

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

Pd—P(1)	2.251 (1)	Pd—P(2)	2.255 (1)
Pd—O(1)	2.065 (2)	Pd—O(3)	2.068 (2)
P(1)—C(11)	1.814 (4)	P(1)—C(21)	1.826 (4)
P(1)—C(31)	1.825 (4)	P(2)—C(41)	1.821 (4)
P(2)—C(51)	1.815 (4)	P(2)—C(61)	1.826 (3)
O(1)—C(1)	1.282 (4)	O(2)—C(1)	1.223 (5)
O(3)—C(2)	1.297 (5)	O(4)—C(2)	1.216 (5)
C(1)—C(2)	1.551 (5)	C(11)—C(12)	1.512 (5)
C(21)—C(22)	1.510 (6)	C(31)—C(32)	1.519 (5)
C(41)—C(42)	1.513 (5)	C(51)—C(52)	1.520 (6)
C(61)—C(62)	1.513 (5)		
P(1)—Pd—P(2)	96.3 (0.4)	P(1)—Pd—O(1)	89.8 (1)
P(2)—Pd—O(1)	172.4 (1)	P(1)—Pd—O(3)	169.7 (1)
P(2)—Pd—O(3)	93.1 (1)	O(1)—Pd—O(3)	81.1 (1)
Pd—P(1)—C(11)	113.9 (1)	Pd—P(1)—C(21)	106.9 (1)
C(11)—P(1)—C(21)	105.2 (2)	Pd—P(1)—C(31)	119.0 (1)
C(11)—P(1)—C(31)	105.5 (2)	C(21)—P(1)—C(31)	105.2 (2)
Pd—P(2)—C(41)	115.4 (1)	Pd—P(2)—C(51)	107.2 (1)
C(41)—P(2)—C(51)	106.0 (2)	Pd—P(2)—C(61)	117.5 (1)
C(41)—P(2)—C(61)	104.8 (2)	C(51)—P(2)—C(61)	104.9 (2)
Pd—O(1)—C(1)	113.1 (2)	Pd—O(3)—C(2)	112.7 (2)
O(1)—C(1)—O(2)	123.1 (3)	O(1)—C(1)—C(2)	116.6 (3)
O(2)—C(1)—C(2)	120.3 (3)	O(3)—C(2)—O(4)	124.4 (3)
O(3)—C(2)—C(1)	115.4 (3)	O(4)—C(2)—C(1)	120.1 (3)
P(1)—C(11)—C(12)	115.2 (3)	P(1)—C(21)—C(22)	113.3 (3)
P(1)—C(31)—C(32)	116.2 (3)	P(2)—C(41)—C(42)	114.2 (3)
P(2)—C(51)—C(52)	116.8 (3)	P(2)—C(61)—C(62)	113.7 (3)

Table 1 gives the atomic coordinates, Table 2 bond distances and angles. Fig. 1 shows the atomic numbering.

Related literature. Modes of oxalato ligand coordination have been reviewed (Scott, Wieghardt & Sykes, 1973). UV irradiation of the title complex

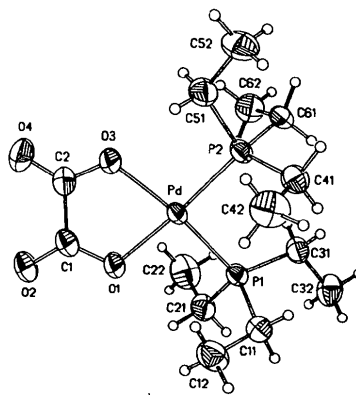


Fig. 1. Structure and labeling scheme for the title complex with 50% probability ellipsoids.

eliminates two molecules of CO₂ and provides a convenient source for Pd(PEt₃)₂ (Paonessa, Prignano & Trogler, 1985).

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Acta Cryst. (1987). **C43**, 168–170

Structure of 4-Benzamido-1-benzoyl-2,3-didehydro-1,2,4-triazolidine

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Abstract. C₁₆H₁₄N₄O₂, *M_r* = 294.31, monoclinic, *P*2₁/*c*, *a* = 14.604 (2), *b* = 10.339 (1), *c* = 9.714 (1) Å, β = 102.56 (1)°, *V* = 1431.7 (3) Å³, *Z* = 4, *D_x* = 1.365 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, μ = 0.728 mm⁻¹, *F*(000) = 616, *T* = 295 K, final *R* = 0.039, *wR* = 0.050 for 2117 independent observed reflections. The triazoline ring is essentially planar with a maximum deviation from a least-squares plane through the ring of 0.04 Å; however, the ring N atom with the benzamido substituent is pyramidal. Bond distances and angles are normal. An intermolecular

hydrogen bond (2.93 Å) occurs between the secondary amine and the benzoyl O atom.

Experimental. Colorless 0.15 × 0.25 × 0.40 mm crystal, from 1:1 benzene/chloroform. Synthesized by G. Kumar and J. Boyer of the University of Illinois at Chicago, m.p. = 489–491 K. Automated Nicolet *R3m* diffractometer with incident-beam graphite monochromator, 25 centered reflections within 30 < 2θ < 60° used for determining lattice parameters. Data corrected for Lorentz and polarization effects, ab-

Table 1. Atom coordinates ($\times 10^4$) and temperature coefficients ($\text{\AA}^2 \times 10^3$)

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

	x	y	z	U_{eq}
N(1)	6108 (1)	2954 (1)	4735 (1)	51 (1)
N(2)	6239 (1)	1619 (1)	5002 (1)	54 (1)
C(3)	5651 (1)	1073 (2)	4013 (2)	51 (1)
N(4)	5148 (1)	1875 (1)	2981 (1)	48 (1)
C(5)	5367 (1)	3204 (2)	3479 (2)	52 (1)
C(6)	6550 (1)	3930 (2)	5525 (2)	49 (1)
C(7)	7345 (1)	3709 (2)	6767 (2)	48 (1)
C(8)	7600 (1)	2536 (2)	7426 (2)	66 (1)
C(9)	8343 (2)	2463 (2)	8580 (2)	78 (1)
C(10)	8846 (1)	3549 (2)	9087 (2)	74 (1)
C(11)	8594 (1)	4714 (2)	8451 (2)	73 (1)
C(12)	7848 (1)	4800 (2)	7310 (2)	62 (1)
O(6)	6294 (1)	5045 (1)	5160 (1)	63 (1)
N(13)	4207 (1)	1595 (1)	2424 (1)	50 (1)
C(14)	3586 (1)	1569 (1)	3273 (2)	44 (1)
O(14)	3839 (1)	1708 (1)	4556 (1)	53 (1)
C(15)	2579 (1)	1378 (1)	2558 (2)	46 (1)
C(16)	2213 (1)	1773 (2)	1187 (2)	58 (1)
C(17)	1265 (1)	1634 (2)	614 (2)	67 (1)
C(18)	681 (1)	1097 (2)	1396 (2)	67 (1)
C(19)	1047 (1)	684 (2)	2752 (2)	69 (1)
C(20)	1992 (1)	822 (2)	3333 (2)	58 (1)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

N(1)—N(2)	1.410 (2)	N(1)—C(5)	1.467 (2)
N(1)—C(6)	1.345 (2)	N(2)—C(3)	1.274 (2)
C(3)—N(4)	1.383 (2)	N(4)—C(5)	1.468 (2)
N(4)—N(13)	1.393 (2)	C(6)—C(7)	1.498 (2)
C(6)—O(6)	1.240 (2)	C(7)—C(8)	1.384 (2)
C(7)—C(12)	1.385 (2)	C(8)—C(9)	1.383 (3)
C(9)—C(10)	1.373 (3)	C(10)—C(11)	1.367 (3)
C(11)—C(12)	1.377 (2)	N(13)—C(14)	1.353 (2)
C(14)—O(14)	1.228 (2)	C(14)—C(15)	1.496 (2)
C(15)—C(16)	1.385 (2)	C(15)—C(20)	1.384 (3)
C(16)—C(17)	1.382 (2)	C(17)—C(18)	1.377 (3)
C(18)—C(19)	1.378 (3)	C(19)—C(20)	1.380 (2)
N(2)—N(1)—C(5)	111.8 (1)	N(2)—N(1)—C(6)	126.9 (1)
C(5)—N(1)—C(6)	121.2 (1)	N(1)—N(2)—C(3)	104.7 (1)
N(2)—C(3)—N(4)	116.5 (1)	C(3)—N(4)—C(5)	106.2 (1)
C(3)—N(4)—N(13)	118.6 (1)	C(5)—N(4)—N(13)	116.6 (1)
N(1)—C(5)—N(4)	100.2 (1)	N(1)—C(6)—C(7)	122.4 (1)
N(1)—C(6)—O(6)	117.2 (1)	C(7)—C(6)—O(6)	120.3 (1)
C(6)—C(7)—C(8)	126.1 (1)	C(6)—C(7)—C(12)	115.9 (1)
C(8)—C(7)—C(12)	118.0 (1)	C(7)—C(8)—C(9)	120.5 (2)
C(8)—C(9)—C(10)	120.7 (2)	C(9)—C(10)—C(11)	119.2 (2)
C(10)—C(11)—C(12)	120.6 (2)	C(7)—C(12)—C(11)	121.0 (2)
N(4)—N(13)—C(14)	120.2 (1)	N(13)—C(14)—O(14)	121.5 (1)
N(13)—C(14)—C(15)	116.2 (1)	O(14)—C(14)—C(15)	122.3 (2)
C(14)—C(15)—C(16)	122.9 (2)	C(14)—C(15)—C(20)	117.7 (1)
C(16)—C(15)—C(20)	119.4 (1)	C(15)—C(16)—C(17)	120.0 (2)
C(16)—C(17)—C(18)	120.4 (2)	C(17)—C(18)—C(19)	119.6 (2)
C(18)—C(19)—C(20)	120.3 (2)	C(15)—C(20)—C(19)	120.2 (2)

sorption ignored. $(\text{Sin}\theta/\lambda)_{\text{max}} = 0.59 \text{ \AA}^{-1}$, range of hkl : $-17 \leq h \leq 4$, $0 \leq k \leq 11$, $-11 \leq l \leq 11$. Standards 800, 040, 004, monitored every 60 reflections with random variation 2.4% over data collection, θ - 2θ mode, scan width $(2.0 + \Delta_{\alpha_1\alpha_2})$, scan rate a function of count rate (8° min^{-1} minimum, $30^\circ \text{ min}^{-1}$ maximum), 3408 reflections measured, 2437 unique, $R_{\text{int}} = 0.014$, 2117 observed [$F_o > 3\sigma(F_o)$].

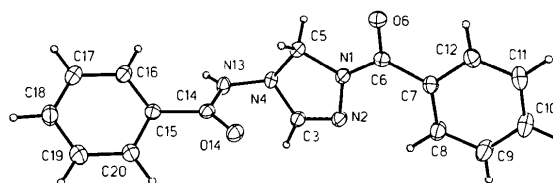


Fig. 1. Thermal-ellipsoid plot drawn at the 20% probability level.

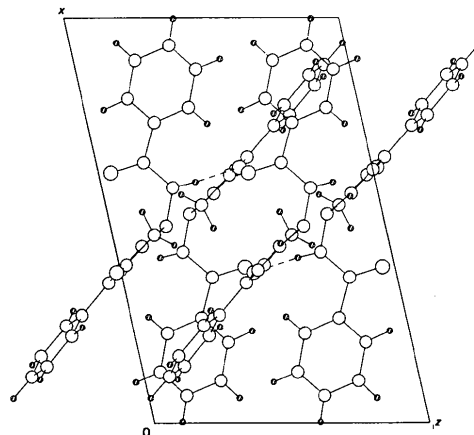


Fig. 2. Unit-cell contents viewed down the b axis. The hydrogen bonds are indicated by dashed lines.

Structure solved by direct methods. The least-squares refinement used *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.0003$, isotropic secondary-extinction value $p = 0.0021 (5)$ in $F_c^* = F_o/[1.0 + 0.002(p)F_o^2/\text{sin}2\theta]^{0.25}$. 256 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for all H, $(\Delta/\sigma)_{\text{max}} = 0.016$. $R = 0.039$, $wR = 0.050$, $S = 1.741$. Final difference Fourier excursions 0.19 and -0.16 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom numbering for Tables 1 and 2, atom coordinates, bond distances, and bond angles follows that shown in Fig. 1. Fig. 2 shows the packing and hydrogen bonding. The hydrogen-bond parameters are: $\text{H}(13)\cdots\text{O}(6)' = 2.14 (2)$, $\text{N}(13)\cdots\text{O}(6)' = 2.934 (4) \text{ \AA}$, and $\angle \text{N}-\text{H}\cdots\text{O} = 153.9 (1.3)^\circ$.

Related literature. The title compound is a minor reaction product obtained from a benzoylhydrazine/formaldehyde/orthoformate reaction which proceeds

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43324 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

through the intermediate compound 1,3,5-tribenzamido-1,3,5-hexahydrotriazine. For the structure of the intermediate see George & Gilardi (1987).

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Ein tricyclisches Molekül mit zentralem anorganischem Phosphor(V)-Hydrazin-Sechsring

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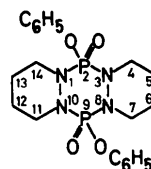
Abstract. *trans*-2,9-Dioxo-2,9-diphenoxy-1,3,8,10-tetraaza-2 λ^5 , 9 λ^5 -diphosphatricyclo[8.4.0.0^{3,8}]tetradecane, C₂₀H₂₆N₄O₄P₂, *M_r* = 448.364, orthorhombic, *Pbca*, *a* = 11.950 (4), *b* = 19.584 (7), *c* = 9.448 (5) Å, *V* = 2211 (3) Å³, *Z* = 4, *D_x* = 1.347 g cm⁻³, λ (Mo *K* α) = 0.71069 Å, μ = 2.74 cm⁻¹, *F*(000) = 944, *T* = 293 K, *R* = 0.059 for 1672 unique contributing reflections. The molecule is the centrosymmetric *E* isomer (*trans* isomer). The central inorganic ring as well as the outer hexahydropyridazine rings adopt a chair conformation similar to that of the corresponding dithio compound [Engelhardt & Stromburg (1985). *Acta Cryst.* C41, 122–126] and in contrast to the *Z* isomer (*cis* isomer) of the latter. The terminal O atoms at P are in equatorial, the phenoxy groups in axial positions. Mean bond distances: N–N 1.446 (5), P–N 1.649 (3), P=O 1.1455 (3), P–O 1.590 (3), N–C 1.469 (6) Å.

Experimentelles. Die Titelverbindung (1) wurde in unserem Laboratorium erstmals dargestellt. Sie entsteht bei der Umsetzung von Bis(hexahydro-1-pyridazinyl)-phosphorsäure-phenylester mit Phenoxyphosphoryldichlorid in Gegenwart von überschüssigem Triethylamin und wasserfreiem Aluminiumchlorid (als Katalysator) in Tetrahydrofuran als Lösungsmittel unter Rückfluß. Die Verbindung kann nach Abfiltrieren des gebildeten Triethylaminhydrochlorids und Abziehen des Lösungsmittels aus dem Rohprodukt mit *n*-Hexan/Toluol extrahiert werden. Nach Umkristallisieren aus *n*-Hexan schmelzen die farblosen Kristalle bei 504 K (korrigiert). Gitterparameter aus 38 Reflexen ($10 < \theta < 15^\circ$, Kleinste-Quadrat-Verfeinerung), automatisches Stoe-Vierkreis-Diffraktometer, θ -2 θ -Abtastung, drei Standardreflexe alle 90 min (600, 080, 00 $\bar{6}$), Meßbereich $0 \leq h \leq +14$, $0 \leq k \leq +23$, $-11 \leq l \leq 0$, $(\sin \theta / \lambda)_{\max}$ 0,5946 Å⁻¹, 1948 kristallographisch unabhängige Reflexe vermessen, Abnahme der Standardreflex-Intensitäten während der gesamten

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SHELDRICK, G. M. (1980). *SHELXTL80*. Minicomputer programs for structure determination. Univ. of Göttingen.

Messzeit < 2%, lineare Korrektur, Kristallabmessungen 0,56 × 0,46 × 0,075 mm Absorptionskorrektur mit sechs Begrenzungsflächen (Unterprogramm *ABSCOR* im Programmsystem *XRAY76*, siehe unten), Transmissionsfaktoren für *F*² 0,980 > *A* > 0,902 (Reflexe 200 bzw. 00 $\bar{2}$), 649 Reflexe mit *I* < 2 σ (*I*) 'unbeobachtet', diese wurden bei der Verfeinerung [Minimalisierung von $\sum w (|F_o| - |F_c|)^2$] nur einbezogen, wenn $|F_o| < |F_c|$. Die Struktur wurde mit Hilfe direkter Methoden gelöst (Programm *MULTAN*; Germain, Main & Woolfson, 1971). Die Wasserstoffatomlagen wurden nach anisotroper Verfeinerung der übrigen Atomlagen mit Hilfe von Differenz-Fourier-Synthesen ermittelt. Die endgültige Verfeinerung aller 189 Parameter mit anisotropen Temperaturfaktoren (H-Atome isotrop) und einer Gewichtung mit $w = K/(A + DF_o + EF_o^2)$ (*K* = 8,50, *A* = 86,367, *D* = -3,5394, *E* = 0,0402) unter Mitverfeinerung eines isotropen Extinktionsfaktors $g = 0,7 (4) \times 10^{-3}$ (Larson, 1967) ergab *R* = 0,059 und *wR* = 0,044 für 'beobachtete' Reflexe, *wR* = 0,054 für alle Reflexe, *S* = 0,76, $(\Delta/\sigma)_{\max}$ im letzten Verfeinerungszyklus 0,05, Reste elektronendichte-Maxima in einer abschließenden Differenz-Fourier-Synthese < 0,361 e Å⁻³; Atomformfaktoren für die neutralen Atome von Cromer & Mann (1968), für H von Stewart, Davidson & Simpson (1965), *f'* = 0,20 für P (*International Tables for X-ray Crystallography*, 1962), Programmsystem *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).



(1)