in Tables 3 and 4, and Figs. 1 and 2 are plots of the molecules.

Related literature. X-ray structure determinations of compounds containing an SF_5 moiety are rare. The structures of two other compounds, *S*-methyl-(pentafluorosulfanyl)thiocarbamate (Bott, Clark, Thrasher & Atwood, 1987), and the dicobalt complex of a trimer $Co_2(CO)_4(HC_2SF_5)_3$ (Wessel, Hartl & Seppelt, 1986) have been reported. In these compounds the SF_5 geometry is consistent with that reported here including the deviation from planar geometry for the S and F atoms; the average ∠NSF = 91·7 (13)° in the thiocarbamate and ∠CSF ranges from 91·9 to 93·2° in the dicobalt complex.

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Structure of 1,3-Bis(trimethylsilyl)-2,4-bis(trimethylsilylamino)-2,4-dithioxocyclodi-λ^5-phosphazane, $[Me_3SiNHP(S)NSiMe_3]_2$

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Abstract. $[(CH_3)_3SiNHP(S)NSi(CH_3)_3]_2$, M_r = 476·88, monoclinic, $P2_1/n$, a = 13·587 (4), b = 6·634 (2), c = 15·328 (4) Å, β = 99·82 (2)°, V = 1361·4 (7) Å^3, Z = 2, D_m = 1·165 Mg m^{-3}, Mo $K\bar{\alpha}$, λ = 0·71073 Å, μ = 0·44 mm^{-1}, $F(000)$ = 512, T = 295 K, R = 0·036 for 2334 unique observed reflections. The structure is composed of isolated molecules situated on centres of symmetry. The molecules are arranged in layers and held together by weak intermolecular forces as demonstrated macroscopically by a softness and plasticity of the crystals.

Experimental. The compound was prepared according to the method of Dostál, Šikola, Meisel & Grunze (1986) and crystals suitable for X-ray work were obtained by recrystallization from benzene. Density of the crystals was determined by flotation in K_2HgI_4–H_2O mixture. Final lattice parameters were refined from 15 reflections, $18 < 2\theta < 32·5°$, collected on a Syntex $P2_1$ diffractometer. The intensities of a unique set of reflections were measured by θ–2θ scan up to $2\theta = 52°$. Two standard reflections were measured after every 50 measured but no significant variation in their intensities was detected. The intensities were corrected for Lp factor but no absorption correction was applied. The structure was solved by direct methods, H atoms were found from a difference Fourier map, and refined by isotropic block-diagonal least-squares refinement based on $|F_o|$ with weights $1/w = \sigma^2(F_o) + (0·025|F_o|)^2$. Non-H atoms were refined anisotropically. The programs used, together with the atomic scattering factors, were part of the *XTL* program system (Syntex, 1971) and the calculations were perfomed on a Laser PC computer.

Additional experimental details are given in Table 1, the atomic parameters are in Table 2, and selected interatomic distances and angles are presented in Table 3. Fig. 1* shows a perspective view of the molecule.

Related literature. A comprehensive review of P—N ring compounds including diphosphazanes is given by Shaw (1978). The structures of alkyl- and aryl-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53207 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Experimental details*

Crystal dimensions	0·35 × 0·42 × 0·58 mm
Range of *h,k,l*	0→16, 0→8, −18→18
Standard reflections	22$\bar{2}$, 328
Reflections: all	2676
observed ($\|F\| > 6\sigma\|F\|$)	2334
Parameters refined	109
R, *wR* (all)	0·041, 0·058
R, *wR* (observed)	0·036, 0·055
S	1·830
$(\Delta/\sigma)_{max}$ in final cycle	0·1
$(\Delta\rho)_{min}/(\Delta\rho)_{max}$	−0·3/0·3 e Å^{-3}

Table 2. *Final atomic parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{1}{3}[B_{22} + 1/\sin^2\beta(B_{11} + B_{33} + 2B_{13}\cos\beta)]$.

	x	*y*	*z*	B_{eq} (Å^2)
P	−875·0 (3)	560·0 (7)	4961·0 (3)	2·80 (2)
Si(1)	498·0 (4)	1453·0 (8)	6816·0 (3)	3·40 (2)
Si(2)	−2909·0 (4)	−766·0 (9)	5441·0 (4)	3·90 (2)
S	−1503·0 (4)	3093·0 (8)	4578·0 (4)	4·20 (2)
N(1)	−1664 (1)	−1039 (2)	5273 (1)	3·60 (6)
N(2)	200 (1)	631 (2)	5706 (1)	3·20 (6)
C(1)	219 (3)	−581 (4)	7556 (2)	6·5 (1)
C(2)	1844 (2)	2023 (4)	7021 (2)	5·0 (1)
C(3)	−258 (2)	3727 (4)	6934 (2)	5·7 (1)
C(4)	−3745 (2)	132 (6)	4435 (2)	6·8 (1)
C(5)	−2981 (2)	957 (4)	6377 (2)	5·9 (1)
C(6)	−3263 (2)	−3327 (5)	5742 (3)	7·4 (2)

Table 3. *Selected interatomic distances* (Å) *and bond angles* (°)

P—S	1·930 (1)	S—P—N(1)	112·1 (1)
P—N(1)	1·635 (1)	S—P—N(2)	117·8 (1)
P—N(2)	1·695 (1)	S—P—N(2^i)	118·8 (1)
P—N(2^i)	1·684 (1)	N(1)—P—N(2)	110·7 (1)
Si(1)—N(2)	1·766 (1)	N(1)—P—N(2^i)	109·0 (1)
Si(1)—C(1)	1·843 (3)	N(2)—P—N(2^i)	85·8 (1)
Si(1)—C(2)	1·841 (3)	P—N(2)—P^i	94·2 (1)
Si(1)—C(3)	1·850 (3)	P—N(2)—Si(1)	133·4 (1)
Si(2)—N(1)	1·764 (2)	P^i—N(2)—Si(1)	132·2 (1)
Si(2)—C(4)	1·850 (3)	P—N(1)—Si(2)	132·0 (1)
Si(2)—C(5)	1·850 (3)	⟨N(2)—Si(1)—C⟩	108 (1)
Si(2)—C(6)	1·845 (3)	⟨N(1)—Si(2)—C⟩	108 (4)

Symmetry code: (i) $-x, -y, 1-z$.

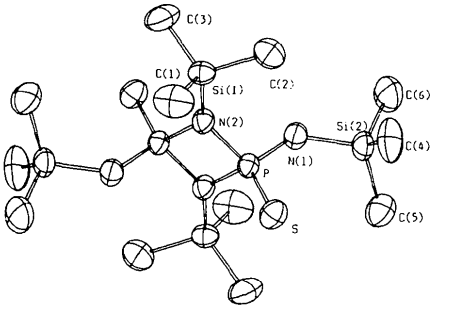

Fig. 1. A perspective view of the molecule.

diphosphazanes are reviewed by Hursthouse, Parkes, Shaw, Shaw & Watkins (1986) and Hursthouse, Ibrahim, Parkes, Shaw, Shaw & Watkins (1986).

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Structure of Ethyl 8-Dimethylamino-1-naphthalenecarboxylate–Picric Acid (1/1)

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Abstract. $C_{15}H_{18}NO_2^+.C_6H_2N_3O_7^-$, $M_r = 472{\cdot}41$, triclinic, $P\bar{1}$, $a = 7{\cdot}843$ (6), $b = 8{\cdot}839$ (2), $c = 15{\cdot}619$ (6) Å, $\alpha = 102{\cdot}93$ (2), $\beta = 92{\cdot}49$ (5), $\gamma = 96{\cdot}94$ (4)°, $V = 1045$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}502$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}0112$ mm^{-1}, $F(000) = 492$, $T = 293$ (1) K, $R = 0{\cdot}048$ for 2518 observed reflections with $I > 3\sigma(I)$. The naphthyl moiety is nearly planar [max. deviation 0·040 (3) Å]. The ethoxycarbonyl group is also planar and its carbonyl O atom is H bonded to the amino H

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