stabilized in addition by van der Waals and non-bonded interactions.

This structure determination formed the summer project of Mr Louis Leone, a summer student in our department in 1983. My thanks are due to him for his participation in this project and also to Ms J. Mann for technical help. My thanks are also due to Dr E. Shefter for the generous gift of the sample used in this study and to Dr R. Parthasarathy for use of the crystallographic facilities. My thanks are also due to the referees and the Co-editor for their constructive criticisms and suggestions.

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Structure of (3-Isopropyl-1-methyl-4-pyrazolyl)diphenylphosphine Oxide

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Abstract. $C_{19}H_{21}N_2OP$, $M_r = 324{\cdot}36$, monoclinic, $P2_1/n$, $a = 10{\cdot}936\ (2)$, $b = 9{\cdot}431\ (2)$, $c = 17{\cdot}708\ (3)$ Å, $\beta = 107{\cdot}62\ (2)°$, $V = 1740{\cdot}7\ (1{\cdot}2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}237$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}124$ mm^{-1}, $F(000) = 688$, $T = 296$ K, final $R_F = 0{\cdot}036$ for 1757 observed independent reflections. The coordination around P is distorted tetrahedral with the P—C bond to the pyrazole ring [1·777 (2) Å] slightly shorter than to the phenyl rings [1·804 (3) and 1·807 (3) Å]. The pyrazole and phenyl rings are planar within 0·03 Å.

Introduction. The method of preparation of the compound has been described by Ben Akacha, Baccar & Ayed (1983). The formula and the conformation of the molecule were established by elemental analysis,* mass spectrometry and ^{1}H, ^{31}P and ^{13}C NMR.

As pyrazole derivatives are used in therapy and the phosphine oxide pyrazoles have interesting properties (Ben Akacha, 1987), we decided to carry out an X-ray structure analysis in order to determine the precise conformation of the molecule and the coordination and the geometry of the ligands around the phosphorus ion.

Experimental. A transparent crystal with irregular shape *ca* 0·13 × 0·33 × 0·36 mm was selected. Precession photographs indicated monoclinic symmetry (Boutiba, 1984). The cell constants were refined from setting angles of 25 reflections. 2747 integrated independent intensities were measured on a CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The range of hkl was $h\ 0 \rightarrow 12$, $k\ 0 \rightarrow 10$, $l\ -19 \rightarrow 19$ up to $2\theta = 46°$, corresponding to $(\sin\theta)/\lambda = 0{\cdot}59$ Å^{-1}. $\omega/2\theta$ scan was used. The standard reflections measured every 2·5 h remained stable. The merging of measured reflections gave 1757 with $I > 2{\cdot}5\sigma(I)$, which were used in the structure analysis. The intensities were corrected for Lorentz–polarization and absorption (empirical by ψ scan). The relative transmission was in the range 0·94–1·0. The structure was solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refinement on F was by *SHELX*76 (Sheldrick, 1976). All H atoms were located in the difference Fourier map and then refined with the same overall isotropic thermal parameter. The maximum and minimum heights in the final difference Fourier synthesis were 0·27 and −0·25 e Å^{-3}. The weighting scheme used was $w(F) = 1/[\sigma^2(F) + 0{\cdot}00146F^2]$ where $\sigma(F)$ was the estimated standard deviation on F. In the last cycle of refinement

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* Centre National d'Analyse, Vernaison, France: registry No. SCA 3137.

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

	x	y	z	B_{eq}*(Å^2)
P	0·2441 (1)	0·0759 (1)	0·6279 (0)	3·02 (10)
O	0·2399 (2)	−0·0402 (2)	0·5706 (1)	3·96 (8)
C(11)	0·2493 (2)	0·2487 (3)	0·5890 (1)	3·02 (7)
C(12)	0·3376 (2)	0·2976 (3)	0·5508 (1)	3·29 (11)
C(13)	0·1750 (3)	0·3651 (3)	0·5907 (1)	3·01 (11)
N(11)	0·3176 (2)	0·4324 (2)	0·5303 (1)	3·60 (9)
N(12)	0·2169 (2)	0·4722 (2)	0·5553 (1)	3·25 (9)
C(21)	0·1056 (2)	0·0739 (3)	0·6632 (1)	3·02 (10)
C(22)	0·1129 (3)	0·0677 (3)	0·7426 (2)	3·98 (12)
C(23)	0·0028 (4)	0·0524 (3)	0·7643 (2)	4·96 (10)
C(24)	−0·1147 (3)	0·0441 (3)	0·7078 (2)	5·29 (12)
C(25)	−0·1237 (3)	0·0504 (3)	0·6286 (2)	4·70 (15)
C(26)	−0·0146 (3)	0·0662 (3)	0·6064 (2)	3·79 (12)
C(31)	0·3857 (2)	0·0640 (3)	0·7126 (1)	3·07 (9)
C(32)	0·4579 (3)	−0·0581 (3)	0·7235 (2)	3·70 (12)
C(33)	0·5712 (3)	−0·0693 (4)	0·7851 (2)	4·80 (14)
C(34)	0·6123 (3)	0·0412 (4)	0·8359 (2)	4·87 (15)
C(35)	0·5414 (3)	0·1630 (4)	0·8270 (2)	4·97 (15)
C(36)	0·4275 (3)	0·1761 (3)	0·7653 (2)	4·29 (12)
C(4)	0·1705 (4)	0·6171 (3)	0·5422 (2)	4·48 (12)
C(5)	0·4467 (3)	0·2179 (3)	0·5349 (4)	4·93 (15)
C(6)	0·5740 (4)	0·2736 (7)	0·5886 (3)	8·46 (25)
C(7)	0·4427 (4)	0·2290 (6)	0·4495 (4)	7·49 (15)

* $B_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

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

P—O	1·484 (2)	C(25)—C(26)	1·371 (4)
P—C(11)	1·777 (2)	C(26)—C(21)	1·396 (4)
P—C(21)	1·804 (3)	C(31)—C(32)	1·377 (4)
P—C(31)	1·807 (3)	C(32)—C(33)	1·386 (4)
C(11)—C(12)	1·412 (3)	C(33)—C(34)	1·362 (5)
C(12)—N(11)	1·323 (3)	C(34)—C(35)	1·368 (5)
N(11)—N(12)	1·357 (3)	C(35)—C(36)	1·394 (4)
N(12)—C(13)	1·340 (3)	C(36)—C(31)	1·392 (4)
C(13)—C(11)	1·372 (4)	C(12)—C(5)	1·507 (4)
C(21)—C(22)	1·385 (4)	N(12)—C(4)	1·452 (4)
C(22)—C(23)	1·378 (4)	C(5)—C(6)	1·524 (6)
C(23)—C(24)	1·373 (5)	C(5)—C(7)	1·503 (2)
C(24)—C(25)	1·377 (5)		
C(11)—P—O	114·2 (1)	C(11)—P—C(21)	105·8 (1)
C(21)—P—O	111·9 (1)	C(11)—P—C(31)	105·0 (1)
C(31)—P—O	111·4 (1)	C(21)—P—C(31)	108·1 (1)

$(\Delta/\sigma)_{max} = 0·01$ and $S = 1·0$. Atomic scattering factors and values of f' and f'' for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974). The calculations were performed on a VAX 11/780 of the Khawarezmi Computer Center of Tunis.

Discussion. The positional and thermal parameters are reported in Table 1, bond lengths and angles at phosphorus in Table 2.* A view of the molecule is shown in Fig. 1 (Johnson, 1965) with atom labelling and thermal ellipsoids with 50% probability. The packing of the molecules in the unit cell is shown in Fig. 2.

The coordination around P is distorted tetrahedral with a mean value of 106·3 (1)° for the C—P—C bond angles. The C—P—O angles are about 112° if the C atom belongs to a phenyl ring and 114° for the C atom belonging to the pyrazole ring. The distortion may be due to electrostatic interaction and steric repulsion between the oxygen atom and the rings. The bond lengths and angles indicate that the two phenyl C atoms are symmetrically disposed relative to the pyrazole carbon.

The P—C bonds to the phenyl rings, 1·807 (3) Å, are longer than the corresponding bond to the pyrazole

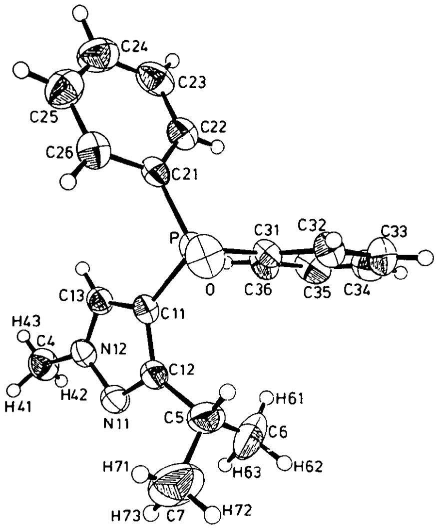

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule with labelling and numbering of atoms. Except for H atoms, thermal ellipsoids are shown with 50% probability.

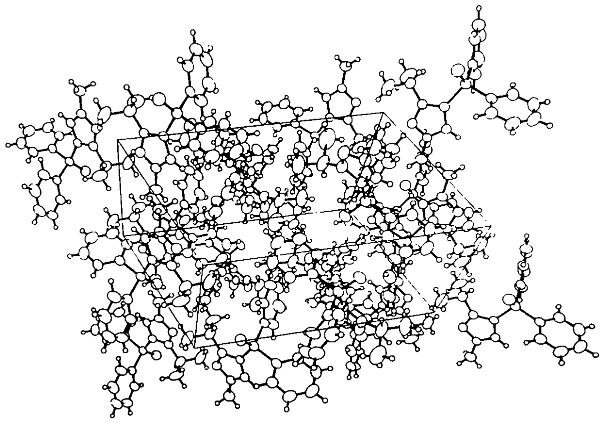

Fig. 2. *ORTEP* view (Johnson, 1965) of the molecules in the unit cell of $C_{19}H_{21}N_2OP$.

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters and bond angles (except at phosphorus) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44382 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ring, 1·777 (2) Å. This difference suggests that 3*d* orbitals on the P atom may interact to a small extent with the π system of the pyrazole ring. As suggested by Cobbledick & Einstein (1975) the small but significant differences between C—C bonds [0·042 (7) Å] and the two C—N bonds [0·017 (6) Å] in the pyrazole ring support this view. The P—O bond is 1·484 (2) Å which makes the coordination tetrahedra more distorted. The C—C distances in the isopropyl group are about 1·51 Å, which is normal for single bonds. The two phenyl and the pyrazole rings are planar with small deviations and the phenyl-ring planes are orthogonal.

The C—C distances in the phenyl rings are in the range 1·362 (5)–1·394 (4) Å as expected for phenyl (Cobbledick & Einstein, 1975; Rodulfo de Gil, Valentina Rivera & Noguera, 1977).

We are indebted to Dr C. J. Gilmore for helpful discussion.

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Structure of 2,5-Dihydro-4-[(4-methylphenyl)amino]-2-oxo-*N*-phenyl-3-furancarboxamide

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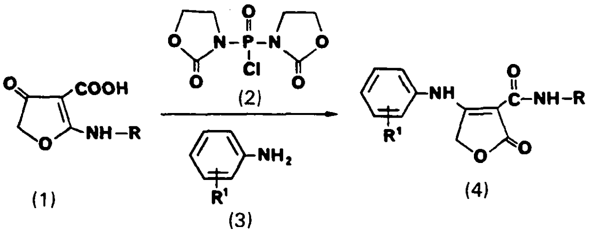
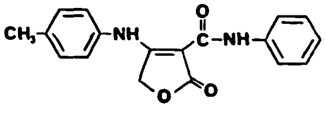
Abstract. $C_{18}H_{16}N_2O_3$, $M_r = 308{\cdot}34$, monoclinic, $C2/c$, $a = 19{\cdot}482\ (3)$, $b = 7{\cdot}642\ (1)$, $c = 21{\cdot}695\ (3)$ Å, $\beta = 110{\cdot}87\ (1)°$, $V = 3018{\cdot}1\ (8)$ Å^3, $Z = 8$, $D_x = 1{\cdot}357$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}88$ cm^{-1}, $F(000) = 1296$, $T = 297$ K, final $R = 0{\cdot}045$ for 1856 unique observed reflections. The title compound was derived from a 4,5-dihydro-4-oxo-2-(phenylamino)-3-furancarboxylic acid *via* a novel 3(2*H*)-furanone–2(5*H*)-furanone rearrangement. The amide and amine N atoms participate in the conjugated π system and the N—H's participate in intramolecular hydrogen bonding.

Introduction. Recently (Mack & Georgiev, 1987), we reported a novel 3(2*H*)-furanone–2(5*H*)-furanone rearrangement which was accomplished by treating 2-(*N*-substituted amino)-4,5-dihydro-4-oxo-3-furancarboxylic acids (1) with 1 equivalent of di(2-oxo-1,3-oxazolidin-3-yl)phosphinic chloride (2) and an appropriately substituted aromatic amine (3), in the presence of triethylamine. The rearrangement resulted in the facile synthesis of a new class of 2(5*H*)-furanone amides (4). An X-ray crystal-structure determination of 2,5-dihydro-4-[(4-methylphenyl)amino]-2-oxo-*N*-phenyl-3-furancarboxamide (4*a*) was undertaken to define unambiguously the structures of 2(5*H*)-furanone amides (4).





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