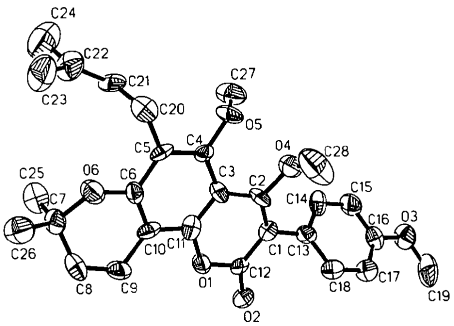

Fig. 1. The structure of di-*O*-methylscandenin. The ellipsoids are shown at the 50% probability level.

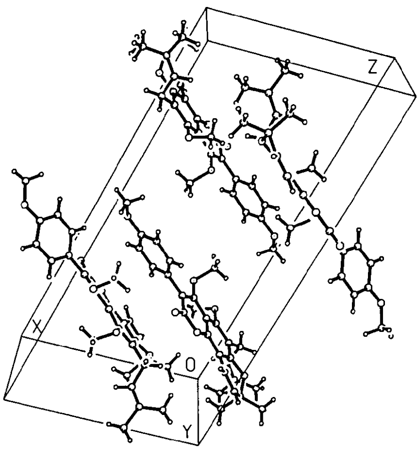

Fig. 2. The unit cell of di-*O*-methylscandenin looking down the *b* axis.

normal values (Murthy, Ramamurthy & Venkatesan, 1988; Schweizer & Dunitz, 1982; Wolska, Borowiak & Cisowski, 1991). The lengths of the C—O bonds in the pyran ring are similar to those in a variety of compounds incorporating the pyran ring (Gambardella, Mascarenhas & Santos, 1983; Jones, Kennard, Kirby & Martin, 1979; Ravikumar & Rajan, 1987).

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Structure of (*E*)-4-Hydroxyiminomethyl-1-(*p*-methoxyphenyl)imidazole

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Abstract. $C_{11}H_{11}N_3O_2$, $M_r = 217.2$, monoclinic, $P2_1/c$, $a = 9.009$ (5), $b = 7.882$ (2), $c = 15.072$ (7) Å, $\beta = 98.61$ (6)°, $V = 1058.3$ (8) Å^3, $Z = 4$, $D_x = 1.363$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.091$ mm^{-1}, $F(000) = 456$, $T = 293$ (3) K, $R = 0.046$, $wR = 0.036$, $S = 1.99$, 4592 unique observed reflections with $F_o > 6\sigma(F_o)$. The structure consists of a monoclinic packing of a hydroxyimino oxime. The angle between the imidazole and phenyl groups is 43.3°. The mean deviations from the imidazole and phenyl planes are 0.0013 and 0.0035 Å, respectively. The molecules related by symmetry are linked by two hydrogen bonds between the hydroxy O atom and the unsubstituted N of the imidazole. C and N of the

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^4$)

	x	y	z	U_{eq}*
C(1)	1654 (3)	−3246 (3)	−1315 (2)	383 (7)
C(2)	856 (2)	−2969 (3)	−611 (2)	375 (7)
C(3)	1239 (3)	−1582 (3)	−45 (2)	370 (7)
C(4)	2387 (2)	−511 (3)	−193 (2)	314 (6)
C(10)	3182 (3)	−803 (3)	−898 (2)	370 (7)
C(11)	2809 (3)	−2164 (3)	−1456 (2)	398 (8)
N(5)	2760 (2)	934 (2)	380 (1)	331 (6)
C(6)	2913 (3)	964 (3)	1294 (2)	410 (8)
N(7)	3333 (2)	2457 (2)	1620 (1)	371 (6)
C(8)	3459 (2)	3458 (3)	880 (1)	297 (6)
C(9)	3098 (2)	2552 (3)	114 (2)	340 (6)
O(81)	5061 (2)	7446 (2)	1676 (1)	442 (5)
C(81)	3987 (2)	5202 (3)	971 (1)	320 (6)
N(81)	4613 (2)	5761 (2)	1727 (1)	370 (6)
O(11)	1361 (2)	−4554 (2)	−1923 (1)	579 (7)
C(12)	151 (4)	−5624 (4)	−1865 (2)	729 (12)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°) *with their standard deviations*

C(1)—C(2)	1.385 (4)	N(5)—C(9)	1.384 (3)
C(1)—C(11)	1.387 (3)	N(7)—C(8)	1.384 (3)
C(1)—O(11)	1.378 (3)	C(8)—C(9)	1.356 (3)
C(2)—C(3)	1.398 (3)	C(8)—C(81)	1.454 (3)
C(3)—C(4)	1.378 (3)	O(81)—N(81)	1.394 (2)
C(4)—C(10)	1.386 (4)	C(81)—N(81)	1.273 (3)
C(4)—N(5)	1.439 (3)	O(11)—C(12)	1.392 (4)
C(10)—C(11)	1.374 (3)	C(6)—N(7)	1.309 (3)
N(5)—C(6)	1.364 (3)		
C(2)—C(1)—C(11)	120.5 (2)	C(4)—N(5)—C(9)	126.7 (2)
C(2)—C(1)—O(11)	123.9 (2)	C(6)—N(5)—C(9)	106.4 (2)
C(11)—C(1)—O(11)	115.5 (2)	N(5)—C(6)—N(7)	112.0 (2)
C(1)—C(2)—C(3)	118.8 (2)	C(1)—O(11)—C(12)	119.0 (2)
C(2)—C(3)—C(4)	120.2 (2)	C(6)—N(7)—C(8)	105.3 (2)
C(3)—C(4)—C(10)	120.6 (2)	N(7)—C(8)—C(9)	110.4 (2)
C(3)—C(4)—N(5)	120.1 (2)	N(7)—C(8)—C(81)	121.9 (2)
C(10)—C(4)—N(5)	119.3 (2)	C(9)—C(8)—C(81)	127.6 (2)
C(4)—C(10)—C(11)	119.5 (2)	N(5)—C(9)—C(8)	105.9 (2)
C(1)—C(11)—C(10)	120.4 (2)	C(8)—C(81)—N(81)	120.4 (2)
C(4)—N(5)—C(6)	126.8 (2)	O(81)—N(81)—C(81)	111.9 (2)

hydroxyiminomethyl substituent are linked by a double bond. The bond lengths and angles are normal and do not reflect delocalization.

Experimental. The synthesis of this compound has already been described (Herrador, 1980). X-ray-quality crystals were prepared by recrystallization from an ethanol solution. A pale white plate-like crystal with dimensions 0.114 × 0.57 × 0.06 mm was used for the X-ray diffractometry. Data collection: Siemens *R3m/V* four-circle diffractometer, highly oriented graphite-crystal-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). Accurate cell parameters were obtained by least-squares fit for 25 reflections measured by a $+\omega$ and $-\omega$ scans in the range $12 < 2\theta < 30°$. Intensity data collection: 293 K, minimum and maximum measuring time 0.5 and 2 s with scan range 1.00° plus $K\alpha$ separation. 2θ range from 3.58 to 60°, index ranges $-12 \leq h \leq 12$, $-11 \leq k \leq 11$, $-21 \leq l \leq 21$, Friedel pairs at $-\omega$. 12 810 reflections collected, $R_{int} = 0.02$, of which 4592 were unique and observed with $F_o > 6\sigma(F_o)$ using ω–2θ mode. Background measurement: stationary counter at beginning and end of scan. Four standard reflections (111, 201, 306, 422) monitored after every 120 min showed no crystal movement or decay. Intensities were corrected for Lorentz–polarization and absorption effects with ψ data-collection method (8 reflections, $\Delta\psi$ 10°). Phase problems were solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1989). Atomic scattering factors used were taken from the program package. All calculations were performed on a MicroVAX II computer. Refinement method: full-matrix least squares. All H atoms were located in difference F synthesis and refined isotropically. Final $R = 0.046$, $wR = 0.036$, weighting scheme: $w^{-1} = \sigma[(F) + 0.004F]$. $S = 1.99$. Maximum $\Delta/\sigma = 0.3$. The final difference Fourier map showed maximum negative and positive peaks of -0.12 and 0.16 e Å^{-3}, respectively.

Final atomic parameters for non-H atoms are given in Table 1.* Fig. 1 shows the structure with the atom labelling. Bond lengths and angles are given in Table 2.

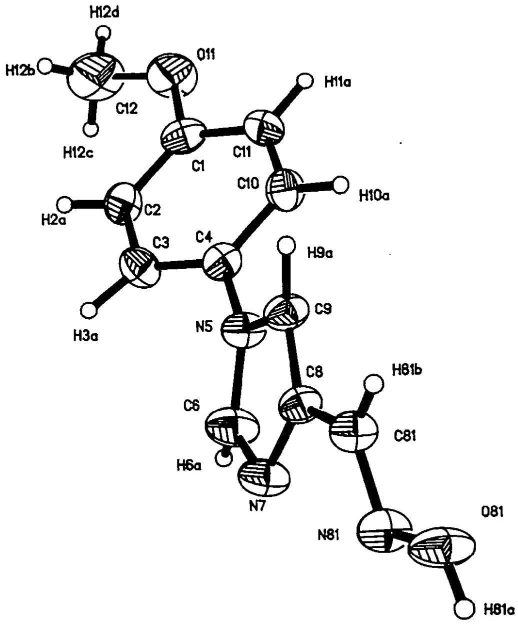

Fig. 1. *XP*-routine *SHELXTL-Plus* plot of the molecule showing the atomic numbering scheme. Thermal ellipsoids are plotted at the 50% probability level. H atoms are shown with arbitrary radii.

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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 54769 (70 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0007]

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}, λ(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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