Lecomte, C., Chadwick, D. L., Coppens, P. & Stevens, E. D. (1983). *Inorg. Chem.* **22**, 2982–2992.
Li, N., Levendis, D., Coppens, P., Kastner, M. E., Carruth, L. E. & Scheidt, W. R. (1987). *Acta Cryst.* C**43**, 1835–1838.
Masuda, H., Taga, T., Osaki, K., Sugimoto, H., Yoshida, Z. & Ogoshi, H. (1980). *Inorg. Chem.* **19**, 950–955.
Molecular Structure Corporation (1988). *TEXSAN. Single Crystal Structure Analysis Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Oumous, H., Lecomte, C., Protas, J., Cocolios, P. & Guilard, R. (1984). *Polyhedron*, **3**, 651–659.
Reed, C. A., Mashkio, T., Bentley, S. P., Kastner, M. E., Scheidt, W. R., Spartalain, K. & Lang, G. (1979). *J. Am. Chem. Soc.* **101**, 2948–2958.
Scheidt, W. R. & Lee Y. J. (1987). *Structure and Bonding*. 64. *Metal Complexes with Tetrapyrrole Ligands* 1, edited by J. W. Buchler, pp. 1–70. New York: Springer-Verlag.
Scheidt, W. R., Lee, Y. J. & Finnegan, M. G. (1988). *Inorg. Chem.* **27**, 4725–4730.
Scheidt, W. R. & Reed, C. A. (1981). *Chem. Rev.* **81**, 543–555.

Acta Cryst. (1995). C**51**, 1494–1495

cis-Dichloro[*N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)-1,3-propanediamine]-chromium(III) Hexafluorophosphate

WING-TAK WONG

Department of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong

WAH-HUNG LEUNG

Department of Chemistry, The University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

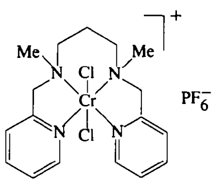
(*Received* 3 *January* 1995; *accepted* 14 *February* 1995)

Abstract

In the title compound, $[CrCl_2(C_{17}H_{24}N_4)]PF_6$, the coordination sphere of the Cr^{3+} ion is a distorted octahedron defined by the four N atoms of the tetradentate ligand and two *cis* Cl^- ions.

Comment

The coordination chemistry of the tetradentate ligand *N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)-1,3-propanediamine (L_1) has been investigated extensively (Che, Tang & Li, 1990; Che, Tang, Lee, Wong & Lau, 1992), but its structural chemistry is less well established. We therefore prepared a chromium complex, (I), of this ligand and determined its structure by X-ray analysis.



(I)

An *ORTEP*II (Johnson, 1976) plot of the complex cation with the atomic numbering scheme is shown in Fig. 1. The Cr—N_{amine} distances [average 2.146 (9) Å] are slightly longer than the Cr—$N_{pyridine}$ distances [average 2.093 (9) Å]. The two Cl^- ligands coordinate to Cr in a *cis* configuration. The six-membered ring defined by atoms Cr, N1, C1, C2, C3 and N2 has a chair conformation. Some disorder of the atoms of the PF_6^- anion is indicated by their large anisotropic displacement parameters.

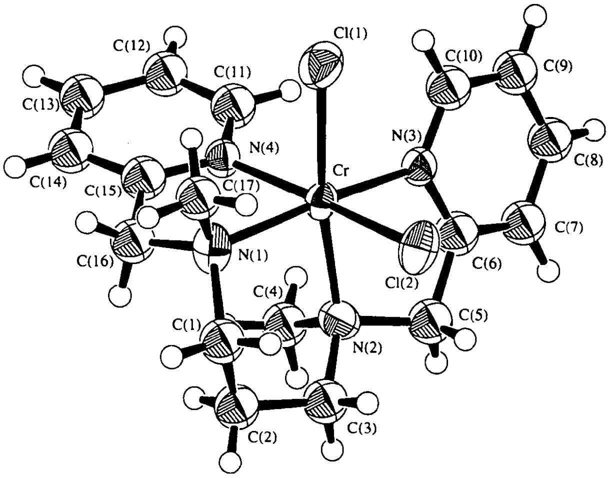


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the complex cation showing the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms are represented as spheres of arbitrary radii.

Experimental

The title complex containing the *N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)-1,3-propanediamine (L_1) ligand was prepared by the interaction of anhydrous $CrCl_3$ and L_1 in dimethylformamide. The dark yellow reaction mixture was heated under reflux overnight. The solvent was then evaporated off *in vacuo* and the dark yellow residue washed with diethyl ether and redissolved in a minimum amount of water. To the green filtrate was added excess $NaPF_6$. The resulting grey precipitate was collected, washed with water and dried in air. Recrystallization from acetonitrile/diethyl ether solution afforded green crystals suitable for X-ray analysis.

Crystal data

$[CrCl_2(C_{17}H_{24}N_4)]PF_6$
$M_r = 552.29$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Orthorhombic
Pbca
$a = 13.029\,(2)$ Å
$b = 25.722\,(9)$ Å
$c = 13.791\,(6)$ Å
$V = 4622\,(3)$ Å^3
$Z = 8$
$D_x = 1.587$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 10$–$14°$
$\mu = 0.84$ mm^{-1}
$T = 293$ K
Block
$0.42 \times 0.40 \times 0.12$ mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.813$, $T_{\max} = 0.999$
6291 measured reflections
4066 independent reflections

2067 observed reflections $[F_o > 3\sigma(F_o)]$
$R_{\rm int} = 0.041$
$\theta_{\max} = 24°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 24$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F
$R = 0.074$
$wR = 0.088$
$S = 3.061$
2067 reflections
195 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(F_o^2) + 0.04F_o^2]^2$
$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 0.94$ e Å^{-3}
$\Delta\rho_{\min} = -0.74$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$B_{\rm iso}$ for C atoms; $B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	$B_{\rm eq}$
Cr	0.2999 (1)	0.17772 (6)	0.6051 (1)	2.56 (3)
Cl(1)	0.4625 (2)	0.2069 (1)	0.6441 (2)	4.64 (7)
Cl(2)	0.3214 (2)	0.2031 (1)	0.4468 (2)	4.29 (6)
P	−0.0624 (3)	0.4631 (1)	0.7722 (3)	4.82 (8)
F(1)	0.0574 (8)	0.4605 (4)	0.785 (1)	10.7 (3)
F(2)	−0.059 (1)	0.5214 (3)	0.776 (1)	12.9 (4)
F(3)	−0.1759 (8)	0.4648 (5)	0.751 (2)	17.6 (6)
F(4)	−0.030 (1)	0.4657 (7)	0.6646 (8)	19.1 (6)
F(5)	−0.0623 (9)	0.4056 (4)	0.767 (2)	22.6 (7)
F(6)	−0.090 (2)	0.4602 (8)	0.8728 (9)	21.1 (7)
N(1)	0.3415 (8)	0.0981 (3)	0.5782 (6)	3.8 (2)
N(2)	0.1419 (7)	0.1632 (3)	0.5721 (7)	3.5 (2)
N(3)	0.2369 (6)	0.2498 (4)	0.6418 (6)	2.9 (2)
N(4)	0.2912 (7)	0.1482 (3)	0.7462 (6)	2.8 (2)
C(1)	0.286 (1)	0.0744 (5)	0.492 (1)	4.8 (3)
C(2)	0.171 (1)	0.0735 (5)	0.498 (1)	5.2 (3)
C(3)	0.121 (1)	0.1272 (5)	0.488 (1)	4.8 (3)
C(4)	0.083 (1)	0.1435 (5)	0.6604 (9)	4.5 (3)
C(5)	0.0967 (9)	0.2143 (5)	0.5454 (9)	4.0 (2)
C(6)	0.1398 (8)	0.2563 (4)	0.6104 (8)	3.5 (2)
C(7)	0.084 (1)	0.3022 (5)	0.629 (1)	5.3 (3)
C(8)	0.135 (1)	0.3416 (6)	0.680 (1)	5.5 (3)
C(9)	0.234 (1)	0.3358 (5)	0.7078 (9)	4.8 (3)
C(10)	0.2835 (8)	0.2889 (4)	0.6880 (8)	3.5 (2)
C(11)	0.2762 (8)	0.1770 (5)	0.8265 (8)	3.7 (2)
C(12)	0.279 (1)	0.1563 (5)	0.9192 (9)	4.7 (3)
C(13)	0.298 (1)	0.1032 (5)	0.9298 (9)	4.8 (3)
C(14)	0.3150 (9)	0.0731 (5)	0.8457 (9)	4.2 (2)
C(15)	0.3107 (8)	0.0980 (4)	0.7573 (8)	3.1 (2)
C(16)	0.321 (1)	0.0654 (5)	0.6656 (9)	4.4 (3)
C(17)	0.454 (1)	0.0940 (6)	0.554 (1)	5.8 (3)

Table 2. *Selected geometric parameters* (Å, °)

Cr—Cl(1)	2.313 (3)	N(4)—C(11)	1.35 (1)
Cr—Cl(2)	2.298 (4)	N(4)—C(15)	1.32 (1)
Cr—N(1)	2.148 (9)	C(1)—C(2)	1.51 (2)
Cr—N(2)	2.144 (9)	C(2)—C(3)	1.54 (2)
Cr—N(3)	2.09 (1)	C(5)—C(6)	1.51 (2)
Cr—N(4)	2.095 (9)	C(6)—C(7)	1.41 (2)
N(1)—C(1)	1.52 (2)	C(7)—C(8)	1.40 (2)
N(1)—C(16)	1.49 (2)	C(8)—C(9)	1.37 (2)
N(1)—C(17)	1.51 (2)	C(9)—C(10)	1.39 (2)
N(2)—C(3)	1.51 (2)	C(11)—C(12)	1.39 (2)
N(2)—C(4)	1.53 (2)	C(12)—C(13)	1.39 (2)
N(2)—C(5)	1.49 (1)	C(13)—C(14)	1.41 (2)
N(3)—C(6)	1.35 (1)	C(14)—C(15)	1.38 (2)
N(3)—C(10)	1.34 (1)	C(15)—C(16)	1.52 (2)
Cl(1)—Cr—Cl(2)	91.0 (1)	C(4)—N(2)—C(5)	106.9 (9)
Cl(1)—Cr—N(1)	96.7 (3)	Cr—N(3)—C(6)	113.7 (8)
Cl(1)—Cr—N(2)	171.0 (3)	Cr—N(3)—C(10)	127.1 (7)
Cl(1)—Cr—N(3)	91.0 (2)	C(6)—N(3)—C(10)	119.2 (9)
Cl(1)—Cr—N(4)	87.1 (3)	Cr—N(4)—C(11)	125.2 (7)
Cl(2)—Cr—N(1)	94.4 (2)	Cr—N(4)—C(15)	116.8 (7)
Cl(2)—Cr—N(2)	88.0 (3)	C(11)—N(4)—C(15)	117.9 (9)
Cl(2)—Cr—N(3)	91.6 (2)	N(1)—C(1)—C(2)	116 (2)
Cl(2)—Cr—N(4)	173.9 (2)	C(1)—C(2)—C(3)	114 (1)
N(1)—Cr—N(2)	92.3 (4)	N(2)—C(3)—C(2)	114 (2)
N(1)—Cr—N(3)	170.2 (4)	N(2)—C(5)—C(6)	109.6 (9)
N(1)—Cr—N(4)	80.2 (3)	N(3)—C(6)—C(5)	117 (1)
N(2)—Cr—N(3)	80.1 (3)	N(3)—C(6)—C(7)	122 (2)
N(2)—Cr—N(4)	94.7 (3)	C(5)—C(6)—C(7)	121 (2)
N(3)—Cr—N(4)	94.2 (3)	C(6)—C(7)—C(8)	117 (1)
Cr—N(1)—C(1)	113.4 (7)	C(7)—C(8)—C(9)	121 (1)
Cr—N(1)—C(16)	110.7 (7)	C(8)—C(9)—C(10)	119 (1)
Cr—N(1)—C(17)	110.6 (7)	N(3)—C(10)—C(9)	122 (2)
C(1)—N(1)—C(16)	108.9 (9)	N(4)—C(11)—C(12)	124 (2)
C(1)—N(1)—C(17)	105 (1)	C(11)—C(12)—C(13)	119 (1)
C(16)—N(1)—C(17)	108.1 (9)	C(12)—C(13)—C(14)	119 (1)
Cr—N(2)—C(3)	116.3 (7)	C(13)—C(14)—C(15)	118 (2)
Cr—N(2)—C(4)	112.0 (7)	N(4)—C(15)—C(14)	124 (1)
Cr—N(2)—C(5)	106.3 (7)	N(4)—C(15)—C(16)	117.1 (9)
C(3)—N(2)—C(4)	108.8 (9)	C(14)—C(15)—C(16)	118.5 (9)
C(3)—N(2)—C(5)	106.3 (9)	N(1)—C(16)—C(15)	112.2 (9)

The structure was solved by Patterson methods. In the final cycles of full-matrix least-squares refinement, anisotropic displacement parameters were included only for Cr, Cl, P, F and N atoms. All calculations were performed using the *SDP* package (Enraf–Nonius, 1985). The high R value is probably due to the disordered hexafluorophosphate anion.

We thank the Hong Kong Research Grants Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1171). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Che, C. M., Tang, W. T., Lee, W. O., Wong, K. Y. & Lau, T. C. (1992). *J. Chem. Soc. Dalton Trans.* pp. 1551–1556.

Che, C. M., Tang, W. T. & Li, C. K. (1990). *J. Chem. Soc. Dalton Trans.* pp. 3735–3739.

Enraf–Nonius (1985). *Structural Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.