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*Acta Cryst.* (1992). **C48**, 1866–1868

# Structure of *cis,cis*-4,6-Diphenyl-2-(2-propenyl)-1,3-dioxa-2-phosphorinane 2-Oxide

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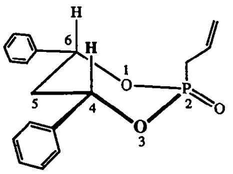
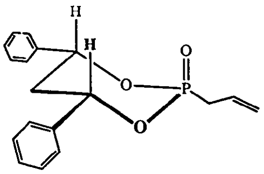
**Abstract.** $C_{18}H_{19}O_3P$, $M_r = 314.32$, monoclinic, $P2_1/n$, $a = 5.726$ (6), $b = 14.234$ (7), $c = 20.08$ (1) Å, $\beta = 92.54$ (7)°, $V = 1635$ (4) Å$^3$, $Z = 4$, $D_x = 1.277$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.71$ cm$^{-1}$, $F(000) = 664$, $T = 296$ K, $R = 0.042$, 1813 unique observed reflections. The X-ray structure determination of the title compound shows that the dioxaphosphorinane ring has a chair conformation in which the phosphoryl O atom (P=O) is equatorial, which explains the absence of substantial NMR deshielding by its P=O group on H(4) and H(6), which are axial.

**Experimental.** The title compound (I) was prepared from the reaction of *meso*-(*R*,*S*)-1,3-diphenyl-1,3-propanediol with 2-propenylphosphonyl dichloride (Kinnear & Perren, 1952) in the presence of 2.2 equivalents of triethylamine in methylene chloride (52% yield). A mixture of 1.28:1 of (I) and its isomer, *trans*$^{2,4}$,*cis*$^{4,6}$-4,6-diphenyl-2-(2-propenyl)-1,3-dioxa-2-phosphorinane 2-oxide (II) (NMR indicates this phosphoryl O atom is in the axial position), was formed. Well formed crystals of (I) were obtained from column chromatographic separation (silica gel; ether/hexane 2/1) followed by recrystallization from methylene chloride. Crystal (I) m.p. 461–463 K; isomer (II) m.p. 362–364 K (the crystals were not suitable for X-ray analysis).



(I)



(II)

The chemical shift of the C(4) and C(6) H atoms of (I) [$\delta$ 5.52 (*dt*); in $CDCl_3$] is at a higher field than those of (II) [$\delta$ 5.81 (*ddd*)] indicating the deshielding effect of the P=O group in (II) on its C(4) and C(6) H atoms.

The crystal used for data collection was 0.43 × 0.35 × 0.22 mm, colorless and equant. Data were

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å$^2$)

$B_{eq} = (8\pi^2/3)[U_{22} + (1/\sin^2\beta)(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| O(1) | 0.0949 (4) | 0.6692 (2) | 0.6746 (1) | 3.5 (1) |
| P(2) | 0.1697 (1) | 0.63227 (6) | 0.74656 (4) | 2.99 (4) |
| O(3) | 0.0947 (3) | 0.7121 (1) | 0.7956 (1) | 3.1 (1) |
| C(4) | −0.1395 (6) | 0.7525 (2) | 0.7857 (2) | 3.3 (2) |
| C(5) | −0.1667 (6) | 0.7892 (2) | 0.7149 (2) | 3.7 (2) |
| C(6) | −0.1336 (6) | 0.7131 (3) | 0.6625 (2) | 3.7 (2) |
| O(7) | 0.4203 (4) | 0.6129 (2) | 0.7536 (1) | 4.3 (1) |
| C(8) | −0.1670 (6) | 0.8280 (2) | 0.8368 (2) | 3.0 (1) |
| C(9) | −0.0074 (6) | 0.8988 (3) | 0.8457 (2) | 4.3 (2) |
| C(10) | −0.0365 (8) | 0.9689 (3) | 0.8910 (2) | 5.2 (2) |
| C(11) | −0.2307 (8) | 0.9678 (3) | 0.9290 (2) | 5.5 (2) |
| C(12) | −0.3909 (7) | 0.8978 (4) | 0.9205 (2) | 5.8 (2) |
| C(13) | −0.3602 (6) | 0.8277 (3) | 0.8745 (2) | 4.4 (2) |
| C(14) | −0.1427 (6) | 0.7544 (3) | 0.5933 (2) | 3.7 (2) |
| C(15) | 0.0337 (7) | 0.8100 (3) | 0.5717 (2) | 5.0 (2) |
| C(16) | 0.017 (1) | 0.8518 (4) | 0.5108 (2) | 6.6 (3) |
| C(17) | −0.176 (1) | 0.8394 (4) | 0.4702 (2) | 7.1 (3) |
| C(18) | −0.353 (1) | 0.7840 (4) | 0.4894 (2) | 6.9 (3) |
| C(19) | −0.3404 (7) | 0.7402 (3) | 0.5518 (2) | 5.1 (2) |
| C(20) | −0.0089 (6) | 0.5325 (2) | 0.7625 (2) | 3.5 (2) |
| C(21) | 0.0330 (7) | 0.4966 (3) | 0.8313 (2) | 5.0 (2) |
| C(22) | −0.118 (1) | 0.4973 (3) | 0.8768 (2) | 7.5 (3) |

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

| | | | |
|---|---|---|---|
| O(1)—P(2) | 1.579 (3) | C(4)—C(5) | 1.516 (5) |
| O(1)—C(6) | 1.461 (4) | C(4)—C(8) | 1.500 (4) |
| P(2)—O(3) | 1.576 (2) | C(5)—C(6) | 1.527 (5) |
| P(2)—O(7) | 1.461 (3) | C(6)—C(14) | 1.508 (5) |
| P(2)—C(20) | 1.787 (3) | C(20)—C(21) | 1.483 (5) |
| O(3)—C(4) | 1.465 (4) | C(21)—C(22) | 1.287 (6) |
| | | | |
| P(2)—O(1)—C(6) | 120.0 (2) | C(4)—C(5)—C(6) | 113.1 (3) |
| O(1)—P(2)—O(3) | 105.2 (1) | O(1)—C(6)—C(5) | 109.0 (3) |
| O(1)—P(2)—O(7) | 112.1 (1) | O(1)—C(6)—C(14) | 108.3 (3) |
| O(1)—P(2)—C(20) | 107.1 (2) | C(5)—C(6)—C(14) | 111.0 (3) |
| O(3)—P(2)—O(7) | 111.7 (1) | C(4)—C(8)—C(9) | 121.8 (3) |
| O(3)—P(2)—C(20) | 106.7 (1) | C(4)—C(8)—C(13) | 119.3 (3) |
| O(7)—P(2)—C(20) | 113.6 (2) | C(6)—C(14)—C(15) | 121.4 (3) |
| P(2)—O(3)—C(4) | 118.1 (2) | C(6)—C(14)—C(19) | 119.3 (4) |
| O(3)—C(4)—C(5) | 108.6 (3) | P(2)—C(20)—C(21) | 111.7 (3) |
| O(3)—C(4)—C(8) | 108.1 (3) | C(20)—C(21)—C(22) | 125.0 (4) |
| C(5)—C(4)—C(8) | 112.8 (3) | | |

collected with a Rigaku AFC-5*S* diffractometer using graphite-monochromated Mo $K\alpha$ radiation, $\omega$ scans, and a scan speed of 4° min$^{-1}$ (in $\omega$). Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to improve accuracy. The lattice parameters were obtained from a least-squares fit of 25 strong reflections in the $2\theta$ range 12–22°. A total of 3328 reflections were measured [$h$ 0 to 6, $k$ 0 to 16, $l$ −23 to 23, $(\sin\theta/\lambda)_{\max} = 0.60$ Å$^{-1}$], 319 reflections were measured twice ($R_{\text{int}} = 0.017$ based on $F^2$), and 1196 reflections (including 142 systematically extinct reflections) were considered unobserved [$I < 3\sigma(I)$], leaving a data set comprised of 1813 unique observed reflections. Three standard reflections ($1\bar{2}0$, 103, $1\bar{1}\bar{1}$) changed by 0.0, 0.0 and −0.2%, respectively; no decay correction was applied. The data were corrected for Lorentz and polarization; no absorption correction was applied. The direct methods program *SHELXS*86 (Sheldrick, 1985) provided the locations of all non-H atoms. Full-matrix least-squares refinement was performed to minimize $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/\text{Lp}^2$ ($S$ = scan rate, $C$ = total integrated peak count, $R$ = ratio of scan time to background counting time, $B$ = total background count, Lp = Lorentz–polarization factor and $p$ = 0.03). After convergence, trigonal planar, methylene, tetrahedral and terminal vinyl H atoms were placed at assumed positions (C—H = 0.95 Å, $B = 1.2 \times B_{eq}$ of associated C atom) and fixed. The final stages of refinement were performed with 200 variables including all non-H positional and anisotropic thermal parameters, one scale factor, and a secondary-

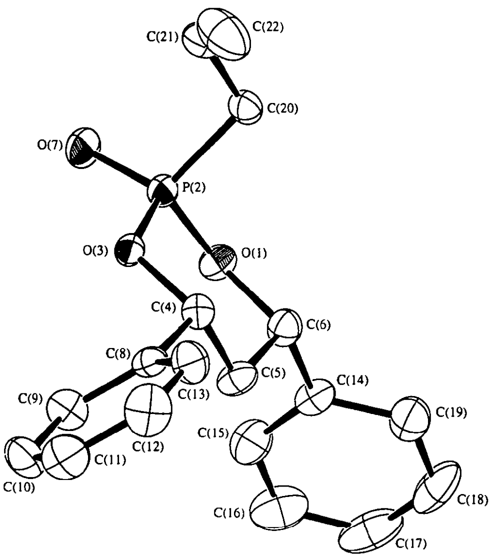

Fig. 1. Molecular configuration and atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level. H atoms have been omitted to improve clarity.

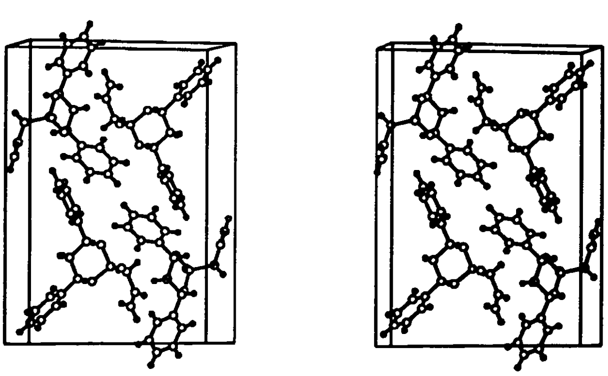

Fig. 2. Stereoscopic illustration of the molecular packing. The origin is at the lower-left-back, the *a* axis is out of the plane of the paper, *b* is horizontal and *c* is vertical.

extinction coefficient [0.4 (2) × $10^{-6}$]. Convergence yielded $R = 0.042$, $wR = 0.052$, $S = 1.75$ and $(\Delta/\sigma)_{max} = 0.001$. Final difference synthesis produced $(\Delta\rho)_{max} = 0.30$ and $(\Delta\rho)_{min} = -0.17$ e Å$^{-3}$. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). All computer programs used were from *TEXSAN* (Molecular Structure Corporation, 1985). Table 1 lists the final atomic coordinates and equivalent isotropic temperature factors while Table 2* presents selected interatomic distances and angles. Fig. 1 shows the atom-numbering scheme and the thermal motion. The 1,3,2-dioxaphosphorinane ring is in the chair conformation and the phosphoryl O atom is equatorial. Fig. 2 illustrates the packing of the molecules within the unit cell.

**Related literature.** As part of the NMR study of five- and six-membered-ring organophosphorus compounds (Hua, Chan-Yu-King, McKie & Myer, 1987), (I) was prepared for studies of the deshielding effect of the P=O group on the adjacent H atoms [*i.e.* C(4) and C(6) H atoms] (*cf.* Cooper, Hall, Harrison & Inch, 1977). Although various crystal structures of 1,3,2-dioxaphosphorinanes are known (Grand & Robert, 1978), compound (I), with 4,6-diphenyl and 2-allyl substitution, has not been previously reported.

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* Lists of structure factors, H-atom parameters, anisotropic thermal parameters, phenyl-ring bond distances and angles, intermolecular distances, torsion angles, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55185 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0403]

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# Structure of a *p*-Nitrobenzoyl Praziquantel Analogue*

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**Abstract.** 2-(*p*-Nitrobenzoyl)-1,2,3,6,7,11b-hexahydro-4*H*-pyrazino[2,1-*a*]isoquinolin-4-one, $C_{19}H_{17}N_3O_4$, $M_r = 351.4$, monoclinic, $C2/c$, $a = 22.704$ (5), $b = 10.330$ (2), $c = 14.808$ (3) Å, $\beta = 105.79$ (3)°, $V = 3341.9$ (6) Å$^3$, $Z = 8$, $D_x = 1.40$ g cm$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.54178 Å, $\mu = 8.0$ cm$^{-1}$, $F(000) = 1472$, $T = 293$ K, final $R = 0.038$ for 1875 unique observed reflections $[F_o > 3\sigma(F_o)]$. The whole molecule adopts a folded shape with the two carbonyls of the amide groups *syn*-related [angle between planes 26.6 (1)°]. The phenyl group in the isoquinoline moiety is planar, while the hydropyrazine and tetrahydropyridine moieties have a sofa conformation with C(1) 0.64 (1) and C(6) 0.66 (1) Å out of the planes formed by the rest of the ring-forming atoms. The nitro and amide [N(2)—C(12)—O(2)—C(13)] groups are tilted 4.7 (1) and 128 (1)° from the planar *p*-substituted phenyl group. Crystal packing results from van der Waals interactions.

**Experimental.** The title compound was synthetized by treatment of the product of hydrolysis of praziquantel with *p*-nitrobenzoyl chloride. A light-yellow plate-like crystal of dimensions 0.12 × 0.20 × 0.40 mm showed systematic absences *hkl* for *h* + *k*

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