Table 3. *Least-squares planes in* (I)

Deviations of atoms from the planes are given in Å.

W* 0·00 (1). Br(1) −0·25 (1). Br(2) 0·25 (1). C(60) 0·27 (1). C(70) −0·26 (1)

Angles between phenyl rings (°): (1) and (2) 80·6 (1). (1) and (3) 89·5 (2), (2) and (3) 65·6 (1). (4) and (5) 78·2 (1). (4) and (6) 86·7 (1), (5) and (6) 88·5 (1).

* Atom not contributing to the plane.

calculated to be far smaller (within 20° of 180°) than that observed for the W (128·5°) and the Mo (127·8°) compounds.

In the present structure the equatorial plane is distorted in that the Br and C atoms are all *ca* 0·25 Å from the plane (Table 3). The angle between W,Br(1),Br(2) and W,C(60),C(70) is 18·1 (1)°. P(1) and P(2) are both bent away from the Br atoms and towards the carbonyls. All four P—W—C angles are in the range 74·2 (6) to 77·5 (7)°. However, the P atoms are not equidistant from the Br atoms. The relevant angles are P(1)—W—Br(1) 121·6 (2), P(2)—W—Br(2) 122·2 (2), and P(1)—W—Br(2) 97·5 (2), P(2)—W—Br(1) 97·2 (2)°. It is not at all clear why this should be so: one explanation is to be found in the orientation of the phenyl rings. The smallest Br—W—P—C torsion angles are concomitant with the larger Br—W—P angles. Thus Br(1)—W—P(1)—C(11) is −12·7 (1)° and Br(2)—W—P(2)—C(61) is −8·6 (1)°. Torsion angles involving the carbonyls and the phenyl rings show several angles of *ca* 30° but none as small as those quoted above.

Attempts to investigate the solution behaviour of this complex and to determine whether it was dynamic were unsuccessful because the low solubility of the com-pound precluded meaningful ^{13}C NMR measurements. However, we were able to confirm from intensity measurements in the carbonyl region (Beck, Melvikoff & Stahl, 1966) that the wide $M(CO)_2$ angle was maintained at 115 ± 5° in solution. In addition, the intense blue colouration of solutions of $WBr_2(CO)_2(PPh_3)_2$ was caused by a broad absorption band centred at 17 900 cm^{-1}. The corresponding tricarbonyl $WBr_2(CO)_3(PPh_3)_2$ was completely trans-parent in this region with no absorption maxima below 30 000 cm^{-1}.

We are currently investigating the structures, and chemical and physical properties of a range of related complexes in an attempt to define further the factors which determine the degree of deformation in the structures of this interesting class of molecules.

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Bis(oxamide oximato)palladium(II) Dihydrate

BY HELMUT ENDRES

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Abstract. $Pd(C_2H_5N_4O_2)_2.2H_2O$, $C_4H_{10}N_8O_4Pd.-2H_2O$, $M_r = 376·6$, monoclinic, $P2_1/c$, $a = 3·899$ (2), $b = 13·475$ (6), $c = 11·433$ (5) Å, $\beta = 95·65$ (4)°, $V = 597·8$ Å^3, $Z = 2$, $d_c = 2·09$ Mg m^{-3}. Final $R = 0·069$ for 1062 observed reflections. The centrosymmetric planar molecules form stacks along **a**, the normals to the planes being inclined at 26° to the stacking axis, resulting in an interplanar distance of 3·51 Å. The water bonds by hydrogen bridges to adjacent com-plexes of a stack, leading to a ladder-like structure.

Introduction. Our studies on the structural chemistry of complexes of oxamide oxime (diaminoglyoxime, oaoH$_2$) (Ephraim, 1889) with the metals of the Ni triad

(Endres & Weiss, 1981, and references cited therein) have shown that these complexes form a variety of crystal and molecular structures, depending on the crystallization conditions. Up to now we have found two different complexes of Pd, both containing charged complex cations (Endres, 1980; Endres & Weiss, 1981). The title compound contains neutral [Pd(oaoH)$_2$] complex molecules, as does the Ni compound of the same composition (Endres, 1979).

The title compound was obtained in the following manner: yellow Pd(oaoH)$_2$ was precipitated by adding dropwise a solution of 1 mmol palladium diacetate in 50 ml acetone to a warm stirred solution of 2·2 mmol oaoH$_2$ in 50 ml acetone. The precipitate and the supernatant liquid were acidified with concentrated HCl and filtered while warm. The pale-yellow residue was dissolved in boiling pyridine. After cooling in a refrigerator for several days a cream-coloured precipitate was filtered off. When the filtrate was allowed to evaporate at room temperature, yellow columns of Pd(oaoH)$_2$.2H$_2$O formed together with a micro-crystalline yellow body which disintegrated in the air.

Lattice parameters were derived from the setting angles of 25 reflections centered on a diffractometer (Syntex $R3$, monochromatic Mo $K\alpha$ radiation). They are very close to those of the analogous Ni compound (Endres, 1979), but the Pd complex has space group $P2_1/c$, whereas the space group of the Ni compound was chosen in the alternative orientation $P2_1/n$. Data collection (θ–2θ scans, background-peak-background, $2\theta < 60°$) yielded 1062 independent observed reflections with $I > 2\sigma(I)$. Lorentz and polarization corrections as well as an empirical absorption correction (ψ scans) were applied.

The similarity of the lattice parameters suggested that the atomic coordinates of the Pd complex should be close to those of the Ni compound, despite the different orientation of the glide. So the coordinates of the Ni compound were taken as starting parameters and refined. Refinement with anisotropic temperature factors by a cascade-matrix least-squares procedure converged with a weighted (unweighted) $R = 0·069$ (0·076).* The weighting scheme was $W = 1/\sigma^2(F)$. Some reasonable positions for H atoms showed up in a difference Fourier map, but they did not refine well and were omitted in the final refinement.

Calculations were carried out on a Nova 3 computer; plots were drawn on a Tektronix plotter. The *SHELXTL* program system (Sheldrick, 1979) was applied, which uses scattering factors from *International Tables for X-ray Crystallography* (1974) and takes anomalous dispersion into account.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36674 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Atomic coordinates are listed in Table 1, bond distances and angles are shown in Fig. 1. The neutral centrosymmetric Pd(oaoH)$_2$ complex molecule exhibits two intramolecular hydrogen bridges, contrary to the charged species described earlier (Endres, 1980; Endres & Weiss, 1981). The molecules form stacks along **a**. The normals to the molecular planes are inclined at 26° to the stacking axis, so that the interplanar distance is 3·51 Å. A projection of the structure onto the *bc* plane is shown in Fig. 2, and Fig. 3 gives a perpendicular projection of two adjacent molecules of a stack onto each other. As in the Ni compound the central metal atom lies nearly perpendicularly above and below oxime N atoms of adjacent molecules, with a Pd–N distance of 3·522 (9) Å. Corresponding values in the Ni compound are: inclination angle 30°, interplanar distance 3·44 Å, intermolecular Ni–N separation 3·478 (3) Å. The slight differences are probably caused by the larger atomic radius of Pd. Similar differences are found, of course, in the intramolecular M–N distances: Pd–N 1·960 (9) and 1·973 (9) Å, Ni–N 1·851 (3) and 1·863 (3) Å, and a smaller N–Pd–N angle, 79·4 (4)°,

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic temperature factors* ($\times 10^3$) *equivalent to the refined anisotropic values*

U is defined as $U = \frac{1}{3}$ trace $\tilde{U}$. $\tilde{U}$ signifying the diagonalized U matrix.

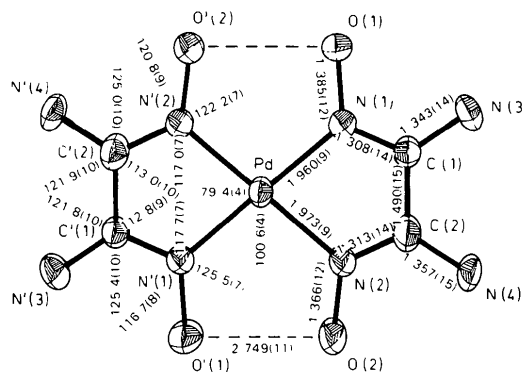|	x	y	z	U (Å^2)
Pd	0	5000	5000	34·8 (3)
N(1)	948 (31)	4621 (7)	6655 (8)	41 (4)
N(2)	2387 (29)	6200 (6)	5646 (8)	39 (3)
N(3)	3629 (29)	5091 (8)	8509 (7)	48 (3)
N(4)	5207 (33)	6942 (7)	7356 (9)	50 (4)
O(1)	89 (27)	3721 (6)	7135 (7)	51 (3)
O(2)	2835 (27)	7019 (6)	4972 (7)	48 (3)
C(1)	2702 (35)	5243 (7)	7360 (10)	36 (4)
C(2)	3533 (35)	6184 (8)	6765 (10)	37 (4)
O(3)	7516 (26)	8363 (6)	4958 (7)	49 (3)

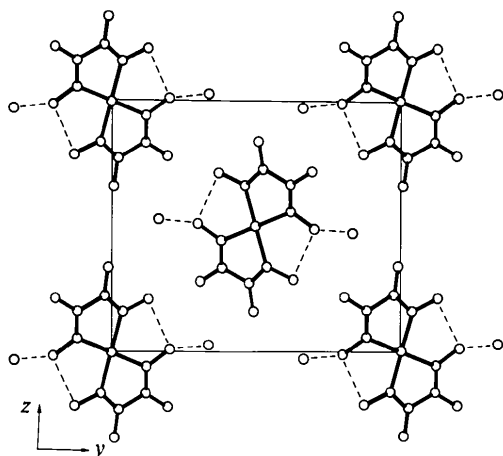Fig. 1. The Pd(oaoH)$_2$ complex molecule with bond distances (Å) and angles (°).
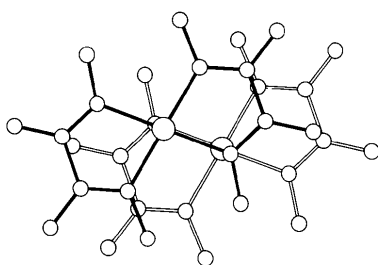
Fig. 2. Projection of the structure onto the bc plane.

Fig. 3. Perpendicular projection of two adjacent molecules of a stack onto each other.

compared with 82·2 (2)° in the Ni compound. As usual the intramolecular hydrogen bridge is considerably longer in the Pd complex [2·75 (1) Å] than in the Ni analogue [2·57 (1) Å].
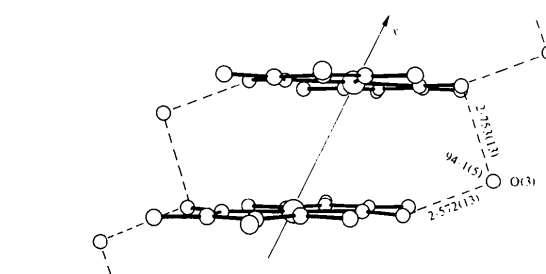

Fig. 4. Perspective view of two molecules of a stack showing the hydrogen-bonded water O atoms which lead to a ladder-like structure (distances in Å, angle in °).

The water molecule is attached by hydrogen bridges to oxime O atoms of adjacent molecules of a stack. This leads to a ladder-like structure, a section of which is shown in Fig. 4.

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Structure of Bis(ethylenediamine)diiodoplatinum(IV) Di-μ-iodo-bis[diiodoargentate(I)]

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Abstract. $[Pt(C_2H_8N_2)_2I_2]_2[Ag_2I_6]$, $M_r = 2115\cdot34$, orthorhombic, Fddd, $a = 15\cdot576$ (3), $b = 18\cdot451$ (3), $c = 24\cdot645$ (4) Å, $V = 7082\cdot79$ Å^3, $Z = 8$, $d_c = 3\cdot96$ Mg m^{-3}, μ(Mo Kα) = 17·690 mm^{-1}. The structure has been solved by Patterson and Fourier methods from diffractometer data and refined to $R = 0\cdot038$ for 1465 observed reflections. The structure consists of $[Pt(en)_2I_2]^{2+}$ cations, which are connected by weak intermolecular I–I contacts, and of isolated $[Ag_2I_6]^{4-}$ anions formed by two AgI$_4$ tetrahedra sharing edges.

Introduction. In an attempt to prepare new Wolffram's salt analogues containing additional anionic metal halide chains (Keller, Martin & Traeger, 1978; Endres,

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