N1—Re—Cl1	85.98 (15)	C2—N1—Re	177.2 (5)
N1—Re—Cl2	85.41 (15)	N1—C2—C3	179.8 (7)
Cl1—Re—Cl2	171.37 (6)	C11—P1—C21	104.3 (3)
N1—Re—Cl3	179.58 (15)	C11—P1—C31	104.0 (3)
Cl1—Re—Cl3	94.41 (6)	C21—P1—C31	101.0 (3)
Cl2—Re—Cl3	94.20 (6)	C11—P1—Re	107.6 (2)
N1—Re—P1	90.03 (13)	C21—P1—Re	120.2 (2)
Cl1—Re—P1	89.54 (5)	C31—P1—Re	118.1 (2)
Cl2—Re—P1	89.87 (5)	C41—P2—C61	102.8 (3)
Cl3—Re—P1	90.14 (5)	C41—P2—C51	104.9 (3)
N1—Re—P2	87.89 (13)	C61—P2—C51	100.4 (3)
Cl1—Re—P2	88.10 (5)	C41—P2—Re	111.3 (2)
Cl2—Re—P2	92.17 (5)	C61—P2—Re	118.2 (2)
Cl3—Re—P2	91.96 (5)	C51—P2—Re	117.4 (2)
P1—Re—P2	176.96 (5)		
N1—Re—P1—C11	−1.4 (3)	N1—Re—P2—C41	0.1 (3)
Cl2—Re—P1—C21	32.1 (3)	Cl1—Re—P2—C61	32.8 (2)
Cl3—Re—P1—C21	−62.1 (3)	Cl3—Re—P2—C61	−61.6 (2)
Cl1—Re—P1—C31	−32.5 (2)	Cl2—Re—P2—C51	−35.4 (2)
Cl3—Re—P1—C31	61.9 (2)	Cl3—Re—P2—C51	58.9 (2)
Re—P1—C11—C12	−86.3 (5)	Re—P2—C41—C42	77.8 (5)
Re—P1—C21—C22	33.9 (6)	Re—P2—C51—C52	−19.2 (6)
Re—P1—C31—C36	−22.8 (6)	Re—P2—C61—C66	36.3 (5)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRC*-2 and *NRC*-2*A* (Ahmed, Hall, Pippy & Huber, 1973). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1973). *NRC Crystallographic Computer Programs for the IBM*/360. Accession Nos. 133–147. *J. Appl. Cryst.* **6**, 309–346.

Drew, M. G. B., Tisley, D. G. & Walton, R. A. (1970). *J. Chem. Soc. Chem. Commun.* pp. 600–601.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Huheey, J. (1972). *Inorganic Chemistry. Principles of Structure and Reactivity*, p. 195. New York: Harper & Row.

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

Parkes, R. L., Payne, N. C. & Sherman, E. O. (1980). *Can. J. Chem.* **58**, 1042–1045.

Parkin, G. (1993). *Chem. Rev.* **93**, 887–911.

Pearson, C. & Beauchamp, A. L. (1995). *Inorg. Chim. Acta*, **237**, 13–18.

Pearson, C. & Beauchamp, A. L. (1997). *Can. J. Chem.* In the press.

Rossi, R., Duatti, A., Magon, L., Casellato, U., Graziani, R. & Toniolo, L. (1982). *J. Chem. Soc. Dalton Trans.* pp. 1949–1952.

Rouschias, G. & Wilkinson, G. (1967). *J. Chem. Soc. A*, pp. 993–1000.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1997). **C53**, 430–432

Aqua(2,2′-bipyridyl-*N*,*N*′)trichloromanganese(III)

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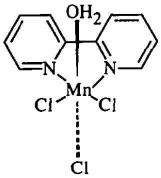
Abstract

In the structure of [$MnCl_3(C_{10}H_8N_2)(H_2O)$], the Mn^{III} atom has a distorted octahedral coordination. In the basal plane, the metal atom is coordinated to the bipyridyl ligand and two Cl atoms. The apical positions are occupied by a Cl atom and an aqua ligand. In the crystal, polymeric chains are formed *via* $H_2O\cdots Cl$ hydrogen bonds.

Comment

Manganese complexes have received considerable attention in recent years due to the presence of manganese centres in biological systems (Weighardt, 1989). The complexation behaviour of 2,2′-bipyridine (bpy) or bpy-related ligands with Mn ions gives rise to a wide variety of coordination complexes depending on the nature of the bpy ligand, the oxidation state of the Mn ion and the nature of the other coordinating ligands. Mn^{II} complexes with bpy-type ligands are well known. There are over 100 structures of this type in the Cambridge Structural Database (Allen & Kennard, 1993). If we look for mononuclear Mn complexes involving bpy and Cl ligands, the number is rapidly reduced to six, two of which form polymers. The majority are Mn^{II} complexes, for example, [$MnCl(bpy)_2(H_2O)$]ClO_4 (Chen, Shi, Mak & Luo, 1995), [$Mn(bpy)_2Cl_2$] (Lumme & Lindell, 1988) or *catena*-bis[μ_2-Cl–bpy–Mn^{II}] (Lubben, Meetsma & Feringa, 1995). Mn^{III} structures with bpy are rarer. The structure of the title compound, (I), can be compared with a basic molecular unit in the polymeric complex *catena*-[(μ_2-chloro)–dichloro–bpy–Mn^{III}] (Perlepes, Blackmann, Huffman & Christou, 1991), henceforth Mn^{III}PBHC.



(I)

In the title compound, the Mn atom is octahedrally coordinated to one bpy ligand, three Cl atoms and an aqua ligand. The coordinated bpy ligand imposes considerable strain on the coordination sphere. The same behaviour was also observed in Mn^{III}PBHC, which was studied at room temperature and 143 K. There is very little difference in the geometrical parameters at the two temperatures. The Mn—N distances and N—Mn—N bond angles are very similar in the two compounds. For example, the Mn—N distances are both 2.062 (6) Å in the title compound compared with 2.055 (4) and 2.056 (4) Å in Mn^{III}PBHC. The N—Mn—N bond angle is 78.5 (2)° compared with 79.0 (2)° [78.9 (2)° at 143 K] in Mn^{III}PBHC. The Mn—Cl2 and Mn—Cl3 distances in the basal plane are 2.209 (2) and 2.255 (2) Å, respectively, while the axial Mn—Cl1 distance is much longer [2.520 (2) Å]. The Mn—OH$_2$ distance is 2.288 (6) Å. In Mn^{III}PBHC, the Mn—Cl distances are 2.504 and 2.762 Å. The long axial Mn—Cl and Mn—OH$_2$ bonds indicate a Jahn–Teller distortion as expected for a d^4 Mn^{III} ion.

In the crystal, the molecules stack in the **a** direction and are hydrogen bonded to form a three-dimensional network. The axial Cl1 atom is hydrogen bonded to the axial aqua ligand (O1) of a molecule displaced by one unit cell in the **a** direction. The same aqua ligand (O1) is also hydrogen bonded to atom Cl2 of a molecule related by a centre of symmetry and also displaced by one unit cell in the **a** direction. The O···Cl distances [3.143 (7) and 3.250 (7) Å] are within the range (3.07–3.29 Å) of similar O···Cl hydrogen bonds observed previously (Chen *et al.*, 1995, and references therein). Details are given in Table 2 and Fig. 2.

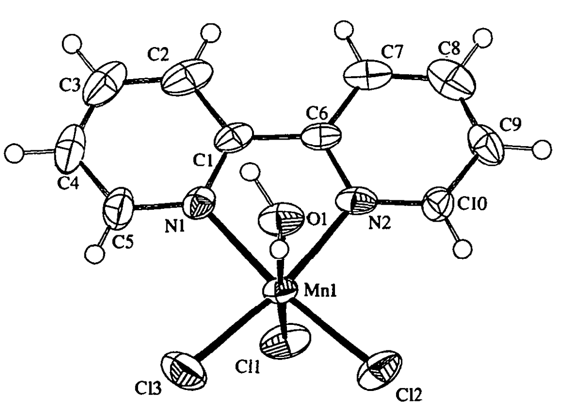


Fig. 1. A perspective view of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

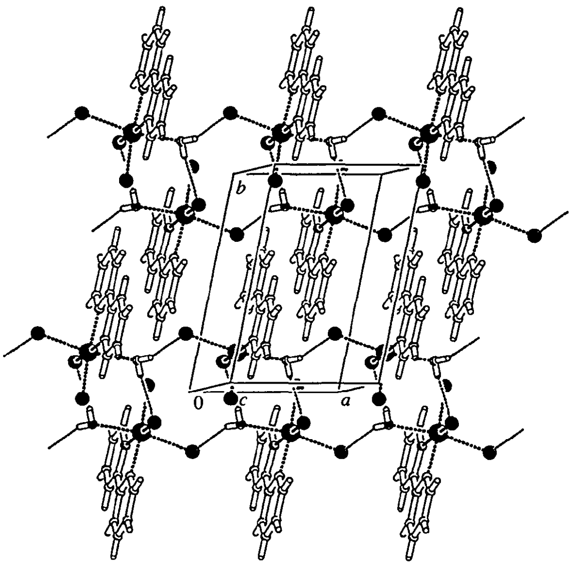


Fig. 2. Packing diagram for the title compound. The intermolecular hydrogen bonds are shown as the thinner dashed lines.

Experimental

The title compound was prepared by adding 1 ml of 1 *M* HCl to a solution of 0.2 g (0.22 mmol) of [Mn$_2$(μ-3-chlorobenzoate)$_2$(bpy)$_2$(H$_2$O)$_2$](NO$_3$)$_2$ in acetonitrile. The resulting red–brown solution was left to cool. After 24 h, a microcrystalline red–brown precipitate was formed and filtered off. The filtrate was kept in a refrigerator for one month, after which time red–brown crystals appeared (total yield: 67%).

Crystal data

[MnCl$_3$(C$_{10}$H$_8$N$_2$)(H$_2$O)]
M_r = 335.49
Triclinic
$P\overline{1}$
a = 6.641 (1) Å
b = 10.005 (2) Å
c = 10.114 (3) Å
α = 84.42 (2)°
β = 73.52 (1)°
γ = 78.45 (1)°
V = 630.8 (2) Å^3
Z = 2
D_x = 1.766 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 28 reflections
θ = 14–19°
μ = 1.628 mm^{-1}
T = 293 (2) K
Rod
0.38 × 0.23 × 0.19 mm
Red–brown

Data collection

Stoe AED-2 four-circle diffractometer

$R_{\rm int}$ = 0.0270
$\theta_{\rm max}$ = 25°

$2\theta/\omega$ scans
Absorption correction: none
2333 measured reflections
2199 independent reflections
1501 reflections with
  $I > 2\sigma(I)$

$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 12$
1 standard reflection
  frequency: 60 min
  intensity decay: 1.5%

Refinement

Refinement on F^2
$R(F) = 0.0691$
$wR(F^2) = 0.2508$
$S = 1.027$
2193 reflections
162 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1321P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.040$
$\Delta\rho_{\max} = 1.074$ e Å^{-3}
$\Delta\rho_{\min} = -1.119$ e Å^{-3}
Extinction correction: none
Scattering factors from
  International Tables for Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Mn1—N2	2.062 (6)	Mn1—Cl2	2.255 (2)
Mn1—N1	2.062 (6)	Mn1—O1	2.288 (6)
Mn1—Cl3	2.209 (2)	Mn1—Cl1	2.520 (2)
N2—Mn1—N1	78.5 (2)	Cl3—Mn1—O1	92.1 (2)
N2—Mn1—Cl3	171.4 (2)	Cl2—Mn1—O1	90.0 (2)
N1—Mn1—Cl3	94.0 (2)	N2—Mn1—Cl1	86.5 (2)
N2—Mn1—Cl2	94.2 (2)	N1—Mn1—Cl1	88.3 (2)
N1—Mn1—Cl2	171.6 (2)	Cl3—Mn1—Cl1	97.61 (9)
Cl3—Mn1—Cl2	92.96 (10)	Cl2—Mn1—Cl1	95.49 (8)
N2—Mn1—O1	83.1 (2)	O1—Mn1—Cl1	168.6 (2)
N1—Mn1—O1	85.1 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Cl1^{i}	0.79 (7)	2.39 (7)	3.143 (7)	160 (6)
O1—H12···Cl2^{ii}	0.76 (9)	2.50 (9)	3.250 (7)	172 (9)

Symmetry codes: (i) $1 + x, y, z$; (ii) $1 - x, -y, -z$.

The aqua H atoms were located from difference maps and refined isotropically. The aromatic H atoms were included in calculated positions as riding atoms with *SHELXL*93 (Sheldrick, 1993) default parameters.

Data collection: *STADI*4 (Stoe & Cie, 1995*a*). Cell refinement: *STADI*4. Data reduction: *XRED* (Stoe & Cie, 1995*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *PLATON*/*PLUTON* (Spek, 1990). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AV1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Chen, X.-M., Shi, K.-L., Mak, T. C. W. & Luo, B.-S. (1995). *Acta Cryst.* C**51**, 358–361.
Lubben, M., Meetsma, A. & Feringa, B. L. (1995). *Inorg. Chim. Acta*, **230**, 169–172.
Lumme, P. O. & Lindell, E. (1988). *Acta Cryst.* C**44**, 463–465.
Perlepes, S. P., Blackmann, A. G., Huffman, J. C. & Christou, G. (1991). *Inorg. Chem.* **30**, 1665–1668.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (1995*a*). *STADI4. Diffractometer Control Program for Windows*. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1995*b*). *XRED. Data Reduction Program for Windows*. Stoe & Cie, Darmstadt, Germany.
Weighardt, K. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 1153–1172.

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Sodium *trans*-Bis(methyliminodiacetato-*O*,*N*,*O*)chromate(III), Na[$Cr(C_5H_7NO_4)_2$]

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Abstract

In the title compound, Na[$Cr^{III}\{R\text{N}(CH_2CO_2)_2\}_2$], where R is CH_3, the methyliminodiacetate anions function as tridentate ligands defining a slightly distorted octahedral coordination at the Cr^{III} ion, with the N atoms in a *trans* configuration. The Cr—N distance of 2.060 (2) Å is slightly shorter than the values found in the complexes where R is bulkier, *i.e* an isopropyl or *tert*-butyl group, whereas the Cr—O bond lengths vary little as the size of R is changed. The methyliminodiacetato derivative appears to be the most symmetrical of the substituted iminodiacetato complexes so far studied.

Comment

Alkyliminodiacetatochromium(III) complexes, [$Cr^{III}\{R\text{-N}(CH_2CO_2)_2\}]^-$, are good models for structural and ligand-field analyses since the size of the R group affects both the Cr—N bond distance and the configuration at the Cr atom. The smallest substituent (R = H) yields a *cis* configuration, but when R is isopropyl, the metal adopts a *trans* configuration (Mootz & Wunderlich, 1980*a*,*b*). The relationship between the Cr—N bond length and ligand-field strength is also of interest. We