

$\theta/2\theta$  scans  $h = -16 \rightarrow 15$   
 Absorption correction: none  $k = 0 \rightarrow 14$   
 4131 measured reflections  $l = 0 \rightarrow 17$   
 3967 independent reflections 3 standard reflections  
 2313 reflections with frequency: 120 min  
 $I > 2\sigma(I)$  intensity decay: 6.1%

#### Refinement

Refinement on  $F^2$   $\Delta\rho_{\max} = 0.151 \text{ e } \text{\AA}^{-3}$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   $\Delta\rho_{\min} = -0.153 \text{ e } \text{\AA}^{-3}$   
 $wR(F^2) = 0.098$  Extinction correction:  
 $S = 0.992$  *SHELXL97*  
 3967 reflections Extinction coefficient:  
 302 parameters 0.0215 (12)  
 H atoms constrained Scattering factors from  
 $w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$  *International Tables for*  
 where  $P = (F_o^2 + 2F_c^2)/3$  *Crystallography* (Vol. C)  
 $(\Delta/\sigma)_{\max} < 0.001$

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C1	1.370 (2)	N21—C26	1.335 (3)
O3—C3	1.364 (2)	N31—C32	1.347 (2)
O5—C5	1.366 (2)	N31—C36	1.336 (2)
N11—C12	1.343 (2)	N41—C42	1.338 (2)
N11—C16	1.336 (2)	N41—C46	1.333 (3)
N21—C22	1.338 (2)		
C2—C1—C6	121.16 (16)	C1—C6—C5	118.69 (17)
C1—C2—C3	119.06 (15)	C12—N11—C16	117.65 (16)
C2—C3—C4	120.84 (16)	C22—N21—C26	117.06 (17)
C3—C4—C5	118.99 (16)	C32—N31—C36	117.53 (16)
C4—C5—C6	121.23 (16)	C42—N41—C46	117.16 (17)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N11	0.82	1.98	2.776 (2)	164
O3—H3...N31	0.82	2.02	2.834 (2)	175
O5—H5...N21 <sup>i</sup>	0.82	1.99	2.785 (2)	162
C44—H44...N41 <sup>ii</sup>	0.93	2.59	3.492 (3)	163

Symmetry codes: (i)  $x, 1 + y, z$ ; (ii)  $-x, \frac{1}{2} + y, -\frac{1}{2} - z$ .

Compound (I) crystallized in the monoclinic system; space group  $P2_1/c$  from the systematic absences. H atoms were treated as riding atoms (C—H 0.93 and O—H 0.82  $\text{\AA}$ ).

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* and *CELDIM* in *CAD-4-PC Software*. Data reduction: *DATRD2* in *NRCVAX96* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *NRCVAX96* and *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *NRCVAX96*, *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 1998). Software used to prepare material for publication: *NRCVAX96*, *SHELXL97* and *WordPerfect* macro *PRPCIF97* (Ferguson, 1997).

We are indebted to Dr J. F. Gallagher and Dublin City University for funds to purchase the X-ray tube used in the data collection. While no direct support for this work was provided by NSERC Canada, we do acknowledge that organization for partially funding the 1992 upgrade of the CAD-4 diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1206). Services for accessing these data are described at the back of the journal.

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#### Pairwise-Interlocked Chains in 4,4'-Sulfonyldiphenol–2,2'-Bipyridyl (1/1)

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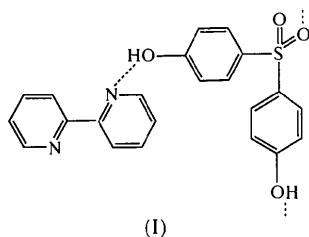
#### Abstract

In the hydrogen-bonded structure of the title complex,  $\text{O}_2\text{S}(\text{C}_6\text{H}_4\text{OH})_2 \cdot \text{C}_{10}\text{H}_8\text{N}_2$  or  $\text{C}_{12}\text{H}_{10}\text{O}_4\text{S} \cdot \text{C}_{10}\text{H}_8\text{N}_2$ , the bis-phenol molecules are linked into  $C(8)$  chains by means of  $\text{O—H} \cdots \text{O}=\text{S}$  hydrogen bonds, and the 2,2'-bipyridyl units, which act as single acceptors only, are pendent from these chains by means of  $\text{O—H} \cdots \text{N}$  hydrogen bonds. Pairs of these chains, related by centres of inversion, are mutually interlocked *via* their pendent arms.

### Comment

The structure of 4,4'-sulfonyldiphenol, O<sub>2</sub>S(C<sub>6</sub>H<sub>4</sub>OH)<sub>2</sub> (Glidewell & Ferguson, 1996), consists of {4,2,1} interwoven nets (Ferguson *et al.*, 1998) built from square R<sub>4</sub><sup>s</sup>(32) rings (Bernstein *et al.*, 1995) in which the molecules are held together by means of O—H···O=S hydrogen bonds. However, in the majority of compounds formed by this bis-phenol with heteroaromatic and other tertiary amines, this O—H···O=S motif is lost, and the predominant mode of intermolecular aggregation is by means of O—H···N hydrogen bonds (see, for example, Coupar *et al.*, 1997).

In the solid state, crystals of 2,2'-bipyridyl have the molecules lying about inversion centres, with the N atoms on opposite sides of the planar molecule (Merritt & Schroeder, 1956; Chisholm *et al.*, 1981), so that this bipyridyl is also potentially a double acceptor of hydrogen bonds. We have now investigated the behaviour of 4,4'-sulfonyldiphenol towards 2,2'-bipyridyl and have found that the resulting 1:1 adduct, O<sub>2</sub>S(C<sub>6</sub>H<sub>4</sub>OH)<sub>2</sub>.C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>, (I), retains the chain-forming O—H···O=S structural motif of pure O<sub>2</sub>S(C<sub>6</sub>H<sub>4</sub>OH)<sub>2</sub>, with the 2,2'-bipyridyl units all pendent from these chains by means of O—H···N hydrogen bonds.



The asymmetric unit of (I) consists of one molecule of each component (Fig. 1). The bis-phenol molecules are linked into a single type of C(8) chain by means of O—H···O=S hydrogen bonds, with four such chains running through the unit cell parallel to the [100] direction. The hydroxy O24 atom at (x, y, z) acts as donor to the sulfone O12 atom at (-1 + x, y, z) (Table 2); the other hydroxy O atom, O14, acts as donor to N31 within the asymmetric unit. Since the C(8) chain is generated by simple translation, all of the pendent 2,2'-bipyridyl units within a given chain lie on the same side of that chain. Although the 2,2'-bipyridyl unit adopts a *transoid* conformation close to planarity, with a dihedral angle between the two ring planes of only 8.4 (1)°, the N41 atom does not act as an acceptor of any hydrogen bonds, hard or soft (Braga *et al.*, 1995).

However, pairs of such chains related by centres of inversion closely imitate the architecture of a zip fastener, with the pendent arms of the two chains mutually interlocked (Fig. 2), and there is evidence of weakly attractive  $\pi$ - $\pi$  interactions (Hunter, 1994) between the rings of neighbouring bipyridyl units. The N31-containing ring at (x, y, z) makes an angle of *ca*

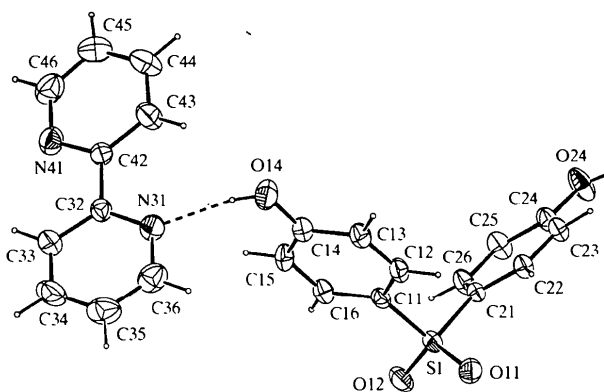


Fig. 1. The asymmetric unit of compound (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

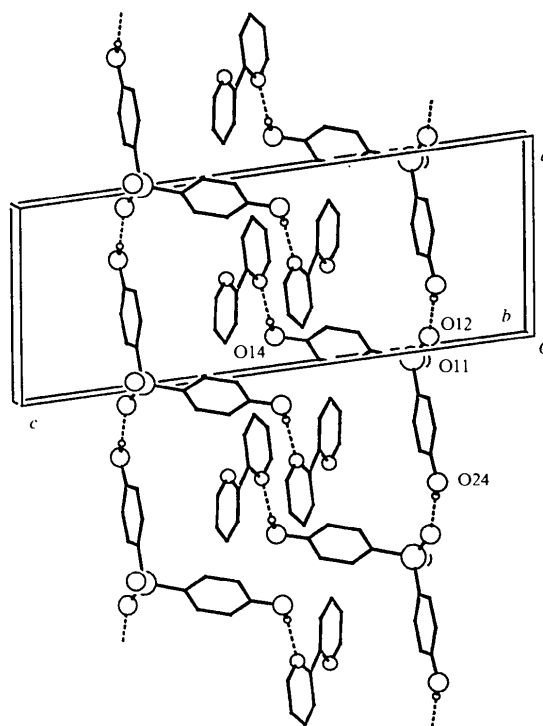


Fig. 2. A view of part of the crystal structure of (I), showing two interlocked chains. H atoms bonded to carbon have been omitted for clarity.

8.4° with the N41-containing ring at (1 - x, -y, 1 - z), and the corresponding ring centroids are *ca* 3.73 Å apart. Two such pairs of linked chains run through each unit cell.

There are fairly short C—H···O hydrogen bonds linking the paired chains (Table 2); atom C12 of the sulfone at (x, y, z) acts as donor to the hydroxy O24 atom at (-1 - x,  $\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ), and repetition of this motif gives a C(9) spiral parallel to [010] around the 2<sub>1</sub> axis at ( $-\frac{1}{2}$ , y,  $\frac{1}{4}$ ), so generating sheets built from

$R_4^1(24)$  rings. Although this structure contains an excess of hard hydrogen-bond acceptors, the N41 and O11 atoms remain unused, participating in no C—H...N or C—H...O interactions.

Examination of the structure with *PLATON* (Spek, 1998) showed that there were no solvent-accessible voids in the crystal lattice.

## Experimental

Equimolar quantities of 4,4'-sulfonyldiphenol and 2,2'-bipyridyl were separately dissolved in methanol. The solutions were mixed and the resulting mixture was set aside to crystallize, producing compound (I). Analysis: found C 64.2, H 4.5, N 6.8%;  $C_{22}H_{18}N_2O_4S$  requires C 65.0, H 4.5, N 6.9%. Crystals suitable for single-crystal X-ray diffraction were selected directly from the analytical sample.

### Crystal data

$C_{12}H_{10}O_4S \cdot C_{10}H_8N_2$   
 $M_r = 406.44$   
 Monoclinic  
 $P2_1/c$   
 $a = 8.4680$  (7) Å  
 $b = 11.0388$  (9) Å  
 $c = 21.7430$  (17) Å  
 $\beta = 97.897$  (6)°  
 $V = 2013.2$  (3) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.341$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107$  Å  
 Cell parameters from 25 reflections  
 $\theta = 10.42$ – $16.78$ °  
 $\mu = 0.192$  mm<sup>-1</sup>  
 $T = 294$  (1) K  
 Plate  
 $0.40 \times 0.40 \times 0.21$  mm  
 Colourless

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction: Gaussian (*ABSO* in *NRCVAX*; Gabe *et al.*, 1989)  
 $T_{\min} = 0.929$ ,  $T_{\max} = 0.963$   
 3870 measured reflections  
 3623 independent reflections

2657 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.008$   
 $\theta_{\text{max}} = 25.13$ °  
 $h = -10 \rightarrow 10$   
 $k = 0 \rightarrow 13$   
 $l = 0 \rightarrow 26$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 1.8%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.096$   
 $S = 1.026$   
 3623 reflections  
 265 parameters  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.3671P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.200$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.236$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0093 (9)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

S1—O11	1.4353 (14)	O24—C24	1.358 (2)
S1—O12	1.4412 (14)	N31—C32	1.339 (2)
S1—C11	1.7567 (19)	N31—C36	1.337 (3)

S1—C21	1.7548 (18)	N41—C42	1.336 (3)
O14—C14	1.353 (2)	N41—C46	1.336 (3)
O11—S1—O12	118.50 (9)	O12—S1—C21	107.50 (9)
O11—S1—C11	107.61 (9)	C11—S1—C21	106.29 (8)
O11—S1—C21	107.86 (8)	C32—N31—C36	118.0 (2)
O12—S1—C11	108.47 (9)	C42—N41—C46	116.9 (2)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O14—H14...N31	0.82	1.93	2.720 (2)	163
O24—H24...O12'	0.82	1.91	2.721 (2)	167
C12—H12...O24''	0.93	2.46	3.361 (2)	164

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $-1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ .

Compound (I) crystallized in the monoclinic system; space group  $P2_1/c$  from the systematic absences. H atoms were treated as riding atoms (C—H 0.93 and O—H 0.82 Å).

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* and *CELDIM* in *CAD-4-PC Software*. Data reduction: *DATRD2* in *NRCVAX96* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *NRCVAX96* and *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *NRCVAX96*, *ORTEP* (Johnson, 1976) and *PLATON* (Spek, 1998). Software used to prepare material for publication: *NRCVAX96*, *SHELXL97* and *WordPerfect* macro *PRPCIF97* (Ferguson, 1997).

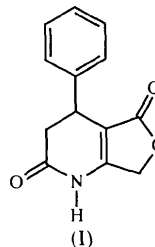
We are indebted to Dr J. F. Gallagher and Dublin City University for funds to purchase the X-ray tube used in the data collection. While no direct support for this work was provided by NSERC Canada, we do acknowledge that organization for partially funding the 1992 upgrade of the CAD-4 diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1207). Services for accessing these data are described at the back of the journal.

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## 5-Oxo-4-phenyl-1,2,3,4,5,7-hexahydrofuro- [3,4-*b*]-2(1*H*)-pyridone†

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### Abstract

The six-membered ring in the title compound, C<sub>13</sub>H<sub>11</sub>NO<sub>3</sub>, adopts a twist conformation and the lactone ring a planar conformation. The aryl group occupies a pseudo-axial position and is orthogonal to the plane through the pyridone ring. The molecules are linked by an intermolecular hydrogen bond between the N atom and the carbonyl-O atom of a neighbouring molecule [ $N1 \cdots O5(x, y-1, z) = 2.820(5) \text{ \AA}$ ].

### Comment

Research on the 1,4-dihydropyridine (1,4-DHP) system is of current interest because of its properties as a calcium channel antagonist (Bossert & Vater, 1989). Substitution on the 1,4-DHP ring has been studied (Kuthan & Kurfurst, 1982) because of the effect of the substituent on the biological properties. It has been found that cyclohexanone and  $\gamma$ -lactone rings fused to the 1,4-DHP moiety result in a striking effect on the entry of calcium ions into the intracellular space (calcium-agonist effect) (Meyer *et al.*, 1976).

The six-membered ring of the title compound, (I), is best described as having a twist conformation; its puckering parameters (Nardelli, 1983a) are  $Q = 0.422(4) \text{ \AA}$ ,  $\theta = 120.6(5)^\circ$  and  $\varphi = -39.9(7)^\circ$  with  $\Delta C_2(N1-C7a) = 0.039(2)$  and  $\Delta C_2(C2) = 0.098(1)$ .

† Alternative name: 4-phenyl-1,2,3,4,5,7-hexahydrofuro[3,4-*b*]pyridine-2,5-dione.

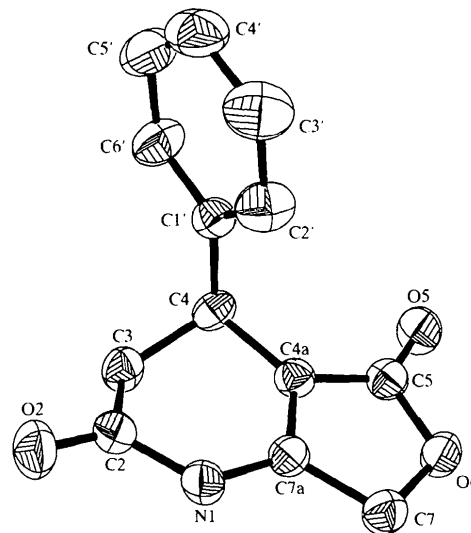


Fig. 1. A perspective view of the molecule with the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

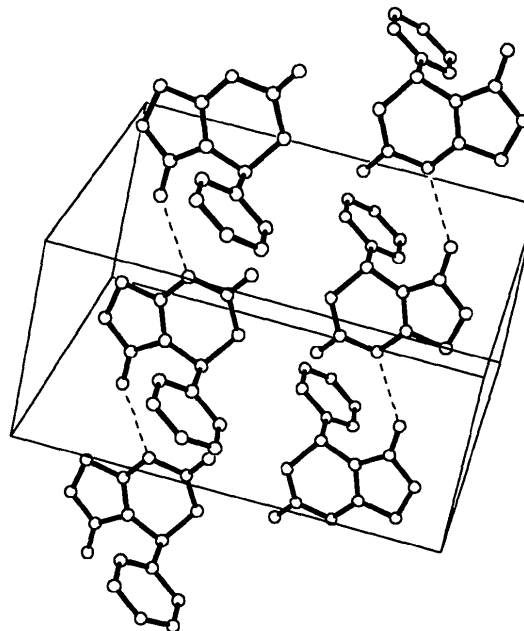


Fig. 2. Crystal structure viewed along the *b* axis. Hydrogen bonds are represented as dashed lines.