

SUPPORTING INFORMATION:

Experimental details¹

Methylene chloride and triethylamine were distilled from calcium hydride, tetrahydrofuran and ether from sodium/benzophenone. Acetonitrile was dried over 4Å molecular sieves. Triflic anhydride was prepared from the acid².

Analyses were performed on the following instruments:

IR: *Perkin Elmer* 683; ¹H NMR *Varian* XL-300 (300 MHz) and *Varian* Nova 400 (400 MHz); ¹³C NMR *Varian* XL-300 (75 MHz) and *Varian* INOVA-400 (100 MHz). GC Mass Spectrometry was performed at the *University of Massachusetts (Boston)*, and elemental analyses were performed by *QTI* (Whitehouse, NJ).

For NMR spectra, chemical shifts are given in ppm (δ) upfield from tetramethylsilane (TMS) for ¹H, ¹³C. ¹H spectra are referenced to TMS directly; ¹³C spectra are referenced to residual protons in CDCl₃ (77.23), DMSO-d₆ (39.15) and methanol-d₄ (49.51). In the description of the spectra, s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet. The prefix b is used to indicate a broad peak. Peaks that are considerably smaller than most in the ¹³C spectra are shown with an asterisk; these typically denote quaternary carbons.

Where necessary, off-resonance decoupling (*Varian* XL-300) or gradient COSY (*Varian* INOVA-400) experiments were run to clarify ¹H NMR assignments. For ¹³C assignments, attached proton test (APT) experiments were routinely used, using a 7ms delay. This sequence causes methyl and methine carbons to appear up (shown in the text as (+)), while methylene and quaternary carbons appear down (shown as (-)).

Preparation of *rac*-1-(benzenesulfinyl)cyclohexene (10)

(a) Preparation of 1-(phenylthio)cyclohexene .

A simplified version of the method of B. Labiad & D. Villemin³ was developed, and this was found to be more effective than the literature method. In this modified procedure, 8 g of Montmorillonite KSF was added into a 500 mL found-bottomed flask along with 3.93 g (40 mmol) of cyclohexanone and 200 mL of toluene. 4.1 mL (4.4 g, 40 mmol) of thiophenol was then added, and the mixture was heated to reflux, with azeotropic removal of water via a Dean and Stark trap. After 3 h, when water collection had ceased, the mixture was cooled and filtered. The toluene layer was washed with 2 x 100 mL of 10% sodium carbonate solution followed by 2 x 100 mL of water. After drying the solution over sodium sulfate, toluene was removed *in vacuo*. The crude product (7.5 g of an amber-colored oil) was then distilled in a Kugelrohr furnace as in the literature procedure (collecting the fraction at 105-115 °C at 0.2mm Hg pressure) to give 5.38 g (70.6% yield) of **44** as a clear colorless liquid: ¹H NMR (CDCl₃) δ 1.56-1.71 (m, 4H), 2.10-2.18 (m, 4H), 6.07 (t, J ~ 1Hz, 1H), 7.15-7.33 (m, 5H); ¹³C NMR (CDCl₃) δ 22.8, 24.7, 127.4, 130.0, 131.2, 132.7*, 133.9, 136.4.

(b) Preparation of racemic 1-(benzenesulfinyl)cyclohexene (10)

1.90 g (10 mmol) 1-(phenylthio)-cyclohexene was dissolved in 15 mL of methylene chloride and the clear, almost colorless solution was cooled to -40 °C. Solid *m*-chloroperbenzoic acid assayed as 63% (2.74 g, 10mmol) was added over approximately 30 minutes at -40 °C. The white slurry was stirred at -40 °C for 3 h, and then allowed to warm slowly to room temperature. The solution (now clear) was washed with saturated NaHCO₃ solution, dried over Na₂SO₄, then evaporated in vacuo to yield 2.29 g pale yellow oil. This was purified using column chromatography to give 1.24 g (60%) of **47** as

a white solid: ^1H NMR (CDCl_3) δ 1.45-1.70 (m, 5H), 2.13-2.28 (m, 3H), 6.69-6.73 (m, 1H), 7.43-7.52 (m, 3H), 7.57-7.61 (m, 2H); APT NMR (CDCl_3) δ 19.5(+), 21.8(+), 22.1(+), 25.9(+), 124.8(+), 129.1(+), 130.6(+), 134.5(+), 142.8*(-), 143.9(-).

Preparation of *rac*-3-(benzenesulfinyl)-2-methyl-2-cyclohexenone⁴ (11b)

(a) Preparation of 2-methyl-3-(phenylthio)-2-cyclohexenone

20 g (80 mmol) of 2-methyl-1,3-cyclohexanedione was added to 70 mL of acetonitrile (dried over 4Å sieves), and 13 mL (8.5 g, 90 mmol) of dry (from CaH_2) triethylamine* was added. The resultant clear yellow solution was cooled to 0-5 °C. Methanesulfonyl chloride (6.5 mL, 9.5 g, 85 mmol) was slowly added (exothermic), keeping the temperature below 10 °C. After stirring for 1 h at 0-10 °C, a further 13 mL of dry triethylamine* was added. Thiophenol (8.5 mL, 9.0 g, 82 mmol) was added slowly (exothermic) maintaining the temperature at around 4-7 °C, then the reaction mixture was allowed to warm up and stir overnight.

From this point onwards our simplified workup procedure was used in place of the Trost³ method. The mixture was concentrated down *in vacuo* (in hood) until very thick, then poured into 250 mL of water and stirred for 1 h at room temperature. The solid product was simply filtered off and recrystallized (not part of Trost's method⁴) from a methanol/water (38 mL/7 mL) solvent pair to yield 12.8 g (73.3% yield) of 2-methyl-3-(phenylthio)-2-cyclohexenone, as large cream-colored crystals m.p. 89-90 °C. (One spot at R_f 0.25, hexane:EtOAc 4:1): ^1H NMR (CDCl_3) δ 1.87 (quintet, $J = 6\text{Hz}$, 2H), 1.97 (t, $J = 1\text{ Hz}$, 3H), 2.18 (td, $J_1 = 6\text{Hz}$, $J_2 = 1\text{Hz}$, 2H), 2.38 (t, 6Hz, 2H), 7.35-

* Trost used diisopropylethylamine as base.

7.43 (m, 3H), 7.46-7.52 (m, 2H); ^{13}C NMR (CDCl_3) δ 9.2, 22.7, 30.2, 37.0, 129.26, 129.33, 130.0*, 135.4, 158.0*, 195.5* (One quaternary aromatic C not observed).

(b) Conversion to racemic 3-(benzenesulfinyl)-2-methyl-2-cyclohexenone (**11b**)

2-Methyl-3-(phenylthio)-2-cyclohexenone, (4.36 g, 10 mmol) was dissolved in 300 mL of methylene chloride and the clear, colorless solution was cooled to 0-5 °C. Solid *m*-chloroperbenzoic acid (4.40 g, 75% by titration, 19.1 mmol) was added over 20-30 minutes at 0-5 °C. The mixture was stirred for 3-4.5 h, allowing the mixture to slowly warm up to room temperature during the first 1-2 h; the white suspension dissolved as the mixture warmed up. After reaction, the solution was washed with 100 mL of saturated NaHCO_3 solution, then dried over Na_2SO_4 . The solvent was removed *in vacuo*, then the residue was purified by flash chromatography through 250 g silica gel. This gave 4.15 g (88.5%) cream-colored solid, m.p. 54.5-57 °C, as well as 0.49 g (11.2%) recovered unreacted thioether. The sulfoxide gave the following spectra: ^1H NMR (CDCl_3) δ 1.90-2.08 (m, 3H), 2.21 (t, 1H, 3H), 2.29-2.40 (m, 1H), 2.47-2.57 (m, 1H), 2.83-2.96 (m, 1H), 7.47-7.67 (m, 5H); APT NMR (CDCl_3) δ 12.7(+), 18.9(-), 22.1(-), 37.6(-), 123.8(+), 129.4(+), 131.0(+), 136.1*(-), 142.2*(-), 160.0*(-), 197.8*(-).

Preparation of *rac*-3-(benzenesulfinyl)-2-cyclohexenone (**11a**)

(a) Preparation of 3-(phenylthio)-2-cyclohexenone.

This was run on a 40 mmol scale (starting from 1,3-cyclohexanedione) using the the Trost⁴ method, but with revised work-up as above for **11b**. After the reaction mixture was stirred overnight, it was worked up without distillation or an aqueous quench. The lower layer (a yellow oil) was simply separated off to give 8.6 g crude product, which after chromatography though 150 g silica gel gave 6.84 g of ivory-colored crystals, m.p.

37.5-41 °C (83.8% yield): ^1H NMR (CDCl_3) δ 2.05 (quintet, $J = 6.3\text{Hz}$, 2H), 2.38 (t, $J = 6.6\text{Hz}$, 2H), 2.53 (tt, $J_1 = 5.8\text{Hz}$, $J_2 = 6.6\text{Hz}$, 2H), 5.48 (t, $J = 1.1\text{Hz}$, 1H), 7.35-7.60 (m, 5H); ^{13}C NMR (CDCl_3) δ 23.1, 30.4, 37.4, 121.0, 128.2*, 130.2, 135.7, 166.9*, 196.2*.

(b) Preparation of racemic 3-(benzenesulfinyl)-2-cyclohexenone (**11a**)

The same method as for **11b** was used, and gave an 85.6% yield of a light tan-colored solid, m.p. 78-81 °C after chromatography: ^1H NMR (CDCl_3) δ 1.93-2.08 (m, 2H), 2.21 (td, $J_1 \sim 7\text{Hz}$, $J_2 \sim 1\text{Hz}$, 2H), 2.34 (td, $J_1 \sim 7\text{Hz}$, $J_2 \sim 1\text{Hz}$, 2H), 6.75 (t, $J \sim 1\text{Hz}$, 1H), 7.51-7.58 (m, 3H), 7.61-7.73 (m, 2H); ^{13}C NMR (CDCl_3) δ 22.4, 22.8, 38.2, 125.5, 126.6, 129.9, 132.3, 141.9*, 167.6*, 196.8*.

Preparation of 2-Iodo-5-(2-nitroethyl)phenol

3-(Nitroethyl)phenol was first prepared from 3-hydroxybenzaldehyde via a Henry reaction⁵ followed by sodium borohydride reduction⁶. Then an *ortho*-iodination was carried out by addition of iodine to 3-(nitroethyl)phenol in the presence of thallium(I) acetate (CAUTION- highly toxic!). The method of P.C. Cambie⁷ was used, except that iodine was added over 24 h to help react all of the starting material. Also, a 25% excess of iodine was used, because the starting material is difficult to separate from the desired product; the 2,6-diiodo compound is much easier to remove by chromatography. This gave 1.45 g (55% yield) of 2-iodo-5-(2-nitroethyl)phenol as an off-white solid: ^1H NMR (CDCl_3) δ 3.25, (t, $J = 8\text{Hz}$, 2H), 4.59 (t, $J = 8\text{Hz}$, 2H), 5.32 (s, 1H), 6.54 (dd, $J_1 = 8\text{Hz}$, $J_2 = 2\text{Hz}$, 1H), 6.85 (d, $J = 2\text{Hz}$, 1H), 7.60 (d, $J = 8\text{Hz}$, 1H); ^{13}C NMR (CDCl_3) δ 33.0, 75.9, 84.5, 115.5, 122.8, 138.3*, 138.9, 155.4.

In addition, a 21.2% yield of 2,6-diiodo-5-(2-nitroethyl)phenol **54b** was isolated as a by-product: $^1\text{H NMR}$ (CDCl_3) δ 3.46 (t, $J = 7.5\text{Hz}$, 2H), 4.61 (t, $J = 7.5\text{Hz}$, 2H), 5.91 (s, 1H), 6.58 (d, $J = 8.1\text{Hz}$, 1H), 7.62 (d, $J = 8.1\text{Hz}$, 1H).

The Pericyclic Reaction

(a) General Methods

Shown for reaction of *rac*-3-(benzenesulfinyl)-2-methyl-2-cyclohexenone (**11b**) with *para*-cresol mediated by trifluoroacetic anhydride

Three methods were used, shown for the successful case of trifluoroacetic anhydride (TFAA) as electrophilic activating agent. When other electrophiles were tested, these were added in place of TFAA.

Method A: Dry methylene chloride (2 mL) (or CDCl_3 , which freezes at -60°C) and trifluoroacetic anhydride (TFAA, 0.70 mL, 0.5mmol) were mixed and cooled to -78°C to -60°C . The TFAA forms a separate layer below about -60°C . Sulfoxide **11b** was added over 5-10 minutes, followed by *p*-cresol over 5-10 minutes, all at -78°C to -60°C . The mixture was slowly warmed up to around 0 - 10°C for about ten minutes. After recooling to -78°C to -60°C , triethylamine was added, then the mixture was allowed to warm up to room temperature and stir (typically overnight). Note: triethylamine serves merely to quench the reaction and prevent degradation of product. The solvent was removed, then the product was isolated by flash chromatography using silica gel. Typically the chromatography was performed twice: once to quickly separate the product, the *p*-cresol, **12** from **11b**, the TFAA salts and other materials, then a second time (using 10:1 hexane:ethyl acetate) more carefully, to purify the product. Often a further recrystallization from methanol was necessary. The product **24** was isolated in 43% yield as a white crystalline solid m.p. 121 - 4°C (97 - 110°C before recrystallization).

Method B: as for method A, but the reaction was run at -40°C to -30°C (dry ice/acetonitrile) throughout, in CH_2Cl_2 . Sulfoxide addition was carried out over 10-15 minutes, whilst phenol addition was performed over 1.5-2 h, then the mixture was allowed to react for 1-1.5 hrs at -40°C to -30°C . Triethylamine was added at -40°C to -30°C before warming up to room temperature. In a few experiments an aqueous workup (water, HCl, then Na_2CO_3) was used, but this was found to be slightly detrimental to the product in a few cases and was therefore it was omitted for most experiments. Generally the same workup was used as for method A, although the methanol recrystallization was often unnecessary. The product was isolated as a white crystalline solid in 55% yield.

Method C: As for method A, but the reaction was performed entirely at room temperature. The reactants were given 5 minutes before triethylamine was added to quench the reaction, then workup was as for method A.

The reaction was also tested in like manner (method A unless stated) with oxalyl chloride, trifluoromethanesulfonic anhydride, methanesulfonic anhydride, thionyl chloride, trifluoroacetyl trifluoromethanesulfonate, N-phenyltrifluoromethanesulfonimide (method B), acetic anhydride (method B). In all cases the reaction gave little or no desired product.

(b) Specific reactions

Reaction of phenyl vinyl sulfoxide 9 with phenol mediated by TFAA

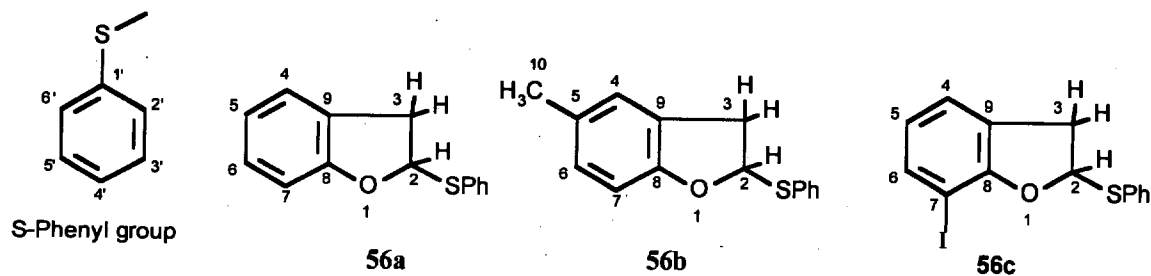
Method A was used, on a 5 mmol scale. The crude product solution (in methylene chloride) was purified by extraction with 2 x 10 mL of 25% sodium hydroxide solution, followed by 10 mL of water. After drying over sodium sulfate, the methylene chloride was removed in vacuo to give a crude yellow oil (0.6 g). This oil was then purified by Kugelrohr distillation (collecting the fraction b.p. 150-160 °C at 20mm Hg) to give 0.441 g (43% yield) of 2-phenylthio-2,3-dihydrobenzofuran as a clear colorless liquid: ¹H NMR (CDCl₃) δ 3.16 (dd, J₁ = 17Hz, J₂ = 5Hz, 1H), 3.63 (dd, J₁ = 17Hz, J₂ = 9Hz, 1H), 6.15 (dd, J₁ = 9Hz, J₂ = 5Hz, 1H), 6.86 (q, J = 8Hz, 2H), 7.11-7.36 (m, 5H), 7.45-7.63 (m, 2H); ¹³C NMR (CDCl₃) δ 36.6, 89.0, 110.1, 121.1, 124.5, 125.7, 127.4, 128.2, 128.8, 131.1*, 131.6, 133.8*, 157.9*.

For NMR spectral assignments for this and related compounds, see Table S1.

Reaction of sulfoxide 11b with *o*-(tosyloxy)phenol

Method A was used, on a 2.5 mmol scale. This gave 0.44 g (41% yield if pure) of the desired product as light brown crystals, contaminated with an impurity, believed to be catechol ditosylate (present in the starting phenol). A trace of this remained even after recrystallization from methanol (gave m.p. 79-81 °C): ¹H NMR (CDCl₃) δ 1.34-1.45 (m, 1H), 1.58 (s, 3H), 1.67-1.77 (m, 1H), 1.92-2.06 (m, 2H), 2.24-2.49 (m, 2H), 2.36 (s, 3H), 6.77 (br s, 2H), 7.01 (approx. d, J = 8Hz, 1H), 7.09-7.19 (m, 2H), 7.24-7.47 (m, 3H), 7.52-7.71 (m, 2H), 7.74-7.79 (m, 2H). Anal. C₂₆H₂₄O₅S₂: calcd. C, 64.98; H, 5.03; S, 13.34; found C, 64.68; H, 4.93; S, 13.34.

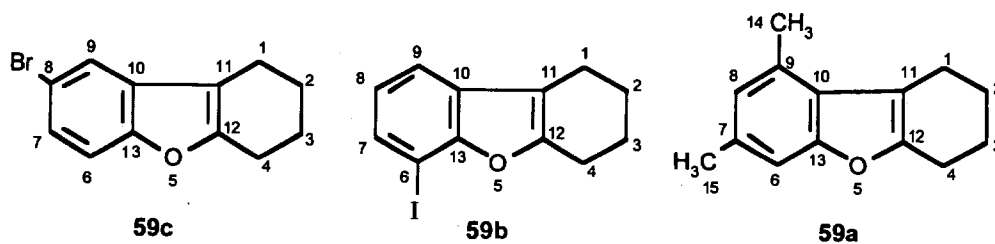
For NMR spectral assignments for related compounds, see Table S1.

Table S1. Cyclization products derived from phenyl vinyl sulfoxide 18

Posn.	56a		56b		56c	
	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C
2	6.15dd 9, 5Hz	89.0	6.15dd 8, 4.9Hz	89.2	6.14dd 9.0, 4.5Hz	89.2
3 (cis/trans are relative to -SPh)	cis 3.16dd 17, 5Hz trans 3.63 dd 17, 9Hz	36.6	cis 3.13dd 16, 4.9Hz trans 3.62 dd 16, 8Hz	36.8	cis 3.26dd 16.8, 4.5Hz trans 3.74dd 16.8, 9.0Hz	38.1
4	7.11-7.36m	125.7	6.98s	127.5	7.09dd 7.3, 1.0Hz	126.3
5	6.86q 8Hz	121.1	-	125.9	6.63t 7.7Hz	124.6
6	7.11-7.36m	124.5	6.94d 8.6Hz	125.2	7.48dd 7.8, 1.0Hz	137.2
7	6.86q 8Hz	110.1	6.74d 8.6Hz	109.8	-	74.9(!)
8	-	157.9	-	156.0	-	158.7
9	-	133.8	-	134.1	-	123.2
10	-	-	2.27s	20.8	-	-
1'	-	131.1	-	130.4	-	132.9
2', 6'	7.43-7.63m	128.8	7.45-7.58m	128.9	7.60-7.72m	129.1
3', 5'	7.11-7.36m	131.6	7.16-7.40m	131.6	7.24-7.42m	133.4
4'	-	128.2	7.16-7.40m	128.7	7.24-7.42m	128.4

Compound numbers given are derived from Ref. 1.

Table S2. Cyclization products derived from 11



Posn.	59c		59b		59a	
	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C
1	2.73tt 6.3, 1.6Hz	23.6	2.76t 6Hz	-	2.81t 6Hz	23.7
2	1.89-1.97m	22.7	1.67-2.12m	-	1.78-1.95m	22.9
3	1.80-1.88m	20.5	1.67-2.12m	-	1.78-1.95m	22.8
4	2.57tt 6.1, 1.6Hz	23.0	2.57t 6Hz	-	2.70t 6Hz	23.3
6	7.22-7.30m	112.4	-	-	6.73s	108.9
7	7.22-7.30m	121.4	7.15- 7.58m [†]	-	-	130.2
8	-	112.8	6.91t 8Hz	-	7.01s	124.9
9	7.53s	125.4	7.15- 7.58m [†]	-	-	133.0
10	-	131.2	-	-	-	125.2
11	-	115.4	-	-	-	113.0
12	-	153.3	-	-	-	152.8
13	-	155.8	-	-	-	154.9
14	-	-	-	-	2.52	19.0 [‡]
15	-	-	-	-	2.38	19.0 [‡]

[†] Exact peak position unclear due to contamination (diphenyl disulfide).

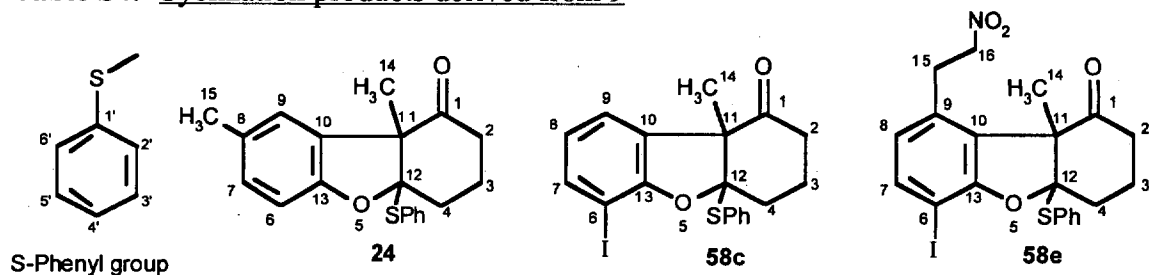
[‡] Assumed to be accidentally equivalent.

Compound numbers given are derived from Ref. 1.

Table S3. Cyclization products derived from **10**

Posn.	60a		60b		60e		60d		60c	
	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C
1	-	194.9	-	-	-	-	-	-	-	194.9
2	3.02t 6.3Hz	38.0	3.01t 6Hz	-	3.04t 6.2Hz	-	2.98t 6Hz	-	3.10t 6.3Hz	38.1
3	2.28 ~quin 6Hz	22.7	2.27 ~quin 6Hz	-	2.28 ~quin 6.4Hz	-	2.22 ~quin 7Hz	-	2.29 quin 6.4Hz	22.7
4	2.60t 6Hz	24.0	2.59t 6Hz	-	2.60t 6.2Hz	-	2.60t 7Hz	-	2.61 ~t 6Hz	24.2
6	7.26-7.37m	111.2	7.33d 8Hz	-	7.33d 8.6Hz	-	7.01d 3Hz	-	-	74.7
7	7.42-7.50m	124.6	7.12dd 8, 1Hz	-	7.41dd 8.6, 2.1Hz	-	-	-	8.01dd 7.7, 1.0Hz	134.3
8	7.26-7.37m	121.9	-	-	-	-	6.93dd 9.3Hz	-	7.08t 7.7Hz	122.0
9	7.42-7.50m	125.1	7.86d 1Hz	-	8.19d 2.1Hz	-	7.89d 9Hz	-	7.66dd 7.7, 1.0Hz	126.5
10	-	123.9	-	-	-	-	-	-	-	124.4
11	-	116.7	-	-	-	-	-	-	-	117.6
12	-	171.0	-	-	-	-	-	-	-	171.1
13	-	154.7	-	-	-	-	-	-	-	159.1
14	-	-	2.43s	-	-	-	3.83s	-	-	-

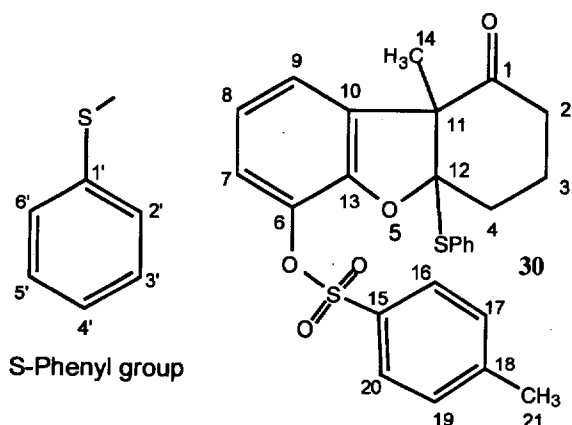
Compound numbers given are derived from Ref. 1.

Table S4. Cyclization products derived from **9**

Posn.	24		58c		58e	
	¹ H	¹³ C	¹ H	¹³ C	¹ H	¹³ C
1	-	209.7	-	209.2	-	210.1
2	2.40-2.46m	37.5	2.34-2.47m	37.5	2.15-2.55m	37.9
3	1.72-1.90m	19.5	1.73-1.92m	19.5	1.75-2.05m	19.5
4	2.17-2.32m	33.2	2.23ddd 16, 14, 6Hz Other H in 2.34-2.47m	33.2	2.15-2.55m	32.5
6	6.78d 7.9Hz	110.2	-	75.0	-	74.9
7	7.05d 7.9Hz	129.0	7.60dd 8, 1.5Hz	132.5	7.30-7.73m	135.5
8	-	125.6 [†]	6.81t 8Hz	123.7	6.61d 8Hz	125.6
9	7.09s	125.6 [†]	7.27dd 9, 1.5Hz	125.3	-	138.7
10	-	130.4	-	129.5	-	129.3
11	-	62.3	-	63.3	-	65.1
12	-	104.3	-	104.6	-	105.0
13	-	154.7	-	157.2	-	158.1
14	1.60s	21.0	1.68s	18.9	1.82s	20.1
15	2.35s	18.6	-	-	3.83t 8Hz	29.3
16	-	-	-	-	4.60-4.70m 4.80-4.90m	37.2
1'	-	131.3	-	131.7	-	135.3
2', 6'	7.56dd 7.3, 1.7Hz	128.6	7.66d 3Hz 7.68d 2Hz	128.8	7.30-7.73m	129.0
3', 5'	7.33-7.41m	137.0	7.36-7.44m	138.0	7.30-7.73m	138.0
4'	7.33-7.41m	129.7	7.36-7.44m	129.3	7.30-7.73m	129.5

Compound #s are derived from Ref. 1. [†] Assumed to be accidentally equivalent.

Table S5 Cyclization products derived from 9(continued)



Posn.	30 (58d)	
	^1H	^{13}C
1	-	209.8
2	2.24-2.49m	37.5
3	1.34-1.45m and 1.67-1.77m	19.1
4	1.92-2.06m	33.1
6	-	146.4
7	6.77br s	121.1
8	6.77br s	118.7
9	7.01 ~d, 8Hz	123.4
10	-	129.0?
11	-	62.6
12	-	106.0
13	-	148.7
14	1.58s	22.0
15	-	137.5
16(&20)	7.74-7.79m	130.2
17(&19)	7.09-7.19m	128.7
18	-	131.4
21	2.36s	21.9
1'	-	129.4
2', 6'	7.52-7.71m	128.9
3', 5'	7.24-7.47m	137.0
4'	7.24-7.47m	129.7

Compound numbers are derived from Ref. 1.

References

- (1) For further information see Walker, M. A. Ph.D. Dissertation, Brandeis University, Waltham, MA, 1999.
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