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}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ 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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Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3025 (1)	2504 (2)	9131 (1)	38 (1)
N(2)	3240 (1)	3771 (1)	8939 (1)	40 (1)
C(3)	2779 (1)	4778 (2)	8858 (1)	46 (1)
C(4)	2144 (1)	4388 (2)	8331 (1)	41 (1)
O(1)	1754 (1)	5238 (1)	8106 (1)	55 (1)
N(5)	2020 (1)	3122 (1)	8138 (1)	42 (1)
C(6)	1413 (1)	2726 (2)	7590 (2)	53 (1)
C(7)	1157 (1)	1733 (2)	8131 (2)	60 (1)
C(7a)	1600 (1)	638 (2)	8442 (1)	45 (1)
C(8)	1397 (1)	−578 (2)	8632 (1)	58 (1)
C(9)	1787 (1)	−1612 (2)	8877 (1)	57 (1)
C(10)	2398 (1)	−1443 (2)	8930 (1)	51 (1)
C(11)	2610 (1)	−256 (2)	8748 (1)	44 (1)
C(11a)	2219 (1)	806 (2)	8518 (1)	39 (1)
C(11b)	2486 (1)	2103 (2)	8326 (1)	37 (1)
C(12)	3824 (1)	4115 (2)	8999 (1)	43 (1)
O(2)	3977 (1)	5255 (1)	9003 (1)	61 (1)
C(13)	4282 (1)	3063 (2)	9039 (1)	41 (1)
C(14)	4179 (1)	2071 (2)	8381 (1)	45 (1)
C(15)	4612 (1)	1125 (2)	8428 (1)	50 (1)
C(16)	5148 (1)	1184 (2)	9139 (1)	50 (1)
N(1)	5601 (1)	133 (2)	9222 (2)	69 (1)
O(3)	5463 (1)	−746 (2)	8666 (2)	99 (1)
O(4)	6065 (1)	185 (2)	9834 (2)	116 (1)
C(17)	5269 (1)	2174 (2)	9789 (2)	59 (1)
C(18)	4830 (1)	3121 (2)	9728 (1)	54 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—N(2)	1.451 (2)	C(1)—C(11b)	1.516 (2)
N(2)—C(3)	1.458 (2)	N(2)—C(12)	1.352 (2)
C(3)—C(4)	1.494 (2)	C(4)—O(1)	1.227 (2)
C(4)—N(5)	1.352 (2)	N(5)—C(6)	1.456 (2)
N(5)—C(11b)	1.464 (2)	C(6)—C(7)	1.510 (3)
C(7)—C(7a)	1.500 (3)	C(7a)—C(8)	1.394 (3)
C(7a)—C(11a)	1.390 (3)	C(8)—C(9)	1.372 (3)
C(9)—C(10)	1.378 (3)	C(10)—C(11)	1.370 (3)
C(11)—C(11a)	1.393 (2)	C(11a)—C(11b)	1.529 (2)
C(12)—O(2)	1.228 (2)	C(12)—C(13)	1.493 (3)
C(13)—C(14)	1.389 (3)	C(13)—C(18)	1.379 (2)
C(14)—C(15)	1.375 (3)	C(15)—C(16)	1.377 (2)
C(16)—N(1)	1.477 (3)	C(16)—C(17)	1.379 (3)
N(1)—O(3)	1.209 (3)	N(1)—O(4)	1.190 (3)
C(17)—C(18)	1.381 (3)		

N(2)—C(1)—C(11b)	109.6 (1)	C(1)—N(2)—C(3)	112.8 (1)
C(1)—N(2)—C(12)	126.8 (1)	C(3)—N(2)—C(12)	119.2 (1)
N(2)—C(3)—C(4)	114.8 (1)	C(3)—C(4)—O(1)	118.1 (2)
C(3)—C(4)—N(5)	119.1 (2)	O(1)—C(4)—N(5)	122.7 (2)
C(4)—N(5)—C(6)	120.1 (1)	C(4)—N(5)—C(11b)	123.9 (1)
C(6)—N(5)—C(11b)	115.4 (1)	N(5)—C(6)—C(7)	109.7 (2)
C(6)—C(7)—C(7a)	110.7 (2)	C(7)—C(7a)—C(8)	120.7 (2)
C(7)—C(7a)—C(11a)	120.7 (2)	C(8)—C(7a)—C(11a)	118.6 (2)
C(7a)—C(8)—C(9)	122.0 (2)	C(8)—C(9)—C(10)	118.9 (2)
C(9)—C(10)—C(11)	120.3 (2)	C(10)—C(11)—C(11a)	121.2 (2)
C(7a)—C(11a)—C(11)	119.0 (2)	C(7a)—C(11a)—C(11b)	122.5 (1)
C(11)—C(11a)—C(11b)	118.5 (2)	C(1)—C(11b)—N(5)	109.9 (1)
C(1)—C(11b)—C(11a)	111.7 (1)	N(5)—C(11b)—C(11a)	111.3 (1)
N(2)—C(12)—O(2)	121.6 (2)	N(2)—C(12)—C(13)	118.1 (2)
O(2)—C(12)—C(13)	120.3 (2)	C(12)—C(13)—C(14)	121.7 (1)
C(12)—C(13)—C(18)	118.4 (2)	C(14)—C(13)—C(18)	119.8 (2)
C(13)—C(14)—C(15)	120.5 (2)	C(14)—C(15)—C(16)	118.5 (2)
C(15)—C(16)—N(1)	118.7 (2)	C(15)—C(16)—C(17)	122.2 (2)
N(1)—C(16)—C(17)	119.0 (2)	C(16)—N(1)—O(3)	116.8 (2)
C(16)—N(1)—O(4)	118.9 (2)	O(3)—N(1)—O(4)	124.3 (2)
C(16)—C(17)—C(18)	118.5 (2)	C(13)—C(18)—C(17)	120.5 (2)

odd, $h0l$ for l odd and $00l$ for l odd. A Nicolet *P3F* diffractometer with Ni-filtered Cu $K\alpha$ radiation was used for data collection. Unit-cell parameters were determined by least-squares fitting of 25 reflections ($8.10 < 2\theta < 29.56°$). Intensity data collection ($h = 0$ to 24, $k = 0$ to 10, $l = -15$ to 15) was by $2\theta/\theta$ scans ($3 < 2\theta < 110°$) with variable scan speed (minimum 4.0, maximum 30.0° min^{-1}). Two standards ($0\bar{2}3$, $5\bar{1}\bar{2}$) measured every 50 reflections showed decay of $< 3\%$, which was ignored. 2346 reflections were collected, of which 2104 were unique and 229 unobserved [$F_o < 3\sigma(F_o)$]. Lp corrections were applied, absorption was ignored. The structure was solved by direct methods. 236 parameters were refined by block-diagonal least squares, including positional and thermal parameters for non-H atoms, a scale factor and a secondary-extinction parameter $\chi = 0.0014$ (2) [$F_c^* = F_c\,(1.0 + 0.002\chi F_c^2/\sin 2\theta)^{-0.25}$]. H atoms with a fixed $U = 0.06$ Å^2 were forced to ride on the C atom to which they were bonded. Final refinement cycles converged to $R = 0.038$ and $wR = 0.049$ with $S = 1.43$. $\sum w(\Delta F)^2$ was the function minimized, where $w = [\sigma(F_o)^2 + 0.001(F_o)^2]^{-1}$. Maximum shift/e.s.d. = 0.868. Residuals in the final difference Fourier map were 0.14 and -0.13 e Å^{-3}. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed using the *SHELXTL* package (Sheldrick, 1983) and *CSU* (Vicković, 1988). Table

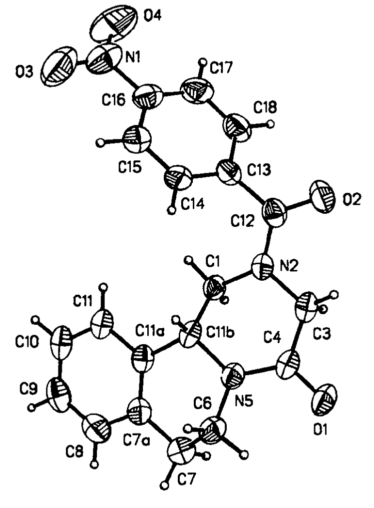

Fig. 1. The molecular conformation of the title compound showing the atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

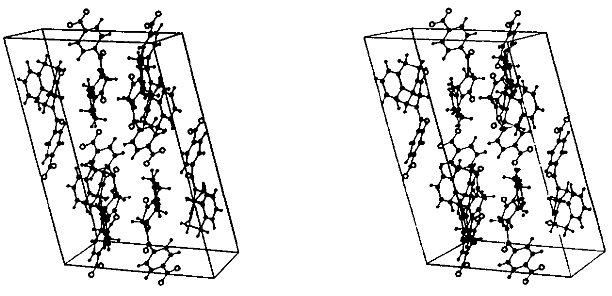

Fig. 2. Stereopacking of the molecule viewed along the *b* axis.

1* lists the final fractional coordinates and the equivalent isotropic thermal parameters. In Table 2 the bond lengths and bond angles are listed. The molecular conformation with the numbering scheme and the crystal packing are shown in Figs. 1 and 2.

Related literature. The conformation observed in this work differs notably from that of the *m*-nitrobenzoyl derivative of praziquantel (Toscano, Rubio, Cetina & Pérez-Ibarra, 1991), while it correlates well with the predicted minimum-energy conformation calculated by molecular mechanics for praziquantel and some alkyl and cycloalkyl derivatives (Rubio, Escobar-Valderrama & Cetina, 1990).

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55175 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0582]

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Structure of 10-Acetyloxy-10-methyl-9(10*H*)-anthracenone

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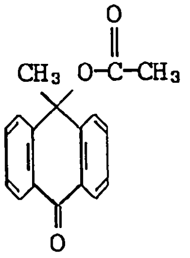
Abstract. 10-Methyl-9-oxo-9,10-dihydroanthracen-10-yl acetate, $C_{17}H_{14}O_3$, $M_r = 266.30$, monoclinic, $P2_1/c$, $a = 7.912$ (1), $b = 27.749$ (8), $c = 13.402$ (3) Å, $\beta = 107.09$ (2)°, $V = 2812$ (1) Å^3, $Z = 8$, $D_x = 1.258$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 0.08$ mm^{-1}, $F(000) = 1119.87$, $T = 298$ K, final $R = 0.046$ for 1825 reflections with $I > 2\sigma(I)$. There are two crystallographically independent molecules in the asymmetric unit, but they are structurally similar. The structure determination confirms that the title compound is one of the products of oxidation of 9-methylanthracene by potassium dichromate.

Experimental. A solution of 9-methylanthracene (3.85 g, 20 mmol) in acetic acid/acetic anhydride (100 ml, 1:1) was cooled to 273 K. Potassium dichromate (15.95 g, 54 mmol) was dissolved in equal volumes of acetic acid and acetic anhydride (100 ml), then added dropwise to the prepared solution of 9-methylanthracene at 273 K. Stirring was continued for 24 h at room temperature. The mixture was poured into 1000 ml of stirred cold water and extracted with hot chloroform. The combined extracts were distilled to yield pale-yellow needles (4.17 g) which were separated on a column chromatograph (silica gel). The two products obtained were recrystallized from ethanol separately. One is 9,10-anthraquinone (54%), the other is 10-acetyloxy-10-methyl-9(10*H*)-anthracenone (I) (37%) whose crystal structure is described.



(I)

A thin colourless plate crystal of dimensions 0.15 × 0.40 × 0.40 mm was selected for study. Cell constants were derived from least-squares refinement of 25 reflections having $19 < 2\theta < 24°$. Intensity data were collected with the $\omega/2\theta$-scan technique with $0 < 2\theta < 45°$ ($h = -8$ to 8, $k = 0$ to 29, $l = 0$ to 14) on a Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Scan rate was

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