[Pt(oaoH)$_2$] (Endres, 1980). No bending of the molecules is observed in this case, as there is only one H bridge between each pair of molecules. H bridges *via* water molecules, leading to a ladder-like structure, are found in the regular stacks of M(oaoH)$_2$.2H$_2$O, M = Ni or Pd (Endres, 1982*a*).

As in some other complexes it is not possible to discriminate between 'M—M' and 'M—L' forms. The arrangement between adjacent pairs of H-bonded dimers, with the long metal–metal distance, is reminiscent of the M—L type of stacking, while the geometry within a dimer is different from the usual M—M pattern, despite the short Pt—Pt distance. There is no twist of adjacent complexes relative to each other and the molecules are nearly eclipsed. The Pt—Pt vector makes an angle of 9·9 (5)° with respect to the normal of the plane through Pt and the four coordinated N atoms. A perpendicular projection of the two molecules of a dimer (Fig. 3, deposited) reveals that it is not an attractive Pt—Pt interaction which leads to the observed arrangement, but the link between the H-bridged O(1) and O(3) atoms of adjacent complexes. The other atoms tend to avoid close intermolecular interactions as far as possible, without losing the bonding O(1)···O(3) contacts.

Further H bridges link the stacks in the other two dimensions (Fig. 4). In the y direction these bridges involve amino and oxime groups, in the z direction the bridging occurs *via* O(6) water molecule.

From a chemical point of view it is interesting to note that the treatment with iodine-containing HI solution does not lead to oxidation of the complex.

This work has been supported by the Stiftung Volkswagenwerk and by the Fonds der Chemischen Industrie. A gift of Pt salt from DEGUSSA, Hanau, is also gratefully acknowledged.

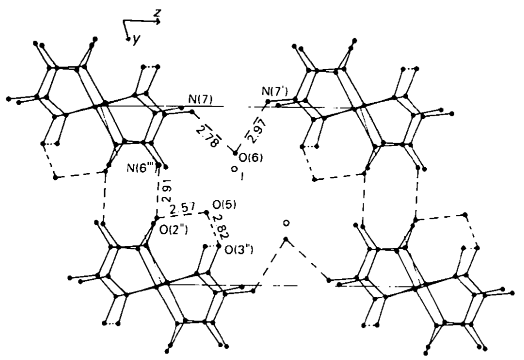

Fig. 4. Parallel projection of the structure along **a** showing probable intermolecular H bridges. The corresponding distances are given, e.s.d. 0·02 Å. Symmetry operations are: (i) $1-x, -y, 1-z$; (ii) $x, 1+y, z$; (iii) $1-x, -y, -z$.

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Structure of an Organoamidoplatinum(II) Compound

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Abstract. Chloro[*N,N*-dimethyl-*N'*-(2,3,5,6-tetrafluorophenyl)-1,2-ethanediaminato(1−)]pyridineplatinum(II), [PtCl($C_{10}H_{11}F_4N_2$)(C_5H_5N)], $M_r = 544·85$, triclinic, $P\bar{1}$, $a = 12·296$ (2), $b = 13·014$ (1), $c = 5·679$ (1) Å, $\alpha = 91·88$ (2), $\beta = 111·74$ (1), $\gamma = 96·23$ (1)°, $V = 836·5$ Å^3, $Z = 2$, $D_m = 2·16$ (2), $D_x = 2·16$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 8·26$ mm^{-1}, $F(000) = 516$, $T = 293$ K. Final $R = 0·037$ for 3798 counter reflections. The structure consists of molecules of the title compound in which approximately square-planar Pt^{II} is

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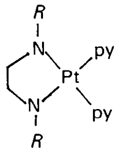
coordinated by chloride, pyridine and the bidentate *N,N*-dimethyl-*N'*-(2,3,5,6-tetrafluorophenyl)-1,2-ethanediaminate(1−) ligand. The Pt to ligand atom distances are: Pt—Cl 2·337 (2), Pt—N(NMe_2) 2·074 (5), Pt—N(pyridine) 2·019 (5) and Pt—N(amido) 2·028 (5) Å.

Introduction. Whereas reaction of $[PtCl_2L_2]$ [*e.g.* L_2 = 2,2'-bipyridyl or *trans*-(pyridine)$_2$] complexes with thallous polyfluorobenzoates in boiling pyridine yield $[R_2PtL_2]$ (*e.g.* $R = C_6F_5$ or *p*-HC_6F_4) or $[RPtClL_2]$ organometallics (Deacon & Grayson, 1982), the complex $[PtCl_2en]$ (en = 1,2-ethanediamine) does not give $[(C_6F_5)_2Pten]$ or $[C_6F_5PtCl(en)]$ with thallous pentafluorobenzoates in pyridine. Unexpectedly, an *N*-(2,3,5,6-tetrafluorophenyl) substituted 1,2-ethanediaminatoplatinum(II) complex (1) is obtained (Deacon, Gatehouse, Grayson & Nesbit, 1984):

$$[PtCl_2en] + 2TlO_2CC_6F_5 + 4py \rightarrow (1) + 2TlCl + 2CO_2 + 2pyHF.$$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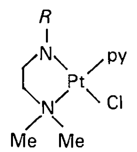
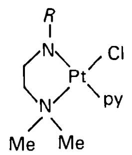

(2)


(3)

R = *p*-HC_6F_4 for (1)–(3)

A similar reaction between $[PtCl_2L_2]$ (L_2 = *N,N*-dimethyl-1,2-ethanediamine) and thallous pentafluorobenzoate yielded the title complex, which may have the structure (2) or (3):

$$[PtCl_2\{(CH_3)_2NCH_2CH_2NH_2\}] + TlO_2CC_6F_5 + 2py \rightarrow (2) \text{ or } (3) + TlCl + CO_2 + pyHF. \quad (1)$$

Since the isomer could not be identified spectroscopically, a crystal-structure determination was undertaken.

Experimental. Details of the preparation by reaction (1) will be given elsewhere. The compound was crystallized from a 1:3 (v/v) mixture of toluene/petroleum ether (b.p. 333–353 K) over 2 d at 243 K. D_m by flotation in CCl_4 and $C_2H_2Br_4$.

Space group $P\bar{1}$ by successful refinement; cell parameters determined with a Philips PW 1100 automatic four-circle diffractometer equipped with a graphite monochromator as described previously (Gatehouse & Miskin, 1974), and are the mean of five refinements of the 24 reflections used to monitor crystal stability during data collection. Three standard reflections measured at 3 h intervals. No decomposition occurred. Data collected using the $\theta/2\theta$ scan technique with a symmetric scan width of $\pm 1{\cdot}20°$ in θ from the calculated Bragg angle, with an allowance for dispersion, at a scan rate of $0{\cdot}05° s^{-1}$. No reflection was sufficiently intense to warrant the insertion of an attenuation filter. Data processed using a program written specifically for the PW 1100 diffractometer (Hornstra & Stubbe, 1972). Valves of I and $\sigma(I)$ corrected for Lorentz and polarization effects. Absorption correction applied, based on indexed crystal faces, max. and min. transmission factors 0·5847 and 0·3955 respectively. 4864 reflections measured to $2\theta = 60°$ from an approximately hexagonal prismatic crystal 0·28 × 0·075 × 0·11 mm, 3798 unique reflections $[I > 3\sigma(I)]$ used in analysis; index range h −17/15, $k \pm 18$, l 0/7. Pt-atom parameters found from Patterson synthesis and all non-H atoms located in subsequent difference Fourier synthesis. Function minimized in full-matrix least-squares refinement $\sum w(|F_o| - |F_c|)^2$, where w is the weight $\{[\sigma^2(F_o)]^{-1}\}$. Pt and Cl atoms refined anisotropically, other non-H atoms refined isotropically (107 variable parameters), final R = 0·037 and wR = 0·036 (for observed reflections); max. Δ/σ in final cycle 0·2; inclusion of H atoms not considered warranted. Final difference Fourier synthesis had $\Delta\rho$ within −1·29 and 1·21 e Å^{-3}. This was in the vicinity of the Pt atom. Scattering factors for neutral atoms and corrections for anomalous dispersion used (*International Tables for X-ray Crystallography*, 1974). All calculations performed on the Monash University VAX 11/780 computer system. Major programs used: *SHELX76* (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964) and *MEANPL* (Blow, 1960). Figure drawn using *ORTEP* (Johnson, 1965).

Discussion. Final fractional coordinates and thermal parameters, interatomic distances and selected angles, and mean-plane data are given in Tables 1, 2 and 3.*

The crystal structure (Fig. 1) shows coordination of chloride, pyridine, and a chelating *N,N*-dimethyl-*N'*-(2,3,5,6-tetrafluorophenyl)-1,2-ethanediaminate(1−) ligand to Pt in an approximately square-planar arrangement. Of the two possible isomers (2) and (3) (R = *p*-HC_6F_4), the former, with pyridine *cis* to the tetrafluorophenylamido group, is present.

The Pt—N(3)(amido) and Pt—N(1)(py) distances (Table 2) are essentially equal by contrast with the situation in $[Pt\{N(p\text{-}HC_6F_4)CH_2\}_2(py)_2]$ where the Pt—N(amido) distance is significantly longer than Pt—N(py) (Deacon *et al.*, 1984). The marginally longer Pt—N(2)(NMe_2) distance is as expected for a neutral N *trans* to an amine ligand {see *e.g.* Pt—N 2·05 Å in *trans*-$[Pt(NH_3)_2Cl_2]$ (Milburn & Truter, 1966)}. Comparison of the Pt—Cl distance with that (2·32 Å) of *cis*-$[Pt(NH_3)_2Cl_2]$ (Milburn & Truter, 1966) suggests that the *trans* influence of the diorganoamido ligand is

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* Lists of structure factors, full bond-angle data and Table 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42142 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

similar to that of an uncharged amine. A similar conclusion can be drawn from the similarity between the Pt—P distances of the *cis*-bis(*o*-aminophenyldiphenylphosphine)platinum(II) cation (Cooper, Downes, Goodwin & McPartlin, 1983) and of *cis*-bis(*o*-amidophenyldiphenylphosphine)platinum(II) (Cooper, Downes, Goodwin, McPartlin & Rosalky, 1983). The stereochemical arrangement at the amido N (Table 2) is substantially distorted from tetrahedral towards triangular, suggesting at least partial delocalization of the (non-coordinating) lone pair on N into the aromatic ring. This is borne out by the shortening of N(3)—C(10) (Table 2) from that [see *e.g.* N(3)—C(9), N(2)—C(8)] expected for an N—C single bond, and by the mean-plane data. This delocalization of charge into the aromatic ring can be partly attributed to the electron-withdrawing character of the F substituents, and may account for the fact that the amido N, though formally charged, is not a markedly better donor than the uncharged N-atom donors. A similar, even more pronounced, effect is observed for [Pt{N(p-HC_6F_4)CH_2}$_2$(py)$_2$] (Deacon *et al.*, 1984).

Table 1. *Fractional coordinates* (×10^4) *and thermal parameters* (Å^2 ×10^3) *with e.s.d.'s in parentheses* (Pt-*atom coordinates and thermal parameters are* ×10^5 *and* 10^4 *respectively*)

	x	y	z	U_{iso}
Pt	32719 (2)	18736 (2)	3297 (5)	†
Cl	2780 (2)	155 (1)	1021 (4)	†
N(1)	1597 (5)	1804 (4)	−2202 (10)	34 (1)
N(2)	4977 (5)	1955 (4)	3011 (10)	34 (1)
N(3)	3785 (5)	3384 (4)	49 (11)	37 (1)
F(1)	2518 (3)	3134 (3)	−5156 (8)	45 (1)
F(2)	993 (4)	4369 (3)	−7610 (9)	57 (1)
F(3)	1983 (5)	6518 (4)	−121 (11)	72 (1)
F(4)	3381 (4)	5220 (3)	2491 (9)	57 (1)
C(1)	1266 (6)	1296 (5)	−4507 (14)	40 (2)
C(2)	82 (7)	1154 (6)	−6215 (16)	50 (2)
C(3)	−750 (7)	1565 (6)	−5481 (17)	57 (2)
C(4)	−415 (7)	2076 (6)	−3114 (17)	58 (2)
C(5)	783 (6)	2205 (5)	−1527 (14)	43 (2)
C(6)	5629 (6)	1102 (5)	2642 (15)	45 (2)
C(7)	4942 (6)	1909 (5)	5640 (15)	46 (2)
C(8)	5636 (6)	2979 (5)	2793 (14)	40 (2)
C(9)	4792 (6)	3789 (5)	2355 (14)	42 (2)
C(10)	3010 (5)	4076 (4)	−1179 (12)	30 (1)
C(11)	2348 (5)	3931 (5)	−3817 (12)	34 (1)
C(12)	1594 (6)	4592 (5)	−5078 (14)	42 (2)
C(13)	1433 (7)	5483 (5)	−3960 (15)	47 (2)
C(14)	2080 (6)	5649 (5)	−1402 (15)	45 (2)
C(15)	2834 (6)	4987 (5)	−50 (14)	40 (2)

† Anisotropic thermal parameters of the type $\exp[-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^* + \cdots)]$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	275 (1)	300 (1)	239 (1)	−21 (1)	63 (1)	22 (1)
Cl	52 (1)	35 (1)	54 (1)	6 (1)	7 (1)	0 (1)

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

Pt—Cl	2·337 (2)	C(12)—C(13)	1·372 (11)
Pt—N(1)	2·019 (5)	C(13)—C(14)	1·370 (10)
Pt—N(2)	2·074 (5)	C(14)—C(15)	1·371 (10)
Pt—N(3)	2·028 (5)	C(15)—C(10)	1·405 (10)
C(1)—C(2)	1·406 (9)	N(3)—C(9)	1·462 (8)
C(2)—C(3)	1·388 (14)	C(9)—C(8)	1·521 (10)
C(3)—C(4)	1·375 (13)	C(8)—N(2)	1·517 (9)
C(4)—C(5)	1·401 (10)	N(2)—C(6)	1·491 (10)
C(5)—N(1)	1·344 (11)	N(2)—C(7)	1·513 (11)
N(1)—C(1)	1·344 (9)	C(11)—F(1)	1·345 (8)
N(3)—C(10)	1·393 (8)	C(12)—F(2)	1·356 (8)
C(10)—C(11)	1·410 (8)	C(14)—F(3)	1·365 (10)
C(11)—C(12)	1·355 (9)	C(15)—F(4)	1·355 (8)
N(1)—Pt—Cl	87·0 (2)	Pt—N(3)—C(10)	123·9 (4)
N(2)—Pt—Cl	92·9 (2)	C(10)—N(3)—C(9)	117·2 (5)
N(2)—Pt—N(3)	83·1 (2)	C(9)—N(3)—Pt	109·6 (4)
N(3)—Pt—N(1)	97·0 (2)	C(8)—N(2)—Pt	106·9 (4)

Torsion angle

N(2)—C(8)—C(9)—N(3) −57·6 (8)°

In the present compound, coordination of the amido group is supported by coordination of the $-NMe_2$ group. Diphenylphosphino groups (Cooper, Downes, Goodwin, McPartlin & Rosalky, 1983) and alkene groups (Cooper, Stevens & McPartlin, 1983) have also been used to support amido coordination. The tetrafluorophenyl group may confer stability to the Pt—N(3)(amido) bond against hydrolysis. Delocalization of the N lone pairs into the aromatic ring would inhibit hydrolytic protolysis at N(3). By contrast, [Pt($NHCH_2$)$_2$bpy] (bpy = 2,2′-bipyridyl) is highly moisture sensitive (Watt & Upchurch, 1968). The delocalization and the stereochemical arrangement at N(3) could promote π-bonding between Pt and the amido N.

This work was carried out during the tenure of a grant from the Anti-Cancer Council of Victoria. This support, that of the Australian Research Scheme, and a loan of Pt from Johnson–Matthey are gratefully acknowledged.

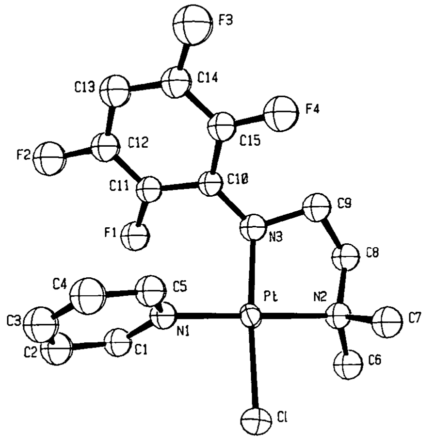

Fig. 1. Drawing of [PtCl{(p-HC_6F_4)$NCH_2CH_2N(CH_3)_2$}(C_5H_5N)] showing the stereochemistry of the molecule and the numbering scheme used.

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Structure of Oxolinic Acid, a Potent Antibacterial Agent. 1-Ethyl-1,4-dihydro-6,7-methylenedioxy-4-oxo-3-quinolinecarboxylic Acid, $C_{13}H_{11}NO_5$*

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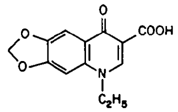
Abstract. $M_r = 261{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}182\,(2)$, $b = 10{\cdot}575\,(2)$, $c = 14{\cdot}758\,(2)$ Å, $\beta = 94{\cdot}26\,(1)°$, $V = 1117{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}552$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}98$ mm^{-1}, $F(000) = 544$, $T = 294$ K, final $R = 0{\cdot}038$ for 1565 observed reflections. The molecule is planar within $\pm 0{\cdot}12$ Å except for the terminal carbon atom of the *N*-ethyl group, which is displaced 1·36 Å from the mean plane. There is considerable double-bond localization in the benzene-ring moiety, and a significant difference between the two N—C bond lengths in the pyridine ring, the bond adjacent to the benzene ring being longer by 0·058 (2) Å. The crystal structure features a bifurcated hydrogen bond with a strong intramolecular component from the carboxylic acid group to the neighbouring carbonyl oxygen and a weak intermolecular component from the same donor to O(9) in the dioxole ring.

Introduction. Oxolinic acid (1) and the less potent but closely related nalidixic acid (2) belong to a group of highly active antibacterial agents. These compounds cause a preferential and reversible inhibition of DNA synthesis by inhibiting DNA gyrase (Sugino, Peebles, Kreuzer & Cozzarelli, 1977; Gellert, Mizuuchi, O'Dea, Itoh & Tomizawa, 1977). The crystal structure of nalidixic acid was determined by Achari & Neidle (1976) from photographic data and refined subsequently from more precise diffractometric data (Huber, Sake Gowda & Acharya, 1980). The crystal structure of 5-aminooxolinic acid (3), which shows some antibacterial activity as well, has also been investigated (Czugler, Argay, Frank, Mészáros, Kutschabsky & Reck, 1976). It is of some interest to compare the structure of oxolinic acid with these, and with the molecular structures of some other closely related antibacterial agents for which no X-ray data are yet available.



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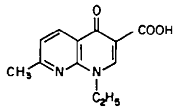

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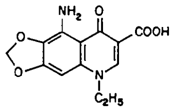

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