

dipyrromethenes, the molecules pack in parallel sheets, approximately 3.5–3.7 Å apart. It is believed that this arrangement stabilizes the planarity of the pyrrolic skeleton because of dispersion forces between the sheets (Sheldrick, Borkenstein, Blacha-Puller & Gossauer, 1977). There are no intermolecular contacts less than 3.5 Å between non-hydrogen atoms.

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Structure of Dibenzoyldisulphane, C₁₄H₁₀O₂S₂

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Abstract. $M_r = 274.35$, monoclinic, $P2_1/a$, $a = 9.079$ (2), $b = 12.073$ (2), $c = 12.370$ (3) Å, $\beta = 107.21$ (2)°, $V = 1295.32$ Å³, $Z = 4$, $D_x = 1.407$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu(\text{Cu } K\alpha) = 3.49$ mm⁻¹, $F(000) = 568$, $T = 298$ K, $R = 0.063$, $R_w = 0.064$ for 1259 reflections. S–S is 2.021 (2) Å and the C–S–S–C dihedral angle is 80.8 (3)°. The compound is monomeric and has a *gauche* confor-

mation with respect to the S–S bond. Short contacts (~ 3.0 Å) are present between the S and O of the two halves of the disulphane.

Introduction. The symmetric disulphane compounds of type RSSR contain the disulphide bridge which plays an important role in stabilizing macromolecular (protein) structures in three dimensions. The title compound

(DBDS) was obtained during the synthesis of the bis(thiobenzoato-S) complexes of Se^{II} (Aravamudan, Subrahmanyam, Seshasayee & Appa Rao, 1983) and Te^{II} (Subrahmanyam, Aravamudan, Rout & Seshasayee, 1983) and also during the slow aerial oxidation of thiobenzoic acid in methanolic solution. The structural study was taken up to determine the configuration of the atoms around the S—S linkage.

Experimental. White cylindrical crystals obtained from a solution of 1:1 (volume) benzene-methanol, crystal 0.30 × 0.26 × 0.40 mm, Enraf-Nonius CAD-4 computer-controlled four-circle diffractometer, graphite monochromator, unit-cell parameters determined by least-squares refinement of the setting angles of 25 high-angle reflections; *hkl* range ±11,15,15, 3 < 2θ < 78°, ω-2θ scan, scan width 1.0° + 0.15° tan θ, two control reflections (243̄ and 114̄) monitored every hour - no significant variation in intensity (< 4%), 3149 reflections collected, 1259 observed *I* > 3σ(*I*) [σ(*I*) is the standard deviation based on counting statistics], *R*_{int} 0.057, no correction for absorption; two sulphur-atom positions located from Patterson map, subsequent Fourier map revealed all the nonhydrogen atoms, refinement by full-matrix least squares, anisotropic thermal parameters for nonhydrogen atoms, all hydrogen-atom positions located from difference Fourier map and refined isotropically, *R* = 0.063, *R*_w = 0.064, *w* = 1/[σ²(*F*_o) + 0.1917 *F*_o²], Σ*w* Δ*F*² minimized, 196 parameters refined, ratio of maximum shift to error in the final cycle of the refinement 0.873; difference Fourier synthesis showed maximum and minimum heights of 0.25 and -0.35 e Å⁻³ respectively; all the calculations carried out with *SHELX76* (Sheldrick, 1976); atomic scattering factors for nonhydrogen atoms taken from Cromer & Mann (1968), anomalous dispersion correction factors from Cromer & Liberman (1970) and for hydrogen atoms from Stewart, Davidson & Simpson (1965).*

Discussion. Tables 1 and 2 contain the atomic coordinates, thermal parameters and intramolecular bonding parameters. Fig. 1 shows the molecular configuration drawn by *ORTEP* (Johnson, 1965). The S—S and S—C distances obtained in BBDS [2.021 (2); 1.811 (6) and 1.827 (6) Å] agree with those reported for similar compounds, namely tetraethylthiuram disulphide [1.995 (5); 1.813 and 1.818 Å] (Karle, Estlin & Britts, 1967) (I), bis-(morpholinothiocarboxyl)disulphane [2.009 (5); 1.787 (16) and 1.769 (15) Å] (Rout, Seshasayee &

Table 1. *The fractional atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10³)*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
S(1)	646 (2)	951 (2)	2176 (1)	60 (1)
S(2)	1839 (2)	1726 (1)	1253 (1)	64 (1)
O(1)	3526 (6)	454 (4)	3344 (4)	68 (2)
O(2)	1798 (6)	-347 (3)	565 (4)	66 (2)
C(1)	2181 (7)	275 (5)	3251 (5)	47 (2)
C(2)	1631 (7)	-479 (5)	3987 (5)	46 (2)
C(3)	2678 (9)	-827 (6)	4996 (6)	57 (2)
C(4)	2240 (10)	-1555 (6)	5701 (6)	66 (3)
C(5)	761 (10)	-1969 (6)	5405 (6)	67 (3)
C(6)	-289 (10)	-1631 (7)	4406 (6)	66 (3)
C(7)	149 (8)	-892 (6)	3706 (5)	53 (2)
C(8)	2125 (8)	578 (5)	372 (5)	47 (2)
C(9)	2840 (7)	893 (5)	-511 (4)	44 (2)
C(10)	3012 (8)	1986 (5)	-794 (6)	55 (2)
C(11)	3700 (10)	2229 (7)	-1625 (6)	73 (3)
C(12)	4282 (9)	1394 (7)	-2138 (6)	69 (3)
C(13)	4090 (9)	297 (6)	-1869 (6)	65 (3)
C(14)	3371 (8)	48 (5)	-1064 (5)	52 (2)

Table 2. *Bond lengths (Å) and angles (°)*

S(1)—S(2)	2.021 (2)	C(8)—C(9)	1.476 (8)
S(1)—C(1)	1.811 (6)	C(9)—C(10)	1.387 (8)
S(2)—C(8)	1.827 (6)	C(10)—C(11)	1.383 (9)
C(1)—O(1)	1.212 (7)	C(11)—C(12)	1.377 (11)
C(8)—O(2)	1.198 (7)	C(12)—C(13)	1.390 (10)
C(1)—C(2)	1.475 (8)	C(13)—C(14)	1.376 (9)
C(2)—C(3)	1.392 (8)	C(14)—C(9)	1.391 (8)
C(3)—C(4)	1.377 (9)	S(1)—...—O(1)	2.655 (5)
C(4)—C(5)	1.376 (11)	S(2)—...—O(2)	2.640 (4)
C(5)—C(6)	1.382 (10)	S(2)—...—O(1)	3.009 (5)
C(6)—C(7)	1.381 (9)	S(1)—...—O(2)	2.959 (6)
C(7)—C(2)	1.380 (9)		
C(1)—S(1)—S(2)	101.5 (2)	C(6)—C(7)—C(2)	121.0 (7)
S(1)—S(2)—C(8)	100.4 (2)	S(2)—C(8)—O(2)	120.1 (5)
S(1)—C(1)—O(1)	121.6 (5)	S(2)—C(8)—C(9)	114.7 (4)
S(1)—C(1)—C(2)	113.8 (4)	O(2)—C(8)—C(9)	125.2 (5)
O(1)—C(1)—C(2)	124.5 (6)	C(8)—C(9)—C(10)	122.6 (5)
C(1)—C(2)—C(3)	118.3 (6)	C(8)—C(9)—C(14)	117.9 (5)
C(1)—C(2)—C(7)	123.4 (6)	C(10)—C(9)—C(14)	119.5 (5)
C(3)—C(2)—C(7)	118.2 (6)	C(9)—C(10)—C(11)	119.9 (6)
C(2)—C(3)—C(4)	120.8 (7)	C(10)—C(11)—C(12)	120.5 (7)
C(3)—C(4)—C(5)	120.4 (7)	C(11)—C(12)—C(13)	119.6 (7)
C(4)—C(5)—C(6)	119.4 (7)	C(12)—C(13)—C(14)	120.1 (6)
C(5)—C(6)—C(7)	120.1 (7)	C(13)—C(14)—C(9)	120.3 (6)

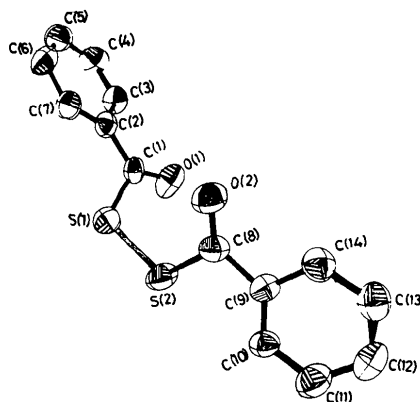


Fig. 1. *ORTEP* plot of the molecule, with thermal ellipsoids of 50% probability.

* Lists of structure factors anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38654 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Aravamudan, 1982) (II), 3,3'-dithiopropionic acid [2.033 (1); 1.819 (31) Å] (Appa Rao, Seshasayee, Aravamudan, Nageswara Rao & Venkatasubramanian, 1982) (III), L-cystine dihydrochloride [2.044; 1.865 Å] (Steinrauf, Peterson & Jensen, 1958) (IV) and *N,N'*-diglycyl-L-cystine dihydrate [2.040 (5); 1.87 (2) Å] (Yakel & Hughes, 1954) (V). The observed carbonyl C—O distances of 1.212 (7) and 1.198 (7) Å are normal. The benzene rings are planar with a maximum deviation of 0.02 (1) Å. The average C—H bond distance is 0.98 (4) Å and the average C—C—H angle is 119 (1)°. The C—S—S—C dihedral angle in DBDS is 80.8 (3)° and can be compared with those reported in (I) (96.4°), (II) (99°), (III) (78.6°), (IV) (101°) and (V) (101°). The dihedral angles C(2)—C(1)—S(1)—S(2) and S(1)—S(2)—C(8)—C(9) are -173.1 (4) and 173.8 (4)°, respectively, indicating the near planarity of the CCSS group in both thiobenzoate fragments. The dihedral angles O(1)—C(1)—S(1)—S(2) and O(2)—C(8)—S(2)—S(1) are 7.8 (5) and -9.2 (5)°, indicating again small deviation from planarity of the OCSS groups. There are two intramolecular contacts, S(1)—O(2) [2.958 (6) Å] and S(2)—O(1) [3.008 (5) Å], probably as a result of the above mentioned dihedral angles. Intermolecular contacts greater than 3.5 Å

are S(1)—O(2)* [3.531 (5) Å] and S(2)—O(2') [3.851 (5) Å].

* The prime denotes the symmetry equivalent (-x, -y, -z).

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Structure of 5'-Deoxy-5',6-epithio-5,6-dihydro-2',3'-O-isopropylideneuridine, C₁₂H₁₆N₂O₅S

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Abstract. $M_r = 300.33$, triclinic, $P1$, $a = 5.635$ (2), $b = 11.077$ (2), $c = 11.582$ (2) Å, $\alpha = 70.48$ (1), $\beta = 88.16$ (3), $\gamma = 80.56$ (3)°, $V = 670.325$ Å³, $Z = 2$, $D_x = 1.49$ Mg m⁻³, $\text{Cu K}\alpha$, $\lambda = 1.54184$ Å, $\mu = 2.308$ mm⁻¹, $F(000) = 316$, $T = 301$ K, $R = 0.054$, $R_w = 0.093$ for 1944 observed counter reflections. The sulphur position with respect to the dihydrouracil ring, which is of possible relevance to the action of thymidylate synthetase, is axial in molecule *A* and equatorial in *B*. Both molecules show the *anti* conformation about the glycosidic bond [torsion angle C(6)—N(1)—C(1')—O(4'), $\chi_{\text{CN}} = 21.6$ (9) and

29.4 (10)°] and have the C(4')-endo, O(4')-exo (⁴T) sugar conformation. The dioxolane-ring conformation is O(2')-endo in *A* and C(7)-endo in *B*. The dihydrouracil rings show self base pairing with hydrogen bonds N(3A)⋯O(2B) and N(3B)⋯O(2A).

Introduction. As part of our systematic studies on modified nucleosides (Gautham, Ramakrishnan, Seshadri, Viswamitra, Salisbury & Brown, 1982), we report here the structure of the title compound (I). A main interest of this study has been to ascertain whether the S atom is equatorial or axial with respect to the