Supporting Information

Dichloroketene-Induced Cyclizations of Vinyl Sulfilimines: Application of the Method in the Synthesis of ($\pm$)-Desoxyeseroline

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Supporting Information Available: ^1H and ^{13}C -NMR spectra for new compounds lacking elemental analyses. Spectroscopic data and experimental details for the preparation of sulfilimines **26**, **28**, **29**, **32**, **33**, **36-39**, **44**, **46**, **50-52**, **61-63**.

General Experimental Section Paragraph: Melting points are uncorrected. Mass spectra were determined at an ionizing voltage of 70eV. Unless otherwise noted, all reactions were performed in flame dried glassware under an atmosphere of dry nitrogen. Solutions were evaporated under reduced pressure with a rotary evaporator and the residue was chromatographed on a silica gel column using an ethyl acetate-hexane mixture as the eluent unless specified otherwise. A CEM Discover Synthesis microwave unit was used with a programmed temperature of 100 °C.

Table of Contents for Supporting Information:

S3	^1H -NMR spectrum of compound 19
S4	^1H -NMR spectrum of compound 21
S5	^1H -NMR spectrum of compound 22
S6	^{13}C -NMR spectrum of compound 24
S7	^1H -NMR spectrum of compound 25
S8	^1H -NMR spectrum of compound 33
S9	^1H -NMR spectrum of compound 35
S10	^1H -NMR spectrum of compound 36
S11	^1H -NMR spectrum of compound 37

S12	¹ H-NMR spectrum of compound 39
S13	¹ H-NMR spectrum of compound 41
S14	¹ H-NMR spectrum of compound 42
S15	¹ H-NMR spectrum of compound 43
S16	¹ H-NMR spectrum of compound 46
S17	¹³ C-NMR spectrum of compound 48
S18	¹ H-NMR spectrum of compound 50
S19	¹³ C-NMR spectrum of compound 51
S20	¹ H-NMR spectrum of compound 52
S21	¹³ C-NMR spectrum of compound 53
S22	¹³ C-NMR spectrum of compound 54
S23	¹ H-NMR spectrum of compound 55
S24	¹ H-NMR spectrum of compound 56
S25	¹³ C-NMR spectrum of compound 57
S26	¹³ C-NMR spectrum of compound 58
S27	¹³ C-NMR spectrum of compound 61
S28	¹³ C-NMR spectrum of compound 62
S29	¹³ C-NMR spectrum of compound 63
S30	¹³ C-NMR spectrum of compound 66
S31	¹ H-NMR spectrum of compound 68
S32	¹³ C-NMR spectrum of compound 69
S33	¹ H-NMR spectrum of compound 71
S34	¹ H-NMR spectrum of compound 73
S35	¹ H-NMR spectrum of compound 82
S36	¹ H-NMR spectrum of compound 83
S37	¹ H-NMR spectrum of compound 84
S38	¹ H-NMR spectrum of compound 79
S39	Experimental Details for all New Sulfilimines

Page 3

4wq-197-major
Pulse Sequence: zgpg30

STANDARD 1H OBSERVE

expl stdh

SAMPLE
 date Jan 5 2005 DEC. & VT
 solvent dmsd 399.942
 title exp
 ACQUISITION
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 in H1
 at 2.202
 np 22912
 sw 5203.3
 th 3000
 bs 16
 tpwr 58
 pw 8.0
 dl 1.000
 tof 207.0
 nt 4
 ct 4
 alock n
 gain not used
 flags n
 tl n
 in n
 dp y
 hs nm
 DISPLAY
 sp -40.3
 wp 3999.2
 vs 144
 sc 0
 wc 250
 hznm 16.00
 ts 500.00
 rfi 1382.2
 rfd 999.9
 th 20
 ins 100.000
 nm cdc ph

DEC. & VT
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 H1
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 PROCESSING
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STANDARD 1H OBSERVE

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 SAMPLE DEC. & VT
 date Jan 21 2005 dfrq 399.940
 solvent cdc13 dn H1
 f1le exp30
 ACQUISITION
 sfrq 399.941 dm
 tn H1
 at 2.202 dmf
 np 22912 dseq 200
 sw 5203.3 dres 1.0
 fh 3000 homo
 bs 16 PROCESSING
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expl stdln

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STANDARD 1H OBSERVE

expt stdh

SAMPLE DEC. 8 VI
 date Jan 15 2005 dfrq 399.940
 solvent cdcl3 dn H1
 file exp 30
 ACQUISITION
 sfrq 399.941 dm -630.7
 tn H1 dm nm
 al 2.202 dmf c
 np 22912 dseq 200
 sw 5203.3 dres 1.0
 fh 3000 homo n
 bs 16 PROCESSING
 tpwr 58 lb wfile 0.20
 pw 8.0 wfile ft
 dl 1.000 proc f
 tof 207.0 fn not used
 nt 4 math f
 cl 0
 alock n
 gain not used
 flags react
 wexp procpot
 wbs
 wrl
 DISPLAY -40.3
 sp wp 3999.2
 vs 144
 sc 0
 wc 250
 hzmm 16.00
 ts 500.00
 rfi 3294.2
 rfp 2903.6
 th 20
 ins 1.000
 nm cdc ph

STANDARD IIR OBSERVE

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 sw 5203.3
 fb 3000
 bs 16
 lpwr 58
 pw 8.0
 dl 1.000
 tof 207.0
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 ct 4
 alock n
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 flags not used
 in n
 in n
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 hs mn
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 wc 250
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 rs 500.00
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 rfp 2903.6
 th 20
 ins 1.000
 nm cdc ph

STANDARD 1H OBSERVE

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SAMPLE date Apr 12 2005 dfrq DEC. & VT 399.940
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 f1file exp 30
 ACQUISITION
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 at 2.202 dmf C
 np 22912 dseq 200
 sw 5203.3 dres 1.0
 fu 3000 homo n
 bs 16 PROCESSING 0.20
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 pw 8.0 wffile
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 tot 207.0 fn not used
 nl 16 math f
 ct 0
 alock n weff react
 gain not used wexp procpilot
 flags not used wbs
 f1 n wnt
 in n
 dp y
 hs na

DISPLAY
 sp -40.3
 wp 3999.2
 vs 144
 sc 0
 wc 250
 hznm 16.00
 is 500.00
 rfi 3294.5
 rfp 2903.6
 th 20
 ins 1.000
 nm cdc ph

STANDARD 1H OBSERVE

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exp1 std1h
SAMPLE
date Aug 27 2004
solvent cdcl3
file exp
ACQUISITION
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in H1
at 1.196
np 9280
sw 3879.1
fb 2200
bs 16
tpwr 55
pw 7.0
dl 1.000
tof 215.9
nt 16
ct wbs
alock wat
gain 20
FLAGS
ii n
in n
dp y
DISPLAY
sp -30.2
wp 3000.6
vs 143
sc 0
wc 12.00
hzmm 438.69
is 229.5
rfi 0
rfp 20
th 1.000
ins nm
cdc ph
DEC. & VT
300.065
H1
30
0
nm
c
200
PROCESSING
0.20
wf file
lb
proc
ft
fn
not used
react
wexp
procpot

```

STANDARD 1H OBSERVE

expt std1h
 SAMPLE
 date Jan 21 2005
 solvent cdcl3
 file ACQUISITION
 srfreq 399.941
 tn H1
 at 2.202
 np 22912
 sw 5203.3
 fb 3000
 bs 16
 tpwr 58
 pw 8.0
 dl 1.000
 tof 207.0
 nt 4
 ct 0
 alock n
 gain not used
 flags not used
 f1 n
 f2 n
 dp y
 hs mn
 DISPLAY
 sp -40.3
 wp 3099.2
 vs 144
 sc 0
 wc 250
 hzmm 16.00
 ts 500.00
 rfl 3203.8
 rfp 2903.6
 th 20
 ins 1.000
 nm cdc ph

DEC. A VT
 dfrq 399.940
 dn H1
 dpr 30
 dof -630.7
 dm nm
 c
 200
 dseq
 dres 1.0
 homo
 PROCESSING
 0.20
 lb
 wtfile
 proc ft
 fn not used
 math f
 werr
 wexp
 procpol
 react
 procpol

STANDARD 1H OBSERVE

expt stdth

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 ln H1 dmm nnn
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 fb 2200 wifile fl
 bs 15 proc not used
 tpwr 55 fn
 pw 7.0 wrr react
 d1 1.000 wexp precipit
 tot 215.9
 nt 8
 ct 8
 alock n
 gain 20

FLAGS n n
 f1 n
 in n
 dp y

DISPLAY
 sp -30.3
 wp 3000.6
 vs 162
 sc 0
 wc 250
 hzmm 12.00
 ts 500.00
 rfl 2406.7
 rfp 2178.5
 th 15
 ins 1.000
 nm cdc ph

1wq-167-C13

Pulse Sequence: szpul

~~38~~
48

Page 17

2-sulfithimine
Pulse Sequence: szpul

5mq-056-A
Pulse Sequence: szpul

Swi-080-8
Pulse Sequence: s2pu1

4wq-156-C13

Pulse Sequence: s2put1

PhS → NTs

62

4wq-198-c13
Pulse Sequence: s2pu1

4wd-200-c13
Pulse Sequence: szpu1

pyrrole
Pulse Sequence: szpu1

Page 31

5WQ-001-B-C13
Pulse Sequence: szpul

STANDARD 1H OBSERVE

Pulse Sequence: szpu1

STANDARD 1H OBSERVE

expt stdh

SAMPLE

DEC. 8 VT

date May 11 2005

399.940

solvent cdc13

H1

file ACQUISITION

H1

sfrq 399.941

30

in H1

nmh

at 2.202

C

np 22912

200

sw 5203.3

1.0

fb 3000

n

bs 16

PROCESSING

tpwr 58

0.20

dl 1.000

F1

to 207.0

not used

ni 4

math

ct 4

f

alock n

react

gain not used

procpot

flags n

il n

in n

dp v

hs nn

sp DISPLAY

-40.3

wp 3999.2

144

vs 144

sc 0

wc 250

hzmm 16.00

fs 757.86

rfi 3293.8

rfp 2903.6

th 20

tms 1.000

nm cdc ph

82

STANDARD 1H OBSERVE

expt stidn

SAMPLE

DEC. 8 VI

date May 11 2005

dfrq 399.940

solvent cdc-13

dn H1

file

dpwr 30

ACQUISITION

dot -630.7

strq 399.941

dm nnn

in H1

dmn c

at 2.202

dnt 200

np 22912

dseq

sw 5203.3

freq 1.0

fb 3000

homo n

bs 16

PROCESSING

tpwr 58

th 0.20

pw 8.0

wtfile

dl 1.000

proc ft

tof 207.0

fn not used

nt 4

math +

ct 4

react

alock n

procpol

gain not used

flags

il n

in n

dp y

hs nn

DISPLAY

sn -40.3

wp 3999.2

vs 144

sc 250

wc 16.00

hzmn 757.86

ls 3293.8

rfl 2903.6

rflp 20

th 1.000

ins

nm cdc ph

STANDARD IN OBSERVE

expl stdln

SAMPLE DEC. & VT
 date May 15 2005 dfrq 399.940
 solvent cdcl3 dp H1
 file exp 30
 ACQUISITION
 sfrq 399.941 dm
 ln H1 dm
 nt 2.202 dmf c
 np 22912 discn 200
 sw 5203.3 dres 1.0
 fh 3000 homo n
 bs 15
 tpwr 38 lh PROCESSING 0.20
 pw 3.0 wtfite
 dl 1.000 proc ft
 tof 207.0 fn not used
 nt 8 math f
 ct 8
 alock react
 gain not used
 flags not used
 werr react
 wexp proplot
 wbs
 wnt

in n
 dp y
 hs nm
 DISPLAY
 sp -40.3
 wp 3999.2
 vs 144
 sc 0
 wc 250
 hzmm 16.00
 is 891.96
 rfi 3293.8
 rfp 2903.6
 th 20
 tns 1.000
 nm edc ph

expt stidh

SAMPLE DFC. 8 VT
 date May 24 2005 dfrq 399.940
 solvent cdc13 dn HI
 file exp dpr 30
 ACQUISITION dof -630.7
 sfreq 399.941 dm nm
 tn HI dmnm c
 at 2.202 dmf 200
 np 22912 dseq
 sw 5203.3 dres 1.0
 fb 3000 homo n
 bs 16 PROCESSING
 tpwr 58 lb wtfile 0.20
 pw 8.0 wtitle
 d1 1.000 proc f1
 tof 207.0 fn not used
 nt 32 math f
 ct 32
 alock n
 gain not used
 flags not used
 11 n
 tn n
 dp v
 hs nm
 display -40.3
 sp 3999.2
 wp 194
 vs 0
 sc 250
 wc 16.00
 hzmm 2432.23
 fs 3293.8
 rfp 2903.6
 th 20
 tns 1.000
 nm cdc ph

S-Ethyl-N-(toluene-4-sulfonyl)-S-1-naphthylsulfilimine (26). To a solution containing 1.5 g (8.0 mmol) of 1-ethylthionaphthalene¹ in 50 mL of MeCN at rt was added 2.2 g (8.0 mmol) of chloramine-T trihydrate. After stirring at rt for 21 h, the reaction mixture was poured into EtOAc, the mixture was filtered and the filtrate was concentrated under reduced pressure. The residue was recrystallized from EtOAc/hexane to give 2.1 g (73%) of **26** as a white solid: mp 134-136 °C; IR (KBr) 1282, 1139, 1089, 983, 810 and 766 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.22 (t, 3H, *J* = 7.2 Hz), 2.31 (s, 3H), 3.03–3.20 (m, 2H), 7.11 (d, 2H, *J* = 8.2 Hz), 7.57–7.77 (m, 3H), 7.75 (d, 2H, *J* = 8.2 Hz), 7.95 (dd, 1H, *J* = 7.6 and 2.0 Hz), 8.01 (d, 1H, *J* = 8.0 Hz), 8.05 (d, 1H, *J* = 8.0 Hz) and 8.28 (dd, 1H, *J* = 7.6 and 1.2 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 7.6, 21.3, 46.9, 121.3, 125.6, 126.2, 126.3, 127.1, 128.1, 129.0, 129.2, 129.7, 132.6, 133.7, 141.2 and 141.5; Anal. Calcd. for C₁₉H₁₉NO₂S₂: C, 63.83; H, 5.36; N, 3.92. Found: C, 63.56; H, 5.28; N, 3.93.

S-2-Benzo[*b*]furan-S-ethyl-N-(toluene-4-sulfonyl)sulfilimine (28). To a solution containing 2.5 g (21 mmol) of benzo[*b*]furan in 200 mL of Et₂O at 0 °C was added 10 mL (25 mmol) of *n*-BuLi (2.5 M in hexane). After the reaction was stirred at rt for 30 min, 2.3 mL (21 mmol) of ethyl disulfide was added. After stirring at rt for 20 h, the reaction was quenched with a saturated aqueous NH₄Cl solution and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 2.4 g (64%) of 2-(ethylthio)benzo[*b*]furan as a yellow oil; IR (neat) 1528, 1445, 1252, 1160, 924 and 749 cm⁻¹; ¹H-NMR (CDCl₃, 600 MHz) δ 1.33 (t, 3H, *J* = 7.2 Hz), 2.96 (q, 2H, *J* = 7.2 Hz), 6.80 (s, 1H), 7.21 (t, 1H, *J* = 7.8 Hz), 7.26 (t, 1H, *J* = 7.8 Hz), 7.44 (d, 1H, *J* = 7.8 Hz), 7.50 (d, 1H, *J* = 7.8 Hz); ¹³C-NMR (CDCl₃, 150 MHz) δ 15.1, 28.9, 110.9, 111.0, 120.3, 122.8, 124.3, 128.6, 150.4 and 156.3.

To a solution containing 1.3 g (7.1 mmol) of above sulfide in 40 mL of EtOH at rt was added 2 g (4.6 mmol) of chloramine-T trihydrate. After stirring at rt for 15 h, the reaction was quenched with H₂O and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure.

The residue was recrystallized from EtOAc/hexane to give 1.1 g (46%) of **28** as a colorless solid; mp 129-130 °C; IR (KBr) 2975, 1278, 1145, 1087, 966, 751 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.30 (t, 3H, *J* = 7.6 Hz), 2.27 (s, 3H), 3.38-3.52 (m, 2H), 7.08 (d, 2H, *J* = 8.0 Hz), 7.23 (brs, 1H), 7.31 (td, 1H, *J* = 7.8 and 1.2 Hz), 7.28 (td, 1H, *J* = 7.8 and 1.2 Hz), 7.48 (d, 1H, *J* = 7.8 Hz), 7.58 (d, 1H, *J* = 7.8 Hz) and 7.70 (d, 2H, *J* = 8.0 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 7.6, 21.3, 44.0, 112.0, 115.2, 122.5, 124.3, 126.0, 126.2, 127.6, 129.0, 140.8, 141.7, 144.5 and 156.8; Anal. Calcd. for C₁₇H₁₇NO₃S₂: C, 58.77; H, 4.93; N, 4.03. Found: C, 58.54; H, 4.83; N, 4.01.

S-2-(1-*tert*-Butoxycarbonyl-3-methyl)indolyl-S-ethyl-(toluene-4-sulfonyl)sulfilimine (29). To a solution containing 3.8 g (13 mmol) of 1-*tert*-butoxycarbonyl-2-

ethylthio-3-methylindole² in 100 mL of MeCN was added 4.0 g (14 mmol) of chloramine-T trihydrate. The mixture was stirred at rt for 14 h and concentrated under reduced pressure. The residue was recrystallized from EtOAc/hexane to give 3.7 g (62%) of **29** as a white solid: mp 158-160 °C; IR (neat) 1712, 1370, 1143 and 956 cm⁻¹; ¹H-NMR (CDCl₃, 600 MHz) δ 1.39 (t, 3H, *J* = 7.2 Hz), 1.69 (s, 9H), 2.18 (s, 3H), 2.54 (s, 3H), 3.40 (dq, 1H, *J* = 12.6 and 7.2 Hz), 3.49 (dq, 1H, *J* = 12.6 and 7.2 Hz), 6.97 (d, 2H, *J* = 8.4 Hz), 7.28 (td, 1H, *J* = 8.4 and 1.2 Hz), 7.41 (td, 1H, *J* = 8.4 and 1.2 Hz), 7.47 (d, 1H, *J* = 8.4 Hz), 7.61 (d, 2H, *J* = 8.4 Hz) and 7.90 (d, 1H, *J* = 8.4 Hz); ¹³C-NMR (CDCl₃, 150 MHz) δ 8.7, 10.1, 21.4, 28.4, 47.0, 86.6, 115.3, 119.9, 123.6, 123.8, 126.2, 127.6, 128.5, 129.0, 130.4, 135.7, 141.1, 141.5 and 150.0; Anal. Calcd. for C₂₃H₂₈N₂O₄S₂: C, 59.97; H, 6.13; N, 6.08. Found: C, 60.08; H, 5.96; N, 6.05.

S-1-Ethyl-S-2-furyl-N-(toluene-4-sulfonyl)sulfilimine (32). To a solution containing 2.6 g (21 mmol) of 2-(ethylthio)furan³ in 100 mL of EtOH was added 5.7 g (20 mmol) of chloramine-T trihydrate. The mixture was stirred at rt for 1 h and concentrated under reduced pressure. The residue was recrystallized from EtOAc/hexane to give 2.9 g (49%) of **32** as a white solid: mp 127-129 °C; IR (neat) 3131, 1280, 1139, 967 and 748 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.20 (t, 3H, J = 7.2 Hz), 2.31 (s, 3H), 3.30 (dq, 1H, J = 12.8 and 7.2 Hz), 3.38 (dq, 1H, J = 12.8 and 7.2 Hz), 6.43 (dd, 1H, J = 3.6 and 2.0 Hz), 6.85 (dd, 1H, J = 3.6 and 0.8 Hz), 7.13 (d, 2H, J = 8.0 Hz), 7.59 (dd, 1H, J = 2.0 and 0.8 Hz) and 7.65 (d, 2H, J = 8.0 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 7.9, 21.5, 44.1, 112.1, 119.1, 126.3, 129.2, 141.1, 141.8, 141.9 and 148.6; Anal. Calcd. for C₁₃H₁₅NO₃S₂: C, 52.50; H, 5.08; N, 4.71. Found: C, 52.66; H, 5.03; N, 4.78.

S-1-Butyl-2-(3-acetoxymethyl)furyl-sulfilimine (33). To a solution containing 3 g (35 mmol) of furan-3-methanol in 200 mL of Et₂O at 0 °C was added 30 mL (75 mmol) of *n*-BuLi (2.5 M in hexane). After stirring at rt for 1 h, 6.7 mL (35 mmol) of *n*-butyl-disulfide was added. Stirring was continued at rt for 15 h and then the reaction was quenched with a saturated aqueous NH₄Cl solution and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure to give 4.5 g of an oil.⁴ This material was dissolved in 100 mL of CH₂Cl₂ and 1.0 mL (12 mmol) of pyridine and 4.9 mL (52 mmol) of Ac₂O were added. After stirring at rt for 18 h, the reaction was quenched with HCl (1.0 M) and extracted with EtOAc. The organic layer was washed with a saturated aqueous NaHCO₃ solution, brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 1.9 g (24%) of 2-butylthio-3-acetoxymethylfuran as a yellow oil: IR (neat) 1743, 1376, 1228, 1023 and 736, cm⁻¹; ¹H-NMR (CDCl₃, 300 MHz) δ 0.89 (t, 3H, J = 7.5 Hz), 1.36–1.45 (m, 2H), 1.48–1.57 (m, 2H), 2.08 (s, 3H), 2.74 (t, 2H, J = 7.2 Hz), 5.02 (s, 2H), 6.47 (d, 1H, J =

1.8 Hz) and 7.46 (d, 1H, $J = 1.8$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 13.6, 20.9, 21.5, 31.9, 35.7, 58.4, 112.2, 125.4, 143.8, 145.0 and 171.0.

To a solution containing 1.1 g (4.6 mmol) of above sulfide in 20 mL of EtOH at rt was added 1.3 g (4.6 mmol) of chloramine-T trihydrate. After stirring at 50 °C for 21 h, the reaction was concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 0.38 g (20%) of **33** as a yellow oil: IR (neat) 1743, 1282, 1225, 1090, 960, 815 and 660 cm^{-1} ; ^1H -NMR (CDCl_3 , 300 MHz) δ 0.87 (t, 3H, $J = 7.2$ Hz), 1.34–1.44 (m, 2H), 1.51–1.66 (m, 2H), 2.07 (s, 3H), 2.36 (s, 3H), 3.24 (ddd, 1H, $J = 12.5$, 8.7 and 6.6 Hz), 3.59 (ddd, 1H, $J = 12.5$, 8.4 and 5.4 Hz), 4.84 (d, 1H, $J = 13.2$ Hz), 5.04 (d, 1H, $J = 13.2$ Hz), 6.48 (d, 1H, $J = 1.9$ Hz), 7.17 (d, 2H, $J = 8.3$ Hz), 7.59 (d, 1H, $J = 1.9$ Hz) and 7.69 (d, 2H, $J = 8.3$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 13.4, 20.7, 21.2, 21.3, 25.0, 48.3, 56.1, 112.7, 126.3, 129.1, 130.1, 139.7, 141.0, 141.7, 148.2 and 170.4.

S-1-Butyl-S-2-furyl-N-(4-nitrobenzenesulfonyl)sulfilimine (36). To a suspension of 0.3 g (2.0 mmol) of 2-butylsulfanylfuran, 0.8 g (4.0 mmol) of 4-nitrobenzenesulfonamide, 0.3 g (8.0 mmol) of MgO and 0.02 g (0.05 mmol) of $\text{Rh}_2(\text{OAc})_4$ in 20 mL of CH_2Cl_2 at rt, was added 1.0 g (3.0 mmol) of $\text{PhI}(\text{OAc})_4$. After stirring at rt for 10 h, the resulting mixture was purified by flash silica gel chromatography to provide 0.59 g (83%) of **36**⁵ as a white solid: mp 113–114 °C IR (KBr) 1530, 1298, 1146, 957 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 0.90 (t, 3H, $J = 7.2$ Hz), 1.39–1.48 (m, 2H), 1.56–1.69 (m, 2H), 3.33 (ddd, 1H, $J = 12.8$, 8.8 and 6.8 Hz), 3.54 (ddd, 1H, $J = 12.8$, 8.4 and 6.0 Hz), 6.49 (dd, 1H, $J = 3.6$ and 2.0 Hz), 6.94 (dd, 1H, $J = 3.6$ and 0.8 Hz), 7.64 (dd, 1H, $J = 2.0$ and 0.8 Hz), 7.96 (d, 2H, $J = 9.0$ Hz) and 8.21 (d, 2H, $J = 9.0$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 13.4, 21.2, 24.9, 49.4, 112.2, 119.7, 123.8, 127.4, 141.3, 148.8, 149.0 and 149.7.

S-1-Butyl-S-2-furyl-N-(benzenesulfonyl)sulfilimine (37). To a solution containing 0.6 g (3.8 mmol) of 2-butylsulfanylfuran in 20 mL of MeCN at rt, was

added 1.1 g (5.1 mmol) of chloramine-T. After stirring at rt for 20 h, the reaction mixture was quenched with H₂O and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 0.74 g (62%) of **37** as a pale yellow oil: IR (neat) 1446, 1300, 1138, 958 and 752 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 0.87 (t, 3H, *J* = 7.2 Hz), 1.33-1.43 (m, 2H), 1.55-1.65 (m, 2H), 3.25 (ddd, 1H, *J* = 12.8, 8.4 and 6.4 Hz), 3.48 (ddd, 1H, *J* = 12.8, 8.4 and 5.6 Hz), 3.80 (s, 3H), 6.46 (dd, 1H, *J* = 3.6 and 1.8 Hz), 6.87 (dd, 1H, *J* = 3.6 and 1.0 Hz), 7.36-7.42 (m, 3H), 7.61 (1H, dd, *J* = 1.8, 1.0 Hz) and 7.81-7.84 (m, 2H); ¹³C-NMR (CDCl₃, 100 MHz) δ 13.4, 21.2, 24.8, 49.3, 112.0, 119.0, 126.2, 128.5, 131.2, 142.1, 143.8 and 148.3.

S-1-Butyl-S-2-furyl-N-(toluene-4-sulfonyl)sulfilimine (38). To a solution containing 3.0 mL (40 mmol) of furan in 200 mL of Et₂O at 0 °C, was added 17 mL (41 mmol) of *n*-BuLi (2.5 M in hexane). After stirring at rt for 30 min, 8.0 mL (40 mmol) of *n*-butyl disulfide was added. The reaction mixture was stirred at rt for 20 h, quenched with a saturated aqueous NH₄Cl and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 4.7 g (72%) of 2-(butylthio)furan as a yellow oil: IR (KBr) 1463, 1150, 1003, 906 and 739 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 0.89 (t, 3H, *J* = 7.6 Hz), 1.38–1.45 (m, 2H), 1.52–1.60 (m, 2H), 2.75 (t, 2H, *J* = 7.2 Hz), 6.36 (dd, 1H, *J* = 3.6 and 2.4 Hz), 6.47 (dd, 1H, *J* = 3.2 and 0.8 Hz) and 7.48 (dd, 1H, *J* = 2.4 and 0.8 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 13.6, 21.5, 31.8, 35.6, 111.3, 116.4, 145.1 and 146.1.

To a solution containing 2.6 g (16 mmol) of above sulfide in 100 mL of EtOH at rt, was added 4.7 g (17 mmol) of chloramine-T trihydrate. After stirring at rt for 20 h, the reaction mixture was quenched with H₂O and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and

concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 4.1 g (75%) of **38** as a white solid: mp 65-67 °C; IR (KBr) 1282, 1140, 1088, 965, 761 and 653 cm⁻¹; ¹H-NMR (CDCl₃, 300 MHz) δ 0.87 (t, 3H, *J* = 9.4 Hz) 1.44–1.32 (m, 2H), 1.62–1.53 (m, 2H), 2.36 (s, 3H), 3.28–3.19 (m, 1H), 3.50–3.41 (m, 1H), 6.47 (dd, 1H, *J* = 4.4 and 1.2 Hz), 6.90 (d, 1H, *J* = 4.4 Hz), 7.17 (d, 2H, *J* = 11.2 Hz), 7.61 (d, 1H, *J* = 1.2 Hz) and 7.70 (d, 2H, *J* = 11.2 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 13.4, 21.2, 21.3, 24.8, 49.4, 112.0, 118.9, 126.2, 129.1, 141.1, 141.6, 142.2 and 148.2; Anal. Calcd. for C₁₅H₁₉NO₃S₂: C, 55.36; H, 5.88; N, 4.30. Found: C, 55.11; H, 5.86; N, 4.32.

S-1-Butyl-S-2-furyl-N-(4-methoxybenzenesulfonyl)sulfilimine (39). To a solution containing 1.0 g (6.4 mmol) of 2-(butylthio)furan in 30 mL of MeCN at rt was added 1.8 g (6.4 mmol) of *N*-chloro-4-methoxybenzenesulfonamide sodium salt⁶. After stirring at rt for 19 h, the reaction was quenched with H₂O and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 1.6 g (69%) of **39** as a pale yellow oil: IR (neat) 1498, 1259, 1140, 962 and 835 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 0.86 (t, 3H, *J* = 7.2 Hz), 1.30-1.41 (m, 2H), 1.51-1.63 (m, 2H), 3.23 (ddd, 1H, *J* = 12.8, 8.8 and 6.8 Hz), 3.44 (ddd, 1H, *J* = 12.8, 8.4 and 5.6 Hz), 3.80 (s, 3H), 6.46 (dd, 1H, *J* = 3.6 and 2.0 Hz), 6.84 (d, 1H, *J* = 8.8 Hz), 6.87 (dd, 1H, *J* = 3.6 and 0.8 Hz), 7.61 (dd, 1H, *J* = 2.0 and 0.8 Hz) and 7.73 (d, 2H, *J* = 8.8 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 13.4, 21.2, 24.8, 49.2, 55.4, 112.0, 113.6, 118.9, 128.2, 135.9, 142.3, 148.3 and 161.7.

S-Ethenyl-S-phenyl-N-(toluene-4-sulfonyl)sulfilimine (44). To a solution containing 0.25 mL (2.4 mmol) of ethyl vinyl sulfide in 50 mL of ethanol was slowly added 0.57 g (2.0 mmol) of chloramine-T trihydrate and 0.1 mL of acetic acid. The reaction mixture was stirred at room temperature for 1 h. The solvent

was removed under reduced pressure and the residue was purified by flash silica gel chromatography to give 0.35 g (70%) of **44** as a white solid: mp 122-124 °C; IR (neat) 1275, 1140, 1088 and 966 cm⁻¹; ¹H-NMR (CDCl₃, 600 MHz) δ 1.22 (t, 3H, *J* = 7.2 Hz), 2.36 (s, 3H), 2.86 (dq, 1H, *J* = 12.0 and 7.2 Hz), 2.92 (dq, 1H, *J* = 12.0 and 7.2 Hz), 6.01 (d, 1H, *J* = 9.0 Hz), 6.17 (d, 1H, *J* = 16.2 Hz), 6.33 (dd, 1H, *J* = 16.2 and 9.0 Hz), 7.20 (d, 2H, *J* = 8.4 Hz) and 7.74 (d, 2H, *J* = 8.4 Hz); ¹³C-NMR (CDCl₃, 150 MHz) δ 7.2, 21.6, 44.5, 126.4, 127.4, 129.4, 130.9, 141.5 and 141.9; Anal. Calcd. for C₁₁H₁₅NO₂S₂: C, 51.33; H, 5.87; N, 5.44. Found: C, 51.54; H, 5.91; N, 5.51.

S-Phenyl-S-1,2-dimethylpropenyl-N-(toluene-4-sulfonyl)sulfilimine (46). To a solution containing 0.85 g (4.8 mmol) of 1,2-dimethylpropenylbenzene sulfide⁷ in 100 mL of EtOH at rt was added 0.13 mL (2.3 mmol) of AcOH and 1.3 g (4.8 mmol) of chloramine-T trihydrate. After stirring at rt for 20 h, the reaction mixture was quenched with H₂O and extracted with EtOAc. The organic layer was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to provide 0.9 g (55%) of **46** as a white solid: mp 92-94 °C; IR (KBr) 1282, 1140, 1088, 965, 761 and 653 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.67 (s, 3H), 1.83 (s, 3H), 2.16 (s, 3H), 2.36 (s, 3H), 7.19 (d, 2H, *J* = 8.2 Hz), 7.46-7.49 (m, 3H), 7.54-7.57 (m, 2H) and 7.79 (d, 2H, *J* = 8.2 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 10.5, 21.3, 22.1, 22.3, 125.9, 126.3, 126.8, 129.0, 129.5, 131.1, 133.5, 141.4, 141.8 and 147.7.

(Z)-S-2-Phenylethenyl-S-phenyl-N-(toluene-4-sulfonyl)sulfilimine (50). To a solution containing 0.75 g (3.5 mmol) of (*Z*)-phenyl styryl sulfide and 0.09 g (0.18 mmol) of hexadecyl-tributylphosphonium bromide in 50 mL of CH₂Cl₂ was slowly added 1 g (3.5 mmol) of chloramine-T trihydrate. The reaction mixture was stirred at room temperature for 24 h, the solvent was removed under reduced pressure and the residue was purified by flash silica gel chromatography to give 0.47 g (35%) of **50** as

a colorless oil: IR (neat) 1445, 1283, 1141 and 959 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 2.32 (s, 3H), 6.32 (d, 1H, $J = 10.4$ Hz), 7.09 (d, 2H, $J = 8.0$ Hz), 7.16 (d, 1H, $J = 10.4$ Hz), 7.37-7.43 (m, 5H), 7.45-7.50 (m, 3H), 7.65 (dd, 2H, $J = 8.0$ and 1.6 Hz) and 7.70 (d, 2H, $J = 8.0$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 21.6, 126.2, 126.3, 126.6, 129.1, 129.3, 129.7, 130.1, 130.5, 132.2, 132.7, 136.3, 141.6, 141.7 and 141.8.

(*E*)-*S*-2-Phenylethenyl-*S*-phenyl-*N*-(toluene-4-sulfonyl)sulfilimine (51). To a solution containing 1.5 g (5.1 mmol) of sulfilimine **17** in 100 mL of THF at -78 $^\circ\text{C}$ was slowly added 2.1 mL (5.1 mmol) of *n*-BuLi solution (2.4 M hexane), 0.6 g (5.1 mmol) of potassium *tert*-butoxide and 0.74 mL (5.1 mmol) of diethyl chlorophosphate. After stirring at -78 $^\circ\text{C}$ for 10 min, 0.5 mL (5.1 mmol) of phenyl acetaldehyde was added dropwise and the mixture was warmed to 0 $^\circ\text{C}$ for 40 min. A saturated aqueous NH_4Cl solution was then added and the aqueous layer was extracted with CH_2Cl_2 . The combined organic layer was washed with brine, dried over MgSO_4 and concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to give 0.38 g (20%) of **51** as a colorless oil: ^1H -NMR (CDCl_3 , 300 MHz) δ 2.28 (s, 3H), 6.58 (d, 1H, $J = 15.3$ Hz), 7.13 (d, 2H, $J = 8.1$ Hz), 7.30-7.36 (m, 6H), 7.43-7.49 (m, 3H), 7.64-7.69 (m, 2H) and 7.76 (d, 2H, $J = 8.1$ Hz); ^{13}C -NMR (CDCl_3 , 75 MHz) δ 21.5, 122.6, 126.4, 126.9, 128.2, 129.1, 129.4, 130.1, 130.9, 132.4, 132.7, 135.4, 141.5, 141.6 and 141.8.

(*E*)-*S*-2-(4-methoxy-phenyl)-ethenyl-*S*-phenyl-*N*-(toluene-4-sulfonyl)sulfilimine (52). To a solution containing 1.0 g (3.4 mmol) of sulfilimine **7** in 100 mL of THF at -78 $^\circ\text{C}$ was slowly added 1.4 mL (3.4 mmol) of *n*-BuLi solution (2.4 M hexane), 0.4 g (3.4 mmol) of potassium *tert*-butoxide and 0.5 mL (3.4 mmol) of diethyl chlorophosphate. After stirring at -78 $^\circ\text{C}$ for 10 min, 0.4 mL (3.4 mmol) of *p*-anisaldehyde was added dropwise and the mixture was warmed to 0 $^\circ\text{C}$ for 40 min. A saturated aqueous NH_4Cl solution was added and the aqueous layer was extracted with CH_2Cl_2 . The combined organic layer was washed with brine, dried over MgSO_4 and

concentrated under reduced pressure. The residue was purified by flash silica gel chromatography to give 0.27 g (19%) of **52** as a colorless oil: IR (neat) 1602, 1512, 1141 and 1088 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 2.28 (s, 3H), 3.79 (s, 3H), 6.43 (d, 1H, J = 15.2 Hz), 6.84 (d, 2H, J = 8.4 Hz), 7.12 (d, 2H, J = 8.0 Hz), 7.25 (d, 1H, J = 15.2 Hz), 7.30 (d, 2H, J = 8.4 Hz), 7.43-7.49 (m, 3H), 7.66 (dd, 2H, J = 8.0 and 1.6 Hz) and 7.75 (d, 2H, J = 8.0 Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 21.5, 55.6, 114.6, 119.9, 125.5, 126.5, 126.8, 129.3, 130.0, 130.1, 132.2, 135.9, 141.7, 141.8, 142.1 and 162.0.

S-1-Cyclopentenyl-S-phenyl-N-(toluene-4-sulfonyl)sulfilimine (61). To a solution containing 0.1 g (0.56 mmol) of 1-(phenylthio)cyclopentene⁸ in 20 mL of ethanol was slowly added 0.16 g (0.56 mmol) of chloramine-T trihydrate which contained 1 drop of acetic acid. The reaction mixture was stirred at room temperature for 1 h. The solvent was removed under reduced pressure and the residue was purified by flash silica gel chromatography to give 0.12 g (60%) of **61** as a colorless oil: IR (neat) 1276, 1127, 968 and 661 cm^{-1} ; ^1H -NMR (CDCl_3 , 300 MHz) δ 1.86-1.97 (m, 2H), 2.05-2.18 (m, 1H), 2.34 (s, 3H), 2.40-2.52 (m, 3H), 6.52-6.55 (m, 1H), 7.16 (d, 2H, J = 8.1 Hz), 7.42-7.52 (m, 3H), 7.62 (dd, 2H, J = 7.5 and 2.1 Hz) and 7.74 (d, 2H, J = 8.1 Hz); ^{13}C -NMR (CDCl_3 , 75 MHz) δ 21.7, 23.0, 29.1, 33.4, 126.4, 126.8, 129.1, 129.3, 132.1, 133.6, 138.0, 141.6 and 143.3.

S-1-Cyclohexenyl-S-phenyl-N-(toluene-4-sulfonyl)sulfilimine (62). To a solution containing 0.1 g (0.53 mmol) of 1-(phenylthio)cyclohexene⁸ in 20 mL of ethanol was slowly added 0.15 g (0.53 mmol) of chloramine-T trihydrate which contained 1 drop of acetic acid. The reaction mixture was stirred at room temperature for 8 h. The solvent was removed under reduced pressure and the residue was purified by flash silica gel chromatography to give 0.13 g (66%) of **62**⁹ as a colorless oil: IR (neat) 1282, 1142, 1088 and 966 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 1.44-1.66 (m, 5H), 2.11-2.25 (m, 3H), 2.35 (s, 3H), 6.67-6.71 (m, 1H), 7.16 (d, 2H, J = 8.0 Hz), 7.43-7.49

(m, 3H), 7.61 (dd, 2H, $J = 7.6$ and 1.6 Hz) and 7.75 (d, 2H, $J = 8.0$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 20.9, 21.3, 21.6, 22.1, 26.5, 126.5, 126.8, 129.2, 129.8, 131.9, 133.3, 135.8, 140.0, 141.6 and 141.9.

S-1-(2-Methyl)-cyclopentenyl-S-phenyl-N-(toluene-4-sulfonyl)sulfilimine (63). To a solution containing 0.03 g (0.16 mmol) of 1-(phenylthio)-2-methylcyclopentene in 10 mL of ethanol was slowly added 0.045 g (0.16 mmol) of chloramine-T trihydrate which contained 1 drop of acetic acid. The reaction mixture was stirred rt for 1 h. The solvent was removed under reduced pressure and the residue was purified by flash silica gel chromatography to give 0.049 g (86%) of **63** as a colorless oil: IR (neat) 1281, 1141, 1088, 959 and 750 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 1.67-1.85 (m, 2H), 1.89-1.95 (m, 1H), 1.96 (s, 3H), 2.35 (s, 3H), 2.34-2.49 (m, 2H), 2.64-2.73 (m, 1H), 7.17 (d, 2H, $J = 8.0$ Hz), 7.44-7.48 (m, 3H), 7.56-7.59 (m, 2H) and 7.76 (d, 2H, $J = 8.0$ Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 15.5, 21.3, 21.6, 28.5, 39.4, 126.1, 126.5, 129.2, 129.7, 129.8, 131.5, 133.7, 141.6, 141.9 and 156.5.

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