This work has received partial support from CONICET and CICPBA, Argentina, and from CNPq, Brazil, through a CONICET–CNPq exchange program. The use of the National Cancer Institute, Frederick Cancer Research Facility, Advanced Scientific Computing Laboratory (ASCL) facilities for the Cambridge Structural Database search is gratefully acknowledged.

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Structure of 6,13-Bis(butylthio)-5,12-dioxa-6,13-dithioxo-6a,7,13a,14-tetraaza-6,13-diphosphadibenz[*a,h*]anthracene

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(*Received* 16 *April* 1991; *accepted* 30 *May* 1991)

Abstract. $C_{22}H_{26}N_4O_2P_2S_4$, $M_r = 568{\cdot}66$, monoclinic, *A*2/*a* (non-standard setting), $a = 14{\cdot}941\ (5)$, $b = 7{\cdot}050\ (1)$, $c = 24{\cdot}832\ (4)$ Å, $\beta = 90{\cdot}48\ (2)°$, $V = 2616\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}444$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 4{\cdot}963$ cm^{-1}, $F(000) = 1184$, room temperature, $R = 0{\cdot}039$, $wR = 0{\cdot}044$ for 1017 observed reflections with $I > 3\sigma(I)$. The molecule is centrosymmetric. The six-membered oxazaphosphorinan ring has a chair conformation, the P═S double bond is equatorial and the P—S single bond is axial. The dihedral angle between the oxazaphosphorinan ring and the phenyl ring is 15·0°.

Experimental. The title compound was synthesized as indicated in *Related literature*. Crystals were obtained by slow evaporation from a trichloromethane solution. A crystal with dimensions 0·2 × 0·2 × 0·3 mm was mounted on a glass fibre. Accurate cell parameters were obtained from centred setting angles of 25 reflections in the range $10 < \theta < 19°$. An Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å) was used. Space group *A*2/*a* with the *b* axis unique is a non-standard setting with equivalent positions $[(0, 0, 0; 0, \frac{1}{2}, \frac{1}{2}) + (x, y, z; -x, -y, -z; \frac{1}{2}-x, y, -z; \frac{1}{2}+x, y, -z)]$. Diffraction intensities in the range $2 < \theta < 25°$ were measured using the ω–2θ scan mode, index range h: −16→16, k: 0→8, l: 0→26. Deviations of three standard reflections, measured after each group of 200 reflections, was less than 3·1%. 2304 unique reflections were collected, of which 1017 were considered observed with $I > 3\sigma(I)$. The intensities were corrected for Lorentz, polarization and absorption effects (with transmission coefficients in the range 0·82–0·97). The structure was solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and the positions of all the H atoms were found in a $\Delta\rho$ map at $R = 0{\cdot}064$. The structure was refined by full-matrix least-squares calculations on F, using unit weights, anisotropic temperature factors for non-H atoms and isotropic temperature factors

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0108-2701/91/122676-02$03.00

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B((3,3) + ac(\cos\beta)B(1,3)]$.

	x	y	z	B_{eq} (Å^2)
P(1)	0·3136 (1)	0·3560 (3)	0·61388 (8)	3·11 (4)
S(1)	0·2566 (1)	0·5198 (3)	0·66251 (9)	4·81 (5)
S(2)	0·3021 (1)	0·0692 (3)	0·62594 (8)	3·75 (4)
O(1)	0·4166 (3)	0·4089 (7)	0·6078 (2)	3·8 (1)
N(1)	0·2821 (3)	0·3755 (8)	0·5496 (2)	3·1 (1)
N(2)	0·1896 (3)	0·3255 (8)	0·5428 (2)	3·0 (1)
C(11)	0·4689 (4)	0·3294 (9)	0·5674 (3)	3·3 (2)
C(12)	0·4329 (4)	0·295 (1)	0·5167 (3)	3·0 (2)
C(13)	0·4899 (4)	0·233 (1)	0·4772 (3)	3·5 (2)
C(14)	0·5795 (4)	0·203 (1)	0·4878 (3)	3·9 (2)
C(15)	0·6126 (4)	0·238 (1)	0·5389 (3)	4·3 (2)
C(16)	0·5585 (4)	0·300 (1)	0·5782 (3)	4·0 (2)
C(21)	0·3381 (4)	0·3309 (8)	0·5060 (3)	2·7 (2)
C(1)	0·3280 (4)	0·046 (1)	0·6973 (3)	4·2 (2)
C(2)	0·4255 (5)	0·061 (1)	0·7113 (3)	5·0 (2)
C(3)	0·4856 (5)	−0·077 (1)	0·6828 (4)	7·1 (3)
C(4)	0·5823 (6)	−0·056 (2)	0·7006 (5)	8·8 (3)

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

P(1)—S(1)	1·880 (2)	P(1)—S(2)	2·052 (1)
P(1)—O(1)	1·592 (3)	P(1)—N(1)	1·667 (3)
S(2)—C(1)	1·818 (5)	O(1)—C(11)	1·394 (5)
N(1)—N(2)	1·434 (4)	N(1)—C(21)	1·409 (5)
N(2)—C(21)	1·280 (5)	C(11)—C(12)	1·387 (5)
C(11)—C(16)	1·379 (5)	C(12)—C(13)	1·375 (5)
C(12)—C(21)	1·460 (5)	C(13)—C(14)	1·379 (5)
C(14)—C(15)	1·380 (6)	C(15)—C(16)	1·344 (6)
C(1)—C(2)	1·499 (6)	C(2)—C(3)	1·506 (9)
C(3)—C(4)	1·516 (8)		

S(1)—P(1)—S(2)	118·22 (8)	S(1)—P(1)—O(1)	111·0 (1)
S(1)—P(1)—N(1)	116·0 (1)	S(2)—P(1)—O(1)	109·1 (1)
S(2)—P(1)—N(1)	101·4 (1)	O(1)—P(1)—N(1)	99·0 (2)
P(1)—S(2)—C(1)	102·4 (2)	P(1)—O(1)—C(11)	121·5 (2)
P(1)—N(1)—N(2)	110·8 (3)	P(1)—N(1)—C(21)	123·5 (3)
N(2)—N(1)—C(21)	115·7 (3)	N(1)—N(2)—C(21)	114·0 (3)
O(1)—C(11)—C(12)	120·5 (3)	O(1)—C(11)—C(16)	118·0 (4)
C(12)—C(11)—C(16)	121·2 (4)	C(11)—C(12)—C(13)	117·7 (4)
C(11)—C(12)—C(21)	120·2 (4)	C(13)—C(12)—C(21)	122·1 (4)
C(12)—C(13)—C(14)	121·3 (5)	C(13)—C(14)—C(15)	119·2 (5)
C(14)—C(15)—C(16)	120·9 (4)	C(11)—C(16)—C(15)	119·7 (5)
N(1)—C(21)—N(2)	123·0 (4)	N(1)—C(21)—C(12)	118·8 (4)
N(2)—C(21)—C(12)	118·2 (4)	S(2)—C(1)—C(2)	114·7 (4)
C(1)—C(2)—C(3)	115·2 (5)	C(2)—C(3)—C(4)	111·8 (7)

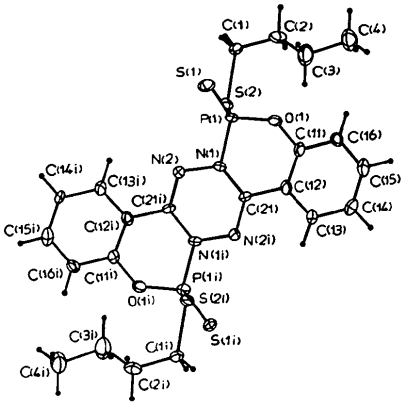

Fig. 1. Perspective drawing and atomic numbering of the title compound. Symmetry code: (i) $-x, -y, -z$.

for H atoms. Structure refinement converged to $R = 0{\cdot}039$, $wR = 0{\cdot}044$ and $S = 2{\cdot}82$ for 207 parameters. Maximum shift/e.s.d. in the final cycle was 1·99 for one of the H atoms. No residual electron density > |0·342 e Å^{-3}| was found. Calculations were carried out on a PDP 11/34 computer with the *SDP* system (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). The fractional coordinates and thermal parameters for the non-H atoms are given in Table 1* and selected bond lengths are in Table 2. A perspective drawing of the molecule is shown in Fig. 1.

Related literature. The *ortho*-difunctional group compounds reacting with P_2S_5 to form stable six-membered phosphacyclic compounds have been reported by Legrand (1968), Walter & Bode (1966) and Sumitomo Chemical Co. Ltd (1980). Many of these compounds have biological activity and some of the crystal structures have been determined by Perales, Fayos & Garcia-Blanco (1979) and Garcia-Blanco & Perales (1972). *Ortho*-polyfunctional group compounds reacted with P_2O_5 have been studied by Chen, Jin, Miao & Wang (1987). The P=S and P—S bonds in the title compound are in agreement with Karolak-Wojciechowska, Wieczorek, Zwierzak & Zawadski (1979).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, C—H bond lengths and calculations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54310 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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