Molecule 1, showing the atom-numbering scheme is in Fig. 1. Final parameters are given in Table 1. Bond lengths and angles are given in Table 2. The ring bonds are shorter than in 5-phenyl-3-isoxazolol (Biagini, Cannas & Marongiu, 1969) and in N-(5-methyl-3-isoxazolyl)sulfanilamide (Bettinetti, Giordano, La Manna, Giuseppetti & Tadini, 1982). Nitro-group parameters are in the range of values found in other compounds. A stereo drawing of the unit cell is shown in Fig. 2. Nitro groups are twisted out of the ring plane by 1·9 and 3·8° for molecule 1, 7·2 and 6·5° for molecule 2, and 12·0 and 8·1° for molecule 3 (e.s.d. ~0·3°). Possible C—H···O hydrogen bonds are given in Table 2. The observed density is lower than the prediction of 1·86 Mg m^{-3} by Stine's (1981) method or 1·92 Mg m^{-3} by Cromer & Ryan (1985).

Related literature. Sutor (1962) discussed the existence of C—H···O hydrogen bonds. Cromer, Coburn, Ryan & Wasserman (1986) note the possibility of this type bond in 1-methyl-2,3,4,5-tetranitropyrrole.

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Dimethyl(thio)[(trimethylsilyl)amino]phosphorane

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Abstract. $C_5H_{16}NPSSi$, $M_r = 181·31$, monoclinic, $P2_1/n$, $a = 6·306$ (1), $b = 10·128$ (6), $c = 16·929$ (6) Å, $\beta = 96·14$ (5)°, $V = 1075·13$ (19) Å^3, $Z = 4$, $D_x = 1·12$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 4·85$ cm^{-1}, $F(000) = 392$, $T = 295$ K. Final $R = 0·064$ for 1016 observed unique reflections. The structure shows that the compound has a distorted trigonal-planar geometry around the central N atom and the N—H and P=S bonds are in trans positions with respect to the N—P bond. The S—P—N—Si and S—P—N—H torsion angles are 176 (3) and −18·5 (6)°. The deviation of the H atom from the P—N—Si plane is 0·23 (5) Å. The bond distances, S=P, P—N, Si—N, av. P—C, and av. Si—C are 1·952 (3), 1·634 (6), 1·741 (7), 1·819 (10), and 1·855 (10) Å, respectively.

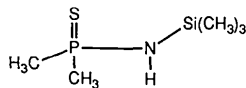**Experimental.** The title compound was synthesized quantitatively by the hydrolysis of $(Me_3Si)_2$-$NP(=S)Me_2$, which is one of the unexpected by-products of the reaction between $(Me_3Si)_2NP(Me)_2$ and $N\equiv SF_3$ (Hosmane & Maldar, 1987, unpublished results). Large well formed colorless crystals of the title compound were grown by sublimation onto a glass surface. A single crystal (0·3 × 0·3 × 0·2 mm) was coated with an epoxy resin and mounted on an Enraf–Nonius CAD-4F diffractometer. The unit-cell parameters were determined by least-squares fit of 15 reflections in the range $10 \le 2\theta \le 24°$, and the space group $P2_1/n$ was assigned on the basis of systematic absences (0k0, k odd, h0l, h + l odd). A total of 2013 independent reflections collected in the range $2 \le 2\theta \le 50°$ (hkl range h 0→7, k 0→11, l −20→20) using graphite-monochromated Mo $K\alpha$ radiation and the $\theta/2\theta$ scan mode, 1016 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity during data collection. Lorentz–polarization corrections applied,

Table 1. Positional and isotropic thermal parameters and their e.s.d.'s

	x	y	z	$B(\text{Å}^2)$
S	1·4751 (4)	0·5511 (2)	0·7338 (2)	4·74 (5)
P	1·6297 (3)	0·7175 (2)	0·7296 (1)	3·24 (4)
Si	1·6240 (4)	0·7501 (2)	0·9108 (1)	3·52 (4)
N1	1·706 (1)	0·7791 (6)	0·8173 (4)	4·0 (1)
C1	1·745 (2)	0·886 (1)	0·9731 (5)	5·8 (2)
C2	1·727 (2)	0·588 (1)	0·9490 (5)	5·4 (2)
C3	1·329 (1)	0·759 (1)	0·9075 (6)	5·8 (2)
C4	1·871 (1)	0·701 (1)	0·6808 (6)	6·5 (2)
C5	·1·467 (2)	0·842 (1)	0·6740 (6)	5·9 (2)
H1A	1·793 (7)	0·861 (5)	0·818 (3)	7 (1)*
H1	0·75 (1)	0·378 (8)	0·483 (5)	5 (2)*
H2	0·65 (2)	0·42 (1)	0·529 (6)	11 (3)*
H4	0·81 (1)	0·076 (8)	0·499 (4)	4 (2)*
H5	0·63 (2)	0·08 (1)	0·556 (6)	8 (3)*
H6	0·83 (1)	0·01 (1)	0·580 (5)	8 (3)*
H7	1·22 (1)	0·197 (7)	0·624 (4)	3 (2)*
H8	1·21 (1)	0·221 (7)	0·553 (4)	4 (2)*
H9	1·24 (2)	0·36 (1)	0·617 (7)	13 (4)*
H10	0·58 (1)	0·263 (8)	0·821 (5)	4 (2)*
H11	0·70 (1)	0·169 (9)	0·859 (5)	10 (2)*
H13	1·03 (2)	0·31 (1)	0·886 (6)	8 (3)*
H14	1·14 (1)	0·35 (1)	0·799 (5)	7 (3)*
H15	0·94 (1)	0·422 (9)	0·833 (5)	7 (2)*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. Bond distances (Å) and angles (°)

Numbers in parentheses are e.s.d.'s in the least-significant digits.

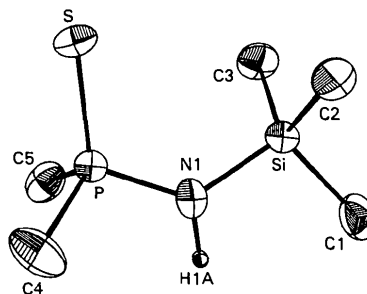|			
S–P	1·952 (3)	Si–C1	1·850 (10)
P–N1	1·634 (6)	Si–C2	1·855 (10)
P–C4	1·814 (10)	Si–C3	1·860 (9)
P–C5	1·825 (10)	N1–H1A	0·99 (5)
Si–N1	1·741 (7)		
S–P–N1	113·3 (3)	N1–Si–C3	111·0 (4)
S–P–C4	112·7 (3)	C1–Si–C2	110·7 (4)
S–P–C5	111·3 (3)	C1–Si–C3	109·3 (5)
N1–P–C4	105·6 (4)	C2–Si–C3	111·5 (5)
N1–P–C5	107·4 (4)	P–N1–Si	132·4 (4)
C4–P–C5	106·2 (5)	P–N1–H1A	116·0 (3)
N1–Si–C1	104·4 (4)	Si–N1–H1A	110·0 (3)
N1–Si–C2	109·8 (4)		

Fig. 1. ORTEP drawing of the molecule. Thermal ellipsoids are scaled to enclose 30% probability.

but no corrections made for absorption or decay. Structure solved by direct-methods program MULTAN11/82 (Frenz, 1982; Hull & Irwin, 1978; Yao, 1981), and refined by full-matrix least squares using the Enraf–Nonius SDP, minimizing $\sum w(|F_o| - |F_c|)^2$; $w = [\sigma(I)^2 + (PI)^2]^{-1/2}$, with $P = 0·07$ chosen to make $\sum w\Delta F^2$ uniformly distributed in $|F_o|$, anisotropic. 14 of the 16 H atoms were located in difference Fourier maps, and refined isotropically; final $R = 0·064$, $wR = 0·071$; $(\Delta/\sigma)_{max} = 0·373$; $\Delta\rho_{max} = 0·519$ e Å^{-3} in final difference Fourier map. Atomic scattering factors from International Tables for X-ray Crystallography (1974).

The final atomic parameters of the non-H atoms and 14 of the 16 H atoms are given in Table 1.* The identification of the atoms and the configuration of the molecule are shown in the ORTEP (Johnson, 1965) drawing of Fig. 1. The bond lengths and bond angles with their standard deviations are given in Table 2.

* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44091 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Several different methods for the preparation of the title compound have been reported (Ullmann & Nöth, 1976; Steinberger & Kuchen, 1974; Wilburn & Neilson, 1977). To date, the structure of this compound has not been determined. Although the structures of compounds containing Si–N–P bonds have been extensively investigated, only a limited number of structural reports were made on the (silylamino)phosphiranes or phosphoranes containing P=S bonds (Caira, Neilson, Watson, Wisian-Neilson & Xie, 1984; Scherer & Jungmann, 1979; Pohl, 1976; Engelhardt & Metter, 1980).

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A Second Modification of Hexaethyl Cyclopropanehexacarboxylate

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Abstract. $C_{21}H_{30}O_{12}$, $M_r = 474.48$, monoclinic, $P2_1$, $a = 8.902$ (1), $b = 9.496$ (1), $c = 14.736$ (1) Å, $\beta = 92.46$ (1)°, $V = 1244.5$ Å^3, $Z = 2$, $D_x = 1.27$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1.5418$ Å, $\mu = 8$ cm^{-1}, $F(000) = 504$, $T = 293$ K. The structure was refined to $R = 0.064$ for 3521 unique observed reflections. The terminal ethyl groups display very high thermal motion, which makes the bond-length precision unreliable. Compared with the orthorhombic modification [Schrumpf, Jones & Sheldrick (1987). *Acta Cryst.* C43, 1758–1760], the following differences are observed: (i) the density of the monoclinic form is 0.04 g cm^{-3} higher; (ii) the mean ring bond length is 1.507 Å monoclinic, 1.516 Å orthorhombic (bond-length e.s.d.'s 0.005, 0.004 Å respectively); (iii) the torsion angle C(11)—O(12)—C(12)—C(13) is −104°, the absolute value being much lower than the other chemically equivalent torsion angles in the monoclinic (137–170°) or orthorhombic (146–162°) form (torsion angle e.s.d.'s <1°).

Experimental. We recently reported the crystal structure of an orthorhombic (*Pccn*) form of the title compound, and the existence of a second, monoclinic form (Schrumpf, Jones & Sheldrick, 1987). At the time, we were unable to obtain good single crystals of the monoclinic form. We have now obtained good crystals (from acetic acid/water) as a by-product of a reaction intended to produce diethyl 2,2,3,3-tetramethylcyclopropane-1,1-dicarboxylate. We are currently studying this unexpected reaction further.
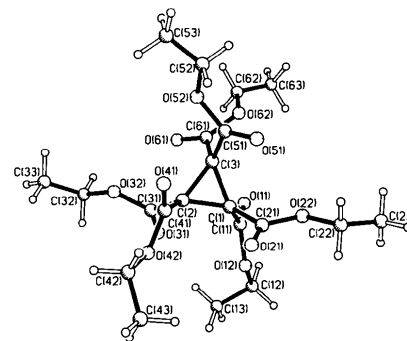

Fig. 1. The title molecule in the crystal, showing the atom-numbering scheme. Radii are arbitrary.

A colourless prism 0.75 × 0.45 × 0.3 mm was mounted on a glass fibre and used to record 4516 profile-fitted intensities (Clegg, 1981) on a Stoe–Siemens four-circle diffractometer (monochromated Cu $K\alpha$ radiation, $2\theta_{max}$ 140°). Merging equivalents gave 3895 unique reflections (R_{int} 0.021), of which 3521 with $F > 4\sigma(F)$ were used for all calculations (programs *SHELX*76 and *SHELXS*84; Sheldrick, 1976, 1984). Index ranges after merging were $|h|, |k| \geq 10, |l| \leq 17$. Cell constants were refined from 2θ values of 88 reflections in the range 60–70°. Three check reflections showed no significant intensity variation.

The structure was solved by routine direct methods