reversal of chirality per column) unless there are vacancies.

If all *y* columns suffered equally from disorder (disorder ratio 1·0:1·0) the space group would be $P2_1/m$.

The disorder ratio of 1·0:0·23 in this specimen can be explained if a fraction *p* of the columns along the *y* axis is free from disorder while the remaining $1-p$ contain just one change in chirality. [These latter columns will thus, on average, contribute $\frac{1}{2}(1-p)$ to the ordered structure.] Hence *p* should be about two-thirds (this gives a disorder ratio of 1·0:0·25). Why some columns are free from disorder and others not, is not clear.

Helpful discussions with Professor Praefcke and financial assistance from the National Research Council of Canada are both gratefully acknowledged.

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The Structure of Spiro[2(1*H*)-naphthalenone-1,2'-naphtho[1,2-*d*][1,3]oxathiole]

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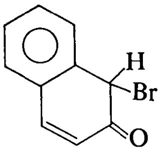
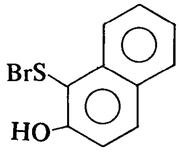
(*Received* 24 *February* 1978; *accepted* 9 *May* 1978)

Abstract. $C_{20}H_{12}O_2S$, $M_r = 316{\cdot}38$, triclinic, $P\bar{1}$; cell dimensions (chosen such that $b > a > c$, α and β obtuse): $a = 9{\cdot}042\,(2)$, $b = 10{\cdot}636\,(3)$, $c = 8{\cdot}309\,(3)$ Å, $\alpha = 93{\cdot}88\,(6)$, $\beta = 108{\cdot}52\,(9)$, $\gamma = 99{\cdot}41\,(6)°$, $Z = 2$, $D_c = 1{\cdot}42$ g cm^{-3} (D_m not determined), μ(Cu $K\alpha$) = 2·25 cm^{-1}. X-ray analysis of the title compound confirms the molecular structure assigned many years ago [Stevenson & Smiles (1930). *J. Chem. Soc.* pp. 1740–1745]. The molecule consists of a non-planar 1,3-oxathiole ring fused to a naphthalene nucleus on one side and spiro-fused to a 2(1*H*)-naphthalenone on the other. The S—C(sp^2) bond length, 1·764 (2) Å, is comparable to that found in other five-ring hetero-sulfur systems, but S—C(sp^3) at 1·874 (2) Å is significantly longer.

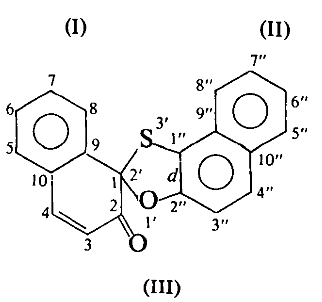
Introduction. Stevenson & Smiles (1930) ascribed to the product of the reaction of 1-bromothio-2-naphthol (II) with 1-bromo-2-naphthol (I, written in the keto-methylene form) the title compound (III, showing chemical numbering):



(I)



(II)



(III)

Praefcke (1976) and co-workers have recently re-investigated this and related reactions. X-ray analysis of compound (III) reported here confirms the original structure assignment.

Irregularly shaped reddish crystals were kindly provided by Professor Praefcke (Berlin). A crystal of dimensions *ca* 0·4 × 0·32 × 0·11 mm was used for data collection. Intensity data were recorded in the θ–2θ scan mode with a computer-controlled Picker four-circle diffractometer using Ni-filtered Cu $K\alpha$ radiation in the range $0 < 2\theta < 115°$. A total of 2987 reflections were recorded of which 2180 were regarded as significant, $I > 2\sigma(I)$, where $\sigma(I)$ was taken as (scan + $\sum B)^{1/2}$ (B is background). Scanning was at 2° min^{-1}. Repeated remeasurements of a standard reflection showed the crystal to be quite stable to X-irradiation.

The structure was solved by direct methods. The percentages of reflections having $\langle E \rangle$ greater than 3·0, 2·0 and 1·0 were 0·27, 4·27 and 31·6 respectively, implying space group $P\bar{1}$ rather than $P1$. A total of 292 reflections with $|E| > 1{\cdot}50$ were used for structure solution using *MULTAN* (Germain, Main & Woolfson, 1971). Sixteen phase sets were obtained based on four starting reflections. The one with the third highest figure of merit (1·168) yielded an E map which led to the correct solution. From this map 23 atoms were found in stereochemically feasible positions. Subsequent difference maps revealed the positions of all the remaining atoms. For least-squares refinement $\sigma(F_o)$ was taken as $\{[\sigma(I)/\mathrm{Lp}]^2 + 0{\cdot}02F_o^4\}^{1/2}/2F_o$ where Lp is the Lorentz–polarization factor. An anomalous dispersion correction $f' = -0{\cdot}319$ was made for sulfur (*International Tables for X-ray Crystallography*, 1974). The final R value with anisotropic thermal factors for non-hydrogen atoms was 0·054. Final atomic coordinates (with crystallographic in place of chemical numbering) are given in Table 1.*

Table 1. *Atomic fractional coordinates* ($\times 10^4$ *for non-hydrogen atoms and* $\times 10^3$ *for hydrogen atoms*)

	x	y	z
S	8785 (1)	3187 (1)	3825 (1)
O(1)	6020 (2)	2051 (2)	1499 (2)
O(2)	5241 (2)	3405 (2)	3886 (3)
C(1)	6884 (3)	3335 (2)	2153 (3)
C(2)	6423 (3)	1228 (2)	2719 (3)
C(3)	7817 (3)	1656 (2)	4032 (3)
C(4)	8370 (3)	876 (2)	5321 (3)
C(5)	7423 (3)	−356 (2)	5200 (3)
C(6)	5982 (3)	−756 (3)	3807 (4)
C(7)	5473 (3)	21 (3)	2574 (4)
C(8)	7953 (4)	−1160 (3)	6455 (4)
C(9)	9381 (4)	−771 (3)	7752 (4)
C(10)	10314 (4)	441 (3)	7862 (4)
C(11)	9833 (3)	1255 (3)	6689 (3)
C(12)	6044 (3)	4018 (3)	3170 (3)
C(13)	6266 (3)	5418 (3)	3241 (3)
C(14)	6781 (3)	6004 (3)	2081 (4)
C(15)	7231 (3)	5335 (2)	777 (3)
C(16)	7236 (3)	4006 (2)	749 (3)
C(17)	7611 (3)	3361 (3)	−524 (3)
C(18)	7975 (4)	4022 (4)	−1785 (4)
C(19)	7995 (4)	5322 (3)	−1748 (4)
C(20)	7636 (3)	5970 (3)	−478 (4)
H(6)	535 (5)	−157 (4)	386 (6)
H(7)	445 (6)	−21 (4)	155 (6)
H(8)	739 (5)	−203 (4)	652 (6)
H(9)	955 (5)	−153 (4)	840 (6)
H(10)	1137 (5)	76 (4)	861 (5)
H(11)	1042 (5)	223 (4)	691 (5)
H(13)	583 (5)	582 (4)	397 (6)
H(14)	679 (5)	688 (4)	214 (6)
H(17)	764 (5)	243 (4)	949 (6)
H(18)	826 (5)	357 (4)	753 (6)
H(19)	818 (5)	572 (4)	735 (6)
H(20)	759 (5)	699 (5)	946 (6)

Discussion. Bond lengths and angles are shown in Fig. 1 and an *ORTEP* plot (Johnson, 1965) of the molecule is in Fig. 2. Equations of weighted least-squares planes of interest and their dihedral angles are listed in Table 2.

The 1,3-oxathiole ring (ring C, Fig. 2) is non-planar. A best molecular fit of this ring (Nyburg, 1974) with that in an analogous compound, (IV), in which the naphthalene nuclei are replaced by tetramethylfuran (Cheng, Wong-Ng & Nyburg, 1978) gives the maximum difference as that of C(2) (Fig. 2), 0·06 Å.

The S—C(sp^3) [S—C(1)] length of 1·874 (2) Å is significantly longer than the S—C(sp^2) [S—C(3)] length of 1·764 (2) Å. The latter is in good agreement with that found elsewhere: 1·773 Å in 9β-3-methoxy-17-acetoxy-7-thiaestra-1,3,5(10),8(14)-tetraene, (V) (van

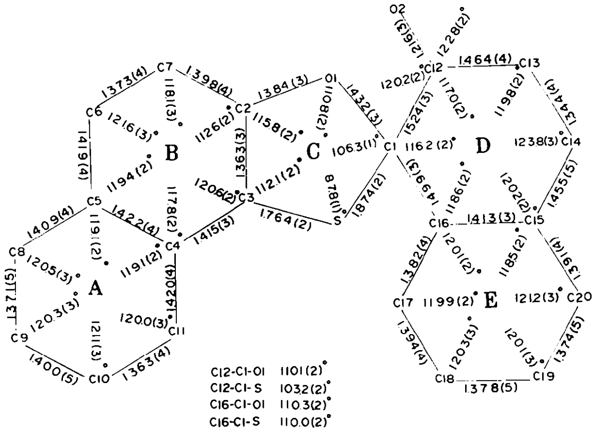


Fig. 1. Bond lengths (Å) and bond angles (°).

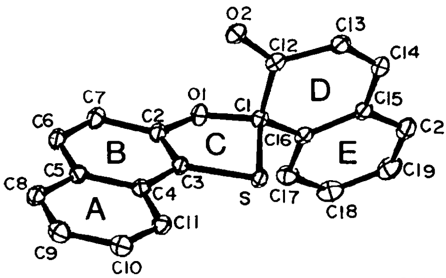


Fig. 2. *ORTEP* (Johnson, 1965) plot showing 20% probability thermal ellipsoids. Hydrogen atoms are omitted.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33608 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Best least-squares planes*

(*a*) Coefficients of planes $AX + BY + CZ = D$ where X,Y,Z are real orthogonal coordinates (in Å) with X along x and Z along z^*

		Equations of planes			
Plane	Atoms	*A*	*B*	*C*	*D*
1	Ring *A*, ring *B*, O(1), S	0·767 (1)	−0·343 (1)	−0·542 (1)	2·261 (0)
2	Ring *E*, C(1), C(14)	−0·809 (2)	−0·145 (1)	−0·569 (2)	−5·515 (1)
3	S, C(1), O(1)	0·714 (4)	0·063 (3)	−0·697 (4)	2·660 (0)
4	C(1), C(12), C(16)	−0·657 (6)	−0·391 (4)	−0·644 (6)	−5·704 (1)

(*b*) Deviations from planes (Å)

Plane								
1	S	0·001 (1)	O(1)	0·018 (2)	C(2)	−0·019 (3)	C(3)	−0·039 (2)
	C(4)	−0·033 (3)	C(5)	−0·012 (3)	C(6)	0·010 (3)	C(7)	0·003 (3)
	C(8)	0·009 (3)	C(9)	0·042 (4)	C(10)	0·026 (3)	C(11)	−0·014 (3)
	C(1)*	−0·402 (3)						
2	C(1)	−0·027 (3)	C(14)	0·034 (3)	C(15)	−0·005 (2)	C(16)	0·012 (2)
	C(17)	0·022 (3)	C(18)	0·021 (3)	C(19)	−0·016 (3)	C(20)	−0·036 (3)
	C(12)*	0·359 (3)	O(2)*	0·798 (2)	C(13)*	0·166 (3)		

(*c*) Dihedral angles (°)

	2	3	4
1	105·2 (1)	25·3 (2)	91·2 (3)
2	–	101·0 (2)	17·2 (3)
3	–	–	92·6 (4)

* These atoms were not included in the best least-squares-plane calculation.

de Ven & Schenk, 1972), 1·749 (6) Å in *N*-(*p*-bromophenylcarbamoyl)thiamine anhydride (Nakai & Koyama, 1972), 1·766 Å in the desaurin from acetone (Lynch, Mellor & Nyburg, 1971), 1·739 and 1·748 Å in 3,5-bis(pivaloylmethylene)-1,2,4-trithiolane (Mellor & Nyburg, 1971) and 1·76 Å in (IV) above.

The S—C(sp^3) bond is longer than those recorded elsewhere: 1·824 Å in (V) above, 1·843 Å in a seven-membered sulfur heterocyclic ring (Irngartinger & Rodewald, 1974). In addition to that in the present compound a number of other crystal structure determinations in this laboratory have revealed longer S—C(sp^3) bonds: 1·897 Å in (IV) above, 1·91 (1) and 1·92 (1) Å in an octamethyl-dioxathia-dispiro-undecanedione (Wong-Ng & Nyburg, 1978) and 1·900 (5) Å in a spiro(naphthalenone-naphtho-oxadithiin) (VI) (Cheng & Nyburg, 1978). There does not seem to be any significant correlation of these lengths with the internal angle at sulfur. Apart from an extra sulfur atom in the heterocyclic ring the last molecule has a structure very similar to that of the compound whose structure is reported here. In both, the spiro carbon atom has a distorted tetrahedral environment. Neither exhibits any short intramolecular distances.

Atoms S and O(1), Fig. 2, are coplanar with all the atoms of rings *A* and *B* (maximum deviation 0·04 Å). Similarly atoms C(1) and C(14) are coplanar with ring *E* (maximum deviation 0·03 Å). Atoms C(12), C(13) and O(2) are displaced in the same sense from this plane [surprisingly towards O(1) with a distance from O(2) of 2·708 (3) Å] giving ring *D* a conformation closely similar to that in (VI) [maximum deviation from best molecular fit, O(2), 0·026 Å].

The bond between C(13) and C(14) at 1·344 (4) Å suggests there is no conjugation with any other atoms in rings *D* or *E*.

There are no abnormal intermolecular distances.

The authors are indebted to Professor K. Praefcke for helpful discussions and to the National Research Council of Canada for financial assistance.

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