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Structure of a Twelve-Membered Heterocycle with Three Disulfide Groups

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Abstract. 1,2,5,6,9,10-Hexathiacyclododecane, $C_6H_{12}S_6$, $M_r = 276{\cdot}55$, $P2_1/c$, $a = 13{\cdot}222\ (3)$, $b = 5{\cdot}258\ (1)$, $c = 17{\cdot}334\ (4)$ Å, $\beta = 106{\cdot}15\ (2)°$, $V = 1157{\cdot}5\ (4)$ Å^3, $Z = 4$, $D_m(298\ K) = 1{\cdot}55$, $D_x(140\ K) = 1{\cdot}59$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 10{\cdot}9$ cm^{-1}, $F(000) = 576$, $T = 140$ K, $R = 0{\cdot}029$, 2661 unique reflections. The three S–S bond lengths and C–S–S–C torsion angles are 2·039 (1) Å, −89·4 (1)°; 2·041 (1) Å, −104·1 (1)°; 2·039 (1) Å, 89·3 (1)°. All other structural features are normal.

Experimental. Recrystallized from methanol; colorless needles, 0·25 × 0·37 × 0·62 mm; density measured by flotation in C_6H_5Cl/CCl_4; Syntex $P2_1$, modified LT-1 low-temperature apparatus; ω scans, 1° range, 1° offset for background counts, 60° min^{-1} (Hope & Nichols, 1981); 12 reflections with $23 < 2\theta < 27°$ for lattice parameters; no absorption correction applied, A^* 1·27–1·46; $(\sin\theta/\lambda)_{max} = 0{\cdot}65$ Å^{-1}; h from 0 to 16, k from 0 to 6, l from −22 to 22; 2 standards (500 and 11$\bar{3}$), no decay; 3141 reflections measured, 2661 unique with $I > 2{\cdot}5\sigma(I)$; $R_{int} = 0{\cdot}013$; direct methods; refinement based on F; H atoms at calculated positions, $U_{iso} = 1{\cdot}2\ U_{iso}$ of bonded C atom; 109 parameters; $R = 0{\cdot}029$; $wR = 0{\cdot}034$, $S = 1{\cdot}09$; $w = [\sigma^2(F) + 0{\cdot}0010F^2]^{-1}$; $(\Delta/\sigma)_{max} = 0{\cdot}006$; $(\Delta\rho)_{max} = 0{\cdot}71$, $(\Delta\rho)_{min} = -0{\cdot}68$ e Å^{-3}; scattering factors and f', f'' from *International Tables for X-ray Crystallography* (1974); computer programs from the *SHELXTL* package (Sheldrick, 1981). Table 1 contains final atom coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms; Table 2 lists bond distances and angles and torsion angles. Figs. 1 and 2 illustrate aspects of the structure.‡

Related literature. The experimental procedure for the synthesis of ring disulfides has been described (Goodrow & Musker, 1981). Two relevant crystal structures are those of 1,2,5,6-tetrathiacyclooctane (Goodrow, Olmstead & Musker, 1982) and 1,2,6,7-tetrathiacyclodecane (Goodrow, Olmstead & Musker, 1983).

Table 1. *Atom coordinates* (×10^4) *and thermal parameters* (Å^2 ×10^3)

	x	y	z	U^*
S(1)	3460 (1)	9092 (1)	4277 (1)	18 (1)
S(2)	3495 (1)	8085 (1)	5419 (1)	20 (1)
S(3)	754 (1)	8222 (1)	4358 (1)	31 (1)
S(4)	283 (1)	5612 (1)	3458 (1)	30 (1)
S(5)	2884 (1)	4078 (1)	2333 (1)	28 (1)
S(6)	3401 (1)	7750 (1)	2421 (1)	25 (1)
C(1)	2543 (2)	5503 (4)	5297 (1)	23 (1)
C(2)	1428 (2)	6351 (5)	5236 (1)	33 (1)
C(3)	1181 (2)	5915 (4)	2830 (1)	26 (1)
C(4)	2035 (2)	3915 (4)	3007 (1)	22 (1)
C(5)	4604 (2)	7727 (4)	3252 (1)	26 (1)
C(6)	4455 (2)	7027 (4)	4061 (1)	19 (1)

* Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Molecular geometry* (Å,°)

S(1)–S(2)	2·039 (1)	S(1)–C(6)	1·822 (2)
S(2)–C(1)	1·823 (2)	S(3)–S(4)	2·041 (1)
S(3)–C(2)	1·823 (2)	S(4)–C(3)	1·827 (3)
S(5)–S(6)	2·039 (1)	S(5)–C(4)	1·834 (2)
S(6)–C(5)	1·824 (2)	C(1)–C(2)	1·516 (3)
C(3)–C(4)	1·511 (3)	C(5)–C(6)	1·516 (3)

S(2)–S(1)–C(6)	102·8 (1)	S(1)–S(2)–C(1)	104·6 (1)
S(4)–S(3)–C(2)	104·5 (1)	S(3)–S(4)–C(3)	106·6 (1)
S(6)–S(5)–C(4)	104·4 (1)	S(5)–S(6)–C(5)	104·7 (1)
S(2)–C(1)–C(2)	114·6 (2)	S(3)–C(2)–C(1)	117·1 (2)
S(4)–C(3)–C(4)	113·0 (2)	S(5)–C(4)–C(3)	113·2 (2)
S(6)–C(5)–C(6)	115·0 (2)	S(1)–C(6)–C(5)	109·4 (1)

S(1)–S(2)–C(1)–C(2)	−89·6 (2)	C(3)–C(4)–S(5)–S(6)	53·8 (1)
S(2)–C(1)–C(2)–S(3)	65·7 (2)	C(4)–S(5)–S(6)–C(5)	89·3 (1)
C(1)–C(2)–S(3)–S(4)	78·3 (2)	S(5)–S(6)–C(5)–C(6)	−63·3 (2)
C(2)–S(3)–S(4)–C(3)	−104·1 (1)	S(6)–C(5)–C(6)–S(1)	−54·0 (2)
S(3)–S(4)–C(3)–C(4)	99·7 (1)	C(5)–C(6)–S(1)–S(2)	−174·5 (1)
S(4)–C(3)–C(4)–S(5)	176·2 (1)	C(6)–S(1)–S(2)–C(1)	−89·4 (1)

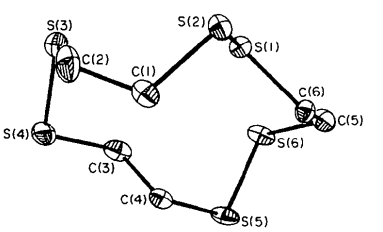

Fig. 1. A computer-generated drawing of $C_6H_{12}S_6$ showing the atom numbering scheme and anisotropic thermal ellipsoids at the 50% probability level.

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‡ Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and synthesis details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42560 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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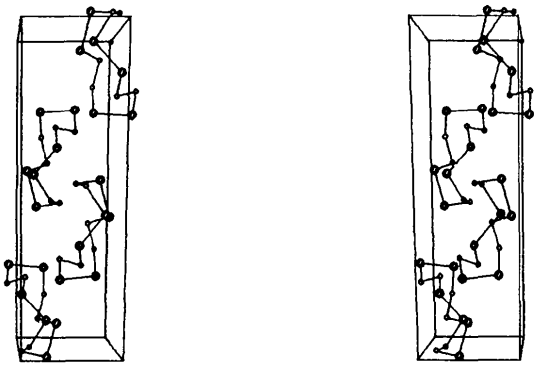

Fig. 2. A stereoview down **a** showing the molecular packing. Sulfurs are drawn slightly larger than carbons.

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Obituary

John E. Derry
30 July 1946—2 September 1985

Dr John E. Derry, Deputy Technical Editor of the International Union of Crystallography, died on 2 September 1985. He was born in Birmingham in 1946. After graduating from Birmingham University in chemistry in 1967 he continued his studies there for a PhD in crystallography under Dr T. A. Hamor. He joined the Union in 1972 as an Editorial Assistant, being promoted to Assistant Technical Editor in 1976, a position which was designated Deputy Technical Editor in 1983.

He was responsible for the editing and production of Section B and Section C of *Acta Crystallographica.* He brought to this work considerable editorial skills and an enviable capacity for handling papers rapidly and with great accuracy on a wide range of complicated structure determinations. He was the Union's expert on chemical nomenclature and recently had been developing checking procedures with various crystallographic data bases for crystal structure papers submitted for publication in the Union's journals.

His other interests included literature, particularly science fiction, films, and hill walking. His quiet efficiency, his dry sense of humour and his concern for the editorial staff working under him made him popular with his colleagues, who held him in high esteem. He is sorely missed.