Application of a Domino Intramolecular Enyne Metathesis/Cross Metathesis Reaction to the Total Synthesis of 8-*epi*-Xanthatin

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Supporting Information

YDk-77

STANDARD IN OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr8"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz

65 repetitions
OBSERVED F1 400.2669779 MHz
DATE ACQUIRED
LINE BROADENING 0.1 Hz
FT size 32768
Total time 10 min, 59 sec

VDK-54
high RF spot

exp2 stdih

SAMPLE		DEC. & VT	
date	May 31, 2005	dfrq	400.268
solvent	CDCl3	dm	H1
file	exp	dpr	30
ACQUISITION		dof	0
tn	400.270	dm	nm
at	2.858	dms	C
np	45732	dmf	200
sw	8000.0	lb	PROCESSING
fb	not used	wfile	0.10
ts	58	proc	ft
dpr	2.0	fn	not used
d1	2.000	werr	
tof	1185.0	wexp	
nt	64	wbs	
ct	64	wnt	
alock	not used		
gain	n		
ll	n		
in	n		
dp	y		
DISPLAY		sp	147.6
wp	3222.9	vs	151
sc	0	wc	250
hzm	12.89	ls	520.19
rf1	3703.4	rf2	2905.2
ins	20	ins	3.000
nm	cdc	ph

VDK-57

exp2 std1h

SAMPLE DEC. & VT
 date Jun 2 2005 dfrq 400.269
 solvent CDCl3 dn 30
 file ACQUISITION exp H1
 sfrq 400.269 dm 0 nm
 tn 2.856 dm 200
 np 32000 lb PROCESSING 0.10
 pw 3002.2 wfile
 bs not used
 tpwr 58 fn not used
 pw 2.0
 d1 2.000 werr
 tof 169.9 wexp
 nt 64 wbs
 ct 64 wnt
 alock
 gain not used
 il n
 in n
 dp y
 DISPLAY
 sp 151.3
 wp 2903.7
 vs 151
 sc 0
 wc 250
 izmm 11.20
 iz 385.22
 rfl 3518.5
 rfp 2905.9
 th 20
 ins 3.000
 nm cdc ph

VOK-81

exp1 s2pu1

SAMPLE	DEC. & VT
date Jul 1 2005	dfrq
solvent CCl ₄	dn
file ACQUISITION	dprw
sfrq 489.349	dof
at H1	dm
nt 4.865	dmm
np 64000	dseq
sw 6577.3	dres
fb 4000	homo
ss 34	temp
tpwr 5.0	lb
pw 24	wf file
d1 2.000	proc
tcf 297.6	f
toft 64	math
ct 64	werr
aalock n	wexp
gain 40	wps
FLAGS	n
l l	n
dp dp	y
hs nm	nm
DISPLAY	
rsp -249.8	
wp 5243.1	
v 133	
sc 0	
wc 250	
zmm 249.8	
iszm 3498.03	
rfl 4129.8	
ff 3615.3	
th 3615.7	
ins 1.000	
ai cdc ph

JB193

exp1 s2pu1

[illegible]

jw

VDK-70
exp1 s2pu1
SAMPLE
date Jun 24 2005 DEC. & VT
solvent CDCl3 dfrq 499.349
file CDC13 dn 30
ACQUISITION exp 30
sfrq 499.349 dm rnm
tn H1 200
at 4.865 dmf c
np 64000 dseq
w 6577.1 dres 1.0
fs 4000 hres n
bs 32 temp 27.0
ss 4 PROCESSING 0.10
tpwr 54 lb
pw 2.0 wtfile
d1 2.000 proc ft
tof -82.2 fn not used
nt 64 math f
ct 64
clock werr
gain flags 40 wexp
il n wbs
in n wnt
dp y
hs nn
DISPLAY
sp 248.8
wp 5245.1
wv 125
sc 250
wc 250
h2mm 20.97
is 2973.71
rfl 4508.6
rfp 3615.3
th
ins 1.000
al cdc ph

5

VDK-88_carbonylation_product

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-40088 "nmr6"

Relax. delay 2.000 sec

Pulse 16.4 degrees

Acq. time 2.856 sec

Width 5602.2 Hz

22640 repetitions

026400000.2669779 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 0 sec

14

VDK-89
f#20-30

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-400BB "nmr6"

Relax. delay 2.000 sec

Pulse 16.4 degrees

Acq. time 2.856 sec

141th S802.2 Hz

141th S802.2 Hz

141th S802.2 Hz

OBSERVE H1 400.2668779 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 43 min, 58 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
93 repetitions
OBSERVED FID 00.2669779 MHz
DATA ACQUISITION
LINE PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 43 min, 58 sec

8-epi-xanthatin

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-400BB "nmr6"

Relax. delay 2.000 sec

Pulse 22.5 degrees

Acq. time 1.280 sec

Width 25188.9 Hz

4048 repetitions

Observed C13, 100.6471877 MHz

Observed H1, 400.2683955 MHz

Processing

Continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FT size 65536

Total time 8 hr, 28 min, 26 sec