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Reaction Between 2-Lithio-1,3-dithiane and Tetrahydrofuran: Structure of Bis[2-(1,3-dithianyl)]methanol

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Abstract. $C_9H_{16}OS_4$, $M_r = 268{\cdot}47$, monoclinic, $P2_1/c$, $a = 6{\cdot}673$ (4), $b = 9{\cdot}806$ (3), $c = 19{\cdot}084$ (5) Å, $\beta = 97{\cdot}12$ (3)°, $V = 1239{\cdot}14$ Å^3, $Z = 4$, $D_m = 1{\cdot}44$, $D_x = 1{\cdot}44$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 65{\cdot}58$ cm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0{\cdot}041$ for 1637 unique observed reflections. The two dithiane rings are in chair conformations having similar geometries: the angle between mean (least-squares) planes through the rings is 57·9°. The S—C distances range from 1·798 (5) to 1·817 (3) Å and the C—C distances from 1·516 (6) to 1·542 (4) Å; the O—C distance is 1·420 (4) Å.

Experimental. Compound isolated unexpectedly (in 25% yield) from reaction between 2-lithio-1,3-dithiane (generated from 1,3-dithiane and *n*-butyllithium) and tetrahydrofuran which occurs on warming of the mixture from 195 to 298 K during 12 h. Crystals obtained by slow diffusion of hexane into a solution in ethyl ethanoate. Cuboid cut to *ca* 0·30 × 0·35 × 0·30 mm and mounted on glass fibre. Density measured by flotation in aqueous sodium bromide solution. Intensities measured by SERC service with an Enraf–Nonius CAD-4 diffractometer and ω–2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $16 < \theta < 28°$. Data collected to $\sin\theta/\lambda$ of 0·59 Å^{-1}, $0 < h < 7$, $0 < k < 11$, $-22 < l < 22$; empirical absorption correction applied, transmission factors 1·000–0·663. Three standard reflections ($0\bar{1}\bar{8}$, 045, $22\bar{7}$) varied ±2%; linear drift correction applied, 2299 reflections measured, 2103 unique ($R_{int} = 0{\cdot}0186$), 466 reflections with $I < 2\sigma(I)$ considered unobserved. Solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain,

Table 1. *Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s*

$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	$\bar{U}$(Å^2)
S(1)	0·1998 (2)	0·3182 (1)	0·59865 (5)	0·0531 (6)
S(3)	0·4288 (2)	0·0630 (1)	0·57485 (4)	0·0499 (6)
S(7)	0·0105 (2)	0·0727 (1)	0·76365 (5)	0·0481 (6)
S(9)	0·3424 (1)	0·2400 (1)	0·84315 (4)	0·0390 (5)
O(14)	0·4172 (4)	0·0122 (3)	0·7306 (1)	0·0474 (15)
C(2)	0·2688 (6)	0·1497 (3)	0·6309 (2)	0·0358 (17)
C(4)	0·6377 (7)	0·1793 (5)	0·5768 (2)	0·0610 (26)
C(5)	0·5793 (7)	0·3221 (5)	0·5495 (2)	0·0629 (27)
C(6)	0·4451 (8)	0·3959 (4)	0·5957 (2)	0·0638 (27)
C(8)	0·2079 (5)	0·1968 (3)	0·7573 (1)	0·0296 (16)
C(10)	0·1367 (6)	0·3103 (4)	0·8850 (2)	0·0413 (19)
C(11)	−0·0397 (6)	0·2116 (4)	0·8878 (2)	0·0458 (20)
C(12)	−0·1451 (6)	0·1734 (4)	0·8148 (2)	0·0483 (21)
C(13)	0·3589 (5)	0·1466 (3)	0·7093 (1)	0·0335 (17)

Table 2. *Bond distances* (Å), *bond angles* (°), *and their e.s.d.'s* (*not including* H *atoms*)

S(1)–C(2)	1·804 (3)	S(1)–C(6)	1·813 (5)
S(3)–C(2)	1·813 (3)	S(3)–C(4)	1·798 (5)
S(7)–C(8)	1·808 (3)	S(7)–C(12)	1·805 (4)
S(9)–C(8)	1·817 (3)	S(9)–C(10)	1·808 (4)
O(14)–C(13)	1·420 (4)	C(2)–C(13)	1·542 (4)
C(4)–C(5)	1·527 (6)	C(5)–C(6)	1·516 (6)
C(8)–C(13)	1·526 (4)	C(10)–C(11)	1·530 (5)
C(11)–C(12)	1·528 (5)		

C(2)–S(1)–C(6)	101·7 (2)	C(2)–S(3)–C(4)	101·7 (2)
C(8)–S(7)–C(12)	98·1 (2)	C(8)–S(9)–C(10)	99·8 (2)
S(1)–C(2)–S(3)	111·9 (2)	S(1)–C(2)–C(13)	113·6 (2)
S(3)–C(2)–C(13)	112·1 (2)	S(3)–C(4)–C(5)	114·2 (3)
C(4)–C(5)–C(6)	112·6 (3)	S(1)–C(6)–C(5)	114·7 (3)
S(7)–C(8)–S(9)	112·3 (2)	S(7)–C(8)–C(13)	111·3 (2)
S(9)–C(8)–C(13)	109·1 (2)	S(9)–C(10)–C(11)	113·8 (2)
C(10)–C(11)–C(12)	113·1 (3)	S(7)–C(12)–C(11)	113·4 (3)
O(14)–C(13)–C(2)	110·8 (3)	O(14)–C(13)–C(8)	107·8 (2)
C(2)–C(13)–C(8)	111·7 (3)		

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Declercq & Woolfson, 1980). Full-matrix least-squares refinement on F values with *SHELX*76 (Sheldrick, 1976). Non-H atoms refined anisotropically, H atoms calculated geometrically (except for OH which was located from the difference map) and allowed to 'ride' on associated heavy atoms with two common isotropic temperature factors for methylene, and methine H and OH, respectively, for a total of 133 variables. $R = 0{\cdot}041$, $wR = 0{\cdot}046$ where $w = 2{\cdot}6203/[\sigma^2(F) + 0{\cdot}0010F^2]$. Final $(\Delta/\sigma)_{max} < 0{\cdot}02$, $\Delta\rho_{max} = 0{\cdot}16$ and $\Delta\rho_{min} = -0{\cdot}19$ e Å^{-3} on final difference map. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom parameters are listed in Table 1 and bond lengths and angles in Table 2. The molecule and numbering scheme are shown in Fig. 1.

Related literature. Structures of 39 compounds containing the 1,3-dithianyl moiety are listed in the Cambridge Crystallographic Database. Of these, seven have the ring system unsubstituted except by a group attached through C at the 2-position. The ring geometry in the functionally simplest derivative, 2-phenyl-1,3-dithiane (Kalff & Romers, 1966), is similar to the ring geometries in the present structure.

* Lists of structure factors, H-atom coordinates and isotropic temperature factors, and anisotropic temperature factors for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43535 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

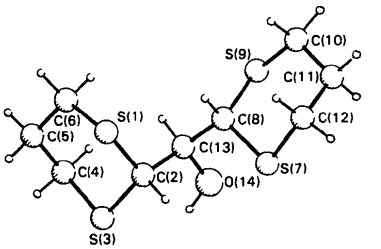

Fig. 1. A view of the molecule drawn with *PLUTO* (Motherwell & Clegg, 1978).

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Structure of a Photochromic Benzoxazine Derivative

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Abstract. 1,3,3,5,6-Pentamethylspiro(indoline-2,3′-3*H*-pyrido[3,2-*f*][1,4]benzoxazine), $C_{23}H_{23}N_3O$, $M_r = 357{\cdot}5$, monoclinic, $P2_1/c$, $a = 11{\cdot}091$ (1), $b = 16{\cdot}115$ (2), $c = 11{\cdot}085$ (1) Å, $\beta = 92{\cdot}83$ (2)°, $V = 1978{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}200$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}55$ mm^{-1}, $F(000) = 760$, $T = 293$ K, $R = 0{\cdot}097$ for 1308 unique observed reflections. The monomeric molecule, with no short intermolecular contacts, consists of a substituted benzoxazine ring linked to an indoline ring through a spiro C atom. There is a 1:1 disorder of NMe and CMe_2 in the indoline ring.

Experimental. Compound prepared by literature method (Kwak & Hurditch, 1984) as a mixture of 1,3,3,5,6- and 1,3,3,4,5-isomers. Crystallization by slow evaporation of an acetone solution at room temperature produces needle-like yellow crystals of the title compound (I) and block yellow crystals of the 1,3,3,4,5-

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