C(5)	0.6506 (6)	−0.181 (1)	0.3039 (2)	3.3 (2)
C(6)	0.7820 (7)	0.005 (1)	0.3404 (2)	3.4 (2)
C(7)	0.2951 (7)	−0.461 (1)	0.2841 (2)	3.4 (2)
C(8)	0.3449 (7)	−0.608 (1)	0.2335 (2)	3.4 (2)
C(9)	0.1788 (7)	−0.787 (1)	0.1953 (2)	3.7 (2)
C(10)	0.2546 (7)	−0.971 (1)	0.1435 (2)	3.4 (2)
C(11)	0.1089 (7)	−1.151 (1)	0.1055 (2)	4.6 (2)
C(12)	0.2112 (8)	−1.309 (1)	0.0603 (2)	4.4 (2)
C(13)	0.4716 (7)	−0.997 (1)	0.1270 (2)	3.8 (2)

Table 2. *Selected geometric parameters* (Å, °)

Cl(1)—C(1)	1.736 (4)	C(4)—C(7)	1.463 (6)
S(1)—C(13)	1.682 (5)	C(5)—C(6)	1.358 (6)
S(1)—C(12)	1.708 (5)	C(7)—C(8)	1.302 (6)
O(1)—C(9)	1.229 (5)	C(8)—C(9)	1.469 (6)
C(1)—C(6)	1.381 (6)	C(9)—C(10)	1.479 (6)
C(1)—C(2)	1.396 (6)	C(10)—C(13)	1.370 (6)
C(2)—C(3)	1.380 (6)	C(10)—C(11)	1.402 (6)
C(3)—C(4)	1.387 (6)	C(11)—C(12)	1.377 (7)
C(4)—C(5)	1.411 (5)		
C(13)—S(1)—C(12)	93.2 (2)	C(8)—C(7)—C(4)	127.5 (4)
C(6)—C(1)—C(2)	121.0 (4)	C(7)—C(8)—C(9)	122.5 (4)
C(6)—C(1)—Cl(1)	120.1 (3)	O(1)—C(9)—C(8)	121.3 (4)
C(2)—C(1)—Cl(1)	118.8 (3)	O(1)—C(9)—C(10)	119.5 (4)
C(3)—C(2)—C(1)	117.9 (4)	C(8)—C(9)—C(10)	119.2 (4)
C(2)—C(3)—C(4)	122.6 (4)	C(13)—C(10)—C(11)	110.7 (4)
C(3)—C(4)—C(5)	117.2 (4)	C(13)—C(10)—C(9)	126.1 (4)
C(3)—C(4)—C(7)	120.8 (4)	C(11)—C(10)—C(9)	123.2 (4)
C(5)—C(4)—C(7)	121.9 (4)	C(12)—C(11)—C(10)	114.7 (4)
C(6)—C(5)—C(4)	121.4 (4)	C(11)—C(12)—S(1)	108.9 (3)
C(5)—C(6)—C(1)	119.8 (4)	C(10)—C(13)—S(1)	112.4 (3)

Data collection was performed with *CONTROL* (Molecular Structure Corporation, 1988) diffractometer control software. Data were corrected for Lorentz and polarization factors. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984), the C, O, S and Cl atoms being located in an *E* map. H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for C, O, S and Cl atoms. Anomalous dispersion-corrections were not applied. All calculations were performed on a MicroVAX II computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The views of the molecule and unit cell (Figs. 1 and 2, respectively) were produced using *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71720 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1058]

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2-(*p*-Butoxyphenyl)-5-pentyl-1,3-dithiane, $C_{19}H_{30}OS_2$

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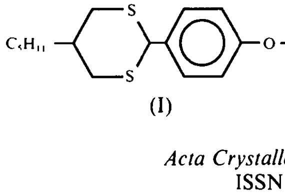
Abstract

The molecules, which are *trans* isomers, are almost linearly extended and are stacked with their axes alternately oriented. The dithiane rings in the molecules are in chair conformations and are situated opposite each other.

Comment

2,5-Disubstituted 1,3-dithianes [*R*-($C_4H_6S_2$)(C_6H_4)-*R′*] have been reported as a new type of liquid-crystal compound (Haramoto & Kamogawa, 1983, 1985*a*,*b*; Haramoto, Nobe & Kamogawa, 1984; Haramoto, Akazawa & Kamogawa, 1984). The title compound (I) with $R = n$-C_5H_{11} and $R' = n$-OC_4H_9 exhibits monotropic behaviour and the nematic liquid-crystal phase appears in the temperature range 316–299 K.



(I)

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The molecule is shown in Fig. 1. The molecules, which are *trans* isomers, are almost linearly extended. The S···S interatomic distance, the mean S—C bond length and the mean C—C bond length in the dithiane ring are 3.008 (2), 1.803 and 1.517 Å, respectively. The dithiane ring is in a chair conformation and is staggered with respect to the benzene ring in order to avoid steric repulsion. The C(2)—C(3) and C(4)—O(1) bonds are in a *gauche* conformation around the C(3)—C(4) bond, although the other C—C bonds in the butoxy and pentyl groups are in the normal *anti* conformations. The *gauche* conformation is believed to be caused by the interaction between the molecules. As the *anti* conformation is generally more stable than the *gauche*, the C(2)—C(3) and C(4)—O(1) bonds are considered to be in the *anti* conformation in the liquid state. The mean C—C bond length, excluding the C—C bonds of the benzene ring, is 1.512 Å. The packing is shown in Fig. 2. The molecules are stacked with their axes alternately oriented. The dithiane rings in neighbouring molecules are opposite each other and the S(1)···S(2) interatomic distance between them is 4.511 (2) Å.

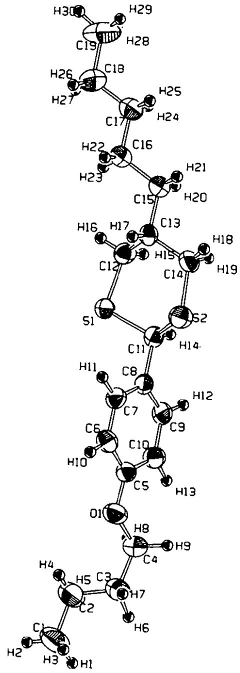

Fig. 1. Perspective drawing (*ORTEP*II; Johnson, 1976), indicating the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

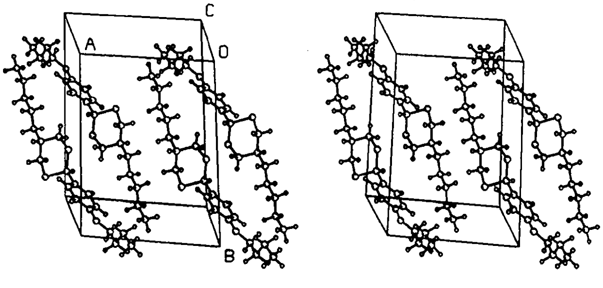

Fig. 2. Stereoscopic view (*PLUTO*; Motherwell & Clegg, 1978) of the unit cell.

Experimental

The synthesis of the title compound has been reported previously (Haramoto, Nobe & Kamogawa, 1984). The compound was recrystallized from chloroform.

Crystal data

$C_{19}H_{30}OS_2$
$M_r = 338.57$
Triclinic
$P\bar{1}$
$a = 11.285\ (1)$ Å
$b = 16.531\ (2)$ Å
$c = 5.4143\ (6)$ Å
$\alpha = 95.36\ (1)^\circ$
$\beta = 91.92\ (1)^\circ$
$\gamma = 103.90\ (1)^\circ$
$V = 974.6\ (2)$ Å^3
$Z = 2$

$D_x = 1.154$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 12.2\text{-}16.4^\circ$
$\mu = 0.262$ mm^{-1}
$T = 296$ K
Rectangular
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
ω-2θ scans [width $(1.73 + 0.30\tan\theta)^\circ$; speed 32° min^{-1} in ω]
Absorption correction: none
4680 measured reflections
4454 independent reflections
1785 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.048$
$\theta_{\rm max} = 27.5^\circ$
$h = 0 \rightarrow 14$
$k = -21 \rightarrow 21$
$l = -6 \rightarrow 6$
3 standard reflections monitored every 150 reflections intensity variation: 3%

Refinement

Refinement on F
$R = 0.046$
$wR = 0.049$
$S = 1.44$
1785 reflections
319 parameters
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\rm max} = 0.41$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{\rm eq}$
S(1)	0.9483 (1)	0.38546 (8)	0.6660 (2)	4.08 (6)
S(2)	0.7424 (1)	0.28405 (8)	0.9299 (3)	4.49 (6)
O(1)	1.1292 (3)	0.0500 (2)	0.8222 (6)	4.6 (2)

C(1)	1.3990 (8)	−0.0975 (5)	0.662 (2)	7.2 (4)
C(2)	1.3382 (7)	−0.0254 (4)	0.726 (2)	6.2 (3)
C(3)	1.2566 (6)	−0.0390 (4)	0.937 (1)	4.9 (3)
C(4)	1.2090 (6)	0.0356 (4)	1.018 (1)	4.6 (2)
C(5)	1.0794 (4)	0.1168 (3)	0.8586 (9)	3.5 (2)
C(6)	0.9996 (5)	0.1265 (3)	0.669 (1)	3.9 (2)
C(7)	0.9438 (5)	0.1915 (3)	0.6890 (9)	3.8 (2)
C(8)	0.9665 (4)	0.2499 (3)	0.8967 (8)	3.0 (2)
C(9)	1.0463 (4)	0.2408 (3)	1.081 (1)	3.6 (2)
C(10)	1.1020 (5)	0.1752 (3)	1.067 (1)	3.9 (2)
C(11)	0.9052 (4)	0.3224 (3)	0.9180 (9)	3.3 (2)
C(12)	0.8675 (5)	0.4660 (3)	0.736 (1)	3.8 (2)
C(13)	0.7307 (4)	0.4373 (3)	0.7531 (9)	3.0 (2)
C(14)	0.7002 (6)	0.3828 (3)	0.965 (1)	4.1 (2)
C(15)	0.6748 (5)	0.5131 (3)	0.786 (1)	3.9 (2)
C(16)	0.6834 (5)	0.5633 (3)	0.565 (1)	4.0 (2)
C(17)	0.6288 (5)	0.6390 (4)	0.596 (1)	4.2 (2)
C(18)	0.6474 (7)	0.6916 (4)	0.385 (1)	5.5 (3)
C(19)	0.593 (1)	0.7666 (5)	0.411 (2)	7.9 (5)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—S(2)	3.008 (2)	C(6)—C(7)	1.368 (6)
S(1)—C(11)	1.803 (4)	C(7)—C(8)	1.387 (6)
S(1)—C(12)	1.806 (5)	C(8)—C(9)	1.365 (6)
S(2)—C(11)	1.800 (5)	C(8)—C(11)	1.519 (6)
S(2)—C(14)	1.802 (5)	C(9)—C(10)	1.376 (6)
O(1)—C(4)	1.443 (6)	C(12)—C(13)	1.511 (6)
O(1)—C(5)	1.356 (5)	C(13)—C(14)	1.522 (6)
C(1)—C(2)	1.531 (8)	C(13)—C(15)	1.532 (6)
C(2)—C(3)	1.491 (8)	C(15)—C(16)	1.512 (7)
C(3)—C(4)	1.496 (7)	C(16)—C(17)	1.520 (6)
C(5)—C(6)	1.388 (6)	C(17)—C(18)	1.491 (7)
C(5)—C(10)	1.389 (6)	C(18)—C(19)	1.508 (13)
C(11)—S(1)—C(12)	100.3 (2)	C(8)—C(9)—C(10)	121.9 (5)
C(11)—S(2)—C(14)	99.1 (2)	C(5)—C(10)—C(9)	119.9 (5)
C(4)—O(1)—C(5)	118.1 (4)	S(1)—C(11)—S(2)	113.2 (3)
C(1)—C(2)—C(3)	113.3 (6)	S(1)—C(11)—C(8)	109.1 (3)
C(2)—C(3)—C(4)	113.1 (5)	S(2)—C(11)—C(8)	110.4 (3)
O(1)—C(4)—C(3)	109.3 (5)	S(1)—C(12)—C(13)	116.7 (4)
O(1)—C(5)—C(6)	116.2 (4)	C(12)—C(13)—C(14)	110.6 (4)
O(1)—C(5)—C(10)	125.4 (4)	C(12)—C(13)—C(15)	110.1 (4)
C(6)—C(5)—C(10)	118.3 (4)	C(14)—C(13)—C(15)	111.0 (4)
C(5)—C(6)—C(7)	120.7 (5)	S(2)—C(14)—C(13)	115.8 (3)
C(6)—C(7)—C(8)	121.0 (5)	C(13)—C(15)—C(16)	114.6 (4)
C(7)—C(8)—C(9)	118.2 (4)	C(15)—C(16)—C(17)	114.9 (4)
C(7)—C(8)—C(11)	121.0 (4)	C(16)—C(17)—C(18)	114.1 (4)
C(9)—C(8)—C(11)	120.8 (4)	C(17)—C(18)—C(19)	115.1 (6)

Azimuthal scans of several reflections revealed no need for an absorption correction; intensities were corrected for Lorentz-polarization effects. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). The non-H atoms were refined with anisotropic displacement parameters. H atoms located by difference Fourier synthesis were refined with isotropic displacement parameters. Full-matrix least-squares refinement minimized $\Sigma w(|F_o|-|F_c|)^2$. All calculations were performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71765 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1072]

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Anomalous Products Formed from *N*-(5,6-Dihydrobenzo[*h*]quinazolin-4-yl)-amidines and Hydroxylamine Hydrochloride

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Abstract

X-ray structure analyses of the ring cleavage and recyclization products of N^1,N^1-dimethyl-N^2-(5,6-dihydrobenzo[*h*]quinazolin-4-yl)acetamidine (I*a*) and *p*-chloro-N^1,N^1-dimethyl-N^2-(5,6-dihydrobenzo[*h*]-quinazolin-4-yl)benzamidine (I*b*) revealed the structures of 2-(3-methyl[1,2,4]oxadiazol-5-yl)-3,4-dihydro-1-naphthylaminoformaldehyde oxime (IV*a*),