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5-Methyl-2,8-dithia-1,3,7-triphosphatricyclo[3.3.1.0^{3,7}]nonane 1-Sulfide

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Abstract. $C_5H_9P_3S_3$, $M_r = 258{\cdot}23$, monoclinic, $A2/a$, $a = 12{\cdot}027\,(5)$, $b = 7{\cdot}785\,(1)$, $c = 22{\cdot}762\,(14)$ Å, $\beta = 102{\cdot}44\,(7)°$, $V = 2081\,(3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}65$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}70926$ Å, $\mu = 10{\cdot}81$ cm^{-1}, $F(000) = 1056$, $T = 295\,(2)$ K, $R = 0{\cdot}025$ for 1843 unique reflections. The molecule consists of a folded five-membered P_3S_2 ring with one exocyclic S atom; the ring is capped by the organic group $CH_3C(CH_2)_3$ which is bonded to the P atoms. A short intermolecular P···P distance of 3·033 (1) Å is found in the crystal structure.

Introduction. The title compound $CH_3C(CH_2P)_3S_2(S)$ was prepared by Ellermann, Demuth & Bauer (1986). These authors postulated a five-membered P_3S_2 ring with an exocylic S atom by spectroscopic methods. The X-ray structure of the analogous arsenic compound $CH_3C(CH_2As)_3S_3$ (Ellermann, Lietz, Merbach, Thiele & Zoubek, 1979) has a six-membered As_3S_3 ring. An X-ray analysis has been performed to confirm the proposed unusual structure of $CH_3C(CH_2P)_3S_2(S)$.

Experimental. Recrystallization from tetrahydrofuran gave colourless crystals; spherical crystal with $r = 0{\cdot}18\,(2)$ mm used for X-ray analysis; Philips PW 1100 diffractometer (graphite monochromator, Mo $K\alpha$ radiation); ω–2θ scan mode, scan width $(1{\cdot}000 + 0{\cdot}345\tan\theta)°$, profile analysis with a modified Lehmann & Larsen (1974) procedure; lattice parameters by least-squares refinement (*GIVER*: Krogmann, 1966) on the basis of 48 reflections with $21 \leq 2\theta \leq 29°$; spherical absorption correction applied with $\mu R = 0{\cdot}20\,(2)$, absorption correction factor $A = 1{\cdot}33\,(3)$; index range h −14/13, k 0/9, l 0/26; six standard reflections (intensity variation 0·3%); 7362 reflections measured, $(\sin\theta_{\max})/\lambda = 0{\cdot}60$ Å^{-1}, after averaging symmetrical equivalents with $I > 3\sigma(I)$ 1843 reflections remained, $R_{\text{int}} = 0{\cdot}01$. Structure solved by direct methods, subsequent Fourier map revealed all C atoms and all H atoms could be located from a difference Fourier map; final refinement by blocked least-squares methods (based on F) of scale factor, coordinates and anisotropic temperature parameters (H atoms isotropic). $R = 0{\cdot}025$, $wR = 0{\cdot}022$, $w = 1/\sigma^2(F_o)$, $S =$

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4·44, max. $\Delta/\sigma = 0{\cdot}04$, $\Delta\rho$ in the final difference Fourier synthesis +0·11 and −0·12 e Å^{-3}; scattering factors from *International Tables for X-ray Crystallography* (1974). Computations employed the *CRYSTAN* system (Burzlaff, Böhme & Gomm, 1977) and *ORTEP*II (Johnson, 1977) for plotting purposes.

Discussion. Coordinates and equivalent isotropic temperature factors are reported in Table 1.* Bond lengths and angles are listed in Table 2.

As shown in Fig. 1, the molecule consists of a folded five-membered P_3S_2 ring with one exocyclic S atom. The ring is capped by the organic group $CH_3C(CH_2)_3$ which bonds to the three P atoms.

The P(2)—P(3) distance of 2·281 (1) Å is slightly longer than the equivalent bonds in 4-methyl-1,2,6-triphosphatricyclo[2.2.1.0^{2,6}]heptane, $C_5H_9P_3$ [2·200 (1) Å] (Zimmermann, Gomm, Köck & Ellermann, 1986), but it is in the range of values commonly found for this type of cyclophosphathiane compound, *e.g.* P_4S_5, P—P: 2·25 Å; P_4S_7, P—P: 2·326 (7) Å (Vos, Olthof, van Bolhuis & Botterweg, 1965). The P(1)—C(1) bond is short at 1·814 (2) Å, whereas the other two [P(2)—C(2): 1·847 (2), P(3)—C(3): 1·849 (2) Å] have values which are comparable with the distance in $C_5H_9P_3$ [1·851 (2) Å]. The relevant C—C bonds show the opposite behaviour [C(1)—C(4): 1·545 (2), C(2)—C(4): 1·527 (2), C(3)—C(4): 1·526 (3) Å].

For a P—S single bond a distance of 2·14 Å is predicted from covalent radii (Pauling, 1973). The values obtained [2·10 Å (mean)] are insignificantly shorter and agree with bond lengths in similar structures (Vos *et al.*, 1965; Lensch, Clegg & Sheldrick, 1984). The P=S bond [P(1)=S(1): 1·931 (1) Å] is also comparable with literature data (Vos *et al.*, 1965; Lensch *et al.*, 1984).

The angles at the C atoms are close to tetrahedral, with the exception of C(1)—C(4)—C(5): 106·9 (1), C(2)—C(4)—C(3): 106·5 (1) and P(1)—C(1)—C(4): 120·7 (1)°. As expected, the C—P—P angles are close to 90°, corresponding to the values in $C_5H_9P_3$ (Zimmermann *et al.*, 1986). The P—S—P angles [P(1)—S(2)—P(2): 97·27 (2), P(1)—S(3)—P(3): 98·25 (2)°] are less than those observed in similar molecules, *e.g.* $(PhP)_3S_3$ 107·4 (1), 108·4 (1)° (Lensch *et al.*, 1984) or P_4S_7 102·4° (mean value) (Vos *et al.*, 1965).

In the crystal, pairs of molecules are arranged about the inversion centres $(000, \frac{1}{2}00, 0\frac{1}{2}\frac{1}{2}, \frac{1}{2}\frac{1}{2}\frac{1}{2})$ so that symmetry-related P(2) atoms are separated by only 3·033 (1) Å. This intermolecular P(2)···P(2) distance is only about 0·75 Å longer than the intramolecular

Table 1. *Atom coordinates* (×10^4) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å^2 × 10^2)

$$U_{eq} = (1/6\pi^2)\textstyle\sum_i\sum_j\beta_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}
S(1)	−193 (1)	−948 (1)	1918 (1)	5·8
S(2)	−1259 (1)	1950 (1)	850 (1)	4·2
S(3)	1015 (1)	−70 (1)	740 (1)	4·0
P(1)	173 (1)	807 (1)	1391 (1)	3·4
P(2)	−389 (1)	3778 (1)	434 (1)	3·7
P(3)	1200 (1)	2322 (1)	324 (1)	3·6
C(1)	1059 (1)	2511 (2)	1785 (1)	4·0
C(2)	372 (1)	5007 (2)	1093 (1)	4·0
C(3)	2083 (1)	3474 (2)	970 (1)	4·0
C(4)	1418 (1)	4043 (2)	1437 (1)	3·6
C(5)	2154 (2)	5228 (2)	1904 (1)	5·6

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

E.s.d.'s are given in parentheses.

P(1)—S(1)	1·931 (1)	C(1)—C(4)	1·545 (2)
P(1)—S(2)	2·087 (1)	C(2)—C(4)	1·527 (2)
P(1)—S(3)	2·082 (1)	C(3)—C(4)	1·526 (3)
P(1)—C(1)	1·814 (2)	C(4)—C(5)	1·537 (2)
P(2)—S(2)	2·108 (1)	C—H	0·93–1·02 (2)
P(2)—P(3)	2·281 (1)		
P(2)—C(2)	1·847 (2)		
P(3)—S(3)	2·122 (1)		
P(3)—C(3)	1·849 (2)		

S(1)···S(1^{i})	3·540 (1)	P(2)···P(3^{iii})	3·525 (1)
S(2)···S(3^{ii})	3·550 (1)	P(3)···P(3^{iv})	3·734 (1)
P(2)···P(2^{iii})	3·033 (1)	S(3)···P(2^{iii})	3·895 (1)

S(1)—P(1)—S(2)	113·50 (3)	S(3)—P(3)—P(2)	102·49 (2)
S(1)—P(1)—S(3)	114·94 (3)	S(3)—P(3)—C(3)	99·69 (4)
S(2)—P(1)—S(3)	100·73 (2)	P(2)—P(3)—C(3)	91·32 (3)
S(1)—P(1)—C(1)	113·48 (6)	P(1)—C(1)—C(4)	120·7 (1)
S(2)—P(1)—C(1)	107·59 (4)	P(2)—C(2)—C(4)	111·8 (1)
S(3)—P(1)—C(1)	105·50 (5)	P(3)—C(3)—C(4)	113·4 (1)
P(1)—S(2)—P(2)	97·27 (2)	C(1)—C(4)—C(2)	110·6 (1)
P(1)—S(3)—P(3)	98·25 (2)	C(1)—C(4)—C(3)	112·3 (1)
S(2)—P(2)—P(3)	102·87 (2)	C(2)—C(4)—C(3)	106·5 (1)
S(2)—P(2)—C(2)	101·06 (4)	C(1)—C(4)—C(5)	106·9 (1)
P(3)—P(2)—C(2)	93·78 (3)	C(2)—C(4)—C(5)	110·4 (1)
		C(3)—C(4)—C(5)	110·3 (1)

Symmetry code: (i) $-x, \frac{1}{2}-y, \frac{1}{2}-z$; (ii) $\frac{1}{2}+x, -y, z$; (iii) $-x, -y, -z$; (iv) $\frac{1}{2}-x, y, -z$.

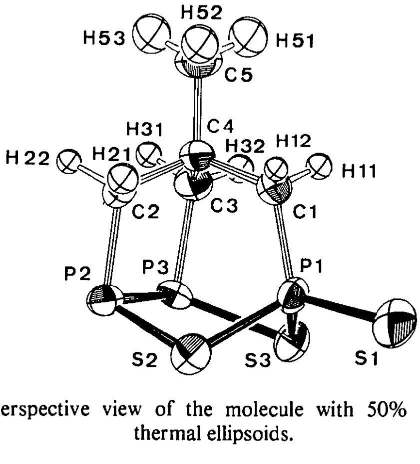

Fig. 1. Perspective view of the molecule with 50% probability thermal ellipsoids.

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and H-atom geometries have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44297 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

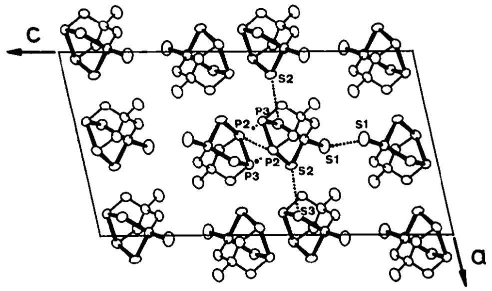

Fig. 2. Projection of the crystal structure along the *b* axis. H atoms have been removed for clarity.

P(2)—P(3) bond. The shortest intermolecular P···P distance in $C_5H_9P_3$ [4·013 (1) Å] is almost 1 Å longer.

The 'dimers' are held together by P(2)···P(3) [3·525 (1) Å] and P(2)···S(3) [3·895 (1) Å] interactions forming columns along **b**. Across the inversion centres ($0\frac{1}{4}\frac{1}{4}$, $\frac{1}{2}\frac{1}{4}\frac{1}{4}$, $0\frac{3}{4}\frac{3}{4}$, $\frac{1}{2}\frac{3}{4}\frac{3}{4}$) these columns are linked by S(1)···S(1) contacts [3·540 (1) Å] along **c**. Short contacts along **a** are exhibited by S(2)···S(3) 3·550 (1) and P(3)···P(3) 3·734 (1) Å. A projection of the unit cell along **b** is illustrated in Fig. 2.

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Trimethoprim Acetate

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Abstract. 2,4-Diamino-5-(3,4,5-trimethoxybenzyl)-1*H*+-pyrimidinium acetate, $C_{14}H_{19}N_4O_3^+.C_2H_3O_2^-$, $M_r = 350{\cdot}38$, monoclinic, $P2_1/c$, $a = 11{\cdot}085\ (4)$, $b = 19{\cdot}137\ (6)$, $c = 9{\cdot}641\ (3)$ Å, $\beta = 122{\cdot}73\ (3)°$, $U = 1720{\cdot}5\ (10)$ Å^3, $Z = 4$, $D_x = 1{\cdot}353\ (1)$, $D_m = 1{\cdot}36\ (1)$ g cm^{-3} (flotation in aqueous KI), $F(000) = 744$, $\mu(\text{Cu}\ K\alpha) = 6{\cdot}4$ cm^{-1}, $\lambda = 1{\cdot}5418$ Å, $T = 293$ K. The structure was solved by the symbolic addition method. Refinement by least squares gave $R = 0{\cdot}0399$ for 1997 independent significant reflections, 0·0438 for all 2337 independent reflections measured. The molecular conformation is close to that in neutral trimethoprim, with the pyrimidine and phenyl rings inclined to the plane C(5)—C(7)—C(1′) at −77·5 and 157·2°, respectively. The pyrimidine ring is protonated at N(1), and molecules are linked in centrosymmetric pairs by hydrogen bonds between N(3) and N(4), and to the acetate groups by hydrogen bonds involving N(1) and N(2). Weaker hydrogen bonds are also present between the amino N atoms and the methoxy O atoms of two neighboring molecules.

Introduction. Trimethoprim (I) is a potent inhibitor of bacterial dihydrofolic acid reductase, but is less effective against mammalian, avian and viral forms of

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