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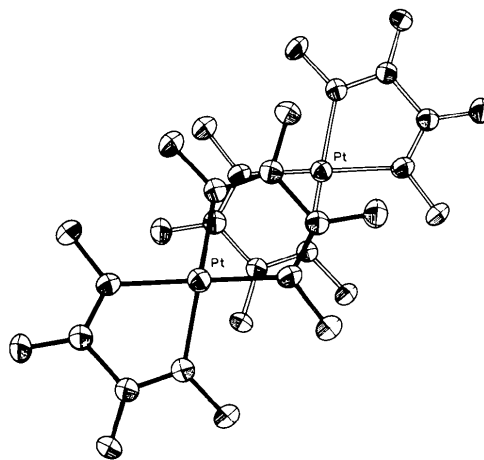

Fig. 2. Perpendicular projection of two adjacent molecules within a stack.

formed (Endres, 1979b, 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).

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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, $\alpha = 103·7 (1)$, $\beta = 84·9 (1)$, $\gamma = 99·0 (1)°$, $U = 1023·3$ Å^3, $Z = 2$, $d_m = 1·51 (2)$, $d_c = 1·52$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·7107$ Å, $\mu = 0·66$ mm^{-1}; space group $P\bar{1}$ from the successful structure determination. The Mo atom in the complex has an

approximately octahedral environment with the allyl group occupying just one coordination site. 2395 independent reflections above back-ground have been refined to an R of 0·070.

Introduction. Crystals of the title compound were prepared as follows. The reactants [Mo(η-allyl)(CO)$_2$Cl(dpa)] (0·42 g, 1·06 mmol) [dpa = di(2-pyridyl)amine] (Brisdon & Griffin, 1975), sodium 2-formylphenolate (0·21 g, 1·49 mmol), aniline (1 ml, 11 mmol) and pyridine (2 ml, 25 mmol) were stirred together in deoxygenated acetone at room temperature and under an atmosphere of dry nitrogen for three days. The resulting mixture was filtered, and water (10 ml) was added to the filtrate which was then rotary evaporated until the product started to crystallize. After standing at 273 K for one hour the product was collected at the filter, washed with water and dried *in vacuo* to give orange crystals of [Mo(η-allyl)(CO)$_2$(py)(salNPh)] (0·43 g, 87% yield) [C$_{23}$H$_{20}$MoN$_2$O$_3$ requires 59·0% C, 4·3% H, 6·0% N; found: 58·9% C, 4·5% H, 5·8% N; IR (Nujol mull): ν_{CO} 1930 vs, 1850 vs; ν_{CN} 1605 cm^{-1} vs].

Crystals suitable for X-ray analysis were obtained by recrystallization from aqueous acetone. A crystal approximately 0·33 × 0·5 × 1·2 mm was mounted with the b^* axis perpendicular to the instrument axis of

a General Electric XRD 5 diffractometer. 2842 independent reflections with $2\theta < 45°$ were measured by the stationary-crystal–stationary-counter method using 10 s counts. Of these, 2395 with $I > 2\sigma(I)$ were used in subsequent calculations.

The positions of the Mo atoms were located from the Patterson map and those of all other atoms except H were obtained from Fourier maps. Mo, C, O and N atoms were refined anisotropically. H atoms in trigonal positions were fixed but their thermal parameters were refined successfully. The H atoms of the allyl group were positioned from difference Fourier maps but could not be refined and their parameters were therefore fixed. The final R was 0·070. The weighting scheme was $w^{1/2} = 1$ for $30 > F_o$ and $30/F_o$ for $F_o > 30$. This gave satisfactory constant values of $w\Delta^2$ over groups of F_o and $\sin\theta/\lambda$. The calculations were carried out on a CDC 7600 computer at the University of London Computer Centre using *SHELX* 76 (Sheldrick, 1976). Atomic scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). The final atomic positions are given in Table 1, bond lengths and angles in Table 2.*

Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z
Mo(1)	2771 (1)	1244 (1)	2674 (1)
C(1)	964 (17)	802 (17)	3290 (18)
O(1)	−165 (12)	482 (17)	3644 (17)
C(2)	2983 (14)	54 (16)	3806 (14)
O(2)	3082 (13)	−690 (13)	4479 (12)
O(3)	2502 (10)	2767 (11)	1654 (10)
C(3)	1585 (22)	3 (20)	849 (18)
C(4)	2923 (19)	−46 (17)	590 (17)
C(5)	3507 (17)	−739 (17)	1384 (18)
N(1)	5036 (11)	2071 (12)	2447 (12)
C(12)	5961 (14)	1548 (18)	2917 (16)
C(13)	7305 (18)	1990 (22)	2769 (18)
C(14)	7739 (17)	3034 (19)	2192 (18)
C(15)	6785 (18)	3602 (18)	1703 (19)
C(16)	5459 (16)	3119 (16)	1861 (16)
C(6)	2401 (16)	4120 (17)	4485 (17)
N(2)	2854 (11)	3017 (12)	4413 (11)
C(21)	2001 (13)	3891 (14)	2065 (15)
C(22)	1902 (14)	4570 (14)	3427 (15)
C(23)	1375 (18)	5814 (18)	3797 (18)
C(24)	947 (18)	6376 (18)	2858 (22)
C(25)	1054 (17)	5702 (18)	1525 (18)
C(26)	1548 (15)	4495 (16)	1111 (15)
C(31)	3285 (15)	2883 (14)	5671 (14)
C(32)	4583 (16)	3379 (17)	6055 (17)
C(33)	4938 (20)	3295 (19)	7290 (18)
C(34)	4022 (20)	2706 (18)	8129 (18)
C(35)	2775 (21)	2185 (20)	7731 (18)
C(36)	2381 (17)	2236 (18)	6502 (16)

Discussion. Recently the crystal and molecular structure of the complex [Mo(η-allyl)(CO)$_2$(pd)(py)] (where pd is 2,4-pentanedionato) has been determined (Brisdon & Woolf, 1978) and, in contrast to many similar (η-allyl)(dicarbonyl)molybdenum complexes (Brisdon & Woolf, 1978, and references therein; Graham, Akrigg & Sheldrick, 1976; Cotton, Frenz & Stanislowski, 1973) which adopt a symmetric geometry for the bidentate ligand (LL_a, Fig. 1a), the pd moiety adopts an unsymmetric geometry (Fig. 1b) in this complex. In order to test whether this unsymmetric geometry is peculiar to pd, or perhaps typical of such three-electron-donor ligands, the structure of the complex [Mo(η-allyl)(CO)$_2$(py)(salNPh)] [where salNPh is N-phenylsalicylideneiminato, (C$_6$H$_5$NCHC$_6$H$_4$O)$^-$] was determined. If it is assumed that the allyl group occupies only one coordination site, this complex has three possible octa-

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34668 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
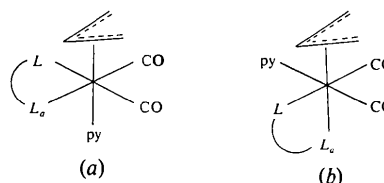

Fig. 1. Isomeric structures of [Mo(η-allyl)(CO)$_2$(LL_a)(py)].

Table 2. *Molecular dimensions* [*distances* (Å) *and angles* (°)]

Mo(1)—C(1)	1·908 (17)	C(1)··Mo(1)·C(2)	79·1 (7)
Mo(1)—C(2)	1·918 (17)	C(1)—Mo(1)—O(3)	98·9 (7)
Mo(1)—O(3)	2·135 (12)	C(1)—Mo(1)—N(1)	166·4 (6)
Mo(1)—N(1)	2·334 (11)	C(1)—Mo(1)—N(2)	85·7 (5)
Mo(1)—N(2)	2·222 (10)	C(2)—Mo(1)—O(3)	172·4 (5)
Mo(1)—C(3)	2·318 (18)	C(2)—Mo(1)—N(1)	96·0 (5)
Mo(1)—C(4)	2·241 (16)	C(2)—Mo(1)—N(2)	91·3 (5)
Mo(1)—C(5)	2·337 (17)	O(3)—Mo(1)—N(1)	84·2 (4)
		O(3)—Mo(1)—N(2)	81·2 (4)
		N(1)—Mo(1)—N(2)	81·7 (4)
C(3)—C(4)	1·37 (3)	C(1)—O(1)	1·19 (2)
C(4)—C(5)	1·42 (3)	Mo—C(1)—O(1)	177·7 (14)
C(3)—C(4)—C(5)	115·3 (17)	C(2)—O(2)	1·16 (2)
		Mo—C(2)—O(2)	178·2 (12)
O(3)—C(21)	1·29 (2)	Mo—O(3)—C(21)	131 (1)
C(22)—C(21)	1·41 (2)	O(3)—C(21)—C(22)	123 (1)
C(21)—C(26)	1·42 (2)	O(3)—C(21)—C(26)	119 (1)
C(22)—C(23)	1·41 (2)	C(22)—C(21)—C(26)	118 (1)
C(23)—C(24)	1·37 (3)	C(6)—C(22)—C(21)	123 (1)
C(24)—C(25)	1·38 (3)	C(6)—C(22)—C(23)	116 (1)
C(25)—C(26)	1·37 (3)	C(21)—C(22)—C(23)	120 (1)
C(22)—C(6)	1·43 (2)	C(22)—C(23)—C(24)	121 (1)
C(6)—N(2)	1·27 (2)	C(23)—C(24)—C(25)	118 (1)
N(2)—C(31)	1·44 (2)	C(24)—C(25)—C(26)	123 (1)
C(31)—C(32)	1·39 (2)	C(25)—C(26)—C(21)	120 (1)
C(32)—C(33)	1·38 (3)	N(2)—C(6)—C(22)	128 (1)
C(33)—C(34)	1·38 (3)	Mo—N(2)—C(6)	126 (1)
C(35)—C(34)	1·35 (3)	Mo—N(2)—C(31)	119 (1)
C(35)—C(36)	1·37 (3)	C(6)—N(2)—C(31)	114 (1)
C(36)—C(31)	1·40 (2)	C(32)—C(31)—C(36)	120 (2)
N(1)—C(12)	1·33 (2)	C(31)—C(32)—C(33)	119 (2)
C(12)—C(13)	1·38 (2)	C(32)—C(33)—C(34)	120 (2)
C(13)—C(14)	1·34 (3)	C(33)—C(34)—C(35)	120 (2)
C(14)—C(15)	1·39 (3)	C(34)—C(35)—C(36)	122 (2)
C(15)—C(16)	1·37 (3)	C(35)—C(36)—C(31)	119 (2)
C(16)—N(1)	1·35 (2)	N(2)—C(31)—C(32)	121 (1)
		N(2)—C(31)—C(36)	119 (1)
		Mo—N(1)—C(12)	121 (1)
		Mo—N(1)—C(16)	121 (1)
		C(12)—N(1)—C(16)	117 (1)
		N(1)—C(12)—C(13)	123 (2)
		C(12)—C(13)—C(14)	121 (1)
		C(13)—C(14)—C(15)	117 (2)
		C(14)—C(15)—C(16)	120 (2)
		C(15)—C(16)—N(1)	122 (2)

this equatorial plane. As usual (Brisdon & Woolf, 1978), the metal-to-carbon lengths in the allyl group show the shortest to be to the unique carbon C(4): 2·24 (2) Å compared to 2·32 (2) and 2·34 (2) Å to C(3) and C(5).

The Mo—CO bond lengths at 1·91 (2) and 1·92 (2) Å are as expected, and the bonds form the usual type of *fac* arrangement with the allyl group. Although we were not able to refine the allyl H atoms, it can be presumed that there is no significant difference in the Mo(allyl)(CO)₂ geometry in this structure and the others tabulated by Brisdon & Woolf (1978).
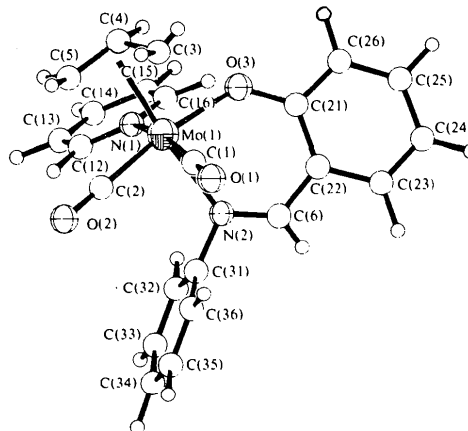
The pyridine group is almost coplanar with the equatorial plane, the angle of intersection being 6·2°. This is in contrast to the pd complex which gives rise to an angle of 36·6° and is probably a result of steric interactions between the H atoms of the pyridine and the methyl H atoms of pd. The bidentate ligand in the present structure is folded about the O(3)···N(2) vector, making an angle of 19·5° between the planes Mo, O(3), N(2) and N(2), C(21), C(22), C(23), C(24),

Table 3. ¹H *NMR spectrum of* [Mo(η-allyl)(CO)₂(py)-(salNPh)]

Assignment*		Chemical shift† $\delta_{p.p.m.}$	
		301 K	343 K
Hₐ η-allyl		1·43 (1,d,8) ⎫	1·50 (2,d,8)
Hₐ η-allyl		1·66 (1,d,9) ⎭	
H_c η-allyl		2·99 (1,m) ⎫	3·4 (3,br)
H_s η-allyl		3·62 (2,m) ⎭	
H_p pyridine		7·66 (1,t,8)	7·72 (1,br)
H_o pyridine		8·30 (2,m)	8·45 (2,m)
H_aromatic	{ salNPh +	6·43 (1,t,7)	6·45 (1,t,7)
	H_m pyridine	6·73–7·44 (10)	6·84–7·56 (10)
HC:N salNPh		7·78 (1,s)	7·88 (1,s)

* *a: anti, c: central, s: syn, o: ortho, p: para, m: meta.*

† Given in parentheses are the relative intensity, multiplicity and the coupling constant (Hz). Tetramethylsilane was used as internal reference ($\delta_{Me_4S} = 0$ p.p.m.).

hedral geometries, the position *trans* to the allyl group being occupied by (i) pyridine (Fig. 1*a*), (ii) the imine N atom [Fig. 1*b*: L = O(3), L_a = N(2)] or (iii) the phenolic O atom [Fig. 1*b*: L = N(2), L_a = O(3)].

In fact, as shown by the crystal structure (Fig. 2), the position is occupied by the imine N atom. Hence the structure is essentially similar to that reported for the related pd complex (Brisdon & Woolf, 1978). The Mo atom is in an approximately octahedral environment with the allyl group occupying one site. If the equatorial plane is taken as N(1), C(2), C(1) and O(3) (deviations 0·05, −0·06, 0·06, −0·05 Å from the least-squares plane), the Mo atom is displaced 0·18 Å towards the allyl group. The allyl group is almost parallel (14·8°) to

Fig. 2. Molecular structure of [Mo(η-allyl)(CO)₂(py)(salNPh)].

C(25), C(26), O(3), C(6). [Deviations of these nine atoms from the least-squares plane are, respectively, −0·02 (1), −0·01 (1), −0·03 (1), −0·01 (1), −0·00 (1), 0·02 (1), 0·00 (1), 0·01 (1), 0·05 (1) Å.] The phenyl ring on N(2) intersects the Mo, N(2), C(6) plane at 88·8°. The Mo–N(2) bond *trans* to allyl at 2·222 (10) Å is very much shorter than Mo–N(1) *trans* to carbonyl at 2·334 (11) Å.

The room-temperature ^{1}H NMR spectrum of [Mo(η-allyl)(CO)$_2$(py)(salNPh)] dissolved in CDCl$_3$ is given in Table 3 and is consistent with any of the three possible octahedral structures. However, it is not consistent with a fluxional molecule as reported for the related pd complex, nor with a mixture of isomers. On raising the temperature, the ^{1}H NMR spectrum simplified and at 343 K the allyl protons H$_a$ and H$_s$ were equivalent in pairs thus indicating a stereochemically non-rigid structure. Hence the ligand arrangement in Fig. 1(*b*) is not peculiar to the pd

complex and must reflect the electronic and/or steric requirements of these complexes.

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Structure of an Adduct of Orthotelluric Acid and Urea

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Abstract. Te(OH)$_6$.2CO(NH$_2$)$_2$, monoclinic, C2/c, a = 14·828 (8), b = 8·891 (6), c = 10·023 (7) Å, β = 129·13 (3)°, Z = 4, D_m = 2·31, D_c = 2·27 Mg m^{-3}. The structure consists of infinite Te(OH)$_6$.2CO(NH$_2$)$_2$ layers parallel to (100). Layers are connected through hydrogen bonds (Te–O–H···O–C). Within the layers the Te(OH)$_6$ molecules are hydrogen bonded to neighbouring Te(OH)$_6$ molecules and to urea molecules.

Introduction. The title compound was studied as part of an investigation of oxygen-containing Te compounds. The adduct was prepared by crystallization from a concentrated aqueous solution of orthotelluric acid and urea in a molar ratio of 1:3. Clear crystals were obtained, some as large, nearly regular prisms, of maximum dimensions 12 × 10 × 6 mm; the crystals had well developed faces, (100), (010) and (10Ī), the largest being (100), and the smallest (10Ī). Intensities were collected ($\omega/2\theta$ scan) in the range 3 ≤ 2θ ≤ 62° on a

Stoe four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. 1523 reflections were measured; those with $I \leq 3\sigma(I)$ were treated as unobserved. The remaining 1498 reflections were corrected for Lp and absorption. The positions of Te atoms were obtained from a Patterson synthesis. The remaining non-H atoms were located by Fourier and ΔF syntheses and refined by least squares to $R = 0·095$ with isotropic and to $R = 0·070$ with anisotropic temperature factors. The positions of the H atoms have not been determined. The final positional parameters are listed in Table 1.* The calculations were performed with *SHELX* 76 (Sheldrick, 1976) on an IBM 370/168 computer. The scattering factor for Te, which is not

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