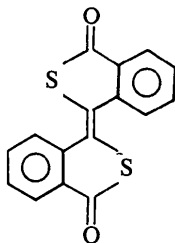




here with the previously determined dioxa structure shows that the dithia molecule maintains its planarity with an S...H intramolecular contact of 2.45 Å.

**Introduction.** The structure of the title compound,



alternatively known as *trans*-dithiabipthalide, became of interest when it was noted that its photochemical stability was quite different from that of the dioxa analog, *trans*-bipthalyl. A literature search has shown that the structure of the dithia compound had apparently been determined by R. E. Davis, but the promised details have never been published (see footnote 24 of Markgraf, Heller & Avery, 1970). So that a better comparison between the two analogs could be made, the structure of the dioxa compound, originally studied by Ramirez, Ricci, Tsuboi, Marecek & Yamanaka (1976), has been redetermined by Whitney (1982)\* using data-collection and refinement conditions similar to those used for the dithia compound. No major discrepancies between Whitney's low-temperature study and the room-temperature work of Ramirez *et al.* were found.

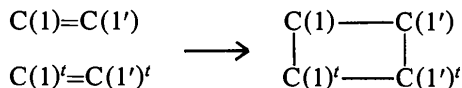
**Experimental.** Crystals of dithiabipthalide were prepared by the method of Bird & Wong (1969). The product was chromatographed on neutral silica gel (Woelm activity I) with xylene as the eluant; with slow evaporation of the solvent, orange-yellow needles (needle axis parallel to the *a* axis) were recrystallized.

Syntex P3 diffractometer with low-temperature apparatus (173 K). Crystal dimensions: 0.05 × 0.09 × 0.25 mm. Unit-cell parameters refined using 50 reflections,  $12 < 2\theta < 25^\circ$ . Data collection: 1224 reflections,  $1^\circ \omega$  scans,  $1.0\text{--}3.0^\circ \text{ min}^{-1}$ ,  $4 < 2\theta < 52^\circ$ ,  $h$  0 to 4,  $k$  -17 to 0,  $l$  -13 to 13. No absorption correction. Standard reflections: 4 every 200 reflections; only statistical variations noted. Solution: direct methods. Refinement: full-matrix least squares;  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = 1/\sigma^2(F_o)$ ,  $\sigma(F_o^2) = [\sigma^2(F_o^2) + (0.02F_o^2)]^{1/2}$ ; 776 reflections,  $F_o^2 > 2\sigma(F_o^2)$ ; 107 variables (S, O, and C anisotropic; H isotropic);  $R_w = 0.043$ ,  $S = 1.45$ .  $(\Delta/\sigma)_{\text{max}} = 0$ . Largest peak in final difference map:  $0.4 \text{ e } \text{Å}^{-3}$ . No correction for secondary extinction. Programs: local modifications of the Enraf-Nonius SDP package (Frenz, 1978). Other computa-

tional details and atomic scattering factors may be found elsewhere (Nugent & Harlow, 1979).

**Discussion.** Atomic positional parameters are given in Table 1, with selected bond distances and angles shown in Table 2.\* Fig. 1 shows a drawing of the molecule with the atomic numbering scheme and a stereo packing diagram is provided in Fig. 2.

The dioxa and dithia compounds have been used in studies related to the photochemical stability of organic pigments. The dioxa compound readily undergoes photodecomposition when exposed to strong UV or visible light; the dithia compound, however, shows virtually no photodegradation under the same conditions. FT-IR studies of the photochemical product strongly suggest that the dioxa compound dimerizes (*via* an interaction between the central C=C moieties of neighboring molecules) to form a substituted cyclobutane (Chase, Amey & Holtje, 1982):



Although the dioxa and dithia derivatives crystallize in the same space group with similar unit-cell dimensions, the crystals are not isostructural. The unique axis in the dioxa structure is the shortest axis ( $< 4 \text{ Å}$ ), while the longest axis is unique in the dithia case. Both, however, pack so as to form columns of molecules with possible  $\pi$  interactions. The small change in crystal packing, brought about by the larger spatial requirements of the S atoms, has increased the C(1)...C(1)' distance within these columns from 3.737 Å in the dioxa case (Whitney's data) to 3.844 Å in the dithia structure.

The relative conformation of the two molecules was of interest because any distortion away from the planar conformation found for the dioxa derivative could affect intermolecular interactions. In fact, the dithia analog is quite planar, although the S atoms do deviate somewhat from the plane,  $\pm 0.045 \text{ Å}$  (the second largest deviation is a mere 0.018 Å). The maintenance of a planar conformation indicates that the dithia molecule is reasonably comfortable with its short intramolecular S...H(3') contact of 2.45 Å. This contact does, however, cause the C(1)–C(2)–C(3) angle to be  $6^\circ$  larger than the corresponding C(6)–C(7)–C(8) angle and C(1)–C(2) to be considerably longer than C(7)–C(8). These would suggest that the S...H contact is more repulsive than attractive. On the other hand, an attractive interaction might be suggested by the fact

\* Tables of anisotropic thermal parameters, mean-plane calculations and structure amplitudes, along with an appendix containing crystal structure information for *trans*-bipthalyl have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38715 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* See deposition footnote.

that the exocyclic angles at C(1) and C(2) are smaller for the S derivative than for the O analog; these angles are necessarily smaller, however, because of the larger endocyclic angular requirements of the S atom.

In conclusion, the only distinguishing feature of the two crystal structures which might explain the photochemical behavior is the different C(1)...C(1)' distances. Electronic effects, *e.g.* charge-transfer processes, may also be involved and this possibility is being explored.

Table 1. *Positional parameters and their estimated standard deviations*

For non-hydrogen atoms  $B_{eq} = \frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$ , where  $a$ ,  $b$  and  $c$  are lattice constants. H atoms were refined isotropically.

	$x$	$y$	$z$	$B_{eq}$ or $B(\text{\AA}^2)$
S	-0.0889 (3)	0.15220 (8)	-0.0248 (1)	1.95 (2)
O	0.0616 (9)	0.2980 (2)	0.1151 (3)	3.41 (8)
C(1)	0.037 (1)	0.0396 (3)	0.0313 (3)	1.46 (8)
C(2)	0.225 (1)	0.0511 (3)	0.1511 (3)	1.62 (8)
C(3)	0.374 (1)	-0.0173 (3)	0.2267 (4)	2.1 (1)
C(4)	0.542 (1)	0.0084 (3)	0.3349 (4)	2.3 (1)
C(5)	0.558 (1)	0.1025 (3)	0.3714 (4)	2.3 (1)
C(6)	0.407 (1)	0.1708 (3)	0.2975 (4)	2.3 (1)
C(7)	0.245 (1)	0.1451 (3)	0.1886 (3)	1.74 (8)
C(8)	0.082 (1)	0.2135 (3)	0.1055 (4)	2.13 (9)
H(3)	0.37 (1)	-0.084 (3)	0.206 (3)	2.1 (9)
H(4)	0.63 (1)	-0.035 (3)	0.384 (3)	3 (1)
H(5)	0.70 (1)	0.121 (3)	0.446 (3)	2.2 (9)
H(6)	0.419 (9)	0.231 (3)	0.318 (3)	1.6 (9)

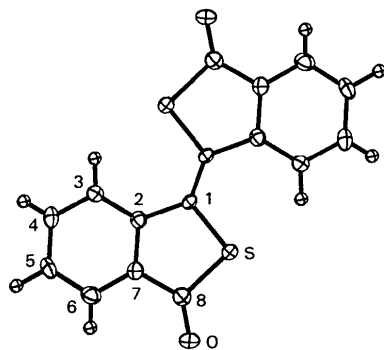


Fig. 1. Drawing of the molecule with the atomic numbering scheme. Spheres for the H atoms correspond arbitrarily to  $B = 1.0 \text{\AA}^2$ .

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### Structure of 1-(5'-O-Acetyl-2',3'-O-isopropylidene- $\beta$ -D-ribofuranosyl)-5-ditosylaminoimidazole-4-carbonitrile, $C_{28}H_{30}N_4O_9S_2$

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**Abstract.**  $M_r = 630.69$ , monoclinic,  $P2_1$ ,  $a = 11.419$  (1),  $b = 13.362$  (4),  $c = 9.677$  (2)  $\text{\AA}$ ,  $\beta = 91.51$  (3) $^\circ$ ,  $V = 1476.1$  (4)  $\text{\AA}^3$ ,  $Z = 2$ ,  $D_m = 1.401$  (4),

Table 2. *Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with estimated standard deviations*

The four C—H distances vary from 0.88 (4) to 1.00 (3)  $\text{\AA}$ .

S—C(1)	1.772 (4)	C(2)—C(7)	1.401 (5)
S—C(8)	1.793 (4)	C(3)—C(4)	1.389 (5)
O—C(8)	1.207 (4)	C(4)—C(5)	1.397 (6)
C(1)—C(1')	1.349 (7)	C(5)—C(6)	1.378 (5)
C(1)—C(2)	1.494 (4)	C(6)—C(7)	1.386 (5)
C(2)—C(3)	1.388 (5)	C(7)—C(8)	1.459 (5)
C(1)—S—C(8)	94.0 (2)	C(4)—C(5)—C(6)	119.1 (4)
S—C(1)—C(1')	121.4 (4)	C(5)—C(6)—C(7)	119.6 (4)
S—C(1)—C(2)	109.1 (2)	C(2)—C(7)—C(6)	122.1 (4)
C(1')—C(1)—C(2)	129.5 (4)	C(2)—C(7)—C(8)	115.2 (3)
C(1)—C(2)—C(3)	129.1 (4)	C(6)—C(7)—C(8)	122.7 (4)
C(1)—C(2)—C(7)	113.0 (3)	S—C(8)—O	122.3 (3)
C(3)—C(2)—C(7)	117.9 (3)	S—C(8)—C(7)	108.7 (3)
C(2)—C(3)—C(4)	120.1 (4)	O—C(8)—C(7)	129.0 (4)
C(3)—C(4)—C(5)	121.2 (4)		

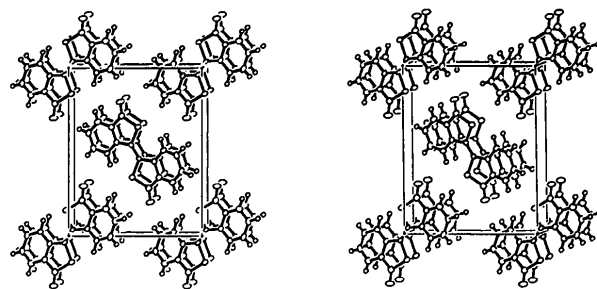


Fig. 2. Stereo packing diagram viewed nearly parallel to the  $a$  axis.

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$D_x = 1.419 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.70924 \text{ \AA}$ ,  $\mu = 0.242 \text{ mm}^{-1}$ ,  $F(000) = 660$ ,  $T = 293 \text{ K}$ . Final  $R = 0.053$  for 2631 observed reflections. The glycosyl

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