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1-Methyl-5-thia-1-thioniacyclooctane Iodide, $C_7H_{15}S_2^+.I^-$

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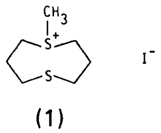
Abstract. $M_r = 290\cdot23$, orthorhombic, $P2_12_12_1$, $a = 6\cdot736$ (2), $b = 12\cdot287$ (3), $c = 13\cdot046$ (4) Å, $V = 1079\cdot7$ (7) Å^3, $Z = 4$, D_m (298 K) = 1·77 (1), D_x(85 K) = 1·79 (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 32\cdot5$ cm^{-1}, $F(000) = 568$, $T = 85$ K. Final $R = 0\cdot020$ for 1447 unique reflections. The compound results from the reaction of 1,5-dithiacyclooctane and excess methyl iodide in diethyl ether. The S···S non-bonded distance of 3·259 (2) Å and C—S···S angle of 174·2 (1)° present in the cation suggest an intramolecular interaction.

Introduction. Certain sulfur-bonded derivatives of 1,5-dithiacyclooctane have been found to have relatively short transannular S···S contact distances in the range 3·121 (5)–3·271 (3) Å. These are 1-acetonyl-5-thia-1-thioniacyclooctane perchlorate (Johnson, Maier & Paul, 1970), 1,5-dithiacyclooctane diiodine adduct (Nichols, 1981) and *trans*-bis(1,5-dithiacyclooctane)tetrachlorotin(IV) (Olmstead, Williams & Musker, 1982). The structure of (1) was of interest in order to determine the transannular distance in the simple *S*-methyl derivative, in which the electronic effects of the derivative group would be minimal.



(1)

Experimental. Sticky, yellow crystals of title compound recovered directly from reaction mixture of methyl iodide and 1,5-dithiacyclooctane, no dialkylation product formed; D_m measured by flotation in $C_2H_2Br_4/CCl_4$; crystal of dimensions 0·37 × 0·42 × 0·75 mm selected for data collection, several others rejected due to twinning; Syntex $P2_1$ diffractometer equipped with a low-temperature apparatus; lattice parameters computed from a least-squares fit of 12 reflections with $30° < 2\theta < 35°$; systematic absences for $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$ indicated space group $P2_12_12_1$ (No. 19); an empirical absorption correction assuming an ellipsoid applied with range of absorption correction factors 6·8–7·8; data collected to $2\theta_{\max} = 55°$ in positive octant with maximum indices h, k, l of 9, 16, 17 respectively; two check reflections showed less than a 2% average fluctuation during data collection; total of 1447 unique reflections measured of which nine having $F < 3\sigma(F)$ rejected; structure solved by Patterson and Fourier techniques, refined by full-matrix least squares (based on F) using $w = 1/[\sigma^2(F) + 0\cdot00008F^2]$; methyl H atoms located on a difference map and refined as a rigid body with the bonded C atom, allowing $U_{\rm iso}$ to vary; remaining H atoms placed at computed positions and assigned $U_{\rm iso} = 1\cdot2\,U_{\rm eq}$ (bonded C), refined using a riding model in which they contribute to the derivatives used in the least-squares procedure; non-H atoms assigned anisotropic thermal parameters, bringing total number of parameters to 94; in final cycle of refinement, maximum shift/e.s.d. —0·379 for overall scale and average shift/e.s.d. 0·037; final difference electron density map showed a maximum and minimum peak height of 0·39 and —0·80 e Å^{-3} respectively in vicinity of the I atom;

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$R = 0{\cdot}020$, $R_w = 0{\cdot}021$, $S = 1{\cdot}81$; computer programs were those of *SHELXTL* (Sheldrick, 1978); scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.*

The results of the crystal-structure determination show the presence of cationic $C_7H_{15}S_2^+$ and I^- ions in the crystal. A drawing of the cation is given in Fig. 1. The 1,5-dithiacyclooctane ring conformation is chair–chair. Torsion angles, together with bond distances and angles, are listed in Table 2. The transannular S(1)···S(2) distance of 3·259 (2) Å and C(1)—S(1)···S(2) angle of 174·2 (1)° may be indicative of a long-range interaction between S atoms (Olmstead *et al.*, 1982).

Non-bonded interactions between cation and anion are presented in Fig. 2. The I^- ion has non-bonded contacts to two S(1) atoms of 3·748 (1) and 3·857 (1) Å, which form an angle of 98·1° with the I^- ion. Each S(1) atom, in turn, interacts with two I^- ions at these distances; one approaches along the line of the S(1)—C(7) bond at an angle of 175·0 (1)° and the other along the line of the S(1)—C(2) bond at an angle of 175·4 (1)°. These contacts are another example of the generalization (Rosenfield, Parthasarathy & Dunitz, 1977) that '... nucleophiles tend to approach along the extension of one of the covalent bonds to S.' We also note that if the three bonded and the three non-bonded atoms surrounding S(1) are taken into account, there is a roughly octahedral arrangement of atoms around S(1).

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Table 1. *Atom coordinates* (× 10⁴) *and equivalent isotropic thermal parameters* (Å² × 10³) *for the non*-H *atoms with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
I	170 (1)	597 (1)	546 (1)	16 (1)
S(1)	6939 (2)	1754 (1)	8361 (1)	14 (1)
S(2)	9999 (2)	2525 (1)	6568 (1)	18 (1)
C(1)	5113 (8)	1265 (4)	9247 (3)	19 (1)
C(2)	7839 (7)	431 (3)	7935 (4)	18 (1)
C(3)	9889 (8)	436 (4)	7419 (4)	20 (1)
C(4)	10038 (9)	1045 (3)	6408 (3)	20 (1)
C(5)	7691 (7)	2929 (4)	5945 (3)	20 (1)
C(6)	6014 (7)	3148 (4)	6697 (4)	18 (1)
C(7)	5288 (8)	2191 (4)	7319 (3)	18 (1)

* $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

Table 2. *Interatomic distances* (Å), *angles* (°) *and torsion angles* (°)

S(1)—C(1)	1·791 (5)	S(1)—C(2)	1·822 (4)
S(1)—C(7)	1·837 (5)	S(2)—C(4)	1·830 (4)
S(2)—C(5)	1·824 (5)	C(2)—C(3)	1·536 (7)
C(3)—C(4)	1·520 (6)	C(5)—C(6)	1·520 (7)
C(6)—C(7)	1·510 (6)	S(1)···S(2)	3·259 (2)
C(1)—S(1)—C(2)	97·2 (2)	C(1)—S(1)—C(7)	99·2 (2)
C(2)—S(1)—C(7)	103·7 (2)	C(4)—S(2)—C(5)	103·4 (2)
S(1)—C(2)—C(3)	115·4 (3)	C(2)—C(3)—C(4)	116·2 (4)
S(2)—C(4)—C(3)	112·9 (3)	S(2)—C(5)—C(6)	113·2 (3)
C(5)—C(6)—C(7)	116·7 (4)	S(1)—C(7)—C(6)	115·4 (3)
C(1)—S(1)···S(2)	174·2 (1)		
C(1)—S(1)—C(2)—C(3)	−161·0 (3)	C(4)—S(2)—C(5)—C(6)	100·9 (4)
C(1)—S(1)—C(7)—C(6)	148·7 (3)	S(2)—C(5)—C(6)—C(7)	−64·5 (5)
S(1)—C(2)—C(3)—C(4)	−65·1 (5)	C(5)—C(6)—C(7)—S(1)	74·4 (5)
C(2)—C(3)—C(4)—S(2)	73·8 (5)	C(6)—C(7)—S(1)—C(2)	−111·5 (4)
C(3)—C(4)—S(2)—C(5)	−114·7 (4)	C(7)—S(1)—C(2)—C(3)	97·7 (4)

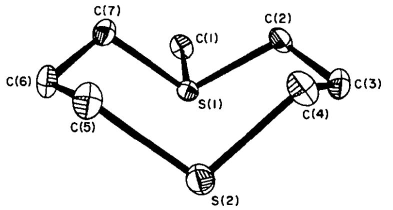

Fig. 1. A perspective drawing of $C_7H_{15}S_2^+$ showing the atom-labeling scheme and anisotropic thermal ellipsoids at the 50% probability level.

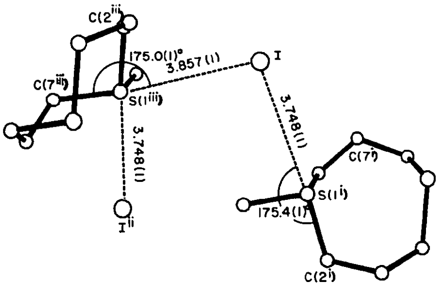

Fig. 2. Non-bonded interactions between I^- and S(1) (distances in Å). Symmetry code: (i) $x-\frac{1}{2}$, $\frac{1}{2}-y$, $1-z$; (ii) $x-\frac{1}{2}$, $\frac{1}{2}-y$, $-z$; (iii) $x-1, y, z-1$.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British library Lending Division as Supplementary Publication No. SUP 38419 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.