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Mg₂Ru₂Cl₁₀O·16H₂O {dimagnesium μ -oxo-bis[pentachlororuthenate(IV)] hexadecahydrate} crystallizes in the monoclinic system (space group $P2_1/c$). The structure consists of layers of [Ru₂Cl₁₀O]⁴⁻ anions, [Mg(H₂O)₆]²⁺ cations and water molecules stacked along the *a* axis. Only the O atom bonded to Ru occupies the $2a$ site with $\bar{1}$ symmetry. All the other atoms occupy general $4e$ sites. The crystal structure is stabilized by O—H···O and O—H···Cl interactions.

Comment

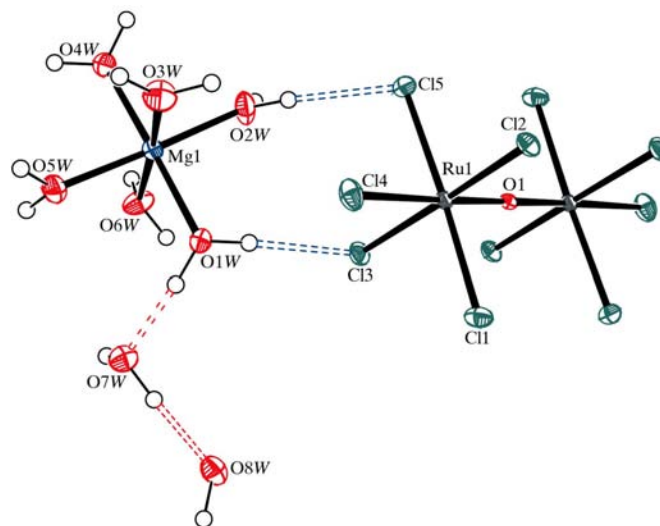
As part of an ongoing study of ruthenium materials in high oxidation states, the chemistry of ruthenium chlorides, fluorides and oxide fluorides is of interest. These materials are also of some value to the nuclear industry (Bourgeois & Cochet-Muchy, 1971).

The structure of Mg₂Ru₂Cl₁₀O·16H₂O presented here is the first example of a salt containing the Ru^{IV} oxychloride anion together with magnesium. Interestingly, few other derivatives of [Ru₂Cl₁₀O]⁴⁻ are known. The only previous structure reports are for K₄Ru₂Cl₁₀O (Deloume *et al.*, 1979) and Cs₄Ru₂Cl₁₀O (Santana Da Silva *et al.*, 1999), where Ru^{IV} is present with anhydrous alkali ions. One goal of the present work was therefore to examine the nature of the interactions between the [Ru₂Cl₁₀O]⁴⁻ anion and hydrated cations capable of forming hydrogen bonds.

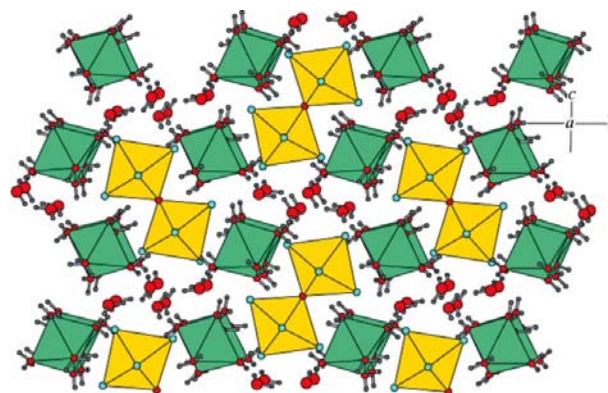
The asymmetric unit contains one [Ru₂Cl₁₀O]⁴⁻ anion, one [Mg(H₂O)₆]²⁺ cation and two water molecules (Fig. 1). The overall structure consists of layers stacked along the *a* direction, with dimeric [Ru₂Cl₁₀O]⁴⁻ units bridging adjacent layers (Fig. 2). The Ru atom resides in a distorted octahedron of one O and five Cl atoms, with an average Ru—Cl distance of

2.362 Å, similar to that in K₄Ru₂Cl₁₀O (2.362 Å), Cs₄Ru₂Cl₁₀O (2.357 Å) and K₄Ru₂Cl₁₀O·H₂O (2.360 Å). As expected, the Ru—O distance compares well with those reported for Cs₄Ru₂Cl₁₀O (1.791 Å), K₄Ru₂Cl₁₀O (1.800 Å) and K₄Ru₂Cl₁₀O·H₂O (1.797 Å). In the latter case, the structure was first determined (Mathieson *et al.*, 1952) from a zero-level Weissenberg photograph for the formula K₄Ru₂Cl₁₀O·H₂O. A subsequent fully anisotropic refinement ($R = 0.034$ for 245 measured reflections) has shown that there is, in fact, no water molecule in the structure (Deloume *et al.*, 1979). The correct formula is therefore K₄Ru₂Cl₁₀O.

The Mg²⁺ ion is surrounded by six water molecules *via* Mg²⁺—OH₂ ion–dipole interactions. The Mg—O distances (average Mg—O = 2.057 Å; Table 1) are longer than the values reported for magnesium bis(triazide) hexahydrate

**Figure 1**

The asymmetric unit of Mg₂Ru₂Cl₁₀O·16H₂O, shown with 50% probability displacement ellipsoids.

**Figure 2**

The structure of Mg₂Ru₂Cl₁₀O·16H₂O, viewed perpendicular to the (100) plane.

(average Mg—O = 2.024 Å; Mautner & Krischner, 1986) and similar to those in [Mg(H₂O)₆](C₁₄H₁₀Cl₂NO₂)·22H₂O (average Mg—O = 2.058 Å), where the Mg²⁺ ion is also hexahydrated (Castellari *et al.*, 1999).

Two types of intermolecular interactions are present in the structure, including O—H···O hydrogen bonds and O—H···Cl dipole–dipole interactions that contribute to hold cations and anions together. The presence of water molecules in the structure results in the presence of additional hydrogen bonds (Table 2). Five O—H···O hydrogen bonds are formed by the two water molecules that ensure the cohesion between the [Mg(H₂O)₆]²⁺ ions located in the same layer. The environment of the [Ru₂Cl₁₀O]^{4−} anion contains 12 O—H···Cl dipole–dipole interactions between the anion and the water molecules. Overall, the various interactions bridge the ruthenate anions within and between layers and form cavities occupied by the [Mg(H₂O)₆]²⁺ cations.

Experimental

The title compound was crystallized from a supersaturated hydrochloric acid solution (50%, 5 ml) prepared using doubly distilled water and a mixture of ruthenium(III) chloride trihydrate (2.61 g) and magnesium chloride (0.95 g) in a 1:1 ratio. Supersaturation was achieved by gentle warming of the solution. Thin brown needle-shaped single crystals of Mg₂Ru₂Cl₁₀O·16H₂O were obtained at ambient temperature by slow evaporation of the solution.

Crystal data

Mg ₂ Ru ₂ Cl ₁₀ O·16H ₂ O	Z = 2
M _r = 906.88	D _x = 2.112 Mg m ^{−3}
Monoclinic, P2 ₁ /c	Mo Kα radiation
a = 8.256 (2) Å	μ = 2.09 mm ^{−1}
b = 16.343 (3) Å	T = 173 (2) K
c = 10.647 (5) Å	Needle, brown
β = 95.499 (3)°	0.45 × 0.06 × 0.03 mm
V = 1430.0 (8) Å ³	

Data collection

Nonius KappaCCD diffractometer	8087 measured reflections
φ scans	2918 independent reflections
Absorption correction: part of the refinement model (ΔF) (Sheldrick, 1990)	2295 reflections with I > 2σ(I)
T _{min} = 0.760, T _{max} = 0.939	R _{int} = 0.038
	θ _{max} = 26.4°

Refinement

Refinement on F ²	w = 1/[σ ² (F _o ²) + (0.0098P) ² + 0.1594P]
R[F ² > 2σ(F ²)] = 0.028	where P = (F _o ² + 2F _c ²)/3
wR(F ²) = 0.063	(Δσ) _{max} = 0.002
S = 1.04	Δρ _{max} = 0.58 e Å ^{−3}
2918 reflections	Δρ _{min} = −0.64 e Å ^{−3}
190 parameters	
H atoms treated by a mixture of independent and constrained refinement	

H atoms were fixed by geometric constraints using the HFIX command and allowed to ride on the attached O atom (O—H = 0.92–0.99 Å).

Data collection: COLLECT (Nonius, 1997); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997);

Table 1

Selected geometric parameters (Å, °).

Ru1—O1	1.7822 (6)	Mg1—O1W	2.093 (2)
Ru1—Cl1	2.3559 (9)	Mg1—O2W	2.051 (3)
Ru1—Cl2	2.3660 (10)	Mg1—O3W	2.056 (3)
Ru1—Cl3	2.3731 (10)	Mg1—O4W	2.065 (2)
Ru1—Cl4	2.3464 (11)	Mg1—O5W	2.047 (3)
Ru1—Cl5	2.3728 (9)	Mg1—O6W	2.048 (2)
O1—Ru1—Cl4	178.14 (2)	O6W—Mg1—O2W	93.02 (11)
O1—Ru1—Cl1	92.81 (4)	O2W—Mg1—O3W	92.14 (12)
O1—Ru1—Cl2	92.22 (2)	O5W—Mg1—O1W	88.18 (10)
O1—Ru1—Cl5	91.68 (3)	O4W—Mg1—O1W	178.25 (11)
O1—Ru1—Cl3	92.92 (2)	Ru1—O1—Ru1 ⁱ	180
O5W—Mg1—O6W	86.60 (11)		

Symmetry code: (i) −x + 1, −y + 1, −z + 1.

Table 2

Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H11W···Cl3	0.94 (3)	2.37 (3)	3.302 (3)	172 (3)
O1W—H21W···O7W	0.96 (3)	1.73 (3)	2.688 (4)	174 (3)
O2W—H12W···Cl5	0.95 (3)	2.25 (3)	3.180 (3)	164 (3)
O2W—H22W···O1W ⁱⁱ	0.96 (2)	2.03 (2)	2.956 (3)	162 (3)
O3W—H13W···Cl2 ⁱⁱⁱ	0.95 (3)	2.74 (3)	3.415 (3)	129 (2)
O3W—H13W···Cl3 ^{iv}	0.95 (3)	2.65 (3)	3.448 (4)	142 (2)
O3W—H23W···Cl1 ⁱⁱⁱ	0.97 (3)	2.82 (3)	3.349 (3)	115 (2)
O4W—H14W···O8W ^v	0.97 (3)	1.79 (3)	2.745 (4)	171 (3)
O4W—H24W···Cl3 ^v	0.96 (3)	2.43 (3)	3.363 (3)	166 (3)
O5W—H15W···Cl2 ^{vi}	0.96 (3)	2.46 (3)	3.191 (3)	133 (2)
O5W—H25W···Cl5 ^{vi}	0.96 (2)	2.44 (3)	3.237 (3)	140 (3)
O6W—H16W···Cl2 ^{vii}	0.92 (2)	2.44 (2)	3.351 (3)	171 (3)
O6W—H26W···O7W ⁱⁱ	0.95 (2)	1.83 (2)	2.750 (3)	164 (3)
O7W—H17W···O8W	0.96 (2)	1.83 (2)	2.777 (3)	168 (3)
O7W—H27W···Cl4 ^{viii}	0.96 (3)	2.29 (3)	3.220 (3)	163 (3)
O8W—H28W···Cl1 ^{viii}	0.96 (3)	2.32 (3)	3.272 (3)	176 (3)
O8W—H18W···Cl4 ^{viii}	0.98 (2)	2.35 (3)	3.313 (3)	166 (2)

Symmetry codes: (ii) x, −y + ½, z + ½; (iii) −x + 1, y − ½, −z + ½; (iv) x, −y + ½, z − ½; (v) −x, y − ½, −z + ½; (vi) x − 1, −y + ½, z − ½; (vii) x − 1, y, z; (viii) −x, −y + 1, −z.

program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and ATOMS (Dowty, 1995); software used to prepare material for publication: WinGX (Farrugia, 1999) and PARST (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BC3021). Services for accessing these data are described at the back of the journal.

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