

# Cycloaddition of Cyclohexa-2,4-dienones, Ring-closing Metathesis and Photochemical Reactions: A Common Stereoselective Approach to Duprezianane, Polyquinane and Sterpurane Frameworks

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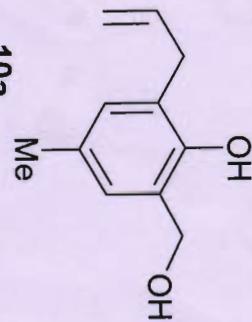
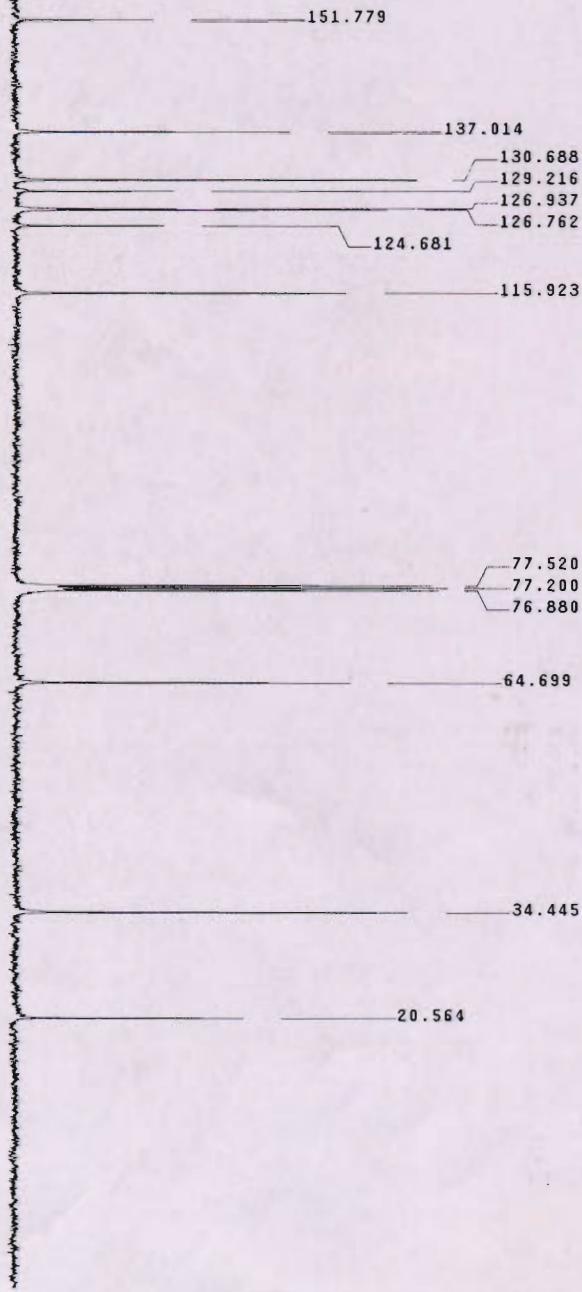
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exp1 s2pu1  
kk-hydrometh

SAMPLE date Feb 9 2004 temp not used  
solvent C0C13 gain not used  
file exp spin not used  
ACQUISITION hst 0.008  
sw 25125.6 pw90 14.000  
at 1.199 alfa 20.000  
np 60270 i1  
fb 13800 i1  
bs 4 in  
di 2.000 dp  
nt 2400 hs  
ct 388 n  
  
TRANSMITTER tn ib 3.50  
sfrq C13 fn not used  
tor 1558.7 sp DISPLAY -1506.1  
tpwr 56 wp 23125.6  
pw 7.000 rf1 9272.1  
  
DECOPPLER dn rfp 7766.0  
dof 0 rp 106.3  
dm 1p -342.2  
dmm yyy w PLOT 250  
dpwr 41 sc 0  
dmf 11900 vs 146  
ai th 5  
no ph



S2

sp-ep

expt s2pu1

SAMPLE 9 2004 temp not used

date Feb 9 2004

solvent CDCl<sub>3</sub>

file /export/home/~/spin

rpvnmr/FEB2004/9~

pw90

FEB/sp-ep-c13.t1d

ACQUISITION a1fa

sw 25125.6

at 1.199

np 60270

fb 13800

bs 4

di 2.000

nt 2400

ct 784

TRANSMITTER

tn C13

sfrq 100.607

tof 1558.7

tpwr 56

pw 7.000

DECOUPLER

dn H1

dot 0

dm 0

dmm 0

dpwr 41

dmrf 11900

SPECIAL

temp not used

gain not used

spin not used

0.008

Hst 14.000

a1fa 20.000

FLAGS

t1 n

fn n

dp y

hs nn

PROCESSING

3.50

nt lb

ct not used

TRANSMITTER

tn C13

sfrq 100.607

tof 1558.7

tpwr 56

pw 7.000

DECOUPLER

dn H1

dot 0

dm 0

dmm 0

dpwr 41

dmrf 11900

DISPLAY

tn -1504.6

sfrq 23125.6

tof 9270.6

tpwr 7766.0

pw 101.9

DECOUPLER

tn 1p

sfrq -342.2

tof PLOT

tpwr 250

dn 0

dot wc

dm 0

dmm vs

dpwr 273

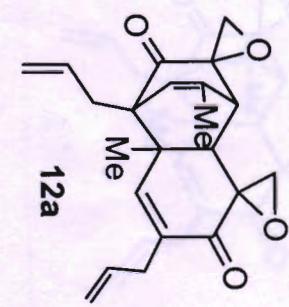
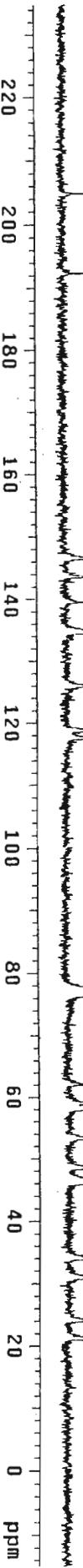
dmrf 5

nt ai

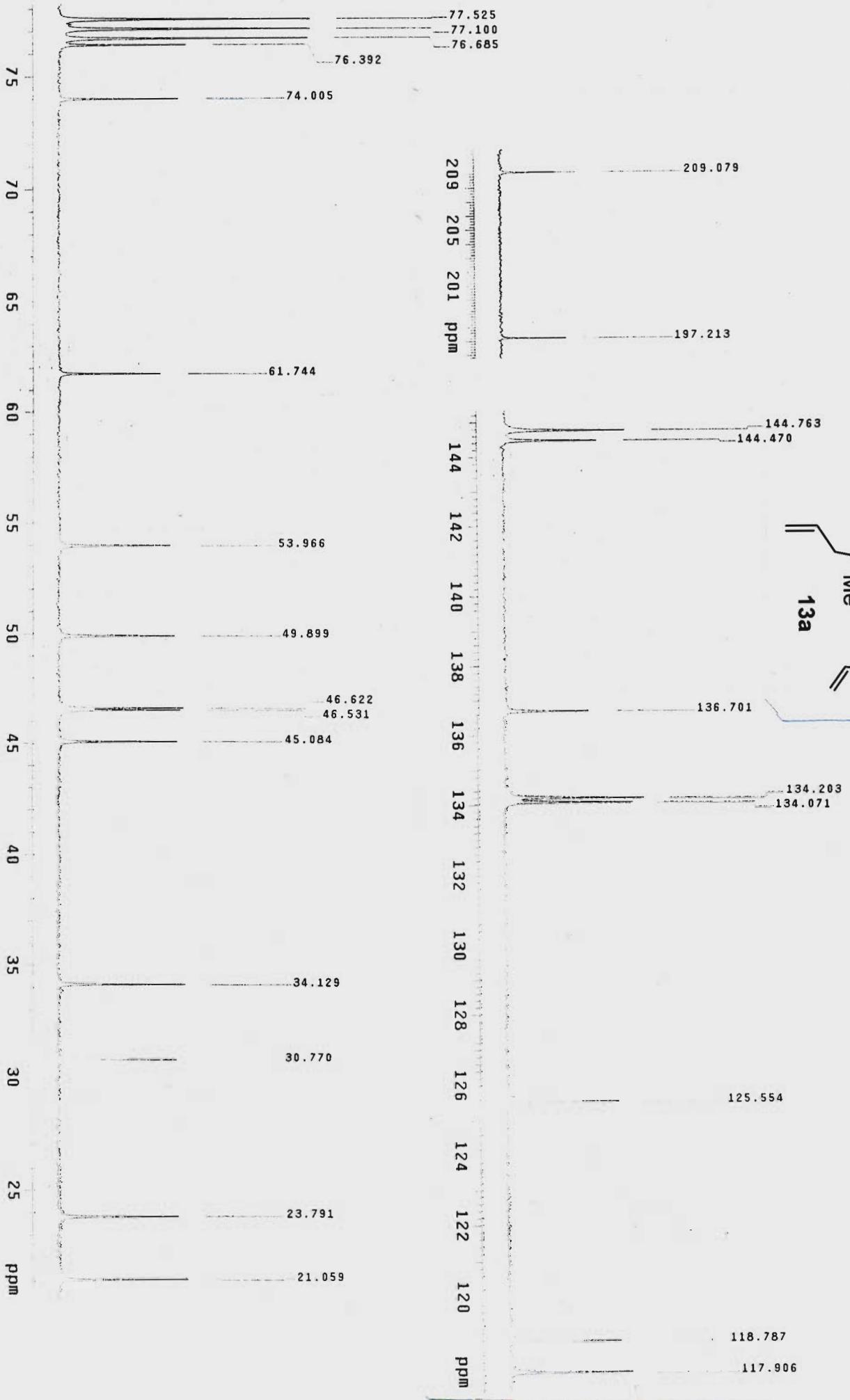
ct no

TRANSMITTER

tn ph



S3



C1-OH-add  
ex<sub>p1</sub> s2pul

SAMPLE

date Oct 25 2004 dn

solvent CDCl<sub>3</sub> dof

file /export/home/~

vnmr1/PROTON/MADHU~ dmm

/25Oct/C1-OH-add.f~ dmrf

id

DEC. & VT

H1 0

nnn

c

200

ACQUISITION 0.10 lb  
sfrq 299.949 fn not used

tn

2.002

werr

np 23980

wexp

sw 5988.0

wbs

fb not used

wnr

bs 2

sp

-55.2

pw 3.0

wp

3057.6

tpwr 60

vs

836

d1 0

sc

0

tof 0.0

wc

250

nt 1600

hzmn

12.23

ct 9082.47

is

9082.47

alock n

rfl

1540.1

gain 6

rrf

0

FLAGS th 1

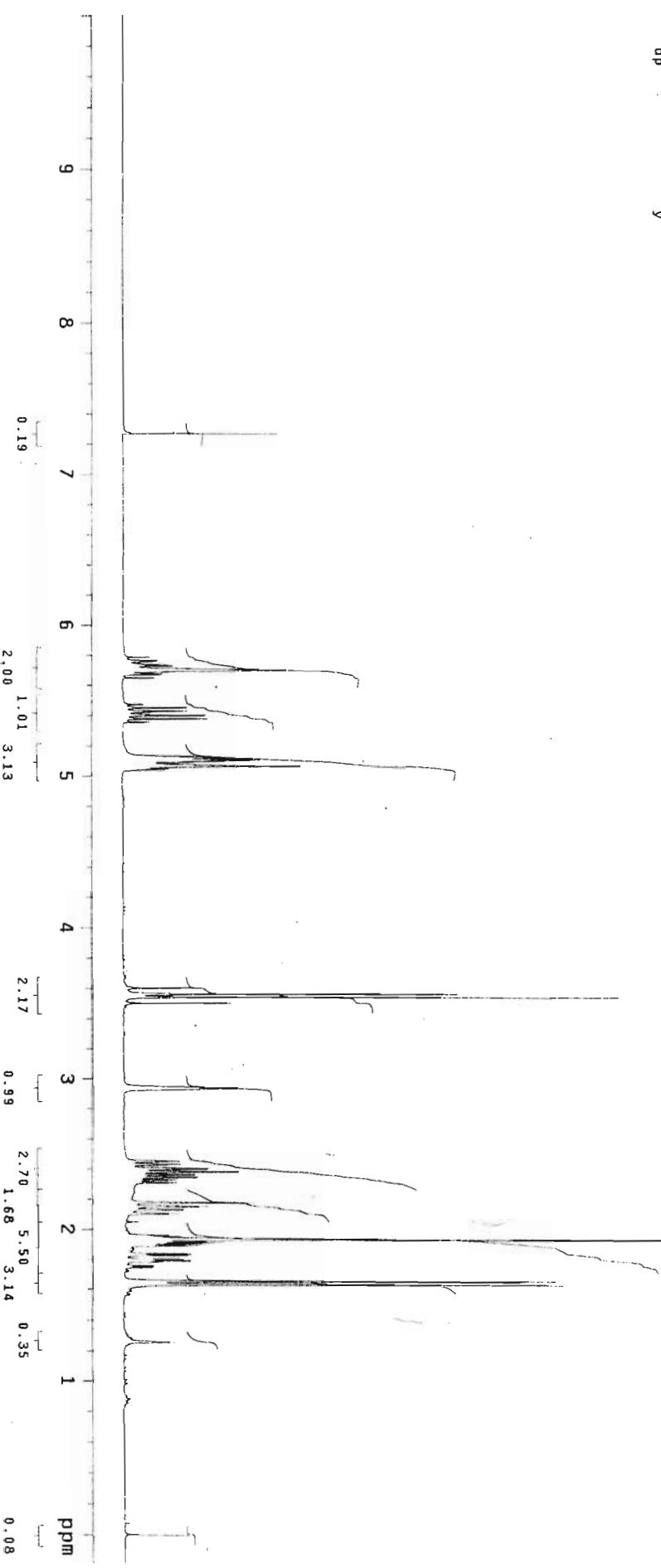
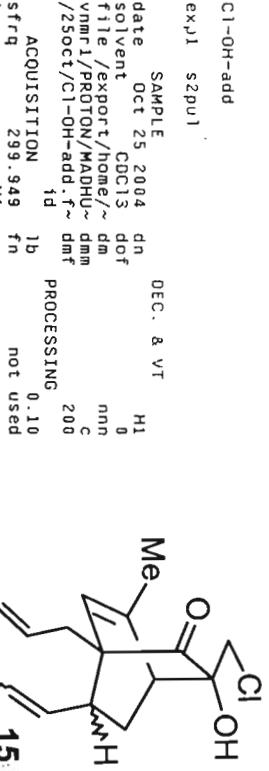
n ins 2.000

in ai

dp ph

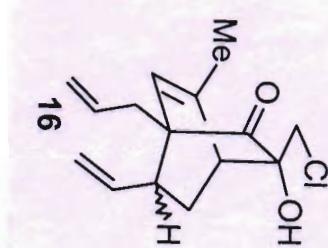
y 2.000

S5

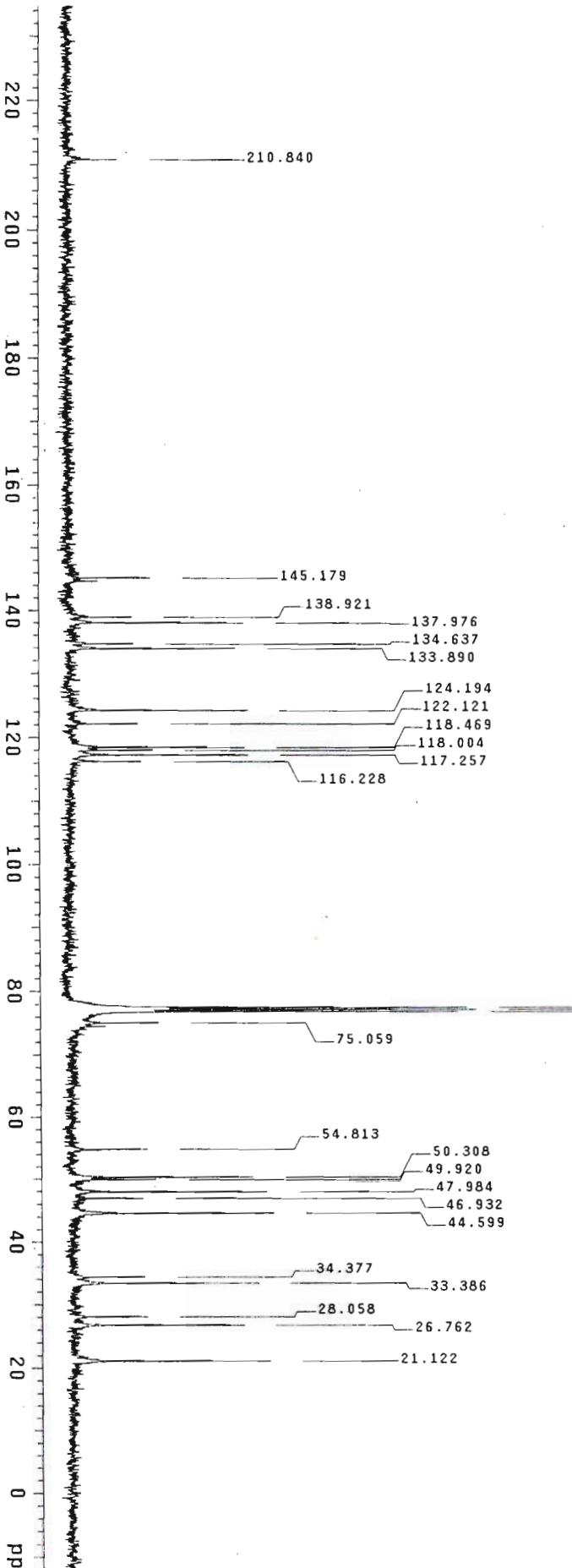


subadd-c13  
exp4 s2ppul

SAMPLE DATE AUG 25 2003 temp not used  
solvent CDCl<sub>3</sub> gain not used  
file /export/home/~spin not used  
guest/August/25-Aug-008  
g/subadd-c13.fid hst  
ACQUISITION pw0 14.000  
SW 25125.6 alfa 20.000  
at 1.199 i1 n  
np 6.079 i1 n  
fb 13800 dp y  
bs 4 hs nn  
d1 2.000 PROCESSING 3.50  
nt 2400 1b fn not used  
ct 924 f1 DISPLAY -1508.3  
TRANSMITTER C13 sp 25125.6  
tn 100.607 wp 1508.3  
sfrq 1558.7 rfi 0  
tof 56 rfp 0  
tpwr pw 7.000 rp 70.9  
DECOUPLER H1 1p -303.8  
dn 0 wc 250  
dof 0 sc 0  
dm 41 vs 159  
dmm 11900 th 6  
dpwr dm<sup>f</sup> ai no  
pw ph



S6

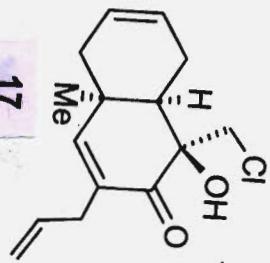


KK-coope

exp2 s2pul

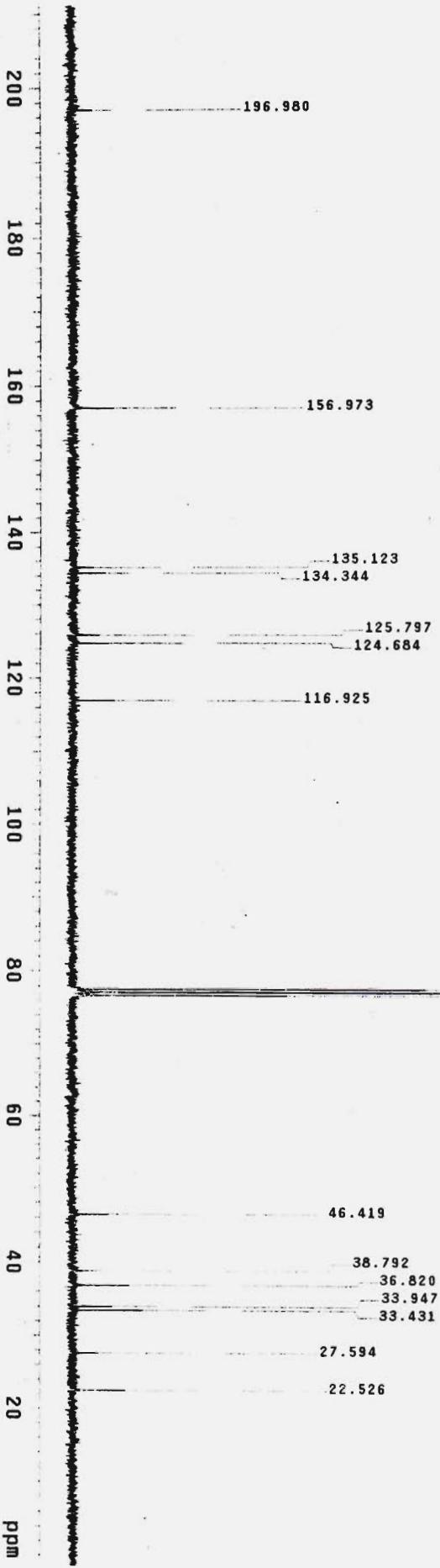
SAMPLE 10 2004 temp not used  
date Feb 10 2004 gain not used  
solvent CDCl<sub>3</sub> spin not used  
file /export/home/~ file not used  
vnmr1/PROTON/0Feb~ bst 9.008  
/KK\_COPE-C13 fid pw90 9.500  
ACQUISITION aifia 20.000

sw 25000.0 FLAGS  
at 1.280 i1 n  
np 64000 in n  
fb 13800 dp y  
bs 4 hs nn  
d1 3.000 1b 1.00  
nt 3200 fn not used  
ct 368 fn not used  
**TRANSMITTER** C13 SP -109.2  
tn 75.430 wd 16030.9  
sfreq 748.9 RT 10031.1  
t0rf 59 RF 5815.0  
tpwr 4.750 TP -151.3  
pw 10 PLOT -301.8  
**DECOPPLER** H1 0 250  
dn 0 WC 0  
dor 0 SC 61  
dm vyy 6  
dmm w vs  
dpwr 39 TH ph  
dmtf 10900 mm



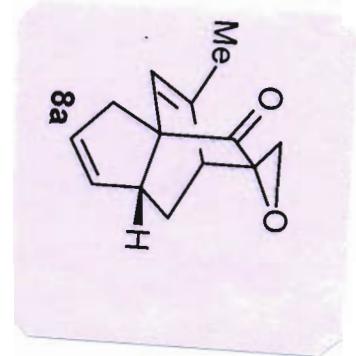
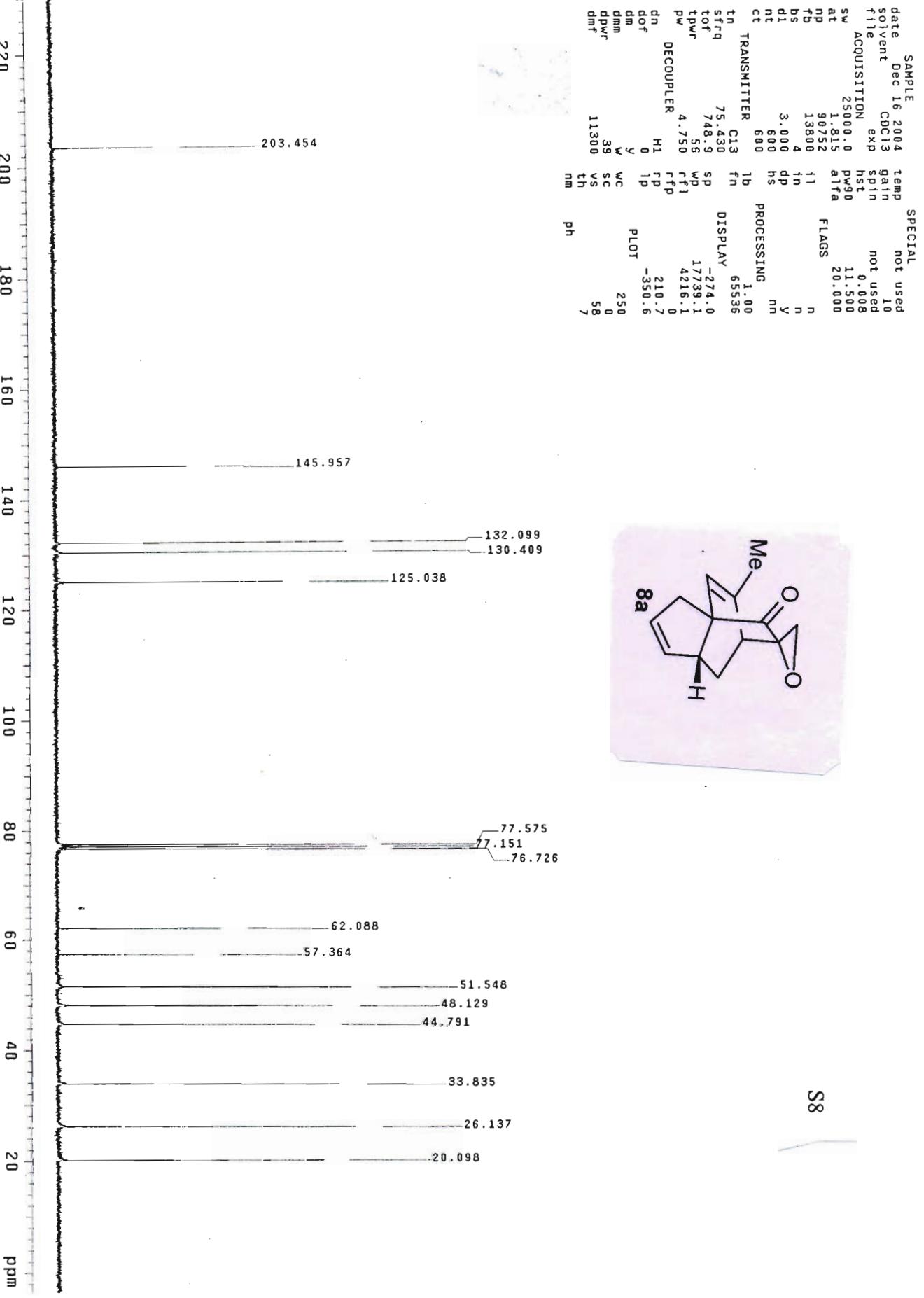
77.515  
77.161  
77.090  
76.675

S7



exp3

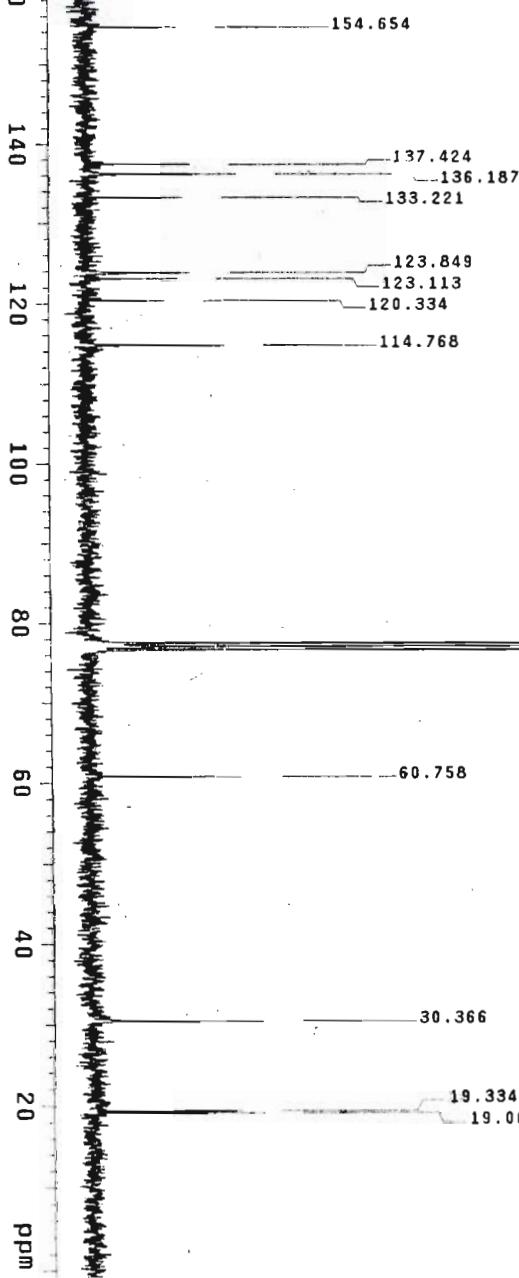
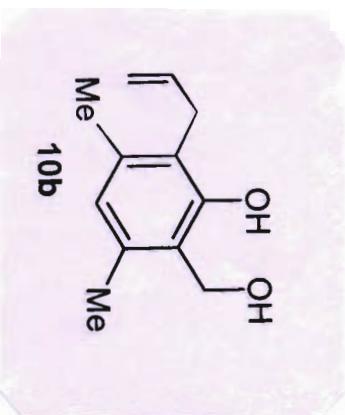
s2pu1



S8

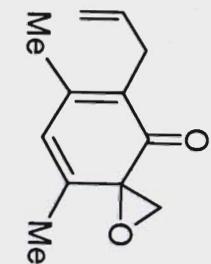
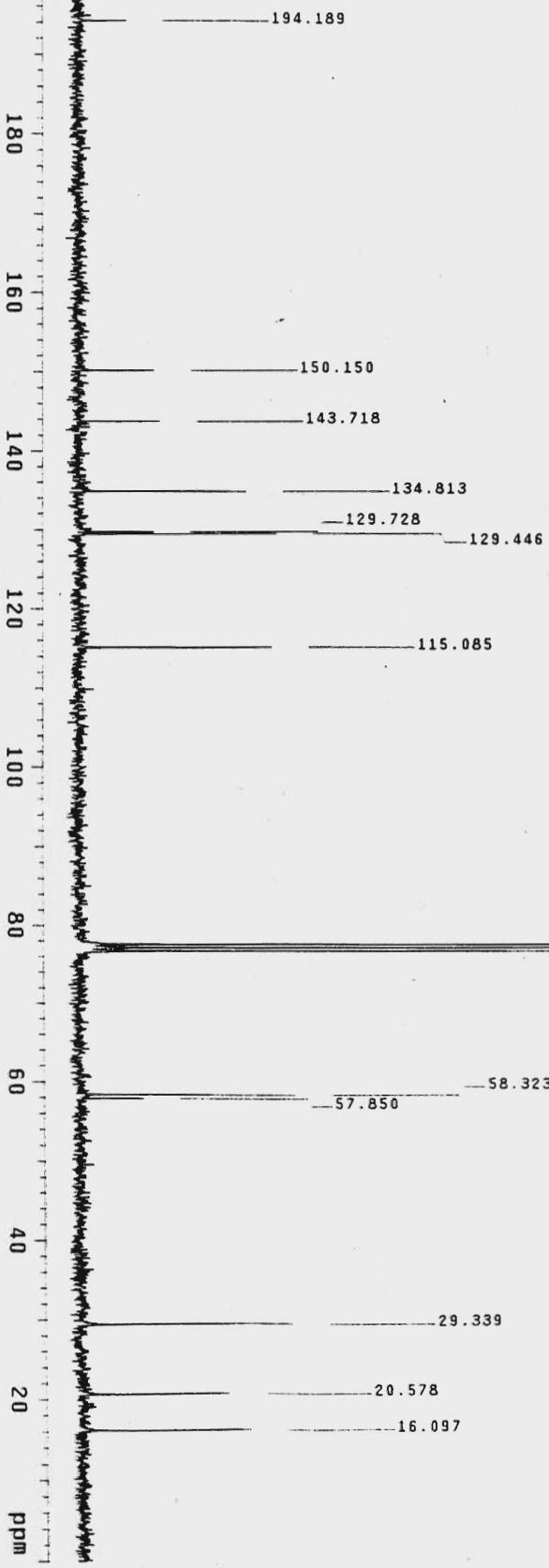
exp3 s2pu1

SAMPLE	Dec 22 2003	temp	SPECIAL
solvent	CDCl <sub>3</sub>	gain	not used
file	exp	spin	not used
ACQUISITION		hst	20
sw	18867.9	pwg0	0.008
at	1.815	alfa	9.500
np	68492	FLACS	20.000
fb	10400		
bs	4	n	
d1	3.000	n	
nt	1600	dp	
ct	432	hs	
tn	C13	1b	PROCESSING
sfrq	75.430	fn	2.00
tof	748.9	DISPLAY	not used
tpwr	59	sp	-65.1
pw	4.750	wp	1523.1
DECOUPLER		r <sub>f1</sub>	8965.2
dn	H1	r <sub>f2</sub>	5815.0
dof	1	r <sub>p</sub>	-157.7
dm	0	1p	-268.0
dmm	yvv	PLOT	77.520
dpwr	39	wc	77.096
dmt	10900	sc	76.672
		vs	
		th	
		nm	
		ph	



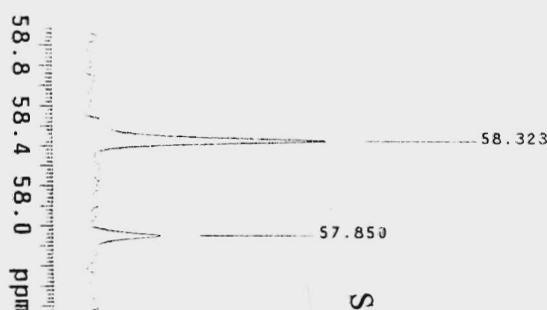
S9

SAMPLE  
date Oct 21 2003 temp  
solvent CDCl<sub>3</sub> gain  
file /export/home/~ spin  
vnmri/PROTON/21Oct~ pw90  
/GDP-Cyhex-C13.fid hst  
ACQUISITION 18867.9 0.008  
sw 18867.9 9.500  
at 1.815 11 alfa  
np 68492 in 20.000  
fb 10400 dp  
bs 4 hs  
di 3.000 PROCESSING nn  
rt 400 1b 2.00  
ct 400 fn not used  
TRANSMITTER C13 DISPLAY  
tn sp -12.1  
strq 75.430 wp 16639.3  
trf 748.9 rfp 6965.5  
tpwr 59 rfp 5815.0  
pw 4.750 rp 193.9  
DECOUPLER H1 -242.4  
dn 0 wc 250  
of 0 sc 90  
dm 0 vs 5  
dpwr 39 th 10900 ph  
dmrf



77.524  
77.100  
76.676

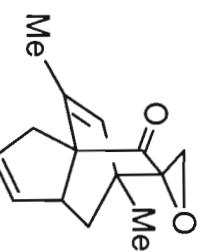
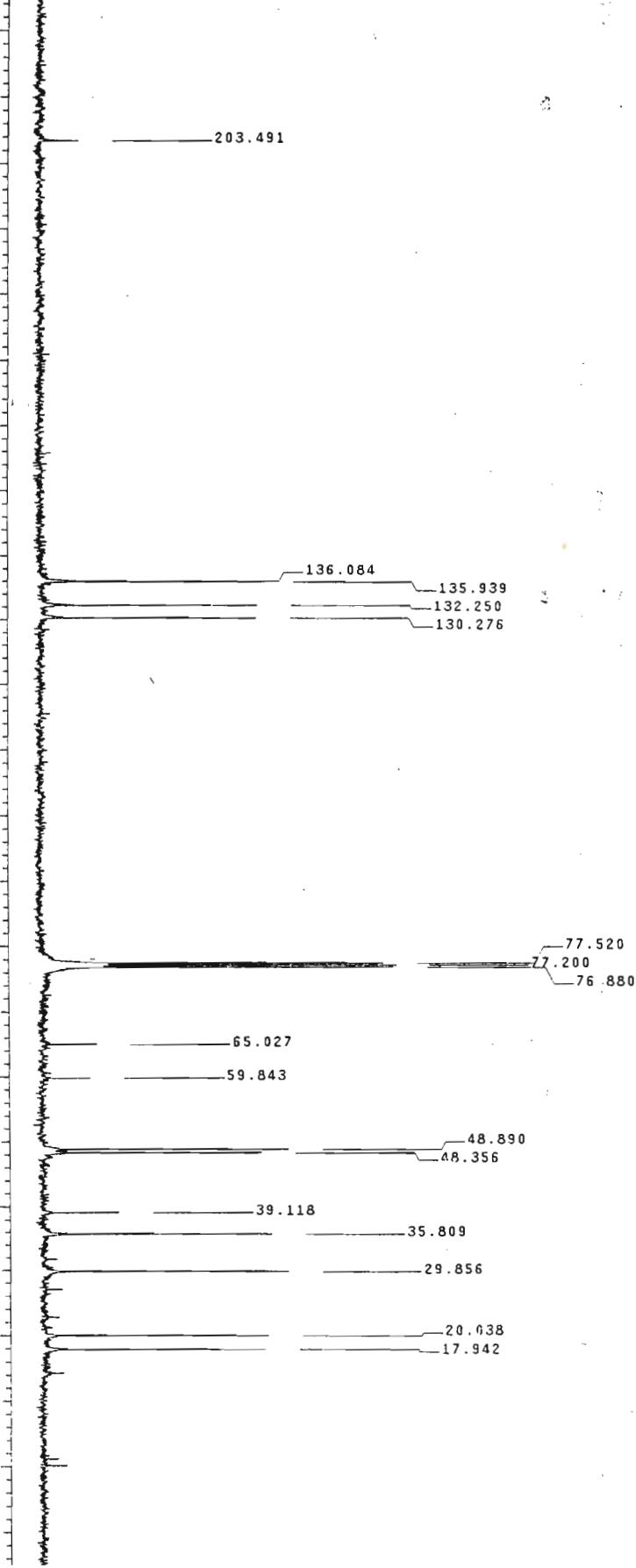
S10



exp4

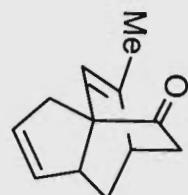
s2pu1

SAMPLE	JAN 12 2004	TEMP	SPECIAL
SOLVENT	CDCl <sub>3</sub>	GAIN	NOT USED
FILE	EXP	SPIN	NOT USED
ACQUISITION	HST	0.008	0.008
SW	25125.6	PW90	14.000
AT	1.199	ALFA	20.000
NP	60270	FLAGS	
FB	13800	II	N
BS	16	IN	N
DI	2.000	DP	Y
NT	2400	HS	NN
CT	704	PROCESSING	3.50
TRANSMITTER	1B	FB	NOT USED
TN	C13	DISPLAY	-1503.8
SFRQ	100.607	SP	25125.6
TOF	1558.7	WP	9269.8
TPWR	56	RFL	7766.0
PW	7.000	RFP	
DECOPPLER		H1	83.9
DH		RP	-299.9
DOF		0	1P
DM		Y/Y	PLOT
DMM		W	WC
DPPW		SC	250
DMF	11900	VS	0
		TH	227
		AI	5
		NO	PH

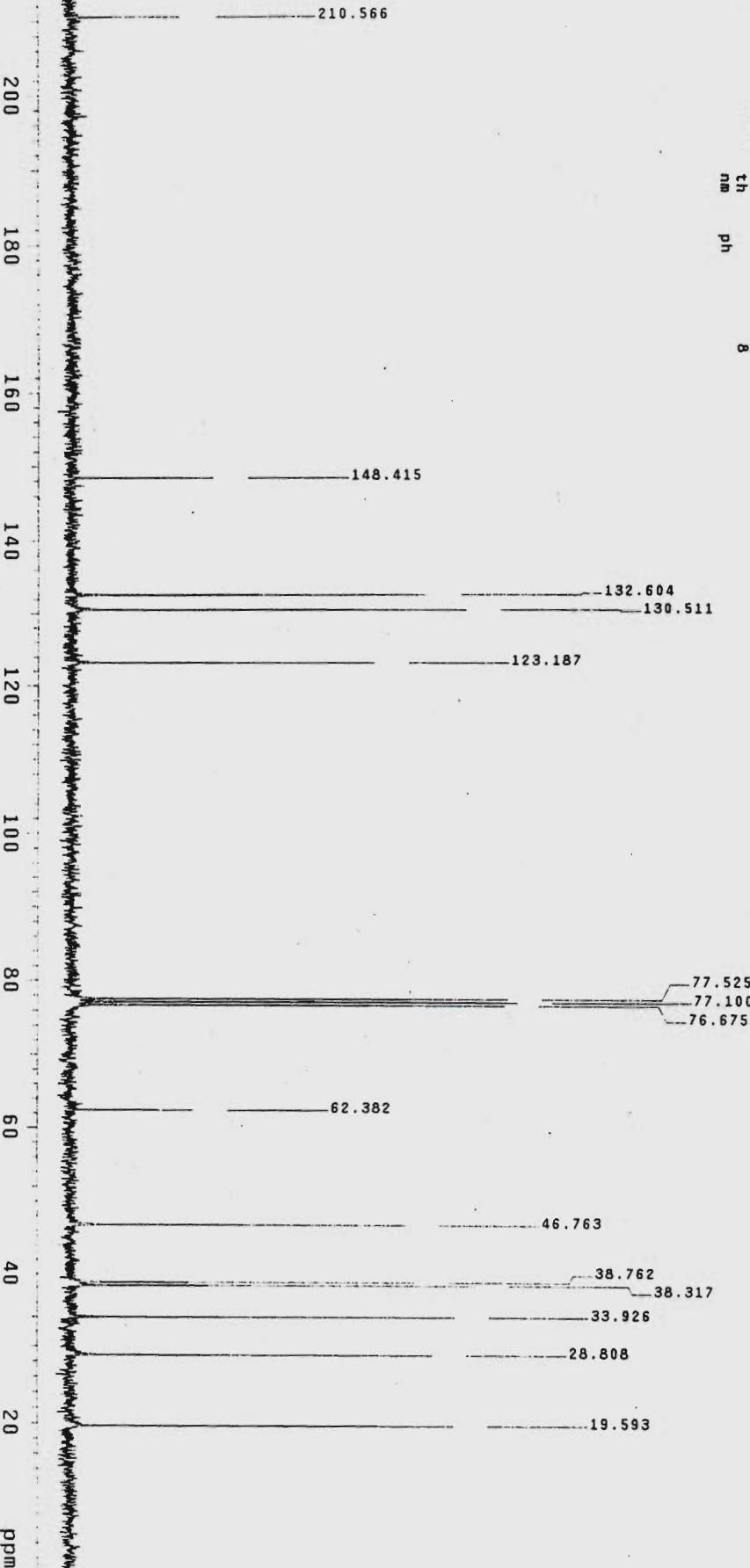


S11

exp3 s2pu1  
 SAMPLE Mar 30 2004 temp not used  
 Solvent CDCl<sub>3</sub> gain 10  
 file exp spin not used  
 ACQUISITION pw90 0.008  
 SW 2500.0 9.500  
 ac 1.280 alfa 20.000  
 np 6400 FLAGS  
 fb 13800 i1 n  
 bs 4 in n  
 d1 3.000 dp y  
 nt 3200 hs nn  
 ct 176 PROCESSING 2.00  
 tn C13 lb not used  
 strq 75.430 fn  
 tof 748.9 sp DISPLAY -38.2  
 tpwr 5.59 wp 16707.6  
 pw 4.750 rri 10032.7  
 DECOUPLER rfp 5815.0  
 dn H1 rp -124.9  
 dof 0 1p -347.0  
 dm vyy PLOT 250  
 dmm w wc 0  
 dpw 39 sc 66  
 dmf vs 8  
 nm th ph



S12

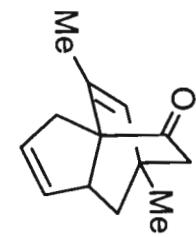
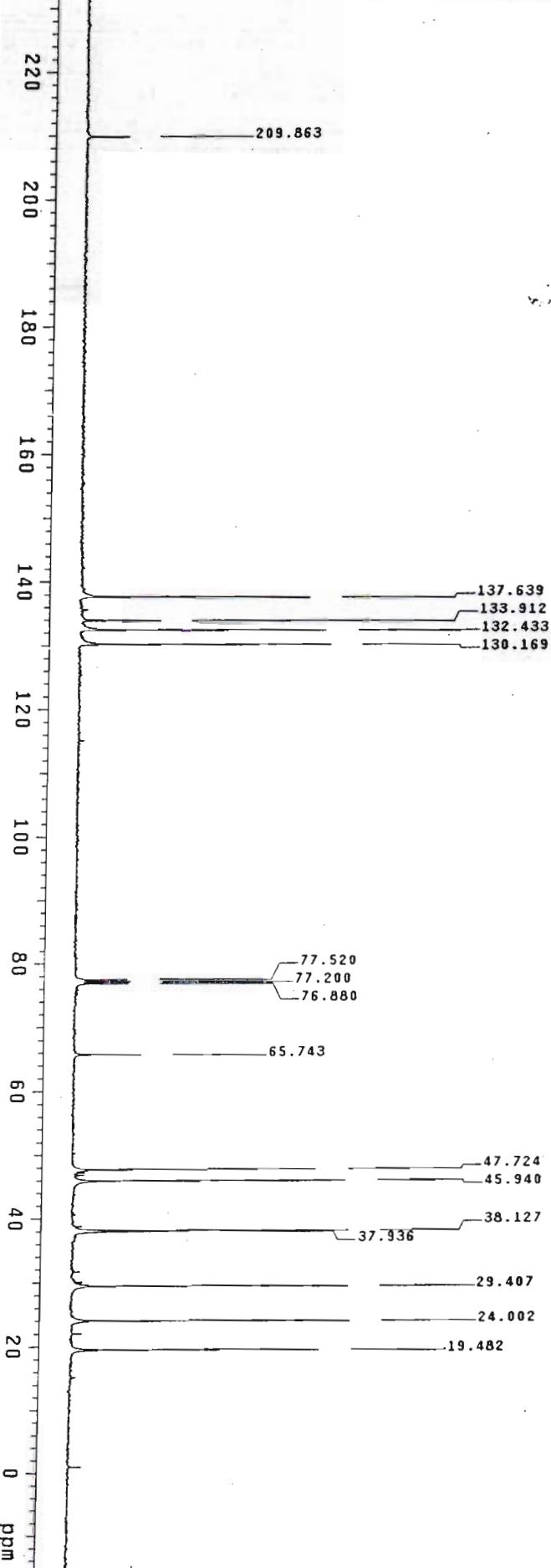


exp2 s2pu1

SAMPLE      DATE      TEMP  
solvent      Feb 26 2004      not used  
file /export/home/~spin  
r1vnmr/FEB-26~.hst  
-FEB/35parent-c13~.pwg0  
fid alfa      20.000

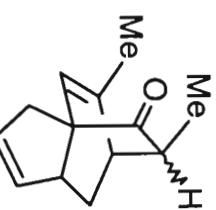
ACQUISITION      FLAGS  
tw 25125.6      11      n  
at 1.199      in      n  
tp 60270      dp      y  
fb 13800      hs      nn  
bs 1.4      PROCESSING  
d1 2.000      1b      3.50  
nt 3200      fn      not used  
ct 104      sp      DISPLAY  
TRANSMITTER      C13      -1517.6  
tn 100.607      wp      25125.6  
strq 1558.7      rfp      9283.6  
tot 7.56      rfp      7.66.0  
tpw 7.000      1p      105.9  
pw 7.000      1p      -296.3

DECOUPLER      H1      PLOT  
dn 0      wc      250  
dor 0      sc      0  
dm 0      vs      27  
dmm 41      th      a1  
dpw 11900      no      ph

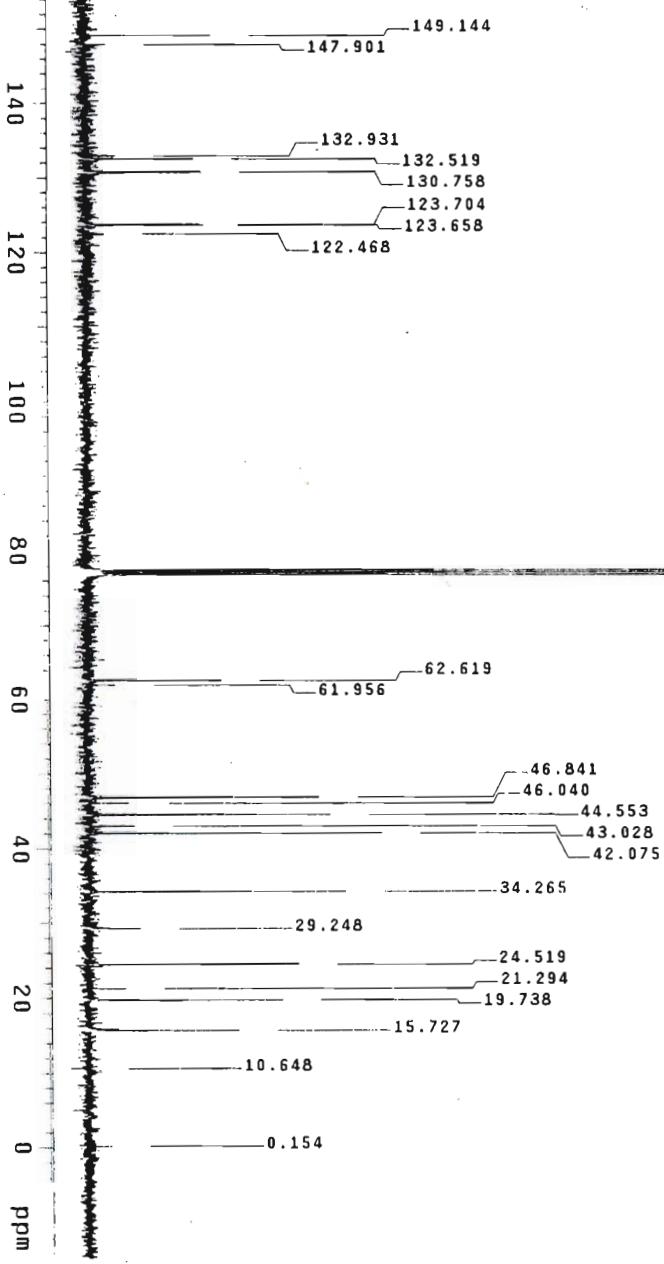


S13

SAMPLE Mar 31 2005 temp SPECIAL  
solvent CDCl<sub>3</sub> gain not used  
file exp not used  
ACQUISITION exp spin  
sw 25125.6 hst 0.008  
at 1.199 pw90 14.000  
np 60270 atra 20.000  
fb 13800 11  
bs 4 in n  
di 1.000 dp y  
nt 3000 hs nn  
ct 3000 PROCESSING 1.00  
TRANSMITTER C13  
tn 1b fn not used  
sfreq 100.561 DISPLAY -1485.1  
t0f 1554.3 sp 25125.6  
tpwr 56 wp 9247.6  
pw 7.000 rf1 7762.5  
DECOUPLER H1 rp 44.3  
dn 0 ip -335.6  
dof 0 pLOT  
dm 41 yy 250  
dmm 41 wC 0  
dpwr 11900 sc 341  
dmf vs 3 ai no  
th ph 3



S14

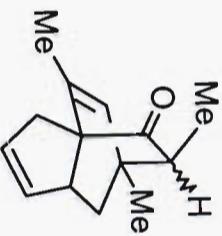


77.513  
71.192  
76.880

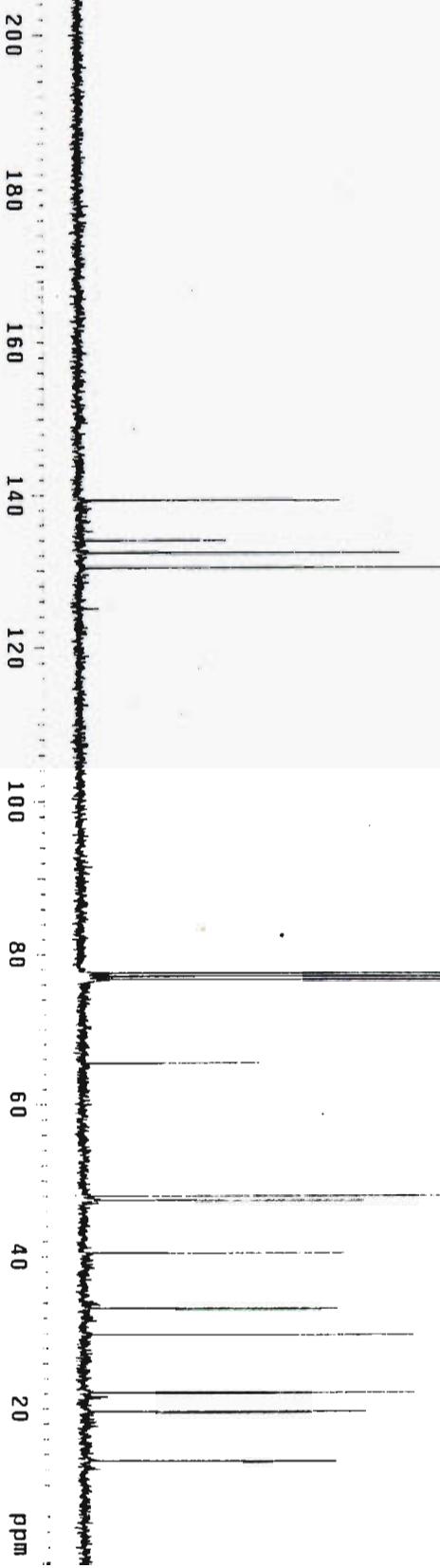
Mono Me  
Scanned for 1.5hours

exp3 s2pu1

SAMPLE	AUG 12 2004	temp	SPECIAL
solvent	CDCl <sub>3</sub>	not used	not used
file		10	10
ACQUISITION		not used	not used
sw	25000.0	hst	0.008
at	1.280	pw90	9.500
np	64000	ari	20.000
fb	13800	FLAGS	
bs	4	i1	n
d1	3.000	i1	n
nt	3200	hs	y
ct	1388		nn
TRANSMITTER		PROCESSING	
tn	C13	lb	1.00
sfrq	75.430	fn	not used
tof	748.9	DISPLAY	
tpwr	59	sp	-12.3
pw	4.750	wp	1.1443.1
DECOUPLER		rfl	10032.7
dn	H1	rfp	5815.0
dof	0	rp	-86.2
dm	vyy	ip	-353.6
dmm	w	PLOT	
dpw	39	wc	250
dif	10900	sc	0
	vs	102	
	th	15	
	nm	ph	



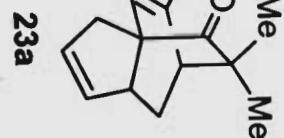
S15



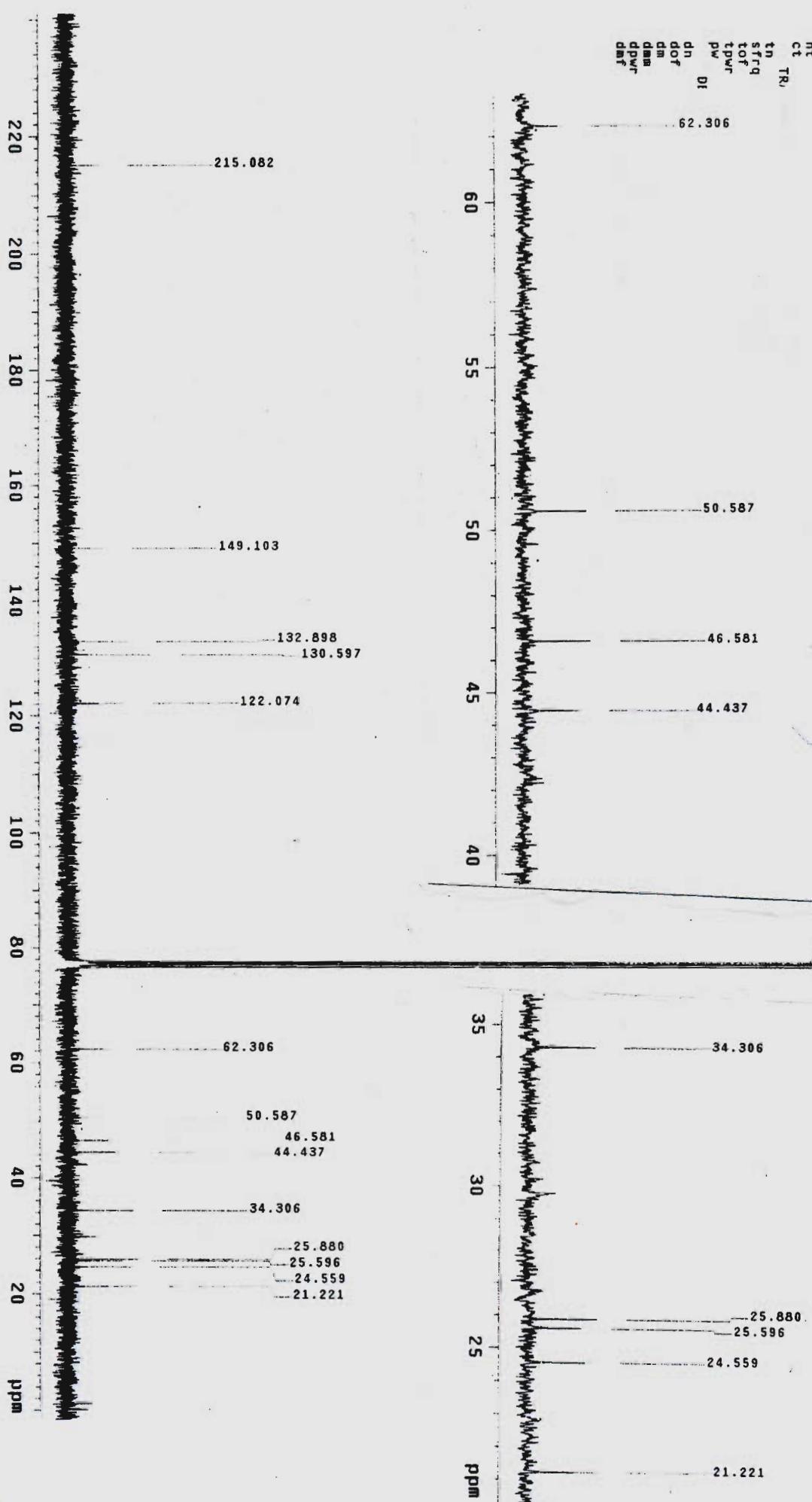
## p-Dime

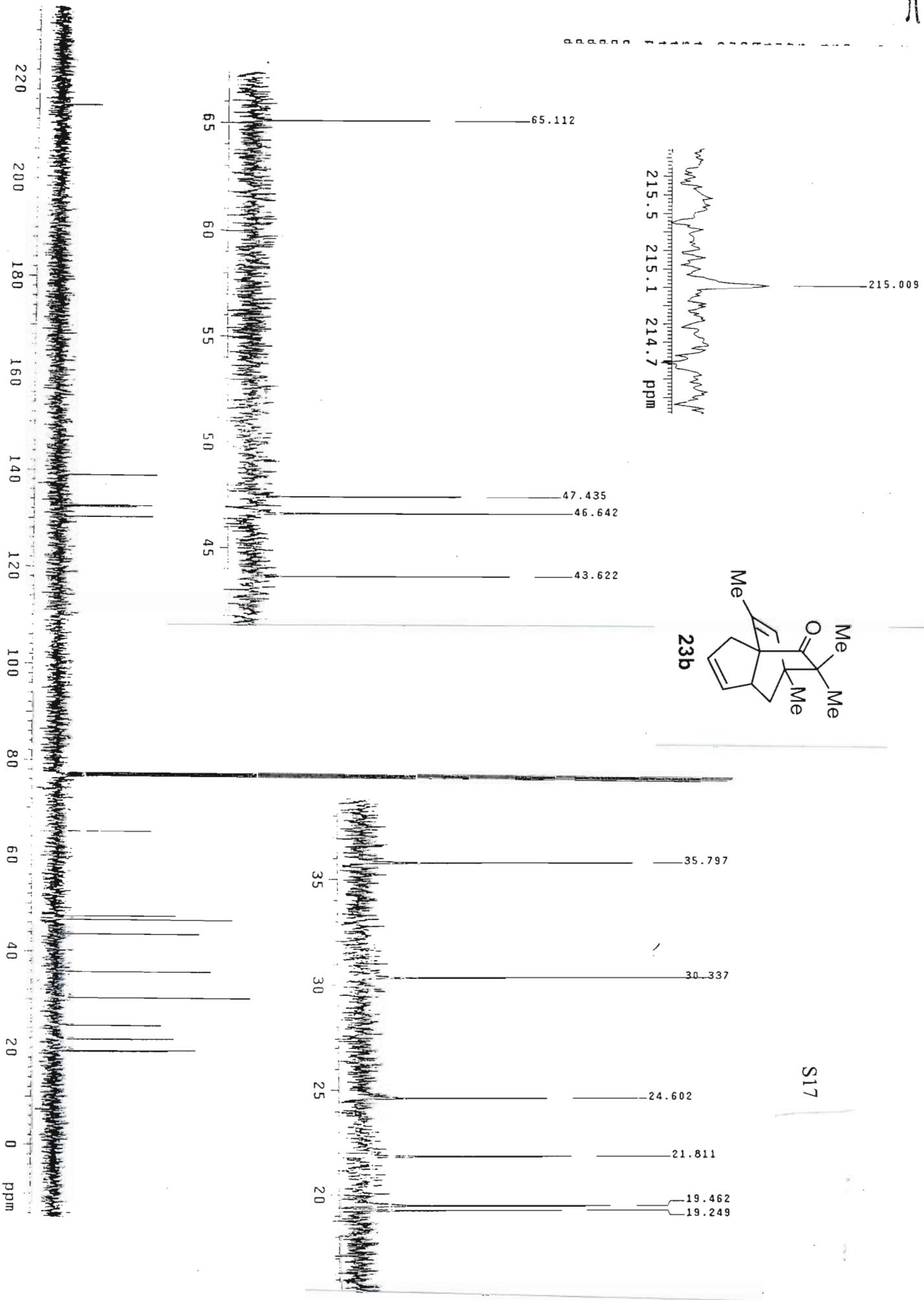
expt s2pu1

SAMPLE Mar 22 2005 temp not used  
 solvent CDCl<sub>3</sub> gain 10  
 file /export/home/~ spin not used  
 vnmr1/March-2005/2~ hist 0.008  
 2Mar/pDime-13C.fid pw90 11.500  
 ACQUISITION 20.000 alfa 20.000  
 SW 25000.0 flags n  
 AT 1.815 t n  
 NO 90752 f n  
 TB 13800 a



S16



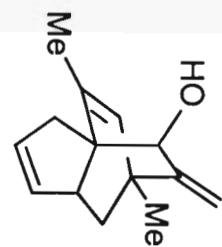
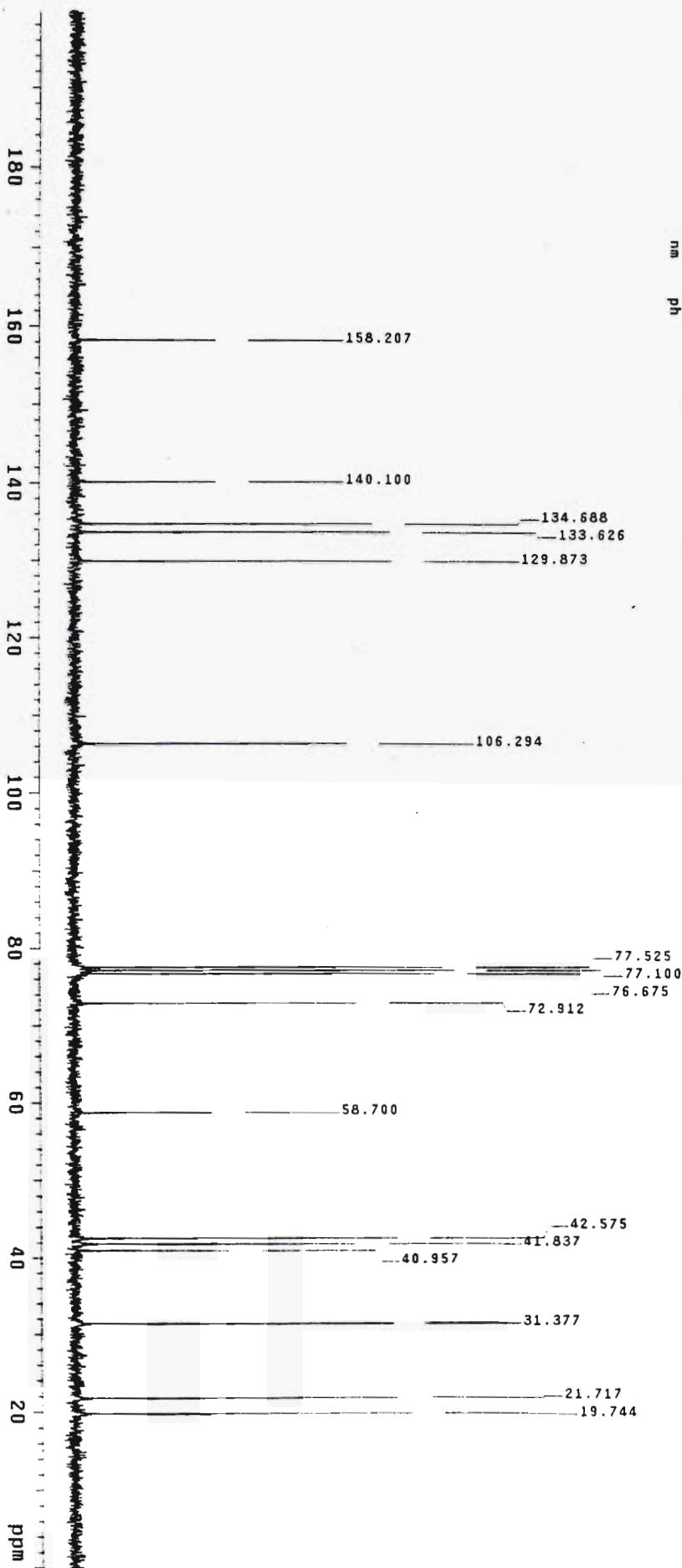


S17

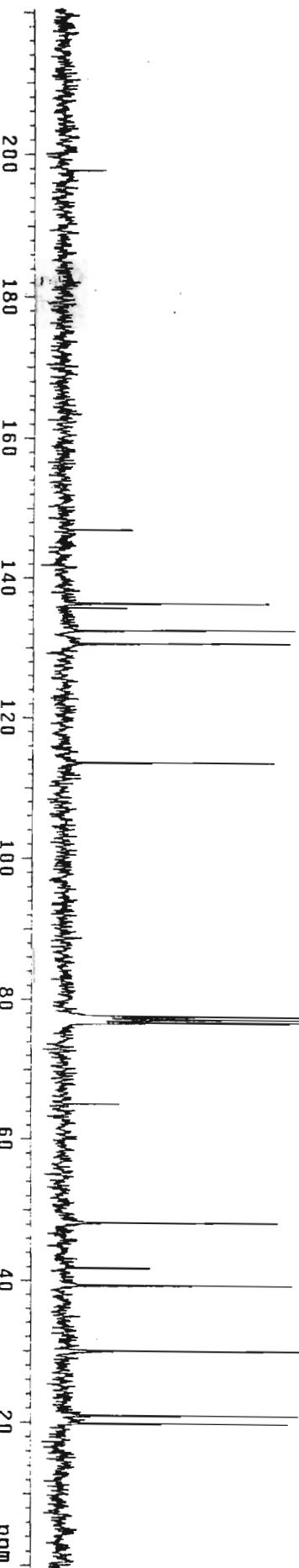
CDP  
Red. SP.-(3)

exp3 s2pu1

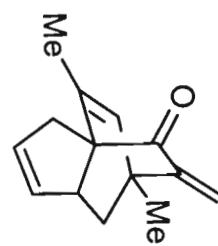
	SAMPLE	SPECIAL	
date	Mar 17 2004	temp	not used
solvent	CDCl <sub>3</sub>	gain	10
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25000.0	pwo	9.500
at	1.280	arfa	20.000
np	64000	FLAGS	
fb	13800	l1	n
bs		in	n
dt	3.000	d1	y
nt	3200	hs	nn
c1	388	PROCESSING	
tn	C13	lb	1.00
sfrq	75.430	fn	not used
tof	748.9	DISPLAY	
tpwr	59	sp	-10.0
pw	4.750	wp	15078.0
DECOUPLER		r1	10030.4
dn	H1	rfp	38015.0
dof	0	rp	-147.0
dm	1p	lp	-321.8
dmm	VYY	PLOT	
dpower	W	wc	250
dmf	39	sc	0
	10900	th	60
	vs	nm	7
	ph	ph	



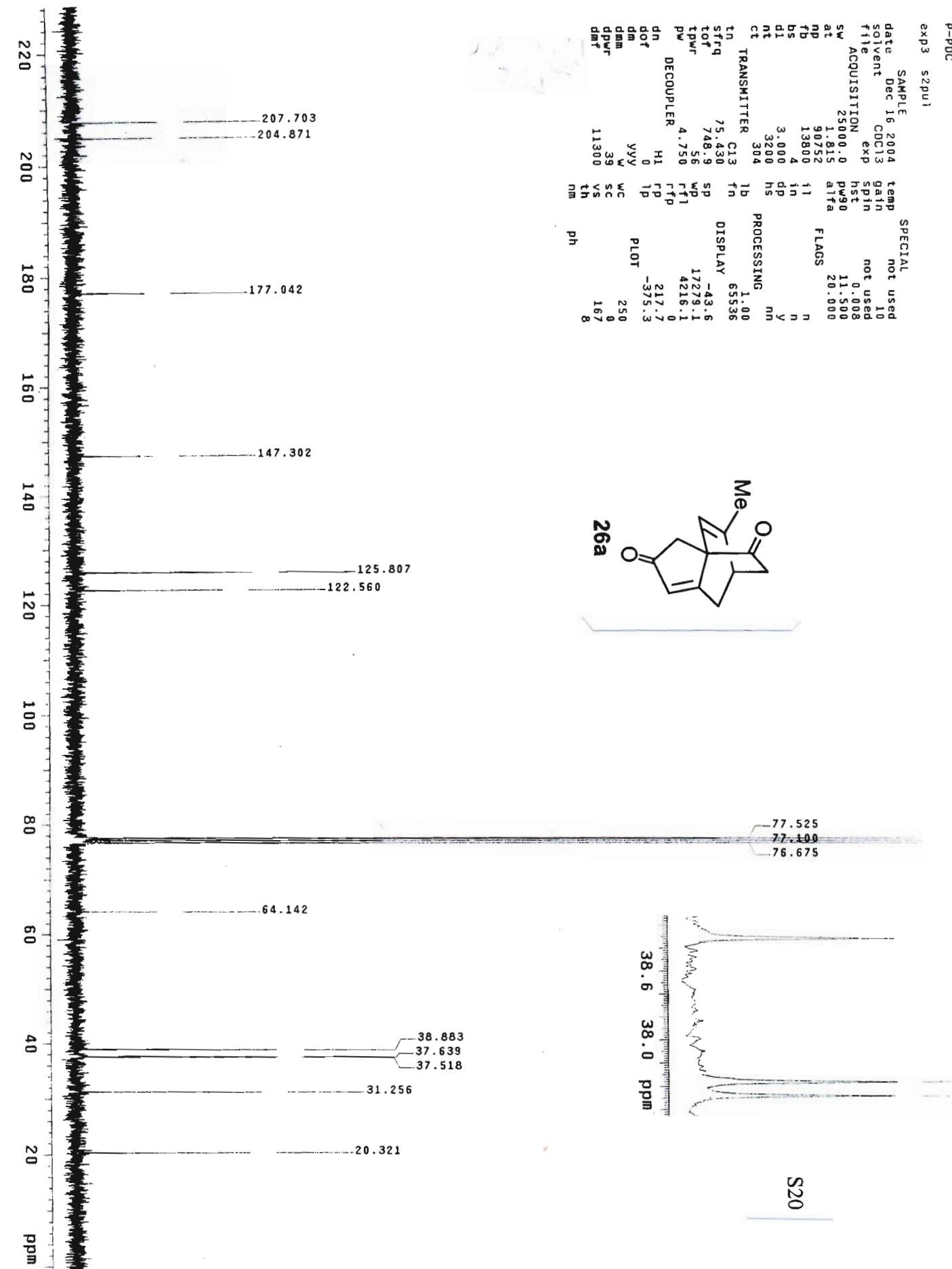
S18



exp3 s2pu1  
 SAMPLE  
 date Oct 27 2004 temp not used  
 solvent CDCl<sub>3</sub> gain 10  
 file exp not used  
 ACQUISITION spin 0.008  
 sw 25000.0 hst 0.008  
 pw 90 9.500  
 at 1.2300 aira 20.000  
 np 64000 a1fa  
 fb 13800 11 FLAGS  
 bs 4 in n  
 d1 3.000 dp y  
 nt 6400 hs nn  
 ct 332 PROCESSING 4.00  
 TRANSMITTER 1b  
 tn C13 fn not used  
 sfrq 75.431 DISPLAY  
 tor 1200.0 sp -51.2  
 tpwr 59 wp 16687.8  
 pw 4.750 rf1 9578.7  
 DECOUPLER H1 rfp 5815.0  
 dn 1p -145.9  
 dof 0 1p -365.3  
 dm yyy PLOT  
 dmm w  
 dpwr 39 sc 250  
 dm<sup>f</sup> 10900 vs 114  
 th nm 5  
 ph



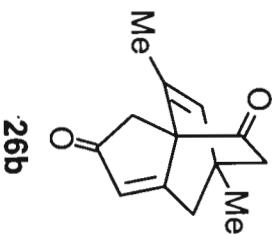
S19



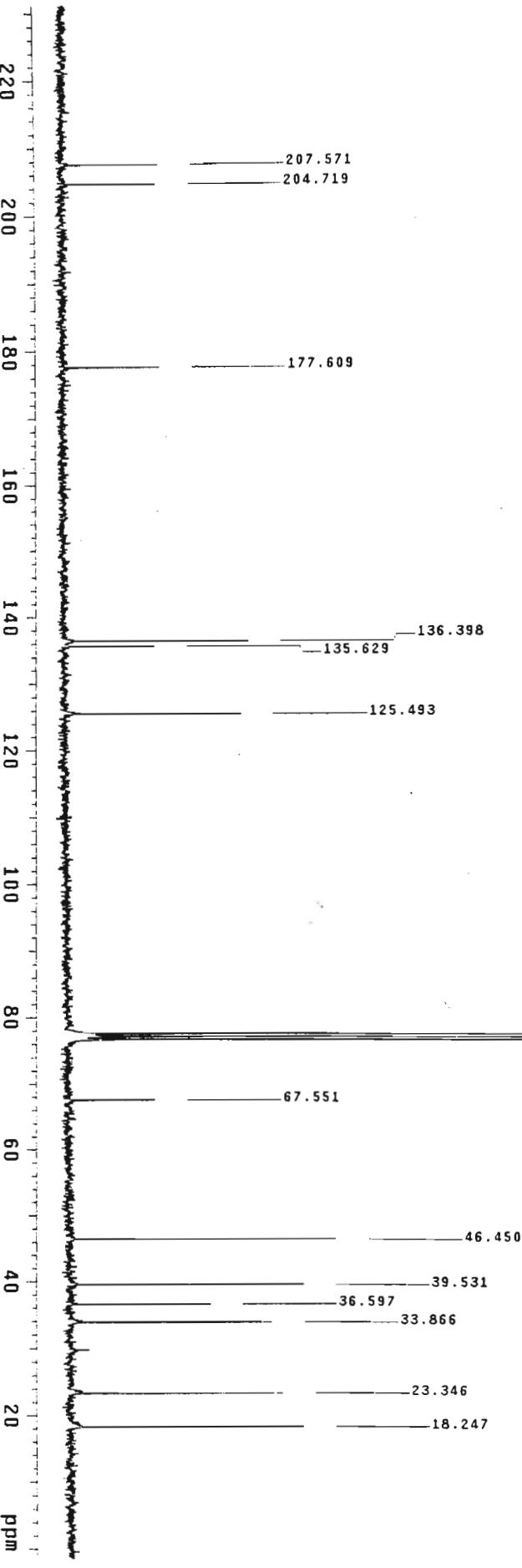
exp3 s2pu1

SAMPLE	JUL 16 2004	SPECIAL	not used
date		temp	10
solvent	CDCl <sub>3</sub>	gain	10
file		spin	not used
ACQUISITION	exp	hst	0.008
sw	25000.0	pw90	9.500
at	0.600	alpha	20.000
np	30000	FLAGS	
fb	13800	l1	n
bs	4	in	n
d1	3.000	dp	y
nt	3200	hs	nn
ct	1576	PROCESSING	2.00
TRANSMITTER	Ib	fn	not used
tn	C13	DISPLAY	-106.9
sfrq	75.430	sp	17541.5
tof	748.9	wp	4214.6
tpwr	59	r <sub>f</sub> 1	
pw	4.750	r <sub>f</sub> p	
DECOUPLER	H1	r <sub>p</sub>	-104.3
dn	0	1p	-349.5
dof	0	PLOT	250
dm	yy	wc	0
dmm	w	sc	105
dpmr	39	th	7
		nm	cdc
		ph	

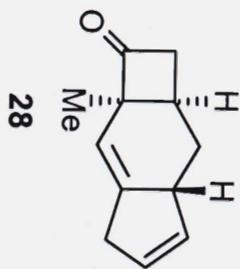
temp	not used
gain	10
spin	not used
hst	0.008
pw90	9.500
alpha	20.000
FLAGS	
l1	n
in	n
dp	y
hs	nn
fn	not used
DISPLAY	-106.9
sp	17541.5
wp	4214.6
r <sub>f</sub> 1	
r <sub>f</sub> p	
r <sub>p</sub>	-104.3
1p	-349.5
PLOT	250
wc	0
sc	105
th	7
nm	cdc
ph	



26b

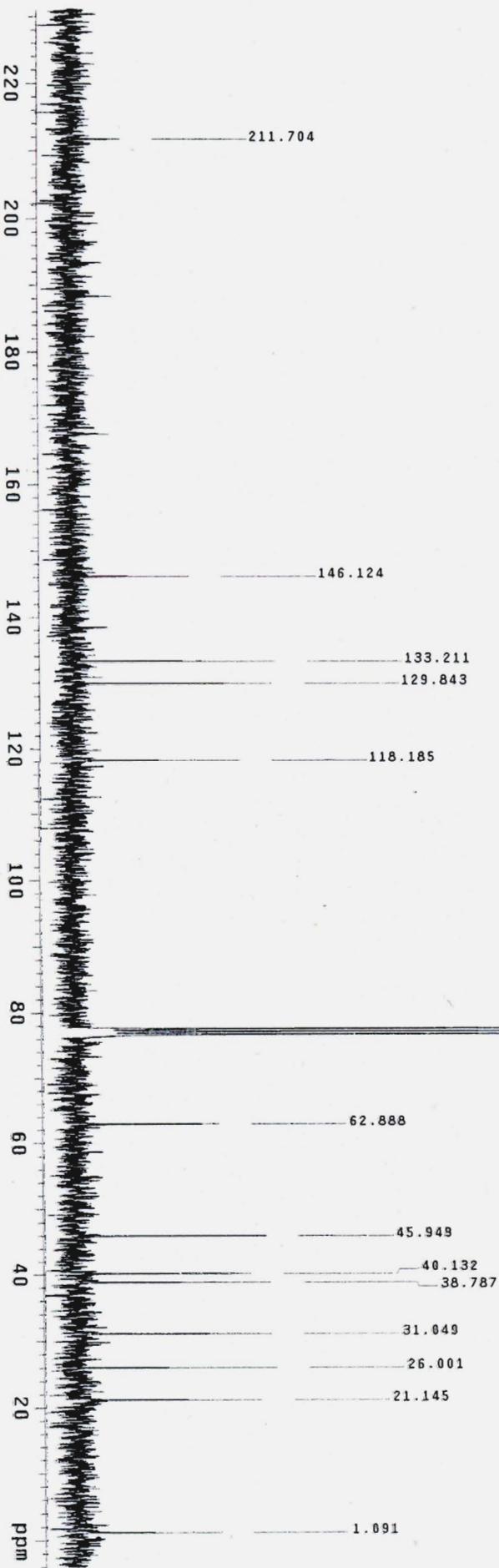


SAMPLE Jan 3 2005 temp not used  
 solvent CDCl<sub>3</sub> gain 10  
 file exp spin not used  
 ACQUISITION hst 0.008  
 sw 2500.0 pw90 11.500  
 at 1.815 alfa 20.000  
 np 90752 flags  
 fb 13800 i1 n  
 bs 4 in n  
 d1 3.000 dp y  
 nt 1600 hs nn  
 ct 704 processing 2.00  
 TRANSMITTER C13 fn not used  
 sfrq 75.430 display -306.4  
 tot 748.9 sp 17739.5  
 tpwr 56 wp 4215.7  
 pw 4.750 rf1 0  
 DECOUPLER H1 rfp -355.1  
 dn 0 lp 116.5  
 dof 1 p 250  
 dm vyy 231  
 dmm w 0  
 dppr 39 sc 8  
 dmrf 11300 vs th  
 nm ph



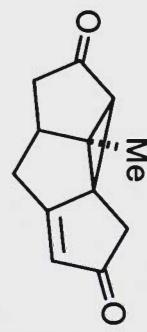
77.520  
77.095  
76.670

S22

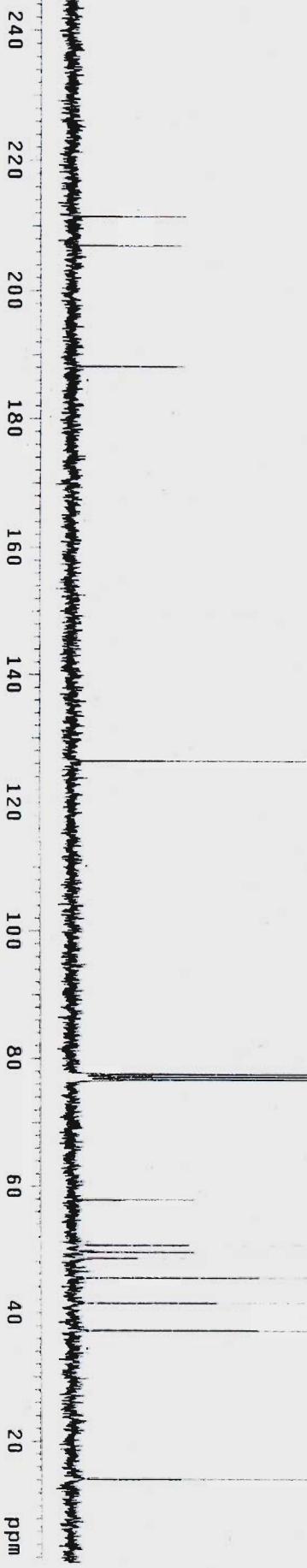


SAMPLE Jan 17 2005 temp not used  
 solvent COCl3 gain 10  
 file exp spin not used  
 not used  
 ACQUISITION hst 0.008  
 sw 25000.0 pw90 11.500  
 at 1.815 alfa 20.000  
 np 90752 aifc  
 fb 13800 i1 n  
 bs 4 in n  
 d1 3.000 dp y  
 nt 1600 hs nn  
 ct 200 PROCESSING 2.00  
 TRANSMITTER 1b  
 tn C13 fn not used  
 sfrq 75.430 DISPLAY 85.0  
 toff 748.9 sp  
 tpwr 56 wp 18774.4  
 pw 4.750 rrf1 10034.9  
 DECOUPLER rfp 5815.0  
 dn H1 rp -245.0  
 dof 0 lp -351.7  
 dm vyy PLOT 250  
 dmm w wc 250  
 dpwr 39 sc 0  
 dmrf 11300 vs 92  
 nm th 7  
 nm ph

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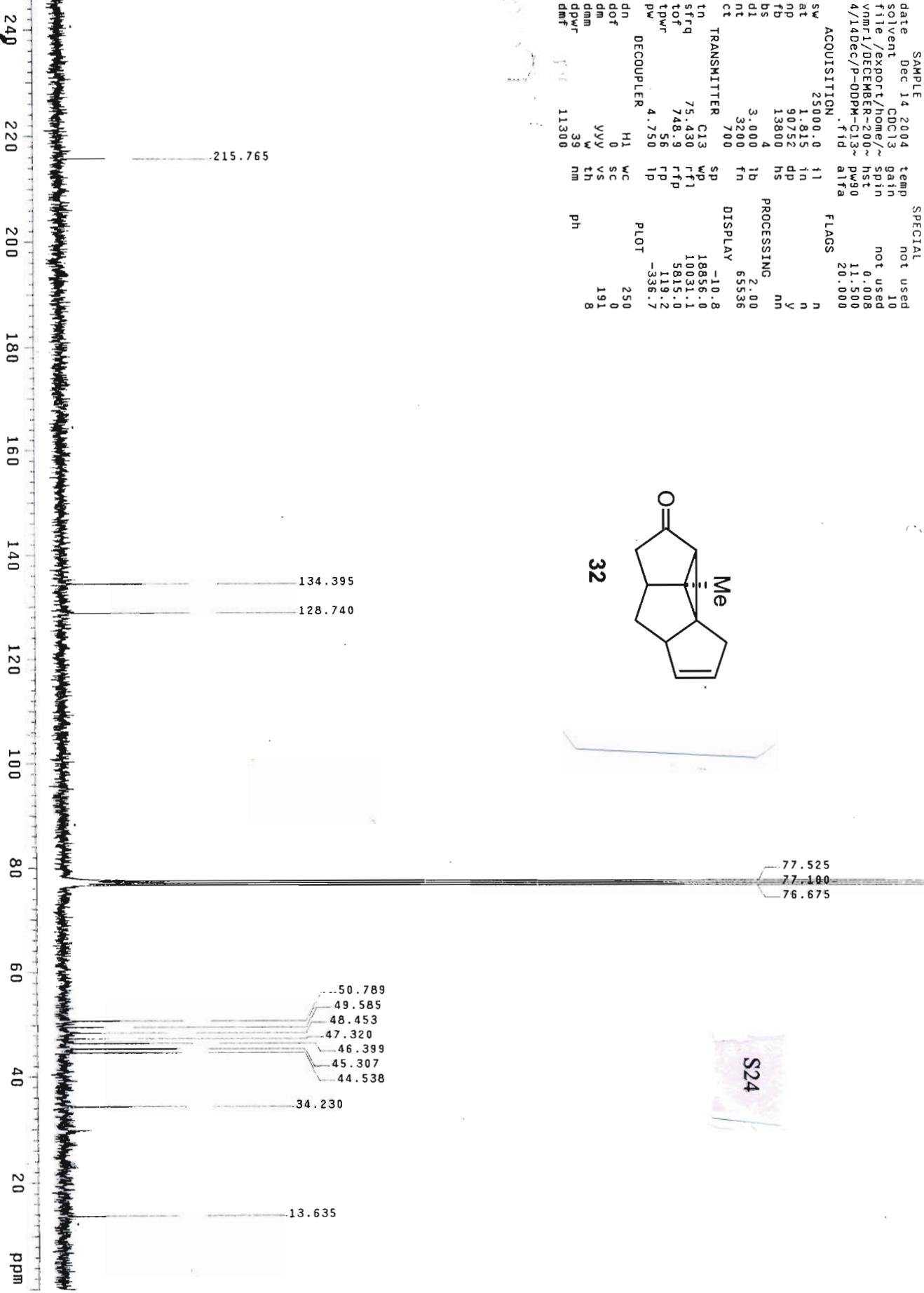


S23



p-DOPM

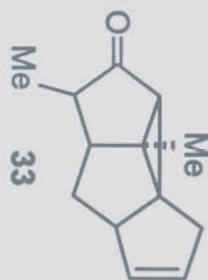
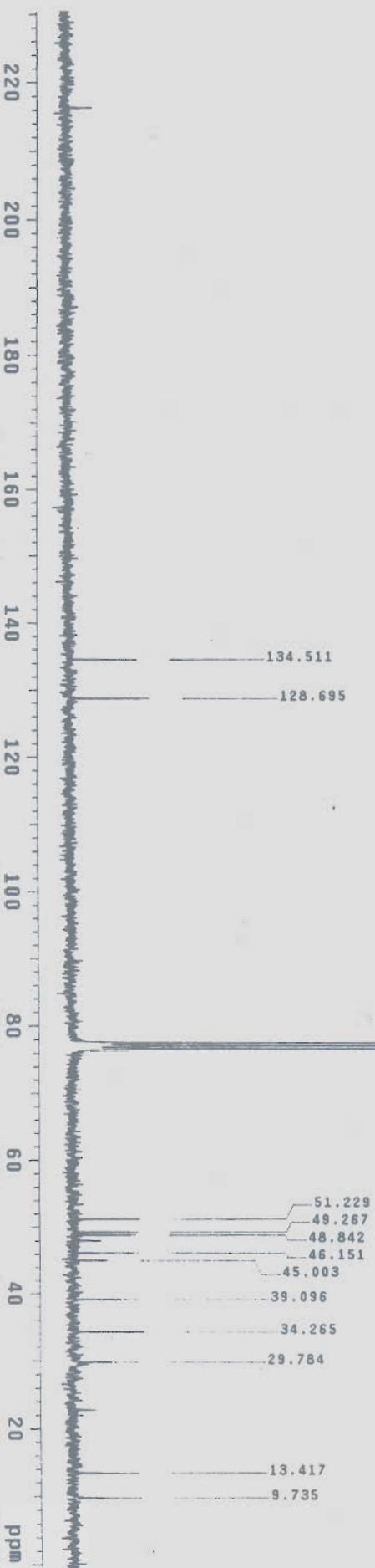
exp1 s2pu1



p-Mono ODPM  
Scanned for 2 hours

exp1 s2pu1

SAMPLE	date	Apr 25 2005	temp	not used
	solvent	CDCl <sub>3</sub>	gain	10
	file /export/home/~	spin	not used	
	vname1/APRIL-2005/2~	hst	0.008	
	SApr/p-MonoODPM-C1~	pw90	11.500	
		alpha	20.000	
ACQUISITION	sw	25000.0	11	FLAGS
	at	1.815	fn	n
	np	90752	dp	y
	rb	13800	hs	nn
	bs	4	PROCESSING	
	d1	3.000	lb	2.00
	nt	1600	fn	not used
TRANSMITTER	ct	1292	DISPLAY	-52.7
	tn	C13	sp	17433.2
	sfrq	75.430	wp	10029.2
	trf	7.48.9	r71	5815.0
	tpr	7.56	r7p	181.8
	pw	4.750	1p	-340.0
DECOPPLER	H1	wc	PLOT	
	dn	0	sc	250
	dof	v	vs	175
	dm	w	th	5
	dmm	nm	ph	
	dprw	39		
	dmf	11300		



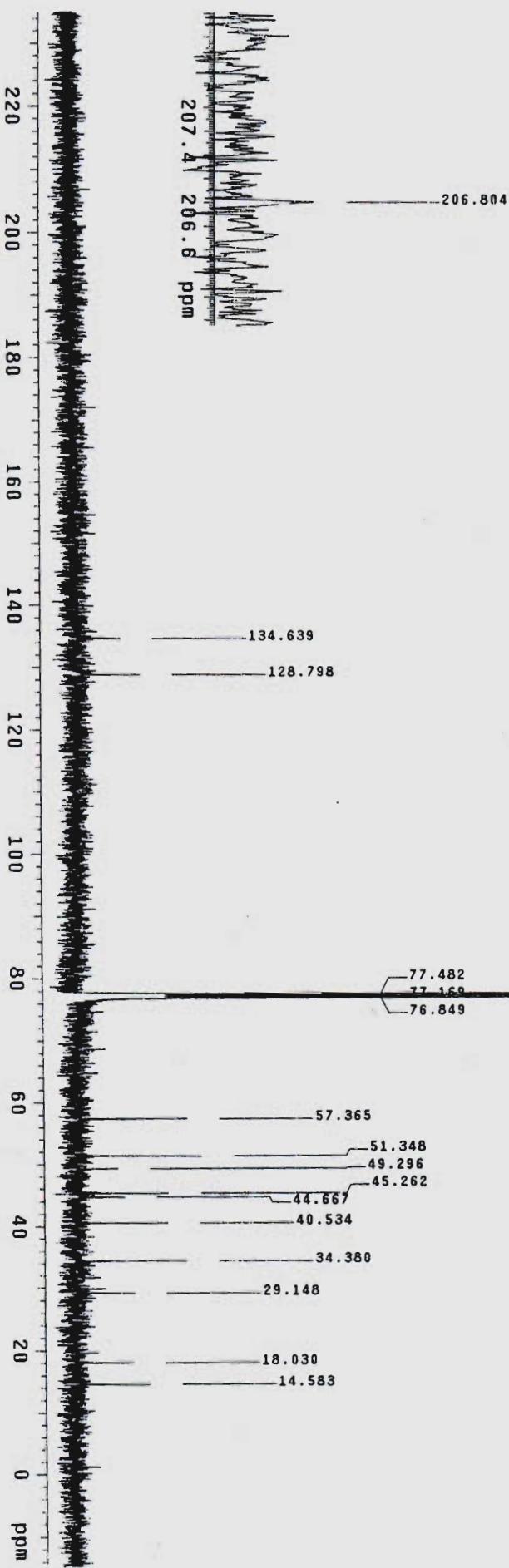
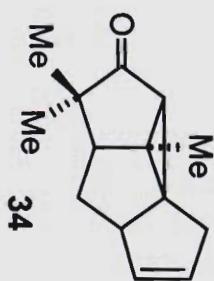
S25

P-Dime-ODPM

exp1 CARBON

SAMPLE  
date Apr 21 2005 temp not used  
solvent CDCl<sub>3</sub> gain not used  
file/export/home/~/ spin not used  
aki/vKS-p-Dime-ODP~ host 0.008  
M. r1d pw90 14.000  
ACQUISITION alfa 20.000

sw 2525.6 FLAGS n  
at 1.199 11 n  
np 60270 in n  
fb 13800 dp y  
bs 4 hs nn  
d1 1.000 1b not used  
nt 10000 1b not used  
ct 8624 fn not used  
TRANSMITTER DISPLAY  
tn C13 sp -1485.9  
sfrq 100.561 wp 25125.6  
tof 1554.3 rf1 9248.3  
tpwr 56 rfp 7762.5  
pw 7.000 rp 16.8  
DECOUPLER 1p -271.4  
dn H1 PLOT  
dot 0 wc 250  
dm yyy sc 0  
dmm vs 979  
dpwr 41 th 6  
dnf 11900 ai no ph



3,5-odpm

exp4 CARBON

SAMPLE	C6C13	temp	not used
date	Apr 14 2005	gain	not used
solvent		spin	not used
file/export/home/~		spin	not used
vksl/gmar/35-odpm1~		hist	0.008
ACQUISITION	3c.fid	pw90	14.000
sw	25125.6	alfa	20.000
at	1.199	FLAGS	n
np	6027.0		n
rb	13800	dip	y
bs	4	hs	nn
dl	1.000	PROCESSING	nn
nt	8.000	1.00	
ct	2024	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1498.2
srfq	100.561	wp	25125.6
tof	1554.3	rtt1	9250.6
tpur	56	rpp	7752.4
pw	7.000	rp	43.5
DECOUPLER	1p		-332.5
dn	H1	PLOT	
dof	0	wc	250
dm	vyy	sc	0
dmm	w	vs	234
dmr	41	th	8
	11900	ai	no ph

