

Crystal structure of dirubidium aquapentachlorochromate(III), $\text{Rb}_2[\text{CrCl}_5(\text{H}_2\text{O})]$

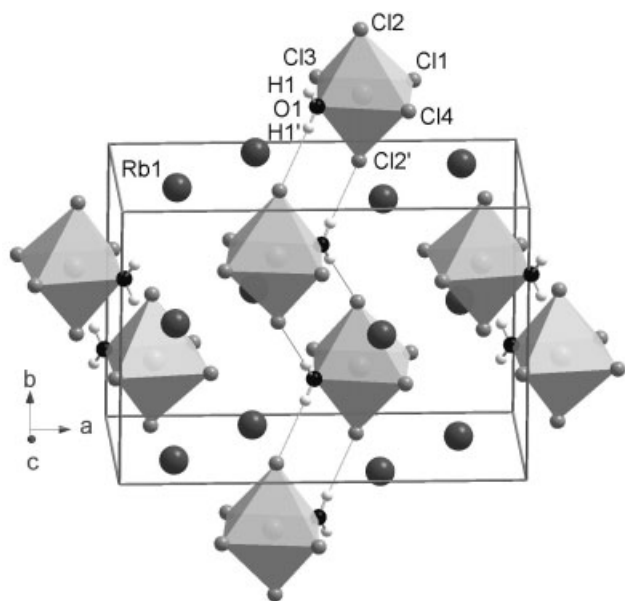
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Abstract

$\text{Cl}_5\text{CrH}_2\text{ORb}_2$, orthorhombic, *Pnma* (no. 62),
 $a = 13.8123(5)$ Å, $b = 9.7705(4)$ Å, $c = 7.1522(4)$ Å,
 $V = 965.2$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.038$,
 $wR_{\text{ref}}(F^2) = 0.073$, $T = 295$ K.

Source of material

The title compound was synthesized from aqueous solution under improved mild hydrothermal conditions. At first a mixture of 1.584 g CrCl_3 , 2.420 g RbCl , and an excess of HCl (37 %) was prepared to make the molar Cr:Rb ratio stoichiometric at 2:1. All starting materials were of analytical grade purity. The mixture was filled into a 20 ml Teflon lined stainless steel autoclave with a filling rate around 50 %. The autoclave was placed in an oven at 413 K for 14 days. The sealing gasket of the autoclave was improved so that the gas inside the autoclave could be slowly released when the pressure was higher than a certain value. This setup enables the aqueous solution to be evaporated in a steady manner. Finally, well-developed crystals were obtained. The title compound is sensitive to moisture and decomposes into hydrated CrCl_3 and RbCl . Therefore it is not easy to prepare crystals of the title compound by room temperature evaporating method or normal hydrothermal conditions.

Discussion

Two series of alkali aquapentachloride compounds $A_2[\text{MCl}_5(\text{H}_2\text{O})]$, where $A = \text{K}, \text{NH}_4, \text{Rb}, \text{Cs}$ and $M = \text{In}, \text{Fe}$, were intensively studied for their interesting properties, e.g. antiferromagnetism in Fe series. Except $\text{Cs}_2[\text{FeCl}_5(\text{H}_2\text{O})]$ compound which crystallizes in the space group *Cmcm* (no. 63), the remaining seven compounds all belong to space group *Pnma* (no. 62) and are actually isotypic [1–6]. We report here the crystal structure along with a modified crystal synthesis for the Rb derivate in the Cr series, because the structure has not yet been specified, though the compound is apparently isotypic with its Fe analogue [7].

The structure of the title compound is characterized by a distorted octahedral coordination of five Cl atoms and one O atom around the Cr atom, with the Cr—O distance (2.027 Å) considerably shorter than the Cr—Cl distances ranging from 2.323 Å to 2.361 Å. The bond lengths indicate that Cr is in the +3 state and the oxygen atom belongs to a water molecule [8]. Neighbouring octahedra are connected via hydrogen bonding between an H atom and a Cl atom, forming one-dimensional zig-zag chains along [010]. The H...Cl distance is 2.379 Å and the O—H...Cl angle is 175.6°. Furthermore, the Rb^+ ion is surrounded by ten Cl atoms and one O atom.

Table 1. Data collection and handling.

Crystal:	dark pink prism, size 0.15 × 0.15 × 0.10 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	125.17 cm ⁻¹
Diffractometer, scan mode:	Rigaku R-axis RAPID, $D = 127.4$ mm, $2\theta = -60^\circ - 60^\circ$, $\chi = 0^\circ$, $\varphi = 0^\circ$, $\omega = 50^\circ - 180^\circ$, $\Delta\omega = 5^\circ$, $\chi = 45^\circ$, $\varphi = 90^\circ$, $\omega = 50^\circ - 180^\circ$, $\Delta\omega = 5^\circ$ 60°
$2\theta_{\text{max}}$:	60°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	12027, 1481
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1112
$N(\text{param})_{\text{refined}}$:	53
Programs:	SHELXL-97 [9], DIAMOND [10]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	8d	-0.029(4)	0.185(5)	-0.039(8)	0.03(1)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Rb(1)	8 <i>d</i>	0.14543(3)	-0.00065(4)	0.65109(5)	0.0300(2)	0.0265(2)	0.0340(2)	-0.0007(2)	-0.0041(2)	0.0034(2)
Cr(1)	4 <i>c</i>	0.11028(6)	1/4	0.1875(1)	0.0183(3)	0.0142(3)	0.0198(4)	0	-0.0041(3)	0
Cl(1)	4 <i>c</i>	0.24266(9)	1/4	0.3877(2)	0.0197(5)	0.0227(5)	0.0228(5)	0	-0.0047(5)	0
Cl(2)	8 <i>d</i>	0.10353(7)	0.00958(8)	0.1785(1)	0.0319(4)	0.0153(3)	0.0365(5)	-0.0007(3)	-0.0083(4)	-0.0019(4)
Cl(3)	4 <i>c</i>	0.00690(9)	1/4	0.4503(2)	0.0230(6)	0.0266(6)	0.0386(7)	0	0.0063(6)	0
Cl(4)	4 <i>c</i>	0.2151(1)	1/4	-0.0693(2)	0.0420(7)	0.0311(7)	0.0199(6)	0	0.0041(5)	0
O(1)	4 <i>c</i>	-0.0065(4)	1/4	0.0158(8)	0.041(2)	0.022(2)	0.057(3)	0	-0.031(2)	0

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