

(Sheldrick, 1986), full-matrix least-squares refinement on F of 364 parameters using *SHELX76* (Sheldrick, 1976) on a VAX11/750 computer. Anisotropic thermal parameters for non-H atoms, H atoms allowed to ride at fixed distance on C and N atoms, refined isotropically. $R = 0.060$, $wR = 0.062$, S was not calculated, $(\Delta/\sigma)_{\max} = 0.001$, $(\Delta\rho)_{\max} = 0.426$, $(\Delta\rho)_{\min} = -0.386 \text{ e } \text{Å}^{-3}$, unit weights; atomic scattering factors as incorporated in *SHELX76*. Table 1* lists atomic positional and equivalent isotropic thermal parameters. Interatomic distances and valence angles are in Table 2. Fig. 1 shows a perspective view of the molecule and Fig. 2 shows the crystal packing.

Related literature. The title compound can be regarded as a heterosilazane analog of 1,3,5-trimethyl-2,2,4,4,6,6-hexakis(methylamino)cyclotri-

* Lists of orthogonal coordinates, structure factors, anisotropic displacement parameters, H-atom parameters and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55415 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0583]

silazane (Andersch & Jansen, 1990a) in which one methylamino group is substituted by an O atom with the six-membered ring being planar. A related siloxazane with respect to the Si₃N₂O core has been reported: 2,2,4,4,6,6-hexamethylcyclotrisiloxazane (Andrianov, Haiduc & Khananashvili, 1963a,b) has been synthesized by co-ammonolysis of 1,3-dichlorotetramethyldisiloxane. An alternative synthesis is by a substitution reaction in the six-membered ring (Wannagat, Bodgusch & Höfler, 1967).

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Structure of [(4,6-Dimethyl-2-pyrimidinyl)thio]acetic Acid Hydrate

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Abstract. C₈H₁₀N₂O₂S.H₂O, $M_r = 216.26$, monoclinic, $P2_1/c$, $a = 7.652(1)$, $b = 10.741(1)$, $c = 13.267(1) \text{ Å}$, $\beta = 100.80(1)^\circ$, $V = 1071.7 \text{ Å}^3$, $Z = 4$, $D_x = 1.341 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ Å}$, $\mu = 2.739 \text{ cm}^{-1}$, $F(000) = 456$, $T = 293 \text{ K}$, final $R = 0.047$ for 1569 unique observed [$I > 3\sigma(I)$] reflections. The S—C bond lengths are 1.758 (3) and 1.789 (3) Å. The thiopyrimidine ring is planar. The crystal cohesion is due to hydrogen bonds involving water molecules.

Experimental. A colorless needle-like crystal (0.15 × 0.22 × 0.59 mm) of the title compound was synthe-

sized by a reported method (Boarland, McOmie & Timms, 1952) and recrystallized from H₂O. Reflection data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. Cell dimensions were refined by 25 accurately centered reflections in the range $22 < 2\theta < 32^\circ$. Intensities were measured using an ω – 2θ scan of 0.79 to $8.24^\circ \text{ min}^{-1}$; scan width $(0.45 + 0.35 \tan \theta)^\circ$; θ range 1 – 25° ($h = 0$ to 9 , $k = 0$ to 13 , $l = -16$ to 16). Three standard reflections monitored periodically showed no significant variation in intensity. Of the 2000 independent reflections measured ($R_{\text{int}} = 0.018$), 1569 with $I > 3\sigma(I)$ were used for the calculations. L_p and ψ -scan absorption corrections

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Table 1. Final positional and equivalent isotropic thermal parameters (\AA^2) of non-H atoms with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
S	0.8180 (1)	0.18138 (8)	0.34667 (6)	4.52 (2)
O(1)	0.6504 (3)	0.3943 (3)	0.4492 (2)	6.47 (7)
O(2)	0.8796 (3)	0.4128 (2)	0.5770 (2)	5.38 (6)
O(W)	0.6902 (3)	0.5683 (2)	0.6617 (2)	5.12 (5)
N(1)	0.7041 (3)	-0.0407 (2)	0.3774 (2)	3.69 (5)
N(2)	0.7947 (3)	0.0829 (2)	0.5281 (2)	3.84 (5)
C(1)	0.7667 (4)	0.0629 (3)	0.4278 (2)	3.49 (6)
C(2)	0.7516 (4)	-0.0117 (3)	0.5852 (2)	4.19 (7)
C(3)	0.6828 (4)	-0.1208 (3)	0.5409 (2)	4.39 (7)
C(4)	0.6607 (4)	-0.1341 (3)	0.4356 (2)	4.03 (7)
C(5)	0.7844 (6)	0.0102 (4)	0.6986 (3)	5.86 (9)
C(6)	0.5941 (5)	-0.2512 (3)	0.3818 (3)	5.67 (9)
C(7)	0.9235 (4)	0.2929 (3)	0.4384 (3)	4.28 (7)
C(8)	0.8012 (4)	0.3710 (3)	0.4879 (3)	4.06 (7)

Table 2. Bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

S—C(1)	1.758 (3)	N(2)—C(2)	1.345 (4)	
S—C(7)	1.789 (3)	C(2)—C(3)	1.372 (5)	
O(1)—C(8)	1.199 (4)	C(2)—C(5)	1.496 (4)	
O(2)—C(8)	1.302 (4)	C(3)—C(4)	1.382 (4)	
N(1)—C(1)	1.340 (4)	C(4)—C(6)	1.489 (5)	
N(1)—C(4)	1.346 (4)	C(7)—C(8)	1.496 (5)	
N(2)—C(1)	1.324 (4)			
C(1)—S—C(7)	101.1 (1)	C(2)—C(3)—C(4)	119.1 (3)	
C(1)—N(1)—C(4)	115.9 (3)	N(1)—C(4)—C(3)	120.4 (3)	
C(1)—N(2)—C(2)	115.6 (3)	N(1)—C(4)—C(6)	117.0 (3)	
S—C(1)—N(1)	113.3 (2)	C(3)—C(4)—C(6)	122.6 (3)	
S—C(1)—N(2)	119.0 (2)	S—C(7)—C(8)	115.7 (2)	
N(1)—C(1)—N(2)	127.7 (3)	O(1)—C(8)—O(2)	124.0 (3)	
N(2)—C(2)—C(3)	121.3 (3)	O(1)—C(8)—C(7)	124.1 (3)	
N(2)—C(2)—C(5)	115.8 (3)	O(2)—C(8)—C(7)	111.9 (3)	
C(3)—C(2)—C(5)	123.0 (3)			
D—H...A	D...A	D—H	H...A	D—H...A
O(2)—H(O2)...O(W)	2.600	1.044	1.565	170.6
O(W)—H(OW)...O(1 ⁱⁱ)	2.771	1.163	1.624	167.4
O(W)—H(OW)...N(1 ⁱⁱⁱ)	2.860	0.906	1.964	169.5

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

were applied (min. and max. transmission factors 0.9358 and 0.9986, respectively).

The structure was solved by direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and completed by difference Fourier methods. Full-matrix least-squares refinement on F included anisotropic thermal parameters for non-H atoms and minimized the function $\sum w(|F_o| - |F_c|)^2$, $w = 1$. Positions of H atoms localized on a difference Fourier map and included in structure-factor calculations but not refined. Final refinements converged with $R = 0.047$, $wR = 0.045$, $S = 0.722$ and $(\Delta/\sigma)_{\text{max}} = 0.27$. A final difference Fourier map revealed a maximum peak density of $< 0.26 \text{ e \AA}^{-3}$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were carried out on a PDP 11/44 computer using the *SDP* package. Final

positional parameters are given in Table 1* with bond lengths, angles and hydrogen-bonding data in Table 2. Fig. 1. shows the title molecule and its molecular packing is represented in Fig. 2.

Related literature. The title compound has been studied by a mass spectrometric method (Shpileva, Tolmachev, Fedotov & Romanov, 1983). The structures of the ligand 4,6-dimethyl-2-pyrimidinethiol as

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and molecular geometry calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55447 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

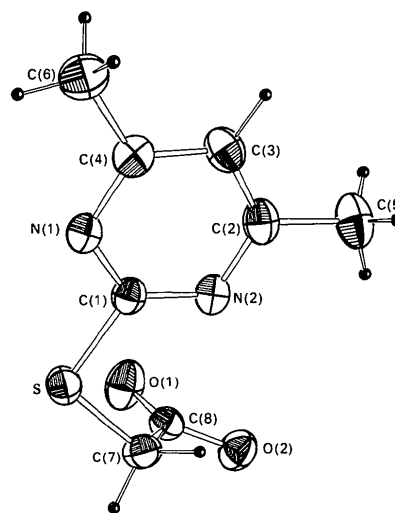


Fig. 1. View of the title molecule showing the atomic numbering scheme (ORTEP; Johnson, 1976).

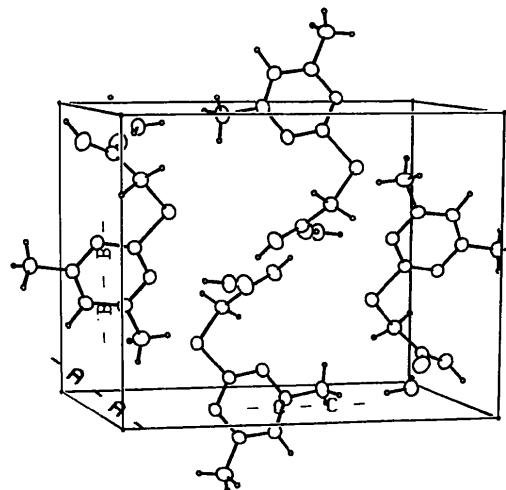


Fig. 2. Packing diagram for the title molecule.

chelate (Cartwright, Langguth & Skapski 1979) and of 2,4-dithiouracil (Shefter & Mautner, 1967) have also been studied.

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