Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Zachariasen, W. H. (1968). *Acta Cryst.* A**24**, 212–216.

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meso-3,3′-Bi(1,2,4-trithiacyclohex-5-enyl)†

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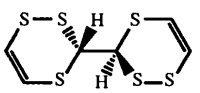
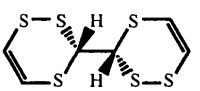
Abstract

The stereochemistry of the title compound, $C_6H_6S_6$, was determined to have *meso* geometry. The molecule has C_i symmetry with a dihedral angle around the disulfide moiety of −64.25 (10)°, a reduction from the normal strain-free geometry (*ca* 90°) of the disulfide.

Comment

Cyclic polysulfides have been the subject of considerable interest (Sato, 1990). In the course of our work on sulfur-containing unsaturated cyclic compounds, 3,3′-bi(1,2,4-trithiacyclohex-5-enyl) was obtained as a diastereomeric mixture (Shimizu, Iwata & Kamigata, 1996). One isomer, the title compound, could be isolated by recrystallization of the diastereomeric mixture. However, if the compound had *meso* or *dl* geometry [(I) and (II), respectively], it could not be determined from spectral data such as ^{1}H and ^{13}C NMR, IR and MS. The geometry of the title compound, isolated as yellow prisms, was therefore determined by X-ray crystallographic analysis to be *meso* form.


(I)


(II)

The atoms of the six-membered ring, except for an S atom at the 2 position, are nearly planar. The dihedral angle around the disulfide moiety is −64.25 (10)°, a reduction from the normal strain-free geometry of the disulfide (*ca* 90°; Dixon, Zeroka, Wendoloski & Wasserman, 1985; Block & Jansen, 1985; Rauk, 1984; Shimizu, Iwata, Kamigata & Ikuta, 1997), whereas the S—C═C—S torsion angle is 2.0 (3)°, indicating an almost strain-free geometry of this part of the molecule.

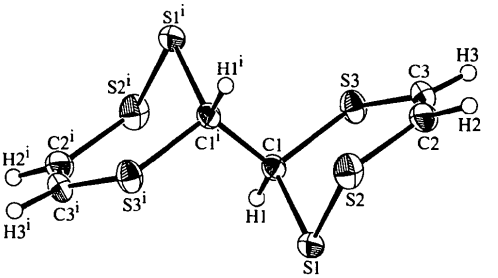


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids. [Symmetry code: (i) $-x, -y, 1-z$].

Experimental

3,3′-Bi(1,2,4-trithiacyclohex-5-enyl) was obtained as a diastereomeric mixture by oxidation of *cis*-disodium ethene-1,2-dithiolate and photochemical reaction of 1,2,5,6-tetrathiacycloocta-3,7-diene. The title compound was isolated as yellow prisms by recrystallization from chloroform (m.p. 400.5–401.5 K). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.77 (*d*, 2H, J = 9.5 Hz), 6.62 (*d*, 2H, J = 9.5 Hz), 4.94 (*s*, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 126.5, 115.4, 56.1; IR (KBr): 3030, 3020, 2905, 1540, 1125, 810 cm^{-1}; UV (cyclohexane): 325 (*sh*, ε 1.4 × 10^3), 247 (ε 6.1 × 10^3) nm; EI MS: *m/z* 271 (M^+ + 1), 207, 180, 147, 135, 116, 90, 58; calculated for $C_6H_6S_6$: C 26.64, H 2.24%; found: C 27.14, H 2.36%.

Crystal data

$C_6H_6S_6$
M_r = 270.47
Monoclinic
$P2_1/c$
a = 8.111 (2) Å
b = 6.146 (1) Å
c = 10.242 (1) Å
β = 103.19 (1)°
V = 497.1 (1) Å^3
Z = 2
D_x = 1.807 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 19.7–19.9°
μ = 1.313 mm^{-1}
T = 293 K
Prism
0.40 × 0.30 × 0.30 mm
Yellow

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.572, T_{max} = 0.674
1340 measured reflections
1257 independent reflections

1073 reflections with $I > 3\sigma(I)$
R_{int} = 0.0123
θ_{max} = 27.55°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 8$
$l = -13 \rightarrow 12$
3 standard reflections
every 150 reflections
intensity decay: 0.29%

† Alternative name: 3,3′-bi(3*H*-1,2,4-trithiinyl).

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = <0.001
R = 0.0286	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
wR = 0.0345	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
S = 3.901	Extinction correction: none
1073 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
67 parameters	
All H atoms refined	
$w = 1/[\sigma^2(F_o) + 0.00002\|F_o\|^2]$	

Table 1. *Selected geometric parameters (Å, °)*

S1—S2	2.0398 (8)	S3—C3	1.731 (2)
S1—C1	1.818 (2)	C1—C1^{i}	1.521 (4)
S2—C2	1.741 (2)	C2—C3	1.316 (3)
S3—C1	1.817 (2)		
S2—S1—C1	99.18 (6)	S1—C1—C1^{i}	113.5 (2)
S1—S2—C2	100.48 (8)	S3—C1—C1^{i}	113.0 (2)
C1—S3—C3	106.75 (9)	S2—C2—C3	125.7 (2)
S1—C1—S3	111.13 (9)	S3—C3—C2	132.5 (2)

Symmetry code: (i) $-x, -y, 1-z$.

The weak reflections [$I < 10\sigma(I)$] were rescanned (maximum five times) and the counts were accumulated to assure good counting statistics. The structure was solved by the direct methods *SHELXS*86 program (Sheldrick, 1985) and expanded using Fourier techniques (*DIRDIF*; Beurskens *et al.*, 1992). The positions of all H atoms were determined from the difference Fourier synthesis map and refined isotropically. The non-H atoms were refined anisotropically. All calculations were performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985, 1992) and *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988) was used for data collection and cell refinement.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1429). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System.* Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Block, R. & Jansen, L. (1985). *J. Chem. Phys.* **82**, 3322–3328.

Dixon, D. A., Zeroka, D., Wendoloski, J. J. & Wasserman, Z. R. (1985). *J. Phys. Chem.* **89**, 5334–5336.

Molecular Structure Corporation (1985, 1992). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Rauk, A. (1984). *J. Am. Chem. Soc.* **106**, 6517–6524.

Sato, R. (1990). *Rev. Heteroatom Chem.* **3**, 193–210, and references therein.

Sheldrick, G. M. (1985). *SHELXS*86. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.

Shimizu, T., Iwata, K. & Kamigata, N. (1996). *Angew. Chem. Int. Ed. Engl.* **35**, 2357–2359.

Shimizu, T., Iwata, K., Kamigata, N. & Ikuta, S. (1997). *J. Chem. Res.* (*S*), 38–39; *J. Chem. Res.* (*M*), 344–354.

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2,5-Diaziridinyl-3-phenyl-*p*-benzoquinone

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Abstract

The crystal structure of the title compound, $C_{16}H_{14}N_2O_2$, a novel anticancer agent, has been determined at 295 (1) K. The aziridine rings form approximately regular triangles, with N—C distances of 1.442 (5)–1.456 (5) Å, C—C distances of 1.462 (6) and 1.467 (6) Å, C—N—C angles of 60.5 (3) and 60.7 (3)°, and C—C—N angles of 59.7 (3)–60.1 (3)°. The aziridine ring planes make dihedral angles of 48.1 (3) and 48.3 (3)° with the quinone ring. The dihedral angle between the quinone ring plane and the phenyl ring is 49.8 (2)°. The molecules are linked by C—H···*X* hydrogen bonding into bilayers which stack along the *c* axis; C···O distances range from 3.355 (4) to 3.631 (5) Å and the unique C···N distance is 3.427 (5) Å.

Comment

Mitomycin C (MC) is an important clinical anticancer agent which is effective for the treatment of a wide range of solid tumors (Crooke & Bradner, 1976; Powis, 1987; Verweij & Pinedo, 1990). Its significant biological activity has, in part, been attributed to its selective activation in cancerous tissues by reducing enzymes, such as DT-diaphorase (DTD) (Siegel, Gibson, Preusch & Ross, 1990). Marked elevation in DT-diaphorase activity and mRNA content have been documented in both preneoplastic and established cancers (Malkinson