2 4100.000
DP 20H DO

LB 1.000
GB .100
CX 20.00
CY 10.00
F1 140.001P
F2 5.007P
HZ/CM 424.528
PPM/CM 6.750
SR -4042.54

E,Z-22



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TD	32768
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HZ/PT	.954
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RD	0.0
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RG	200
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TE	297
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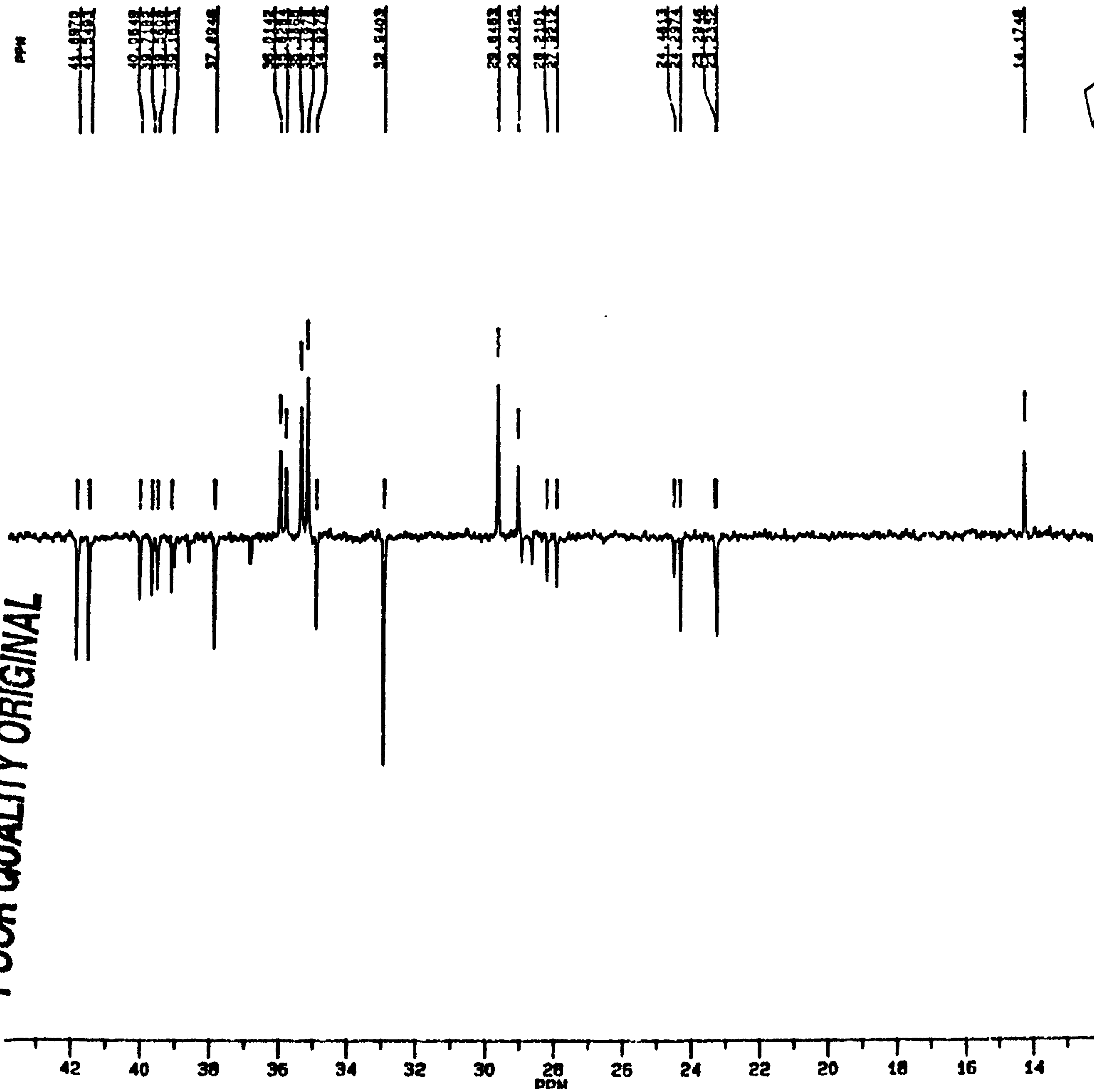

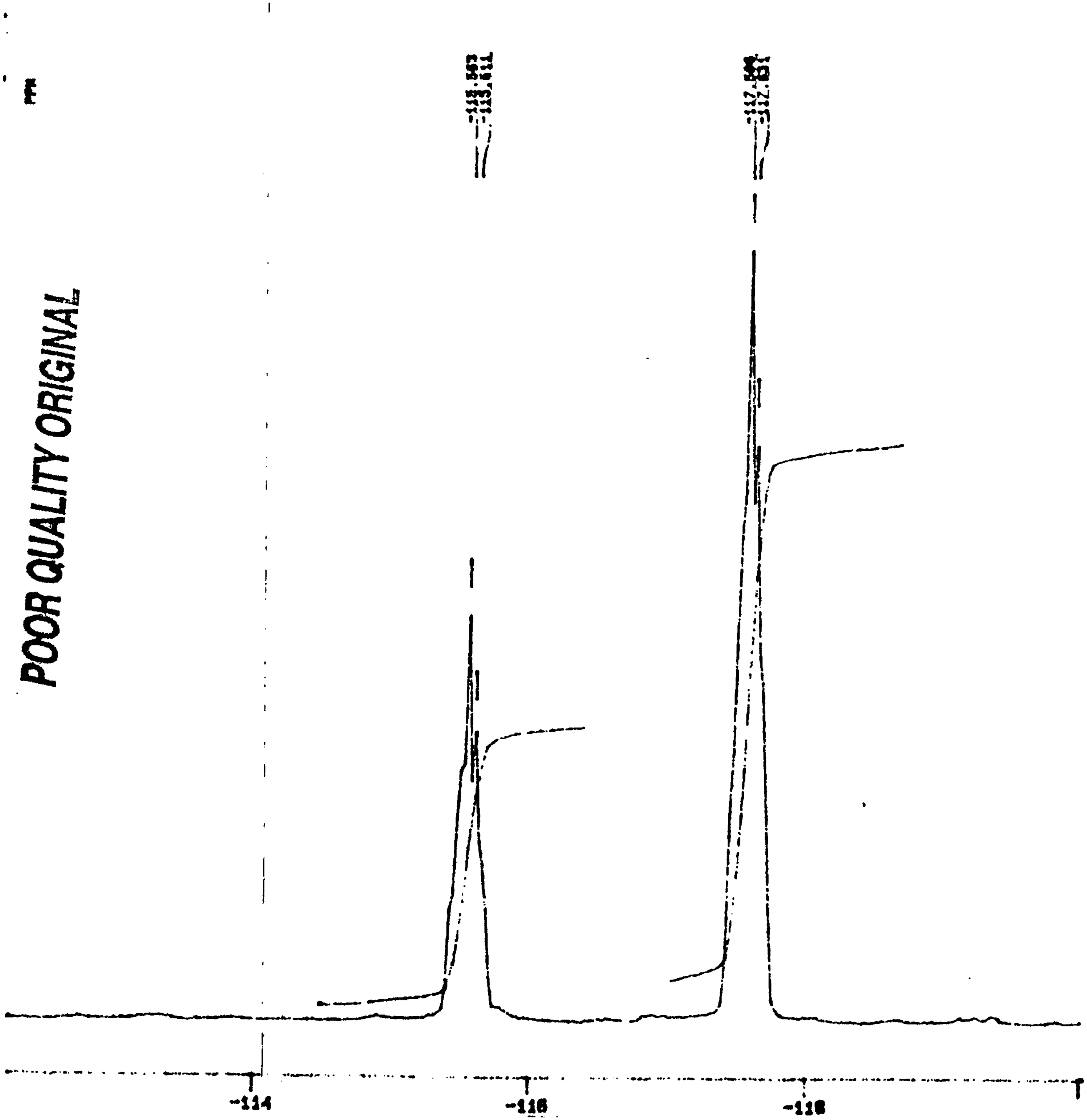

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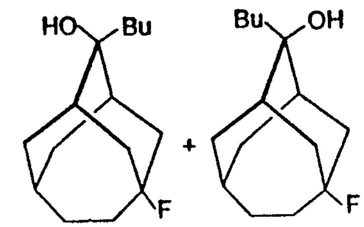

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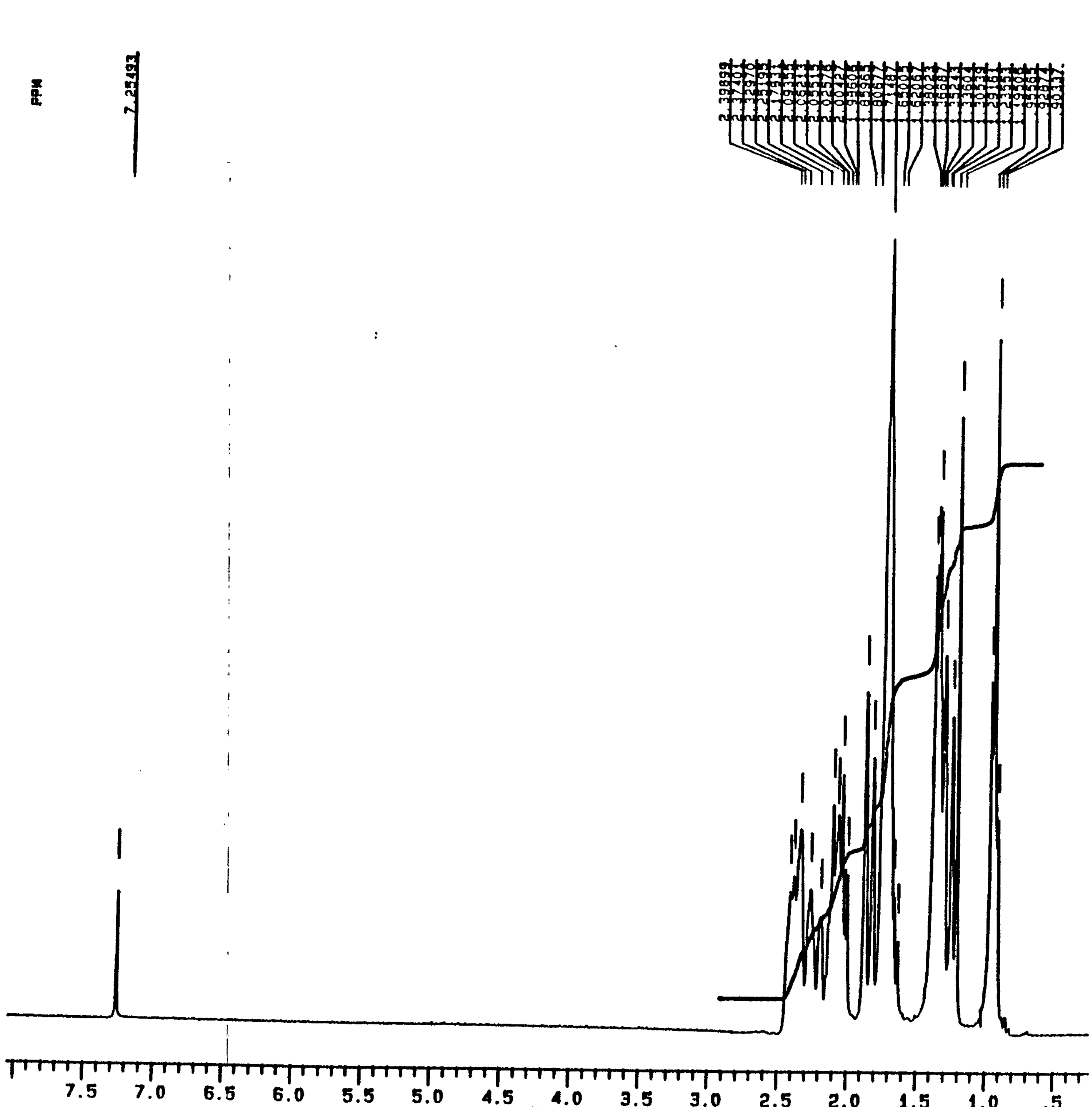

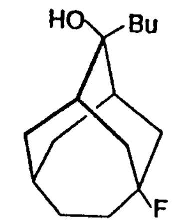

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NS	64
TE	297
FW	3000
O2	3210.000
DP	63L PO
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GB	.100
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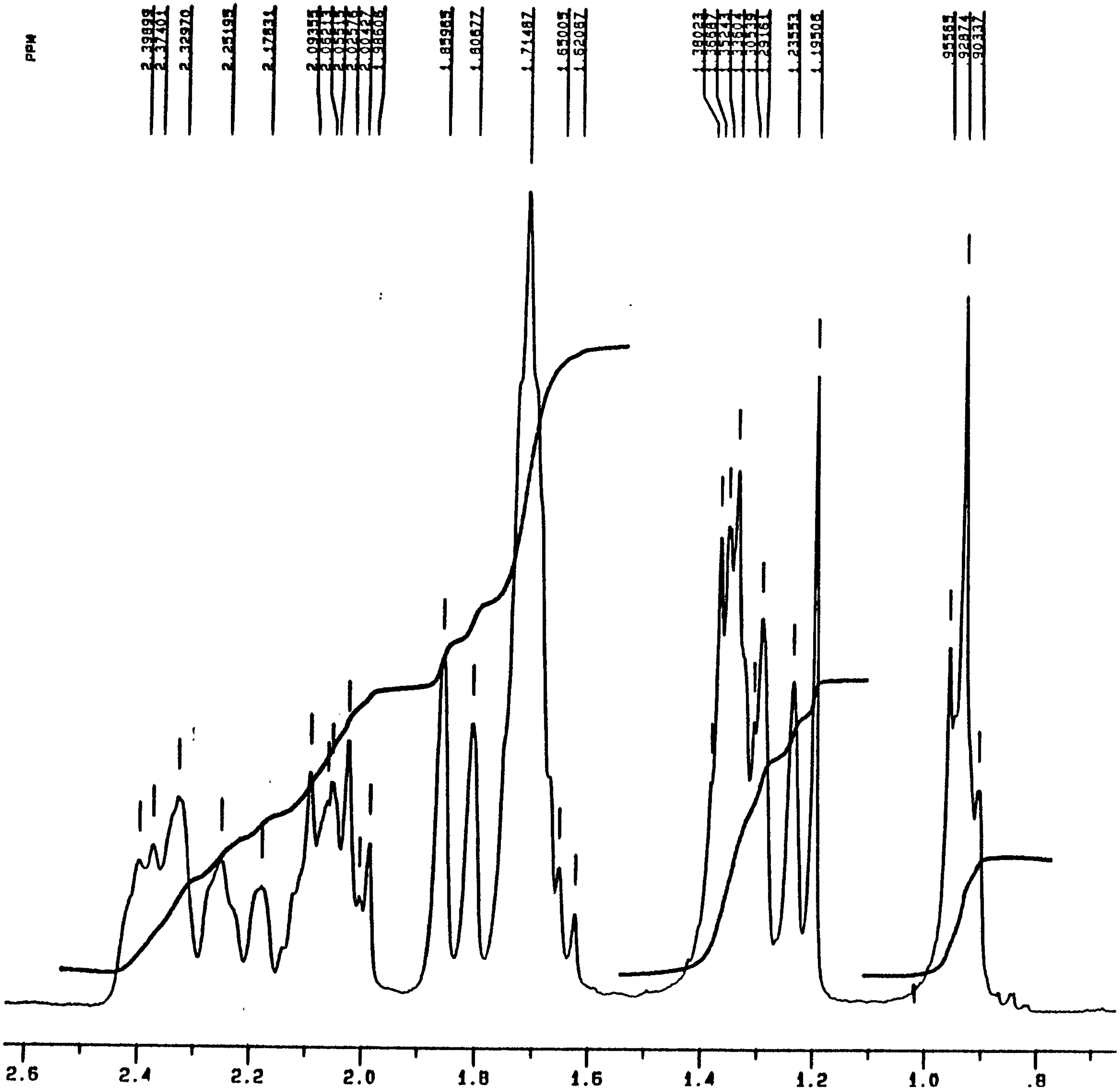

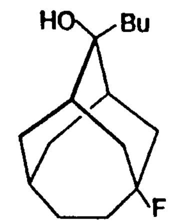

E-22

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SW 2994.012
HZ/PT .365

PW 3.0
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AQ 2.736
RG 32
NS 64
TE 297

FW 3000
O2 3210.000
DP 63L PO

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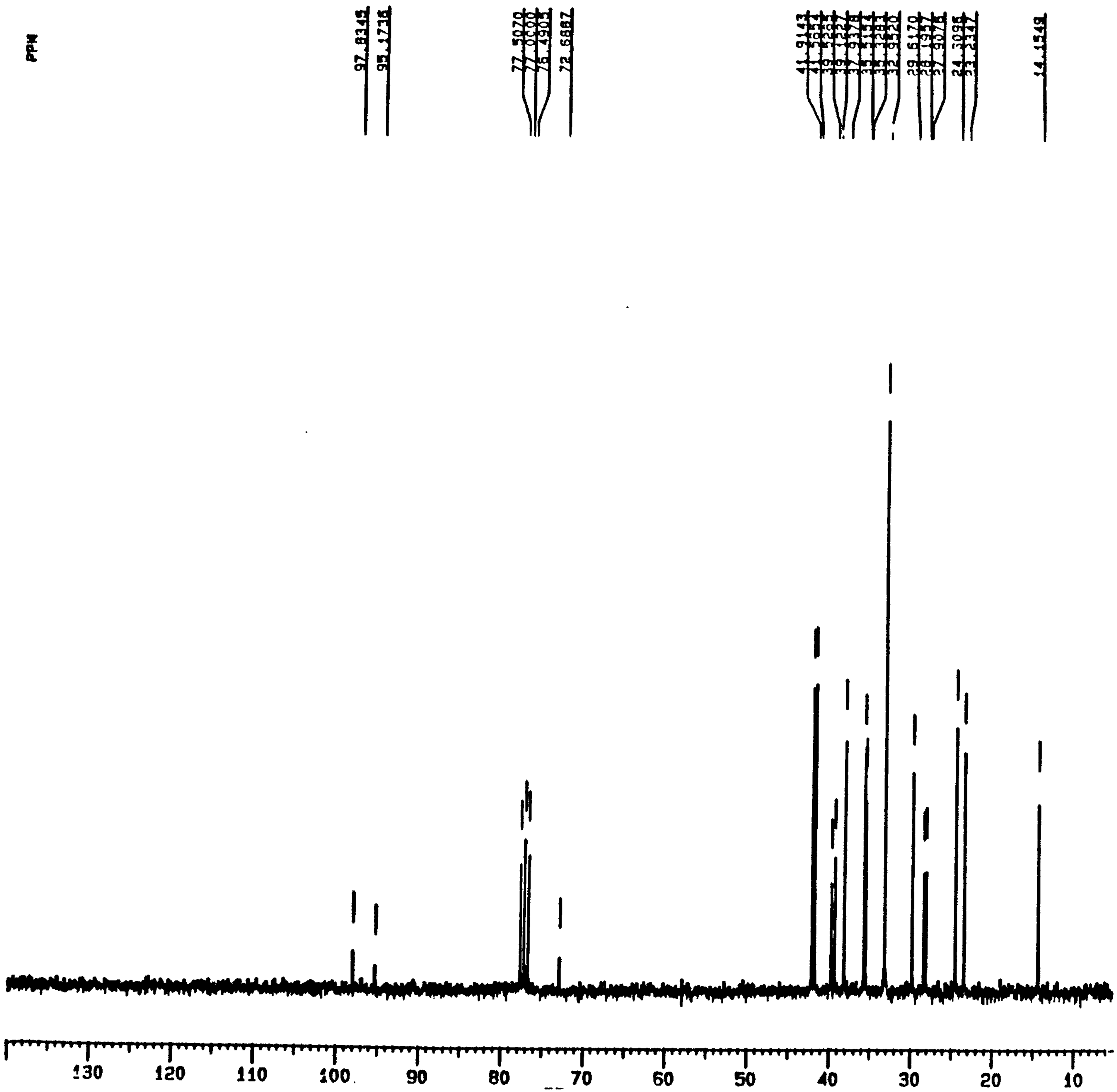

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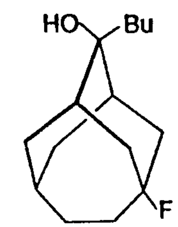

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SI   32768
TD   32768
SW   15625.000
HZ/PT     .954

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AQ       1.049
RG     200
NS     598
TE     297

FW   19600
O2    4100.000
DP   20H CPD

LB       1.000
GB        .100
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HZ/CM  424.528
PPM/CM   6.750
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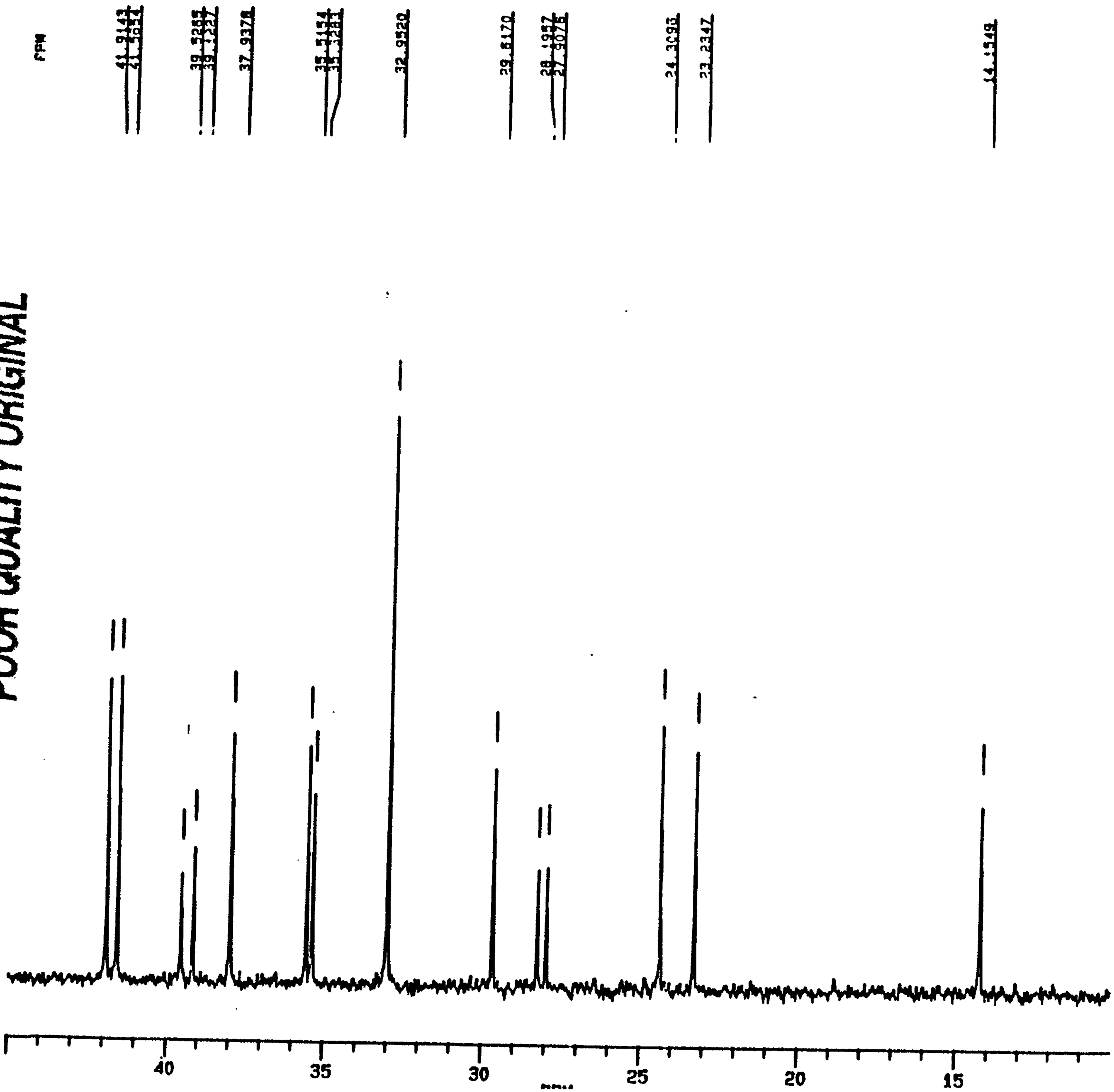
POOR QUALITY ORIGINAL



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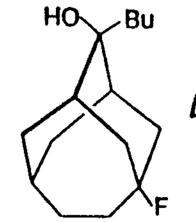

E-22

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SI 32768
TD 32768
SW 15625.000
HZ/PT .954

PW 5.0
RD 1.000
AQ 1.049
RG 200
NS 598
TE 297

FW 19600
O2 4100.000
DP 20H CPD

LB 1.000
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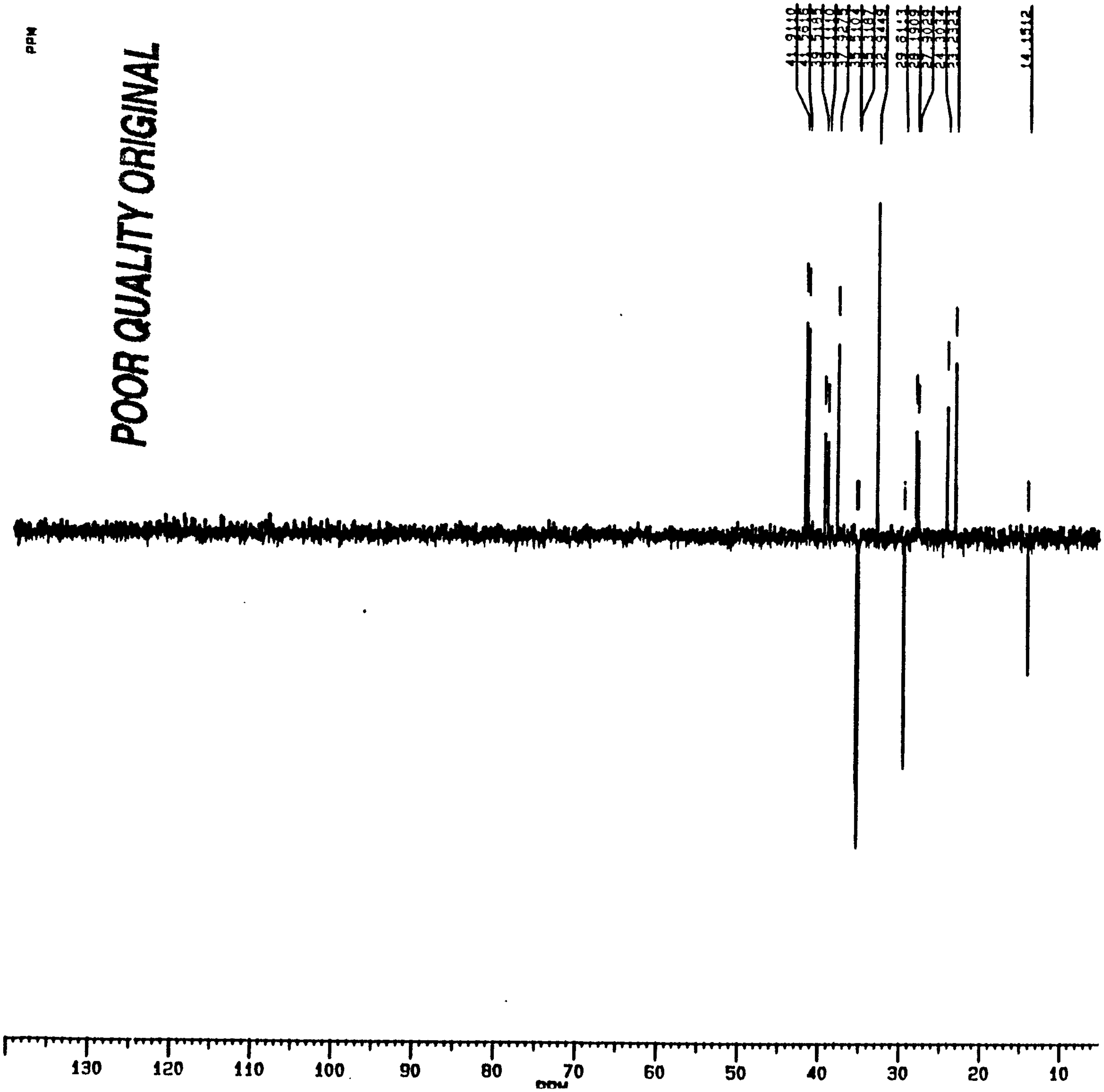
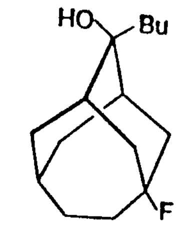
SR -4042.54





= E-22

89

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O1 3218.018
SI 32768
TD 32768
SW 15625.000
HZ/PT .954

PW 0.0
RD 0.0
AQ 1.049
RG 1600
NS 353
TE 297

FW 19600
O2 4100.000
DP 20H DO

LB 1.000
GB .100
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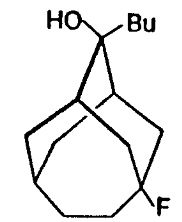
SR -4042.54

E-22



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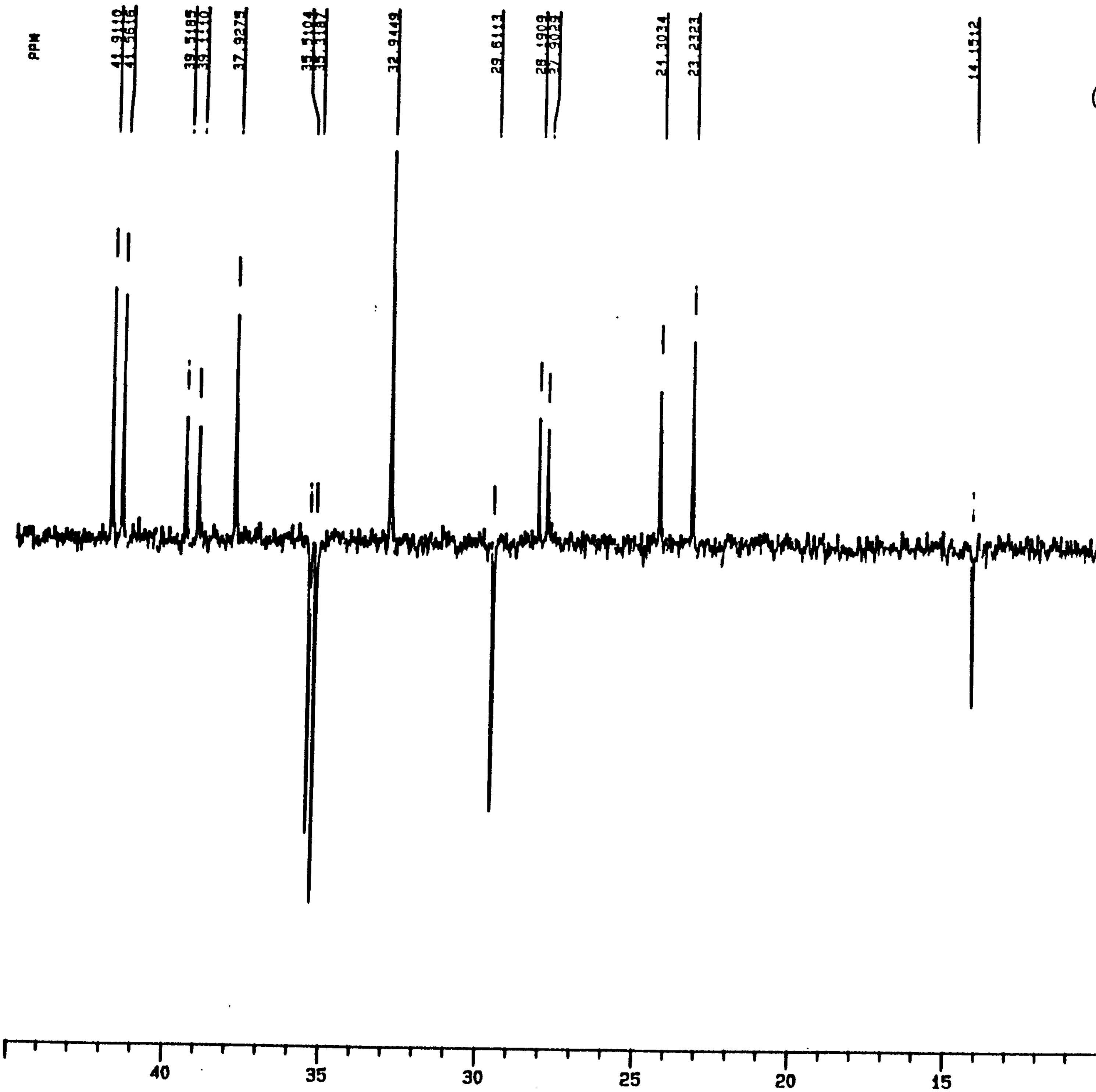

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RG 1600
NS 353
TE 297

FW 19600
O2 4100.000
DP 20H DO

LB 1.000
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SR -4042.54

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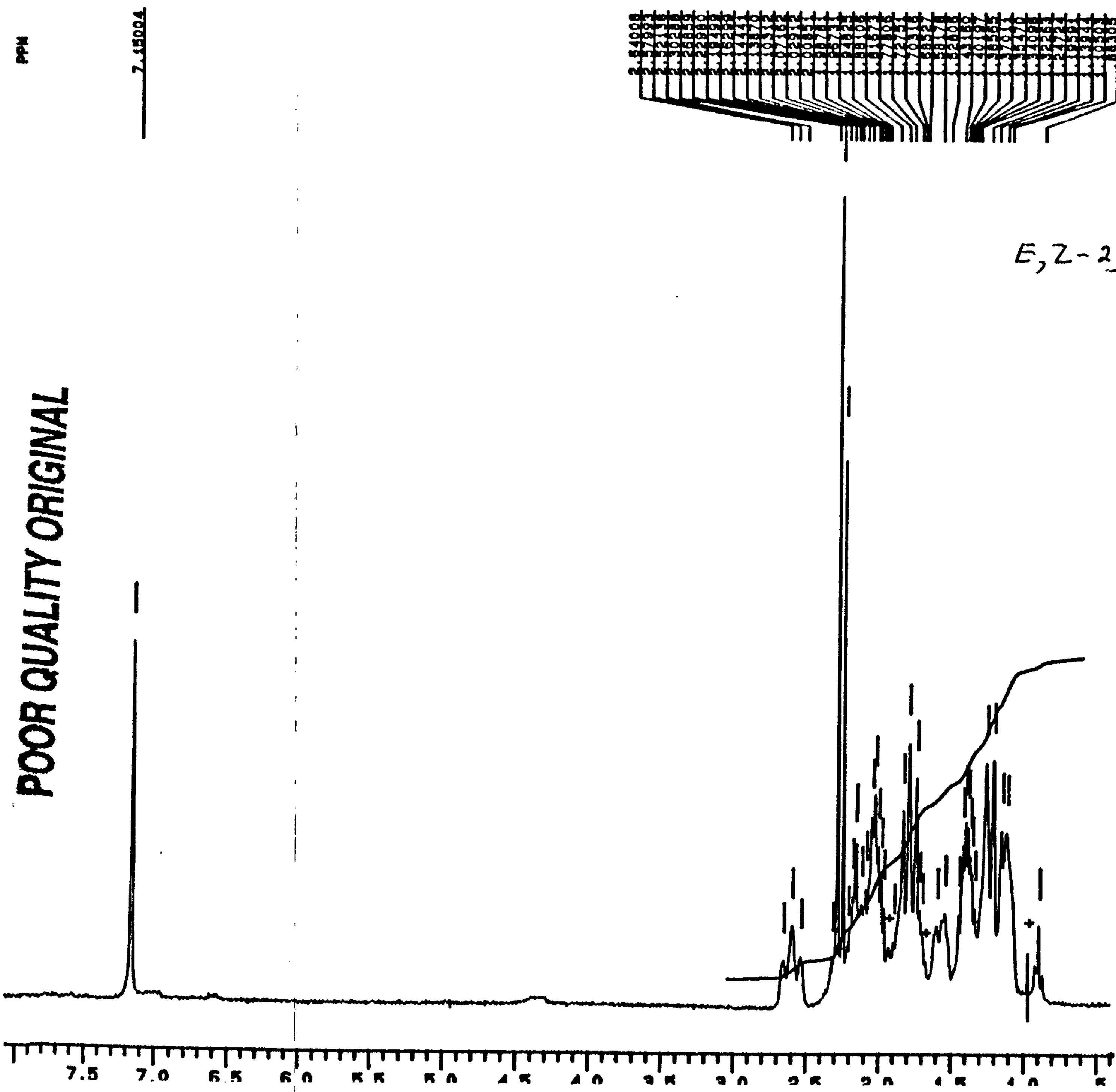

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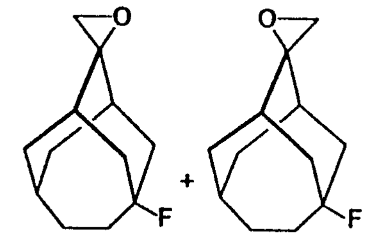

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TD 16384
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HZ/PT .365

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RD 1.000
AQ 2.736
RG 2
NS 64
TE 297

FW 3000
O2 3210.000
DP 63L PO

LB .100
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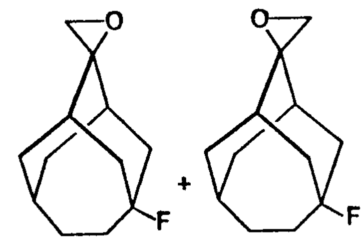
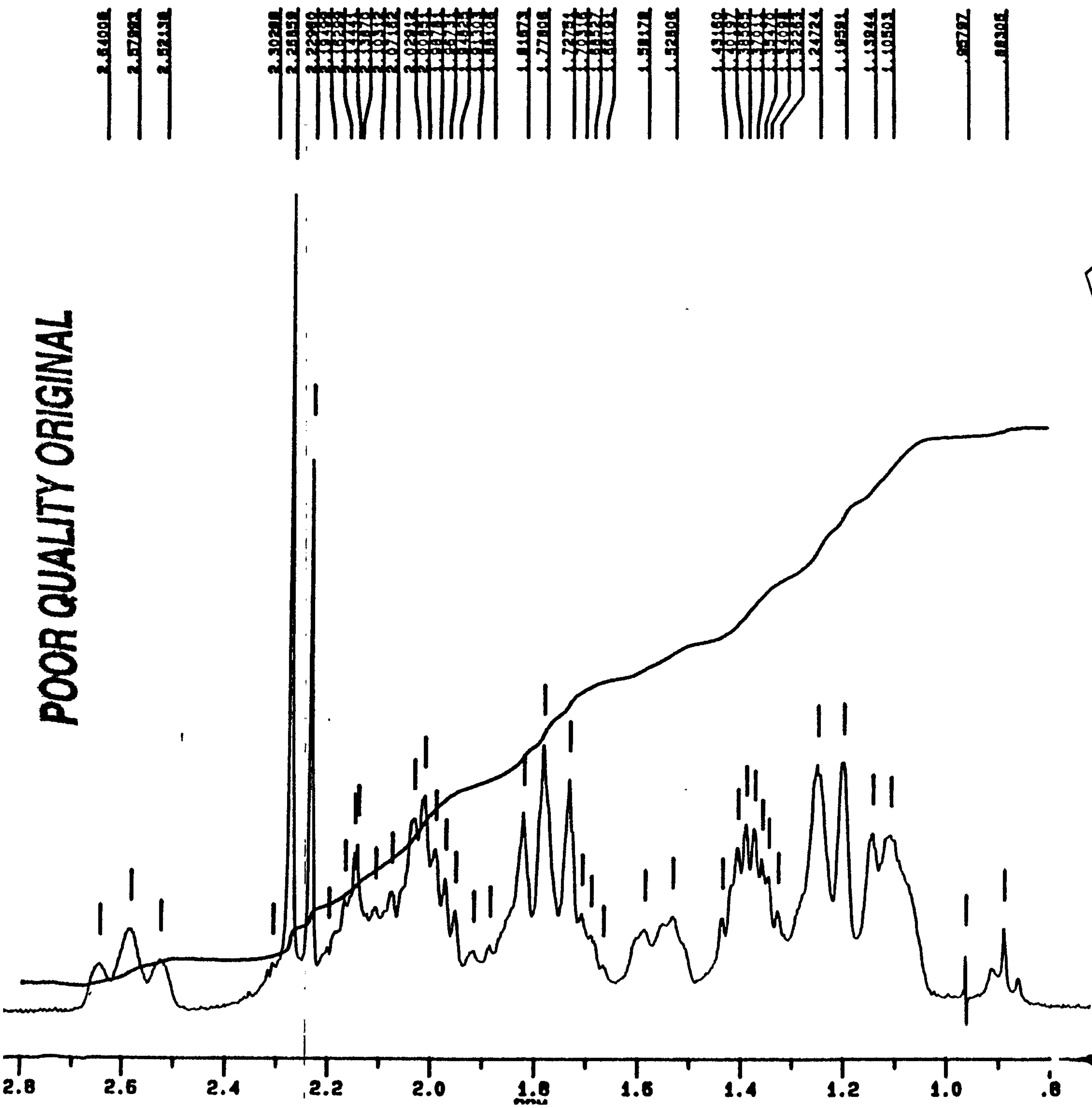
92

E, Z-23



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SF     250.133
SY  100.0
O1    4102.190
SI   16384
TD   16384
SW    2994.012
HZ/PT     .365

PW        3.0
RD        1.000
AQ        2.736
RG        2
NS       64
TE      297

FW    3000
O2    3210.000
DP   63L PO

LB         .100
GB         .100
CX       20.00
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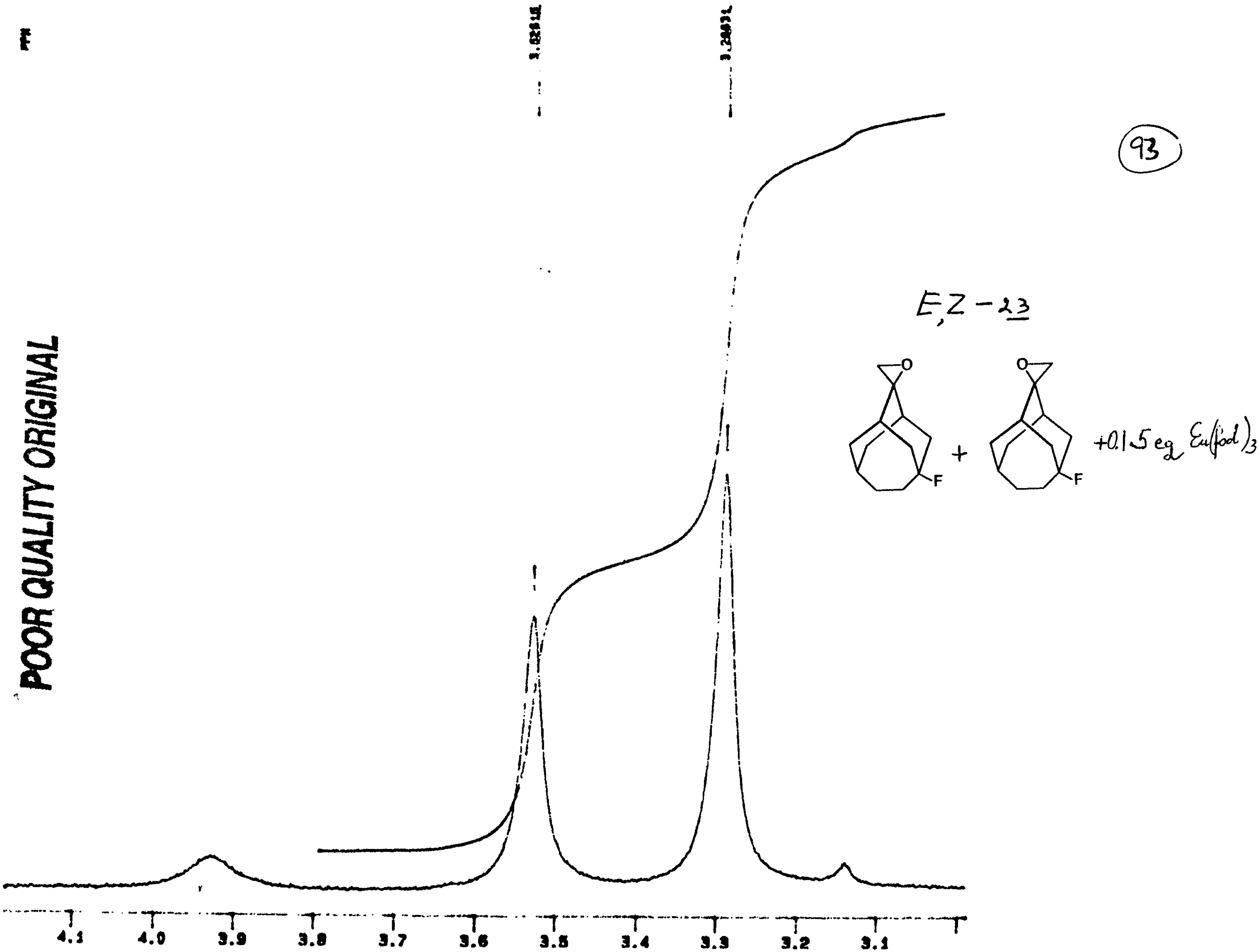

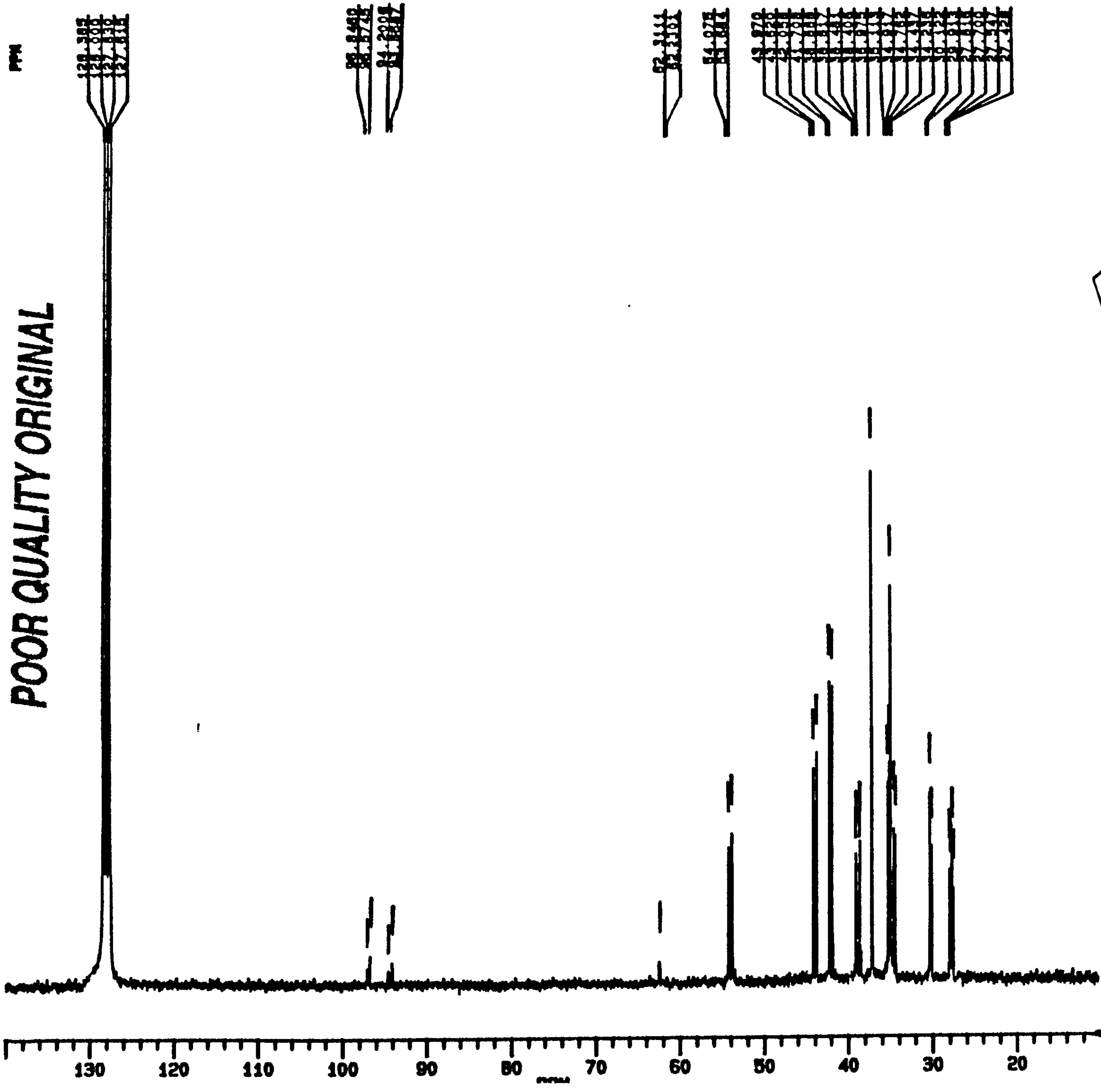

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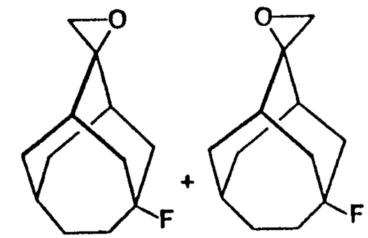
E,Z-23



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HZ/PT .954

PW 5.0
RD 2.000
AQ 1.049
RG 200
NS 2105
TE 297

FW 19600
O2 4100.000
DP 20H CPD

LB 1.000
GB .100
CX 20.00
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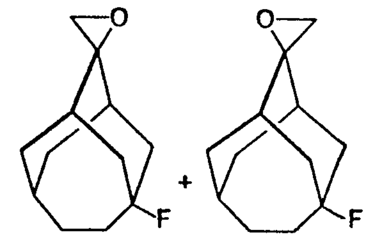
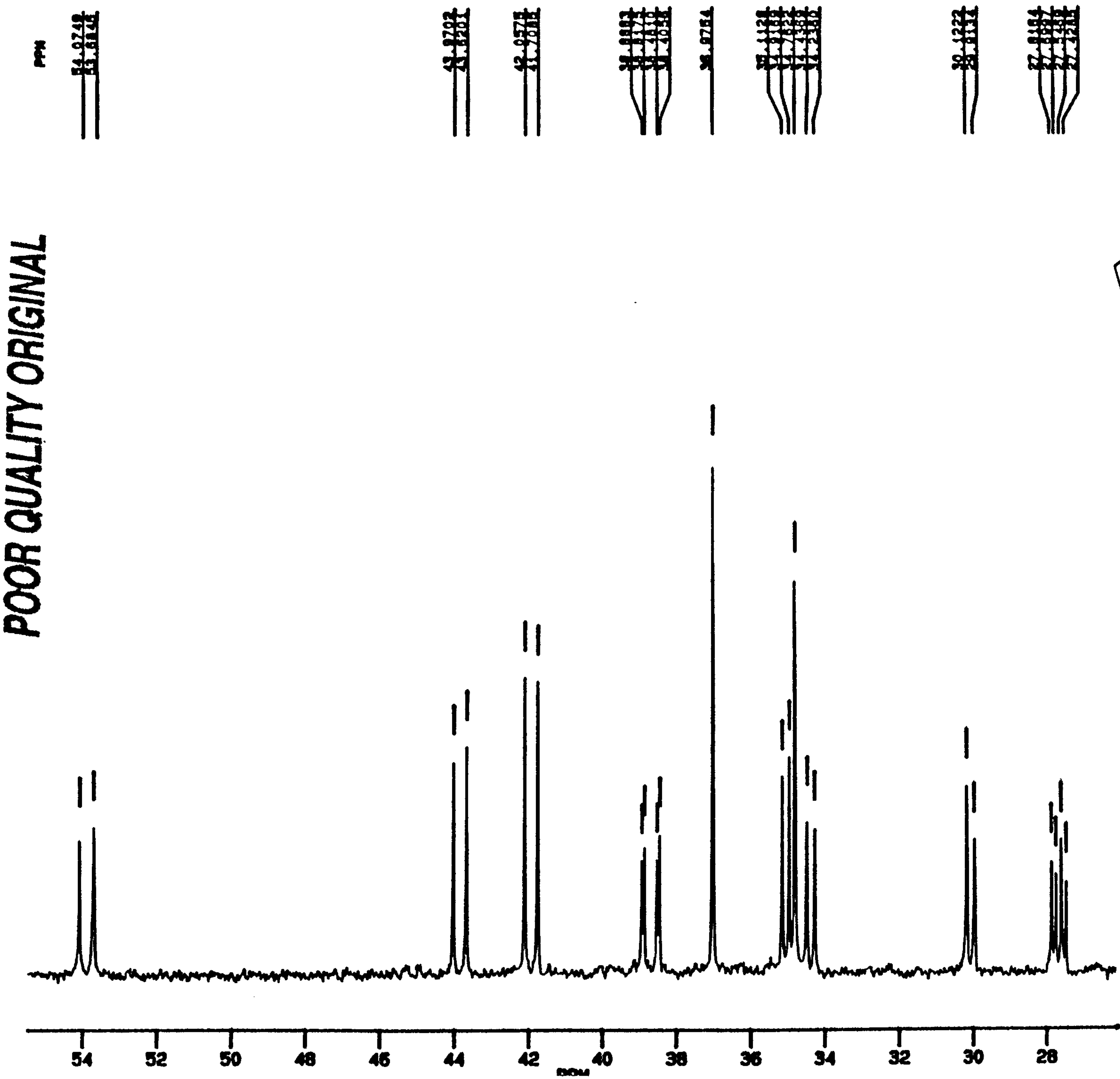
(95)

E,Z- 23





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RG 200
NS 2105
TE 297

FW 19600
O2 4100.000
DP 20H CPD

LB 1.000
GB .100
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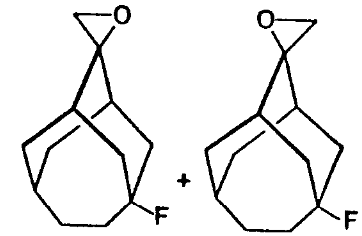
E,Z-23



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RG 200
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TE 297

FW 19600
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DP 20H DO

LB 1.000
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PPM/CM 6.500
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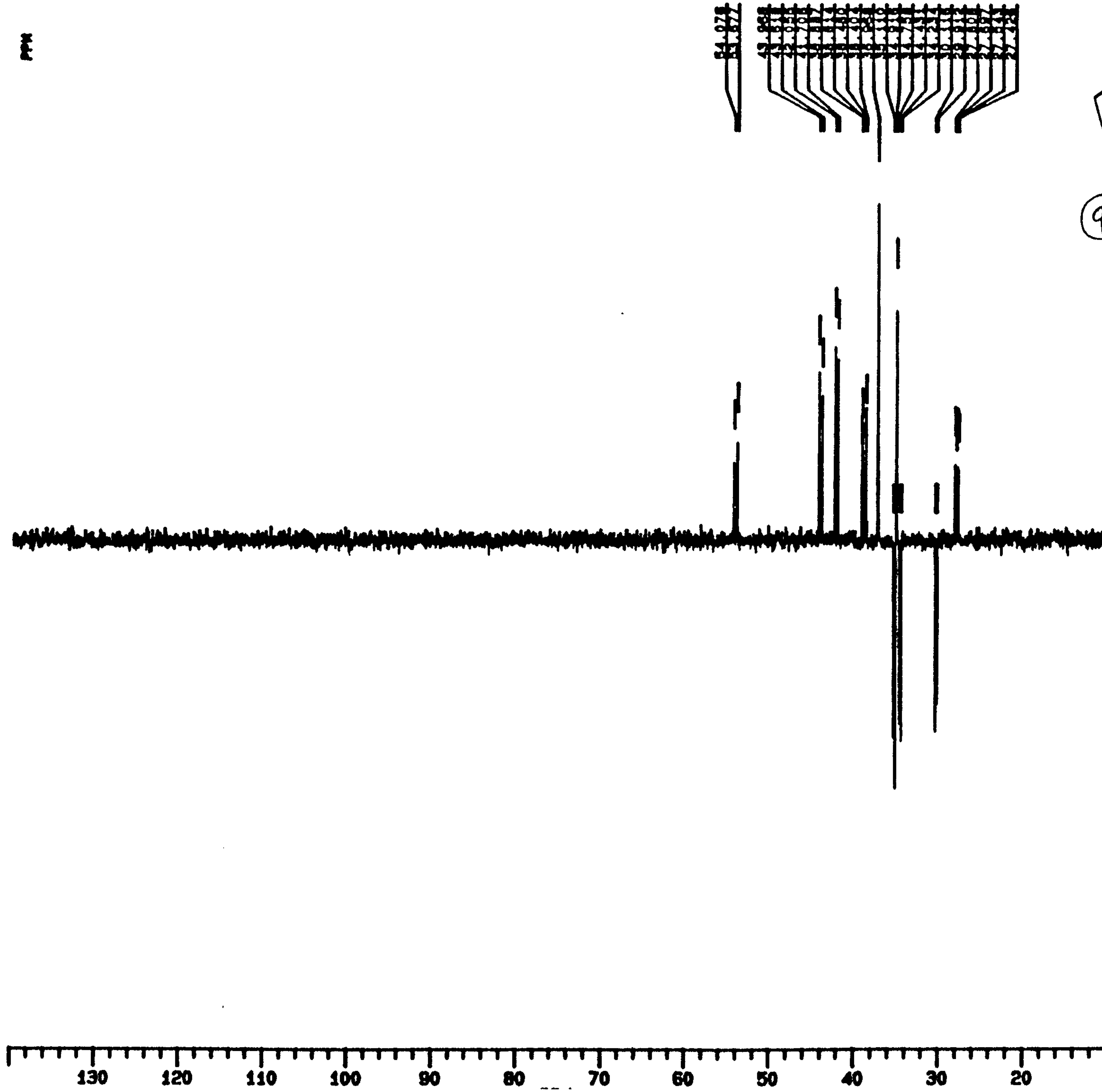
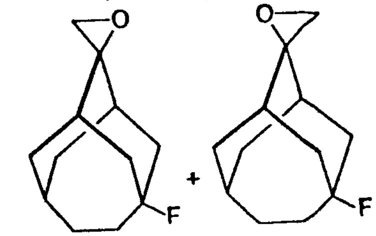


E,Z - 23



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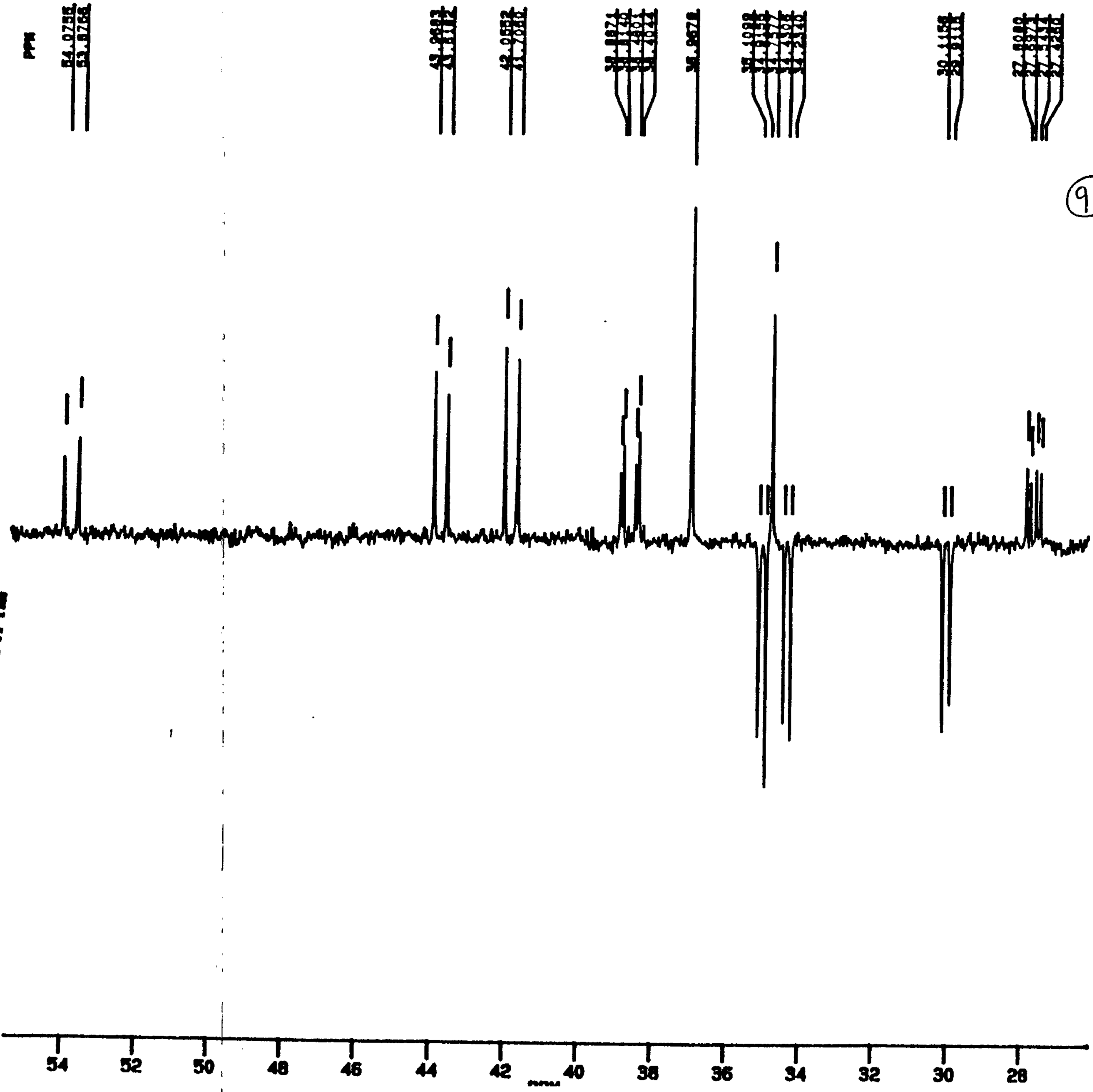

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TD 32768
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RG 200
NS 955
TE 297

FW 19600
O2 4100.000
DP 20H DO

LB 1.000
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CX 20.00
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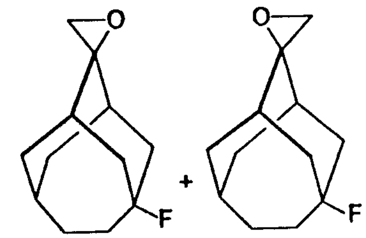
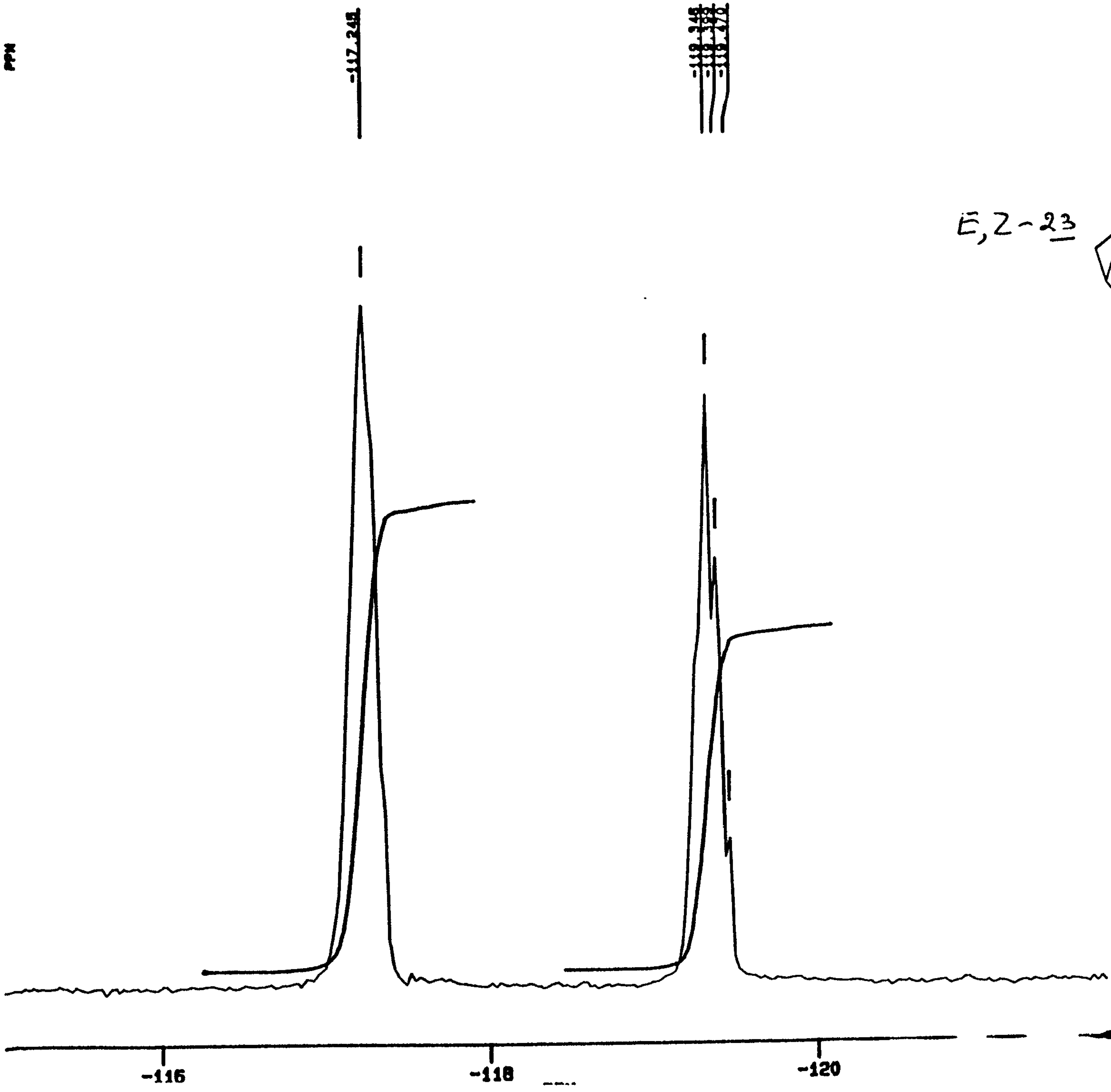
98

E,Z-23





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HZ/PT 6.104

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AQ .164
RG 160
NS 205
TE 297

FW 150000
O2 3210.000
DP 63L PO

LB 1.000
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CY 12.00
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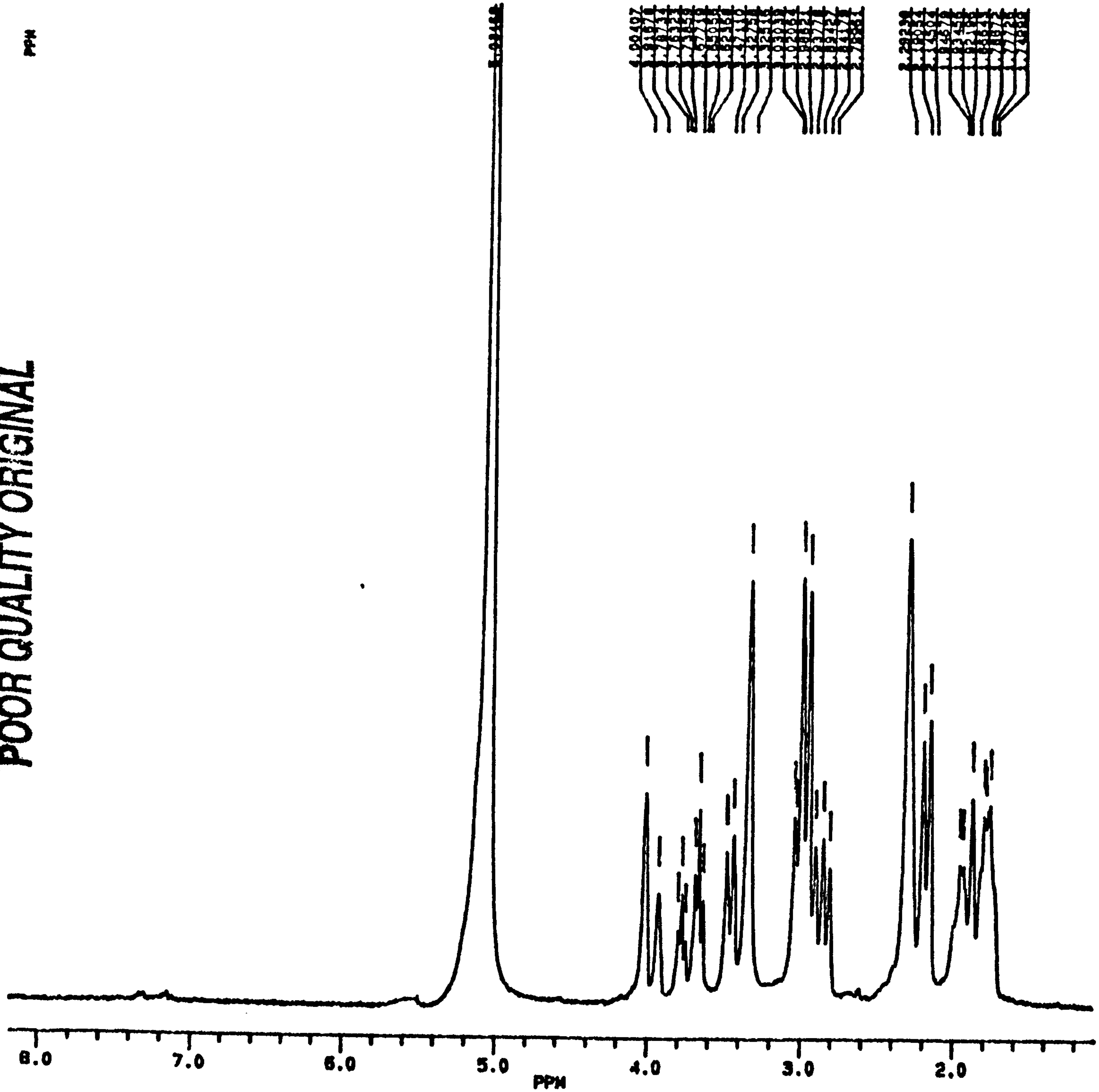

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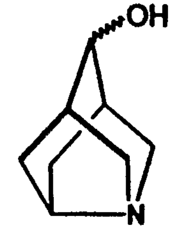
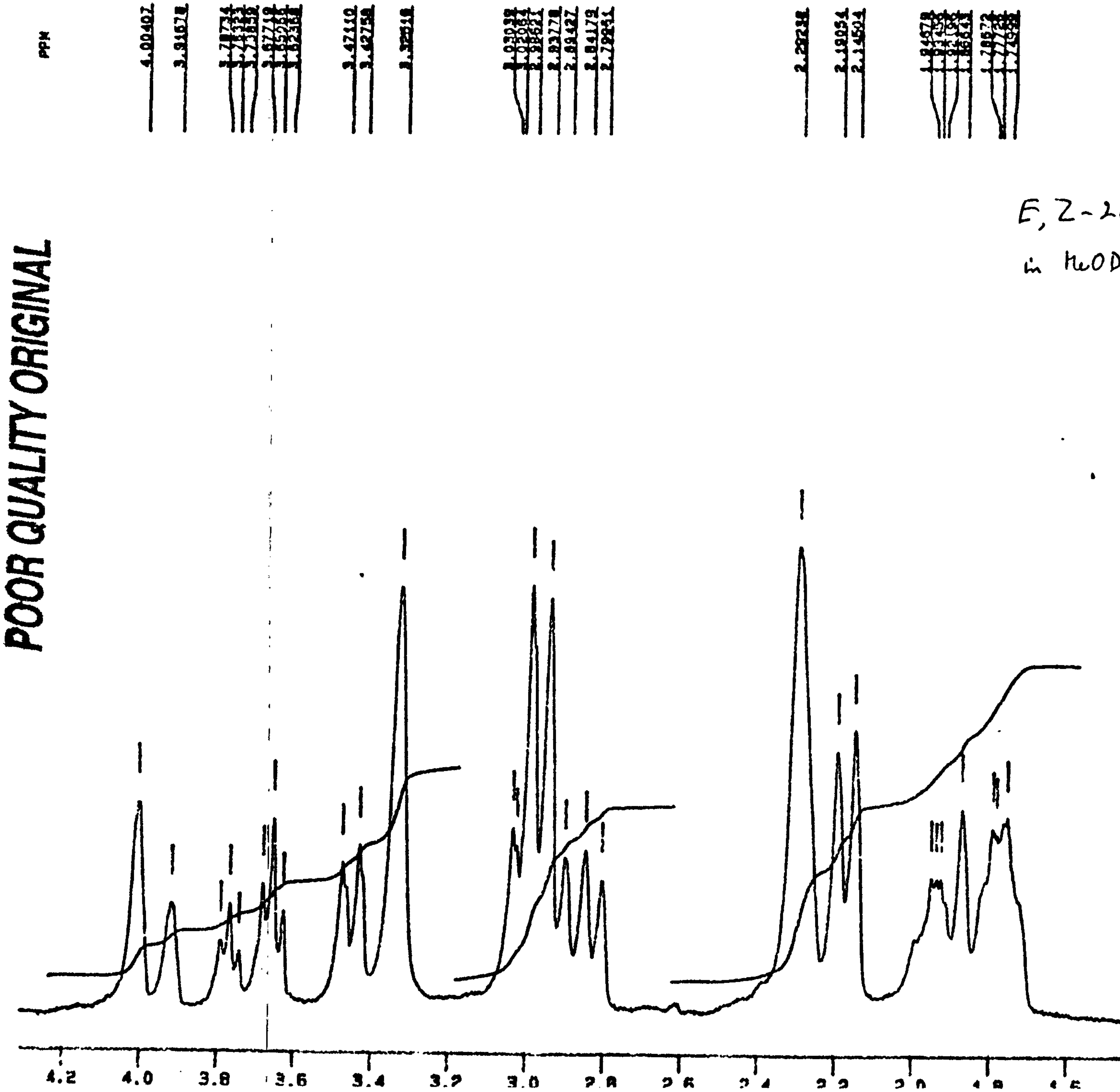
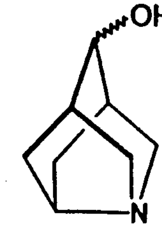


E, Z-24

in MeOD-d_4

POOR QUALITY ORIGINAL





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E, Z-24
in MeOD-d_4

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TD	16384
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HZ/PT	.365
PW	3.0
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AQ	2.736
RG	160
NS	208
TE	297
FW	3000
O2	3210.000
DP	63L PO
LB	.100
GB	.100
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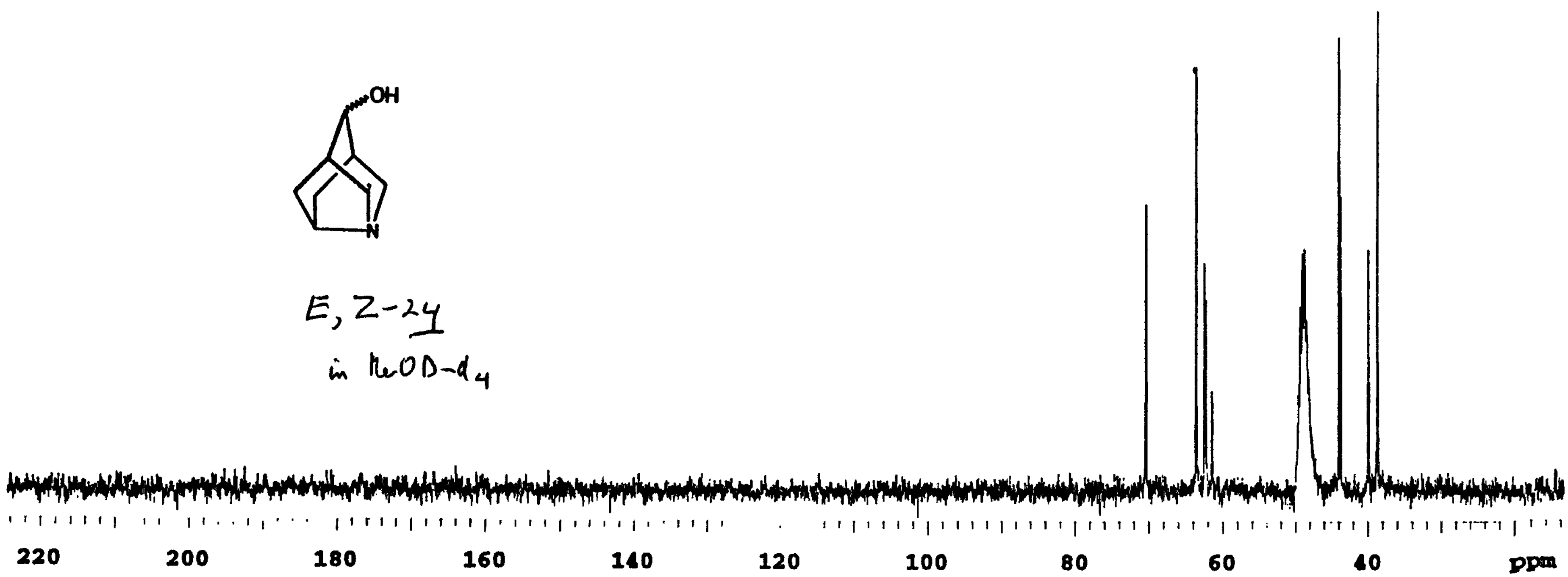
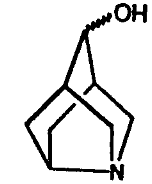
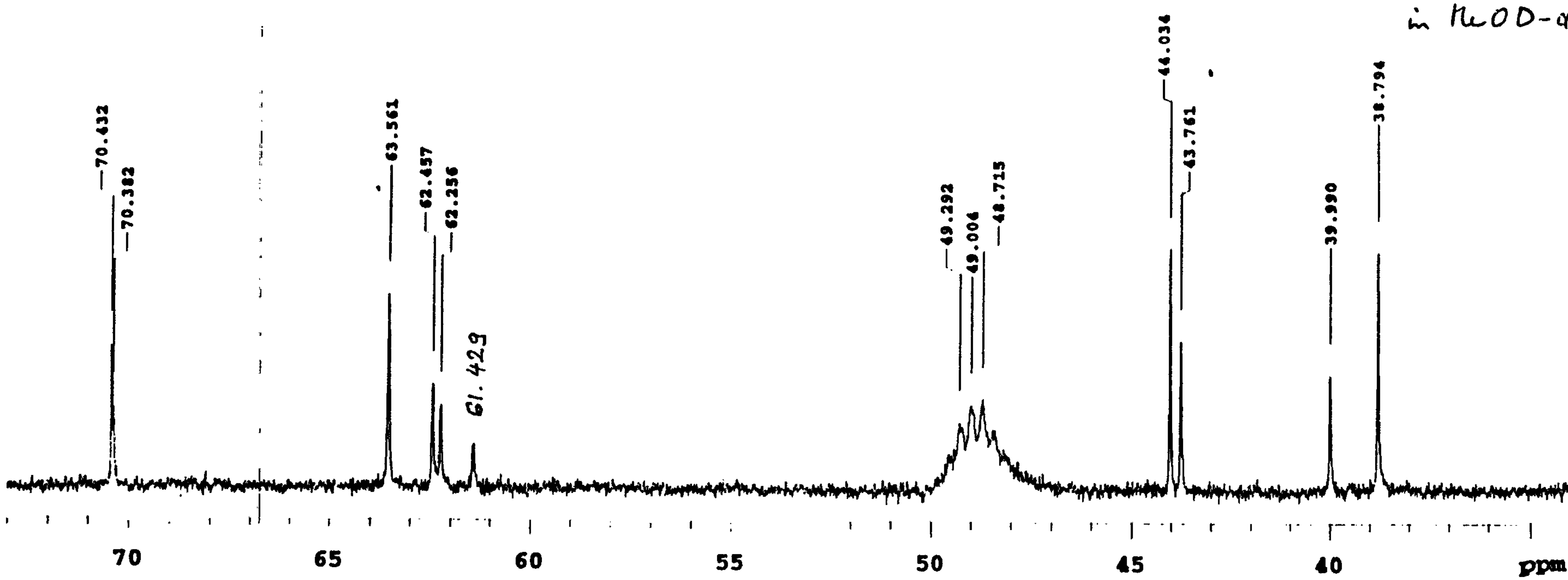


E, Z-24
in MeOD-d_4

102



E,Z-24
in MeOD-d_4



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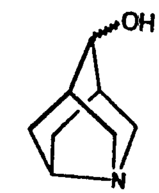
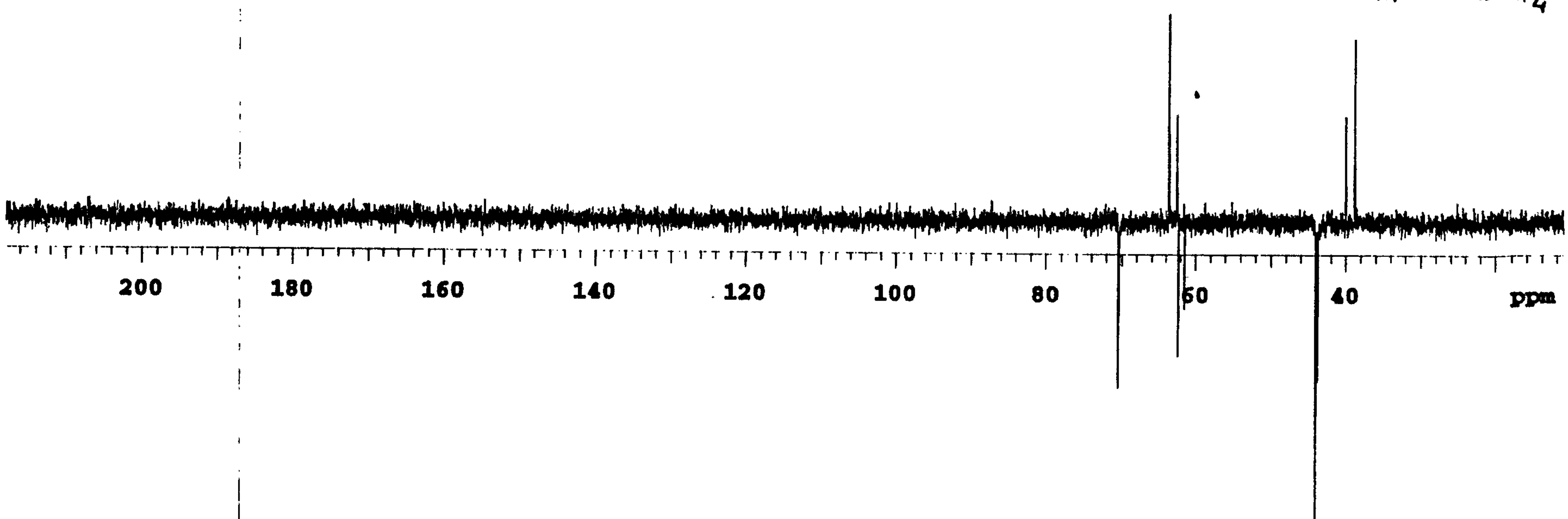
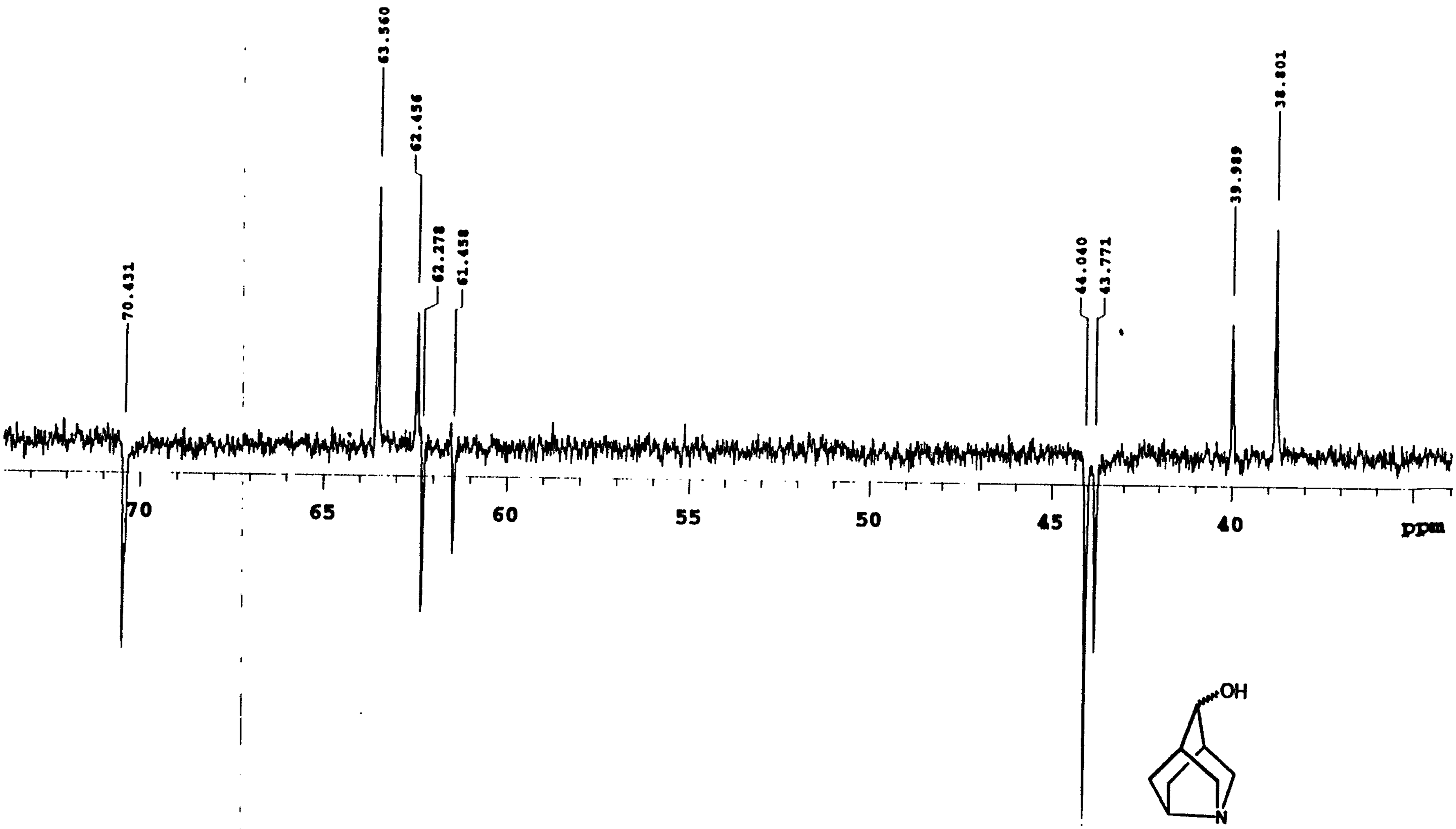


E, Z-24

in MeOD-d_4





E, Z-24
in MeOD-d_4

STANDARD 1H OBSERVE

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file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.070	dm	nnn
tn	H1	dmm	c
at	3.747	dmf	200
np	33728	PROCESSING	
sw	4500.5	wtfile	
fb	2600	proc	ft
bs	16	fn	not used
tpwr	49		
pw	2.0	werr	
d1	0	wexp	
tof	0	wbs	
nt	32	wnt	
ct	32		
alock	s		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-800.3		
wp	4500.5		
vs	9317		
sc	0		
wc	250		
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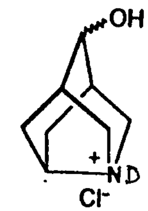
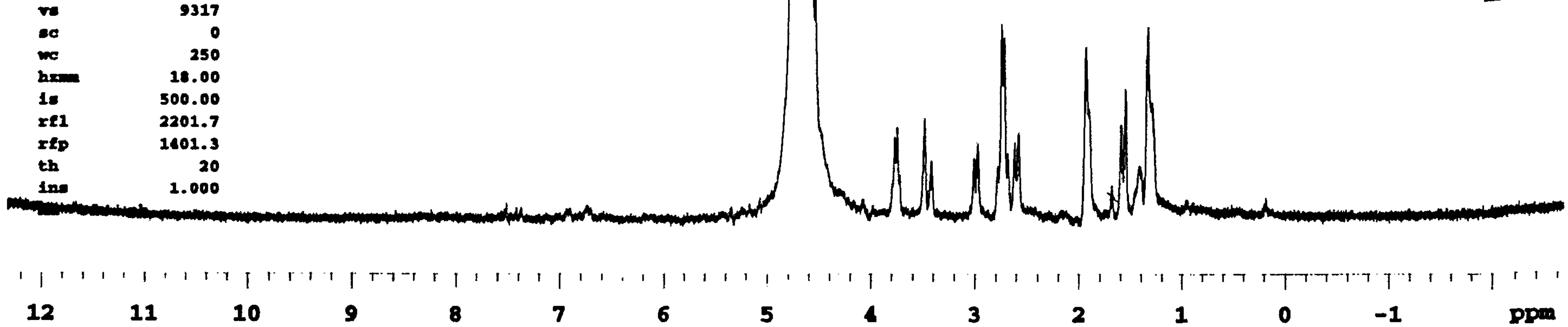
is	500.00		
rfl	2201.7		
rfp	1401.3		
th	20		
ins	1.000		

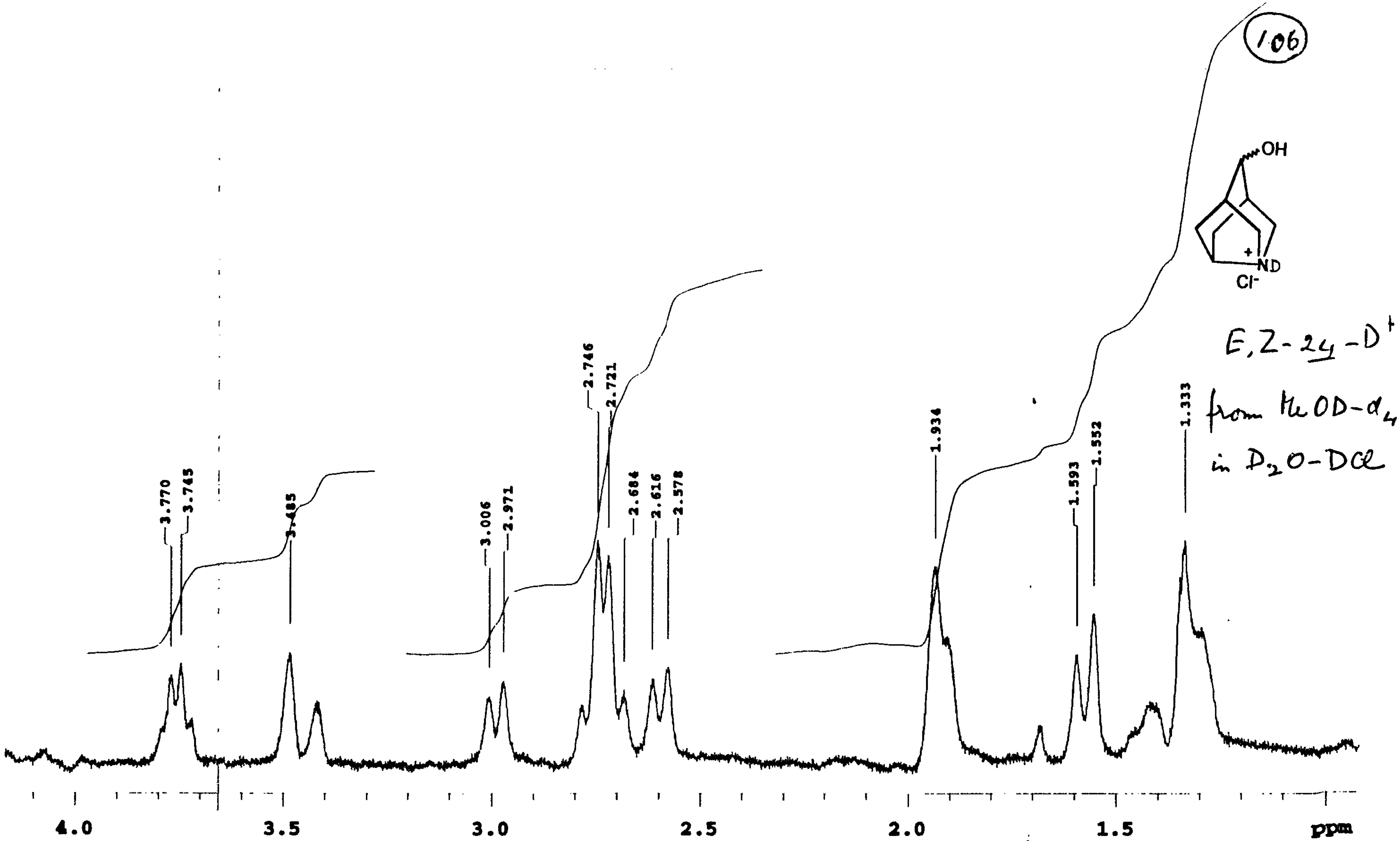
105



E,Z-24 D⁺

from MeOD-d_4 - in D_2O-DCl



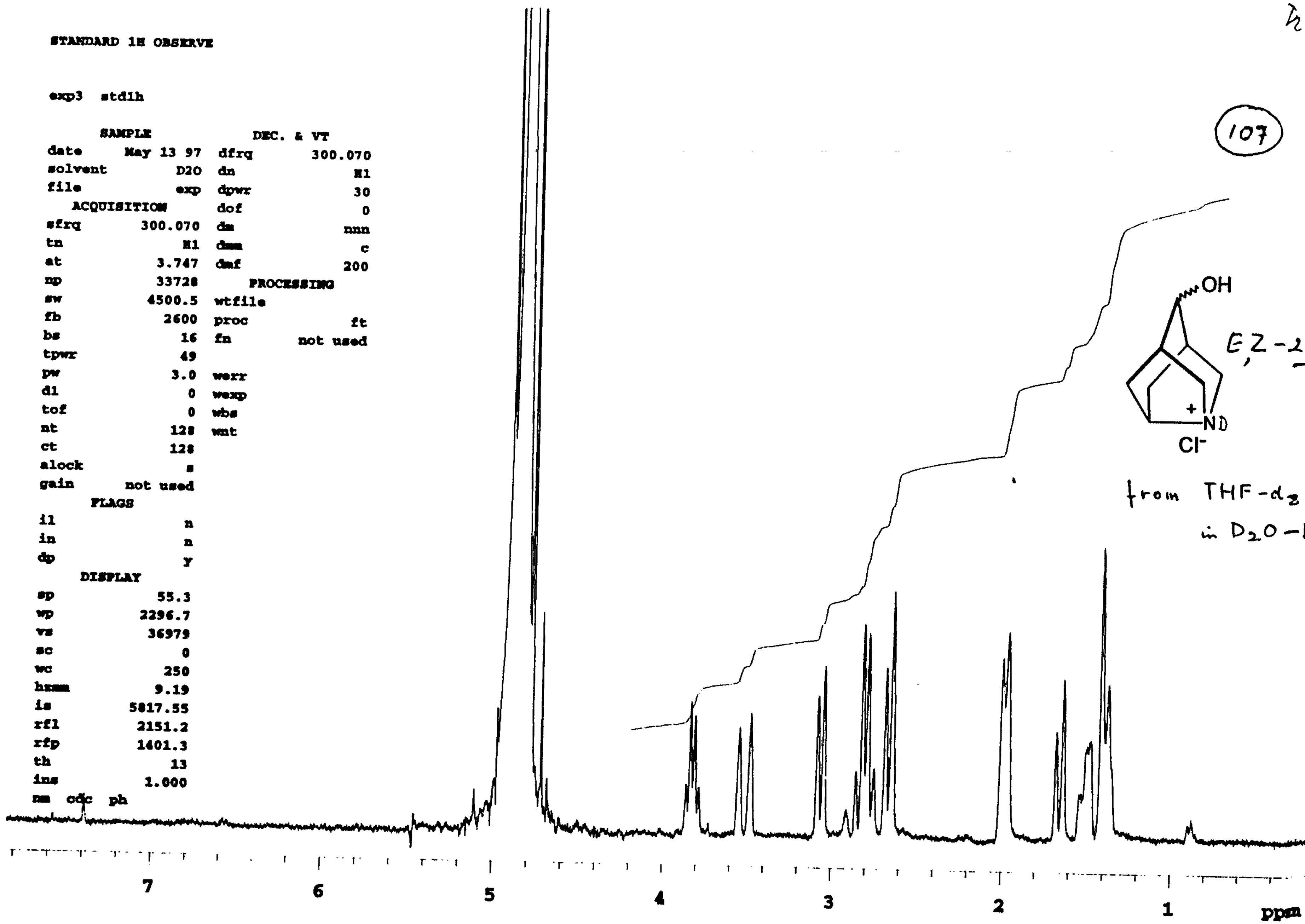

106
OH
ND
Cl⁻
E,Z-24-D+
from MeOD-d4
in D2O-DCl
3.770
3.745
3.485
3.006
2.971
2.746
2.721
2.684
2.616
2.578
1.934
1.593
1.552
1.333
4.0
3.5
3.0
2.5
2.0
1.5
ppm

107

STANDARD 1H OBSERVE

exp3 std1h

SAMPLE		DEC. & VT	
date	May 13 97	dfrq	300.070
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.070	dm	nnn
tn	H1	dmm	c
at	3.747	dmf	200
np	33728	PROCESSING	
sw	4500.5	wtfile	
fb	2600	proc	ft
bs	16	fn	not used
tpwr	49		
pw	3.0	werr	
d1	0	wexp	
tof	0	wbs	
nt	128	wnt	
ct	128		
alock	s		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	55.3		
wp	2296.7		
vs	36979		
sc	0		
wc	250		
hzmm	9.19		
is	5817.55		
rfl	2151.2		
rfp	1401.3		
th	13		
ins	1.000		
nm cdc ph			



E,Z-24-D+

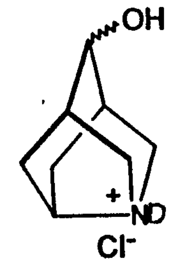
from THF-d8 -
in D2O-DCl

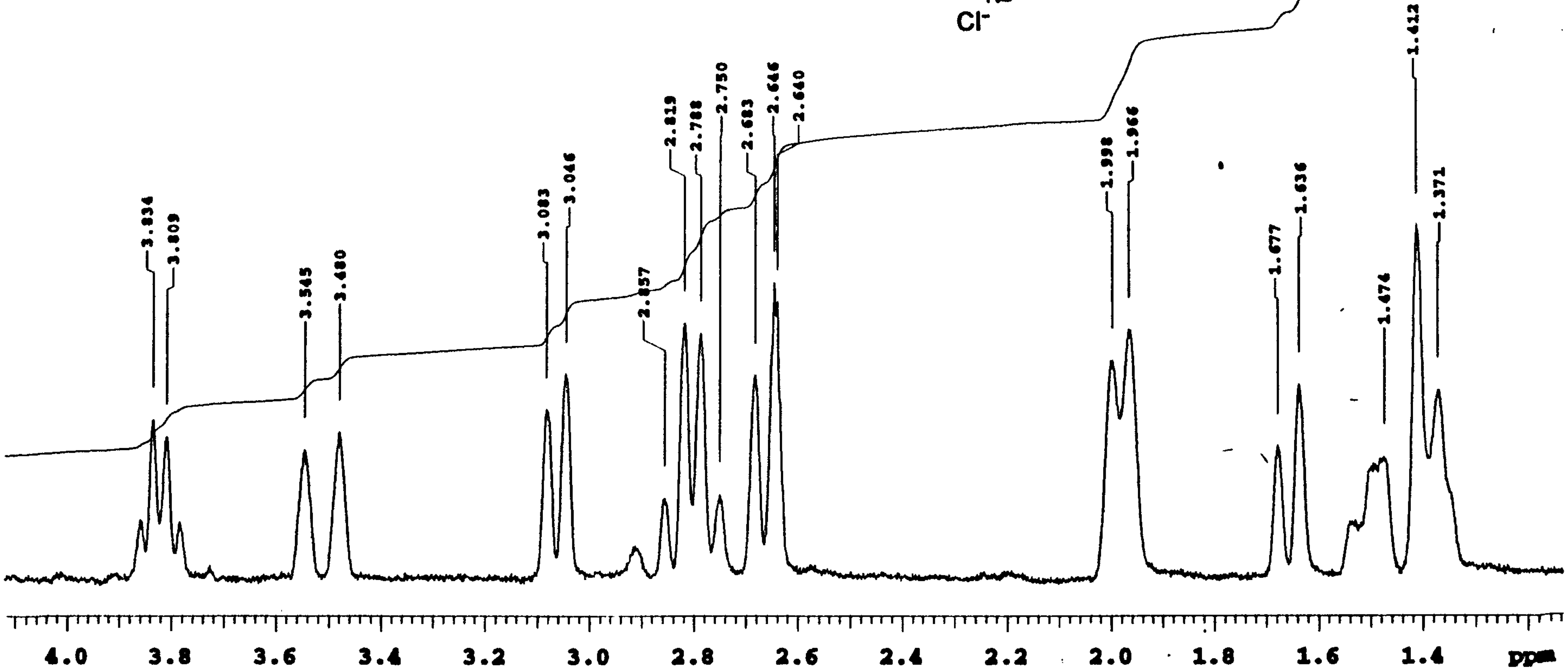
108

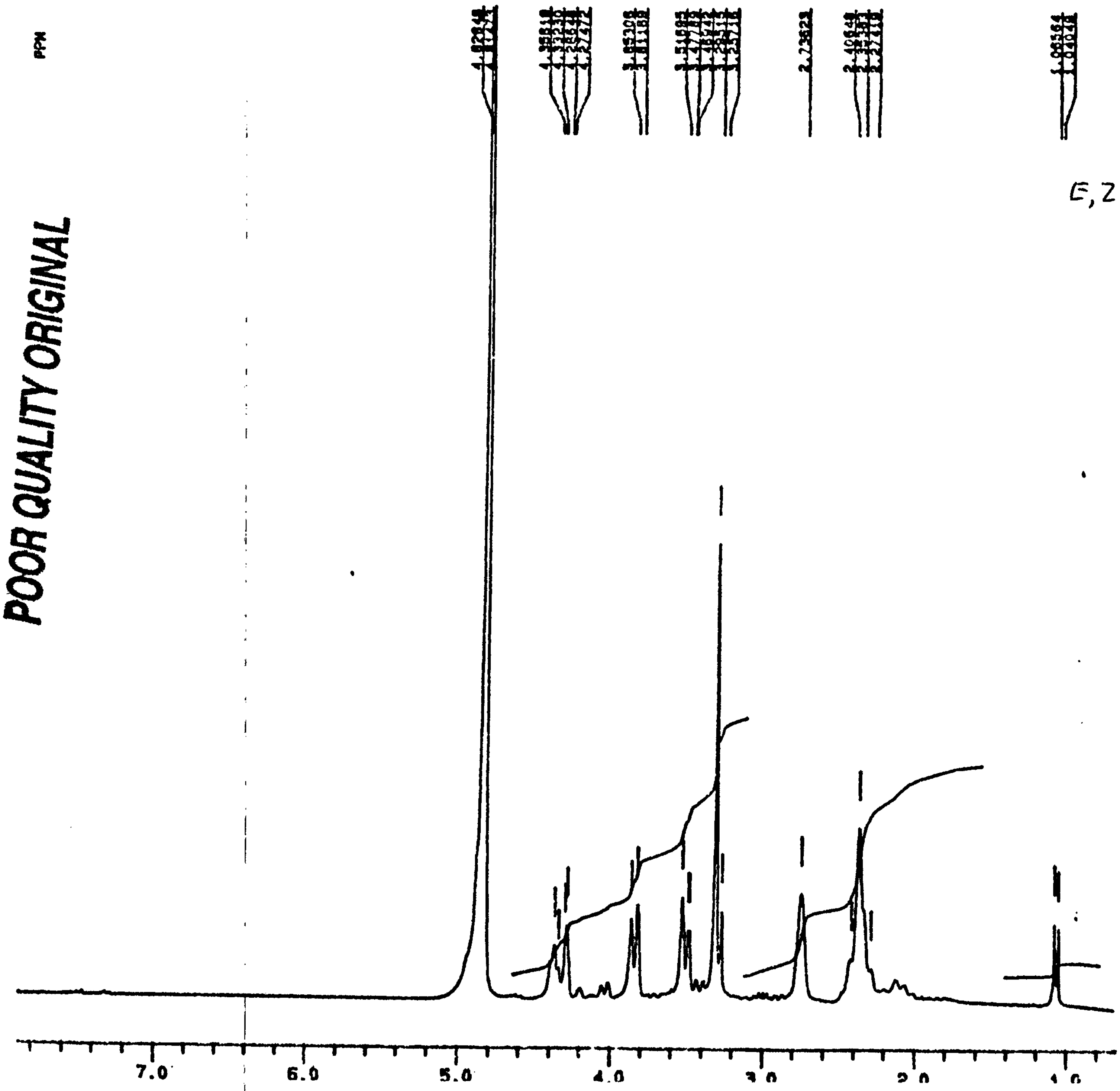
E,Z-24 D^+

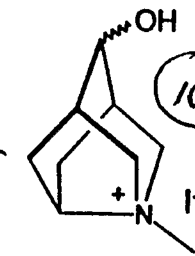
from THF-d_8-

in D_2O-DCl









E,Z-25

109

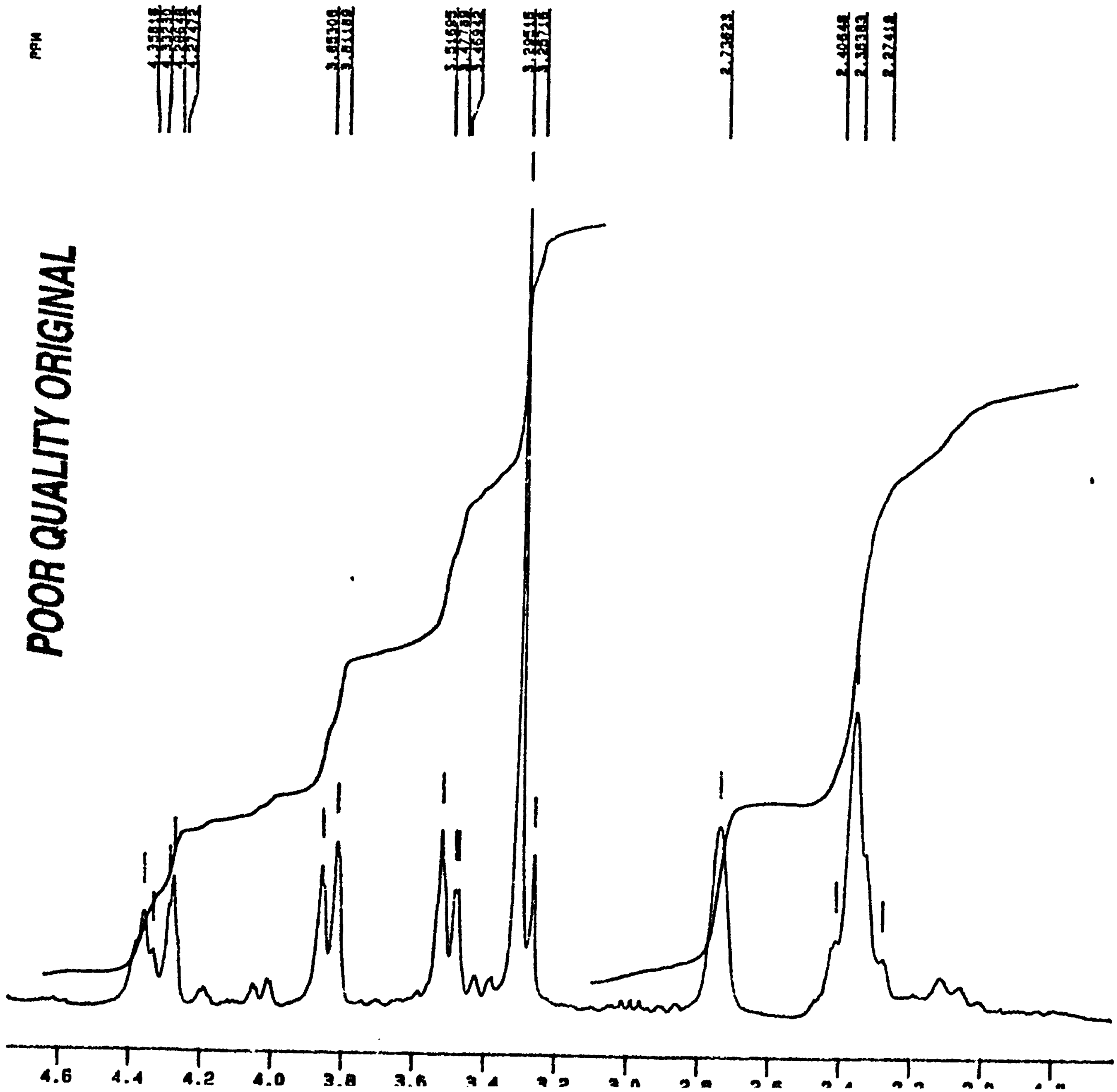
SF 250.133
SY 100.0
O1 4584.030
SI 16384
TD 16384
SW 2994.012
HZ/PT .365

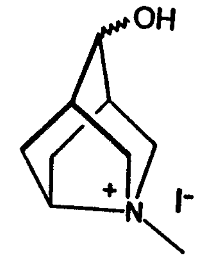
PW 3.0
RD 1.000
AQ 2.736
RG 80
NS 172
TE 297

FW 3000
O2 3210.000
DP 63L PO

LB .100
GB .100
CX 20.00
CY 8.00
F1 7.879P
F2 .680P
HZ/CM 90.036
PPM/CM .360
SR 3458.69

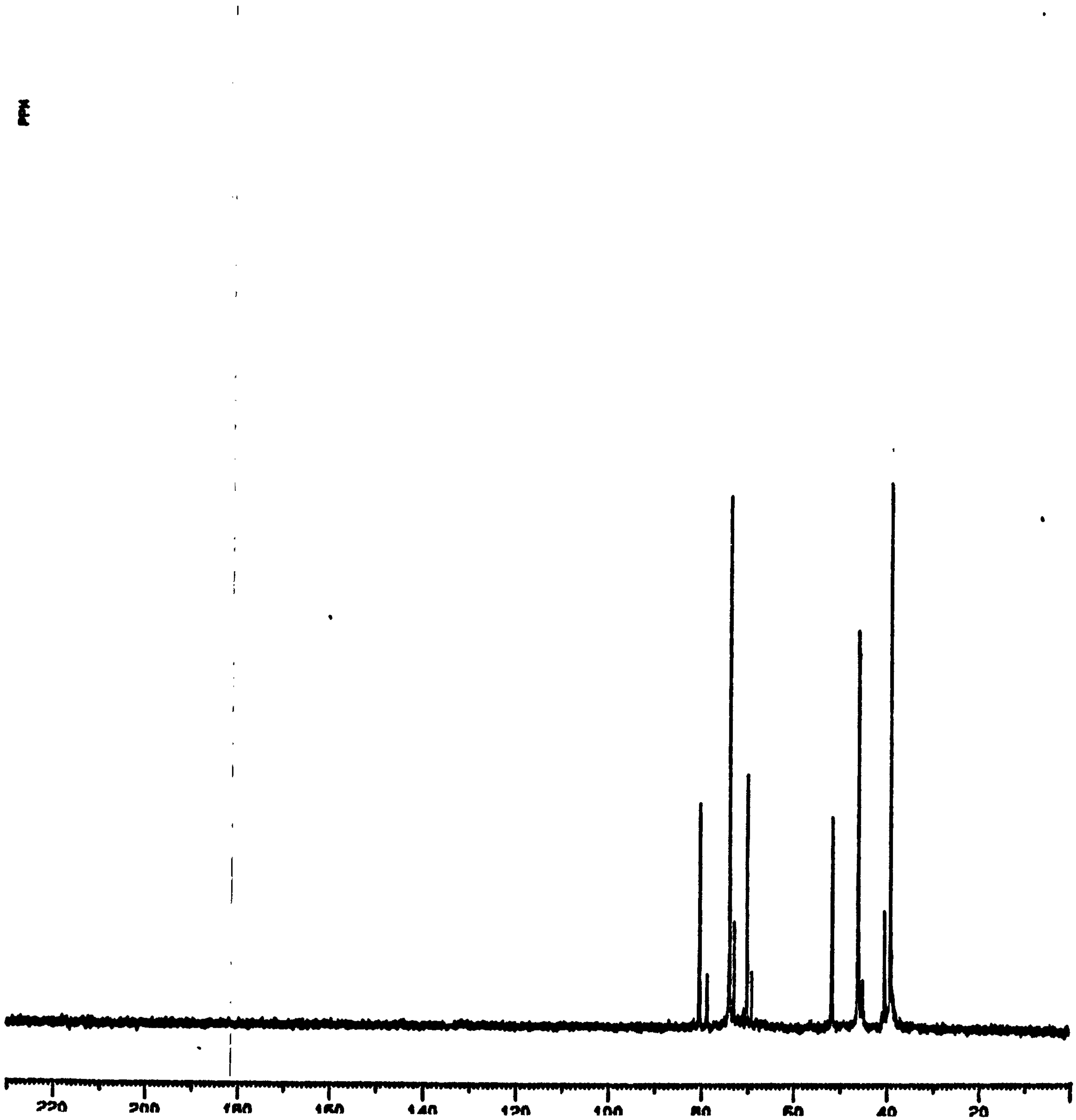
POOR QUALITY ORIGINAL

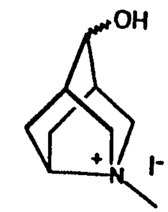




E,Z-25

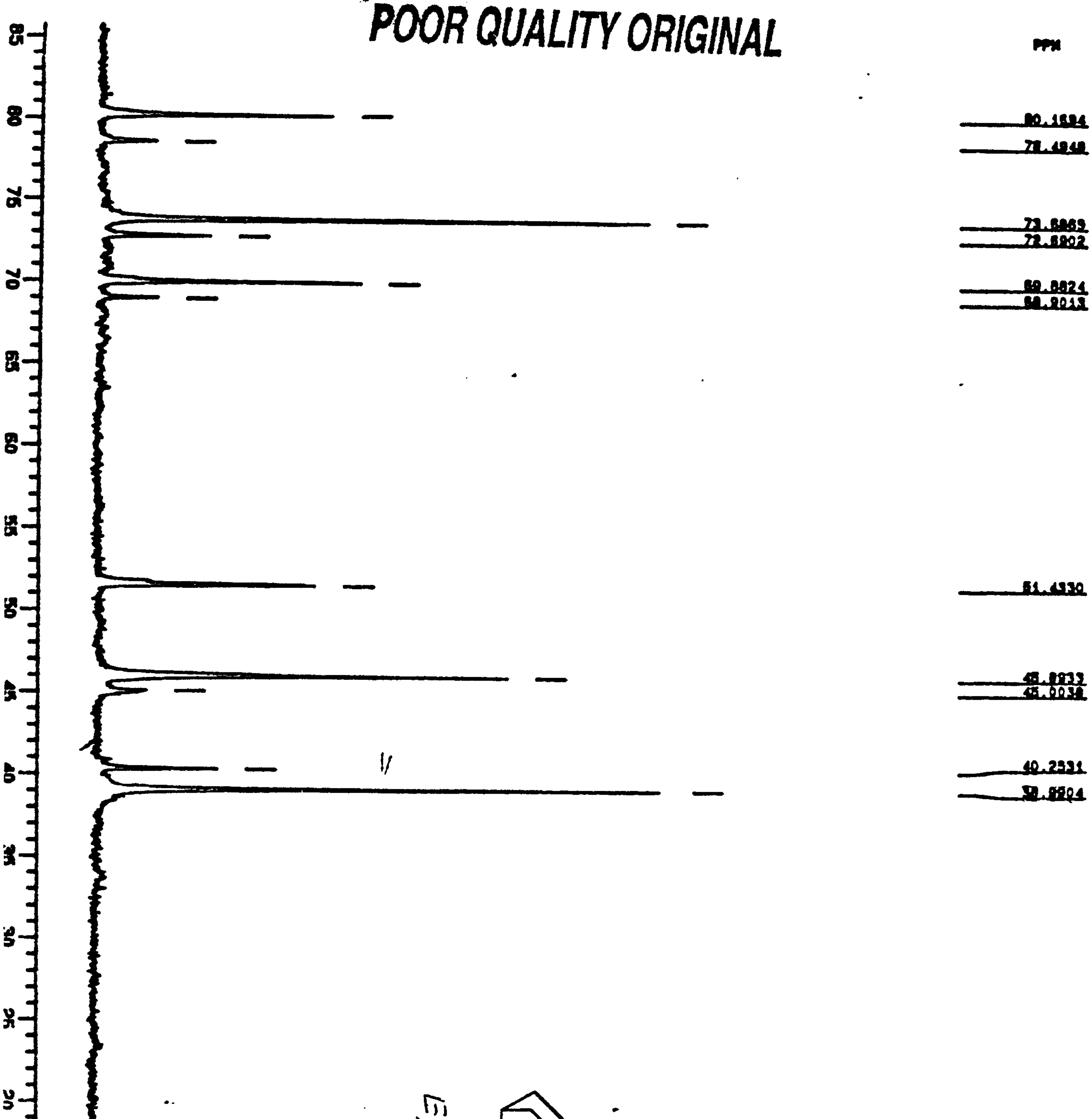
110

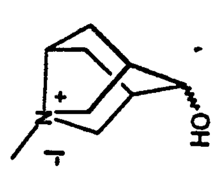




E,Z-25

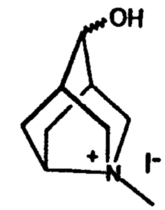
POOR QUALITY ORIGINAL



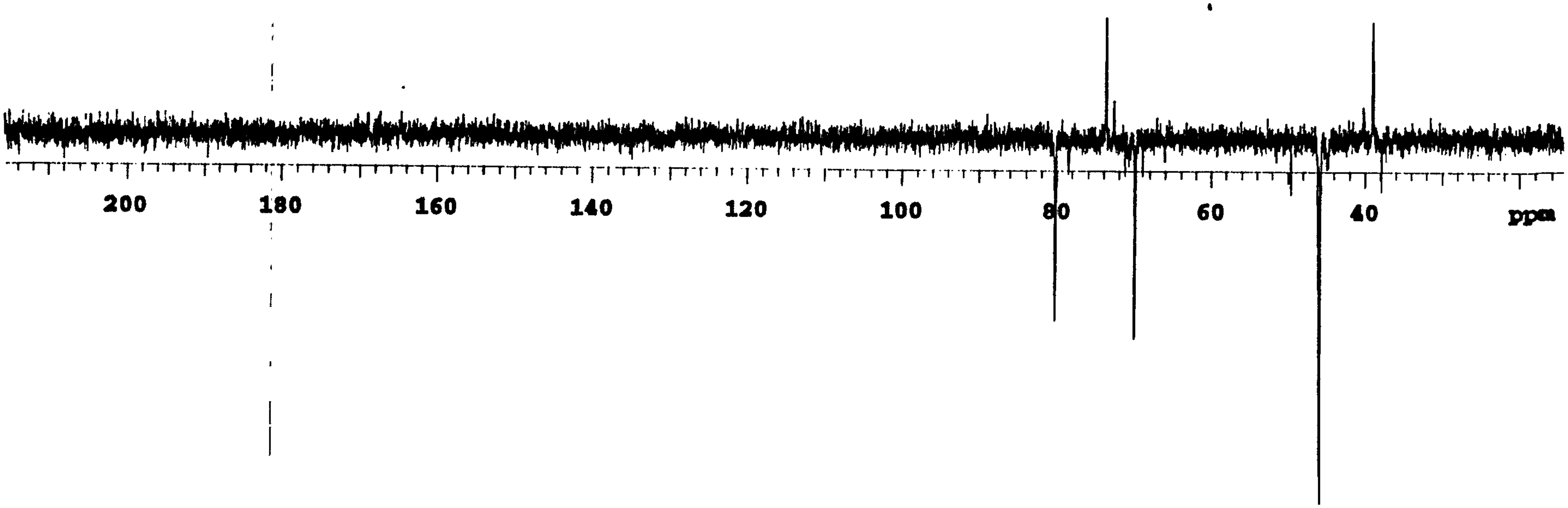


E,Z-25

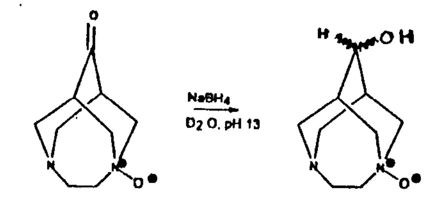
112



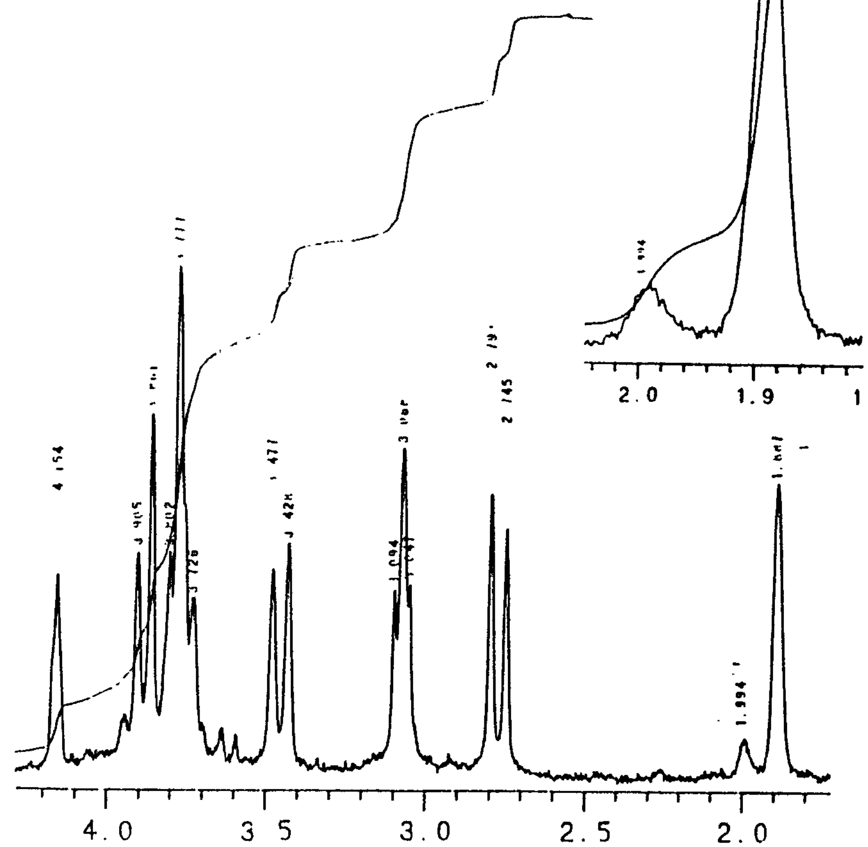
E,Z-25

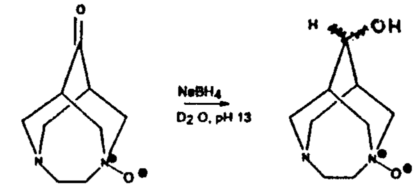


114



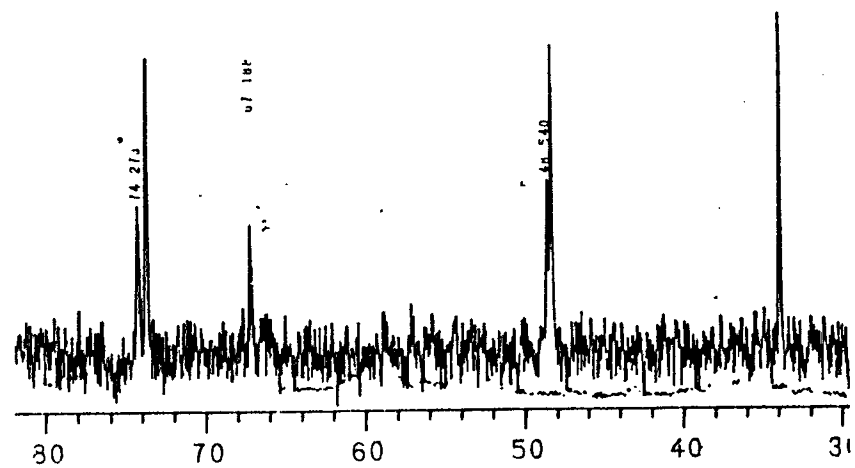
EZ-26



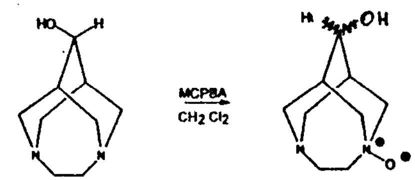


115

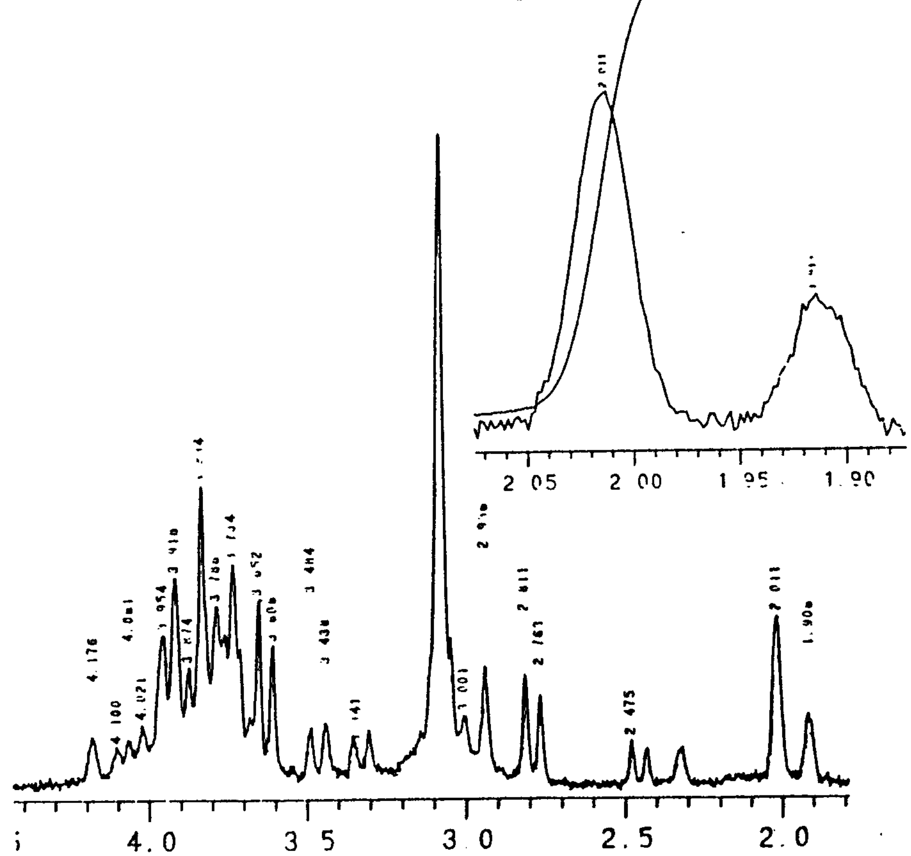
EZ-26



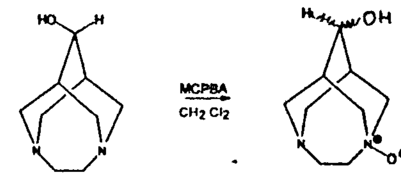
116



E2-26

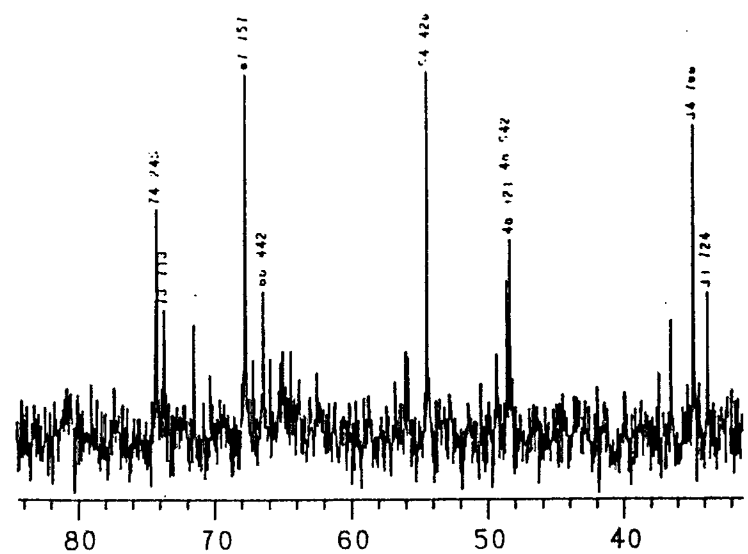


Spectra recorded in D_2O, pH=13



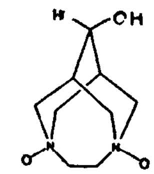
EZ-26

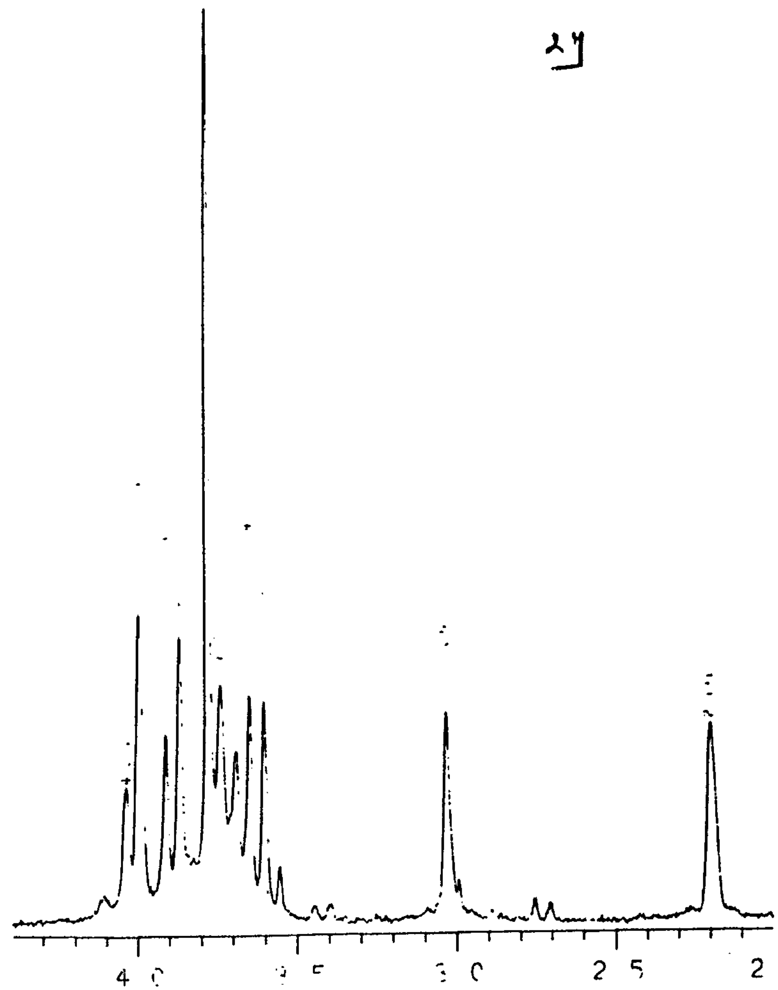
(117)



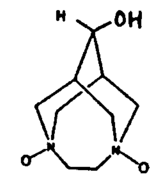
Spectra recorded in D_2O, pH=13

118



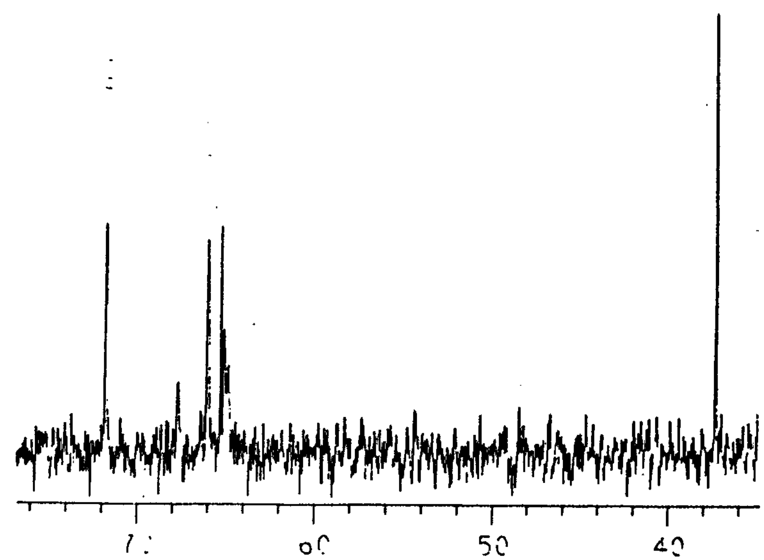


^1H NMR, D_2O, pH 13



27

119



^{13}C NMR, D_2O