HAMILTON, W. C. & IBERS, J. A. (1974). Editors. *International Tables for X-ray Crystallography*, Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1971). *ORTEPII*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Structure of (1α,3β,5α)-5-Chloro-1,3-diphenyl-5-bis(trimethylsilyl)methyl-1λ⁶,3λ⁶,2,4,6,5λ⁵-dithiatriazaphosphorine 1,3-Dioxide

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Abstract. $C_{19}H_{29}ClN_3O_2PS_2Si_2$, $M_r = 518.17$, monoclinic, $P2_1/n$, $a = 10.751$ (3), $b = 22.48$ (1), $c = 10.942$ (3) Å, $\beta = 108.58$ (2)°, $V = 2507$ (2) Å³, $Z = 4$, $D_x = 1.373$ g cm⁻³, Mo $K\bar{\alpha}$, $\lambda = 0.71073$ Å, $\mu = 4.9$ cm⁻¹, $F(000) = 1088$, $T = 130$ K, $R = 0.069$ for 2831 observed reflections with $I > 2.5\sigma(I)$. The inorganic ring skeleton has an envelope conformation. The S–N and P–N bond lengths (mean values) are 1.562 (6) and 1.590 (8) Å, respectively. One of the phenyl rings exhibits a disordered behaviour.

Experimental. Colourless crystals obtained by recrystallization from diethyl ether/pentane (1:1). Crystal (0.13 × 0.20 × 0.35 mm) was glued on top of a glass fibre and transferred into the cold nitrogen stream of the low-temperature unit on an Enraf–Nonius CAD-4F diffractometer interfaced to a PDP-11/23. Graphite-monochromated Mo $K\bar{\alpha}$ radiation. Cell dimensions from setting angles of 22 reflections ($7.02 < \theta < 13.94°$). Intensity data of 4390 reflections were collected; $h -13 \rightarrow 11$, $k\, 0 \rightarrow 24$, $l\, 0 \rightarrow 13$; $[(\sin\theta)/\lambda]_{max} = 0.6379$ Å⁻¹; $\omega/2\theta$ scan mode with $\Delta\omega = (0.90 + 0.35\tan\theta)°$. Three reference reflections measured every 167 min ($43\bar{6}$: r.m.s.d. 2.9%; $\bar{4}36$: r.m.s.d. 2.4%; $45\bar{4}$: r.m.s.d. 1.7%) showed no indication of crystal decomposition during the 160 h of X-ray exposure time. Intensities corrected for scale variation and for Lorentz and polarization effects. No absorption correction applied. Variance $\sigma^2(I)$ calculated on the basis of counting statistics plus the term $(PI)^2$, where P ($= 0.040$) is the instability constant as derived from the

excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). Data set averaged in a set of 4046 unique reflections ($R_{int} = 0.070$). Structure solved by direct methods using *SHELXS* (Sheldrick, 1986) and refined on F by block-diagonal least-squares techniques with anisotropic thermal parameters for non-hydrogen atoms ($R = 0.079$, $wR = 0.092$). The parameters of one of the phenyl rings [C(7)–C(12)] show unrealistic values; bond lengths (varying from 1.24 to 1.54 Å), bond angles (varying from 114 to 124°) and high temperature factors suggest some degree of disorder. Constrained group refinement introduced for [C(7)–C(12)] (C–C = 1.38, C–H = 1.0 Å and C–C–C = 120°). H atoms located on a difference Fourier map and included in the final refinement with one overall isotropic temperature factor. Convergence with 332 parameters was reached at $R = 0.069$, $wR = 0.076$, $w = 1$, $S = 3.163$; 2831 observed reflections with $I > 2.5\sigma(I)$; average $\Delta/\sigma = 0.028$, maximum $\Delta/\sigma = 0.197$. Minimum and maximum residual densities in final Fourier map −0.63 and 0.79 e Å⁻³. In the vicinity of the disordered phenyl ring the densities are −1.35 and 1.75 e Å⁻³, respectively. The observed conformation of the PNS ring approaches that of an envelope (Boeyens, 1978) with torsion angles ranging from −27.3 (6) to 29.4 (6)° and lowest asymmetry parameter value $\Delta C_s[P(1)] = 3.1$ (5)° (Duax, Weeks & Rohrer, 1976). Puckering parameters are $Q = 0.243$ (5) Å, $\theta = 128$ (1)°, $\varphi = 309$ (2)° [(Cremer & Pople, 1975) e.s.d. following Norrestam (1981)]. Scattering factors from Cromer & Mann (1968). Anomalous-dispersion factors were taken from Cromer & Liberman (1970). All calculations carried

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Table 1. *Final fractional atomic coordinates and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å²)
Cl(1)	0·7985 (2)	0·09262 (9)	0·0797 (2)	0·0302 (6)
S(1)	0·6071 (2)	0·22392 (9)	0·1538 (2)	0·0248 (5)
S(2)	0·6573 (2)	0·13624 (9)	0·3454 (2)	0·0255 (6)
P(1)	0·8359 (2)	0·15762 (9)	0·2173 (2)	0·0196 (5)
Si(1)	1·0463 (2)	0·22743 (9)	0·1427 (2)	0·0201 (6)
Si(2)	1·1137 (2)	0·1032 (1)	0·3048 (2)	0·0241 (6)
O(1)	0·5093 (5)	0·2206 (3)	0·0303 (5)	0·044 (2)
O(2)	0·6598 (6)	0·1426 (4)	0·4760 (6)	0·059 (2)
N(1)	0·7496 (5)	0·2130 (3)	0·1515 (5)	0·025 (2)
N(2)	0·5691 (6)	0·1833 (3)	0·2545 (8)	0·040 (3)
N(3)	0·7990 (5)	0·1315 (3)	0·3372 (6)	0·025 (2)
C(1)	0·6063 (7)	0·2965 (3)	0·2106 (7)	0·021 (2)
C(2)	0·7122 (7)	0·3177 (4)	0·3101 (7)	0·028 (3)
C(3)	0·7081 (8)	0·3737 (4)	0·3553 (8)	0·041 (3)
C(4)	0·5997 (9)	0·4095 (4)	0·3022 (9)	0·041 (3)
C(5)	0·4961 (8)	0·3877 (4)	0·2056 (8)	0·036 (3)
C(6)	0·4977 (7)	0·3318 (4)	0·1593 (7)	0·029 (3)
C(7)	0·579 (1)	0·0694 (3)	0·290 (1)	0·042 (3)
C(8)	0·499 (1)	0·0614 (3)	0·1649 (9)	0·058 (4)
C(9)	0·438 (1)	0·0073 (5)	0·1268 (7)	0·068 (4)
C(10)	0·458 (1)	−0·0389 (3)	0·214 (1)	0·20 (1)
C(11)	0·539 (1)	−0·0311 (3)	0·3392 (9)	0·54 (3)
C(12)	0·599 (1)	0·0231 (5)	0·3772 (7)	0·40 (2)
C(13)	1·0036 (6)	0·1713 (3)	0·2569 (6)	0·018 (2)
C(14)	0·9477 (7)	0·2131 (4)	−0·0262 (7)	0·027 (3)
C(15)	1·2212 (7)	0·2215 (4)	0·1527 (8)	0·032 (3)
C(16)	1·0188 (8)	0·3023 (4)	0·1982 (7)	0·030 (3)
C(17)	1·2740 (8)	0·1302 (4)	0·4134 (8)	0·039 (3)
C(18)	1·1328 (8)	0·0637 (4)	0·1624 (8)	0·038 (3)
C(19)	1·0495 (8)	0·0490 (4)	0·3968 (8)	0·034 (3)

out on the CDC Cyber 170/760 computer of the University of Groningen with the program packages *XTAL* (Stewart & Hall, 1983), *EUCLID* (Spek, 1982; calculation of geometric data) and a locally modified version of the program *PLUTO* (Meetsma, 1986; Motherwell & Clegg, 1978; preparation of illustrations). Final fractional atomic coordinates and equivalent isotropic thermal parameters for the non-hydrogen atoms are presented in Table 1.* Intramolecular bond

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms and an *ORTEP* plot (Johnson, 1965) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44329 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
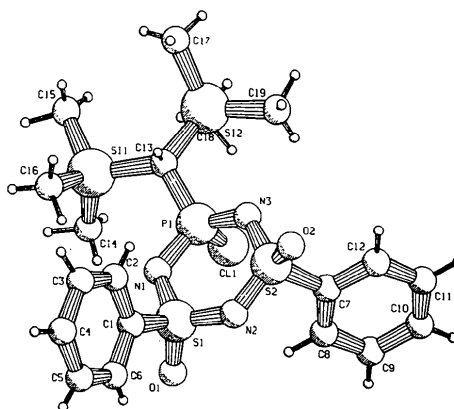

Fig. 1. *PLUTO* drawing illustrating the puckering and the adopted numbering scheme.

Table 2. *Selected data on the geometry: bond distances* (Å) *and angles* (°) *and torsion angles* (°)

Cl(1)—P(1)	2·044 (3)	C(7)—S(2)	1·735 (8)
S(1)—N(1)	1·560 (6)	C(7)—C(8)	1·38 (1)
S(1)—O(1)	1·425 (6)	C(7)—C(12)	1·38 (1)
S(1)—N(2)	1·582 (8)	C(8)—C(9)	1·38 (1)
S(2)—N(2)	1·551 (8)	C(9)—C(10)	1·38 (1)
S(2)—N(3)	1·558 (6)	C(10)—C(11)	1·38 (1)
S(2)—O(2)	1·428 (7)	C(11)—C(12)	1·38 (1)
P(1)—N(1)	1·582 (7)	C(13)—P(1)	1·742 (7)
P(1)—N(3)	1·598 (7)	C(13)—Si(1)	1·931 (7)
C(1)—S(1)	1·747 (7)	C(13)—Si(2)	1·904 (7)
C(1)—C(2)	1·39 (1)	C(14)—Si(1)	1·842 (8)
C(1)—C(6)	1·37 (1)	C(15)—Si(1)	1·853 (8)
C(2)—C(3)	1·36 (1)	C(16)—Si(1)	1·845 (9)
C(3)—C(4)	1·38 (1)	C(17)—Si(2)	1·856 (9)
C(4)—C(5)	1·36 (1)	C(18)—Si(2)	1·861 (9)
C(5)—C(6)	1·36 (1)	C(19)—Si(2)	1·849 (9)

N(1)—S(1)—N(2)	113·2 (4)	Cl(1)—P(1)—C(13)	104·7 (2)
O(1)—S(1)—C(1)	107·5 (4)	P(1)—N(1)—S(1)	123·6 (4)
N(2)—S(2)—N(3)	114·8 (4)	S(1)—N(2)—S(2)	126·8 (5)
O(2)—S(2)—C(7)	106·8 (5)	S(2)—N(3)—P(1)	121·7 (4)
N(1)—P(1)—N(3)	113·6 (3)		

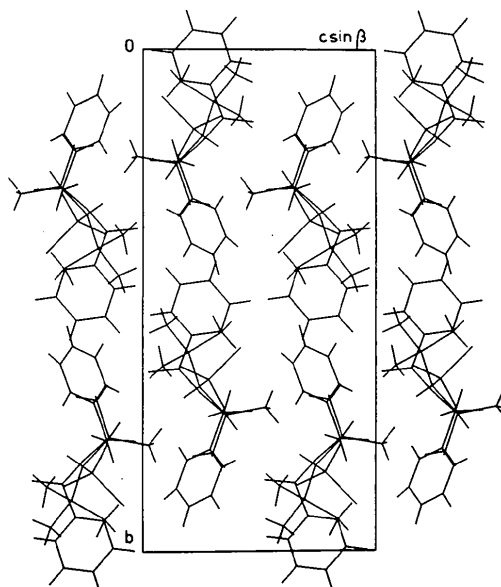|			
O(1)—S(1)—N(1)—P(1)	−114·3 (5)	N(2)—S(2)—N(3)—P(1)	−17·8 (6)
N(2)—S(1)—N(1)—P(1)	13·5 (6)	O(2)—S(2)—N(3)—P(1)	−147·0 (5)
C(1)—S(1)—N(1)—P(1)	127·9 (5)	C(7)—S(2)—N(3)—P(1)	96·8 (6)
N(1)—S(1)—N(2)—S(2)	−0·1 (7)	Cl(1)—P(1)—N(1)—S(1)	90·5 (4)
O(1)—S(1)—N(2)—S(2)	129·4 (6)	N(3)—P(1)—N(1)—S(1)	−27·3 (6)
C(1)—S(1)—N(2)—S(2)	−114·8 (6)	C(13)—P(1)—N(1)—S(1)	−155·4 (4)
N(3)—S(2)—N(2)—S(1)	2·4 (8)	Cl(1)—P(1)—N(3)—S(2)	−87·7 (5)
O(2)—S(2)—N(2)—S(1)	130·7 (6)	N(1)—P(1)—N(3)—S(2)	29·4 (6)
C(7)—S(2)—N(2)—S(1)	−114·2 (7)	C(13)—P(1)—N(3)—S(2)	158·0 (4)

Fig. 2. Projection of the crystal structure down [100].

distances and angles are summarized in Table 2. Fig. 1 shows the molecular structure and Fig. 2 the arrangement of the compound in the unit cell viewed along **a**.

Related literature. The structural parameters of the inorganic ring skeleton can be compared with those found in related structures (NSOPh)₂NP*XY* (van Bolhuis, Cnossen-Voswijk & van de Grampel, 1981; van Bolhuis, van den Berg & van de Grampel, 1981; Meetsma, Spek, Olthof-Hazekamp, Winter, van de Grampel & de Boer, 1985; Meetsma, Spek, Winter, Cnossen-Voswijk, van de Grampel & de Boer, 1986; Meetsma, Spek, Winter, van de Grampel & de Boer, 1986; Winter, van de Grampel, de Boer, Meetsma & Spek, 1987).

References

BOEYENS, J. C. A. (1978). *J. Cryst. Mol. Struct.* **8**, 317–320.

BOLHUIS, F. VAN, VAN DEN BERG, J. B. & VAN DE GRAMPEL, J. C. (1981). *Cryst. Struct. Commun.* **10**, 1031–1035.

BOLHUIS, F. VAN, CNOSSEN-VOSWIJK, C. & VAN DE GRAMPEL, J. C. (1981). *Cryst. Struct. Commun.* **10**, 69–72.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. ALLINGER & E. L. ELIEL, pp. 271–383. New York: John Wiley.

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

MCCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* A**31**, 245–249.

MEETSMA, A. (1986). Extended version of the program *PLUTO*. Univ. of Groningen, The Netherlands.

MEETSMA, A., SPEK, A. L., OLTHOF-HAZEKAMP, R., WINTER, H., VAN DE GRAMPEL, J. C. & DE BOER, J. L. (1985). *Acta Cryst.* C**41**, 1801–1804.

MEETSMA, A., SPEK, A. L., WINTER, H., CNOSSEN-VOSWIJK, C., VAN DE GRAMPEL, J. C. & DE BOER, J. L. (1986). *Acta Cryst.* C**42**, 365–368.

MEETSMA, A., SPEK, A. L., WINTER, H., VAN DE GRAMPEL, J. C. & DE BOER, J. L. (1986). *Acta Cryst.* C**42**, 368–371.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NORRESTAM, R. (1981). *Acta Cryst.* A**37**, 764–765.

SHELDRICK, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

SPEK, A. L. (1982). *The EUCLID package.* In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.

STEWART, J. H. & HALL, S. R. (1983). *The XTAL system.* Tech. Rep. TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland.

WINTER, H., VAN DE GRAMPEL, J. C., DE BOER, J. L., MEETSMA, A. & SPEK, A. L. (1987). *Phosphorus Sulfur*, **32**, 145–151.

Structure of 3-{2-[4-(2-Methoxyphenyl)-1-piperazinyl]ethyl}-2,4(1*H*,3*H*)-quinazolinedione Monohydrate (SGB1534)

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Abstract. $C_{21}H_{24}N_4O_3.H_2O$, $M_r = 398.47$, monoclinic, $P2_1/c$, $a = 19.199$ (4), $b = 13.667$ (2), $c = 7.559$ (2) Å, $\beta = 97.05$ (2)°, $V = 1968.5$ Å³, $Z = 4$, $D_x = 1.344$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 7.36$ cm⁻¹, $F(000) = 848$, $T = 298$ K, final $R = 0.059$ for 1295 unique reflections $[F_o^2 > 1.8\sigma(F_o^2)]$. Phenyl and quinazolinedione moieties of an SGB1534 molecule are arranged parallel to each other, and attached to the N atoms of a piperazine ring (chair form) at equatorial positions. Water molecules are held in the hole running through the crystal along the direction [001].

Experimental. Colorless thin plates of SGB1534 grew from a mixed solvent of methanol/water (=2:1, v/v).

Crystal size $0.33 \times 0.25 \times 0.03$ mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu *K*α radiation, graphite monochromator, θ–2θ scan with scan speed 1.27–$2.75°$ min⁻¹ in θ, scan width $(0.50 + 0.14\tan\theta)°$. Range of indices, $-22 \le h \le 22$, $0 \le k \le 16$, $0 \le l \le 8$ $(2\theta < 130°)$. Lattice parameters determined based on 22 2θ values $(24 < 2\theta < 64°)$. Variation of standard $< 1.2\%$; 3338 reflections measured; 1295 observed reflections with $F_o^2 > 1.8\sigma(F_o^2)$. Systematic absences $h0l$, l odd; $0k0$, k odd. No corrections for absorption. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refined by full-matrix least squares. The locations of H atoms were calculated stereochemically, except for those of the water molecule. Non-H atoms refined with anisotropic thermal parameters, and

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