NMR Spectra

Metal-Catalyzed Cycloisomerization of En-Yne Functionalities via a 1,3-Alkylidene Migration

Ming-Yuan Lin, Arindam Das and Rai-Shung Liu*

Department of Chemistry, National Tsing-Hua University, Hsinchu, Taiwan, ROC.
..... *E-mail: rsliu@mx.nthu.edu.tw*

2

Current Data Parameters
NAME: Yvan-130
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20060307
Time: 13:55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 16
DS: 0
SWH: 17019.274 Hz
F2: 500.136188 MHz
F1: 101.626188 MHz
AQ: 1.363188 sec
RG: 32
DM: 41.600 usec
DE: 8.50 usec
TE: 300.2 K
D1: 1.00000000 sec
d11: 0.00000000 sec
d12: 0.00000000 sec
d13: 0.00000000 sec
d14: 0.00000000 sec
d15: 0.00000000 sec
d16: 0.00000000 sec
d17: 0.00000000 sec
d18: 0.00000000 sec
d19: 0.00000000 sec
d20: 0.00000000 sec
d21: 0.00000000 sec
d22: 0.00000000 sec
d23: 0.00000000 sec
d24: 0.00000000 sec
d25: 0.00000000 sec
d26: 0.00000000 sec
d27: 0.00000000 sec
d28: 0.00000000 sec
d29: 0.00000000 sec
d30: 0.00000000 sec
d31: 0.00000000 sec
d32: 0.00000000 sec
d33: 0.00000000 sec
d34: 0.00000000 sec
d35: 0.00000000 sec
d36: 0.00000000 sec
d37: 0.00000000 sec
d38: 0.00000000 sec
d39: 0.00000000 sec
d40: 0.00000000 sec
d41: 0.00000000 sec
d42: 0.00000000 sec
d43: 0.00000000 sec
d44: 0.00000000 sec
d45: 0.00000000 sec
d46: 0.00000000 sec
d47: 0.00000000 sec
d48: 0.00000000 sec
d49: 0.00000000 sec
d50: 0.00000000 sec
d51: 0.00000000 sec
d52: 0.00000000 sec
d53: 0.00000000 sec
d54: 0.00000000 sec
d55: 0.00000000 sec
d56: 0.00000000 sec
d57: 0.00000000 sec
d58: 0.00000000 sec
d59: 0.00000000 sec
d60: 0.00000000 sec
d61: 0.00000000 sec
d62: 0.00000000 sec
d63: 0.00000000 sec
d64: 0.00000000 sec
d65: 0.00000000 sec
d66: 0.00000000 sec
d67: 0.00000000 sec
d68: 0.00000000 sec
d69: 0.00000000 sec
d70: 0.00000000 sec
d71: 0.00000000 sec
d72: 0.00000000 sec
d73: 0.00000000 sec
d74: 0.00000000 sec
d75: 0.00000000 sec
d76: 0.00000000 sec
d77: 0.00000000 sec
d78: 0.00000000 sec
d79: 0.00000000 sec
d80: 0.00000000 sec
d81: 0.00000000 sec
d82: 0.00000000 sec
d83: 0.00000000 sec
d84: 0.00000000 sec
d85: 0.00000000 sec
d86: 0.00000000 sec
d87: 0.00000000 sec
d88: 0.00000000 sec
d89: 0.00000000 sec
d90: 0.00000000 sec
d91: 0.00000000 sec
d92: 0.00000000 sec
d93: 0.00000000 sec
d94: 0.00000000 sec
d95: 0.00000000 sec
d96: 0.00000000 sec
d97: 0.00000000 sec
d98: 0.00000000 sec
d99: 0.00000000 sec
d100: 0.00000000 sec

— 19.329
— 25.116
— 22.047

— 61.242
— 64.464
— 67.258
— 72.816
— 76.679
— 77.000
— 77.305
— 83.001

— 118.807
— 128.716
— 132.198
— 138.947

13C OBSERVE
exp2 std13c
SAMPLE
date Feb 29 2006 DEC. & VT
solvent CDCl3 dn 400.454
file CUC13 exp 42
ACQUISITION
sfrq 100.705 dm yyy
tn C13 dm
at 0.435 dm 8000
sp 25188.9 lb PROCESSING 3.00
fb 13800 wfile
bs 4 proc ft
tpwr 58 fn not used
pw 6.5
di 2.000 werr
or 20000 wexp
ct 1000 wps
nt 40 wnt
a lock n
gain not used
flags n
ll n
ln n
dp n
DISPLAY
sp -0.2
wp 20136.2
vs 45
sc 0
h2mm 250
ls 500.00
rfp 8852.9
th 7753.4
ins 8
nm 100.000
ph

Yuan-121
exp2 std13c

SAMPLE		DEC. & VT	
date	Feb 19 2006	dfrq	400.454
solvent	CDCl3	dn	H1
file	exp	ddr	4
ACQUISITION			
sfrq	100.705	dm	yyv
tn	0.635	dmf	w
at	32000	lb	8000
pw	25186.9	wfllt	3.00
bs	13800	fn	not used
tpwr	58	fn	not used
pw	6.5	fn	not used
d1	2.000	werr	
tof	2000.0	wexp	
ct	1000	wps	wft
alock	0	wnt	
gain	not used	n	
FLAGS			
fl	n	n	
in	n	n	
dp	DISPLAY	y	
sp	-0.2		
wp	22152.3		
vs	50		
sc	0		
wc	250		
l2mm	1.46		
l2mm	50.46		
rf1	8860.8		
rfp	7753.4		
th	2		
ins	100.000		
nm	no	ph

29.941
29.834
24.627
22.444
22.260
16.779

83.673
82.970
77.321
77.000
76.695
72.786
72.603
62.586
62.494

142.092
141.039
137.466
137.023
133.633
132.492
131.358
131.114
128.106
128.029
127.525
127.128
127.037
126.701
125.495

2.779
2.757
2.131
2.130
2.128
1.813
1.804
1.796
1.789
1.780
1.764
1.551

S. 315
 S. 314
 S. 022
 S. 019
 S. 015
 S. 011
 S. 008

Current Data Parameters
NAME YUM-124
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060221
Time 15:55
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
PCPDPRG2 zgpg30
SOLVENT CDCl3
NS 328
DS 0
SWH 45045.467 Hz
FIDRES 0.34655 Hz
AQ 0.333748 sec
RG 1024
IM 11.100 usec
DE 6.50 usec
TE 300.2 K
T1 3.00000000 sec
d11 0.01000000 sec
DELTA 2.90000010 sec
PCPRG2 waltz16
PCPDPRG2 waltz16
NUC1 13C
P1 4.50 usec
PL1 0.00 dB
SFO1 150.7609749 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 19.00 dB
PL12 13.30 dB
PL13 14.00 dB
SFO2 500.13605475 MHz
F2 - Processing parameters
SI 32768
SF 150.7444032 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 0.50
1D MR plot parameters
CX 20.00 cm
CY 4.00 cm
CT 20.00 cm
F1 3048.88 Hz
F2 0.00000000 Hz
F2 0.00 Hz
F2 10.00000000 ppm/cm
H2CN 1507.44397 Hz/cm

1.825
1.821
1.817
1.809
1.551
1.546

2.794
2.779
2.755

5.439
5.435
5.432
5.408
5.404
5.400

DEC	8	VI	400	454	H1	H2
7.388						
7.384						
7.380						
7.375						
7.370						
7.367						
7.363						
7.358						
7.352						
7.345						
7.343						
7.338						
7.332						
7.329						
7.324						
7.313						
7.240						
7.090						
7.067						
7.050						
7.031						

[illegible]

2.751
2.749
2.702
2.697
2.684
2.679
2.670
2.665
2.660
2.654
2.630
2.523
2.512
2.493
2.488
2.038
2.021
2.019
1.983
1.963
1.807
1.794
1.792
1.765
1.775
1.541
1.538

6.105
6.105

7.239
7.199
7.180
7.164
7.124
6.995

exp9 stdlh
SAMPLE
date Feb 16 2006 DEC. & VT
solvent CDC13 dn- 400.454
file H1
ACQUISITION exp 42
sfrq 400.455 dm nnn
tn H1 dmm
ns 2.600 dmf
sw 32016 lb PROCESSING 0.10
fb 3400 wtfile
bs 4 proc ft
tpwr 58 fn not used
pwr 6.0
tq 1000 werr
tof 5000 wbsp
nt 100 wbs
ct 12 wnt wft aph
alock gain not used
flags
il n
in n
dp y
DISPLAY
sp -200.4
wp 4204.5
vs 45
wc 0
h2mm 25.0
h2mm 2.16
ls 126.80
rf1 495.3
rfp 0
in 3
ns 100.000
nm ph

33.254
29.483
29.162
23.360
23.283

77.305
77.000
76.679

142.474
136.794
135.878
134.167
128.976
126.197
124.899
122.836

exp2 std13c
date Feb 16 2006
time 12:13
file CUC13
solvent CDCl3
exp 4
dof 6
sfrq 100.705 dm vvv
tn C13 dm
at 0.635 dm
np 32000 lb PROCESSING 8000
w 251883 lb
fs 1580 wfile 3.00
bs 4 proc
tpwr 58 fn not used
pw 6.5
dl 2.000 werr
tof 2000.0 wexp
ct 100 wps
cl 12 wnt
alock n
gain not used
flags
f1 n
f2 n
f3 n
dp DISPLAY y
sp -0.2
wp 20136.2
vs 45
sc 0
wc 250
ic 86.00
lc 500.00
rfi 8840.6
rfp 7753.4
th 4
ins
nm 100.000
ph

STANDARD 1H OBSERVE

expt pulse sequence: aldh

Sample

date	file	1.06	dn	HI	DEC. & UT
solvent	CDC13	dof	0	min	
file	exp	da	286	38	
ACQUISITION	da	286	38		
afreq	399.952	def	286	38	
ts	HI	dpr	38		
at	1.365	PROCESSING			
np	16384	lb	0.38		
au	5999.7	wtfile			
fb	3390	proc	ft		
ba	4	fn	not used		
tpur	55	meth	f		
pu	7.8				
dl	1.000	uerr			
tof	8	uerr			
nt	100	uba			
ct	88	unt			
clock	n				
gain	not used	sp	286.5		
FLGS		sp	4190.3		
il	n	uc	6		
in	n	ac	8		
dp	u	uc	258		
ha	an	hnam	2.43		
		ia	67.76		
		rfl	3477.8		
		rfp	2895.7		
		th	7		
		ina	1.000		
		nr	ph

Current Data Parameters
NAME Yvan-128
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060301
Time 19.20
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 500
DS 4
SWH 65045.847 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
GB 11.100 usec
PC 1.0000000 sec
YC 320.4 K
O1 3.00000000 sec
d11 0.03000000 sec
DECTA 2.90000010 sec
MCBET 0.00000000 sec
MCURT 0.01000000 sec
===== CHANNEL F1 =====
NUC1 13C
P1 4.50 usec
PL1 0.00 dB
SFO1 150.7609769 MHz
===== CHANNEL F2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 11.30 dB
PL13 14400 dB
SFO2 599.5029575 MHz
F2 - Processing parameters
SI 32768
SF 150.744023 MHz
WDW EM
SS 3.00 Hz
LB 0
GB 0
PC 0.50
1D MR plot parameters
SI 32768
SF 150.744023 MHz
WDW EM
SS 3.00 Hz
LB 0
GB 0
PC 0.50
F1 200.000 ppm
F1 30148.88 Hz
F2 0.000 ppm
F2 0.000 Hz
F3 10.0000 ppm/cm
F3 1507.44397 Hz/cm

555.5
555.5

```

exp1 std1h
SAMPLE
date Feb 14 2018
solvent CDCl3
file CDCl3
acquisition exp 42
sfreq 400.555
in H1
at 2.564
np 32016
pw 6005.6
bs 3414
bs proc
tpwr 50
dl 1.000
tor 500.0
ct 100
clock not used
main not used
flags n
in n
in n
sp -200.4
wp 4204.5
v6 37
ec 251
h1 15.82
ls 62.98
rfq 3385.3
rfp 2693.3
th 11
ns 2.000
na ph
  
```

Current Data Parameters
 NAME Activator-15
 EXPTNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060305
 Time 18.07
 INSTRUM spect
 PULPROG zgpg30
 TD 32768
 SCAVST CQCL3
 DS 16
 SS 0
 SFR 12015.230 Hz
 FIDRES 0.366778 Hz
 AQ 1.3613988 sec
 RG 128
 DW 41.600 usec
 DE 6.50 usec
 TE 323.7 K
 D1 1.50000000 sec
 DELT 0.00000000 sec
 ACQRES 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.10 usec
 PL1 0.00 dB
 SFO1 599.5024580 MHz

F2 - Processing parameters
 SI 32768
 SF 599.5000271 MHz
 WDW Hanning
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 30.00 cm
 F1P 10.000 ppm
 F2 5995.00 MHz
 F2P -0.500 ppm
 ZF 299.75 Hz
 ZFREQ 0.552500 ppm/cm
 HZCN 314.71749 Hz/cm

CC(C)=Cc1ccc2c(c1)c3ccccc3c2C4=CC=CC=C4

1.460
1.455
1.444
1.410
1.382
1.373
1.356
1.281
1.242
0.956
0.945
0.941
0.934
0.928
0.923
0.916
0.899
0.884
0.867
0.859
0.854
0.846
0.842
0.838
0.825
0.821

7.772
7.749
7.731
7.649
7.577
7.573
7.556
7.425
7.421
7.405
7.385
7.382
7.248
7.235
6.544
6.504
6.385
6.343
6.326

das	expl	etdih
SAMPLE	Feb 68	STDIH
solvent		
ACQUISITION		
sfrsfq		
at		
eat		
np		
pw		
bs		
tper		
pw		
dldl		
stef		
ctt		
malock		
main		
in		
dp		
sp		
ep		
sc		
wc		
hzwa		
is		
rpl		
tl		
tp		
is		
nm		
ph