mit den Anionen $[Ni(HN_2S_2)(N_2S_2)]^-$ und $[Ni(N_2S_2)_2]^{2-}$: Weiss (1983, 1984).

Literatur

International Tables for X-ray Crystallography (1974). Bd. IV. Birmingham: Kynoch Press. (Gegenwärtiger Verteiler D. Reidel, Dordrecht.)

MAYER, K. F. & WEISS, J. (1978). *Acta Cryst.* B**34**, 1999–2000.

SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. Göttingen.

WEISS, J. (1983). *Z. Anorg. Allg. Chem.* **502**, 165–170.

WEISS, J. (1984). *Angew. Chem.* **96**, 232; *Angew. Chem. Int. Ed. Engl.* **23**, 225.

WEISS, J. & THEWALT, U. (1968). *Z. Anorg. Allg. Chem.* **363**, 159–165.

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Oxalato-*cis*-bis(triethylphosphine)palladium(II)

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Abstract. $[\{(C_2H_5)_3P\}_2Pd\{OC(=O)C(=O)O\}]$, $M_r = 430{\cdot}74$, monoclinic, $P2_1/n$, $a = 8{\cdot}554\ (5)$, $b = 15{\cdot}444\ (9)$, $c = 14{\cdot}400\ (10)$ Å, $\beta = 95{\cdot}93\ (5)°$, $U = 1892\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}512$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 11{\cdot}4$ cm^{-1}, $F(000) = 888$, $T = 292$ K, $R_F = 0{\cdot}0253$ for 2246 observed reflections. The Pd atom coordination geometry is square planar; the oxalato group bonds as a chelating, bidentate ligand to form a five-membered metallocycle. The O—Pd—O angle is acute, 81·1 (1)°, and the P—Pd—P angle obtuse, 96·3 (1)°.

Experimental. Synthesized from *cis*-$Pd(PEt_3)_2Cl_2$ by addition of $Ag_2C_2O_4$ (Parshall, 1970) and recrystallized from ethyl ether/methylene chloride. Off-white, 0·24 × 0·26 × 0·33 mm, mounted on glass fiber. Nicolet *R*3, graphite monochromator, unit cell from least-squares fit of angular settings of 25 reflections ($23 \le 2\theta \le 29°$). 2805 reflections, $4 \le 2\theta \le 45°$, collected for $-10 \le h \le 10$, $0 \le k \le 17$, $0 \le l \le 16$ at 4° min^{-1} using ω scans. 8% decay in the intensities of three standard reflections (linear correction applied), no correction for absorption (transmission 0·91 to 1·00). 2246 independent observed $F_o \ge 3\sigma(F_o)$ reflections, 223 rejected, $R_{int} = 0{\cdot}019$ for 111 duplicates. Multisolution direct methods (*SOLV*), blocked-cascade least squares on *F*, $w^{-1} = \sigma^2(F) + 0{\cdot}0006F$, complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Non-hydrogen atoms anisotropic, hydrogen atoms fixed, C—H = 0·96 Å, U(H) = 1·2 U_{eq}(C), 191 parameters (a parameter was refined for a secondary-extinction correction, but its value was insignificantly small), $R = 0{\cdot}025$, $wR = 0{\cdot}029$, slope of normal probability plot = 1·071, max. final $\Delta/\sigma = 0{\cdot}053$, max. $\Delta(\rho) = 0{\cdot}26$, min. $\Delta(\rho) = -0{\cdot}28$ e Å^{-3}. *SHELXTL* program package (Sheldrick, 1983).†

† Lists of anisotropic thermal parameters, hydrogen-atom parameters, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43298 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* (× 10^4) *and thermal coefficients* (Å^2 × 10^3)

	x	*y*	*z*	U_{eq}*
Pd	6382·2 (3)	2226·8 (2)	4976·8 (2)	30 (1)
P(1)	7070 (1)	3256 (1)	6049 (1)	33 (1)
P(2)	4022 (1)	1925 (1)	5468 (1)	33 (1)
O(1)	8563 (3)	2330 (2)	4491 (2)	48 (1)
O(2)	9872 (3)	1868 (2)	3348 (2)	59 (1)
O(3)	6031 (3)	1393 (2)	3851 (2)	44 (1)
O(4)	7122 (3)	1083 (2)	2555 (2)	73 (1)
C(1)	8667 (4)	1904 (2)	3735 (2)	40 (1)
C(2)	7172 (4)	1413 (2)	3324 (3)	45 (1)
C(11)	8098 (4)	2843 (2)	7123 (2)	47 (1)
C(12)	9314 (5)	2153 (3)	6999 (3)	58 (2)
C(21)	8477 (4)	3964 (2)	5553 (3)	48 (1)
C(22)	7824 (5)	4386 (3)	4649 (3)	68 (2)
C(31)	5556 (4)	3977 (2)	6411 (2)	41 (1)
C(32)	6123 (5)	4715 (2)	7057 (3)	56 (2)
C(41)	4065 (4)	1606 (2)	6687 (2)	45 (1)
C(42)	5148 (5)	853 (3)	6961 (3)	65 (2)
C(51)	3216 (4)	1007 (2)	4791 (3)	48 (1)
C(52)	1768 (4)	579 (3)	5116 (3)	61 (2)
C(61)	2505 (4)	2759 (2)	5308 (2)	39 (1)
C(62)	2565 (5)	3288 (3)	4428 (3)	53 (1)

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

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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

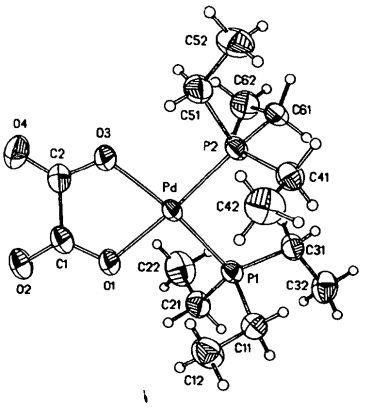

Fig. 1. Structure and labeling scheme for the title complex with 50% probability ellipsoids.

eliminates two molecules of CO_2 and provides a convenient source for $Pd(PEt_3)_2$ (Paonessa, Prignano & Trogler, 1985).

References

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

PAONESSA, R. S., PRIGNANO, A. L. & TROGLER, W. C. (1985). *Organometallics*, **4**, 647–657.

PARSHALL, G. (1970). *Inorg. Syn.* **12**, 26–27.

SCOTT, K. L., WIEGHARDT, K. & SYKES, A. G. (1973). *Inorg. Chem.* **12**, 655–663.

SHELDRICK, G. M. (1983). *SHELXTL Users Manual*, revision 4. Nicolet XRD Corporation, Madison, Wisconsin.

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

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Abstract. $C_{16}H_{14}N_4O_2$, $M_r = 294{\cdot}31$, monoclinic, $P2_1/c$, $a = 14{\cdot}604$ (2), $b = 10{\cdot}339$ (1), $c = 9{\cdot}714$ (1) Å, $\beta = 102{\cdot}56$ (1)°, $V = 1431{\cdot}7$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}365$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}728$ mm^{-1}, $F(000) = 616$, $T = 295$ K, final $R = 0{\cdot}039$, $wR = 0{\cdot}050$ 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 $30 < 2\theta < 60°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, ab-

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