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Structure of 4-Oxo-5,6,7,8-tetrahydro-3*H*-cyclohexathieno[2,3-*d*]pyrimidine

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Abstract. $C_{10}H_{10}N_2OS$, $M_r = 206{\cdot}3$, monoclinic, $P2_1/n$, $a = 6{\cdot}943\ (2)$, $b = 13{\cdot}216\ (2)$, $c = 10{\cdot}342\ (2)$ Å, $\beta = 101{\cdot}1\ (2)°$, $V = 931{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}47$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 7{\cdot}5$ cm^{-1}, $F(000) = 432$, $T = 296$ K, $R = 0{\cdot}06$ for 1610 observed reflections. Molecular geometry is normal. The thiophene ring is planar, and is coplanar with the 3*H*-pyrimidinone ring [dihedral angle = 0·7 (1)°]. The fused cyclohexene ring adopts a half-chair conformation. The C—S distances are 1·726 (3) and 1·738 (3) Å. The C—S—C bond angle is 91·6 (1)°.

Experimental. 2-Aminothiophenes were prepared by adopting the following procedure: a cycloalkanone was allowed to undergo a Cope reaction (Cope, Hofmann, Wyckoff & Hardenbergh, 1941), followed by a Gewald reaction in the presence of sulfur, to form substituted cycloalka[*b*]thiophenes (Gewald, 1962, 1965; Gewald, Schinke & Bottcher, 1966). 2-Amino-3-(ethoxycarbonyl)-4,5,6,7-tetrahydrocyclohexa[*b*]thiophene was dissolved in formamide. The mixture was boiled under reflux at 473 K for 18 h. On cooling to room temperature a crystalline precipitate was obtained, which was filtered, washed with 2-propanol, and recrystallized from chloroform–methanol–propanol. The 4-oxo-5,6,7,8-tetrahydro-3*H*-cyclohexathieno[2,3-*d*]pyrimidine was obtained as light brown needles.

A specimen of dimensions 0·29 × 0·20 × 0·25 mm was chosen. Lattice parameters and their e.s.d.'s derived from a least-squares treatment of 25 reflections ($25 < \theta < 35°$). Intensity data were collected on an Enraf–Nonius CAD-4 single-crystal X-ray diffractometer, graphite-crystal-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode for h $0\rightarrow8$, k $-8\rightarrow14$, l $-12\rightarrow12$, $2\theta_{\max} = 140°$. Two standard reflections monitored every 100 measurements varied less than 2%. Of the 2052 independent reflections collected 1610 were judged significant [$I > 3\sigma(I)$]. $R_{\mathrm{int}} = 0{\cdot}01$. Intensities were corrected for Lp effects, absorption corrections were ignored. Structure solved by Patterson methods using *SHELXS*86 (Sheldrick, 1986). Positional and thermal parameters of non-hydrogen

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms, with e.s.d.'s in parentheses*

$$B_{\mathrm{eq}} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
S(1)	0·1758 (1)	0·1089 (1)	0·2659 (1)	4·0
C(2)	−0·0235 (3)	0·0500 (2)	0·3105 (2)	3·0 (1)
N(3)	−0·0859 (3)	−0·0437 (2)	0·2652 (2)	3·5
C(4)	−0·2348 (4)	−0·0757 (2)	0·3106 (2)	3·4 (1)
N(5)	−0·3282 (3)	−0·0235 (2)	0·3939 (2)	2·5
C(6)	−0·2721 (3)	0·0725 (2)	0·4428 (2)	3·0 (1)
C(7)	−0·1053 (3)	0·1105 (2)	0·3956 (2)	2·8 (1)
C(8)	−0·0040 (3)	0·2048 (2)	0·4257 (2)	3·0 (1)
C(9)	−0·0500 (4)	0·2849 (2)	0·5174 (2)	3·7 (1)
C(10)	0·1209 (6)	0·3561 (3)	0·5599 (4)	5·3 (1)
C(11)	0·2126 (6)	0·3894 (2)	0·4459 (4)	5·4 (1)
C(12)	0·2906 (5)	0·3002 (2)	0·3746 (3)	4·7 (1)
C(13)	0·1498 (4)	0·2138 (2)	0·3625 (2)	3·5 (1)
O(14)	−0·3629 (3)	0·1144 (1)	0·5177 (2)	3·8

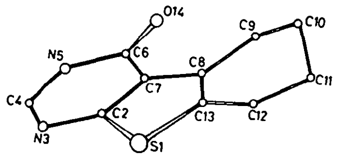

Fig. 1. Perspective view of the molecule showing atomic nomenclature.

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Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°), *with e.s.d.'s in parentheses*

S(1)—C(2)	1·726 (3)	C(6)—O(14)	1·223 (3)
S(1)—C(13)	1·738 (3)	C(7)—C(8)	1·435 (3)
C(2)—N(3)	1·365 (4)	C(8)—C(9)	1·496 (3)
C(2)—C(7)	1·389 (3)	C(8)—C(13)	1·360 (4)
N(3)—C(4)	1·287 (4)	C(9)—C(10)	1·512 (5)
C(4)—N(5)	1·362 (3)	C(10)—C(11)	1·509 (6)
N(5)—C(6)	1·394 (4)	C(11)—C(12)	1·543 (5)
C(6)—C(7)	1·433 (3)	C(12)—C(13)	1·493 (4)

C(2)—S(1)—C(13)	91·6 (1)	C(6)—C(7)—C(8)	128·7 (2)
S(1)—C(2)—N(3)	122·4 (2)	C(9)—C(8)—C(13)	121·7 (2)
S(1)—C(2)—C(7)	111·0 (2)	C(7)—C(8)—C(13)	111·6 (2)
N(3)—C(2)—C(7)	126·7 (2)	C(7)—C(8)—C(9)	126·6 (2)
C(2)—N(3)—C(4)	113·4 (2)	C(8)—C(9)—C(10)	111·9 (2)
N(3)—C(4)—N(5)	125·3 (3)	C(9)—C(10)—C(11)	112·3 (3)
C(4)—N(5)—C(6)	123·9 (2)	C(10)—C(11)—C(12)	113·0 (3)
N(5)—C(6)—C(7)	112·5 (2)	C(11)—C(12)—C(13)	109·9 (3)
N(5)—C(6)—O(14)	120·0 (2)	C(12)—C(13)—C(8)	126·1 (2)
C(7)—C(6)—O(14)	127·4 (2)	C(12)—C(13)—S(1)	121·3 (2)
C(6)—C(7)—C(2)	118·1 (2)	C(8)—C(13)—S(1)	112·6 (2)
C(8)—C(7)—C(2)	113·2 (2)		

C(8)—C(9)—C(10)—C(11)	45·2 (4)	C(11)—C(12)—C(13)—C(8)	−12·1 (4)
C(9)—C(10)—C(11)—C(12)	−59·3 (4)	C(13)—C(8)—C(9)—C(10)	−16·7 (4)
C(10)—C(11)—C(12)—C(13)	40·5 (4)	C(12)—C(13)—C(8)—C(9)	−0·1 (5)

atoms refined by full-matrix least-squares refinement using *SHELX76* (Sheldrick, 1976). The hydrogen atoms, located from a difference Fourier map, were refined isotropically. At the final stage of refinement, weights were introduced resulting in $R = 0{\cdot}06$, $wR = 0{\cdot}07$. $w = 1/(\sigma^2|F_o| + 0{\cdot}00219|F_o|^2)$, $S = 1{\cdot}9$. Shift/e.s.d. < 0·1. Final difference Fourier map was featureless with $\Delta\rho$ within ± 16 e Å^{-3}. The atomic scattering factors used for all the atoms were as provided in the *SHELX76* program. Computer programs: *PARST* (Nardelli, 1983) for geometrical calculations. Table 1 lists the final atomic coordinates and equivalent isotropic thermal parameters of non-hydrogen atoms.* The numbering scheme and molecular connectivity are given in the perspective view of Fig. 1. The bond lengths, bond angles and selected torsion angles are shown in Table 2.

Related literature. Biological activities of 2-aminothiophene and thieno[2,3-*d*]pyrimidine have attracted the attention of medicinal chemists (Nakanishi, Imamura & Maruyama, 1970; Wellings, 1972; Manhas, Sharma & Amin, 1972).

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* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom coordinates, bond lengths and angles involving hydrogen atoms, torsion angles and least-squares-planes equations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52134 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,4,7,10-Tetraazacyclododecane Tetrahydrochloride

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Abstract. $[C_8H_{24}N_4]Cl_4$, $M_r = 318{\cdot}1$, orthorhombic, *Pbcn*, $a = 13{\cdot}788$ (3), $b = 9{\cdot}511$ (2), $c = 10{\cdot}643$ (2) Å, $V = 1395{\cdot}7$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}51$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 8{\cdot}35$ cm^{-1}, $F(000) = 672$, $T = 150$ K, $R = 0{\cdot}033$, $wR = 0{\cdot}045$ for 1136 unique observed reflections. The cations exhibit crystallographic twofold rotation symmetry and [3333] quadrangular conformation, with protonated N atoms occupying corner positions. Chloride counterions connect adjacent cations through hydrogen bonding.

Experimental. The title compound (also known as cyclen tetrahydrochloride, $[H_4cyclen]Cl_4$) was obtained by a literature method (Hay & Norman, 1979),

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