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4*H*-Dinaphtho[2,1-*f*:1',2'-*h*][1,5]dithionin-*S*,*S*'-tetroxide: Preparation, Structure Elucidation and Facile Intramolecular [2+2]-Photocyclization

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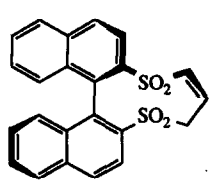
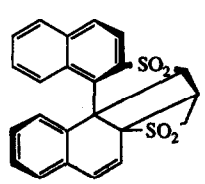
Abstract: The preparation of compound **1**, a conformational study in solution and the unexpectedly facile rearrangement into **2** via photochemical intramolecular [2+2]-cycloaddition is described: the structure of these compounds have been investigated by NMR and *X*-ray diffraction.

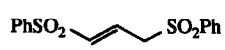
Small molecular entities are often highly activated by the introduction of two arylsulfonyl groups. As examples, bis(phenylsulfonyl)methane,[1] -ethylene,[2] -acetylene,[3] -propene,[4] -butadiene,[5] *etc.* have found practical applications in organic synthesis. The advantages of sulfonyl groups as activating functionalities are the effective activation, the high crystallinity of the derivatives, and especially the fact that they can be easily removed and substituted with hydrogen atoms,[6] thus formally behaving as equivalents of the respective hydrocarbonic moieties. Since we are interested in developing chiral derivatives of these reagents utilizing 1,1'-binaphthalene-2,2'-dithiol as the chiral auxiliary,[7] we have prepared reagent **1** with the aim of generating a chiral version of 1,3-bis(phenylsulfonyl)propenes **3**. Though these reagents were scarcely studied,[4,8] they are, in principle, precursors of the allyl anion **4**. Specifically, compound **1** represents a chiral C_2-symmetrical form of these reagents.



1



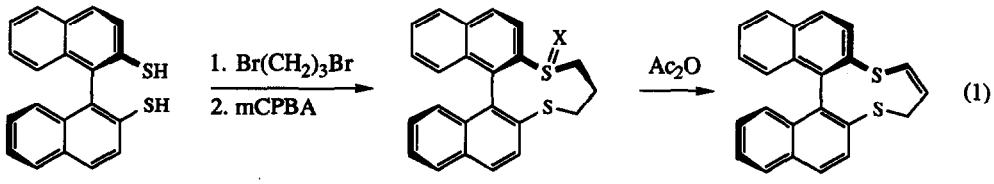
2



3

4

The synthesis of propene **1** was accomplished by oxidation of the corresponding sulfide **7**[9] which, in turn, was obtained by Pummerer rearrangement of the propane **6**, readily available from 1,1'-binaphthalene-2,2'-dithiol and 1,3-dibromopropane followed by oxidation (Eq 1).

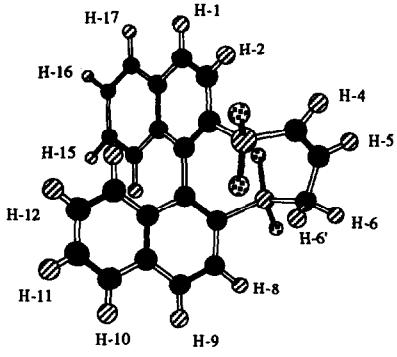


(1)

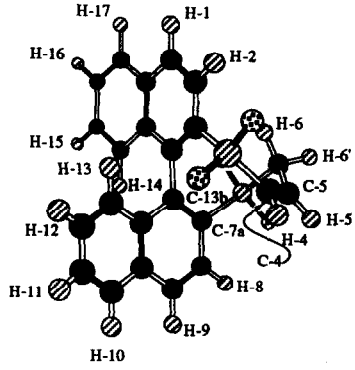
5 (X = lone pair)
6 (X = O)

7

The 400 MHz ^{1}H NMR spectrum of bis(sulfone) **1** in CD_2Cl_2 at room temperature shows two distinct signals for the geminal methylenic protons, as expected on consideration of the atropisomeric nature of the system. The low-field signal is, however, in the form of a very broad singlet, suggesting the possibility of a dynamic process.[10] As a matter of fact, at temperatures between 223 and 203 °K the system is "frozen", displaying the sharp signals of two isomers in *ca.* 10 : 1 ratio, with associated $DG°_{213°K}$ = 1.0 kcal mol^{-1}. Indeed, the presence of two conformers of **1**, deduced by inspection of Dreiding models, could be supported through MM2 force field computational analysis.[11] The two calculated bottom energy structures are shown below.



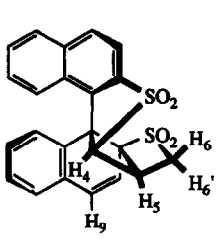
1a



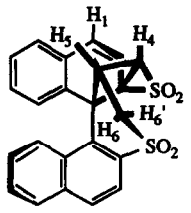
1b

In the more stable (by 1.5 kcal mol^{-1}, in qualitative agreement with the energy difference from NMR data) conformer **1a**, the olefinic double bond is relatively far from both naphthyl residues, while in the less stable conformer **1b**, the olefinic bond is close to the 7a-13b bond of the naphthylsulfonyl residue in allylic position (estimated distances from MM2 calculations: C-4 - C-13b = 3.2 Å; C-5 - C-7a = 2.9 Å), and it is therefore conveniently positioned for undergoing [2+2]-cycloaddition.

On standing in the light (daylight is sufficient), compound **1** converted rapidly into compound **2**, whose structure was investigated by ^{1}H NMR spettroscopy. In the hypothesis of a [2+2]-photocycloaddition,[12] the two intramolecular adducts **2** and **2'** can be anticipated, arising from addition of the olefinic bond to the allylic or vinylic naphthylsulfonyl residue respectively.



2



2'

An exhaustive n.O.e. investigation on **2** (Figure 1) allowed discrimination between the two possibilities.

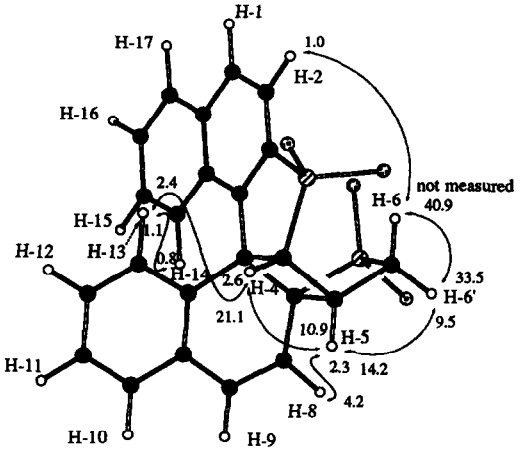


Figure 1 - Main n.O.e. enhancements observed for **2** with atom labels used in the ^{1}H NMR data description.

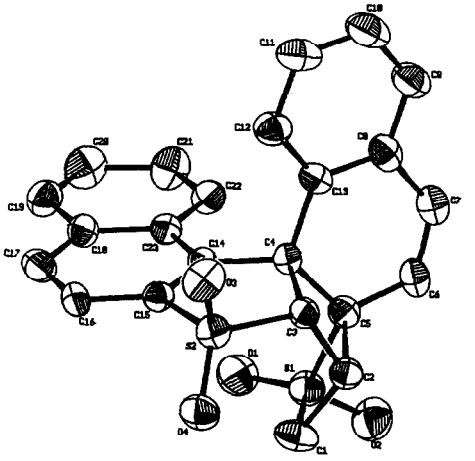
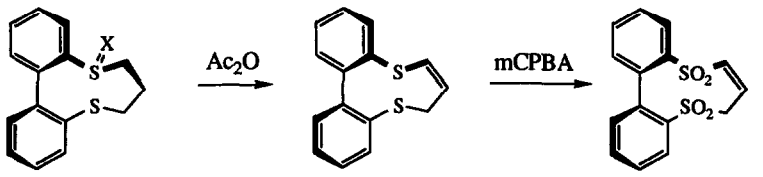


Figure 2 - ORTEP drawing of structure **2** with the atom labels used in the X-ray data. Hydrogen atoms are omitted.

From the consideration of selected dipolar interactions, it is possible to assign the "external" (H-8 and H-9) and "internal" (H-13 and H-14) naphthyl resonances, as well as the methylenic (H-6) and cyclobutyl (H-4 and H-5) resonances. Diagnostic interactions are found between H-5 and the "external" H-8 resonances, and between H-4 and the "internal" (H-13) ones, thus allowing the assignment of structure **2**. Structure **2'** would have required the reversed interaction scheme. The structure of **2** was confirmed by diffractometric analysis. The ORTEP view is reported in Figure 2.

The conversion of **1** into **2** brings about the generation of two strained four-membered rings and a net loss of aromaticity. Nevertheless, the formation of two new σ bonds compensates for the destabilization. For the sake of comparison, we have prepared the biphenyl derivative **8** with the same procedure used for **1** (Eq 2).



(2)

9 (X = lone pair)
10 (X = O)

11

8

Also **8** presents some broadened signals in the ^{1}H NMR spectrum at room temperature, suggesting the possibility of a dynamic process similar to that of **1**. In this instance, however, no conversion to a [2+2] internal adduct was observed, probably because the energy due to the loss of aromaticity is now excessive.

Experimental Section

2,3,4-Trihydrodinaphtho[2,1-*f*:1',2'-*h*][1,5]dithionin (5): 1,1'-Dinaphtho-2,2'-dithiol[13] (1.0 g, 3.14 mmol) was added at 0 °C to a solution of sodium ethoxide obtained from sodium (0.15 g, 6.5 mmol) in 70 mL of ethanol. After 10 min 1,3-dibromopropane (0.32 mL, 3.14 mmol) diluted in 10 mL of absolute ethanol was added dropwise at the same temperature. The reaction mixture was kept stirring at 0 °C for 2 h and let reaching room temperature overnight. After evaporating most of the solvent in a rotary evaporator and adding water (100 mL), the reaction mixture was extracted with dichloromethane (3 x), dried (Na_2SO_4) and concentrated to afford a solid which was recrystallized from dichloromethane-*n*-hexane (1.06 g, 94% yield): mp 281-2 °C. ^{1}H NMR ($CDCl_3$, 300 MHz), δ 2.00-2.08 (2 H, m), 2.62-2.72 (2 H, m), 2.79-2.87 (2 H, m), 7.03-7.07, 7.23-7.29, 7.44-7.50, 7.77-7.81, 7.90-7.97 (12 H, series of m, Ar). IR (KBr, cm^{-1}) 3035, 2949, 2909, 1493, 1400, 1382, 1341, 1313, 1201, 1017, 824, 817, 754. *Elem. Anal.* (Calcd for $C_{23}H_{18}S_2$): C, 76.8 (77.0); H, 5.3 (5.1).

2,3,4-Trihydrodinaphtho[2,1-*f*:1',2'-*h*][1,5]dithionin-*S*-oxide (6): A dichloromethane solution (30 mL) of *m*-chloroperbenzoic acid (mCPBA, 70-75%, 0.148 g, 0.64 mmol) was added dropwise at -30 °C within 3 h to a solution of **5** (0.23 g, 0.64 mmol) in the same solvent (30 mL). After 7 h the reaction mixture was brought to room temperature, washed with sodium bisulfite (1 x), potassium carbonate (1 x) and water (2 x). After drying ($MgSO_4$) and evaporating the solvent, a crude solid material consisting of a single diastereomeric sulfoxide was obtained which was recrystallized from dichloromethane-ethyl ether (0.228 g, 95% yield): mp 275-6 °C. ^{1}H NMR ($CDCl_3$, 300 MHz), δ 1.66-1.80 (1 H, m), 2.20-2.42 (2 H, m), 2.86-2.95 (1 H, m), 3.10-3.19 (1 H, m), 3.36-3.44 (1 H, m), 7.10-7.71, 7.92-8.02, 8.23-8.30 (12 H, series of m, Ar). ^{13}C NMR ($CDCl_3$, 75.4 MHz) δ 22.65, 33.17, 57.52, 118.81, 126.19, 126.43, 127.03, 127.47, 127.56, 127.58, 127.63, 128.02, 128.50, 130.07, 130.10, 130.31, 131.78, 131.99, 132.55, 132.78, 134.42, 135.46, 138.54. IR (KBr, cm^{-1}) 3040, 2988, 1490, 1588, 1496, 1421, 1400, 1302, 1255, 1056, 1035, 860, 841, 773, 752. *Elem. Anal.* (Calcd for $C_{23}H_{18}OS_2$): C, 73.5 (73.8); H, 5.2 (4.8).

4*H*-Dinaphtho[2,1-*f*:1',2'-*h*][1,5]dithionin (7): A solution of **6** (1.7 g, 4.54 mmol) in 150 mL of acetic anhydride was heated to reflux for 5 h in the dark. The solvent was removed under reduced pressure and the residue was diluted with dichloromethane and washed several times with sodium carbonate and water. After evaporation of the solvent the residue was flash-chromatographed eluting with *n*-hexane-dichloromethane (3:1) to obtain a solid which was recrystallized from dichloromethane-*n*-hexane (0.30 g, 20% yield): mp 241-2 °C. ^{1}H NMR ($CDCl_3$, 300 MHz), δ 2.74-2.80 (1 H, m), 3.18-3.26 (1 H, m), 5.35-5.46 (2 H, m), 7.03-7.05, 7.25-7.32, 7.44-7.55, 7.59-7.62, 7.80-7.98 (12 H, series of m, Ar). IR (KBr, cm^{-1}) 3040, 1495, 1395, 810, 715. *Elem. Anal.* (Calcd for $C_{23}H_{16}S_2$): C, 77.4 (77.5); H, 4.9 (4.5).

4*H*-Dinaphtho[2,1-*f*:1',2'-*h*][1,5]dithionin-*S*,*S*'-tetroxide (1): A dichloromethane solution (100 mL) of mCPBA (70-75%, 5 g, 20.25 mmol) was added dropwise within 3 h and at 0 °C to a solution of compound **7** in the same solvent (50 mL). After 20 h in the dark, the reaction mixture was washed with sodium bisulfite (1 x), potassium carbonate (1 x) and water (2 x), dried ($MgSO_4$) and rotoevaporated to give a solid which was recrystallized from dichloromethane-*n*-hexane (1.86 g, 86% yield): mp 332-3 °C. ^{1}H NMR ($CDCl_3$, 300 MHz, *ca.* 300 °K), δ 3.64-3.75 (2 H, m), 5.99-6.09 (1 H, m), 6.39 (1 H, d, J = 11.5 Hz), 7.11-7.18, 7.35-7.46, 7.60-7.73, 7.97-8.07, 8.18-8.41 (12 H, series of m, Ar). IR (KBr, cm^{-1}) 3020, 2970, 1332, 1160, 1120, 810, 707. UV (CH_2Cl_2, nm) λ_{max} (ε) 292.8 (12732), 300.8 (12219), 337.6 (8415), 324.0 (6927). *Elem. Anal.* (Calcd for $C_{23}H_{16}S_2O_4$ x 1/3 CH_2Cl_2 as verified by ^{1}H NMR): C, 62.1 (62.4); H, 3.7 (3.7). The 400 MHz ^{1}H NMR spectrum at 213 °K in CD_2Cl_2 showed the presence of two conformers in *ca.* 10 : 1 ratio. Major (only aliphatic resonancies): δ 3.56 (1 H, t, J = 13 Hz), 3.67 (1 H, dd, J = 13 and 8 Hz), 6.07 (1 H, m), 6.39 (1 H, d, J = 12 Hz). Minor (only aliphatic resonancies): δ 4.09 (1 H, dd, J = 14.5 and 12 Hz), 5.15 (1 H, dd, J = 14.5 and 9 Hz), 6.35 (1 H, m), 6.75 (1 H, d, J = 12 Hz).

Photorearrangement of 1 into 2: A solution of compound **1** (*ca.* 30 mg) in deuterochloroform (*ca.* 0.6 mL) into an NMR tube was subjected to sunlight. After three days conversion was complete, as shown by ^{1}H NMR monitoring: mp 312-3 °C from dichloromethane-ethanol. ^{1}H NMR ($CDCl_3$, 400 MHz), δ 3.52 (1 H, m, H-5), 4.17 (1 H, dd, J =15 and 10 Hz, H-6'), 4.50 (1 H, d, J = 9 Hz, H-4), 4.75 (1 H, dd, J = 15 and 5 Hz, H-6), 6.52 (1 H, d, J = 8 Hz, H-13), 6.58 (1 H, d, J = 10 Hz, H-8), 6.88 (1 H, d, J = 10 Hz, H-9), 7.05 (1 H, m, H-12), 7.25 (2 H, m, H-10 and H-11), 7.38 (1 H, d, J = 8.5 Hz, H-14), 7.43 (1 H, m, H-15), 7.61 (1 H, m, H-16), 7.88 (1 H, d, J = 10 Hz, H-2), 7.98 (1 H, d, J = 10 Hz, H-17), 8.20 (1 H, d, J = 10 Hz, H-1). IR (KBr, cm^{-1}) 2924, 2852, 1385, 1319, 1304, 1196, 1151, 1111, 865, 836, 823. *Elem. Anal.* (Calcd for $C_{23}H_{16}S_2O_4$ x 1/10 CH_2Cl_2 as verified by ^{1}H NMR): C, 64.5 (64.8); H, 3.7 (3.8).

Crystal Data for Cycloadduct **2:** Single crystals were grown by slow evaporation from dichloromethane/ethanol solution: $C_{23}H_{16}O_4S_2$, M = 420.5, monoclinic, a = 16.773(2), b = 10.412(2), c = 10.604(2) Å, β = 96.8(2)°, V = 1838.9(9) Å^3, Z = 4, D_c = 1.52 g cm^{-3}, F(000) = 876. Space group $P2_1/n$, Mo-K$_\alpha$ radiation, μ = 2.66 cm^{-1}, R = 0.045, R_w = 0.045 for 4439 unique reflections. The structure was solved by direct methods using the program SHELX86.[14] SHELX76[15] was used for refinement calculations. Further data are provided in Tables I-III.

Table I. Positional and thermal ($Å^2x10^3$) parameters for cycloadduct 2. Standard deviations are given in parentheses. Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor

Atom	*x*	*y*	*z*	*Ueq*	Atom	*x*	*y*	*z*	*Ueq*
S(1)	0.05940(7)	0.2037(1)	0.9585(1)	0.0411(4)	C(10)	0.1103(3)	0.5905(5)	1.4887(5)	0.054(2)
S(2)	0.16655(6)	0.0443(1)	1.3209(&)	0.0347(3)	C(11)	0,1026(3)	0.4677(5)	1.5338(4)	0.053(2)
O(1)	-0.0197(2)	0.1561(3)	0.9662(3)	0.053(1)	C(12)	0.0962(3)	0.3647(4)	1.4509(4)	0.043(2)
O(2)	0.0752(2)	0.2685(3)	0.8437(3)	0.057(1)	C(13)	0.0990(2)	0.3836(4)	1.3214(4)	0.031(1)
O(3)	0.1969(2)	0.0574(3)	1,4533(3	0.048(1)	C(14)	0.0337(2)	0.1717(3)	1.2586(3)	0,028(1)
O(4)	0.1924(2)	-0.0655(3)	1.2537(3)	0.048(1)	C(15)	0.0612(2)	0.0554(4)	1.3043(3)	0.029(1)
C(1)	0.1405(3)	0.0979(4)	1.0104(4)	0.046(2)	C(16)	0.0105(3)	-0.0413(4)	1.3418(4)	0.037(1)
C(2)	0.1811(2)	0.2030(4)	1.0973(4)	0.034(1)	C(17)	-0.0692(3)	-0.0181(4)	1.3329(4)	0.038(2)
C(3)	0.1795(2)	0,1934(4)	1.2425(4)	0.030(1)	C(18)	-0.1017(2)	0.0998(4)	1.2828(4)	0.033(1)
C(4)	0.0975(2)	0.2683(3)	1.2333(4)	0.028(1)	C(19)	-0.1856(2)	0.1243(4)	1.2687(4)	0.043(2)
C(5)	0.1082(2)	0.3012(4)	1.0907(4)	0.031(1)	C(20)	-0.2167(2)	0.2321(5)	1.2147(4)	0.052(2)
C(6)	0.1135(2)	0.4372(4)	1.0577(4)	0.038(1)	C(21)	-0.1658(3)	0.3280(4)	1.1742(4)	0.047(2)
C(7)	0.1117(3)	0.5305(4)	1.1406(4)	0.042(1)	C(22)	-0.0844(2)	0.3107(4)	1.1898(4)	0.036(1)
C(8)	0.1071(2)	0.5083(4)	1.2759(4)	0.035(1)	C(23)	-0.0505(2)	0.1970(4)	1.2432(3)	0.029(1)
C(9)	0.1122(3)	0.6103(4)	1.3608(5)	0.048(2)					

Table II. Interatomic distances (Å) for cycloadduct 2. Standard deviations are given in parenthesis

S(1)-O(1)	1.427(3)	S(1)-O(2)	1.444(3)	S(1)-C(1)	1.785(5)	S(1)-C(5)	1.842(4)
S(2)-O(3)	1.442(3)	S(2)-O(4)	1.441(3)	S(2)-C(3)	1.786(4)	S(2)-C(15)	1.758(4)
C(1)-C(2)	1.537(6)	C(2)-C(3)	1.546(6)	C(2)-C(5)	1.588(5)	C(3)-C(4)	1.575(5)
C(4)-C(5)	1.581(6)	C(4)-C(13)	1.520(5)	C(4)-C(14)	1.516(5)	C(5)-C(6)	1.463(6)
C(6)-C(7)	1.313(6)	C(7)-C(8)	1.464(6)	C(8)-C(9)	1.389(6)	C(8)-C(13)	1.397(6)
C(9)-C(10)	1.376(7)	C(10)-C(11)	1.376(7)	C(11)-C(12)	1.383(6)	C(12)-C(13)	1.393(6)
C(14)-C(15)	1.365(5)	C(14)-C(23)	1.427(5)	C(15)-C(16)	1.405(6)	C(16)-C(17)	1.351(6)
C(17)-C(18)	1.420(6)	C(18)-C(19)	1.422(6)	C(18)-C(23)	1.422(6)	C(19)-C(20)	1.338(7)
C(20)-C(21)	1.413(7)	C(21)-C(22)	1.366(6)	C(22)-C(23)	1.403(6)		

Table III. Bond angles (deg) for cycloadduct 2. Standard deviations are given in parenthesis

C(1)-S(1)-C(5)	81.5(2)	O(2)-S(1)-C(5)	105.9(2)	O(2)-S(1)-C(1)	109.8(3)
O(1)-S(1)-C(5)	118.7(2)	O(1)-S(1)-C(1)	116.6(2)	O(1)-S(1)-O(2)	118.3(3)
C(3)-S(2)-C(15)	94.2(2)	O(4)-S(2)-C(15)	111.1(3)	O(4)-S(2)-C(3)	113.5(2)
O(3)-S(2)-C(15)	109.0(3)	O(3)-S(2)-C(3)	108.8(2)	O(3)-S(2)-O(4)	117.6(2)
S(1)-C(1)-C(2)	90.7(3)	C(1)-C(2)-C(5)	98.5(4)	C(1)-C(2)-C(3)	119.5(4)
C(3)-C(2)-C(5)	89.0(3)	S(2)-C(3)-C(2)	122.5(3)	C(2)-C(3)-C(4)	91.4(3)
S(2)-C(3)-C(4)	107.8(3)	C(3)-C(4)-C(14)	106.9(3)	C(3)-C(4)-C(13)	113.7(4)
C(3)-C(4)-C(5)	88.2(3)	C(13)-C(4)-C(14)	112.1(4)	C(5)-C(4)-C(14)	118.4(3)
C(5)-C(4)-C(13)	114.9(3)	C(2)-C(5)-C(4)	89.6(3)	S(1)-C(5)-C(4)	121.1(3)
S(1)-C(5)-C(2)	87.1(2)	C(4)-C(5)-C(6)	117.1(3)	C(2)-C(5)-C(6)	124.4(5)
S(1)-C(5)-C(6)	112.7(3)	C(5)-C(6)-C(7)	123.3(4)	C(6)-C(7)-C(8)	123.2(4)
C(7)-C(8)-C(13)	120.3(4)	C(7)-C(8)-C(9)	120.5(4)	C(9)-C(8)-C(13)	119.2(4)
C(8)-C(9)-C(10)	121.2(4)	C(9)-C(10)-C(11)	119.7(5)	C(10)-C(11)-C(12)	120.2(4)
C(11)-C(12)-C(13)	120.5(4)	C(8)-C(13)-C(12)	119.2(4)	C(4)-C(13)-C(12)	119.6(4)
C(4)-C(13)-C(8)	121.1(4)	C(4)-C(14)-C(23)	124.5(3)	C(4)-C(14)-C(15)	115.8(5)
C(15)-C(14)-C(23)	119.6(4)	S(2)-C(15)-C(14)	112.8(4)	C(14)-C(15)-C(16)	123.1(5)
S(2)-C(15)-C(16)	124.0(4)	C(15)-C(16)-C(17)	118.6(4)	C(16)-C(17)-C(18)	120.9(5)
C(17)-C(18)-C(23)	120.5(5)	C(17)-C(18)-C(19)	121.7(4)	C(19)-C(18)-C(23)	117.8(4)
C(18)-C(19)-C(20)	121.7(5)	C(19)-C(20)-C(21)	120.3(6)	C(20)-C(21)-C(22)	120.1(4)
C(21)-C(22)-C(23)	120.7(5)	C(18)-C(23)-C(22)	119.3(5)	C(14)-C(23)-C(22)	123.4(5)
C(14)-C(23)-C(18)	117.2(4)				

2,3,4-Trihydrodibenzo[*f*,*h*][1,5]dithionin (9): Was obtained with the same procedure described for **5** starting from 2,2'-dibenzodithiol[16] and 1,3-dibromopropane in 94% yield: mp 65-6 °C (CH_2Cl_2-*n*-hexane). ^1H NMR ($CDCl_3$, 300 MHz), δ 1.96-2.08 (2 H, m), 2.62-2.82 (4 H, m), 7.16-7.66 (8 H, m, Ar). IR (KBr, cm^{-1}) 3052, 2915, 1442, 1430, 1420, 790, 770, 765. *Elem. Anal.* (Calcd for $C_{15}H_{14}S_2$): C, 69.5 (69.7); H, 5.6 (5.5).

2,3,4-Trihydrodibenzo[*f*,*h*][1,5]dithionin-*S*-oxide (10): Was obtained as described for **6** as a single diastereoisomer in 96% yield. The sulfoxide did not crystallize easily and was used as such in the following transformation. ^1H NMR ($CDCl_3$, 300 MHz), δ 1.66-1.82 (1 H, m), 2.15-2.32 (2 H, m), 2.82-2.93 (2 H, m), 3.16 (1 H, ddd, *J* = 22, 8, 2.5 Hz), 7.12-7.68 (6 H, series of m, Ar), 8.02-8.11 (2 H, m, Ar). IR ($CHBr_3$, cm^{-1}) 3044, 2919, 1451, 1430, 1398, 1080, 1061, 1029, 845, 759, 701.

4*H*-Dibenzo[*f,h*][1,5]dithionin (11): The Pummerer reaction was performed as for **7** in 24% yield: mp 85-6 °C (CH_2Cl_2-*n*-hexane). ^1H NMR ($CDCl_3$, 300 MHz), δ 2.69 (1 H, ddd, J = 12, 3.5, 1 Hz), 3.22 (1 H, t, J = 12 Hz), 5.30-5.42 (1 H, m), 5.54 (1 H, d, J = 9 Hz), 7.10-7.50 (7 H, m, Ar), 7.65-7.72 (1 H, m, Ar). IR (KBr, cm^{-1}) 3051, 2920, 1440, 1422, 1350, 754, 742, 714. *Elem. Anal.* (Calcd for $C_{15}H_{12}S_2$): C, 69.9 (70.3); H, 4.8 (4.7).

4*H*-Dibenzo[*f,h*][1,5]dithionin-*S,S'*-tetroxide (8): Was obtained as described for **1** in 95% yield: mp 294-5 °C (CH_2Cl_2-*n*-hexane). ^1H NMR ($CDCl_3$, 300 MHz), δ 3.40-3.90 (2 H, broad m), 5.85-6.03 (1 H, broad m), 6.37 (1 H, d, J = 12 Hz), 7.48-7.88, 8.06-8.14, 8.26-8.36 (8 H, series of m, Ar). IR (KBr, cm^{-1}) 3100, 3020, 2950, 1455, 1320, 1180, 1165, 790, 770, 720. UV (CH_2Cl_2, nm) λ_{max} (ε) 272.0 (2671). *Elem. Anal.* (Calcd for $C_{15}H_{12}S_2O_4$ x 1/10 CH_2Cl_2 as verified by ^1H NMR): C, 55.3 (55.1); H, 3.5 (3.7).

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