C4*A*	0.3961 (14)	0.1581 (8)	0.3109 (8)	0.049 (3)
C5*A*	0.3243 (14)	0.0673 (8)	0.3030 (7)	0.052 (3)
C6*A*	0.2622 (13)	0.0041 (7)	0.3866 (7)	0.043 (3)
C7*A*	0.1060 (13)	−0.0531 (7)	0.7562 (7)	0.047 (3)
C8*A*	0.0932 (17)	−0.0163 (8)	0.8454 (9)	0.064 (3)
C9*A*	0.1668 (17)	0.0724 (9)	0.8482 (8)	0.067 (4)
C10*A*	0.2532 (14)	0.1275 (8)	0.7629 (7)	0.048 (3)
C11*A*	0.2667 (11)	0.0925 (6)	0.6695 (6)	0.033 (2)
C12*A*	0.1899 (11)	0.0005 (6)	0.6666 (7)	0.037 (2)
C13*A*	0.1977 (11)	−0.0388 (6)	0.5716 (7)	0.033 (2)
C14*A*	0.2663 (11)	0.0294 (6)	0.4820 (7)	0.034 (2)
C15*A*	0.3422 (11)	0.1206 (7)	0.4901 (7)	0.034 (2)
C1*B*	−0.1166 (12)	0.4978 (6)	0.6888 (6)	0.030 (2)
C2*B*	−0.2993 (11)	0.4958 (7)	0.7591 (6)	0.035 (2)
C3*B*	−0.2077 (13)	0.6174 (7)	0.8953 (7)	0.045 (3)
C4*B*	−0.1724 (15)	0.6775 (9)	0.9633 (9)	0.058 (3)
C5*B*	−0.1854 (15)	0.6404 (10)	1.0617 (9)	0.062 (3)
C6*B*	−0.2310 (14)	0.5424 (9)	1.0956 (8)	0.051 (3)
C7*B*	−0.3432 (15)	0.2035 (8)	1.0174 (8)	0.057 (3)
C8*B*	−0.3474 (18)	0.1398 (9)	0.9462 (9)	0.070 (4)
C9*B*	−0.3197 (18)	0.1803 (9)	0.8449 (10)	0.070 (4)
C10*B*	−0.3016 (14)	0.2815 (8)	0.8172 (7)	0.048 (3)
C11*B*	−0.3033 (11)	0.3498 (7)	0.8886 (6)	0.036 (2)
C12*B*	−0.3221 (12)	0.3092 (7)	0.9903 (6)	0.038 (3)
C13*B*	−0.3120 (12)	0.3729 (8)	1.0656 (7)	0.043 (3)
C14*B*	−0.2678 (11)	0.4796 (7)	1.0301 (7)	0.037 (3)
C15*B*	−0.2583 (11)	0.5177 (7)	0.9269 (7)	0.036 (2)
O8	0.0841 (9)	0.7262 (5)	0.4496 (6)	0.055 (2)
O7†	0.163 (3)	0.6875 (11)	0.6801 (11)	0.052 (4)
O71†	0.249 (2)	0.6800 (10)	0.6620 (10)	0.041 (4)

† The assumed occupancy factor for atoms O7 and O71 is 0.5.

Table 5. *Selected geometric parameters (Å) for (II)*

Pb—O2*B*	2.501 (6)	Pb···Pb^{i}	4.046 (3)
Pb—O1*A*	2.523 (6)	Pb···Pb^{ii}	4.411 (3)
Pb—O2*B*^{i}	2.538 (6)	O8···O1*A*^{i}	2.743 (9)
Pb—O1*B*	2.576 (6)	O8···O3*A*^{iii}	2.838 (9)
Pb—O71	2.611 (13)	O7···O1*B*	2.957 (16)
Pb—O2*A*	2.739 (6)	O7···O3*A*^{iii}	2.735 (15)
Pb—O7	2.753 (14)	O7···O3*B*^{iv}	2.795 (16)
Pb—O8	2.824 (6)	O71···O3*A*^{iii}	2.832 (14)
Pb—O2*A*^{ii}	2.878 (6)	O71···O3*B*^{iv}	2.682 (14)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, 1+y, z$; (iv) $-x, 1-y, 2-z$.

Data collection, cell refinement and data reduction were performed using Kuma KM4 diffractometer software. In the case of (II), the standard reflections decreased in intensity by approximately 10% over the period of data collection; the data were therefore rescaled using the intensities of the standards. The structure of $[Ca(CMA)_2(H_2O)_2]$ was solved by direct methods. The refinement of $[Pb(CMA)_2(H_2O)_2]$ was initiated using the heavy-atom coordinates of $[Ca(CMA)_2(H_2O)_2]$. In both structures the C-bonded H atoms were included in geometrically calculated positions. The water H atoms in the Ca salt were found in a difference map and were refined with the restraint O—H = 0.97 Å. In the case of the Ca crystal, after refinement an additional significant peak was found on a difference Fourier map near the O(7) water molecule. It was admitted as a partially disordered O7 atom and assigned as O70. In the case of the Pb crystal, one water molecule was found to be disordered over two positions with occupancy factors of 0.5; the water H atoms were not resolvable. Refinement was performed using *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(1,10-phenanthroline)ruthenium(II) Bis(hexafluorophosphate)

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Abstract

$[Ru(phen)_3](PF_6)_2$ (phen = 1,10-phenanthroline), $[Ru(C_{12}H_8N_2)_3](PF_6)_2$, is homostructural with the perchlorate monohydrates of the Cu^{II}, Co^{II}, Os^{II} and Fe^{II} analogues. This homomorphism is notable in view of the widely differing metal-atom environments and counterion sizes, and suggests that the packing arrangement is a general and accommodating one among tris(1,10-phenanthroline)metal(II) complexes. While $[Ru(bpy)_3](PF_6)_2$ is built up from homochiral layers, $[Ru(phen)_3](PF_6)_2$ is composed of

rigorously racemic layers of complex cations parallel to the *bc* plane, with counterions sandwiched between. The average length of the Ru—N bonds [2.063 (4) Å] and the average ligand bite angle [79.8 (2)°] compare well with the values published for $[\mathrm{Ru(bpy)_3}](\mathrm{PF_6})_2$ [2.056 (2) Å and 78.7 (1)°, respectively].

Comment

In the course of our work on chiral recognition among trisdiimine–metal complexes (Breu & Catlow, 1995), the crystal structure of the title compound, (I), was determined in order to explore the influence of the ligand system on the self-assembly process of these conformationally rigid propeller-shaped compounds. Moreover, despite the extensive scrutiny that trisdiimine complexes of $\mathrm{Ru^{II}}$ have received (*e.g.* Yersin, Huber & Wiedenhofer, 1994), there is no structure reported for $[\mathrm{Ru(phen)_3}]^{2+}$ (phen = 1,10-phenanthroline) in the Cambridge Structural Database (Allen, Kennard & Taylor, 1983).

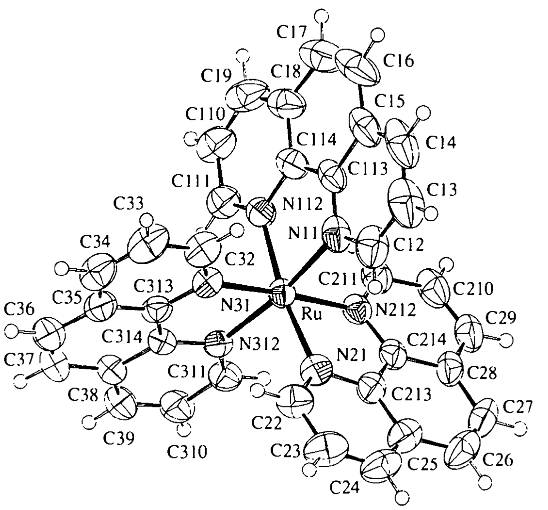
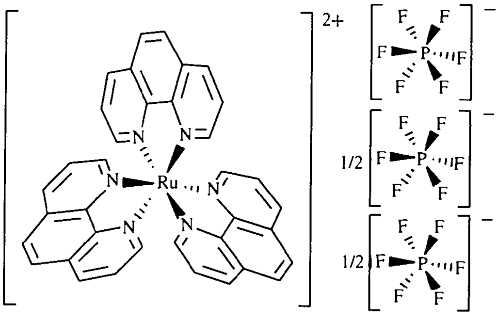


Fig. 1. Molecular structure showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level; H atoms are drawn as small circles of arbitrary radii. Anions are omitted for clarity.



(I)

Although the complex cation occupies a general site in the lattice, its molecular geometry corresponds to approximate D_3 symmetry (Fig. 1). The maximum standard deviation of the mean of the pseudo-symmetry-related bonds is 0.014 Å for C15—C16 and its equivalents, and the maximum standard deviation in the ligand bite and ligand angles is 1.2° for C12—C13—C14 and its equivalents. The largest deviations from D_3 symmetry are observed for the non-chelating angles in the Ru coordination sphere, with a maximum standard deviation of 2.3°, and are probably caused by packing effects.

Each phenanthroline ligand is planar; the maximum deviation from the best planes is 0.042 (5) Å. Bond lengths and angles in the phenanthroline ligands are identical within experimental error to those found in other structures (Anderson, 1973; Boys, Escobar & Wittke, 1984; Goodwin, Kepert, Patrick, Skelton & White, 1984; Koh *et al.*, 1994).

The average length of the Ru—N bonds [2.063 (4) Å] and the average ligand bite angle [79.8 (2)°] compare well with the values published for $[\mathrm{Ru(bpy)_3}](\mathrm{PF_6})_2$ (bpy = 2,2′-bipyridine) [2.056 (2) Å and 78.7 (1)°; Rillema, Jones, Woods & Levy, 1992]. Along the pseudo C_3 axis the molecular dimensions of $[\mathrm{Ru(phen)_3}]^{2+}$ and $[\mathrm{Ru(bpy)_3}]^{2+}$ are essentially the same. However, perpendicular to this axis, the $Cm6$–$Cm7$ bridges lead to a more disc-like shape of $[\mathrm{Ru(phen)_3}]^{2+}$, whereas $[\mathrm{Ru(bpy)_3}]^{2+}$ is almost spherical.

The most interesting feature of the crystal structure is the packing of the complex cations with respect to their chirality. As for many structures of trisdiimine complexes of this kind (Breu & Range, 1994), $[\mathrm{Ru(phen)_3}](\mathrm{PF_6})_2$ is composed of layers of complex cations parallel to the *bc* plane with counterions sandwiched between. The complex cations are oriented with their pseudo C_3 axis almost perpendicular to the layers. Due to the rigid planar structure of the ligand, $[\mathrm{Ru(phen)_3}]^{2+}$ is far from being completely spherical; it has three major pockets along the pseudo threefold axis which allow penetration of neighbouring complexes in a close-packed array. The cation layers now consist of zigzag chains of complex cations running along the *c* axis (Fig. 2) in which the intruding phenanthroline ligands are almost parallel. The tilt angle between these phenanthroline ligands is 1.56°, and the perpendicular distance between the centroid of the ring defined by C15, C16, C17, C18, C114 and C113, and the ring defined by N112, C111, C110, C19, C18 and C114 is only 3.410 Å. As a consequence of chirality, this kind of mutual penetration between complexes with parallel pseudo C_3 axes is only feasible if the complexes have an alternating configuration (Kuroda, Mason, Rodger & Seal, 1981).

This zipper-shaped structure leads to a shifted π-stacked arrangement of ligands in neighbouring complexes. Owing to the inherent polarity of aromatic sys-

Fig. 2. Space-filling packing diagram of the complex cation layers composed of zigzag chains of complex cations running from left to right.

tems, the electron-rich core being surrounded by an electron-poor torus of H atoms, this is a favourable 'π–π interaction' (Jorgensen & Severance, 1990) and may be responsible for the formation of the complex cation layers despite the charges of the $[Ru(phen)_3]^{2+}$ ions.

The neighbouring chains in the layers are once again connected *via* two pairs of almost parallel (tilt angle 25.9°) ligands. The Ru···Ru distance within a chain is 9.425 (2) Å, whereas to the adjacent chain it is 10.711 (3) Å.

Within the layers, each complex is surrounded by four other complexes of opposite chirality. Rigorously racemic layers are a common building block in all published $[M(phen)_3]^{n+}$ structures (Breu & Range, 1994). $[Ru(phen)_3](PF_6)_2$ is homostructural with the perchlorate monohydrates of the Cu^{II} (Anderson, 1973), Co^{II} (Boys *et al.*, 1984), Os^{II} (Goodwin *et al.*, 1984) and Fe^{II} (Koh *et al.*, 1994) analogues. This homomorphism is notable in view of the widely differing metal-atom environments and counterion sizes, and suggests that the packing arrangement is a general and accommodating one among tris(1,10-phenanthroline)-metal(II) complexes. In contrast to the above structures, $[Ru(phen)_3](PF_6)_2$ does not contain lattice solvent water and there is little, if any, disorder of the anions. The two anions in the 4*e* positions refine well, while the anion in the general position deviates somewhat from ideal octahedral geometry. However, no satisfactory model for a disorder of this site could be developed.

Experimental

$[Ru(phen)_3](PF_6)_2$ was obtained by metathesis of the chloride salt, which was prepared by refluxing $RuCl_2(Me_2SO)_4$ with four equivalents of 1,10-phenanthroline in water (Kahn, Bhardwaj & Bhardwaj, 1990). Suitable single crystals were obtained by recrystallization from ethanol/acetone (5:1).

Crystal data

$[Ru(C_{12}H_8N_2)_3](PF_6)_2$
$M_r = 931.62$
Monoclinic
$C2/c$
$a = 37.086$ (9) Å
$b = 16.045$ (5) Å
$c = 12.128$ (5) Å
$\beta = 101.425$ (15)°
$V = 7074$ (4) Å^3
$Z = 8$
$D_x = 1.750$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 757 reflections
$\theta = 2.11$–$23.77°$
$\mu = 0.636$ mm^{-1}
$T = 293$ (2) K
Prism
0.15 × 0.12 × 0.11 mm
Orange

Data collection

Stoe IPDS (image plate) diffractometer
Rotation scans
Absorption correction: none
9729 measured reflections
5217 independent reflections

3542 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0353$
$\theta_{max} = 23.77°$
$h = -40 \rightarrow 40$
$k = -18 \rightarrow 17$
$l = -10 \rightarrow 13$

Refinement

Refinement on F^2
$R(F) = 0.0435$
$wR(F^2) = 0.1142$
$S = 0.914$
5217 reflections
516 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0751P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.526$ e Å^{-3}
$\Delta\rho_{min} = -0.426$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ru	0.12193 (1)	0.27513 (2)	0.37931 (3)	0.0403 (1)
N11	0.14741 (10)	0.3509 (3)	0.2807 (4)	0.0544 (16)
N21	0.13260 (10)	0.1666 (3)	0.3004 (4)	0.0504 (16)
N31	0.16661 (9)	0.2703 (2)	0.5101 (4)	0.0436 (14)
N112	0.10808 (10)	0.3904 (2)	0.4347 (4)	0.0446 (14)
N212	0.07757 (9)	0.2726 (3)	0.2463 (3)	0.0438 (14)
N312	0.09984 (10)	0.2087 (2)	0.4962 (3)	0.0416 (14)
C12	0.16779 (15)	0.3289 (4)	0.2057 (5)	0.069 (2)
C13	0.1845 (2)	0.3855 (6)	0.1443 (6)	0.090 (3)
C14	0.1798 (2)	0.4687 (6)	0.1630 (6)	0.091 (3)
C15	0.1584 (2)	0.4960 (4)	0.2402 (6)	0.073 (3)
C16	0.1513 (2)	0.5789 (5)	0.2653 (8)	0.099 (3)
C17	0.1299 (2)	0.5999 (4)	0.3381 (8)	0.090 (3)
C18	0.1147 (2)	0.5382 (3)	0.4022 (6)	0.067 (2)
C19	0.0932 (2)	0.5547 (4)	0.4817 (7)	0.080 (3)
C22	0.16032 (14)	0.1123 (3)	0.3288 (6)	0.0605 (19)
C23	0.1661 (2)	0.0463 (4)	0.2615 (7)	0.074 (3)
C24	0.1430 (2)	0.0344 (4)	0.1610 (6)	0.071 (3)
C25	0.11334 (14)	0.0873 (4)	0.1275 (5)	0.0583 (19)
C26	0.0869 (2)	0.0800 (4)	0.0242 (5)	0.073 (3)
C27	0.0596 (2)	0.1346 (4)	−0.0037 (5)	0.067 (3)
C28	0.05498 (14)	0.2027 (4)	0.0670 (4)	0.0551 (19)
C29	0.0271 (2)	0.2633 (4)	0.0438 (5)	0.063 (2)
C32	0.20020 (13)	0.3038 (3)	0.5164 (5)	0.0596 (19)
C33	0.22724 (15)	0.2996 (4)	0.6137 (6)	0.073 (3)
C34	0.2204 (2)	0.2600 (4)	0.7062 (6)	0.068 (2)
C35	0.18561 (14)	0.2246 (3)	0.7051 (5)	0.0573 (19)
C36	0.1749 (2)	0.1865 (4)	0.7995 (6)	0.074 (3)

C37	0.1403 (2)	0.1567 (4)	0.7923 (5)	0.076 (3)
C38	0.1137 (2)	0.1615 (3)	0.6912 (5)	0.056 (2)
C39	0.0779 (2)	0.1279 (4)	0.6770 (5)	0.067 (2)
C110	0.0797 (2)	0.4912 (4)	0.5359 (6)	0.068 (2)
C111	0.08782 (14)	0.4093 (3)	0.5108 (5)	0.0559 (19)
C113	0.14287 (13)	0.4324 (3)	0.2980 (5)	0.053 (2)
C114	0.12153 (13)	0.4548 (3)	0.3800 (5)	0.0548 (19)
C210	0.02496 (14)	0.3242 (4)	0.1181 (5)	0.065 (2)
C211	0.05037 (13)	0.3280 (3)	0.2204 (5)	0.0540 (19)
C213	0.10899 (13)	0.1530 (3)	0.1998 (4)	0.0472 (17)
C214	0.07970 (13)	0.2111 (3)	0.1710 (4)	0.0467 (17)
C310	0.05507 (15)	0.1343 (3)	0.5752 (5)	0.0585 (19)
C311	0.06647 (12)	0.1755 (3)	0.4860 (5)	0.0499 (19)
C313	0.15942 (12)	0.2326 (3)	0.6039 (4)	0.0442 (16)
C314	0.12353 (13)	0.2005 (3)	0.5975 (4)	0.0438 (17)
P1	0	0.03919 (11)	1/4	0.0455 (6)
F11	0.04360 (8)	0.0391 (2)	0.2846 (3)	0.0724 (14)
F12	−0.00296 (10)	0.0390 (2)	0.3785 (3)	0.0791 (16)
F13	0	0.1389 (2)	1/4	0.0677 (18)
F14	0	−0.0606 (2)	1/4	0.0668 (18)
P2	0	0.5851 (2)	1/4	0.0786 (10)
F21	0.0309 (2)	0.5179 (3)	0.2664 (5)	0.151 (3)
F22	0.0297 (2)	0.6519 (4)	0.2754 (7)	0.196 (4)
F23	0.0002 (2)	0.5841 (4)	0.3789 (5)	0.165 (3)
P3	0.24086 (5)	0.0400 (2)	0.0536 (2)	0.1000 (9)
F31	0.24495 (13)	0.0426 (4)	0.1833 (4)	0.133 (2)
F32	0.27711 (14)	−0.0087 (4)	0.0608 (5)	0.148 (3)
F33	0.2157 (2)	−0.0320 (6)	0.0502 (7)	0.233 (5)
F34	0.23781 (13)	0.0428 (6)	−0.0761 (4)	0.216 (5)
F35	0.2063 (2)	0.0939 (7)	0.0440 (7)	0.278 (6)
F36	0.2658 (2)	0.1198 (5)	0.0579 (7)	0.209 (4)

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

Ru—N11	2.061 (5)	Ru—N112	2.067 (4)
Ru—N21	2.063 (5)	Ru—N212	2.064 (4)
Ru—N31	2.056 (4)	Ru—N312	2.068 (4)
N11—Ru—N112	80.38 (17)	N31—Ru—N312	79.62 (15)
N21—Ru—N212	79.47 (17)		

H atoms were placed in calculated ideal positions and refined using a riding model, each with an isotropic displacement parameter set to 1.2 times the equivalent isotropic displacement parameter of the atom to which it is attached.

Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990), *INSIGHT*II (Biosym Technologies, 1993). Software used to prepare material for publication: *PLATON*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1107). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Chromium(III) Cyclam Complexes. 6. *trans*-Aquahydroxy(1,4,8,11-tetraazacyclotetradecane)chromium(III) Dibromide Hydrate

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Abstract

The X-ray analysis of the title compound, $[Cr(OH)(H_2O)(C_{10}H_{24}N_4)]Br_2.H_2O$, indicates that the cyclam moiety exists in a chair conformation. The *trans*-axial O atoms bonded to Cr both carry one well defined H atom, with each oxygen also bearing a partially occupied (50%) H atom. The O—Cr—O bond angle is almost linear [179.6 (1)°] and the O···O vector is tilted by 1.2 (2)° from the perpendicular to the CrN_4 plane.

Comment

The title compound, (I), is one of a series of salts of the complex ion *trans*-$[Cr(cyclam)X_2]^+$, where cy-