Fig. 1. The packing arrangement in the crystal viewed perpendicular to the ac plane.

Fig. 2. The molecular geometry and atomic labelling of a single molecule.

$F \leq 2 \cdot 00\sigma(F_o)$. The final ΔF map showed no peaks or troughs greater than $+0 \cdot 03\,\text{e}\,\text{Å}^{-3}$. $(\Delta/\sigma)_{max} = 0 \cdot 01$.

Final atomic parameters are listed in Table 1, while the bond angles and bond lengths appear in Table 2.* The crystal packing is shown in Fig. 1. The atom-numbering scheme is shown in Fig. 2. Molecular geometry calculations were performed using CALC (Gould & Taylor, 1985).

Related literature. We recently reported the structures of 1,4-bis(4-pyridyl)butadiyne (Allan, Beaumont,

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51174 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
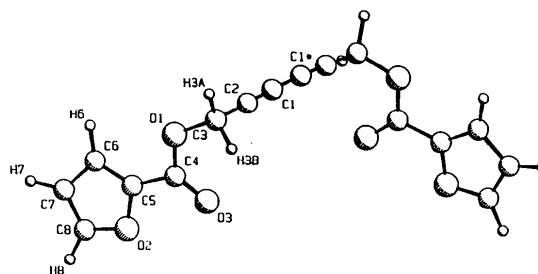
Macindoe, Milburn, Werninck & Barrow, 1988) and 1,6-diamino-1,1,1,6-tetramethyl-2,4-hexadiyne (Allan, Beaumont, Macindoe, Milburn, Welch & Werninck, 1988) as part of a study to investigate the possibility of 1,4 addition polymerization between adjacent diacetylene monomers.

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Structure of a Dithiolane

BY D. MENTZAFOS,* M. POLISIOU AND M. GEORGIADIS

Agricultural University of Athens, Iera Odos 75, 118 55 Athens, Greece

Abstract. Tetrahydropyran-3-spiro-(1',3'-dithiolane)-2-spirocyclohexane, $C_{12}H_{20}OS_2$, $M_r = 244 \cdot 4$, monoclinic, $P2_1/n$, $a = 9 \cdot 0929$ (4), $b = 9 \cdot 9160$ (8), $c = 14 \cdot 015$ (1) Å, $\beta = 100 \cdot 422$ (5)°, $V = 1242 \cdot 8$ (2) Å^3, $Z = 4$, $D_m = 1 \cdot 32$, $D_x = 1 \cdot 306$ g cm^{-3}, $\lambda(\text{Cu}\,K\bar{\alpha}) = 1 \cdot 54178$ Å, $\mu = 35 \cdot 02$ cm^{-1}, $F(000) = 528$, $R = 0 \cdot 0387$ for 1799 observed reflections with $F_o \geq 4 \cdot 0\sigma(F_o)$, $T = 296$ (2) K. The five-membered-ring atoms exhibit significant thermal motion possibly due to disorder. The structure displays no other unusual features.

Experimental. The compound was provided by Dr Tsekouras (Tsekouras, 1986). Density measured by

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* To whom correspondence should be addressed.

Table 1. *Positional and equivalent isotropic thermal parameters* $(\times 10^4)$ *of the non-hydrogen atoms*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{eq}(\text{Å}^2)$
O	3154 (2)	1959 (1)	2553·9 (9)	410
C(2)	2178 (3)	2655 (3)	1798 (2)	511
C(3)	706 (3)	2969 (3)	2086 (2)	518
C(4)	952 (2)	3727 (2)	3048 (2)	443
C(5)	2052 (2)	2972 (2)	3828 (1)	365
C(6)	3538 (2)	2677 (2)	3461 (1)	351
C(7)	4583 (3)	1723 (2)	4114 (2)	454
C(8)	6031 (3)	1472 (3)	3749 (2)	581
C(9)	6828 (3)	2776 (3)	3597 (3)	699
C(10)	5819 (3)	3724 (3)	2954 (2)	624
C(11)	4377 (2)	3993 (2)	3330 (2)	428
C(13)	1068 (5)	3184 (4)	5538 (3)	1107
C(14)	938 (5)	1772 (4)	5311 (2)	1081
S(1)	2424·7 (6)	3970·0 (6)	4946·7 (4)	530
S(2)	1136·6 (6)	1391·4 (5)	4108·3 (4)	505

Fig. 1. An *ORTEP* diagram with 50% probability thermal ellipsoids for the non-H atoms.

Table 2. *Bond distances* (Å) *and bond angles* (°)

Maximum e.s.d.'s are 0·004 Å for distances and 0·2° for angles.

O–C(2)	1·430	C(14)–S(2)	1·768
C(2)–C(3)	1·499	S(2)–C(5)	1·850
C(3)–C(4)	1·525	C(6)–C(7)	1·523
C(4)–C(5)	1·536	C(7)–C(8)	1·517
C(5)–C(6)	1·559	C(8)–C(9)	1·516
C(6)–O	1·443	C(9)–C(10)	1·497
C(5)–S(1)	1·833	C(10)–C(11)	1·522
S(1)–C(13)	1·785	C(11)–C(6)	1·539
C(13)–C(14)	1·436		

C(2)–O–C(6)	115·7	O–C(6)–C(11)	111·1
O–C(2)–C(3)	111·4	C(5)–C(6)–C(11)	111·0
C(2)–C(3)–C(4)	110·2	C(7)–C(6)–C(11)	109·2
C(3)–C(4)–C(5)	111·2	C(5)–S(1)–C(13)	97·2
C(4)–C(5)–C(6)	110·4	S(1)–C(13)–C(14)	111·0
C(4)–C(5)–S(1)	110·0	C(13)–C(14)–S(2)	113·6
C(6)–C(5)–S(1)	110·3	C(14)–S(2)–C(5)	98·6
C(4)–C(5)–S(2)	107·7	C(6)–C(7)–C(8)	112·6
C(6)–C(5)–S(2)	111·2	C(7)–C(8)–C(9)	112·0
S(1)–C(5)–S(2)	107·2	C(8)–C(9)–C(10)	111·3
O–C(6)–C(5)	107·3	C(9)–C(10)–C(11)	112·0
O–C(6)–C(7)	104·4	C(10)–C(11)–C(6)	111·7
C(5)–C(6)–C(7)	113·7		

flotation. Syntex $P2_1$ diffractometer. Crystal size 0·2 × 0·5 × 0·5 mm. Lattice parameters from 15 automatically centered reflections, $50° < 2\theta < 58°$. Ni-filtered Cu $K\bar\alpha$ radiation, θ–2θ scan. $2\theta \le 120°$, h −10→10, k 0→11, l 0→15. Variable scan speed 2·0–16·0 $(2\theta)°$ min^{-1}. Scan range 1·8° (2θ) plus $\alpha_1 - \alpha_2$ separation, background counting time 0·5 of scan time. Three standard reflections monitored periodically showed less than 3% intensity fluctuation. Lp and numerical absorption correction applied. Transmission factors 0·5317–0·3247. Data collected/unique/R_{int}: 2367/1848/C·023. Structure solved and refined by *SHELX*76 (Sheldrick, 1976). Positions of the S and five C atoms deduced by direct methods. Remaining non-H atoms revealed from ΔF calculations. The high thermal parameters for C(13) and C(14) suggest that these atoms are disordered, but a difference Fourier map of this region could not resolve the disorder. Such disorder is not uncommon in $(CH_2)_2$ groups (Psycharis,
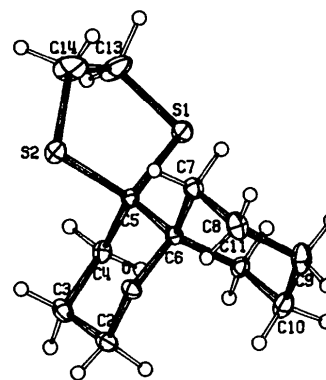
Hountas, Terzis & Papavassiliou, 1988; Terzis, Psycharis, Hountas & Papavassiliou, 1988). Refinement based on F. Positions of the H atoms except those on C(13) and C(14) located from ΔF map and refined; those on C(13) and C(14) fixed with C–H distance of 1·08 Å. Least-squares refinement minimized $\sum wF^2$, $1/w = \sigma^2(F_o) + 0·00096|F_o|^2$. Non-H atoms anisotropic. $|\Delta/\sigma|_{max} = 0·019$. $\Delta\rho_{max}/\Delta\rho_{min}$, $0·44/−0·34$ e Å^{-3}. R/wR, $0·0387/0·0571$ for 1799 observed reflections. R/wR, $0·0391/0·0571$ for all data. Atomic scattering factors from *SHELX*76. The final atomic parameters of the non-H atoms are given in Table 1.* Bond distances and angles are given in Table 2. A view of the molecule is shown in Fig. 1 (Johnson, 1976).

Related literature. The compound was an intermediate step for the synthesis of dacus oleae pheromone analogues (Mori, Uematsu, Watanabe, Yanagi & Minobe, 1984) and the structure needed to be ascertained before proceeding with the next step in the synthesis.

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