shown in Fig. 2. There are no unusual intermolecular contacts in the structure.

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Structure of Bis(1,3-di-*o*-anisyl-2-thioxo-1,3,2λ^5-diazaphosphorinan-2-yl) Oxide*

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Abstract. $C_{34}H_{40}N_4O_5P_2S_2$, M_r = 710·80, monoclinic, $P2_1/c$, a = 12·122 (4), b = 18·844 (4), c = 7·738 (3) Å, β = 106·99 (2)°, V = 1690·5 (9) Å^3, Z = 2, D_x = 1·40 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 3·01 cm^{-1}, $F(000)$ = 748, T = 291 K, R = 0·032 for 2067 observed reflections. The centrosymmetric molecule possesses a linear P—O—P linkage with a P—O bond length of 1·593 (1) Å. The diazaphosphorinane ring adopts a chair conformation with the thiophosphoryl sulfur atom in the equatorial position and the bridging oxygen atom in the axial position. The aromatic substituent at N(3) is axially oriented whereas the substituent at N(1) is rather equatorial. Both nitrogen atoms are pyramidally coordinated.

Introduction. As part of our studies on macrocyclic phosphoramide derivatives (Dutasta & Simon, 1987), we have investigated the synthesis of rigidified molecules containing the 1,3,2-diazaphosphorinane ring as the binding subunit. The present structure determination was undertaken to identify the main compound (1) formed in the reaction between hexamethylphosphorus triamide (HMPT) and N,N'-di-*o*-anisyl-1,3-propanediamine. The unexpected dimeric structure of (1) containing two 1,3,2-diazaphosphorinane rings was first suggested from the NMR and mass spectroscopy data. Knowledge of structures of 1,3,2-diazaphosphorinanes is still rather limited and detailed structural data previously available mainly concern compounds (2) [R = OC_2H_5, R' = t-C_4H_9, X = S, Y = CH_3; Belov *et al.*, 1981; R = $Si(C_6H_5)_3$, R' = t-C_4H_9, X = lone pair, Y = H; Nifant'ev, Sorokina, Vorob'eva, Borisenko & Nevskii, 1985].

The determination of the molecular structure of the title compound is of particular interest as, to our knowledge, no thiopyrophosphoramidic structures have been reported so far. Only the X-ray structures of the acyclic octamethylpyrophosphoramide (4) in metal complexes have been described (Hussain, Joesten & Lenhert, 1970; Kepert, Patrick & White,

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* P,P'-Oxybis(1,3-di-*o*-methoxyphenyl-1,3,2λ^5-diazaphosphorinane 2-sulfide).

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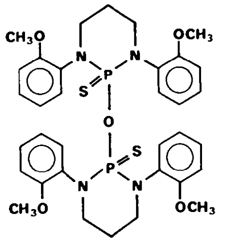
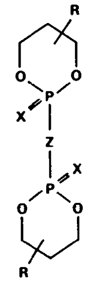
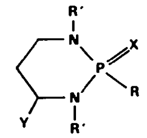
1983). This contrasts with the well documented 1,3,2-dioxaphosphorinane systems (3) for which X-ray studies have been completed with pyrophosphates ($X = O$, $Z = O$; Bukowska-Strzyzewska & Dobrowolska, 1978; Cook & White, 1976; Jones, Sheldrick, Kirby & Briggs, 1985), thiopyrophosphates ($X = S$, $Z = O$; Wieczorek, Sheldrick, Karolak-Wojciechowska, Mikolajczyk & Ziemnicka, 1979; Bukowska-Strzyzewska & Dobrowolska, 1980; Mikolajczyk, Ziemnicka, Karolak-Wojciechowska & Wieczorek, 1983; Mikolajczyk, Ziemnicka, Wieczorek & Karolak-Wojciechowska, 1984) and sulfide derivatives ($X = O$, $Z = S$; Bukowska-Strzyzewska, Michalski, Mlotkowska & Skoweranda, 1976).



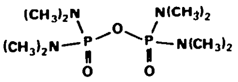
(1)



(3)



(2)



(4)

Experimental. The compound was obtained from the reaction of equimolar quantities of hexamethylphosphorus triamide and *N*,*N'*-di-*o*-anisyl-1,3-propanediamine in toluene, at reflux temperature. After heating for five days, sulfur (10% excess) was added. After cooling to room temperature, the solvent was removed and the residue was triturated in chloroform. The precipitate which formed was removed by filtration. Dissolution in hot dimethyl sulfoxide followed by slow cooling gave the compound as clear colourless crystals [m.p.: 521–522 K; δ ^{31}P ($CDCl_3$) = 51·5].

A parallelepiped crystal with dimensions 0·24 × 0·21 × 0·18 mm was used for data collection. Lattice parameters were refined using 15 reflections in the range $5 \leq 2\theta \leq 30°$. The intensities were collected on a Syntex $P2_1$ diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. 2507 independent reflections were measured with $\sin\theta/\lambda \leq 0·56$ Å^{-1}; $-13 \leq h \leq 13$, $0 \leq k \leq 21$, $0 \leq l \leq 8$; 2067 with $I \geq 2·5\sigma(I)$. Standard reflection ($25\bar{3}$) checked every 50 reflections: no significant deviation. Structure solved by *SHELXS*86 (Sheldrick, 1985). Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using *F*. Hydrogen atoms from difference Fourier synthesis and refined isotropically with a common temperature factor. Final $R = 0·032$, $wR = 0·034$, for 2067 observed reflections [$w = 1/(\sigma^2 + 0·00006F^2)$]. Final maximum shift to e.s.d. = 0·3. $S = 2·14$. Maximum and minimum peak heights in final difference Fourier synthesis are 0·27 and $-0·19$ e Å^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic temperature factors* (Å²)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
N(1)	1950 (2)	10623 (1)	1337 (3)	2·21 (4)
P(2)	1351 (1)	9847 (1)	662 (1)	1·70 (1)
N(3)	1470 (2)	9404 (1)	2559 (2)	1·98 (3)
C(4)	2503 (2)	8993 (1)	3356 (3)	2·43 (4)
C(5)	3585 (2)	9214 (2)	3299 (4)	3·24 (5)
C(6)	4555 (3)	8804 (2)	4098 (4)	4·41 (6)
C(7)	4438 (3)	8188 (2)	4933 (5)	5·30 (7)
C(8)	3387 (3)	7965 (2)	5041 (4)	4·46 (6)
C(9)	2400 (2)	8370 (1)	4272 (3)	3·06 (5)
C(10)	2217 (2)	11096 (1)	55 (3)	2·41 (4)
C(11)	1375 (2)	11507 (1)	−1111 (4)	3·21 (5)
C(12)	1660 (3)	11975 (2)	−2290 (4)	4·09 (6)
C(13)	2773 (3)	12024 (2)	−2323 (4)	4·18 (6)
C(14)	3628 (3)	11618 (2)	−1179 (4)	3·63 (6)
C(15)	3361 (2)	11152 (1)	34 (3)	2·75 (5)
C(16)	1677 (3)	10977 (2)	2887 (4)	3·34 (5)
C(17)	1775 (3)	10478 (2)	4463 (4)	3·29 (5)
C(18)	1068 (2)	9813 (1)	3918 (3)	2·57 (5)
O(19)	1320 (2)	8197 (1)	4328 (3)	3·87 (4)
C(20)	1234 (4)	7699 (2)	5684 (5)	5·61 (8)
O(21)	4144 (1)	10742 (1)	1241 (3)	3·74 (4)
C(22)	5322 (3)	10789 (2)	1243 (6)	4·75 (7)
S(23)	1871 (1)	9371 (1)	−1132 (1)	3·06 (1)
O(24)	0	10000	0	2·75 (4)

Discussion. Final atomic coordinates are given in Table 1. Table 2 reports the main interatomic distances, bond angles and torsion angles.* Fig. 1 is a view of the molecule, showing the numbering of the atoms (program *PLUTO*; Motherwell & Clegg, 1978). The atom O(24) lies on a crystallographic center of symmetry so that the asymmetric part of the unit cell contains only one half of the molecule. This results in the linear P—O—P sequence, a somewhat unusual feature for neutral pyrophosphate derivatives. The P—O bond length of 1·593 Å is in agreement with the values obtained for parent compounds. Except for rare examples of linear P—O—P sequences in inorganic pyrophosphate ions, only one organic diphosphonium salt with a linear P—O—P group has been reported (Aaberg, Gramstad &

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* 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 52175 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) (σ = 1)

P(2)—N(1)	1·649 (2)	C(10)—N(1)	1·439 (3)
C(16)—N(1)	1·491 (3)	N(3)—P(2)	1·657 (2)
S(23)—P(2)	1·908 (1)	O(24)—P(2)	1·593 (1)
C(4)—N(3)	1·447 (3)	C(18)—N(3)	1·496 (3)
C(5)—C(4)	1·389 (4)	C(9)—C(4)	1·395 (4)
C(6)—C(5)	1·391 (4)	C(7)—C(6)	1·356 (5)
C(8)—C(7)	1·368 (5)	C(9)—C(8)	1·398 (4)
O(19)—C(9)	1·362 (3)	C(11)—C(10)	1·385 (4)
C(15)—C(10)	1·396 (3)	C(12)—C(11)	1·383 (4)
C(13)—C(12)	1·361 (4)	C(14)—C(13)	1·381 (4)
C(15)—C(14)	1·391 (4)	O(21)—C(15)	1·362 (3)
C(17)—C(16)	1·517 (4)	C(18)—C(17)	1·507 (4)
C(20)—O(19)	1·435 (4)	C(22)—O(21)	1·430 (3)

C(10)—N(1)—P(2)	119·9 (2)	C(16)—N(1)—P(2)	118·1 (2)
C(16)—N(1)—C(10)	115·1 (2)	N(3)—P(2)—N(1)	104·5 (1)
S(23)—P(2)—N(1)	116·0 (1)	S(23)—P(2)—N(3)	116·5 (1)
O(24)—P(2)—N(1)	104·9 (1)	O(24)—P(2)—N(3)	101·1 (1)
O(24)—P(2)—S(23)	112·3 (1)	P(2)—O(24)—P(2)	180·0
C(4)—N(3)—P(2)	119·2 (1)	C(18)—N(3)—P(2)	113·7 (2)
C(18)—N(3)—C(4)	113·2 (2)	C(5)—C(4)—N(3)	122·2 (2)
C(9)—C(4)—N(3)	118·2 (2)	C(9)—C(4)—C(5)	119·6 (2)
C(6)—C(5)—C(4)	120·2 (3)	C(7)—C(6)—C(5)	119·7 (3)
C(8)—C(7)—C(6)	121·4 (3)	C(9)—C(8)—C(7)	120·2 (3)
C(8)—C(9)—C(4)	118·9 (3)	O(19)—C(9)—C(4)	116·6 (2)
O(19)—C(9)—C(8)	124·5 (3)	C(11)—C(10)—N(1)	121·6 (2)
C(15)—C(10)—N(1)	118·6 (2)	C(15)—C(10)—C(11)	119·7 (2)
C(12)—C(11)—C(10)	120·5 (3)	C(13)—C(12)—C(11)	119·7 (3)
C(14)—C(13)—C(12)	120·9 (3)	C(15)—C(14)—C(13)	120·2 (3)
C(14)—C(15)—C(10)	118·9 (3)	O(21)—C(15)—C(10)	116·4 (2)
O(21)—C(15)—C(14)	124·6 (2)	C(17)—C(16)—N(1)	112·7 (2)
C(18)—C(17)—C(16)	112·7 (2)	C(17)—C(18)—N(3)	110·5 (2)
C(20)—O(19)—C(9)	117·1 (3)	C(22)—O(21)—C(15)	117·4 (2)

C(10)—N(1)—P(2)—N(3)	167	S(23)—P(2)—N(3)—C(18)	179
C(10)—N(1)—P(2)—S(23)	38	O(24)—P(2)—N(3)—C(4)	163
C(10)—N(1)—P(2)—O(24)	−87	O(24)—P(2)—N(3)—C(18)	−59
C(16)—N(1)—P(2)—N(3)	−43	P(2)—N(3)—C(4)—C(5)	36
C(16)—N(1)—P(2)—S(23)	−173	P(2)—N(3)—C(4)—C(9)	−147
C(16)—N(1)—P(2)—O(24)	63	C(18)—N(3)—C(4)—C(5)	−102
P(2)—N(1)—C(10)—C(11)	79	C(18)—N(3)—C(4)—C(9)	75
P(2)—N(1)—C(10)—C(15)	−103	P(2)—N(3)—C(18)—C(17)	−61
C(16)—N(1)—C(10)—C(11)	−72	C(4)—N(3)—C(18)—C(17)	79
C(16)—N(1)—C(10)—C(15)	106	C(4)—C(9)—O(19)—C(20)	−161
P(2)—N(1)—C(16)—C(17)	47	C(8)—C(9)—O(19)—C(20)	19
C(10)—N(1)—C(16)—C(17)	−162	C(10)—C(15)—O(21)—C(22)	180
N(1)—P(2)—N(3)—C(4)	−88	C(14)—C(15)—O(21)—C(22)	0
N(1)—P(2)—N(3)—C(18)	49	N(1)—C(16)—C(17)—C(18)	−54
S(23)—P(2)—N(3)—C(4)	41	C(16)—C(17)—C(18)—N(3)	61

Husebye, 1980). The P(S)—O—P(S) angle values in the bis-1,3,2-dioxaphosphorinane derivatives (3) lie in the range 131·7 to 138·2° depending on the orientations of the substituents with respect to the rings (Wieczorek *et al.*, 1979; Mikolajczyk *et al.*, 1983, 1984). Values of 132·1 and 132·4° are observed for the bis(phosphoramide) (4) (Hussain *et al.*, 1970; Kepert *et al.*, 1983). In the present case the configuration of the P—O—P sequence is probably due to severe steric interactions between the aromatic amino groups on the phosphorus atoms.

The conformation of the six-membered heterocycle is a chair with torsion angles −43, 50, −61, 61, −54 and 47°. The P—OP bond is axial whereas the thiophosphoryl sulfur atom is equatorial. The average P—N bond length of 1·653 Å falls in the range of the P—Nsp^2 bond (Aaberg *et al.*, 1980; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) which indicates a certain amount of double-bond character arising from $p\pi$—$d\pi$ interaction. However the ring nitrogen atoms deviate from the planar configuration. The sum of the bond angles around N(1) is 353·1°; an increased p character is observed for N(3) with its bond angles summing to 346·1°. Consequently the P(2)—N(3) bond is longer than the P(2)—N(1) bond (Rømming & Songstad, 1978, 1979). The substituent at N(3) is oriented axially to the ring whereas the substituent at N(1) is rather equatorial. The *o*-anisyl groups in the asymmetric unit are very close to planarity, the angle between the two aromatic rings is 16°. In the situation depicted in Fig. 1, the *o*-anisyl groups of one molecule are arranged in such a way that there are no methoxy groups near one another. No unusually short intermolecular distances were detected between the molecules.

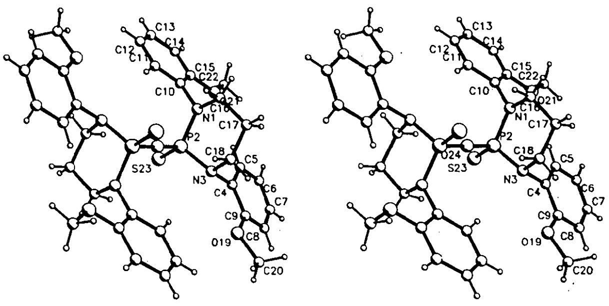

Fig. 1. Three-dimensional view of molecule (1), with atom numbering.

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Structure of D-Perseitol (D-*glycero*-D-*galacto*-Heptitol)

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Abstract. $C_7H_{16}O_7$, $M_r = 212{\cdot}20$, monoclinic, $P2_1$, $a = 9{\cdot}748\ (4)$, $b = 4{\cdot}851\ (2)$, $c = 20{\cdot}013\ (1)$ Å, $\beta = 99{\cdot}61\ (2)^\circ$, $V = 933{\cdot}1\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}510$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 11{\cdot}4$ cm^{-1}, $F(000) = 456$, $T = 295$ K, final $R = 0{\cdot}039$ for 1713 unique observed data. The asymmetric unit consists of two independent molecules which have identical planar extended carbon-chain conformations. All 14 hydroxyl groups are involved as donors and acceptors in intermolecular hydrogen bonds. There are three bifurcated hydrogen bonds involving one of the independent molecules; each consists of a strong intermolecular and a weaker intramolecular interaction. Some of the C—C—O—H torsion angles deviate substantially from the ideal staggered conformations.

Introduction. In the class of alditols the crystal structures of all possible pentitols and hexitols are known (Jeffrey & Kim, 1970). Of the class of heptitols, which consists of 16 members, three occur in plants, D-volemitol (D-*glycero*-D-*manno*-heptitol) in the mushroom *Lactarius volemus*, *β*-sedoheptitol (D-*glycero*-D-*gluco*-heptitol) in the stem and leaves of *Sedum spectabile*, and D-perseitol (D-*glycero*-D-*galacto*-heptitol) in *Laureus persea* L. (avocado) (Hough & Richardson, 1967; Ferrier, 1983). Of the 16 possible heptitols four are *meso* forms and 12 consist of six enantiomeric pairs. Thirteen heptitols are known in the literature.

So far only the crystal structures of *meso*-L-*glycero*-L-*gulo*-heptitol (GGH) (Nimgirawath, James & Mills, 1976) and of D-*glycero*-L-*allo*-heptitol (GAH) (Angyal, Saunders, Grainger, Le Fur & Williams, 1986) have been determined. GGH crystallizes in space group $P2_1/c$ as an assembly of equal numbers of enantiomorphous non-planar conformers and GAH in space group $P2_12_12$ with non-planar conformers of one handedness. Two other *meso*-alditols have been shown to crystallize as enantiomorphous conformers: ribitol crystallizes in space group $P2_1/c$ where both enantiomers are present (Kim, Jeffrey & Rosenstein, 1969) and xylitol has space group $P2_12_12_1$ and contains conformers of only one handedness (Kim & Jeffrey, 1969). We now report the crystal structure of D-perseitol (D-*glycero*-D-*galacto*-heptitol).

Experimental. Crystals suitable for X-ray analysis were obtained from a commercial sample of D-perseitol by crystallization from 1-propanol/water. A colourless thin plate of dimensions $0{\cdot}2 \times 0{\cdot}1 \times 0{\cdot}05$ mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation. Lattice parameters were determined from the setting angles of 25 reflections with $13{\cdot}9 < \theta < 18{\cdot}4^\circ$. The intensity data of 1790 reflections were collected in one quarter of the reflection sphere ($-11 \leq h \leq 0$, $-5 \leq k \leq 0$, $-24 \leq l \leq 24$) with $2\theta_{\max} = 140^\circ$, of which 1713 were above the $2{\cdot}5\sigma(I)$ level. The ω–θ scan mode was applied with $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)^\circ$. Two reference reflections, $\bar{1}\bar{1}\bar{6}$ and $2\bar{1}5$, measured every hour showed no significant variations during 11 h of X-ray exposure

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