

the planarity of the entire system [excluding C(5) and C(6)]. Atomic parameters are given in Table 2.\* Principal computer programs are cited in Gadol & Davis (1982); the least-squares-planes program was written by Cordes (1983).

**Related literature.** The structure was previously reported (Simonsen, 1972), but there was some difficulty with disorder in the cyclohexeno ring. In the present structure determination, the disorder shows up in the final electron density difference map in which peaks of 0.35 and 0.31 e Å<sup>-3</sup> form a reasonable C(4)—C(5)—C(6)—C(7) chain with C—C distances of 1.66, 1.63 and 1.45 Å through the sequence. This fraction, amounting to approximately 5%, was ignored. The angle between the thiophene ring and the carboxyl group in the title compound differs greatly from the 79° found in 2-(*N,N*-diethylamino)-3-carbomethoxy-naphtho[2,3-*b*]thiophene-4,9-dione (DCMT) (Gibbons, Lerbscher & Trotter, 1972). DCMT cannot hydrogen

bond intramolecularly and, in fact, severe steric interactions prevent the carbomethoxy group from being coplanar with the thiophene ring.

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\* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51190 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of a 2,4-Dithiinopyran Derivative

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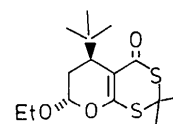
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**Abstract.** (5*SR*,7*RS*)-7-Ethoxy-5,6-dihydro-2,2-dimethyl-5-*tert*-butyl-8*H*-[1,3]dithiinol[4,5-*b*]pyran-4-one, C<sub>15</sub>H<sub>24</sub>O<sub>3</sub>S<sub>2</sub>, *M<sub>r</sub>* = 316.47, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 10.233 (2), *b* = 12.647 (2), *c* = 12.898 (3) Å, β = 95.12 (3)°, *V* = 1662.42 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.264 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.31 mm<sup>-1</sup>, *F*(000) = 680, *T* = 298 K, *R* = 0.038 for 2580 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR.

**Experimental.** Title compound (I): crystal size 0.2 × 0.3 × 0.4 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo *K*α radiation, profile-



(I)

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

	x	y	z	$U_{eq}$
S(4)	-5745 (1)	-1326 (1)	-4112 (1)	48 (1)
S(2)	-8596 (1)	-1729 (1)	-4063 (1)	56 (1)
O(6)	-3762 (1)	240 (1)	-1230 (1)	45 (1)
O(5)	-4925 (1)	62 (1)	-2829 (1)	51 (1)
C(8A)	-7236 (2)	-169 (2)	-2797 (1)	36 (1)
O(1)	-9308 (2)	-814 (1)	-2455 (1)	69 (1)
C(8')	-7728 (2)	1793 (2)	-2448 (2)	46 (1)
C(3)	-7310 (2)	-1376 (2)	-4885 (2)	44 (1)
C(1)	-8405 (2)	-823 (2)	-3007 (2)	45 (1)
C(4A)	-6073 (2)	-404 (2)	-3160 (1)	38 (1)
C(7)	-6001 (2)	657 (2)	-1289 (1)	47 (1)
C(8)	-7294 (2)	697 (2)	-1991 (1)	40 (1)
C(6)	-4786 (2)	669 (2)	-1861 (1)	41 (1)
C(3'')	-7615 (2)	-338 (2)	-5450 (2)	55 (1)
C(6'')	-1475 (2)	70 (2)	-761 (2)	60 (1)
C(8'C)	-9077 (2)	1683 (2)	-3061 (2)	63 (1)
C(8'B)	-7877 (2)	2565 (2)	-1553 (2)	65 (1)
C(3')	-7196 (2)	-2288 (2)	-5654 (2)	66 (1)
C(6')	-2501 (2)	352 (2)	-1618 (2)	50 (1)
C(8'A)	-6771 (2)	2256 (2)	-3178 (2)	59 (1)

Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

fitting mode involving variable scan width and speed (Clegg, 1981). 8763 reflections measured,  $2\theta_{max}$   $50^\circ$ ,  $\pm h \pm k + l$ , three check reflections with no significant intensity change. 2919 unique reflections ( $R_{int} = 0.018$ ), of which 2580 with  $F > 4\sigma(F)$  were used for all calculations (SHELXS86, Sheldrick, 1985; SHELX76, Sheldrick, 1976). Cell constants refined from  $\pm 2\theta$  values of 50 reflections in the range  $20$ – $25^\circ$ . Absorption correction was not necessary. Extinction correction was applied yielding a value of 0.0007 for the secondary-extinction coefficient  $x$ , where  $F_c^* = F_c \times (1 + 0.002x F_c^2 / \sin^2\theta)^{-0.25}$ . Structure solution by direct methods. Refined on  $F$  to  $R = 0.038$ ,  $wR = 0.055$ ; all non-H atoms anisotropic, H atoms were included using a riding model [ $C-H$   $0.96 \text{ \AA}$ ,  $U(H) = 0.08 \text{ \AA}^2$ ]. We refined 182 parameters,  $S = 1.95$ , weighting scheme  $w^{-1} = \sigma^2(F) + 0.0006F^2$  which led to a featureless analysis of variance in terms of  $\sin\theta$  and  $F_o$ , max.  $\Delta/\sigma = 0.002$ , max. and min. height in final  $\Delta\rho$  map  $0.23$  and  $-0.17 \text{ e \AA}^{-3}$  respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.† Fig. 1 shows a thermal ellipsoid plot with the atom numbering.

**Related literature.** For the preparation of the compound see Schäfer (1987). For the preparation of some related compounds see Holla (1985).

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51141 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

S(4)–C(3)	1.810 (2)	S(4)–C(4A)	1.747 (2)
S(2)–C(3)	1.818 (2)	S(2)–C(1)	1.777 (2)
O(6)–C(6)	1.380 (2)	O(6)–C(6')	1.432 (2)
O(5)–C(4A)	1.350 (2)	O(5)–C(6)	1.462 (2)
C(8A)–C(1)	1.459 (3)	C(8A)–C(4A)	1.351 (3)
C(8A)–C(8)	1.514 (3)	O(1)–C(1)	1.216 (2)
C(8')–C(8)	1.555 (3)	C(8')–C(8'C)	1.535 (3)
C(8')–C(8'B)	1.530 (3)	C(8')–C(8'A)	1.534 (3)
C(3)–C(3'')	1.520 (3)	C(3)–C(3')	1.533 (3)
C(7)–C(8)	1.536 (2)	C(7)–C(6)	1.501 (3)
C(6'')–C(6')	1.498 (3)		
C(4A)–S(4)–C(3)	101.2 (1)	C(1)–S(2)–C(3)	104.7 (1)
C(6')–O(6)–C(6)	114.6 (1)	C(6)–O(5)–C(4A)	121.0 (1)
C(4A)–C(8A)–C(1)	122.9 (2)	C(8)–C(8A)–C(1)	117.1 (2)
C(8)–C(8A)–C(4A)	119.1 (2)	C(8'C)–C(8')–C(8)	109.1 (2)
C(8'B)–C(8')–C(8)	109.1 (2)	C(8'B)–C(8')–C(8'C)	107.5 (2)
C(8'A)–C(8')–C(8)	113.4 (2)	C(8'A)–C(8')–C(8'C)	108.2 (2)
C(8'A)–C(8')–C(8'B)	109.4 (2)	S(2)–C(3)–S(4)	109.8 (1)
C(3'')–C(3)–S(4)	111.6 (1)	C(3'')–C(3)–S(2)	111.3 (1)
C(3')–C(3)–S(4)	105.5 (2)	C(3')–C(3)–S(2)	107.1 (1)
C(3')–C(3)–C(3'')	111.3 (2)	C(8A)–C(1)–S(2)	122.7 (1)
O(1)–C(1)–S(2)	114.6 (2)	O(1)–C(1)–C(8A)	122.6 (2)
O(5)–C(4A)–S(4)	107.3 (1)	C(8A)–C(4A)–S(4)	128.4 (1)
C(8A)–C(4A)–O(5)	124.3 (2)	C(6)–C(7)–C(8)	114.7 (2)
C(8')–C(8)–C(8A)	114.3 (2)	C(7)–C(8)–C(8A)	107.2 (2)
C(7)–C(8)–C(8')	117.0 (2)	O(5)–C(6)–O(6)	107.9 (2)
C(7)–C(6)–O(6)	109.0 (2)	C(7)–C(6)–O(5)	113.1 (2)
C(6'')–C(6')–O(6)	108.2 (2)		

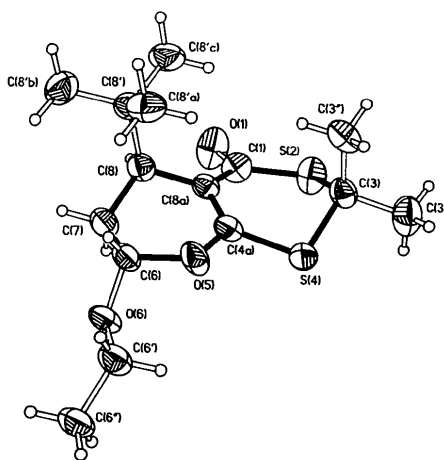


Fig. 1. View of (I) showing the atomic numbering.

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*Acta Cryst.* (1988). **C44**, 2039

**On the structure of 2-hydroxybiphenyl.** By MOSHE KAPON and GEORGE M. REISNER, *Department of Chemistry, Technion-Israel Institute of Technology, Haifa 32000, Israel*

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### Abstract

The crystal structure of 2-hydroxybiphenyl recently reported in space group *Cc* [Perrin, Bekkouch & Thozet (1987). *Acta Cryst.* **C43**, 980–982] has been refined in *Fdd2*. The increase in Laue symmetry – from *2/m* to *mmm* – has caused no significant changes in the structural parameters.

The structure of 2-hydroxybiphenyl,  $C_{12}H_{10}O$ , has been reported (Perrin, Bekkouch & Thozet, 1987) in space group *Cc* [ $a = 5.882$  (2),  $b = 12.867$  (2),  $c = 12.313$  (3) Å,  $\beta = 103.76$  (3)°,  $Z = 4$ ] and refined to an *R* of 0.064 for 751 observed reflections. The vectors [0,1,0], [1,0,2] and [1,0,0] define an orthorhombic cell ( $a' = 12.867$ ,  $b' = 23.919$ ,  $c' = 5.882$  Å,  $Z = 8$ ) and the corresponding coordinate transformation  $x' = y - 0.1250$ ,  $y' = 0.5z - 0.4692$ ,  $z' = x - 0.5z$  led to a structure in space group *Fdd2* that we have refined based on the original data recovered from SUP 43666. Refinement on *F* by the *SHELX77* system of programs (Sheldrick, 1977) converged routinely to *R* = 0.056 for 441 averaged reflections and 79 parameters. Final *Fdd2* coordinates are given in Table 1.\*

The change in space group requires the molecule to have an exact twofold axis which bisects the C4–C4' bond joining the phenyl rings. The oxygen atom was again found to be disordered with occupancies 0.4 and 0.1. Since the space-group change involves only an increase in Laue symmetry, there are no significant alterations in the bond lengths and

\* Anisotropic temperature factors of the non-hydrogen atoms and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51146 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates ( $\times 10^4$ ) and  $U_{eq}$  ( $\text{Å}^2 \times 10^3$ ) for the carbon and O1 atoms, atomic coordinates ( $\times 10^3$ ) and  $U_{iso}$  ( $\text{Å}^2 \times 10^3$ ) for O2 and the hydrogen atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j |U_{ij}(a_i^* a_j^*)| (a_i \cdot a_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}/U_{iso}$
C1	2223 (4)	–877 (2)	4100	78 (1)
C2	2046 (4)	–509 (2)	5883 (14)	75 (1)
C3	1172 (3)	–172 (2)	5864 (15)	65 (1)
C4	466 (3)	–182 (2)	4068 (11)	54 (1)
C5	657 (3)	–558 (2)	2300 (14)	63 (1)
C6	1530 (4)	–893 (2)	2335 (16)	76 (1)
O1*	–56 (5)	–622 (3)	664 (17)	72 (2)
O2†	101 (2)	14 (1)	741 (6)	100 (7)
H1	286 (3)	–113 (1)	404 (8)	57 (10)
H2	256 (4)	–50 (2)	725 (12)	101 (17)
H3†	101 (2)	14 (1)	741 (6)	100 (7)
H6	154 (4)	–117 (2)	152 (12)	110 (23)

\* Refined with occupancy of 0.4.

† Refined as an average atom of 0.1 oxygen + 0.9 hydrogen.

angles (Schomaker & Marsh, 1979; Marsh & Herbstein, 1988). E.s.d.'s of bonds are usually smaller (by  $\sim 0.002$  Å) than those originally reported. E.s.d.'s of angles reported by Perrin *et al.* are twice as large as those found for the present refinement.

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