Supporting information

(NMR spectra)

Convergent Total Synthesis of (+)-Mycalamide A

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(4S,5R,6R)-5,6-Dimethyl-4-(phenylselenenylmethyl)tetrahydro-2H-pyran-2-one	
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(4R,5R,6R)-5,6-Dimethyl-4-(phenylselenenylmethyl)tetrahydro-2H-pyran-2-one	

¹ H NMR of 31	S69
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¹ H NMR of 2	S71
¹³ C NMR of 2	S72
¹ H NMR of 47	S73
¹³ C NMR of 47	S74
¹ H NMR of 48	S75
¹³ C NMR of 48	S76
¹ H NMR of 49	S77
¹³ C NMR of 49	S78
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11

No2, P5	No2, P5	exp2	stdin	date	May 18 01	dec.	& VT	299.954	1	7.272	2191.1374	8.7
		solvent	CDCl3	exp	exp	def	def	def	2	4.322	1295.3208	3.9
		file	exp	exp	exp	def	def	def	3	4.314	1294.1232	3.9
		ACQUISITION	exp	exp	exp	def	def	def	4	4.304	1291.1015	3.8
		sfreq	299.953	exp	exp	def	def	def	5	4.298	1289.1785	3.7
		tn	3.841	exp	exp	def	def	def	6	4.228	1267.4770	5.0
		at	32768	exp	exp	def	def	def	7	4.208	1262.2577	5.8
		np	4500.5	exp	exp	def	def	def	8	4.144	1243.0285	7.5
		sw	2250	exp	exp	def	def	def	9	4.122	1235.4357	4.7
		fb	16	exp	exp	def	def	def	10	4.115	1234.2381	9.9
		bs	10.6	exp	exp	def	def	def	11	4.093	1227.6452	6.4
		pw	1.359	exp	exp	def	def	def	12	3.944	1183.1434	8.4
		cl	600.0	exp	exp	def	def	def	13	3.927	1177.9241	7.6
		nt	15	exp	exp	def	def	def	14	3.916	1174.6276	8.2
		clock	not used	exp	exp	def	def	def	15	3.898	1169.1336	5.2
		gbin	15	exp	exp	def	def	def	16	2.505	751.3112	5.2
		flags	15	exp	exp	def	def	def	17	2.496	748.5641	14.3
		11	n	exp	exp	def	def	def	18	2.488	746.3665	19.4
		in	n	exp	exp	def	def	def	19	1.845	493.3658	5.1
		dp	y	exp	exp	def	def	def	20	1.470	440.6974	35.6
		sp	-30.2	exp	exp	def	def	def	21	1.388	415.4489	38.0
		wp	2599.5	exp	exp	def	def	def	22	1.387	415.6895	37.1
		vs	203	exp	exp	def	def	def	23	0.000	0.0000	4.8
		sc	0	exp	exp	def	def	def				
		wc	380	exp	exp	def	def	def				
		hzmm	7.85	exp	exp	def	def	def				
		15	12796.77	exp	exp	def	def	def				
		rfl	570.8	exp	exp	def	def	def				
		rfp	0	exp	exp	def	def	def				
		th	20	exp	exp	def	def	def				
		ins	1.000	exp	exp	def	def	def				
		nm	cdc	exp	exp	def	def	def				
		ph		exp	exp	def	def	def				

INDEX	FREQUENCY (PPM)	Hz	HEIGHT
1	7.272	2191.1374	8.7
2	4.322	1295.3208	3.9
3	4.314	1294.1232	3.9
4	4.304	1291.1015	3.8
5	4.298	1289.1785	3.7
6	4.228	1267.4770	5.0
7	4.208	1262.2577	5.8
8	4.144	1243.0285	7.5
9	4.122	1235.4357	4.7
10	4.115	1234.2381	9.9
11	4.093	1227.6452	6.4
12	3.944	1183.1434	8.4
13	3.927	1177.9241	7.6
14	3.916	1174.6276	8.2
15	3.898	1169.1336	5.2
16	2.505	751.3112	5.2
17	2.496	748.5641	14.3
18	2.488	746.3665	19.4
19	1.845	493.3658	5.1
20	1.470	440.6974	35.6
21	1.388	415.4489	38.0
22	1.387	415.6895	37.1
23	0.000	0.0000	4.8

DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
acetylene
Sat Sep 10 18:35:55 2005
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
96
1.2083 sec
1.7920 sec
5.50 usec
1H
20.5 c
CDCL3
77.00 ppm
0.12 Hz
25

11

12

No2.p8

exp1 stdth

SAMPLE DEC. & VT
 date May 23 01 dfrq 299.954
 solvent CDCl3 H1
 file exp 0
 ACQUISITION exp 0
 sfrq 299.955 H1
 tn 200
 st 3.641
 up 32769 wfile
 sw 4500.5 proc ft
 fu 2250 fu 32768
 bs 15
 pw 10.5 warr react
 d1 1.359 wexp wft
 tot 600.0 wbs
 nt 15 wnt
 ct 15
 alock n
 gain not used
 FLAGS
 ll n
 .n n
 dp y
 DISPLAY
 sp -30.2
 wp 2999.5
 va 203
 sc 0
 wc 380
 hzmm 7.88
 lb 23703.51
 rf1 574.4
 rfp 0
 th 20
 ins 1.000
 nm cdc ph

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
Br-alkene
Sat Sep 10 19:01:51 2005
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
96
1.2083 sec
1.7920 sec
5.50 usec
1H
20.6 c
CDCL3
77.00 ppm
0.12 Hz
25

12

--- ACQUISITION PARAMETERS ---
File Name = 1d_spectrum.479
Author =
Sample ID = Kagawa
Content = Single Pulse Experiment
Creation Date = 26-JUN-2006 18:14:13
Revision Date = 28-JUN-2006 04:05:26
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = 1
Dim Title = 1H
Dim Size = 16384
Dim Units = [ppm]
Scans = 8
Mod_return = 1
X_dmain = 1H
X_offset = 5 [ppm]
X_freq = 500.18241602 [MHz]
X_sweep = 7.50750751 [kHz]
Solvent = CHLOROFORM-D
Spin_get = 15 [Hz]
Temp_get = 23.1 [°C]
Recvr_gain = 25
Field_strength = 11.7473579 [T]
Filter_mode = BUTTERWORTH
Filter_width = 3.75119936 [kHz]

13

TBDPS ka

exp1 std1h

SAMPLE		DEC. & VT	
date	Jan 12 99	dfrq	299.954
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nmn
sfrq	299.955	dms	c
tn	H1	daf	200
at	3.641	PROCESSING	
np	32768	wfile	
sw	4500.5	proc	ft
fb	2250	fn	32768
bs	16		
pw	5.0	werr	react
d1	1.359	wexp	wft
tof	600.0	wbs	
nt	15	wit	
ct	15		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-30.2		
wp	2999.5		
vs	428		
sc	0		
mc	360		
hzman	7.89		
ls	21758.18		
rfl	574.1		
rfd	0		
th	20		
ins	1.000		
nm	cdc	ph

TBDPS

40 mg

exp1 std13c

SAMPLE DEC. & VT
date Jan 14 99 dirq 299.954
solvent CDCl3 dn H1
file exp1 exp1 1
ACQUISITION
sfrq 75.431 dm yyy
tn C13 dm W
at 0.873 dmf 11050
np 32768
sw 18761.7 lb PROCESSING
fb 9500 wfile 1.00
bs 16 proc. ft
pw 10.0 fn 32768
d1 1.127
tof 0 werr
nt 1024 wexp wft
ct 67 wds wft
alock n
gain not used
FLAG

il n
in n
op y
SP -1882.5
MP 18761.7
VS 214
SC 0
WC 400
HZ 400
H2 0.47
IS 500.00
RF 7708.3
RFD 5815.7
TH 10
INS 1.000
NM no ph

14

S11

OCC(CO)C(O)[C@H](OBn)COPIV

15

exp2 std13c.

SAMPLE		DEC. & VT	
date	May 11 98	dfrq	299.954
solvent	CDCl3	dn	h1
file	exp	dhp	i
ACQUISITION		doF	0
sfrq	75.431	dm	yyy
tn	C13	dmm	w
at	0.873	dmf	11050
np	32768	PROCESSING	
sw	18761.7	lb	1.00
fb	9500	wtfile	
bs	16	proc	ft
pw	10.0	fn	32768
d1	1.127		
tof	0	werr	wft
nt	200	wexp	
ct	200	wos	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1965.3		
wp	18761.7		
vs	214		
sc	0		
mc	400		
hzmag	0.54		
is	600.00		
rfl	7722.0		
rfl	5815.7		
th	30		
ins	1.000		
nm	no ph		

51

513

methyleneacetat] ka

methyleneacetat] ka

exp2 std13c

SAMPLE DEC. & VT
date Nov 7 98 dfrq 299.954
solvent CDCl3 dn H1
file exp ddp 1
ACQUISITION
sfrq 75.431 dm vvy 0
tn C13 dmm W
at 0.873 cmf 11050
np 32768
sw 18761.7 lb 1.00
fb 9500 wfile
bs 16 proc ft
pw 10.0 fn 32768
di 1.127
tof 0 werr
nt 300 wexp wft
ct 258 wds wft
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1892.5
wd 18761.7
vs 214
sc 0
wc 400
hzmm 0.47
ls 500.00
rfl 7708.3
rfd 5815.7
th 5
ins 1.000
nm no ph

16

S15

EXMOD SGBCM
OBNUC 13C
OBERQ 125.65 MHz
OBSET 117.00 KHz
OBFIN 13000.0 Hz
OBATN 0
IRNUC 1H
IRSET 152.00 KHz
IRFIN 10400.0 Hz
IRATN 5
IRRPW 50 us
FREQU 31250.0 Hz
POINT 32768
RGAIN 29
ACQTM 1.049 sec
PD 2.000 sec
SCANS 600
PW1 6.5 us
PW2 24.0 us
PW3 20.0 us
PI1 3.450 ms
PI2 0.100 ms
PI3 1.80 ms
LOOP1 1
SLVNT CDCL3
EXREF 77.10 ppm
EXP:SBELL:SSQUA
BF 0.10 Hz
249c: ()'c

18b

18b

519

exp3	stdt3c	SAMPLE	DEC. & VT
date	Dec	8 98	qfng
solvent	CDCl3	exp	dn
file	ACQUISITION	exp	thp
strng	75.431	dm	daf
tn	C13	dmm	yyy
at	0.673	dmt	w
np	32768		11093
sw	16761.7	lb	PROCESSING
fb	9500	wfile	1.00
bs	15	proc	ft
pw	10.0	fn	32768
d1	1.127		
tof	0	werr	wft
mnt	303	wexp	wft
ct	203	wbs	
alock	n	wrt	
gain	not used		
FLA6S			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1699.5		
wd	16761.7		
vs	214		
sc	0		
wc	400		
hzmm	0.90		
is	500.00		
rfl	7715.2		
rnp	5815.7		
th	14		
ins	1.000		
nm	no	ph	

7-epi-tms ether

exp1 std1h

date Jan 29 03
solvent CDCl3
file exp1
ACQUISITION
sfrq 299.955
tn H1
at 3.641
np 32768
sw 4500.5
fb 2250
bs 15
pw 11.3
di 1.359
tof 600.0
nt 16
ct 16
alock not used
gain not used
flags n n y
display -30.1
sp 2999.5
wp 203
vs 0
sc 380
mc 7.89
hzmm 17022.93
is 2752.2
rfl 2177.7
rfp 20
ins 1.000
nm cdc ph

DEC. & VT 299.954
dfrq H1
dn 0
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile ft
proc 32768
fn react
wft

7-epi-tms ether

Wed Jan 29 17:26:06 JST 2003

INDEX	FREQUENCY (PPM)	HZ	HEIGHT
1	7.260	2177.6740	9.2
2	3.635	1090.4017	25.7
3	1.290	386.8888	11.9
4	1.217	364.9126	11.3
5	1.083	324.8060	43.7
6	0.224	67.1352	59.3

19a

12-epi-TMS-al001

exp1 std13c

SAMPLE DEC. & VT
date Feb 10 03 dfreq 299.954
solvent CDCl3 dn H1
file exp dhp 1
ACQUISITION daf 0
sfrq 75.431 dm yyy
tn C13 dmm w
at 0.873 dmf 9720
np 32768
sw 18761.7 lb 1.00
fb 9500 wfile
bs 15 pproc ft
pw 8.4 fn 32768
d1 1.127
tof 0 werr
nt 10000 maxp wft
ct 480 wds wft
alock n
gain not used
FLAG5
t1 n
in n
dp y
DISPLAY
sp -754.9
wp 17348.5
vs 214
sc 0
wc 400
hzmm 43.37
is 500.00
rf1 7705.0
rfp 5808.2
th 14
ins 1.000
nm no ph

19a

m3, p51, No2-CSApure

exp1 std1h

SAMPLE DEC. & VT
date Jun 14 02 dfrc 299.949
solvent CDCl3 dn H1
file exp 0
ACQUISITION dm nnn
sfrq 299.949 dnm c
tn H1 dmf 200
at 3.641 PROCESSING
np 32769 wfile
sw 4500.5 proc ft
fb 2250 fn 32769
bs 15
pw 10.5 warr react
d1 1.359 wexp wft
tof 600.0 wds
nt 128 wnt
ct 128
alock n
gain not used
FLAG5
il n
in n
dp y
DISPLAY
sp -30.2
wp 2999.5
vs 203
sc 0
wc 380
hzmm 7.89
ls 11257.47
rfj 573.6
rfp 0
th 20
ins 1.000
nm cdc ph

19b

196

12-epi

exp2 std13c

SAMPLE		DEC. & VT	
date	Jan 10 03	dfrq	299.954
solvent	CDCl3	dn	H1
file	exp	dhp	1
ACQUISITION		dof	0
sfrq	75.431	dm	yyy
tdn	613	dmm	w
at	0.873	dmf	9720
np	32768	PROCESSING	
sw	18761.7	lb	1.00
fb	9500	wtfile	ft
bs	16	prdc	32768
pw	8.4	fn	
d1	1.127		
tof	0	went	wft
nt	512	wexp	wft
ct	432	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-38.0		
wp	16593.9		
vs	214		
sc	0		
wc	400		
hnm	41.49		
is	500.00		
rfl	7707.2		
rpf	5808.2		
th	19		
ins	1.000		
nm			
no			
ph

19c

diol		exp1		stdn		SAMPLE		DEC. & VT			
date	Jan	6	03	dfreq	299.954	dn	H1	0	nnn	c	200
solvent	CCCC13	exp	df	dm	299.955	dmm	H1	3.641	32768	wfile	ft
file	ACQUISITION	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
sfreq	299.955	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
tn	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
np	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
sw	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
fb	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
bs	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
pw	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
d1	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
tof	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
nt	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
ct	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
gain	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
alock	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
not used	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
FLAGS	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
il	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
in	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
dp	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
sp	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
wp	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
vs	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
sc	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
wc	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
hzmm	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
is	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
rfl	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
rpf	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
th	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
ins	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
nm	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
cdc	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768
ph	at	df	dm	dmf	299.955	dmm	H1	32768	4500.5	proc	32768

diol
Tue Jan 7 08:54:56 JST 2003

exp1 stdi3c		SAMPLE		DEC. & VT	
date	Jan 5 03	dfrq	299.954		
solvent	CDCl3	dn			
file	exp	dhp			
ACQUISITION					
sfrq	75.431	dm	yyy		
tn	C13	dmm	w		
at	0.873	dmf	9720		
qp	32768	PROCESSING			
sw	18761.7	lb	1.00		
fb	9500	wtfile			
bs	15	proc	ft		
pw	B.4	fn	32768		
d1	1.127				
tof	0	warr			
nt	15360	wexp	wft		
ct	14736	wbs	wft		
alock	n	wnt			
gain	not used				
FLAGS					
tl	n				
ln	n				
dq	y				
DISPLAY					
sp	-1897.8				
wp	16593.9				
vs	214				
sc	0				
wc	400				
hzmm	41.48				
is	500.00				
rfl	7706.0				
rpf	5808.2				
th	4				
ins	1.000				
nm	no				
ph					

INDEX	FREQUENCY (PPM)	H _z	HEIGHT
1	177.445	13384.7789	0.1
2	135.861	10248.0904	0.3
3	135.724	10237.7637	0.2
4	133.842	10095.7795	0.0
5	129.591	9775.1250	0.0
6	127.647	9626.5400	0.4
7	101.231	7635.9011	0.1
8	93.640	7053.3037	0.1
9	80.811	6095.6141	0.1
10	78.609	5929.5609	0.1
11	77.425	5940.2357	1.6
12	77.000	5808.1703	1.7
13	76.575	5776.1048	1.6
14	72.294	5453.1599	0.1
15	62.683	4728.2516	0.1
16	52.562	4719.0900	0.1
17	51.600	3892.2594	0.1
18	44.571	3362.0342	0.1
19	27.056	2041.5246	0.1
20	26.626	2008.4140	0.4
21	24.075	1816.0212	0.1
22	19.247	1451.8493	0.1
23	18.397	1387.7184	0.0
24	-0.155	-11.7096	0.0

20 (n=1)

cycloheptanone, aldol

exp1 stdin

SAMPLE		DEC. & VT	
date	Mar 28 03	dfreq	299.954
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nmh
sfrq	299.955	dmp	C
tn	H1	dmf	200
at	3.641	PROCESSING	
np	32768	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	32768
bs	15	war	
pw	11.3	wexp	react
d1	1.359	wft	
tof	500.0	wbs	
nt	15	wnt	
ct	15		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-30.2		
wp	2999.5		
vs	214		
sc	0		
wc	400		
hzmm	7.50		
is	20425.71		
rfl	538.7		
rfl	0		
th	2		
ins	1.000		
nm	cdc	ph

20 (n=2)

aldol, B, TMSaheer

exp1 stdth

SAMPLE		DEC. & VT	
date	3 03	dfrq	299.954
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nmh
sfrq	299.955	dmm	c
tn	H1	dmf	200
at	3.641	PROCESSING	
np	32768	wtfile	
sw	4500.5	prac	ft
fb	2250	fn	32768
bs	16		
pw	11.3	wnrr	react
d1	1.359	wexp	wit
tof	600.0	wbs	
nt	32	wnt	
ct	32		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-90.0		
wp	2999.5		
vs	203		
sc	0		
mc	360		
hzmm	7.89		
is	13254.66		
rfl	2747.3		
rfp	2177.7		
th	2		
ins	1.000		
nm	cdc	ph

C:\My Documents\kagawa\Michael,7.als
Michael,7
Fri Apr 04 20:40:54 2003

OBNUC 1H
EXMOD NON
OBFQ 399.65 MHz
OBSE 124.00 KHz
OBFN 10500.0 Hz
POINT 32768
FREQ 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PWL 8.3 us
IRNUC 1H
CTEMP 25.0 C
SLVAT CDCL3
EXREF 0.00 ppm
BF 0.50 Hz
RGAIN 15

21 (n=1)

DFILE C:\My Documents\kagawa\Michael,8.als
 COMPT Michael,8
 DATIM Sat Apr 05 10:22:37 2003
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.0 Hz
 POINT 32768
 FREQU 7993.6 Hz
 SCANS 16
 ACQTM 4.099 sec
 PD 2.901 sec
 PM1 8.3 us
 IRNUC 1H
 CTMP 23.2 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.50 Hz
 RGAIN 14

21 (n=2)

23 (major isomer)

p188, upper

Tue Mar 11 09:58:10 JST 2003

INDEX	FREQUENCY (PPM)	H _z	HEIGHT
1	9.540	2861.6831	2.0
2	9.533	2859.4854	2.9
3	7.444	2232.6892	3.3
4	7.426	2227.3951	2.5
5	7.420	2225.7469	4.9
6	7.412	2223.2746	3.3
7	7.407	2221.6264	2.4
8	7.391	2216.9564	2.0
9	7.353	2205.6936	2.3
10	7.334	2199.9249	4.7
11	7.329	2198.2767	5.2
12	7.306	2191.4091	5.9
13	7.268	2180.1463	6.9
14	7.265	2179.3222	6.7
15	7.260	2177.6740	19.5
16	7.249	2174.3775	2.6
17	7.244	2173.0040	3.5
18	7.241	2171.9052	3.1
19	5.697	1708.7569	2.2
20	5.672	1701.3400	2.3
21	1.558	467.3765	4.8
22	1.205	361.3415	2.7
23	1.013	303.9287	9.0
24	0.990	287.0511	9.0
25	0.946	283.8754	8.3
26	0.924	277.2825	8.0

p188, upper

exp1 std th

SAMPLE 11 03 DEC. & VT 299.954

date Mar 11 03

solvent CDCl₃ dn H1

file exp dof 0

ACQUISITION dm nmh

sfreq 299.955 dmm c

tn H1 dmf 200

at 3.541 PROCESSING

np 32768 wfile

sw 4500.5 prac

fb 2250 fn 32768

bs 16

pw 11.3 werr

d1 1.359 wexp

tof 600.0 wbs

nt 16 wnt

ct 16

clock not used

gain n

FLAG

11 n

1n n

dp y

DISPLAY

sp -30.1

wp 2999.5

vs 203

sc 0

wc 380

hzmm 7.89

is 11381.76

rfl 2752.0

rfp 2177.7

th 20

ins 1.000

nm cdc ph

DFIL2 C:\My Documents\kagawa\oxazolidinone-
CCMT oxazolidinone-upper
DATIM Wed Apr 23 12:19:23 2003
OENUC 13C
EXMOD BCM
OBERQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27173.9 Hz
SCANS 262
ACQTM 1.206 sec
PD 1.794 sec
PWL 6.0 us
IRNUC 1H
CTEMP 23.0 C
SLANT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 22

23 (major isomer)

S31

23 (minor isomer)

p188, lower													
Tue Mar 11 10:12:53 JST 2003													
p188, lower													
exp1 stdth													
SAMPLE													
date Mar 11 03													
DEC. & VT													
299.954													
H1													
COC13													
exp 0													
dm													
nmn													
c													
200													
ACQUISITION													
sfrq 299.955													
H1													
3.641													
PROCESSING													
32768													
wtfile													
4500.5													
proc													
2250 fn													
32768													
ft													
16													
react													
11.3													
werr													
1.359													
wexp													
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DEFILE
COMPT oxazolidinone-lower
DATIM Wed Apr 23 12:32:43 2003
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQ 27173.9 Hz
SCANS 111
ACQTM 1.206 sec
PD 1.794 sec
PW 6.0 us
IRNUC 1H
CTEMP 22.6 c
SLVT CDCl3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 23

23 (minor isomer)

24

exp1 std13ts

SAMPLE		DEC. & VT	
date	Dec	9 98	dfreq
solvent	CUC13	exp	dn
file			dfp
ACQUISITION			
strq	75.43j	dm	dof
trn	c13	dmm	w
et	0.873	dmf	11050
np	32768	PROCESSING	
sw	18761.7	lb	1.00
fb	9590	wfile	ft
bs	16	prac	32768
pw	10.0	fn	
d1	1.127		
tof	0	werr	mft
nt	1024	wexp	mft
ct	195	wbs	
alock	n	wnt	
gain	not used		
FLASS			
il	n		
in	n		
dp	y		
DISPLAY			
sd	-1896.0		
nd	18761.7		
vs	214		
sc	0		
mc	400		
hzmm	0.80		
is	500.00		
rfl	7711.7		
rtp	5815.7		
th	8		
ins	1.000		
nm			
no			
ph

535

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02

○

88

05

○

1

wittig
MePhBr, BuLi, THF, 0 C

exp1 st01h

SAMPLE DEC. & VT
date Dec 18 99 dfrq 299.954
solvent CDCl3 dn H1
file exp dof 0
ACQUISITION exp dm nm
sfrq 299.955 dnm c
tn H1 dmf 200
at 3.641 wtfile
np 32768 ft
sw 4500.5 prac 32768
fb 2250 fn
bs 16
pw 5.0 warr react
d1 1.359 wexp wft
tof 600.0 wbs
nt 15 wnt
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -30.2
wp 2999.5
vs 407
sc 0
wc 380
hzmm 7.89
is 12500.97
rfl 575.8
rfp 0
th 20
ins 1.000
nm cdc ph

25

S36

DDQ, pH
exo-olefine

exp1 st01h

SAMPLE		DEC. & VT	
date	Dec	20 98	
solvent	CDC13		
file	exp		
ACQUISITION			
sfrq	299.955		
tn	H1		
at	3.641		
np	32768		
sw	4500.5		
fb	2250		
bs	15		
pw	8.0		
d1	1.359		
tof	600.0		
nt	24		
ct	24		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-30.2		
wp	2999.5		
vs	361		
sc	0		
hc	360		
hzmm	7.89		
is	18411.29		
rfl	572.5		
rff	0		
th	20		
ins	1.000		
nm			
cdc			
ph			

26

338

```
DDQ, PHH
exo-olefine
exp1 std13c
```

SAMPLE		DEC. & VT	
date	Dec 20 98	dfrq	299.954
solvent	CDC13	dn	
file	exp	dhp	1
ACQUISITION		dof	0
sfreq	75.431	dm	yyy
tn	C13	dmm	w
at	0.873	dmf	11050
np	32768	PROCESSING	
sw	18761.7	lb	1.00
fb	9500	wtfile	
bs	15	proc	ft
pn	10.0	fn	32768
d1	1.127		
tof	0	werr	wft
nt	1024	wexp	wft
ct	901	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1890.3		
wp	18761.7		
vs	214		
sc	0		
wc	400		
hzmm	0.61		
is	500.00		
rfl	7706.0		
rfp	5815.7		
th	14		
ins	1.000		
nm	no ph		

26

239

27

m3, p76, acetyl

m3, p76, acetyl1

[illegible]

DFILE COMINT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\ECA600No.2\Kagawa\Kagawa_1H1
Kagawa Test
22-09-2004 14:16:00
1H
single-pulse-ex2
600.17 MHz
5.30 KHz
5.47 Hz
32768
1126126 Hz
8
2.9098 sec
5.0000 sec
6.45 usec
1H
24.3 g
ODCL3
0.00 ppm
0.12 Hz
36

28

DFILE
COMNT
DATIM
GENUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVMT
EXREF
BF
RGAIN

D:\MECA600No.2\Kagawa\Kagawa_13C_1
Kagawa_13C_Test
22-09-2004 14:55:49
13C
single_pulse_dec
150.92 MHz
8.52 kHz
1.74 Hz
32788
47343.49 Hz
800
0.8921 sec
2.0000 sec
3.42 usec
1H
25.5 °
CDCL3
77.00 ppm
0.12 Hz
80

28

DFILE C:\My Documents\kagawa\crutius.pro.als
 COMNT crutius.pro.
 DATIM Thu Oct 31 17:08:39 2002
 CENUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.0 Hz
 POINT 32768
 FREQU 7993.6 Hz
 SCANS 16
 ACQTM 4.099 sec
 PD 2.901 sec
 PW1 6.9 us
 IRNUC 1H
 CTEMP 25.4 C
 SLVNT CDCl3
 EXREF 0.00 ppm
 RF 0.50 Hz
 RGAIN 17

29

29

crutius.pro.

Tru Oct 31 18:12:22 JST 2002

INDEX	FREQUENCY (PPM)	Hz	HEIGHT
1	135.770	10241.2492	0.5
2	115.185	8763.9180	0.5
3	86.550	6528.4978	0.3
4	79.475	5994.8370	0.3
5	78.473	5919.2541	0.2
6	77.425	5840.2357	1.6
7	77.000	5808.1703	1.7
8	76.575	5776.1048	1.7
9	76.362	5760.0721	0.3
10	74.723	5636.3910	0.2
11	70.669	5330.8240	0.2
12	63.863	4918.7220	0.2
13	61.803	4661.8303	0.3
14	41.826	3139.8664	0.1
15	33.109	2497.4121	0.5
16	23.028	1737.0028	0.3
17	17.577	1325.8779	0.4
18	13.251	999.4974	0.2

crutius.pro.

exp1 std13c

date	Oct 31 02	DEC. & VT	299.954
solvent	CDCl3	dn	H1
file	exp	chn	1
ACQUISITION	def	def	0
sfrq	75.431	dm	yyy
tn	C13	dmm	w
at	0.873	dmf	9720
np	32768	PROCESSING	
sw	18751.7	lb	1.00
fb	9500	wtfile	
bs	16	proc	ft
pw	8.4	fn	32768
d1	1.127		
tof	0	werr	
nt	512	wexp	wft
ct	512	wbs	wft
alock		n	
gain	not used	n	
FLAGS			
il	n		
in	n		
dp	y		
sp	-8.3		
wd	15085.7		
vs	214		
sc	0		
wc	400		
hzmm	37.72		
is	100505.96		
rfl	7706.0		
rfp	5808.2		
th	13		
ins	1.000		
nm	no	ph

m5, p132, upper

exp2 std1h

SAMPLE B 04 dfrq 299.954
data May CDE13 dn H1
solvent exp dof 0
file exp dm mn
ACQUISITION dm c
sfrq 299.955 dm 200
tn H1
at 3.641
np 32768 wfile
sw 4500.5 proc ft
fb 2250 fn 32768
bs 16
pw 11.3 werr react
d1 1.359 wexp wft
tof 600.0 wds
nt 16 wnt
ct 16
clock n
gain not used
FLAGS
il n
in n
cp y
DISPLAY
sp -150.0
wp 2999.5
vs 203
sc 0
wc 380
hzmm 7.89
is 20396.19
rfl 573.0
rfp 0
th 20
jns
nm cdc ph 1.000

DEC. & VT 299.954
H1
0
mn
c
200

m5, p132, upper

Sat May 8 14:18:26 JST 2004

INDEX FREQUENCY (PPM) HZ HEIGHT
1 7.255 2179.2144 20.1
2 3.898 1169.1336 23.8
3 3.583 1074.6359 21.5
4 1.553 455.8953 8.4
5 1.013 303.8209 13.5
6 0.877 263.1649 14.0
7 0.062 48.6798 51.5
8 0.051 45.3833 8.5
9 0.000 0.0000 13.6

DFILE
COMMIT
DATIM
CENUC
EXMCD
CEFRQ
CBSET
CBFEN
POINT
FREQU
SCANS
AQCTM
PD
PWI
IRNUC
CTEMP
SUNVT
EXREF
BF
RGAIN

D:\AL400 NMR\kgawa\Teoc-oxaly-Cals
Teoc-oxaly-H
Thu Jul 08 17:58:08 2004
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27173.90 Hz
325
1.2059 sec
1.7940 sec
5.50 usec
IH 28.9 °
CDCl₃
77.00 ppm
0.12 Hz
23

30

3

m5, TBAF
Fri Jul 16 17:58:41 JST 2004

INDEX	FREQUENCY (PPM)	HZ	HEIGHT
1	7.271	2180.8627	5.8
2	5.736	1720.4514	2.9
3	5.170	1550.6953	2.9
4	5.147	1543.8277	3.5
5	4.901	1470.2074	4.3
6	4.878	1463.0652	4.5
7	3.941	1182.0446	28.4
8	3.577	1072.9877	27.7
9	3.474	1041.9463	3.0
10	1.017	305.1944	16.2
11	0.885	265.3626	17.0
12	0.000	0.0000	4.1

expi stdth
 date Jul 16 04
 solvent CDCl3
 file exp
 ACQUISITION
 sfreq 299.955
 tn H1
 at 3.641
 md 32768
 sw 4500.5
 fb 2250
 bs 15
 pw 11.3
 d1 1.358
 taf 600.0
 nt 16
 ct 16
 alock not used
 gain FLABS
 il n
 in n
 dp y
 sp -150.0
 wd 2999.5
 vs 203
 sc 0
 mc 380
 hzmm 7.89
 is 28082.32
 rfi 571.4
 rfp 0
 th 20
 ins 1.000
 nm cdc ph

DYAL400 NMR	ppm
oxalyl-C	100.40 MHz
oxalyl-C	125.00 KHz
oxalyl-C	1050.00 Hz
oxalyl-C	32768
oxalyl-C	27173.50 Hz
oxalyl-C	293
oxalyl-C	1.2059 sec
oxalyl-C	1.7940 sec
oxalyl-C	5.50 msec
oxalyl-C	26.2 °C
oxalyl-C	CDCl ₃
oxalyl-C	77.00 ppm
oxalyl-C	0.12 Hz
oxalyl-C	24

3

----- ACQUISITION PARAMETERS -----
File Name = id_spectrum.277
Author =
Sample ID = epoxide
Content = Single Pulse Experiment
Creation Date = 13-SEP-2005 08:35:34
Revision Date = 14-SEP-2005 13:05:31
Spec Site = ECP500
Spec Type = DELTA NMR
Data Format = 1D REAL
Dimensions = X
Dim Title = 1H
Dim Size = 18384
Dim Units = [ppm]
Scans = 16
Mod_return = 1
X_domain = 1H
X_offset = 5[ppm]
X_freq = 500.16241602[MHz]
X_sweep = 7.50750751[kHz]
Solvent = CHLOROFORM-D
Spin_get = 14[HZ]
Temp_get = 22.2[degC]
Recvr_gain = 23
Field_strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 3.75119936[MHz]

36

S49

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
epoxyalcohol
Sat Sep 10 18:49:32 2005
13C
BOB
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
96
1.2083 sec
1.7920 sec
5.50 usec
1H
21.7 c
CDCL3
77.00 ppm
0.12 Hz
24

36

पृ. ४५

exp1 std1h

SAMPLE		DEC. & VT	
date	May 24 02	dfrq	299.948
solvent	CDCl3	dn	H1
file	exp	daf	0
ACQUISITION		dm	nmn
sfrq	299.948	dmm	200
tn	H1	dmf	PROCESSING
at	3.641	wtfile	fl
np	32768	proc	32768
sw	4500.5	fn	react
fb	2250		wft
bs	16		
pw	10.5	werr	
d1	1.359	waxp	
tof	600.0	wbs	
nt	16	wnt	
ct	16		
clock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-30.2		
wp	2999.5		
vs	203		
sc	0		
wc	380		
hzmm	7.89		
is	10789.07		
rfl	572.2		
rpf	0		
th	20		
ins	1.000		
nm	cdc		
ph

551

m2, p50, recrystal

exp1 stdin

SAMPLE DEC. & VT
data Sep 20 01 dfrq 299.954
solvent CDCl3 dn H1 0
file exp dof 0
ACQUISITION dn nnn
sfrq 299.955 dnm c
tn H1 dnf 200
at 3.641 PROCESSING
nd 32768 wtflla
sw 4800.5 proc ft
fb 2250 fn 32768
bs 15
pw 10.5 wexp react
d1 1.359 wexp wft
tof 600.0 wds
nt 15 wnt
ct 15
alock n
gain not used
FLAG5
ll n
in n
ds y
DISPLAY
sp -30.2
wp 2999.5
vs 400
sc 0
wc 380
hzmm 7.88
is 21744.00
rfj 573.3
rfp 0
th 20
ins 1.000
nm cdc ph

41

41

m2, p87	expl	std13c																												
			SAMPLE		DEC. & VT																									
			date	Oct 13 01	dfreq	299.954																								
			solvent	CDCl3	dn	H1																								
			file	exp	exp	1																								
			ACQUISITION				0																							
			sfrq	75.431	dm	yyy	0																							
			tn	C13	dm	w																								
			at	0.873	dm	11050																								
			np	32768	dm	PROCESSING																								
			sw	18761.7	lb	1.00																								
			fb	9500	wtfile																									
			bs	16	proc	ft																								
			pw	10.0	fn	32768																								
			d1	1.127																										
			tof	0	warr																									
			nt	512	wexp	wft																								
			ct	320	wbs	wft																								
			alock	n	mnt																									
			gain	not used																										
			flags																											
			al	n																										
			in	n																										
			dp	y																										
			DISPLAY																											
			sp	-8.3																										
			wp	15085.7																										
			vs	247																										
			sc	0																										
			wc	400																										
			hzmm	37.72																										
			is	100142.38																										
			rfl	7708.3																										
			rfp	5808.2																										
			th	5																										
			ins	1.000																										
			nm	no	ph																									

m2, p87	Sat Oct 13 10:08:46	JST 2001										
			INDEX	FREQUENCY (PPM)	HZ							
			1	135.648	10232.0577	3.5						
			2	135.557	10225.1865	3.3						
			3	132.900	10024.7774	0.8						
			4	132.718	10011.0351	0.8						
			5	130.046	9809.4808	2.0						
			6	130.016	9807.1904	2.2						
			7	127.936	9650.2987	8.1						
			8	109.171	8234.8380	1.0						
			9	85.715	6465.5121	2.0						
			10	77.425	5840.2357	2.3						
			11	77.000	5808.1703	2.4						
			12	76.575	5776.1048	2.3						
			13	76.180	5746.3297	1.3						
			14	67.759	5111.8918	1.0						
			15	63.670	4802.6892	1.0						
			16	39.470	2977.2488	1.4						
			17	39.438	2899.3755	1.4						
			18	26.702	2014.1399	6.1						
			19	25.413	1992.3812	1.1						
			20	25.442	1919.0888	1.2						
			21	19.035	1435.8166	1.2						
			22	11.140	840.3153	1.6						

INDEX	FREQUENCY (PPM)	H _z	HEIGHT
1	135.648	10232.0577	3.5
2	135.557	10225.1865	3.3
3	132.500	10024.7774	0.8
4	132.718	10011.0351	0.8
5	130.046	9809.4808	2.0
6	130.016	9807.1904	2.2
7	127.936	9650.2987	8.1
8	109.171	8234.8380	1.0
9	85.715	5455.5121	2.0
10	77.425	5840.2357	2.3
11	77.000	5808.1703	2.4
12	75.575	5775.1048	2.3
13	75.180	5746.3297	1.3
14	67.759	5111.8918	1.0
15	63.670	4902.6902	1.0
16	39.470	2977.2488	1.4
17	38.438	2899.3755	1.4
18	26.702	2014.1399	6.1
19	25.413	1992.3812	1.1
20	25.442	1919.0888	1.2
21	19.035	1435.8166	1.2
22	11.140	840.3153	1.5

m2.p87

Sat Oct 13 10:08:46 JST 2001

m3.p48

exp1 stdih

SAMPLE DEC. & VT
date Jun 1 02 dfrq 299.948
solvent CDCl3 dn H1
file exp 0
ACQUISITION dm nm
sfrq 299.949 dnm c
tn H1 dnf 200
at 3.641 wtfile
nd 32768
sw 4500.5 proc ft
fb 2250 fn 32768
bs 16
pw 10.6 werr react
d1 1.359 wexp wft
tof 600.0 wbs
nt 8 wnt
ct 8
alock not used
gain FLAG
il n
in n
op y
DISPLAY
sp -30.2
wb 2999.5
vs 203
sc 0
wc 380
hzmm 7.89
is 11215.29
rfl 572.2
rfp 0
th 20
ins 1.000
nm cdc ph

42

S54

42

m3.p4B

Sat Jun 1 15:48:59 JST 2002

INDEX	FREQUENCY (PPM)	Hz	HEIGHT
1	159.364	12020.7244	1.1
2	130.078	9811.5437	1.1
3	129.273	9750.9484	8.8
4	113.878	8589.7209	9.9
5	77.607	5853.8505	1.9
6	77.425	5840.1082	1.7
7	77.000	5808.0427	1.8
8	76.575	5775.9773	1.7
9	73.630	5553.8095	2.8
10	70.198	5294.9955	2.8
11	64.824	4889.5955	2.6
12	55.107	4156.8719	1.1
13	37.829	2853.4402	2.7
14	14.403	1086.4047	2.7
15	12.232	922.6418	3.3

exp1 std13c
SAMPLE
date Jun 1 02 dfreq 299.948
solvent CDCl3 dn H1
file exp dhp 1
ACQUISITION ddf 0
sfreq 75.429 dm yyy
tn 613 dm W
at 0.873 dm 11050
np 32768
sw 18761.7 lb wtfile 1.00
fb 16 proc ft
bs 10.0 fn 32768
pw d1 1.127
tof 0 werr
nt 256 wexp wft
ct 256 wbs wft
alock n
gain not used
flags

al n
in n
dp y

DISPLAY

sp -8.4

wp 15085.7

vs 193

sc 0

wc 400

hzmm 37.71

ls 62048.23

rfl 7733.5

rfd 5808.0

th 20

ins 1.000

nm no ph

S55

DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\AL400 NMR\Kagawa\M-cyclopropane-C
M-cyclopropane-C
Sat Oct 16 12:32:08 2004
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27173.90 Hz
311
1.2059 sec
1.7940 sec
5.50 usec
1H
24.6 c
CDCL3
77.00 ppm
0.12 Hz
23

D:\AL400 NMR\kagawa\olefine-MPM\als
olefine-MPM
Sat Jul 10 18:38:01 2004

NON	389.85 MHz		
	124.00 KHz		
	10500.00 Hz		
	32768		
	7993.80 Hz		
	18		
	4.0993 sec		
	2.9010 sec		
1H	5.90 used		
	28.4 c		
CDCl3			
	0.00 ppm		
	0.10 Hz		
	17

43

DFILE
CONAT
DATIM
CENUC
EXMOD
EXPRQ
OBSEY
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\AL400 NMR\kgawa\olefine-MPM-Cals
olefine-MPM-C
Sat Jul 10 18:57:37 2004
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32788
27773.90 Hz
302
1.3059 sec
1.7940 sec
9.50 usec
IH 25.0 c
CDCL3
77.00 ppm
0.10 Hz
24

43

S59

DEFILE
C:\listar
COMNT
DATM 24-09-2004 18:15:48
CNUC
EXMOD
OBFRQ
CSSET
CSFN
POINT
FREQ
SCANS
AQCTM
PD
F1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\ECA800\No.2\Kagawa\gawa_C_dlistar.1
C_dlistar
24-09-2004 18:15:48
single pulse dec
150.92 MHz
8.52 kHz
1.74 Hz
32768
47348.49 Hz
800
0.8921 sec
2.0000 sec
3.42 usec
1H 25.6 g
CDCL3
77.00 ppm
0.12 Hz
60

46

S61

DFILE
COMNT
DATM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
diethyl-dimalonate-OH-up
Tue Jul 05 07:42:54 2005
1H
NON

399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
8
4.1001 sec
2.9010 sec
5.80 usec
1H
20.3 c
CDCL3
7.26 ppm
0.12 Hz
13

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27210.88 Hz
137
1.2042 sec
1.7940 sec
5.50 usec
1H
20.4 c
CDCL3
77.00 ppm
0.12 Hz
23

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
diethyl-dimalonate-OH-low
Tue Jul 05 07:56:28 2005
1H
NON

399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
8
4.1001 sec
2.9010 sec
5.80 usec
1H
20.0 c
CDCL3
7.26 ppm
0.12 Hz
14

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\WINNMR98\COMMON\DEFAULT.ALS
diethyl-dimalonate-OH-low
Tue Jul 05 08:37:22 2005
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27210.88 Hz
147
1.2042 sec
1.7940 sec
5.50 usec
1H
20.0 c
CDCL3
77.00 ppm
0.12 Hz
25

DFILE
COMMT
DATM
OBNUC
EXMOD
OBFRQ
ORSET
ORFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
RF
RGAIN

NON
399.95 MHz
124.00 KHz
10500.00 Hz
32768
7993.80 Hz
18
4.0983 sec
2.9010 sec
5.90 usec
1H
27.8 c
CDCl₃
7.24 ppm
0.12 Hz
18

(4S,5R,6R)-5,6-Dimethyl-4-(
(phenylselenylmethyl)-
tetrahydro-2H-pyran-2-one

DFILE
COMNT
DATIM
CINUC
EXMOD
OBFREQ
OBSSET
OBSFEN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\XAL400 NMR\Kagawa\alpha-lactone-C-als
alpha-lactone-C
Fri Jul 09 17:58:02 2004
13C
BCM
100.40 MHz
125.00 KHz
105000.00 Hz
32788
27173.90 Hz
334
1.2059 sec
1.7940 sec
5.50 usec
IH
27.8 °
CDCL₃
77.00 ppm
0.12 Hz
23

(4*S*,5*R*,6*R*)-5,6-Dimethyl-4-(
(phenylselenenylmethyl)-
tetrahydro-2*H*-pyran-2-one

DFILE
COMNT
DATIM
EXMOD
OBFREQ
OBSSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\ECA600\No.2\kagawa\gawa_Sa.1
Se H
24-09-2004 18:41:30
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
32768
11261.25 Hz
16
2.9088 sec
5.0000 sec
8.45 usec
1H
24.4 °C
CDCl₃
0.00 ppm
0.12 Hz
38

31

DETE
COMET
DATE
13C
EXHND
EXHND
OBSCT
OBSCT
OBSCT
POINT
FREQ
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\ECA600No.2\Kagawa\gawa.C, Se.1
Se. C
24-09-2004 19:27:07
13C
single pulse dec
150.02 MHz
8.52 kHz
1.74 Hz
32758
47348.49 Hz
1000
0.0821 sec
2.0000 sec
3.42 usec
1H 25.0 c
CDCL3
77.00 ppm
0.12 Hz
80

31

S70

```

===== ACQUISITION PARAMETERS =====
File Name      = 1d_spectrum.616
Author         =
Sample ID      = left
Content        = Single Pulse Experiment
Creation Date   = 23-SEP-2005 11:09:19

Revision Date  = 24-SEP-2005 15:52:37
Spec Site      = ECP500

Spec Type      = DELTA_XMR
Data Format     = 1D NMR
Dimensions     = X
Dim title      = 1H
Dim size       = 16384
Dim units      = [ppm]
Scans          = 4
Mod return     = 1
X domain       = 1H
X offset       = 5 [ppm]
X freq         = 500.16341602 [MHz]
X sweep        = 7.50750751 [MHz]
Solvent        = CHLOROFORM-D
Spin get       = 15 [Hz]
Temp_get       = 22.9 [C]
Recrv_gain     = 23
Field_strength = 11.7473579 [T]
Filter_mode     = BUTTERWORTH
Filter_width    = 3.751199936 [MHz]

```

2

S71

DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\AL400 NMR\Kagawa\mix-Sn-Cals
mix-Sn-C
Fr Aug 27 15:54:04 2004
13C
BOB
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27173.90 Hz
297
1.2059 sec
1.7940 sec
5.30 usec
1H
24.9 °
CDCl₃
77.00 ppm
0.12 Hz
24

2

S72

DFILE
COMINT
DATIM
OSNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
OTEMP
SLVNT
EXREF
BF
RGAIN

D:\AL400 NMR\Kagawa\coupling-Hals
coupling-H
Sat Feb 12 15:08:45 2005
1H
NON
399.65 MHz
124.00 KHz
10500.00 Hz
32768
7993.80 Hz
16
4.0993 sec
2.9010 sec
5.90 usec
1H 22.4 °
ODCL3
0.00 ppm
0.12 Hz
12

DFILE
CONNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFTN
POINT
FREQU
SCANS
AQTM
PD
PWT
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

D:\AL400 NMR\Kagawa\coupling-Cals
coupling-H
Sat Feb 12 15:29:44 2005
13C
ECM
100.40 MHz
125.00 KHz
10500.00 Hz
32788
27173.90 Hz
392
1.2059 sec
1.7940 sec
5.50 uscs
1H 22.2 g
CDCL3
77.00 ppm
0.12 Hz
23

C:\THERM\DATA\COMMON\DEFAULT.D

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Wed Jun 22 07:32:43 2005

1H
NON

399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
8
4.1001 sec
2.9010 sec
5.80 usec
23.3 C
7.16 ppm
0.12 Hz
15

48

S75

----- ACQUISITION PARAMETERS -----
File Name = benzosta.3
Author =
Sample ID = 050908
Content = Single Pulse with Broad
Creation Date = 9-SEP-2005 08:29:13
Revision Date = 10-SEP-2005 13:02:26
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = 1D REAL
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 19000
Mod Return = 1
X_domain = 13C
X_offset = 100 [ppm]
X_freq = 125.77787547 [MHz]
X_sweep = 31.4654088 [kHz]
Solvent = BENZENE-D6
Spin_get = 17 [Hz]
Temp_get = 23.2 [dC]
Recvr_gain = 29
Field_strength = 11.7473579 [T]
Filter_mode = BUTTERWORTH
Filter_width = 15.72066241 [kHz]

D:*ECA6000\2\kagawa
 14-03-2005 21:10:38
 1H single_pulse, ex2
 600.17 MHz
 5.30 KHz
 5.47 Hz
 32768
 11261.26 Hz
 256
 2.9098 sec
 5.0000 sec
 6.75 usec
 25.3 c
 7.15 ppm
 0.12 Hz
 50
 DF IL COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 BF
 RGAIN

DFILE D:\VAL400 NMR\kagawa\m7, p1, major-C
COMNT m7, p1, major-C
DATIM Fri Mar 18 08:58:56
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27173.90 Hz
SCANS 13909
AQTM 1.2059 sec
PD 1.7940 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT C6D6
EXREF 128.00 ppm
BF 0.41 Hz
RGAIN 18

----- ACQUISITION PARAMETERS -----
File Name = mycalamide_A-H.3
Author =
Sample ID = 8/9
Content = Single Pulse Experiment
Creation Date = 9-AUG-2005 21:42:44
Revision Date = 11-AUG-2005 01:35:34
Spec Site = ECP500
Spec Type = DELTA NMR
Data Format = 1D REAL
Dimensions = 1
Dim Title = 1H
Dim Size = 16384
Dim Units = [ppm]
Scans = 64
Mod return = 1
X domain = 1H
X offset = 5 [ppm]
X freq = 500.16241602 [MHz]
X sweep = 7.50750751 [kHz]
Solvent = CHLOROFORM-D
Spin get = 16 [Hz]
Temp set = 23.1 [C]
Recvr gain = 30
Field strength = 11.7473579 [T]
Filter mode = BUTTERWORTH
Filter width = 3.75119936 [kHz]

500.0 400.0 300.0 200.0 100.0 0

(Millions)

9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0

X : parts per Million : 1H

----- ACQUISITION PARAMETERS -----
 File Name = mycalamide_A.4
 Author =
 Sample ID = 050903
 Content = Single Pulse with Broad
 Creation Date = 5-SEP-2005 06:53:25
 Revision Date = 6-SEP-2005 12:44:34
 Spec Site = ECP500
 Spec Type = DELTA NMR
 Data Format = ID REAL
 Dimensions = X
 Dim Title = 13C
 Dim Size = 32768
 Dim Units = [ppm]
 Scans = 65000
 Mod return = 1
 X_domain = 13C
 X_offset = 100 [ppm]
 X_freq = 125.77787547 [MHz]
 X_sweep = 31.44654088 [Hz]
 Solvent = CHLOROFORM-D
 Spin_get = 15 [Hz]
 Temp_get = 25.1 [deg]
 Recvz_gain = 30
 Field_strength = 11.7473579 [T]
 Filter_mode = HETTERORTH
 Filter_width = 15.72066221 [kHz]

mycalamide A 1a

S80

(Billions)

X : parts per Million : 13C