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Bis(oxamide oximato)platinum(II)–Hydrogen Chloride (1:2)

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Abstract. $[Pt(C_2H_5N_4O_2)_2].2HCl$, $C_4H_{10}N_8O_4Pt.2HCl$, $M_r = 502{\cdot}17$, $P\bar{1}$, $a = 5{\cdot}244$ (2), $b = 8{\cdot}317$ (3), $c = 8{\cdot}500$ (5) Å, $\alpha = 61{\cdot}57$ (3), $\beta = 80{\cdot}53$ (4), $\gamma = 81{\cdot}23$ (3)°, $Z = 1$, $d_c = 2{\cdot}60$ Mg m^{-3}; final $R = 0{\cdot}052$ for 1996 reflections. The planar complex units form stacks along **a** with interplanar distances of 3·415 Å. The normals of the planes are inclined at 49·4° to the stacking axis.

Introduction. In the search for new materials with highly anisotropic physical properties and with a feasible chemistry we are presently investigating the structural variability of complexes of Ni, Pd, and Pt with oxamide oxime (diaminoglyoxime, oaoH$_2$) (Endres, 1978, 1979*a*,*b*, 1980). The title compound is prepared by adding drop-wise a solution of $K_2[PtCl_4]$ in 10% HCl to an aqueous solution of oxamide oxime (Ephraim, 1889) at 343–353 K with stirring. The reaction mixture is concentrated by evaporation on a warm plate to about $\frac{1}{4}$ of its original volume and allowed to cool. The yellow precipitate is filtered off, washed with ethanol, dried, and dissolved in warm 10% HCl. The solution is allowed to cool slowly in a Dewar vessel (1–3 days). Two kinds of crystal form: long bright-orange columns (which are still under investigation) and a small quantity of rhombohedrally shaped crystals, which are the subject of this paper. Elementary analysis indicated the presence of two moles of Cl per mole of [Pt(oaoH)$_2$] in the mixture.

A crystal 0·17 × 0·12 × 0·05 mm was used for the X-ray investigation. Rotation and Weissenberg photographs showed the crystal to be triclinic and gave an estimate for the lattice constants. Exact lattice parameters were derived from the setting angles of 25 reflections centred on a Syntex *R*3 diffractometer. Data collection [monochromatic Mo $K\alpha$ radiation, θ–2θ background–peak–background step-scan mode, 2θ(max.) = 60°] yielded 1996 observed independent reflections with $I > 3{\cdot}0\sigma(I)$; these were corrected for Lorentz and polarization factors, but not for absorption ($\mu = 11{\cdot}96$ mm^{-1}). A Patterson synthesis favoured the choice of the centrosymmetric space group and revealed the Cl position. Pt was placed at 0,0,0. Fourier syntheses showed the positions of the light atoms. Refinement with isotropic temperature factors resulted in $R = 0{\cdot}106$; refinement by full-matrix least squares with anisotropic temperature factors converged with $R = 0{\cdot}052$ (maximum shift/error 0·08).* A final difference synthesis showed no maxima attributable to solvent molecules. H atoms could not be identified. Calculations were performed on Nova 3 and IBM 370/168 computers with the XRAY system (Stewart, Kundell & Baldwin, 1970) and scattering factors from *International Tables for X-ray Crystallography* (1974). Unit weights were used for all reflections. Plots were drawn by *ORTEP* (Johnson, 1965).

Discussion. Atomic coordinates are listed in Table 1; bond distances and angles with the numbering scheme are shown in Fig. 1. As usual, the oaoH species acts as a bidentate ligand *via* the oxime N atoms. The distance between the O atoms is relatively long [2·95 (1) Å], so that the existence of a H bridge may be questioned in this case. There are other adducts of [Pt(oaoH)$_2$] where only one of the two possible intramolecular H bridges exists, and instead of the second *intramolecular* H bridge an *intermolecular* one is

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34723 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$)

	x	*y*	*z*
Pt	0	0	0
Cl	682 (6)	3239 (4)	4108 (5)
O(1)	3223 (17)	−2266 (11)	−1672 (14)
O(2)	1435 (17)	3709 (11)	−838 (13)
N(1)	1996 (17)	2182 (12)	−1175 (13)
N(2)	2840 (18)	−645 (12)	−1527 (14)
N(3)	6631 (18)	200 (13)	−3493 (15)
N(4)	5757 (18)	3497 (12)	−3020 (15)
C(1)	4137 (20)	2196 (14)	−2226 (15)
C(2)	4588 (19)	483 (14)	−2456 (15)

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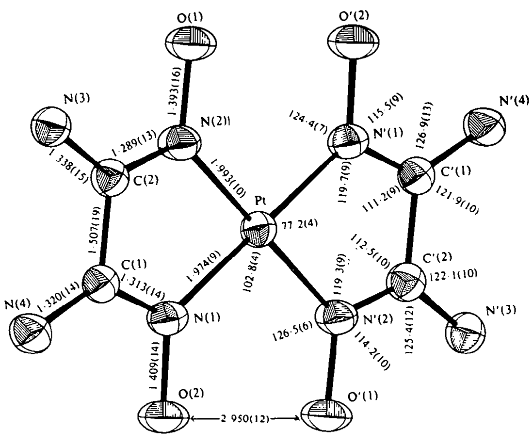

Fig. 1. The bis(oxamide oximato)platinum(II) molecule with bond distances (Å) and angles (°).

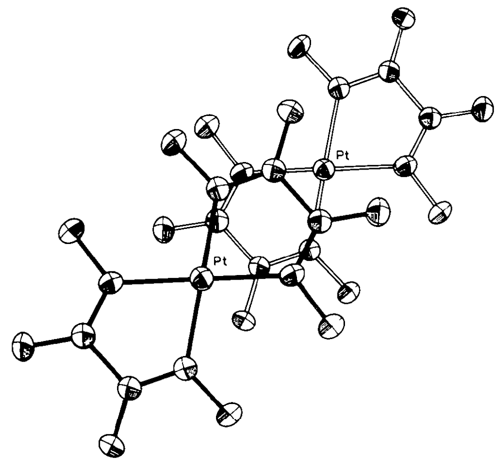

Fig. 2. Perpendicular projection of two adjacent molecules within a stack.

formed (Endres, 1979*b*, 1980). The complex molecule is practically planar: if the molecular plane is defined by Pt and the four oxime N, the maximum deviation of an atom from this plane is 0·16 Å. The molecules form regular stacks along **a** with an interplanar separation of 3·415 Å. The normals of the molecular planes are tilted at 49·4° to the stacking axis. Fig. 2 shows a perpendicular projection of two adjacent complex molecules indicating the mode of overlap. Pt has no axial interaction with adjacent molecules. Due to the very inclined stacking angle, each half of one molecule overlaps with one half of the molecule above and below. The stacks form channels in the lattice which accommodate the Cl species. The closest contact of Cl with an atom of the complex occurs to O(2), 2·973 (9) Å. This indicates H bonding, and one could argue that the H of the HCl is involved in this bond. This would explain the long intramolecular O'(1)–O(2) distance of 2·95 (1) Å, indicating the weak tendency of O(2) to form another H bridge to O(1). Other short contacts involving Cl are: 3·21 (1) Å to N(4) of the same molecule as above, and 3·22 (1) Å to N(3) of two other molecules. By this network of short distances, different stacks are linked in the *y* and *z* directions. The distances may be compared to the N–Cl distances in crystalline NH_4Cl, 3·36 Å (Wyckoff, 1963).

References

ENDRES, H. (1978). *Acta Cryst.* B**34**, 2306–2309.
ENDRES, H. (1979*a*). *Acta Cryst.* B**35**, 625–627.
ENDRES, H. (1979*b*). *Acta Cryst.* B**35**, 3032–3034.
ENDRES, H. (1980). *Acta Cryst.* B**36**. In the press.
EPHRAIM, J. (1889). *Chem. Ber.* **22**, 2305–2306.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.
WYCKOFF, R. W. G. (1963). *Crystal Structures*, p. 104. New York, London, Sydney: John Wiley.

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(η-Allyl)dicarbonyl(*N*-phenylsalicylideneiminato)pyridinemolybdenum(II)

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Abstract. $[Mo(C_3H_5)(C_5H_5N)(C_{13}H_{10}NO)(CO)_2]$, $C_{23}H_{20}MoN_2O_3$, M_r = 468·4, triclinic, a = 10·153 (7), b = 10·232 (8), c = 10·279 (11) Å, α = 103·7 (1), β = 84·9 (1), γ = 99·0 (1)°, U = 1023·3 Å^3, Z = 2, d_m = 1·51 (2), d_c = 1·52 Mg m^{-3}, Mo $K\alpha$ radiation, λ = 0·7107 Å, μ = 0·66 mm^{-1}; space group $P\bar{1}$ from the successful structure determination. The Mo atom in the complex has an

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