Related literature. The structure has a similar conformation to that of the glyoxime (Calleri, Ferraris & Viterbo, 1966), the formamide oxime (Hall, 1965) and the dimethylglyoxime (Hamilton, 1961). The latter was determined by neutron diffraction. The oncological applications of the coordination compounds of the transition elements with the oxime group justifies our interest in the structure determined.

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Structure of 5-Phenyldibenzophosphole*

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Abstract. $C_{18}H_{13}P$, $M_r = 260.3$, orthorhombic, $Pbc2_1$ [alternative setting of $Pca2_1$ (No.29)], $a = 8.486\,(2)$, $b = 12.387\,(3)$, $c = 26.244\,(5)$ Å, $V = 2759\,(1)$ Å^3, $Z = 8$, $D_x = 1.25$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.8$ cm^{-1}, $F(000) = 1088$, $T = 288$ K, $R = 0.050$ for 1512 observed reflections. The structure contains two 5-phenyldibenzophosphole molecules per asymmetric unit and these have almost identical conformations in the crystal lattice [as defined by the torsion angles C(11)—P(1*A*)—C(31)—C(32) 32.0 (5) and C(41)—P(2*A*)—C(61)—C(62) 34.1 (6)°]. The central five-membered rings in both molecules have envelope conformations, with the P atom 0.136 (15) and 0.104 (15) Å from the relevant four-C-atom plane; the dibenzophosphole moieties are both bowed slightly, with the aromatic rings bent in a direction away from the pendant phenyl ring bonded to the P atom [dihedral angles between the plane of the C atoms of the central ring and the aromatic rings are in the range 1.1 (4)–3.0 (4)°]. There is also a small rotation about the central C—C bond as shown by the relevant torsion angles which are in the range 0.2 (5)–3.3 (6)°. Difference maps show that there is disorder in the crystal lattice in that each molecule is disordered over two sites [a major and a minor one in each case; occupancies 0.89 and 0.11 for molecule (1) and 0.95 and 0.05 for molecule (2)]. For the minor sites, only the P atoms could be detected. Main dimensions are: P—C(phenyl) 1.841 (9), 1.846 (10) Å, P—C(dibenzophosphole) 1.808 (9)–1.838 (10), mean 1.819 (9) Å, phosphole ring C—P—C 88.9 (4) and 89.5 (4)°, exocyclic C—P—C 101.7 (4)–104.2 (4), mean 103.0 (4)°.

Experimental. The title compound was prepared as described previously (Affandi, Green, Hsieh, Holt, Nelson & Alyea, 1987). The material was recrystallized from ethanol and a colourless block crystal measuring 0.30 × 0.35 × 0.32 mm was selected for the diffraction study. Accurate cell dimensions and crystal orientation matrix were determined from a least-squares analysis of 25 reflections in the range $8 < \theta < 18°$. Intensities of reflections with indices h 0 to 10, k 0 to 15, l 0 to 33, with $2 < 2\theta < 54°$, were measured; ω–2θ scans; ω-scan width $(0.9 + 0.35\tan\theta)°$; graphite-monochromated Mo $K\alpha$ radiation. The intensities of three standard reflections (119, 342, 337) measured at 1 h intervals showed no significant movement or decay. Data were corrected for Lorentz and polarization effects. 3062 reflections measured, 1512 with $I > 3\sigma(I)$ were labelled 'observed'. The systematic absences ($0kl$: $k = 2n + 1$ and $h0l$: $l = 2n + 1$) allow the space group to be either $Pbcm$ (No. 57) or $Pbc2_1$ [an alternative setting of $Pca2_1$ (No. 29)]. We were unable to solve the structure in the centrosymmetric space group but the solution was readily obtained by direct methods in the non-centrosymmetric $Pbc2_1$ system. Refinement was by full-matrix least-squares calculations (on F), with H atoms allowed for (as riding atoms, C—H 0.95 Å) with anisotropic thermal parameters for P and C atoms. Difference maps calculated during the course of the refinement showed two significant

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Table 1. *Positional and thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

B_{iso} is the mean of the principal axes of the thermal ellipsoid. The site occupancy is 1.0 unless otherwise specified.

	x	y	z	B_{iso}	Occupancy
P(1*A*)	0.70517 (33)	0.70563 (17)	0.0*	2.95 (10)	0.89 (1)
P(2*A*)	0.20790 (35)	0.52606 (18)	0.29543 (10)	3.26 (10)	0.95 (1)
P(1*B*)	0.2108 (58)	0.0613 (32)	0.2973 (18)	9.8 (11)	0.11 (1)
P(2*B*)	1.2942 (35)	0.7444 (22)	0.0040 (11)	0.7 (4)	0.05 (1)
C(11)	0.8808 (10)	0.7829 (7)	0.0146 (4)	3.5 (4)	
C(12)	1.0164 (10)	0.7453 (12)	0.0393 (5)	4.5 (5)	
C(13)	1.1517 (11)	0.8105 (9)	0.0407 (5)	4.9 (5)	
C(14)	1.1473 (13)	0.9112 (9)	0.0197 (5)	5.1 (5)	
C(15)	1.0128 (12)	0.9508 (7)	−0.0047 (5)	4.2 (5)	
C(16)	0.8792 (10)	0.8852 (7)	−0.0068 (4)	3.2 (4)	
C(21)	0.6224 (11)	0.8250 (7)	−0.0301 (3)	3.6 (4)	
C(22)	0.4768 (13)	0.8365 (10)	−0.0547 (6)	5.2 (6)	
C(23)	0.4403 (17)	0.9305 (11)	−0.0790 (4)	6.0 (6)	
C(24)	0.5482 (20)	1.0122 (14)	−0.0810 (5)	6.2 (7)	
C(25)	0.6907 (15)	1.0049 (8)	−0.0576 (4)	4.9 (5)	
C(26)	0.7298 (11)	0.9090 (6)	−0.0324 (3)	3.5 (4)	
C(31)	0.5960 (12)	0.7017 (7)	0.0606 (3)	3.3 (4)	
C(32)	0.6015 (12)	0.7856 (8)	0.0957 (4)	4.6 (4)	
C(33)	0.5110 (14)	0.7830 (11)	0.1389 (6)	5.8 (7)	
C(34)	0.4152 (15)	0.6934 (12)	0.1483 (5)	6.4 (7)	
C(35)	0.4104 (16)	0.6101 (9)	0.1143 (6)	6.6 (6)	
C(36)	0.5009 (13)	0.6165 (8)	0.0712 (6)	5.4 (6)	
C(41)	0.3893 (11)	0.4514 (7)	0.2805 (3)	3.1 (4)	
C(42)	0.5205 (14)	0.4903 (10)	0.2568 (5)	4.3 (5)	
C(43)	0.6533 (12)	0.4243 (10)	0.2534 (4)	5.1 (5)	
C(44)	0.6500 (13)	0.3227 (9)	0.2746 (5)	5.1 (5)	
C(45)	0.5216 (15)	0.2843 (7)	0.2992 (6)	4.8 (5)	
C(46)	0.3886 (11)	0.3477 (7)	0.3019 (4)	3.5 (4)	
C(51)	0.1293 (11)	0.4064 (7)	0.3256 (3)	3.5 (4)	
C(52)	−0.0192 (11)	0.3935 (9)	0.3461 (5)	4.2 (5)	
C(53)	−0.0572 (15)	0.2934 (10)	0.3688 (5)	5.1 (5)	
C(54)	0.0483 (16)	0.2124 (8)	0.3711 (5)	4.8 (5)	
C(55)	0.2016 (12)	0.2239 (7)	0.3505 (4)	4.3 (4)	
C(56)	0.2385 (10)	0.3205 (6)	0.3270 (3)	3.4 (4)	
C(61)	0.0983 (12)	0.5282 (7)	0.2347 (4)	3.8 (4)	
C(62)	0.1023 (12)	0.4443 (9)	0.2000 (4)	4.9 (5)	
C(63)	0.0144 (13)	0.4497 (12)	0.1557 (6)	6.0 (7)	
C(64)	−0.0815 (18)	0.5336 (11)	0.1473 (5)	6.6 (7)	
C(65)	−0.0863 (15)	0.6194 (9)	0.1811 (5)	6.3 (6)	
C(66)	0.0016 (11)	0.6170 (8)	0.2259 (5)	4.5 (5)	

* Set at 0.0 to define the origin.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

The $C_{ar}—C_{ar}$ bond lengths are within the range 1.377 (20)–1.415 (17), mean 1.382 (16) Å.

P(1*A*)—C(11)	1.812 (9)	P(2*A*)—C(41)	1.838 (10)
P(1*A*)—C(21)	1.818 (9)	P(2*A*)—C(51)	1.808 (9)
P(1*A*)—C(31)	1.841 (9)	P(2*A*)—C(61)	1.846 (10)
C(16)—C(26)	1.465 (13)	C(46)—C(56)	1.473 (13)

C(11)—P(1*A*)—C(21)	88.9 (4)	C(41)—P(2*A*)—C(51)	89.5 (4)
C(21)—P(1*A*)—C(31)	101.7 (4)	C(51)—P(2*A*)—C(61)	101.8 (4)
C(41)—P(2*A*)—C(61)	104.1 (4)	C(11)—P(1*A*)—C(31)	104.2 (4)
P(1*A*)—C(11)—C(12)	126.7 (8)	P(2*A*)—C(41)—C(42)	127.1 (8)
P(1*A*)—C(11)—C(16)	112.9 (7)	P(2*A*)—C(41)—C(46)	111.8 (7)
C(11)—C(16)—C(26)	112.3 (7)	C(41)—C(46)—C(56)	113.1 (8)
C(15)—C(16)—C(26)	127.1 (9)	C(45)—C(46)—C(56)	127.0 (9)
P(1*A*)—C(21)—C(22)	128.6 (8)	P(2*A*)—C(51)—C(52)	127.1 (7)
P(1*A*)—C(21)—C(26)	112.1 (7)	P(2*A*)—C(51)—C(56)	112.8 (7)
C(16)—C(26)—C(21)	113.5 (8)	C(46)—C(56)—C(51)	112.6 (7)
C(16)—C(26)—C(25)	126.4 (9)	C(46)—C(56)—C(55)	126.5 (8)
P(1*A*)—C(31)—C(32)	122.4 (7)	P(2*A*)—C(61)—C(62)	123.1 (7)
P(1*A*)—C(31)—C(36)	119.8 (9)	P(2*A*)—C(61)—C(66)	116.9 (8)

peaks (1.0 and 0.7 e Å^{-3}) which were very close to atoms in the dibenzophosphole framework. We concluded that the only reasonable explanation was that each molecule was disordered over two sites (a major and a minor one, from peak heights) and that we were only able to locate the P atoms of the minor isomer in each case. Refinement of the occupancy factors for the P atoms yielded 0.89/0.11 and 0.95/0.05 for the occupancies of molecules (1) and (2) respectively. In the final refinement cycles, the P atoms of the minor sites were refined isotropically, all other non-H atoms were allowed anisotropic motion. The final refinement cycle included 350 variable parameters; $R = 0.050$, $wR = 0.063$, goodness of fit 1.31, $w = 1/[\sigma^2(F_o) + 0.0015(F_o)^2]$, maximum shift/e.s.d. < 0.02. Maximum and minimum residues in final ΔF synthesis were 0.29 and −0.27 e Å^{-3}, respectively. Scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a Silicon Graphics 4*D*-380 computer using the *NRCVAX* suite of programs (Gabe, Le Page, Charland, Lee & White, 1989). Atomic coordinates and selected bond lengths and angles are given in Tables 1 and 2 respectively.* Fig. 1 is a view of the two molecules prepared using *ORTEP*II (Johnson, 1976).

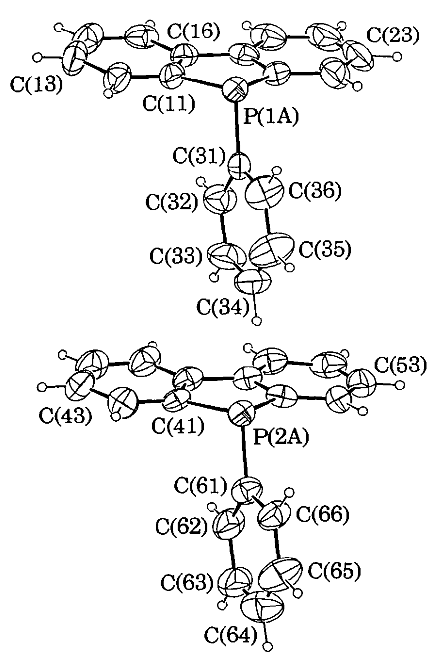

Fig. 1. A view of both 5-phenyldibenzophosphole molecules showing the numbering scheme. For clarity, H atoms are shown as small spheres of an arbitrary size and the C and P atoms are shown as thermal ellipsoids drawn at the 50% probability level.

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* Full details of molecular dimensions, anisotropic thermal parameters, calculated H-atom coordinates, torsion angles, mean-planes data, sections of difference maps showing the minor P atoms, and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54897 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0078]

Related literature. For earlier studies on phospholes and dibenzophosphole see reviews by Mathey, Fischer & Nelson (1983) and Holah, Hughes & Wright (1975). NMR (^{1}H, ^{13}C) spectroscopic characterization was reported by Nelson, Affandi, Gray & Alyea (1987). Multinuclear NMR and structural studies of coordination compounds of this ligand are of current interest (Attar, Bearden, Alcock, Alyea & Nelson, 1990; Affandi, Nelson, Alcock, Howarth, Alyea & Sheldrick, 1988; Alyea, Malito & Nelson, 1987). Structural analysis of the corresponding selenide provided an assessment of the steric properties of 1-phenylphosphole (Alyea, Ferguson, Malito & Ruhl, 1986).

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N-(4-Chlorobenzylidene)-2-(4-imidazolyl)ethylamine

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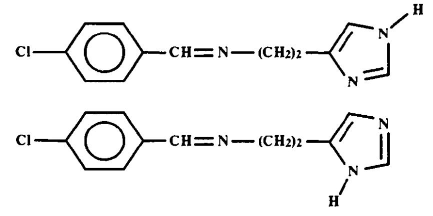
Abstract. $C_{12}H_{12}ClN_3$, $M_r = 233.7$, triclinic, $P\bar{1}$, $a = 13.768$ (2), $b = 9.947$ (2), $c = 9.825$ (3) Å, $\alpha = 110.98$ (3), $\beta = 105.54$ (2), $\gamma = 94.45$ (3)°, $V = 1187.7$ Å^3, $Z = 4$, $D_x = 1.30$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 26.8$ cm^{-1}, $F(000) = 488$, $T = 296$ K, $R = 0.054$, $wR = 0.057$, $S = 0.58$, for 1438 unique observed reflections. In both molecules of the asymmetric unit the heterocyclic ring is planar and the phenyl ring forms small dihedral angles with respect to the C7,N8,C9 (methylideneaminomethyl) plane [2.8 (5) and 1.1 (6)°]. The two independent molecules form a dimer linked by an NH···N hydrogen bond [N14—H···N14′ = 1.93 (10) Å, ∠N14—H···N14′ = 172 (8)°]. These dimers are joined to others through N12—H···N12 bonds [N12′—H···N12$^{\rm i}$ = 1.94 (9) Å, ∠N12′—H···N12$^{\rm i}$ = 172 (8)°; (i) = x, y, $z + 1$].



Experimental. Prismatic crystal 0.11 × 0.23 × 0.37 mm, recrystallized from methanol, mounted on a Siemens AED single-crystal diffractometer equipped with IBM PS2/30 computer (Belletti, Cantoni & Pasquinelli, 1988). Lattice parameters from 24 well centered reflections, $13.7 \leq \theta \leq 39.6°$, one standard reflection measured every 50 revealed a decomposition of about 32% during the data-collection time, 4506 reflections collected with a modified version of the Lehmann & Larsen (1974) method, $-14 \leq h \leq 14$, $-11 \leq k \leq 10$, $0 \leq l \leq 11$, θ range 3–70°, 1438 unique observed, $I \geq 2\sigma(I)$, data corrected for the decay and for Lp effects. Structure solved with *SHELXS*86 (Sheldrick, 1986), absorption correction following Walker & Stuart (1983) with the program *ABSORB* (Ugozzoli, 1987), 0.722 and 1.268 minimum and maximum correction in φ–μ, 0.956 and 1.103 in θ; block-matrix least-squares refinement on 396 parameters with *SHELX*76 (Sheldrick, 1976), non-H atoms anisotropic, H atoms from ΔF map, $\sum w(\Delta F)^2$ refined. Minimum and maximum peaks in

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