Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($\times 10^4$) *of the non-H atoms*

$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	$U_{eq}(\text{Å}^2)$
O(1)	1791 (5)	4447 (1)	3583 (2)	355 (7)
C(2)	1888 (8)	4525 (2)	1998 (3)	380 (9)
C(3)	3236 (8)	4013 (1)	1131 (3)	333 (9)
C(4)	4674 (7)	3351 (1)	1853 (3)	311 (9)
C(5)	4468 (7)	3296 (1)	3518 (3)	306 (9)
C(6)	3062 (7)	3833 (1)	4310 (3)	299 (9)
O(7)	5921 (6)	2886 (1)	1016 (2)	451 (8)
O(8)	3310 (7)	4118 (1)	−432 (2)	502 (9)
C(9)	2628 (9)	3867 (2)	6041 (3)	353 (11)
O(10)	4499 (6)	3324 (1)	6940 (2)	387 (8)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(2)	1·360 (1)	C(4)—O(7)	1·244 (1)
O(1)—C(6)	1·352 (1)	C(5)—C(4)	1·431 (2)
C(3)—C(2)	1·337 (2)	C(5)—C(6)	1·343 (2)
C(3)—O(8)	1·346 (2)	C(9)—C(6)	1·502 (2)
C(4)—C(3)	1·444 (2)	C(9)—O(10)	1·406 (2)

C(6)—O(6)—C(2)	119·4 (1)	O(7)—C(4)—C(3)	119·4 (1)
O(1)—C(2)—C(3)	121·9 (1)	C(4)—C(5)—C(6)	121·0 (1)
C(2)—C(3)—C(4)	120·8 (1)	O(1)—C(6)—C(9)	109·8 (1)
C(4)—C(3)—O(8)	119·4 (1)	C(9)—C(6)—C(5)	128·2 (1)
O(8)—C(3)—C(2)	119·7 (1)	C(5)—C(6)—O(1)	122·1 (1)
C(3)—C(4)—C(5)	114·8 (1)	C(6)—C(9)—O(10)	113·1 (1)
C(5)—C(4)—O(7)	125·9 (1)		

formed using an M4030-1 computer, Slovak Technical University, Bratislava, Czechoslovakia, with *SHELX*76 (Sheldrick, 1976). Structure and atomic numbering shown in Fig. 1* (drawn by use of *ORTEP;* Johnson, 1965). Projection of crystal structure in Fig. 2 (Pavelčik, Kettmann & Majer, 1985). Fractional atomic coordinates for non-H atoms are in Table 1; bond distances and angles are in Table 2.

Related literature. Kojic acid was prepared by transformation of saccharides with cultivated Aspergillus tamaril strain (Uher, 1987). Kojic acid can be isolated by thickening of the ultrafiltered medium, followed by crystallization from methanol, acetone or ethyl acetate.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53194 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Structure of a Phosphinohydrazone

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Abstract. $C_{27}H_{20}ClN_2O_3P$, $M_r = 486·9$, orthorhombic, $P2_12_12_1$, $a = 5·3832$ (4), $b = 15·884$ (2), $c = 27·67$ (1) Å, $V = 2366$ (1) Å^3, $Z = 4$, $D_x = 1·37$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1·5418$ Å (graphite monochromator), $\mu = 23·3$ cm^{-1}, $F(000) = 1008$, $T = 293$ K, final $R = 0·042$ for 1911 reflections with $I >$

Table 1. *Atomic coordinates, equivalent isotropic temperature factors and e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cl	0·4065 (3)	-0·00163 (9)	0·46510 (5)	6·80 (8)
P	0·2645 (3)	0·03232 (8)	0·13735 (5)	3·85 (6)
O1	0·5218 (6)	0·0601 (2)	0·1433 (1)	4·4 (2)
O7	0·2287 (6)	-0·0655 (2)	0·1293 (1)	4·7 (2)
O8	0·1191 (7)	0·0678 (2)	0·0923 (1)	4·1 (2)
N15	0·083 (1)	0·0559 (3)	0·1828 (2)	4·1 (2)
N16	0·1755 (7)	0·0399 (2)	0·2283 (1)	3·7 (2)
C1	0·369 (1)	-0·1186 (3)	0·0997 (2)	4·0 (3)
C2	0·575 (1)	-0·0938 (4)	0·0738 (2)	5·1 (3)
C3	0·702 (1)	-0·1529 (4)	0·0461 (2)	5·8 (4)
C4	0·628 (1)	-0·2350 (4)	0·0458 (2)	5·9 (4)
C5	0·420 (2)	-0·2590 (4)	0·0715 (2)	5·8 (4)
C6	0·291 (1)	-0·2014 (3)	0·0982 (2)	4·6 (3)
C9	0·117 (1)	0·1549 (3)	0·0826 (2)	3·7 (2)
C10	0·302 (1)	0·1895 (4)	0·0557 (2)	4·6 (3)
C11	0·295 (1)	0·2747 (4)	0·0449 (2)	5·1 (3)
C12	0·101 (1)	0·3236 (3)	0·0609 (2)	4·9 (3)
C13	-0·089 (1)	0·2876 (4)	0·0870 (2)	5·3 (3)
C14	-0·081 (1)	0·2029 (4)	0·0980 (2)	4·5 (3)
C17	0·055 (1)	0·0720 (3)	0·2640 (2)	3·6 (2)
C18	0·141 (1)	0·0532 (3)	0·3133 (2)	3·8 (3)
C19	0·349 (1)	0·0037 (4)	0·3218 (2)	4·8 (3)
C20	0·432 (1)	-0·0138 (3)	0·3680 (2)	5·0 (3)
C21	0·306 (1)	0·0201 (3)	0·4066 (2)	4·4 (3)
C22	0·101 (1)	0·0690 (3)	0·4005 (2)	5·6 (3)
C23	0·021 (1)	0·0860 (3)	0·3535 (2)	4·8 (3)
C24	-0·163 (1)	0·1247 (3)	0·2580 (2)	4·0 (3)
C25	-0·329 (1)	0·1735 (3)	0·2533 (2)	4·2 (3)
C26	-0·525 (1)	0·2342 (3)	0·2492 (2)	3·8 (2)
C27	-0·617 (1)	0·2708 (4)	0·2902 (2)	6·1 (4)
C28	-0·802 (1)	0·3300 (4)	0·2879 (3)	6·9 (4)
C29	-0·897 (1)	0·3517 (4)	0·2443 (3)	6·4 (4)
C30	-0·811 (1)	0·3158 (4)	0·2043 (2)	6·1 (4)
C31	-0·621 (1)	0·2579 (4)	0·2057 (2)	5·3 (3)

$3\sigma(I)$. The structure provides the first crystallographic data for a molecule containing the $(R—O)_2P(=O)N—$ unit.

Experimental. Yellowish crystals of 3-phenyl-1-(4-chlorophenyl)-2-propyn-1-one diphenylphosphinohydrazone from ethanol; $0·1 \times 0·2 \times 0·4$ mm specimen for X-ray measurements; Picker FACS-I diffractometer; cell parameters from 9 reflections centered manually at $\pm 2\theta$ in the range $25·7 < 2\theta < 42·6°$; intensity data measured with $2\theta - \theta$ scan; 2θ scan speed of $2°$ min^{-1}; 2θ scan range of $(1·6 + 0·29\tan\theta)°$; 20 s background measurements made at the ends of the scans; 4 standard reflections measured at 200 reflection intervals; 2393 data (includes 104 standards and systematically absent data) measured from $\theta = 2 - 126°$; index range for h, k, l of 0–6, 0–17, 0–31; 2188 unique data without systematically absent data; 1911 reflections with $I > 3\sigma(I)$; average change in standard intensities of $0·1\%$ with range of $-1·0$ to $+1·9\%$; absorption ignored; structure solved with the *SAYTAN–MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) direct-methods procedures; refinement by full-matrix least squares with anisotropic temperature factors for C, N and O and isotropic terms for H; $\Sigma[1/\sigma(F_o)]^2(F_o - F_c)^2$ minimized; secondary extinction (Zachariasen, 1968) refined; final R, wR

Table 2. *Bond lengths (Å), angles (°) and e.s.d.'s in parentheses*

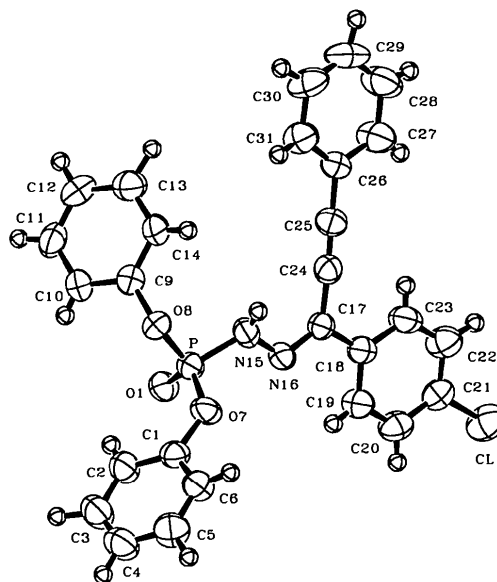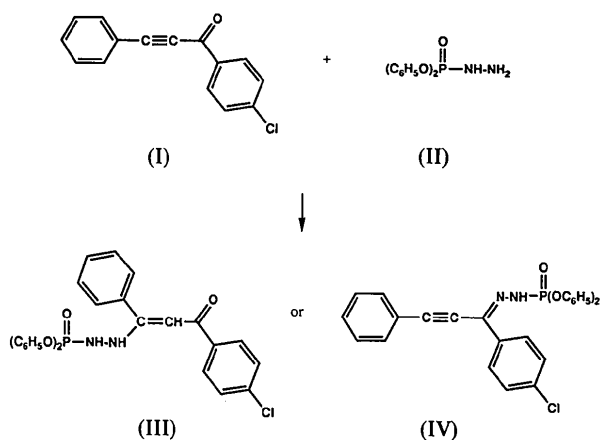|			
Cl—C21	1·740 (5)	C12—C13	1·377 (8)
P—O1	1·463 (3)	C13—C14	1·381 (7)
P—O8	1·575 (3)	C17—C24	1·453 (6)
P—O7	1·581 (3)	C17—C18	1·468 (6)
P—N15	1·636 (5)	C18—C23	1·389 (7)
O7—C1	1·397 (5)	C18—C19	1·390 (7)
O8—C9	1·409 (5)	C19—C20	1·380 (7)
N15—N16	1·377 (6)	C20—C21	1·374 (7)
N16—C17	1·286 (6)	C21—C22	1·360 (7)
C1—C2	1·379 (7)	C22—C23	1·397 (8)
C1—C6	1·379 (6)	C24—C25	1·192 (6)
C2—C3	1·392 (7)	C25—C26	1·433 (7)
C3—C4	1·365 (8)	C26—C31	1·364 (7)
C4—C5	1·378 (9)	C26—C27	1·365 (7)
C5—C6	1·365 (8)	C27—C28	1·372 (9)
C9—C10	1·361 (7)	C28—C29	1·354 (9)
C9—C14	1·379 (7)	C29—C30	1·328 (8)
C10—C11	1·386 (7)	C30—C31	1·376 (8)
C11—C12	1·375 (8)		
O1—P—O8	116·8 (2)	C9—C14—C13	119·5 (6)
O1—P—O7	115·3 (2)	N16—C17—C24	123·2 (4)
O1—P—N15	114·1 (3)	N16—C17—C18	118·4 (5)
O8—P—O7	100·3 (2)	C24—C17—C18	118·4 (5)
O8—P—N15	103·3 (2)	C23—C18—C19	116·8 (6)
O7—P—N15	105·1 (2)	C23—C18—C17	121·5 (5)
C1—O7—P	127·3 (3)	C19—C18—C17	121·7 (5)
C9—O8—P	120·4 (3)	C20—C19—C18	122·1 (6)
N16—N15—P	116·5 (4)	C21—C20—C19	118·9 (6)
C17—N16—N15	116·6 (4)	C22—C21—C20	121·6 (6)
C2—C1—C6	120·1 (5)	C22—C21—Cl	118·7 (5)
C2—C1—O7	124·5 (5)	C20—C21—Cl	119·6 (4)
C6—C1—O7	115·4 (5)	C21—C22—C23	118·6 (6)
C1—C2—C3	119·3 (6)	C18—C23—C22	121·9 (6)
C4—C3—C2	120·2 (7)	C25—C24—C17	174·6 (5)
C3—C4—C5	120·0 (7)	C24—C25—C26	177·5 (6)
C6—C5—C4	120·3 (6)	C31—C26—C27	118·6 (6)
C5—C6—C1	120·1 (6)	C31—C26—C25	122·1 (5)
C10—C9—C14	120·9 (5)	C27—C26—C25	119·2 (6)
C10—C9—O8	119·7 (5)	C26—C27—C28	121·0 (7)
C14—C9—O8	119·3 (5)	C29—C28—C27	119·4 (7)
C9—C10—C11	119·5 (6)	C30—C29—C28	120·0 (6)
C12—C11—C10	120·2 (7)	C29—C30—C31	121·6 (6)
C11—C12—C13	119·9 (6)	C26—C31—C30	119·3 (6)
C12—C13—C14	119·9 (6)		

Fig. 1. *ORTEP* drawing of (IV). The C, N and O atoms are displayed as 50% ellipsoids and the H atoms as $B = 1·5$ Å^2 spheres.

and goodness-of-fit = 0·042, 0·031, 1·82; maximum Δ/σ = 0·51; maximum and minimum values in final difference map of 0·23 and −0·23 e Å⁻³. The preliminary crystallographic calculations were performed on a UNISYS 1100/92 computer with the *XRAY*76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); final calculations were carried out with the *TEXSAN* program system (*TEXSAN*, 1989) on a Digital Equipment Corp. MicroVAX II computer. Atomic coordinates and isotropic temperature factors are listed in Table 1; bond lengths and angles are given in Table 2.* An *ORTEP* drawing (Johnson, 1965) is shown in Fig. 1; the *PLOTMD* program (Luo, Ammon & Gilliland, 1989) was used to label the drawing and prepare a file which was printed on a Hewlett-Packard Laserjet II printer.

Related literature. Phosphino-hydrazine (II) could react with acetylenic ketone (I) *via* a Michael addition to yield enone (III) or to form hydrazone (IV) (El-Sayed, Refatt, Ammon, Vlassi, Fouli & Heiba, 1988). This determination has confirmed the product as hydrazone (IV). A search of the Cambridge Structural Database current to December 1989 revealed no other examples of a molecule containing the $(R{-}O)_2P(={O})N{-}$ moiety.

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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 53202 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 8-Chloro-1,2,3,4-tetrahydrocinnolin-4-one and its 2-Chloroacetyl Derivative

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Abstract. (1) $C_8H_7ClN_2O$, m.p. 397–398 K, M_r = 182·6, orthorhombic, *Pbca*, a = 4·873 (1), b = 13·438 (2), c = 25·526 (3) Å, V = 1671·5 Å³, Z = 8, D_x = 1·451 Mg m⁻³, λ(Cu $K\alpha$) = 1·54178 Å, μ = 3·7 mm⁻¹, $F(000)$ = 752, T = 293 K, final R = 0·045 for 1479 observed reflections. (2) $C_{10}H_8Cl_2N_2O_2.\frac{1}{2}H_2O$, M_r = 268·1, orthorhombic, *Pbcn*, a = 18·740 (3), b = 8·839 (2), c = 14·124 (2) Å, V = 2339·5 Å³, Z = 8, D_x = 1·522 Mg m⁻³, λ(Cu $K\alpha$) = 1·54178 Å, μ = 5·05 mm⁻¹, $F(000)$ = 1096, T = 293 K, final R = 0·051 for 1941 observed reflections. The structures were solved by direct methods. Enhanced conjugation of N(1) lone-pair electrons with the benzene π system in (1) is evidenced by shortening of the C(benzene)—N(1) bond to 1·370 (2) compared with 1·400 (3) Å in (2). The hybridization state of the N(2) atom in (2) is sp^2 [the sum of appropriate bond angles at N(2) is 359·2°]