REVISED

Supporting Information for

Monoalkylation of C₆₀ and C₇₀ with Zn and Active Alkyl Bromides

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B. ^1H NMR of 5a	S50
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D. MS of 5a	S52

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B. ^1H NMR of 5b	S54
C. ^{13}C NMR of 5b	S55
D. MS of 5b	S56

HPLC-5PBB-100% Tol

UV-Vis

Compound 3a

compound 3a

56

compound 3a

WZVH3-1,2Dichlorobenzene-13C-110201

exp4 std13c

exp4 std13c

SAMPLE DEC. & VT
date 1 2001 dfrq 399.729
solvent Benzene dn H1
file exp 42
ACQUISITION
100.521 dm yyy w
tn C13 dmm 8135
at 0.800 dmf
np 40000 dseq
sw 25000.0 dres 1.0
fb 14000 homo n
bs 16 PROCESSING
57 lb 1.00
tpwr 6.0 wfile
pw 1.000 proc ft
dl 0 fn. not used
tof 0 math
nt 100000 verr
ct 30208 wexp
a-lock not used wbs
gain not used wnt

FLAGS n n
nn y
nn

DISPLAY
sp -2988.9
wp 25000.0
vs 1642
sc 0
wc 250
hzmm 4.01
ts 7.45
rfl 2988.9
rfp 0
th 39
ins 100.000
nm ph

[Mass Spectrum]
 Date : 21-Sep-2001 15:31
 Sample: WZMG68b2
 Note : (NBR) #01-0808
 Inlet : Direct Ion Mode : FAB-
 Spectrum Type : Normal Ion (MF+Linear)
 RT : 0.41 min Scan# : (3.6)
 BP : m/z 720.3016 Int. : 92.73
 Output m/z range : 640.9495 to 961.4243
 3889206
 100
 90
 80
 70
 60
 50
 40
 30
 20
 10
 0

Cut Level : 0.00 %
 720.3

compound 3a

HPLC-Buckyprep-100% Toluene

UV-Vis

Compound 3b

wjwg506-7065230CD43-082001

420823-40mer1-13pt11-12-1R

exp2 s2pu1

SAMPLE		DEC. & VT	
date	Aug 20 2001	dfrq	399.732
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	399.732	dm	nnn
tn	H1	dmm	C
at	3.415	dmf	200
np	32768	dseq	
sw	4757.0	dres	1.0
fb	3000	homo	n
tpwr	60	wi	file
pw	5.8	proc	ft
di	0	fn	not used
tof	0	math	f
nt	16		
ct	16		
alock	n	werr	
gain	not used	wexp	
flags	wbs	wnt	
ll	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-390.9		
wp	4797.0		
vs	144		
sc	0		
wc	250		
hzmm	0.05		
ls	1317.93		
rfl	390.9		
rfd	0		
th	64		
ins	100.000		
nm	no	ph

compound b

S10

compound 3t

145.5 (2) 141.7 (2)

compound 3b

Date : 31-Jul-2001 11:41
 Data : F8120
 Sample: Z. Wang WZKGS06
 Note : (NBR) #01-0673
 Inlet : Direct
 Ion Mode : F8B-
 Spectrum Type : Normal Ion [MF-Linear]
 Scan# : (10,17)
 RT : 1.46 min
 BP : m/z 719.8693 Int. : 40.79
 Output m/z range : 558.8709 to 961.8219
 Cut Level : 0.00 %
 3654169

512

UV-Vis

HPLC-Buckyprep-100% Toluene

Compound 3c

compound 3c

WZVG84b-80CS220CDCL3-1H-092301

exp2 s2pu1

SAMPLE		DEC. & VT	
date	Sep 23 2001	dfrq	399.732
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	399.732	dm	nnn
tn	H1	dmf	c
at	3.415	dmf	200
np	32768	dseq	1.0
sw	4797.0	dres	1.0
fb	3000	homo	n
tpwr	60	PROCESSING	
pw	6.0	wtfile	
dl	0	proc	ft
tof	0	fn	not used
nt	32	math	f
ct	32	werr	
alock	n	wexp	
gain	not used	wbs	
fl	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-388.3		
wp	4797.0		
vs	72		
sc	0		
wc	250		
hzmm	19.19		
ts	2115.43		
rfl	388.3		
rfd	0		
th	22		
ins	22.033		
nm	ph

S14

$\text{H}-\text{CO}_2\text{CH}_2\text{CH}$

compound 3c

VZV084B-80CS22DCDC13-13C-092401
 exp1 std13c
 SAMPLE
 date Sep 23 2001
 solvent CDCl3
 file exp
 ACQUISITION
 sfrq 100.522
 tn 0.800
 at 40000
 np 25000.0
 sw 14000
 fd 16
 bs 57
 tpwr 6.0
 pw 1.000
 di 0
 tof 100000
 nt 40224
 ct alock
 gain 60
 FLAGS
 ll n
 in n
 dp y
 hs nn
 DISPLAY
 sp -3010.7
 wp 25000.0
 vs 25536
 sc 0
 wc 250
 hzmm 151
 ls 1542.27
 rfi 3010.7
 rfp 0
 th 31
 ins 100.000
 nm ph

DEC. & VT

dfrq, 399.732

dn H1

dpr 42

dof 0

vvy w

C13 8135

dmm 1.0

dseq n

dres 0.50

homo n

lb 57

wfile 6.0

proc ft

fn not used

math f

werr n

wexp 60

wbs n

wnt nn

200
180
160
140
120
100
80
60
40
20
0
ppm

compound 3c

[Mass Spectrum]
Date : 26-Oct-2001 14:52
Data : FAB160
Sample: WZMG84b
Note : (NBR) #01-0891
Inlet : Direct
Ion Mode : FAB-
Spectrum Type : Normal Ion [M⁺-Linear]
Scan# : (4,6)
RT : 1.14 min
BP : m/z 152.3387
Int. : 91.86
Output m/z range : 698.2706 to 1001.2326
Cut Level : 0.00 %
225840

514

HPLC-Buckyprep-100% Toluene

UV-Vis

Compound 3d

WZVG94b-75CS225CDC13-1H-100501

exp5 s2pul

date	Oct 5 2001	DEC. & VT	400.397
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION	exp	dof	0
sfrq	400.397	dm	nnn
tn	H1	dmm	C
at	3.412	dmf	200
np	23456	dseq	
sw	4316.4	dres	1.0
fb	2000	homo	n
tpwr	60	PROCESSING	
pw	5.8	wtfile	
dl	0	proc	ft
tof	0	fn	not used
nt	4	math	f
ct	4	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	v		
hs	nn		
DISPLAY			
sp	-149.9		
wp	4316.4		
vs	162		
sc	0		
wc	250		
hzmm	6.72		
ls	500.00		
rfl	3056.8		
rfp	2306.9		
th			
ins	100.000		
nm	no	ph

compound 3d

13C Std. Param.
 for CDC13
 230ppm width
 thing1 aswptg
 07/13/00 WJL
 expl std13c
 SAMPLE DEC. & VT
 date Oct 5 2001 dfrq 400.337
 solvent CDC13 dn H1
 file exp 42
 ACQUISITION
 sfrq 100.630 dm vvv
 tn 0.373 dm 11148
 at 17182 dseq
 np 23048.1 dres 1.0
 sw 13000 homo
 fb 400
 bs 57 lb
 tpwr 3.9 wfile
 pw 1.000 proc ft
 dl 1104.2 fn not used
 nt 4000 math
 ct 3600
 alock not used n
 gain not used n
 FLAGS f1 n
 in n
 dp y
 hs nn
 DISPLAY
 sp -953.2
 wp 23048.1
 vs 10390
 sc 0
 wc 250
 hzmm 25.58
 ts 500.00
 rfl 953.2
 rfp 0
 th 0
 ins 5
 nm 100.000
 no ph

S19

[Mass Spectrum]
Date : 26-Oct-2001 15:43
Data : F8166
Sample: W2WGS4b
Note : (NBR) #01-0896
Inlet : Direct Ion Mode : FAB-
Spectrum Type : Normal Ion (MF-Linear)
RT : 1.62 min Scan# : (12,15)
BP : m/z 720.2709 Int. : 31.60
Output m/z range : 677.2042 to 1002.3585
Cut Level : 0.00 %

UV-Vis

HPLC-5PBB-100% Toluene

Compound 3e

compound 3e

WJW671b-750525 CDCl₃-1H-071101

58-2
exp2 s2pu1
SAMPLE
date Sep 11 2001
solvent CDCl₃
file
ACQUISITION
sfrq 399.732
th 3.415
at 32768
np 4797.0
sw 3000
fb 60
tdwr 6.0
pw 0
di 0
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
sp -388.3
wp 4797.0
vs 148
sc 0
wc 250
hzmm 0.17
fs 16919.83
rfl 388.3
rfp 0
th 15
ins 22.033
nm ph
DEC. & VT
dfrq 399.732
dh H1
dwr 40
dof 0
dm nnn
dm c
dseq 200
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt

522

compound 3e

exp1 std13c

SAMPLE
date Sep 11 2001
solvent CDCl₃
file exp

DEC. & VT
dfrq 399.732
dn H1
dpwr 42
dof 0
yv 0

ACQUISITION
sfrq 100.522
tn Cl₃
at 0.800
np 40000
sw 25000.0
fb 14000
bs 16
tpwr 57
pw 6.0
dl 1.000
tof 0
nt 100000
ct 27840

PROCESSING
lb 57
vtrfile
proc not used
fn
math
werr
wexp
wbs
wnt

FLAGS
ll n
in n
dp v
hs nn

DISPLAY
sp -3010.7
wp 25000.0
vs 9156
sc 0
wc 250
hzmm 15.16
ls 6.2526e+08
rfl 3010.7
rfp 0
th 7
ins 100.000
nm ph

523

compound 3e

[Mass Spectrum]
 Date : 21-Sep-2001 16:24
 Data : FAB146
 Sample: WZMG71b
 Note : (NBR) #01-0810
 Inlet : Direct
 Ion Mode : FAB-
 Spectrum Type : Normal Ion (MF-Linear)
 RT : 0.82 min Scan# : (7,10)
 BP : m/z 720.0000 Int. : 53.35
 Output m/z range : 640.9496 to 954.0059
 Cut Level : 0.00 %

524

UV-Vis

HPLC-Buckyprep-100% Toluene

Compound 3f

S25

compound 3f

VZWG86b-75CS222SCDC13-1H-092801

exp2 s2pu1

SAMPLE
 date Sep 28 2001
 solvent CDCl3
 file ACQUISITION exp
 sfrq 399.732
 tn 3.415
 at 32768
 np 4797.0
 sw 3000
 fb 60
 tpwr 6.0
 pw 0
 di 0
 tof 32
 nt 32
 ct 32
 alock not used
 gain not used
 flags n
 l1 n
 tn n
 dp y
 hs nn
 display
 sp -388.3
 wd 4797.0
 vs 147
 sc 0
 wc 250
 hzmm 19.19
 ls 2115.43
 rfl 388.3
 rfp 0
 th 10
 lns 22.033
 nm ph

DEC. & VT
 dn 399.732
 dpwr 40
 cor 0
 dm nnn
 dmf 200
 dseq 1.0
 dres n
 homo
 wfile
 proc ft
 fn not used
 math
 werr
 wexp
 wbs
 wnt

526

compound 3f

WZV086b-75CS225CDC13-13C-082901

expl std13c

SAMPLE
 date Sep 28 2001
 solvent CDCl3
 file exp
 ACQUISITION
 sfrq 100.522
 tn 0.800
 at 0.800
 np 40000
 sw 25000.0
 fb 14000
 bs 16
 tpwr 57
 pw 6.0
 dl 1.000
 tof 0
 nt 100000
 ct 38288
 alock n
 gain 60
 flags n
 ll n
 ln n
 dp y
 hs nn
 DISPLAY
 sp -3010.7
 wd 25000.0
 vs 44608
 sc 0
 wc 250
 hzmm 18.16
 fs 1542.27
 rfl 3010.7
 rfp 0
 th 11
 ins 100.000
 nm ph

DEC. & VT
 dn 399.732
 HI 42
 dpr 0
 dpr yvy
 dm w
 C13 8135
 dmf
 dseq
 dres 1.0
 homo n
 PROCESSING
 lb 0.50
 wtfile
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt

S29

compound 3f

[Mass Spectrum]
 Date : 25-Oct-2001 15:17
 Data : FABI63
 Sample: WZWC86b
 Note : (NBR) #01-0893
 Inlet : Direct
 Ion Mode : FAB-
 Spectrum Type : Normal Ion [MF⁺Linear]
 Scan# : (15,19)
 RT : 2.07 min
 BP : m/z 611.9991
 Int. : 14.86
 Output m/z range : 695.1200 to 806.6323
 446337

528

HPLC-Buckyprep-100% Toluene

UV-Vis

Compound 3g

compound 3g

VZVG85B-75CS222SCDC13-1H-092701
 exp2 s2pul
 SAMPLE
 date Sep 27 2001
 solvent CDCl3
 file exp
 ACQUISITION
 sfrq 399.732
 tn 3.415
 at 32768
 np 4792.0
 sw 3000
 fb 60
 tpwr 6.0
 pw 0
 dl 0
 nt 32
 ct 32
 alock not used
 gain not used
 flags
 ll n
 ln n
 dp n
 hs nn
 DISPLAY
 sp -388.3
 wd 4797.0
 vs 72
 sc 0
 wc 250
 hzmm 19.19
 fs 2115.43
 rfl 388.3
 rfp 0
 th 21
 fns 22.033
 nm ph
 DEC. & VT
 dfrq 399.732
 dn H1
 dpwr 40
 dm 0
 dmm nn
 dmf C
 dseq 200
 dres 1.0
 homo n
 wfile
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt

530

compound 3g

WZVG85b-75CS225C0C13-13C-092801
 exp1 std13c
 SAMPLE date Sep 27 2001 DEC. & VT
 solvent CDC13 dn H1
 file exp 42
 ACQUISITION sfrq 100.522 dm yv 8135
 tn C13 dmm
 at 0.800 dmf
 np 40000 dseq
 sw 25000.0 dres 1.0
 fb 14000 homo
 bs 16
 tpwr 57 lb PROCESSING 0.50
 pw 6.0 wfile
 dl 1.000 proc ft
 tof 0 fn not used
 nt 100000 math
 ct 29360
 alock n werr
 gain 60 wexp
 FLAGS n wnt
 ll n
 in n
 dp y
 hs nn
 DISPLAY
 sp 3010.7
 wp 25000.0
 vs 27862
 sc 0
 wc 250
 hzmm 100.00
 ls 1542.27
 rfl 3010.7
 rfp 0
 th 21
 ins 100.000
 nm ph

531

compound 3

[Mass Spectrum]
 Date : 26-Oct-2001 15:06
 Data : F8162
 Sample: WZKGB5b
 Note : (NBR) #01-0892
 Inlet : Direct
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 0.55 min Scan# : (3,9)
 BP : m/z 720.219 Int. : 30.93
 Output m/z range : 594.2136 to 902.9674
 2270418
 720.22
 Cut Level : 0.00 %

532

UV-Vis

HPLC-5PBB-100% Toluene

Compound 3h

compound 3I

58-2
exp2 s2pu1

SAMPLE
date Sep 19 2001
solvent CDCl3
file exp
ACQUISITION
sfrq 399.732
tn 3.415
at 3.415
np 32768
sw 4797.0
fb 3000
tpwr 60
pw 6.0
d1 0
tof 0
nt 32
ct 32
alock n
gain not used
FLAGS
f1 n
f2 n
dp n
hs n
DISPLAY
sp -388.3
wp 4797.0
vs B2
sc 0
wc 250
hzmm 0.48
fs 2115.43
rfl 388.3
rfp 0
th 9
fns 22.033
nm

DEC. & VT
dfrq. 399.732
dn HI
dpwr 40
dof 0
dm nnn
dmm C
dmf 200
dseq 1.0
dres n
homo n
PROCESSING
wtfile
proc ft
fn not used
math
werr
wexp
wbs
wnt

S34

compound 3h

wjw 750 - 750s 25 CDCl3 - 13C - 092001

WJW 750 - 750s 25 CDCl3 - 13C - 092001

expt	std13c	date	19 Sep 2001	DEC. & VT	dfrq	399.732
solvent	CDCl3	file	exp	dn	H1	42
ACQUISITION	exp	exp	exp	gpr	0	0
sfrq	100.522	dm	0	dy	8135	0
tn	0.800	dmm	0	dy	8135	0
at	40000	dseq	0	dy	8135	0
np	25000.0	dres	1.0	dy	8135	0
sw	14000	homo	0	dy	8135	0
fb	16	lb	0.50	dy	8135	0
bs	57	proc	not used	dy	8135	0
tpwr	1.000	fn	not used	dy	8135	0
pw	100000	math	not used	dy	8135	0
dl	29056	werr	0	dy	8135	0
to	60	wexp	0	dy	8135	0
nt	60	wbs	0	dy	8135	0
ct	60	wnt	0	dy	8135	0
alock	60	nn	0	dy	8135	0
gain	60	nn	0	dy	8135	0
FLAGS	60	nn	0	dy	8135	0
ll	60	nn	0	dy	8135	0
in	60	nn	0	dy	8135	0
dp	60	nn	0	dy	8135	0
hs	60	nn	0	dy	8135	0
DISPLAY	60	nn	0	dy	8135	0
sp	-3010.7	nn	0	dy	8135	0
wp	25000.0	nn	0	dy	8135	0
vs	20717	nn	0	dy	8135	0
sc	250	nn	0	dy	8135	0
wc	250	nn	0	dy	8135	0
hzmm	0.37	nn	0	dy	8135	0
ls	1542.27	nn	0	dy	8135	0
rfl	3010.7	nn	0	dy	8135	0
rfl	3010.7	nn	0	dy	8135	0
rfl	3010.7	nn	0	dy	8135	0
th	70	nn	0	dy	8135	0
ins	100.000	nn	0	dy	8135	0
nm	100.000	nn	0	dy	8135	0

535

compound : 1

[Mass Spectrum]
 Date : 26-Oct-2001 14:35
 Data : FABI59
 Sample: WZMG75b
 Note : (NBR) #01-0890
 Inlet : Direct
 Ion Mode : FAB-
 Spectrum Type : Normal Ion (MF-Linear)
 Scan# : (25,27)
 RT : 2.50 min
 BP : m/z 720.3033 Int. : 49.12
 Output m/z range : 694.6588 to 902.3739
 Cut Level : 0.00 %
 1545247

S36

HPLC-5PBB-100% Toluene

UV-Vis

Compound 3i

compound 3

VZV091b-70CS230CDC13-1H-100201

exp2 s2pu1

SAMPLE		DEC. & VT	
date	Oct 2 2001	dfrq	399.732
solvent	CDC13	dn	H1
file		dpwr	40
ACQUISITION		dof	0
sfrq	399.732	dm	nmn
tn	3.415	dmf	C
at	32768	dseq	200
np	4797.0	dres	1.0
sw	3000	homo	n
fb	60	PROCESSING	
tpwr	6.0	wtfile	
pw	0	proc	ft
di	0	fn	not used
nt	32	math	f
ct	32	werr	
alock	n	wexp	
gain	not used	wbs	
flags		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-388.3		
wp	4797.0		
vs	453		
sc	0		
wc	250		
hzmm	19.19		
fs	2115.43		
rfl	388.3		
rfp	0		
th	28		
ins	22.033		
nm			
	ph

S38

compound 3

WZVG91b-70CS230CDC13-13C-100301

expt std13c

SAMPLE		DEC. & VT	
date	Oct 2 2001	dfrq'	399.732
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	100.522	dm	yyy
tn	0.013	dmm	w
at	0.800	dmr	8135
np	40000	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	57	lb	0.50
pw	6.0	wtfile	
dl	1.000	proc	ft
tof	0	fn	not used
nt	100000	math	
ct	30112	n	
alock	60	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	y		
do	n		
hs	nm		
DISPLAY		sp	-3010.7
wp	25000.0	vs	8349
sc	0	wc	250
h2mm	8.15		
ls	1542.27		
rfl	3010.7		
rfp	0		
th	10		
ins	100.000		
nm	ph

539

compound 3

[Mass Spectrum]
 Date : 26-Oct-2001 15:27
 Data : FAB164
 Sample : WZK091b
 Note : (NBR) #01-0894
 Inlet : Direct
 Ion Mode : FAB-
 Spectrum Type : Normal Ion [MF⁻Linear]
 RT : 1.49 min Scan# : (10,15)
 BP : m/z 720.2488 Int. : 23.93
 Output m/z range : 593.4718 to 1004.7478
 Cut Level : 0.00 %
 1505723
 100

S40