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Tetrasulphurtetraimide: a Refinement

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Abstract. $S_4N_4H_4$, orthorhombic, *Pnma*, $a = 7{\cdot}988$ (2), $b = 12{\cdot}244$ (3), $c = 6{\cdot}743$ (2) Å, $V = 659{\cdot}5$ Å^3, $D_m = 1{\cdot}89$, $D_c = 1{\cdot}896$ g cm^{-3}, $Z = 4$, $F(000) = 348{\cdot}0$, $\mu(\text{Mo } K\alpha) = 12{\cdot}35$ cm^{-1}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å. The structure has been refined by full-matrix least-squares methods to an R of 0·034 for 867 intensities measured on a diffractometer. Our results confirm the earlier structure and give interatomic distances and angles with greater accuracy.

Introduction. The structure of $S_4N_4H_4$ was described by Lund & Svendsen (1957) and Sass & Donohue (1958). While the former workers did not attempt to locate the H atoms, the latter postulated their positions based on packing considerations but failed to locate them on a difference map. The lowest R was 0·118 indicating scope for significant improvement. We became interested in $S_4N_4H_4$ because it has been found to form metal complexes (Nabi, Hursthouse & Malik, 1977). Samples of $S_4N_4H_4$ used in our study were recrystallized from acetonitrile. They were colourless thin plates elongated along [101]. Cell parameters were determined by least-squares refinement of 15 setting angles measured on a diffractometer. The intensities ($1{\cdot}5 < \theta < 35°$) were collected from a crystal 0·30 × 0·20 × 0·07 mm on a Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and an ω–2θ scan. The reflexions of two different octants (hkl and $h\bar{k}l$) of the reciprocal sphere were measured. The intensities were corrected for $(Lp)^{-1}$ factors, reduced to F_o values and merged to obtain 1699 unique reflexions (internal consistency, $R = 0{\cdot}0148$). 867 obeyed the condition $I > 1{\cdot}5\sigma(I)$ and were used in the final refinement. Absorption effects were ignored.

The final positional parameters of the S and N atoms listed by Sass & Donohue (1958) and a U_{iso} of 0·04 Å^2 were taken as the starting parameters. Isotropic and anisotropic least-squares refinement reduced R to 0·10 and 0·043 respectively. The H atoms were located on a difference map and included in the refinement with isotropic temperature factors to give a final R of 0·034.* In the final cycle, the largest shift/e.s.d. was less than 0·02. Unit weights were used and gave a flat analysis of variance. Scattering factors were those of Stewart, Davidson & Simpson (1965) for H and Cromer & Mann (1968) for N and S. The final positional parameters are given in Table 1. All calculations were done on the Queen Mary College ICL 1904S computer with programs written by G. M. Sheldrick and W. D. S. Motherwell (University of Cambridge).

Table 1. *Final atomic fractional coordinates* (S *and* N × 10^5, H × 10^4)

	x	y	z
S(1)	4217 (10)	12986 (6)	18103 (11)
S(2)	13701 (8)	12997 (5)	−24203 (10)
N(1)	23038 (39)	25000*	−28159 (50)
N(2)	10609 (57)	25000*	26798 (58)
N(3)	16496 (34)	9293 (19)	−712 (37)
H(1)	3368 (56)	2500*	−2883 (66)
H(2)	1805 (58)	2500*	3262 (68)
H(3)	2383 (37)	589 (24)	142 (45)

* Held invariant because of space-group requirements.

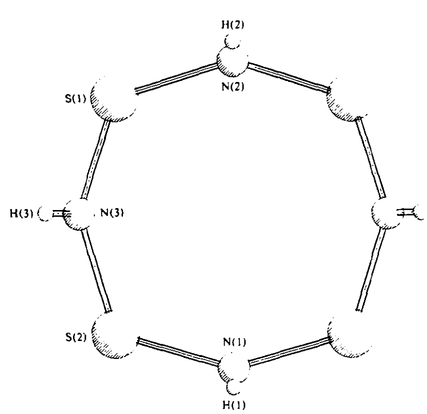

Fig. 1. Molecular structure of $S_4N_4H_4$ showing the atom labelling.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32593 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

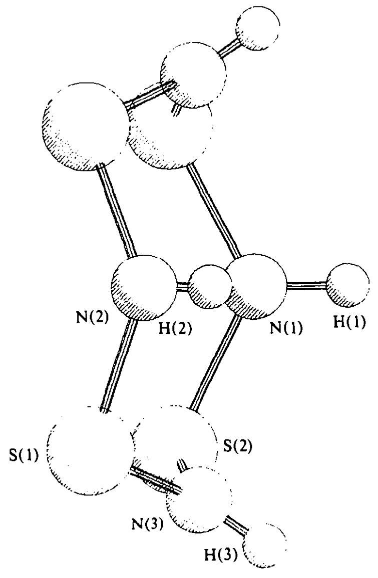

Fig. 2. A view of the molecule looking down [001].

Discussion. The structure of a single molecule and the atom labelling are shown in Fig. 1. An alternative view of the molecule is shown in Fig. 2. The molecular dimensions are listed in Table 2. Our study confirms the earlier analysis. Of the three independent H atoms, only H(2) is favourably positioned to form a weak N(2)—H(2)···N(1) hydrogen bond between adjacent molecules lying along **c**. The interatomic distances and angles in the H bond are N(2)—H(2) = 0·712, N(2)···N(1) = 3·196, H(2)···N(1) = 2·675 Å and N(2)—H(2)···N(1) = 132°.

The individual S—N lengths are equal, within the limits of experimental error, and average 1·665 (1) Å; similarly the N—H lengths [average 0·76 (2) Å] and the S—N—S, N—S—N and S—N—H angles [average, 124·2 (2), 109·3 (1) and 117·1 (10)° respectively] also show insignificant variations.

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

The primed atoms are generated by *m* symmetry.

S(1)–N(2)	1·664 (2)	N(2)–S(1)–N(3)	109·1 (2)
S(1)–N(3)	1·666 (2)	N(1)–S(2)–N(3)	109·4 (1)
S(2)–N(3)	1·663 (3)	S(1)–N(3)–S(2)	124·9 (2)
S(2)–N(1)	1·669 (2)	S(1)–N(2)–S(1′)	124·3 (3)
N(1)–H(1)	0·85 (4)	S(2)–N(1)–S(2′)	123·4 (2)
N(2)–H(2)	0·71 (4)	S(2)–N(1)–H(1)	117·0 (5)
N(3)–H(3)	0·73 (3)	S(1)–N(2)–H(2)	116·8 (6)
S(1)···S(2)	2·952	S(1)–N(3)–H(3)	118·4 (25)
S(1)···S(1′)	2·942	S(2)–N(3)–H(3)	116·7 (25)
S(2)···S(2′)	2·939		
N(1)···N(3)	2·720		
N(2)···N(3)	2·713		

N(3)–S(1)–N(2)–S(1′)	87·6
N(3)–S(1)–N(2)–H(2)	−75·0
N(2)–S(1)–N(3)–S(2)	−86·2
N(2)–S(1)–N(3)–H(3)	92·3
N(3)–S(2)–N(1)–S(2′)	−87·5
N(3)–S(2)–N(1)–H(1)	73·8
N(1)–S(2)–N(3)–S(1)	86·7
N(1)–S(2)–N(3)–H(3)	−91·7

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