Les atomes de tellure ont donc deux types d'environnements. Les atomes de Te(1) ont un environnement de quatre atomes Te(2) situés à 3,027 Å. Cette valeur est assez proche de celle observée dans le tellure élémentaire (2,74 Å), et très inférieure à celle observée lors du contact de deux ions Te^{2+} (4,42 Å) dans les tellures ioniques. La liaison Te(1)—Te(2) a donc un caractère principalement covalent. Les atomes de Te(2) ont un environnement plus compliqué que le précédent: chacun d'eux est lié à deux atomes de gallium de deux tétraèdres superposés et à l'atome Te(1), ces trois liaisons étant approximativement perpendiculaires les unes aux autres. Les distances entre atomes Te(2) (3,780 Å) sont très supérieures à la distance Te(1)—Te(2) et ne peut correspondre à des liaisons covalentes.

La structure tridimensionnelle de Ga_2Te_5 est formée de chaînes infinies de tétraèdres $GaTe_4$, réunies les unes aux autres par des atomes de tellure interstitiels qui constituent des points Te(2)—Te(1)—Te(2) covalents.

Nous remercions Monsieur le Professeur N. Rodier qui a bien voulu nous apporter son aimable collaboration lors des mesures expérimentales.

Références

Alapini, F., Guittard, M., Julien-Pouzol, M. & Flahaut, J. (1976). *C. R. Acad. Sci. Paris, Sér. C*, **282**, 543–545.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104–109.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Newman, P. C., Brice, J. C. & Wright, A. C. (1961). *Philips Res. Rep.* **16**, 41–48.

Acta Cryst. (1977). B**33**, 2272–2273

cyclo-Tri-μ-nitrido-[bis(trimethylsilylamino)phosphorus]disulphur

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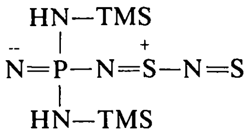
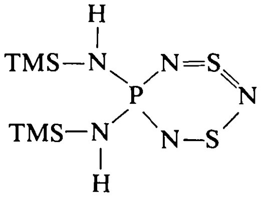
Abstract. $S_2N_5PSi_2C_6H_{20}$, triclinic, $P\bar{1}$, $a = 7{\cdot}281\ (3)$, $b = 10{\cdot}667\ (5)$, $c = 11{\cdot}911\ (7)$ Å, $\alpha = 108{\cdot}10\ (4)$, $\beta = 110{\cdot}62\ (4)$, $\gamma = 87{\cdot}56\ (4)^\circ$, $V = 821$ Å^3, $D_x = 1{\cdot}26$ g cm^{-3}, $Z = 2$. The structure was determined from diffractometer data by direct methods and refined by least squares to $R = 0{\cdot}073$ for 2612 independent reflexions. The molecule contains a six-membered ring comprising one P, two S and three N atoms.

Introduction. Appel & Halstenberg (1976) synthesized a S–N–P-containing compound which was remarkable because of its deep-blue colour. They proposed two different reaction paths which should lead to different structures [(I) and (II)]. This determination was carried out to establish the molecular structure and thus to decide between the proposed reaction mechanisms.



(I)



(II)

$[TMS = -Si(CH_3)_3]$

A crystal 0·18 × 0·28 × 0·46 mm was mounted in a glass capillary over P_4O_{10} (because of the pronounced moisture sensitivity).

Lattice constants were determined from the θ values of 37 reflexions by least squares (Berdesinski & Nuber, 1966); intensities were measured on an automatic single-crystal diffractometer (AED, Siemens) by the five-value method. In a θ range up to 30°, 2612 independent reflexions with $I > 2{\cdot}58\sigma(I)$ were observed. They were corrected with Lorentz and polarization factors, but no further corrections were applied.

The structure was solved with *MULTAN* (Main, Germain & Woolfson, 1971), implemented in the local version of the XRAY system (Stewart, Kundell & Baldwin, 1970), and subsequent Fourier syntheses. Least-squares refinement with anisotropic temperature factors converged at $R = 0{\cdot}073$ (maximum shift/error

Table 1. *Positional parameters* (× 10^4)

	x	*y*	*z*
S(1)	2627 (3)	4017 (2)	331 (2)
S(2)	3268 (3)	1341 (2)	9916 (2)
P	9754 (2)	2188 (2)	114 (2)
Si(1)	6570 (3)	1628 (2)	7478 (2)
Si(2)	74 (3)	3132 (2)	2896 (2)
N(1)	752 (8)	3693 (5)	573 (6)
N(2)	3976 (9)	2842 (7)	139 (7)
N(3)	874 (7)	1121 (5)	9355 (5)
N(4)	7507 (7)	2063 (5)	9134 (5)
N(5)	9717 (7)	1966 (5)	1403 (5)
C(1)	4168 (14)	2386 (12)	7063 (10)
C(2)	8291 (18)	2286 (14)	6909 (10)
C(3)	6147 (15)	9801 (9)	6810 (9)
C(4)	2703 (12)	3789 (8)	3741 (8)
C(5)	9297 (15)	2179 (9)	3747 (8)
C(6)	8480 (13)	4527 (8)	2712 (9)

0·087, average shift/error 0·021). The positional parameters are listed in Table 1.*

The location of the H atoms was tried by difference syntheses and steric considerations. Atomic coordinates could be found which diminished *R* to 0·065, but some resulted in unreasonably short bond lengths, others in large temperature factors. Therefore the H positions were not inserted in the final refinement cycles.

Discussion. The structure determination proved (II) to be correct. Bond lengths and angles are summarized in Tables 2 and 3. Fig. 1 is an *ORTEP* plot of the molecule.

The P atom has a normal, though slightly deformed, tetrahedral configuration. The six-membered ring (II) contains S(1) in oxidation state four and S(2) in oxidation state two, as shown by the different bond lengths (Table 2) between the S atoms and their N neighbours. The ring is puckered; the deviations from a plane through the three N atoms are: S(1) −0·41, S(2) +0·53, P +0·27 Å.

Table 2. *Bond lengths* (Å)

S(1)–N(1)	1·564 (7)	Si(1)–N(4)	1·755 (6)
S(1)–N(2)	1·570 (7)	Si(1)–C(1)	1·864 (11)
S(2)–N(2)	1·615 (8)	Si(1)–C(2)	1·868 (16)
S(2)–N(3)	1·630 (5)	Si(1)–C(3)	1·855 (9)
P–N(1)	1·637 (6)	Si(2)–N(5)	1·762 (5)
P–N(3)	1·605 (6)	Si(2)–C(4)	1·867 (8)
P–N(4)	1·623 (5)	Si(2)–C(5)	1·864 (12)
P–N(5)	1·635 (7)	Si(2)–C(6)	1·867 (9)

Table 3. *Bond angles* (°)

N(1)–S(1)–N(2)	115·0 (4)	N(4)–Si(1)–C(1)	105·9 (4)
N(1)–S(2)–N(3)	110·9 (3)	N(4)–Si(1)–C(2)	110·5 (4)
N(1)–P–N(3)	112·1 (3)	N(4)–Si(1)–C(3)	108·5 (4)
N(1)–P–N(4)	110·7 (3)	C(1)–Si(1)–C(2)	110·5 (6)
N(1)–P–N(5)	104·8 (3)	C(1)–Si(1)–C(3)	109·5 (5)
N(3)–P–N(4)	105·3 (3)	C(2)–Si(1)–C(3)	111·8 (6)
N(3)–P–N(5)	115·3 (3)	N(5)–Si(2)–C(4)	111·4 (3)
N(4)–P–N(5)	108·7 (3)	N(5)–Si(2)–C(5)	103·7 (3)
S(1)–N(1)–P	122·9 (4)	N(5)–Si(2)–C(6)	110·0 (3)
S(1)–N(2)–S(2)	122·3 (5)	C(4)–Si(2)–C(5)	111·2 (4)
S(2)–N(3)–P	115·8 (3)	C(4)–Si(2)–C(6)	109·6 (4)
P–N(4)–Si(1)	130·5 (4)	C(5)–Si(2)–C(6)	110·7 (5)
P–N(5)–Si(2)	130·7 (4)		

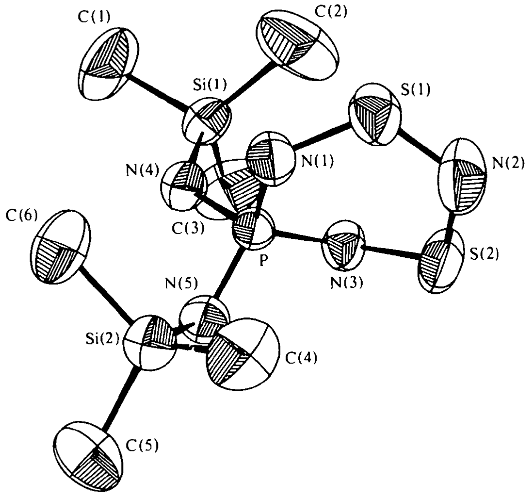


Fig. 1. An *ORTEP* plot (Johnson, 1965) of the molecule.

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References

Appel, R. & Halstenberg, M. (1976). *Angew. Chem.* **88**, 763–764; *Angew. Chem. Int. Ed.* **15**, 695–696.

Berdesinski, W. & Nuber, B. (1966). *Neues Jb. Miner. Abh.* **104**, 113–146.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Main, P., Germain, G. & Woolfson, M. M. (1971). *MULTAN. A Computer Program for the Automatic Solution of Crystal Structures*, Dept. of Physics, Univ. of York, England.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32541 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.