

The authors are indebted to Dr J.-C. Molimard (Centre de Recherches Clin Midy, Montpellier, France) for fruitful discussions and for supplying the samples. The authors thank the National Belgian Foundation for Scientific Research (FNRS), IBM-Belgium and the Facultés Universitaires N.-D. de la Paix (FNDP), for the use of the Namur Scientific Computing Facility. GG would like to thank the FNDP for his doctoral fellowship.

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Acta Cryst. (1989). C45, 457–460

Short S...O Contacts: Structure of 2,5-Bis(*p*-methoxyphenylhydroxymethyl)thiophene

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(Received 2 February 1988; accepted 2 September 1988)

Abstract. C₂₀H₂₀O₄S, $M_r = 356.4$, monoclinic, $P2_1/c$, $a = 5.045$ (3), $b = 29.115$ (7), $c = 11.924$ (2) Å, $\beta = 94.82$ (3)°, $V = 1745.2$ Å³, $Z = 4$, $D_x = 1.357$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 17.86$ cm⁻¹, $F(000) = 752$, $T = 298$ K, $R = 0.038$, $wR = 0.044$ for 1212 reflections, $I > 3\sigma(I)$. In the crystal, the molecule adopts a conformation in which one of the two hydroxymethyl fragments is synplanar to S and the other is anticlinal to S. Although the two fragments are otherwise equivalent, the C–C–O bond angles in the two hydroxymethyl fragments are significantly different from each other [106.7 (3) and 110.7 (3)°]. The decrease of 4° in the bond angle is for the C–C–OH fragment that makes a short S...O contact and must indicate a non-bonded attractive interaction between the two atoms.

Introduction. The approach of presumed electrophiles and nucleophiles has been shown to exhibit a preferred geometry and angular orientation towards a divalent sulfur center (Rosenfield, Parthasarathy &

Dunitz, 1977; Guru Row & Parthasarathy, 1981; Chatterjee & Parthasarathy, 1983). These interactions, though not very strong, exhibit directional preferences at distances close to or less than the sum of van der Waals radii of the two interacting atoms. Most of these interactions occur in crystal structures along with stronger overwhelming interactions like coordination, H bonding *etc.* We have undertaken crystal structure determinations of compounds which are likely to exhibit intra- and/or intermolecular interactions where the complexity of other stronger interactions is minimized. In the present paper we discuss the structure of one such compound.

Experimental. The title compound was prepared following a reported procedure (Ulman & Manassen, 1979); needle-shaped crystals obtained from ethanol at room temperature; crystal used: 0.55 × 0.30 × 0.15 mm; CAD-4 diffractometer; unit-cell dimensions from least-squares fit of 25 reflections ($16 < 2\theta < 42^\circ$); intensity data [$2\theta_{\text{max}} = 154^\circ$ for $\lambda(\text{Cu } K\alpha)$]; $\omega/2\theta$ scan; ω scan width ($1.20 + 0.14 \tan\theta$)°; aperture width ($3.0 + 1.2 \tan\theta$) mm; maximum time spent on any reflection is 100 s; faster scan for strong reflections;

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Table 1. Fractional atomic coordinates with equivalent isotropic thermal parameters for non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms (*e.s.d.*'s are given in parentheses)

$$B_{\text{eq}} = \frac{1}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}/B_{\text{iso}}(\text{\AA}^2)$
S(1)	0.1845 (3)	0.24641 (4)	0.64137 (8)	3.07 (2)
O(1)	0.4220 (7)	0.18198 (9)	0.4956 (2)	3.59 (7)
O(2)	-0.1120 (7)	0.3100 (1)	0.8950 (2)	3.89 (7)
O(3)	0.4758 (8)	-0.0224 (1)	0.6947 (3)	5.17 (9)
O(4)	0.0587 (8)	0.4299 (1)	0.4553 (3)	5.22 (9)
C(2)	0.3111 (9)	0.1940 (1)	0.6827 (3)	2.67 (9)
C(3)	0.239 (1)	0.1830 (2)	0.7869 (3)	3.8 (1)
C(4)	0.079 (1)	0.2169 (2)	0.8323 (3)	4.0 (1)
C(5)	0.0336 (9)	0.2541 (2)	0.7639 (3)	2.99 (9)
C(6)	0.495 (1)	0.1686 (1)	0.6108 (3)	2.98 (9)
C(7)	0.4883 (9)	0.1176 (1)	0.6275 (3)	2.66 (9)
C(8)	0.677 (1)	0.0963 (2)	0.6993 (4)	3.7 (1)
C(9)	0.668 (1)	0.0499 (2)	0.7200 (4)	4.3 (1)
C(10)	0.468 (1)	0.0235 (2)	0.6672 (4)	3.6 (1)
C(11)	0.278 (1)	0.0437 (2)	0.5952 (4)	4.4 (1)
C(12)	0.288 (1)	0.0909 (2)	0.5748 (4)	3.9 (1)
C(13)	0.273 (1)	-0.0512 (2)	0.6435 (5)	5.8 (2)
C(14)	-0.140 (1)	0.2951 (2)	0.7800 (3)	3.1 (1)
C(15)	-0.0879 (9)	0.3333 (1)	0.6989 (3)	2.8 (1)
C(16)	0.116 (1)	0.3644 (2)	0.7196 (3)	3.7 (1)
C(17)	0.171 (1)	0.3979 (1)	0.6423 (4)	4.0 (1)
C(18)	0.021 (1)	0.3989 (1)	0.5403 (3)	3.4 (1)
C(19)	-0.183 (1)	0.3687 (2)	0.5165 (4)	4.0 (1)
C(20)	-0.239 (1)	0.3357 (2)	0.5963 (4)	3.7 (1)
C(21)	0.272 (1)	0.4614 (2)	0.4722 (4)	5.4 (1)
H(O1)	0.583 (9)	0.183 (1)	0.463 (3)	5 (1)*
H(O2)	0.05 (1)	0.308 (1)	0.917 (4)	7 (1)*
H(C3)	0.281 (8)	0.155 (1)	0.821 (3)	3.0 (9)*
H(C4)	0.007 (9)	0.214 (1)	0.903 (3)	5 (1)*
H(C6)	0.674 (8)	0.179 (1)	0.630 (3)	2.7 (8)*
H(C8)	0.840 (9)	0.114 (1)	0.731 (3)	5 (1)*
H(C9)	0.807 (9)	0.035 (1)	0.759 (3)	5 (1)*
H(C11)	0.142 (9)	0.025 (1)	0.557 (3)	4 (1)*
H(C12)	0.14 (1)	0.106 (1)	0.525 (3)	6 (1)*
H(C14)	-0.338 (8)	0.283 (1)	0.777 (3)	4 (1)*
H(C16)	0.22 (1)	0.368 (1)	0.790 (3)	5 (1)*
H(C17)	0.308 (9)	0.422 (1)	0.664 (3)	5 (1)*
H(C19)	-0.32 (1)	0.372 (2)	0.450 (4)	7 (1)*
H(C20)	-0.39 (1)	0.311 (1)	0.588 (4)	6 (1)*
H(C13)1	0.08 (1)	-0.039 (2)	0.662 (4)	7 (1)*
H(C13)2	0.32 (1)	-0.083 (1)	0.671 (4)	7 (1)*
H(C13)3	0.29 (1)	-0.050 (1)	0.570 (3)	6 (1)*
H(C21)1	0.45 (1)	0.444 (2)	0.485 (4)	8 (1)*
H(C21)2	0.27 (1)	0.480 (2)	0.403 (4)	8 (1)*
H(C21)3	0.25 (1)	0.479 (1)	0.530 (3)	6 (1)*

* B_{iso} .

three standards every hour of X-ray exposure; variation in intensity less than 2% during data collection; check on orientation matrix every 100 reflections; 4093 reflections measured (*h*: 0→6, *k*: 0→36, *l*: -15→15), out of which 1212 were unique and significant [$I > 3\sigma(I)$]; $R_{\text{symm}} = 0.033$; Lp correction applied; intensities of three reflections at $\chi \approx 90^\circ$ measured for different values of φ in the range from 0 to 360° and the resultant curve of transmission as a function of φ used to calculate absorption for all reflections; max. and min. transmissions are 0.95 and 0.80 and average 0.88.

Structure solved using direct-methods program MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); full-matrix refinement with anisotropic thermal parameters for non-hydrogen atoms led to R 0.069; H atoms from difference electron density map; final cycle of refinement with anisotropic

Table 2. Bond distances (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

S(1)–C(2)	1.711 (4)	C(18)–C(19)	1.366 (7)
S(1)–C(5)	1.718 (4)	C(19)–C(20)	1.398 (6)
C(2)–C(3)	1.362 (6)	C(21)–O(4)	1.415 (7)
C(2)–C(6)	1.508 (6)	O(1)–H(O1)	0.93 (5)
C(3)–C(4)	1.411 (7)	O(2)–H(O2)	0.83 (5)
C(4)–C(5)	1.365 (6)	C(3)–H(C3)	0.93 (3)
C(5)–C(14)	1.500 (6)	C(4)–H(C4)	0.95 (4)
C(6)–O(1)	1.446 (5)	C(6)–H(C6)	0.96 (4)
C(6)–C(7)	1.499 (6)	C(8)–H(C8)	1.02 (4)
C(7)–C(8)	1.375 (6)	C(9)–H(C9)	0.92 (4)
C(7)–C(12)	1.384 (6)	C(11)–H(C11)	0.95 (4)
C(8)–C(9)	1.375 (7)	C(12)–H(C12)	1.01 (4)
C(9)–C(10)	1.377 (7)	C(13)–H(C13)1	1.08 (5)
C(10)–C(11)	1.365 (7)	C(13)–H(C13)3	0.89 (4)
C(10)–O(3)	1.375 (5)	C(13)–H(C13)2	1.01 (4)
C(11)–C(12)	1.396 (6)	C(14)–H(C14)	1.06 (4)
C(13)–O(3)	1.421 (7)	C(16)–H(C16)	0.96 (4)
C(14)–O(2)	1.434 (5)	C(17)–H(C17)	1.00 (4)
C(14)–C(15)	1.512 (6)	C(19)–H(C19)	1.02 (5)
C(15)–C(16)	1.378 (7)	C(20)–H(C20)	1.03 (5)
C(15)–C(20)	1.389 (6)	C(21)–H(C21)1	1.03 (5)
C(16)–C(17)	1.384 (6)	C(21)–H(C21)2	0.99 (5)
C(17)–C(18)	1.378 (6)	C(21)–H(C21)3	0.87 (4)
C(18)–O(4)	1.381 (5)		
C(2)–S(1)–C(5)	93.1 (2)	C(9)–C(10)–C(11)	119.7 (4)
S(1)–C(2)–C(3)	110.5 (3)	C(10)–C(11)–C(12)	119.9 (5)
S(1)–C(2)–C(6)	120.4 (3)	C(7)–C(12)–C(11)	120.7 (5)
C(3)–C(2)–C(6)	128.8 (4)	O(2)–C(14)–C(5)	110.7 (3)
C(2)–C(3)–C(4)	113.0 (4)	O(2)–C(14)–C(15)	112.4 (3)
C(3)–C(4)–C(5)	113.5 (4)	C(5)–C(14)–C(15)	111.7 (4)
S(1)–C(5)–C(4)	109.9 (3)	C(14)–C(15)–C(16)	122.4 (4)
S(1)–C(5)–C(14)	121.2 (3)	C(14)–C(15)–C(20)	119.3 (4)
C(4)–C(5)–C(14)	128.7 (4)	C(16)–C(15)–C(20)	118.2 (4)
O(1)–C(6)–C(2)	106.7 (3)	C(15)–C(16)–C(17)	122.1 (4)
O(1)–C(6)–C(7)	112.7 (3)	C(16)–C(17)–C(18)	118.6 (5)
C(2)–C(6)–C(7)	112.9 (4)	C(17)–C(18)–C(19)	121.1 (4)
C(6)–C(7)–C(8)	120.6 (4)	O(4)–C(18)–C(17)	124.3 (4)
C(6)–C(7)–C(12)	121.4 (4)	O(4)–C(18)–C(19)	114.6 (4)
C(8)–C(7)–C(12)	118.0 (4)	C(18)–C(19)–C(20)	119.6 (4)
C(7)–C(8)–C(9)	121.6 (5)	C(15)–C(20)–C(19)	120.4 (5)
C(8)–C(9)–C(10)	120.1 (5)	C(10)–O(3)–C(13)	117.7 (4)
O(3)–C(10)–C(9)	115.4 (4)	C(18)–O(4)–C(21)	117.9 (4)
O(3)–C(10)–C(11)	124.9 (4)		

thermal parameters for non-hydrogen atoms and isotropic thermal parameter for H atoms; final $R = 0.038$ and $wR = 0.044$ for 1212 reflections; $S = 1.487$; function minimized $\sum w[|F_o| - (1/k)|F_c|]^2$ where $w = 4|F_o|^2/\sigma^2(|F_o|^2)$, $\sigma^2(|F_o|^2) = [\sigma(I) + s^2I^2]/Lp$, $s = 0.04$, and $\sigma(I)$ is based on counting statistics, k is the scale factor; max. $\Delta/\sigma = 0.07$; final $\Delta\rho = +0.17$ and -0.27 e \AA^{-3} . Programs from Enraf–Nonius *Structure Determination Package* (Frenz, 1979); atomic scattering factors from *International Tables for X-ray Crystallography* (1974); also *ORTEP* (Johnson, 1965).

Discussion. The final atomic parameters are given in Table 1.* Bond distances and angles are given in Table 2. A perspective view of the molecule and the numbering scheme used is shown in Fig. 1.

* Lists of anisotropic thermal parameters, torsional angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51378 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

S...O interaction

The two hydroxyl groups are twisted in opposite directions about C(2)–C(6) and C(5)–C(14) bonds resulting in O(1) being synplanar and O(2) anticlinal to S(1) [S(1)–C(2)–C(6)–O(1) angle is -29.6 (5) and S(1)–C(5)–C(14)–O(2) is -145.0 (3) $^\circ$]. O(1) makes a short intramolecular contact with S(1) with the H of the OH group pointing away from S(1) [Fig. 1; S...O distance is 2.884 (4) Å]. A molecular model shows that when the value of the torsion angle S(1)–C(2)–C(6)–O(1) is about -30° and C(2)–C(6)–O(1)–H(O1) is favourable, the $p\pi$ lone-pair orbital on O(1) points towards the back of the S(1)–C(5) bond, suitably located to interact with S(1) (Rosenfield *et al.*, 1977). The torsion angle C(2)–C(6)–O(1)–H(O1) of 144 (3) $^\circ$ orients the $p\pi$ orbital nearly towards the S(1) atom. This torsion angle also orients the other lone-pair orbital towards H(O2) resulting in a hydrogen bond with C(6)–O(1)...H(O2) 128 (2) $^\circ$.

The geometry of this S...O interaction may be described in terms of the nucleophilic approach of O to a divalent sulfur center (Rosenfield *et al.*, 1977) as follows: O(1) is nearly in the plane of the sulfide (azimuthal angle θ measured with respect to the normal to the sulfide plane is 81.1°) and nearly along the back of the S(1)–C(5) bond [φ measured from the bisector of C(2)–S(1)–C(5) is 101.7°]. Ideally, in intermolecular contacts, the preferred values of θ and φ are 80 and 135° , but due to the constraints of intramolecular interactions in this structure, the ideal value for φ is not realized.

The environments of the two hydroxyl groups are otherwise similar as indicated both by the hydrogen bonding in which they are involved and by van der Waals interactions with other atoms in the molecule (Table 3). Though the corresponding bond lengths and angles in the two methoxyphenyl groups and the thiophene ring and all bond lengths in the two hydroxymethyl groups agree within the 3σ level, two angles in the two hydroxymethyl groups [106.7 (3) $^\circ$ for C(2)–C(6)–O(1) and 110.7 (3) $^\circ$ for C(5)–C(14)–

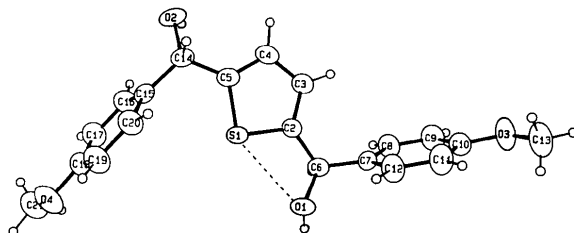


Fig. 1. A view of the molecule showing the relative orientation of the two methoxyphenylhydroxymethyl fragments. Note the S(1)...O(1) short contact [2.884 (3) Å] shown by the dotted line and the orientation of the H atom attached to O(1) turned away from S(1). The parentheses in the atomic numbering have been omitted from this figure for clarity.

Table 3. Intra- and intermolecular interactions involving O(1) and O(2) atoms showing the similarity in their environment

a	Atom			Distance (Å)			Angle ($^\circ$) a–b–c	Symmetry code
	b	c	a–b	a–c	b–c			
Hydrogen bonds								
O(1)	H(O1)	O(2)	0.93 (4)	2.737 (4)	1.81 (4)	175 (4)	(i)	
O(2)	H(O2)	O(1)	0.84 (5)	2.865 (4)	2.05 (5)	165 (4)	(v)	
van der Waals contacts								
O(1)	C(12)	H(C12)	2.914 (5)	2.66 (4)			(iii)	
O(1)	H(C16)		2.96 (4)				(iv)	
O(1)	H(C14)		3.13 (4)				(i)	
O(2)	C(16)	H(C16)	2.936 (5)	2.75 (4)			(iii)	
O(2)	H(C12)		3.11 (4)				(v)	
O(2)	H(C6)		3.10 (3)				(ii)	

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

O(2)] deviate by 4° (*i.e.* $> 13\sigma$). This reduction in the value of the bond angle C(2)–C(6)–O(1) brings the S(1) and O(1) atoms closer by 0.101 Å. This decrease in the value of C(2)–C(6)–O(1), when compared with C(5)–C(14)–O(2) taken as an internal standard, is possible only if the S(1)...O(1) interaction is attractive.

Such S...O interactions have been reported in two thiamine derivatives (Pletcher, Sax, Blank & Wood, 1977; Shin, Pletcher & Sax, 1979). However, in these two compounds the S atom carries a formal charge of $+0.5$. Short intramolecular S...O interactions (S...O distances in the range 2.4 – 3.0 Å) have been found in several other structures (Kálmán & Párkányi, 1980). The characteristic feature of these structures that exhibit short contacts is a divalent S atom and an O atom held in a nearly coplanar environment containing a conjugated system resulting in a five-membered hetero ring closed by the S...O interaction. The significant variation in the equilibrium S...O distance in these systems has been explained in terms of the nature of the atom Z attached to the S and the strength of the coupling between the Z–S antibonding orbital and the O lone-pair orbital (Cohen-Addad, Lehmann, Becker, Párkányi & Kálmán, 1984). Their conclusion agrees with ours about how nucleophiles interact with S.

Geometry and packing

The bond distances and angles observed in the present structure for the thiophene ring and methoxyphenyl rings agree well with the geometry found in many thiophene derivatives and methoxyphenyl rings found in the literature. The average geometry of the methoxy group attached to a phenyl ring, based on a survey of the 1986 Cambridge Data Base (Allen *et al.*, 1979) is as follows: O–C_{sp3} is 1.423 (10) Å; O–C_{sp2} is 1.368 (11) Å and C_{sp2}–O–C_{sp3} is 117.7 (7) $^\circ$. These values agree with the values observed for the two such moieties in the present structure [1.421 (7),

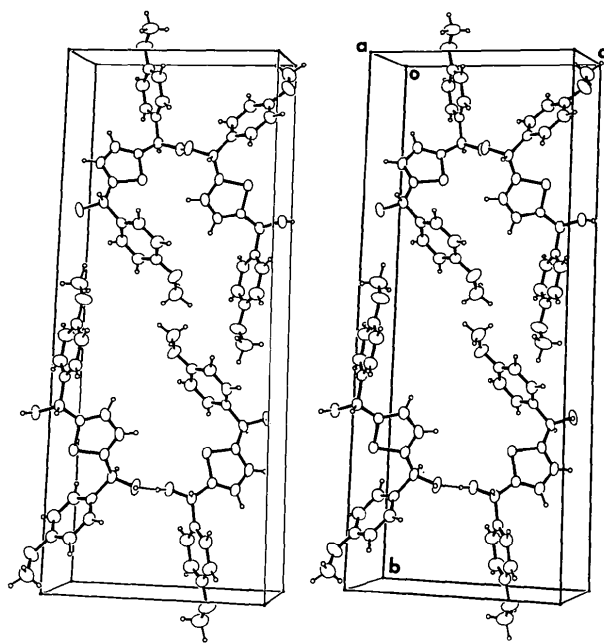


Fig. 2. A view of the packing of the molecules in the unit cell.

1.415 (7) Å; 1.375 (5), 1.381 (5) Å; and 117.7 (4), 117.9 (4)° respectively].

The crystal structure is stabilized by two hydrogen bonds (Table 3) which form a linear O—H...O—H...O—H chain running parallel to the crystallographic *a* axis. A view of the packing of the molecules is shown in Fig. 2.

The work presented here was supported by a grant from NIH, CA 23704.

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Acta Cryst. (1989). **C45**, 460–462

Structure of Bis(2-benzimidazolyl) Disulfide

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(Received 14 June 1988; accepted 26 September 1988)

Abstract. $C_{14}H_{10}N_4S_2$, $M_r = 298.40$, orthorhombic, *Pcc2*, $a = 9.964$ (3), $b = 9.967$ (3), $c = 12.890$ (3) Å, $V = 1280.26$ Å³, $Z = 4$, $D_x = 1.548$, $D_m = 1.540$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 3.56$ mm⁻¹,

$F(000) = 632$, $T = 293$ K, $R = 0.053$, $wR = 0.046$ for 1089 observed reflections. The S—S bond length is 2.077 (3) Å, longer than those in most disulfides which is indicative of the title compound's instability in

0108-2701/89/030460-03\$03.00

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