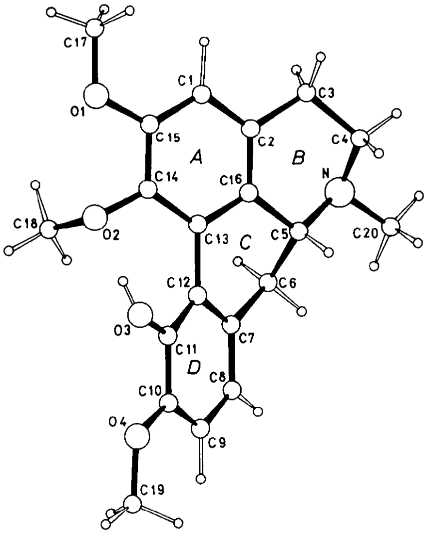

Fig. 1. Perspective view of the molecule.

Related literature. According to Berezhinskaya, Aleshinskaya & Aleshkima (1968) isocorydine exhibits adrenolytic action in anaesthetized cats and rabbits and also anti-adrenergic effects. The dihedral angle between the least-squares planes for phenyl rings *A* and *D* is 32.3 (1)°, whereas this angle is 15.5 and 21.1° in leucoxine and isoboldine hydrobromide (Brown & Hall, 1977), 30.2 (1)° in bulbocapnine methiodide (Wei, Basu, Einstein & Hingerty, 1984), 34.4 (2)° in bulbocapnine (Ribár, Mészáros, Gašić, Kanyó & Engel, 1991) and 24.9° in nanteine (Ribár, Mészáros, Engel, Gašić & Kanyó, 1991).

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Structure of 3,3-Diethyl-1,2-diphenyl-1,4,2-diazaphospholidin-5-one 2-Oxide

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Abstract. $C_{18}H_{21}N_2O_2P$, $M_r = 328.35$, monoclinic, $P2_1/c$, $a = 9.232$ (3), $b = 14.561$ (1), $c = 12.446$ (3) Å, $\beta = 91.07$ (1)°, $V = 1673$ (2) Å^3, $Z = 4$, $D_x = 1.304$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.54$ mm^{-1}, $F(000) = 696$, room temperature, $R = 0.050$ for 2123 reflections with $I > 3\sigma(I)$. The five-membered ring is close to planar with deviations from the least-squares plane in the range −0.069 (2) to 0.075 (1) Å. The P—N and P—C distances within the ring are 1.687 (1) and 1.862 (1) Å, respectively. The phenyl group attached to P(1) adopts an axial position while the O atom and phenyl group attached to C(1) and N(1), respectively, adopt equatorial positions. Two molecules (related by a centre of symmetry) are connected by two intermolecular hydrogen bonds of length 2.879 (3) Å between N(2) and O(2), thus forming dimers in the crystal.

Experimental. A crystal with dimensions 0.3 × 0.3 × 0.3 mm was used for X-ray analysis on an Enraf–

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Table 1. *Non-H-atom fractional coordinates and displacement parameters*

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

	x	y	z	B_{eq}(Å^2)
P(1)	0.5561 (1)	0.2184 (1)	0.7747 (1)	2.53 (1)
O(1)	0.5185 (3)	0.2044 (2)	0.6599 (2)	3.65 (3)
O(2)	0.3574 (2)	0.0898 (1)	0.9893 (2)	3.43 (3)
N(1)	0.4243 (2)	0.1994 (1)	0.8642 (2)	2.83 (3)
N(2)	0.5776 (3)	0.0833 (2)	0.9096 (2)	3.59 (4)
C(1)	0.4468 (3)	0.1209 (2)	0.9274 (2)	2.86 (3)
C(2)	0.6772 (3)	0.1299 (2)	0.8365 (2)	2.86 (3)
C(3)	0.8036 (3)	0.1722 (2)	0.9007 (3)	3.86 (5)
C(4)	0.9060 (4)	0.1017 (3)	0.9530 (4)	5.48 (7)
C(5)	0.7247 (3)	0.0616 (2)	0.7495 (3)	4.04 (5)
C(6)	0.8377 (4)	0.1013 (4)	0.6737 (3)	5.40 (7)
C(11)	0.6168 (3)	0.3333 (1)	0.8009 (2)	2.70 (3)
C(12)	0.6747 (3)	0.3822 (2)	0.7142 (2)	3.52 (4)
C(13)	0.7218 (4)	0.4718 (2)	0.7299 (3)	4.28 (5)
C(14)	0.7102 (4)	0.5129 (2)	0.8286 (4)	4.62 (6)
C(15)	0.6520 (4)	0.4655 (2)	0.9138 (3)	4.30 (5)
C(16)	0.6057 (3)	0.3750 (2)	0.9018 (2)	3.40 (4)
C(21)	0.2936 (3)	0.2521 (2)	0.8689 (2)	2.82 (3)
C(22)	0.2232 (3)	0.2764 (3)	0.7746 (2)	3.90 (5)
C(23)	0.0972 (3)	0.3321 (3)	0.7796 (3)	4.67 (6)
C(24)	0.0450 (3)	0.3584 (2)	0.8762 (4)	4.51 (6)
C(25)	0.1184 (4)	0.3335 (3)	0.9697 (3)	4.90 (6)
C(26)	0.2424 (3)	0.2799 (2)	0.9670 (2)	3.99 (5)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

P(1)—O(1)	1.479 (1)	C(11)—C(12)	1.407 (1)
P(1)—N(1)	1.687 (1)	C(11)—C(16)	1.400 (1)
P(1)—C(2)	1.862 (1)	C(12)—C(13)	1.388 (1)
P(1)—C(11)	1.793 (1)	C(13)—C(14)	1.373 (2)
O(2)—C(1)	1.226 (1)	C(14)—C(15)	1.383 (2)
N(1)—C(1)	1.402 (1)	C(15)—C(16)	1.393 (2)
N(1)—C(21)	1.432 (1)	C(21)—C(22)	1.378 (2)
N(2)—C(1)	1.348 (1)	C(21)—C(26)	1.378 (1)
N(2)—C(2)	1.470 (1)	C(22)—C(23)	1.420 (2)
C(2)—C(3)	1.532 (1)	C(23)—C(24)	1.359 (3)
C(2)—C(5)	1.540 (1)	C(24)—C(25)	1.384 (2)
C(3)—C(4)	1.532 (2)	C(25)—C(26)	1.386 (2)
C(5)—C(6)	1.533 (2)		

O(1)—P(1)—N(1)	117.19 (5)	C(2)—C(3)—C(4)	114.2 (1)
O(1)—P(1)—C(2)	115.61 (5)	C(2)—C(5)—C(6)	113.3 (1)
O(1)—P(1)—C(11)	111.76 (4)	P(1)—C(11)—C(12)	117.04 (7)
N(1)—P(1)—C(2)	92.83 (4)	P(1)—C(11)—C(16)	122.65 (7)
N(1)—P(1)—C(11)	105.03 (4)	C(12)—C(11)—C(16)	120.29 (9)
C(2)—P(1)—C(11)	112.77 (4)	C(11)—C(12)—C(13)	119.4 (1)
P(1)—N(1)—C(1)	113.75 (7)	C(12)—C(13)—C(14)	120.4 (1)
P(1)—N(1)—C(21)	123.94 (6)	C(13)—C(14)—C(15)	120.4 (1)
C(1)—N(1)—C(21)	122.01 (8)	C(14)—C(15)—C(16)	120.9 (1)
C(1)—N(2)—C(2)	119.15 (8)	C(11)—C(16)—C(15)	118.6 (1)
O(2)—C(1)—N(1)	124.02 (9)	N(1)—C(21)—C(22)	119.16 (9)
O(2)—C(1)—N(2)	124.73 (8)	N(1)—C(21)—C(26)	119.81 (9)
N(1)—C(1)—N(2)	111.24 (8)	C(22)—C(21)—C(26)	121.0 (1)
P(1)—C(2)—N(2)	101.37 (6)	C(21)—C(22)—C(23)	118.9 (1)
P(1)—C(2)—C(3)	112.53 (7)	C(22)—C(23)—C(24)	120.2 (1)
P(1)—C(2)—C(5)	109.47 (7)	C(23)—C(24)—C(25)	119.6 (1)
N(2)—C(2)—C(3)	109.96 (9)	C(24)—C(25)—C(26)	121.3 (1)
N(2)—C(2)—C(5)	109.06 (8)	C(21)—C(26)—C(25)	118.9 (1)
C(3)—C(2)—C(5)	113.72 (9)		

Nonius CAD-4 diffractometer. Accurate cell parameters were obtained from centred setting angles of 25 reflections in the range $12 < \theta < 22°$ with graphite-monochromated Cu $K\alpha$ radiation. Diffraction intensities in the range $2 < \theta < 70°$ were collected using ω–2θ scan mode, index range h $-11 \rightarrow 11$, k $0 \rightarrow 17$, l $0 \rightarrow 15$. The orientation of the crystal was automatically checked every 200 reflections and an intensity monitor was measured every 100 min of X-ray exposure. Deviation in intensity of three standard reflections was less than 1.1%. 3462 unique reflections were collected of which 2123 were considered observed [$I > 3\sigma(I)$]. The intensities were corrected for Lorentz and polarization effects; and empirical absorption correction (North, Phillips & Mathews, 1968) was applied with max. and min. values 0.9989 and 0.9803, respectively.

The structure was solved by direct methods using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All H atoms were located from $\Delta\rho$ maps. The structure was refined by full-matrix least-squares calculation on F, using unit weights with Dunitz & Seiler (1973) modified weights $\{w = 1,\ w' = w\exp[20(\sin\theta/\lambda)^2]\}$, anisotropic displacement factors for non-H atoms and isotropic for H atoms. Structure refinement converged at $R = 0.050$, $wR = 0.050$ and $S = 9.08$

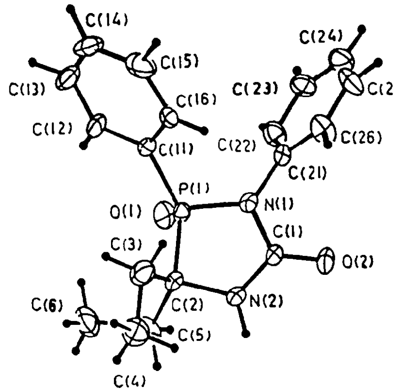

Fig. 1. Perspective drawing of $C_{18}H_{21}N_2O_2P$.

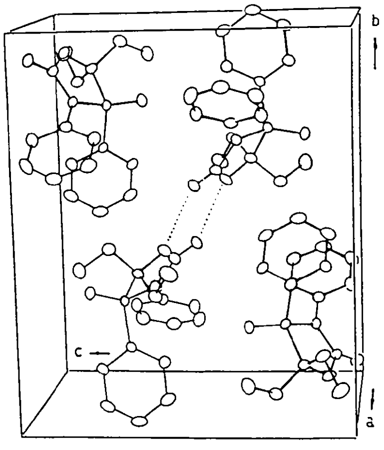

Fig. 2. Packing diagram of $C_{18}H_{21}N_2O_2P$. Dotted lines show hydrogen bonds.

for 292 variables. Maximum shift/e.s.d. in final cycle was 0.63 and no residual electron density $>|0.385|$ e Å^{-3} was found. Calculations were carried out on a PDP11/34 computer with *SDP* (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 non-H atoms are listed in Table 1* and selected bond lengths and angles in Table 2. A perspective drawing of the molecule is shown in Fig. 1 and the packing diagram in Fig. 2.

Related literature. This is the first crystal structure of a 1,4,2-diazaphospholidine derivative to be reported. The planarity of the five-membered ring is in good agreement with results reported by Hutton, Modro, Niven & Scaillet (1986) for 1,3,2-diazaphospholidine-2,4,5-trione. Similar five-membered rings with envelope and half-chair conformations have been reported by Prange, Pascard, Devillers & Navech (1977) and Marre, Sanchez, Wolf, Jaud & Galy (1984). P=O, P—N and P—C bond lengths compare well with values reported by Perales & Garcia-Blanco (1977) and Marre, Sanchez, Wolf, Jaud & Galy (1984).

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* Lists of structure factor amplitudes, anisotropic displacement parameters for non-H atoms, H-atom parameters, C—H and N—H bond lengths, and calculations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54714 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0242]

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Structure of a Dibenzo[*d*,*d*′]benzo[1,2-*b*:4,3-*b*′]dithiophene (DBBDT) Complex with 7,7,8,8-Tetracyano-*p*-quinodimethane (TCNQ)*

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Abstract. $C_{18}H_{10}S_2.C_{12}H_4N_4$, $M_r = 494.59$, monoclinic, $P2_1/c$, $a = 16.159$ (7), $b = 17.958$ (2), $c = 16.159$ (7) Å, $\beta = 96.41$ (2)°, $V = 4660$ (3) Å^3, $Z = 8$, $D_x = 1.410$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.45$ cm^{-1}, $F(000) = 2032$, $T = 294$ K, $R = 0.067$ for 2241 observed reflections. The asymmetric unit contains two non-planar DBBDT molecules and two planar TCNQ molecules. The crystal structure consists of stacks along the *a* axis of alternating DBBDT and TCNQ molecules.

Experimental. Crystals of DBBDT–TCNQ were produced by dissolving equal amounts of DBBDT and TCNQ (gradient sublimed) in hot acetonitrile followed by slow cooling. A single crystal having the form of a prism elongated along [100] with dimensions 0.33 × 0.10 × 0.13 mm was selected for data collection using an Enraf–Nonius CAD-4 diffractometer with graphite monochromator. Unit-cell

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* IUPAC name for TCNQ is 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile.

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