Crystal structure of dirubidium aquapentachlorochromate(III), $Rb_2[CrCl_5(H_2O)]$

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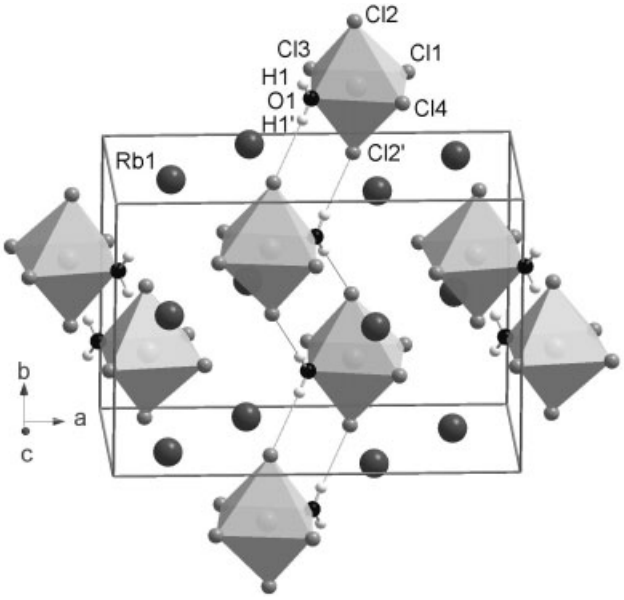

Abstract

$Cl_5CrH_2ORb_2$, orthorhombic, *Pnma* (no. 62),
a = 13.8123(5) Å, b = 9.7705(4) Å, c = 7.1522(4) Å,
V = 965.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.038,
$wR_{ref}(F^2)$ = 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 $A_2[MCl_5(H_2O)]$, where A = K, NH_4, Rb, Cs and M = In, Fe, were intensively studied for their interesting properties, e.g. antiferromagnetism in Fe series. Except $Cs_2[FeCl_5(H_2O)]$ 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, 2θ = −60° − 60°, χ = 0°, φ = 0°, ω = 50° − 180°, $\Delta\omega$ = 5°, χ = 45°, φ = 90°, ω = 50° − 180°, $\Delta\omega$ = 5°
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12027, 1481
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1112
$N(param)_{refined}$:	53
Programs:	SHELXL-97 [9], DIAMOND [10]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

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

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rb(1)	8*d*	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*c*	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*c*	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*d*	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*c*	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*c*	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*c*	–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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