positional parameters and the equivalent values of the anisotropic temperature factors U_{ij} [$U_{\rm eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23}\cos\alpha + 2U_{13}\cos\beta + 2U_{12}\cos\gamma)$] or the isotropic temperature factors U are given in the column headed U in Table 1. Bond lengths and angles are given in Table 2.

Through ligand substitution of the stronger π-acceptor CO for $P(C_6H_5)_3$ at the Mn atom *trans* to the Sn^{IV} atom, the Sn—Mn bond is shortened compared with those in $[SnCl\{Mn(CO)_5\}_3]$ (Tsai, Flynn & Boer, 1967) [Sn—Mn: 2·720 (6), 2·746 (6), 2·753 (6), 2·703 (6), 2·745 (6), 2·758 (6) Å] and $[SnBr\{Mn(CO)_5\}_3]$ (Haupt, Preut & Wolfes, 1978) [Sn—Mn: 2·739 (2), 2·722 (2), 2·758 (2) Å]. In the crystalline state $[SnCl\{Mn[(C_6H_5)_3P](CO)_4\}_3]$ has significantly different Sn—Mn and Mn—P distances which are probably brought about by intermolecular interactions. The bond angles at the central Sn atom agree with those in $[SnCl\{Mn(CO)_5\}_3]$ and $[SnBr\{Mn(CO)_5\}_3]$.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Haupt, H.-J., Preut, H. & Wolfes, W. (1978). *Z. Anorg. Allg. Chem.* **446**, 105–118.

Haupt, H.-J. & Schwab, B. (1978). Unpublished.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Sheldrick, G. M. (1976). *SHELX*. A program for crystal structure determination. Univ. of Cambridge, England.

Tsai, J. H., Flynn, J. J. & Boer, F. P. (1967). *Chem. Commun.* pp. 702–703.

Acta Cryst. (1981). B**37**, 690–692

Di-μ-chloro-dichlorobis(2,6-lutidine)diplatinum(II)* Dichloromethane Solvate

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(*Received* 19 *August* 1980; *accepted* 27 *October* 1980)

Abstract. $[Pt_2(C_7H_9N)_2Cl_4].CH_2Cl_2$, $C_{14}H_{18}Cl_4N_2Pt_2.CH_2Cl_2$, $M_r = 831{\cdot}24$, monoclinic, $C2/c$, $a = 20{\cdot}010\,(16)$, $b = 7{\cdot}574\,(5)$, $c = 14{\cdot}631\,(7)$ Å, $\beta = 96{\cdot}77\,(5)°$, $V = 2202\,(3)$ Å^3, $Z = 4$, $D_x = 2{\cdot}507$, $D_m = 2{\cdot}49\,(2)$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 14·07 mm^{-1} and $T = 295$ K. The structure was refined by full-matrix least-squares analysis to a conventional R factor of 0·036 and $R_w = 0{\cdot}027$ for 1670 reflexions. The molecule is dimeric with two chlorine bridges and a *trans* configuration for the two 2,6-lutidine ligands as required by the crystallographic inversion centre. The coordination around Pt is square planar. The terminal Pt—Cl bonds [2·272 (3) Å] are slightly shorter than normal, while the bridged bonds are slightly longer [2·320 (3) and 2·321 (3) Å]. The Pt—N bond is 2·020 (10) Å and the 2,6-lutidine ligand is perpendicular to the Pt coordination plane (88·5°). The dimeric molecule crystallizes with a molecule of solvent (dichloromethane). The C atom in CH_2Cl_2 is located on a C_2 axis.

Introduction. In attempting to synthesize complexes of the type *cis*-[Pt(am)(am′)Cl_2] (where am and am′ are different amines) to increase the screening range for anti-tumour properties of Pt compounds, we have isolated a new series of complexes [Pt(dmf)LCl_2], where dmf = dimethylformamide and L = a pyridine derivative (Kong & Rochon, 1979). In order to determine the configuration of the compounds, we studied the crystal structure of [Pt(dmf)(2,6-lutidine)Cl_2] (Rochon, Kong & Melanson, 1980).

When [Pt(dmf)LCl_2] is dissolved in certain solvents, such as chloroform, a dimer $[Pt_2L_2Cl_4]$ is partially formed. [Pt(dmf)(2,6-lut)Cl_2] (2,6-lut = 2,6-lutidine) was synthesized as described previously (Kong & Rochon, 1979) and dissolved in dichloromethane. Six weeks later, red crystals which proved to be the dimer $[Pt_2(2,6\text{-lut})_2Cl_4]$ were obtained at room temperature. Their crystal structure is reported here.

A set of precession photographs indicated that hkl, $h + k = 2n + 1$ and $h0l$, $l = 2n + 1$ are systematic absences. These extinctions are found for space groups Cc and $C2/c$. The cell parameters were obtained by least-squares refinement from the setting angles of 15 automatically centred reflections on a Syntex $P\bar{1}$ diffractometer using graphite-monochromatized Mo $K\alpha$ radiation.

The intensity data were collected from a crystal with eight faces and approximate dimensions 0·16 × 0·17 × 0·44 mm elongated along c. 2178 independent reflections were measured in the range $2\theta < 52°$ by the $2\theta/\theta$ scan technique using Mo $K\alpha$ radiation on a Syntex $P\bar{1}$

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* 2,6-Lutidine = 2,6-dimethylpyridine.

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diffractometer. The scan rate was 1° (2θ) min^{-1} for most reflections, but rates as high as 24° min^{-1} were selected for strong reflections by the autocollection program. A 2θ scan range of 1° below $K\alpha_1$ and 1° above $K\alpha_2$ was selected. The background time to scan time ratio was 0·40. During the data collection three standard reflections, measured after every 47 reflections, showed intensity fluctuations within ±3% of their mean values.

The 1670 reflections with $I > 2{\cdot}5\sigma(I)$ were considered observed, where $\sigma(I)$ was calculated as already described (Melanson & Rochon, 1975). An absorption correction based on the equations of the crystal faces was applied to all reflections. The transmission factors varied from 0·089 to 0·233. The data were then corrected for the Lorentz and polarization effects. The scattering factors of Cromer & Waber (1965) were used for Pt, Cl, N and C, those of Stewart, Davidson & Simpson (1965) for H. The anomalous-dispersion terms (Cromer & Liberman, 1970) of Pt and Cl were included in the calculations.

The Pt atom was easily located from the three-dimensional Patterson map which indicated space group $C2/c$ with a centre of symmetry between the two Pt atoms. The positions of all the other atoms, except the H atoms, were obtained by structure-factor and Fourier-map calculations. Some residual peaks were identified as a molecule of dichloromethane, which had been used to crystallize the compound. The C atom of the CH_2Cl_2 molecule is located on the C_2 axis. There is one molecule of CH_2Cl_2 per dimer or four molecules per unit cell. Refinement of the parameters was carried out by full-matrix least-squares methods on $\sum w(F_o - F_c)^2$ where $w = (a + b|F_o| + cF_o^2)^{-1}$. The constants were adjusted to make the distribution of $\langle w|\Delta F|^2\rangle$ almost invariable with respect to $|F_o|$ and $\sin\theta/\lambda$ (a = 116·5, b = −0·825 and c = 0·0015). The H atoms in the 2,6-lutidine ligand and in the CH_2Cl_2 molecule were fixed at their calculated positions* (C—H distance = 0·95 Å) and assigned isotropic B = 6·0 Å^2. The refinement converged to $R = \sum||F_o| - |F_c||/\sum|F_o|$ = 0·036 and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum|F_o|^2]^{1/2}$ = 0·027. The final residual electron density was within ±0·6 e Å^{-3}.

The calculations were carried out with a Cyber 73 computer and the programs used have already been described (Melanson & Rochon, 1975).

Table 1. *Positional parameters, with their e.s.d.'s, and temperature factors* (*all* × 10^4)

	x	y	z	U_{eq}* (Å^2)
Pt	825·1 (2)	4669·9 (6)	432·7 (3)	333·7
Cl(1)	1824 (1)	5969 (4)	247 (2)	519
Cl(2)	−213 (1)	3408 (4)	580 (2)	486
Cl(3)	151 (2)	1160 (7)	6561 (3)	1033
N	1326 (4)	2968 (14)	1330 (6)	384
C(1)	1140 (6)	5077 (19)	2550 (7)	525
C(2)	1406 (6)	3369 (16)	2236 (8)	381
C(3)	1732 (6)	2180 (19)	2846 (8)	448
C(4)	1989 (6)	667 (22)	2551 (10)	643
C(5)	1893 (6)	309 (22)	1634 (8)	611
C(6)	1566 (5)	1454 (17)	993 (8)	444
C(7)	1436 (7)	1082 (18)	−1 (9)	738
C(8)	0	23 (31)	7500	1168

* $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$.

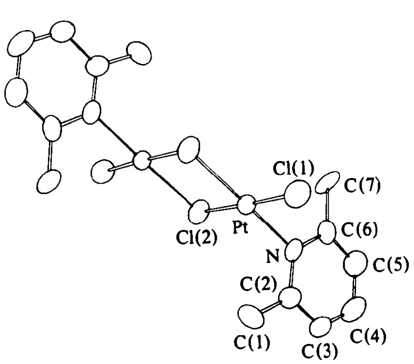

Fig. 1. Labelled diagram of the $[Pt_2(2,6\text{-lutidine})_2Cl_4]$ molecule.

Table 2. *Bond distances* (Å) *and angles* (°)

Pt—Cl(1)	2·272 (3)	C(3)—C(4)	1·35 (2)
Pt—Cl(2)	2·321 (3)	C(4)—C(5)	1·36 (2)
Pt—Cl(2′)	2·320 (3)	C(5)—C(6)	1·38 (2)
Pt—N	2·020 (10)	C(1)—C(2)	1·49 (2)
N—C(2)	1·35 (1)	C(6)—C(7)	1·48 (2)
N—C(6)	1·36 (2)	C(8)—Cl(3)	1·678 (13)
C(2)—C(3)	1·38 (2)		

Cl(1)—Pt—Cl(2)	177·9 (1)	N—C(2)—C(1)	119 (1)
Cl(1)—Pt—Cl(2′)	93·3 (1)	C(1)—C(2)—C(3)	122 (1)
Cl(2)—Pt—Cl(2′)	84·7 (1)	C(2)—C(3)—C(4)	121 (1)
Pt—Cl(2)—Pt′	95·3 (1)	C(3)—C(4)—C(5)	118 (1)
Cl(1)—Pt—N	88·8 (3)	C(4)—C(5)—C(6)	123 (1)
Cl(2)—Pt—N	93·3 (3)	N—C(6)—C(5)	116 (1)
Cl(2′)—Pt—N	177·9 (3)	N—C(6)—C(7)	119 (1)
Pt—N—C(2)	119·2 (8)	C(5)—C(6)—C(7)	124 (1)
Pt—N—C(6)	118·2 (7)	Cl(3)—C(8)—Cl(3′)	118·3 (8)
N—C(2)—C(3)	119 (1)		

Discussion. The refined atomic parameters are listed in Table 1. A view of the molecule is shown in Fig. 1. The molecule is dimeric with two Cl bridges and a *trans* configuration of the two 2,6-lutidine ligands, as required by the crystallographic inversion centre. The bond distances and angles are listed in Table 2.

The coordination around Pt is square planar. The weighted best plane was calculated through the five atoms. The deviations from this plane are: Pt, −0·0001 (5); Cl(1), 0·005 (3); Cl(2), 0·005 (3); Cl(2)′,

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* Lists of structure factors, anisotropic thermal parameters and calculated hydrogen positions have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35773 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

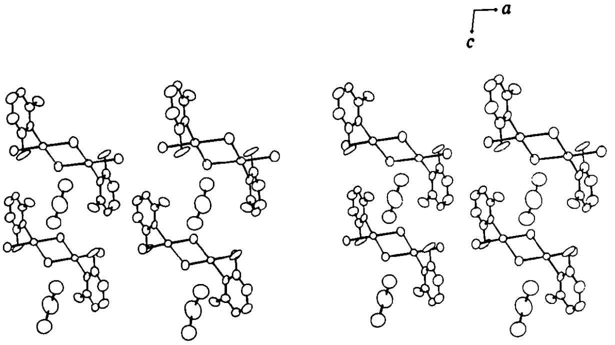

Fig. 2. Stereoscopic diagram of the packing in the $[Pt_2(2,6\text{-lutidine})_2Cl_4].CH_2Cl_2$ crystal.

−0·002 (3) and N, −0·017 (9) Å. The angles around the Pt atom are close to the expected 90 and 180° but there are some slight distortions. The Cl(2)—Pt—Cl(2)′ angle (84·7°) is smaller than expected for an ideal square plane. This deviation is probably caused by a slight strain inside the four-membered ring. The internal angle at the bridge Cl atom is 95·3°.

The terminal Pt—Cl bonds [2·272 (3) Å] are shorter than normal (2·29–2·30 Å) while the bridging bonds [2·320 (3) and 2·321 (3) Å] are longer than normal Pt—Cl bonds. These values agree well with those found in other Cl-bridged Pt complexes (Watkins, 1970; Elder, Cruea & Morrison, 1976; De Renzi, Di Blasio, Paiaro, Panunzi & Pedone, 1976; Black, Mais & Owston, 1969). Since the *trans* influence of nitrogen ligands is quite small, the two bridging Pt—Cl bonds are equal.

The Pt—N distance of 2·020 Å agrees well with published Pt—N distances in pyridine derivative compounds (Rochon, Kong & Melanson, 1980; Melanson & Rochon, 1976, 1977, 1978*a*,*b*). The 2,6-lutidine ligand is planar and is perpendicular to the Pt coordination plane (88·5°).

The distances within the 2,6-lutidine ligand are normal and agree with published structures mentioned above. The N—C distances are 1·35 (1) and 1·36 (2) Å while the aromatic C—C vary from 1·35 to 1·38 Å. The exocyclic C—CH_3 distances are 1·48 (2) and 1·49 (2) Å. The angles are close to the expected 120°.

A stereoscopic view of the packing in the crystal is shown in Fig. 2. There is one molecule of dichloromethane for each dimeric molecule. The H atoms are not shown in Fig. 2. The C atom in CH_2Cl_2 is located on a C_2 axis. The C—Cl(3) distance [1·678 (13) Å] is significantly shorter than the C—Cl bond length found in dichloromethane (1·768 ± 0·013 Å) at 153 K by Kawaguchi, Tanaka, Takeuchi & Watanabe (1973). It is not too far from the value (1·722 ± 0·009 Å) found by Oliver & Davis (1977) in a Pt complex where CH_2Cl_2 is a molecule of solvent. The angle Cl(3)—C(8)—Cl(3′) (118°) is slightly larger than the tetrahedral value. In CH_2Cl_2, the angle was found to be 112 ± 1°. The thermal parameters of C(8) and Cl(3) in dichloromethane are fairly large, indicating considerable agitation of the solvent molecules.

The dichloromethane molecules are packed between the four-membered Pt rings, contributing to the lattice energy of the crystal. There is no hydrogen bonding, all Cl···H—C distances being larger than the sum of the van der Waals radii.

Grateful acknowledgements are made to the Natural Sciences and Engineering Research Council of Canada, to the Ministère de l'Education (FCAC) for financial support and to Johnson Matthey Inc. for the sample of potassium tetrachloroplatinate.

References

BLACK, M., MAIS, R. H. B. & OWSTON, P. G. (1969). *Acta Cryst.* **B25**, 1760–1766.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

DE RENZI, A., DI BLASIO, B., PAIARO, G., PANUNZI, A. & PEDONE, C. (1976). *Gazz. Chim. Ital.* **106**, 765–768.

ELDER, R. C., CRUEA, R. D. P. & MORRISON, R. F. (1976). *Inorg. Chem.* **15**, 1623–1626.

KAWAGUCHI, T., TANAKA, K., TAKEUCHI, T. & WATANABE, T. (1973). *Bull. Chem. Soc. Jpn*, **46**, 62–66.

KONG, P. C. & ROCHON, F. D. (1979). *Can. J. Chem.* **57**, 682–684.

MELANSON, R. & ROCHON, F. D. (1975). *Can. J. Chem.* **53**, 2371–2374.

MELANSON, R. & ROCHON, F. D. (1976). *Can. J. Chem.* **54**, 1002–1006.

MELANSON, R. & ROCHON, F. D. (1977). *Acta Cryst.* **B33**, 3571–3573.

MELANSON, R. & ROCHON, F. D. (1978*a*). *Acta Cryst.* **B34**, 941–943.

MELANSON, R. & ROCHON, F. D. (1978*b*). *Acta Cryst.* **B34**, 1125–1127.

OLIVER, J. D. & DAVIS, R. E. (1977). *J. Organomet. Chem.* **137**, 373–382.

ROCHON, F. D., KONG, P. C. & MELANSON, R. (1980). *Can. J. Chem.* **58**, 97–101.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

WATKINS, S. F. (1970). *J. Chem. Soc. A*, pp. 168–172.