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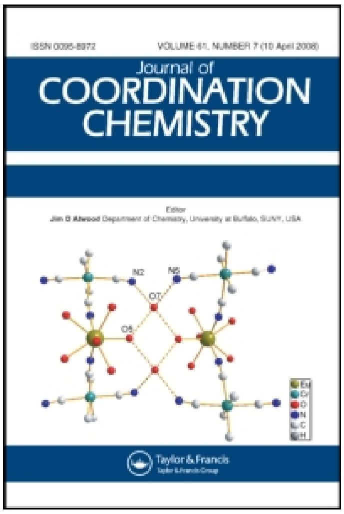

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CRYSTALLOGRAPHIC INVESTIGATIONS OF DIAQUATETRACHLORO-COBALT(II) AND NICKEL(II) COMPLEXES

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CRYSTALLOGRAPHIC INVESTIGATIONS OF DIAQUATETRACHLORO-COBALT(II) AND NICKEL(II) COMPLEXES

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The crystal structures of $2RbCl\cdot CoCl_2\cdot 2H_2O$ (**1**) and $2RbCl\cdot NiCl_2\cdot 2H_2O$ (**2**) have been determined by X-ray diffraction analyses. Both crystallize in triclinic space group $P\bar{1}$ with $a = 5.596(3)$, $b = 6.486(3)$, $c = 7.009(3)$ Å, $\alpha = 65.35(2)$, $\beta = 87.68(2)$, $\gamma = 84.58(3)^\circ$, $Z = 1$ for **1**, and $a = 5.571(2)$, $b = 6.450(3)$, $c = 6.968(2)$ Å, $\alpha = 65.52(4)$, $\beta = 87.57(3)$, $\gamma = 84.22(3)^\circ$, $Z = 1$ for **2**. The Co^{2+} and Ni^{2+} atoms are both coordinated in distorted *trans*-octahedral geometries by four Cl^- and two water molecules, and Rb^+ is surrounding by eight Cl^- ions. The average interatomic distances between the first transition metal(II) and chloride ions in *trans*-$[M(II)Cl_4(OH_2)_2]^{2-}$ (M(II) = Co, Ni) moieties are 2.479 and 2.437 Å for **1** and **2**, respectively, and the M(II)-O bond lengths are 2.107 and 2.070 Å for **1** and **2**, respectively. With the same M(II) cations, the M(II)-Cl distance is significantly longer than those of *cis*- and *trans*-$[M(II)Cl_2(OH_2)_4]$ octahedral moieties reported previously in $M(II)Cl_2\cdot 4H_2O$ and $M(II)Cl_2\cdot 6H_2O$ crystals.

KEYWORDS: X-ray structure, double salt, cobalt(II), nickel(II), chloride

INTRODUCTION

Tetrachloro complexes of cobalt(II) usually have tetrahedral geometry in both solution and solid states, and have been widely studied in many fields of chemistry.[1,2] It is also well known that the reaction of $[Co(OH_2)_6]^{2+}$ with chloride ions gives $[CoCl_4]^{2-}$ with structure change from octahedral to tetrahedral in aqueous solution and in the course of stepwise complex formation the step where

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the structural change occurs has been discussed repeatedly.[2] Krauß and Lehmann[3] suggested that the change is performed though an octahedral $[CoCl_4(OH_2)]^{2-}$ intermediate.

The double salt $2RbCl \cdot CoCl_2 \cdot 2H_2O$ (**1**) is known to crystallize from $RbCl—CoCl_2—H_2O$ ternary solution.[4,5] The structure of **1** has been suggested to be isomorphous with $2RbCl \cdot MnCl_2 \cdot 2H_2O$, which consists of one octahedral $[MnCl_4(OH_2)]^{2-}$ unit and two Rb^+ ions per unit cell,[6] because of the similarity of lattice parameters.[5] Therefore, an octahedral $[CoCl_4(OH_2)_2]^{2-}$ complex should exist in **1**. However, the crystal structure of **1** has not yet been established, and if the structure of $[CoCl_4(OH_2)_2]^{2-}$ was confirmed by X-ray diffraction methods, it would be the first verified $[CoCl_4(OH_2)_2]^{2-}$ complex with octahedral geometry.

In this paper, the crystal structure of **1** has been determined by X-ray diffraction methods to clarify the complete structure of $[CoCl_4(OH_2)_2]^{2-}$ and which might help to elucidate at which stage the change from octahedral to tetrahedral geometry occurs. The crystal structure of $2RbCl \cdot NiCl_2 \cdot 2H_2O$ (**2**) containing $[NiCl_4(OH_2)_2]^{2-}$ has been previously determined by Sproul,[7,8] but regrettably it was not published, and the lattice parameters have only recently been redetermined.[9] Thus, the structure of **2** has also been investigated and the $[M(II)Cl_4(OH_2)_2]^{2-}$ (M(II) = Co, Ni) structures are discussed in comparison with those of octahedral $[M(II)Cl_2(OH_2)_4]$ complexes reported previously.[10]

EXPERIMENTAL

Purple crystals of **1** suitable for X-ray examination were isolated in a plate-like form from an $RbCl—CoCl_2—H_2O$ solution at 25°C. Crystals of **2** were obtained in a yellow prismatic form from an $RbCl—NiCl_2—H_2O$ solution at 25°C. Experimental details and crystal data for the two crystals are listed in Table I. X-ray peak intensities were monitored by three standard reflections every 100 reflections. There was no significant variation in intensities during the data collections. The intensities were converted to F_o data in the usual manner. All reflections were corrected for Lorentz and polarization effects. An empirical absorption correction using DIFABS[11] was, after isotropic refinements for **1** and **2**, applied with corrections in the range 0.87–1.17 and 0.96–1.01, respectively. The structures were determined by the heavy atom method and refined by full-matrix least-squares methods. Several cycles of refinement were carried out including anisotropic thermal parameters for non-hydrogen atoms. All H atoms were included with isotropic thermal parameters in the structure factor calculations at the final stage of refinement; their positions were located from difference-Fourier syntheses. Atomic scattering factors and anomalous dispersion terms were taken from International Tables for X-ray Crystallography.[12] Calculations were performed on an Indigo computer at the Coordination Chemistry Laboratories, Institute for Molecular Science, using the program system teXsan[13] and partially on the CRAY C916 supercomputer at the Supercomputer Laboratory of the Institute for Chemical Research of Kyoto University. Final atomic parameters are summarized in Table II.

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Table I Crystal data and experimental details for $2RbCl \cdot CoCl_2 \cdot 2H_2O$ and $2RbCl \cdot NiCl_2 \cdot 2H_2O$.

	$2RbCl \cdot CoCl_2 \cdot 2H_2O$	$2RbCl \cdot NiCl_2 \cdot 2H_2O$
Formula weight	407.71	407.47
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	5.596(2)	5.571(2)
b (Å)	6.486(3)	6.450(3)
c (Å)	7.009(3)	6.968(2)
α (°)	65.35(2)	65.52(4)
β (°)	87.68(2)	87.57(3)
γ (°)	84.58(3)	84.22(3)
V (Å^3)	230.2(2)	226.7(2)
Z	1	1
$D_{calc.}$ (g cm^3)	2.941	2.948
T/K	296	296
$F(000)$	189	190
μ(Mo Kα) (cm^{-1})	134.4	136.6
Radiation (Mo Kα)	0.71069	0.71069
Diffractometer	AFC5R	AFC5R
Unit cell determination	25 reflections $28.15 \leq 2\theta \leq 29.54$	25 reflections $21.26 \leq 2\theta \leq 24.56$
Scan mode	ω–2θ	ω–2θ
Maximum 2θ (°)	60	60
hkl range	+ 8, ±9, ±10	+ 8, ±9, ±10
Scanning rate (°min^{-1})	6.0	6.0
Scan width (°)	$1.52 + 0.50\tan\theta$	$1.73 + 0.50\tan\theta$
Cutoff type	$\|I_o\| > 3\sigma \|I_o\|$	$\|I_o\| > 3\sigma \|I_o\|$
Total reflections measured	1472	1443
Reflection after cutoff	989	849
Parameters refined	46	46
R	0.044	0.048
R_w	0.051	0.056
Weighting scheme	$1/(\sigma^2(F_o))$	$1/(\sigma^2(F_o))$

RESULTS AND DISCUSSION

The coordination structures of complexes **1** and **2** are shown in Figure 1(a) and 1(b) with atomic numbering, respectively. The crystal structure in a unit cell of **1** is shown in Figure 2 with hydrogen bonds represented by broken lines. That of **2** is similar to that of **1**. The unit cell contains two Rb^+ ions and one slightly distorted *trans* octahedral $[M(II)Cl_4(OH_2)_2]^{2-}$ ion with the M(II) ion on an inversion centre. Selected interatomic distances and angles for **1** and **2** are listed in Table III.

The Co^{2+} and Ni^{2+} ions are hexa-coordinated by four Cl^- ions at equatorial positions in a crystallographically enforced planar fashion and by two water molecules in the axial positions. The average M(II)-Cl bond lengths, 2.479 and 2.437 Å for Co^{2+} and Ni^{2+} compounds, respectively, are longer than those of *trans*- and *cis*-$[M(II)Cl_2(OH_2)_4]$ complexes which found in $M(II)Cl_2 \cdot 6H_2O$[10] and $M(II)Cl_2 \cdot 4H_2O$[10(a),10(c)] crystals, respectively (Table IV), although the average M(II)—O distance of the $[M(II)Cl_4(OH_2)_2]^{2-}$ unit in the respective double salt crystals is similar to those of the *trans*- and *cis*-$[M(II)Cl_2(OH_2)_4]$ octahedra.[10] The longer M(II)—Cl distance is also found in the Mn^{2+} complexes, *i.e.*, 2.56 Å for

Table II Atomic parameters for $2RbCl\cdot CoCl_2\cdot 2H_2O$ and $2RbCl\cdot NiCl_2\cdot 2H_2O$.[a]

	x/a	y/b	z/c	B_{eq} or B_{iso} (Å^2)
$2RbCl\cdot CoCl_2\cdot 2H_2O$				
Co	0	0	0	1.5(1)
Rb	0.2436(2)	- 0.3317(1)	- 0.3874(1)	2.39(6)
Cl(1)	- 0.2332(4)	0.3773(3)	- 0.1252(3)	2.1(1)
Cl(2)	0.2530(4)	0.1350(3)	- 0.3197(3)	2.0(1)
O	0.237(1)	0.1125(9)	0.1550(9)	2.1(4)
H(1)	0.2865	0.2547	0.1345	8(3)
H(2)	0.3630	- 0.0259	0.2031	8(2)
$2RbCl\cdot NiCl_2\cdot 2H_2O$				
Ni	0	0	0	1.5(2)
Rb	0.2414(3)	- 0.3270(2)	- 0.3892(2)	2.3(1)
Cl(1)	- 0.2309(6)	0.3722(5)	- 0.1218(5)	2.0(2)
Cl(2)	0.2537(6)	0.1371(5)	- 0.3140(5)	1.9(2)
O	0.231(2)	0.109(1)	0.156(1)	2.1(6)
H(1)	0.2350	0.2600	0.1420	8(2)
H(2)	0.3540	- 0.0190	0.1850	11(2)

[a] $B_{eq} = (8/3)\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$. H atoms were fixed and refined isotropically.

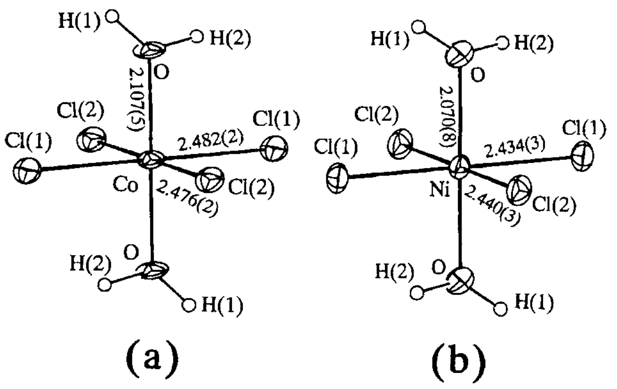

Figure 1 ORTEP drawings[14] of the molecular structure of (a) $[CoCl_4(OH_2)_2]^{2-}$ and (b) $[NiCl_4(OH_2)_2]^{2-}$ with thermal ellipsoids scaled at the 30% probability level. The bond lengths are in Å (see Table III). H atoms are represented by circles with radius 0.1 Å.

$[MnCl_4(OH_2)_2]^{2-}$ in $2RbCl\cdot MnCl_2\cdot 2H_2O$[6] *vs* 2.488 Å for *cis*-$[MnCl_2(OH_2)_4]$ in $MnCl_2\cdot 4H_2O$ (Table IV).[15] In the $[M(II)Cl_4(OH_2)_2]^{2-}$ units, the negative charge on the metal complex is dispersed and the central metal ion is neutralized. The elongation may be explained by the decrease of charge on central metal ion coordinated by four Cl^- ions and by an increase of electrostatic repulsion among Cl^- ions within the equatorial plane.

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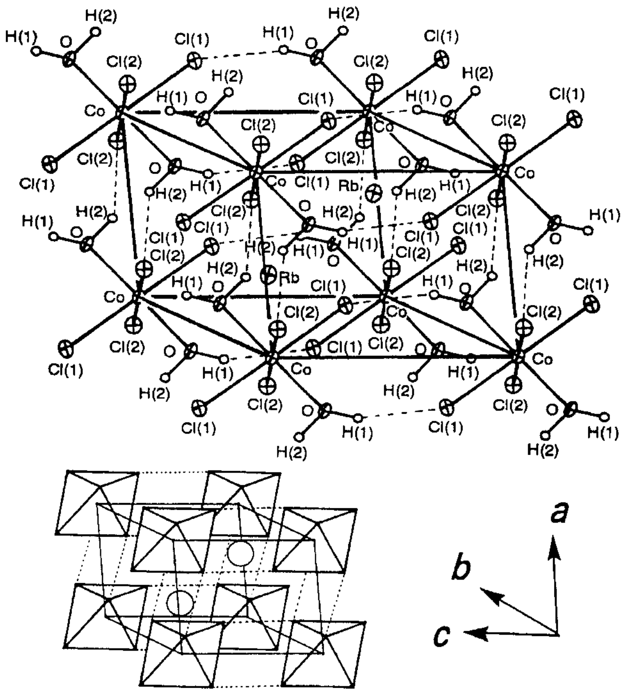

Figure 2 The crystal structure of $2RbCl \cdot CoCl_2 \cdot 2H_2O$ in the unit cell. In the ORTEPdrawing[14] (upper), non-H and H atoms are represented by thermal ellipsoids at 30% probability level and small circles with 0.1 Å radius, respectively. In the coordination polyhedra diagram (lower), octahedral polyhedra and open circles are $[CoCl_4(OH_2)_2]^{2-}$ and Rb^+ ions, respectively, Dotted lines indicate hydrogen bonds.

In both complexes **1** and **2**, the Rb^+ ion is surrounded with eight Cl^- ions from neighboring $[M(II)Cl_4(OH_2)_2]^{2-}$ complexes which form a distorted cube (Figure 3) Interatomic distances between Rb^+ and Cl^- ions vary from 3.226 to 3.553 Å for **1** and from 3.244 to 3.541 Å for **2**, respectively. The average distances of 3.405 and 3.400 Å for the Co^{2+} and Ni^{2+} compounds, respectively, are similar, and are also comparable to those of the Mn^{2+} complex (3.41 Å).[6]

In both crystals, the Cl^- ions and water molecules form a hydrogen bond network. The average hydrogen bond distances between Cl^- and $O(H_2)$ are 3.187 and 3.197 Å for **1** and **2**, respectively, as shown in Table III; these are slightly longer than typical Cl—O hydrogen bond distances, 3.12 Å, reported previously,[16] and that of $2RbCl \cdot MnCl_2 \cdot 2H_2O$ (3.23 Å)[6] seems also to be slightly longer than the typical value. Some degree of flexibility of hydrogen bonds would be important to maintain the structures of $2RbCl \cdot M(II)Cl_2 \cdot 2H_2O$ double salts including the Mn^{2+} complex.

Although no solution X-ray data have been reported for $[M(II)Cl_4(OH_2)_2]^{2-}$, tetrachloro complexes may possibly form not only tetrahedral species such as $[M(II)Cl_4]^{2-}$ but also *trans*-octahedral ones as described here in $RbCl—M(II)Cl_2—H_2O$ solutions under suitable conditions.

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Table III Interatomic distances and angles.[a]

	$2RbCl\cdot CoCl_2\cdot 2H_2O$	$2RbCl\cdot NiCl_2\cdot 2H_2O$
	M = Co	M = Ni
Distances (Å)		
M-Cl(1)	2.482(2)	2.434(3)
M-Cl(1)^{b}	2.482(2)	2.434(3)
M-Cl(2)	2.476(2)	2.440(3)
M-Cl(2)^{b}	2.476(2)	2.440(3)
M-O	2.107(5)	2.070(8)
M-O^{b}	2.107(5)	2.070(8)
Rb-Cl(1)^{c}	3.424(3)	3.432(4)
Rb-Cl(1)^{d}	3.466(2)	3.491(4)
Rb-Cl(1)^{b}	3.478(2)	3.448(3)
Rb-Cl(1)^{e}	3.553(2)	3.541(3)
Rb-Cl(2)	3.259(2)	3.253(3)
Rb-Cl(2)^{c}	3.285(3)	3.269(3)
Rb-Cl(2)^{e}	3.342(2)	3.331(4)
Rb-Cl(2)^{f}	3.436(2)	3.434(4)
Cl(1)-O^{g}	3.226(6)	3.244(8)
Cl(2)-O^{h}	3.147(6)	3.147(9)
Cl(1)-H(1)^{g}	2.351(2)	2.315(3)
Cl(2)-H(2)^{h}	2.271(2)	2.330(3)
O-H(2)	1.030(5)	0.929(7)
O-H(1)	0.939(5)	0.985(8)
Angles (°)		
O-M-O^{b}	180	180
O-M-Cl(1)	89.4(2)	89.1(2)
O-M-Cl(1)^{b}	90.6(2)	90.9(2)
O-M-Cl(2)	90.6(2)	90.1(2)
O-M-Cl(2)^{b}	89.4(2)	89.9(2)
O^{b}-M-Cl(1)	90.6(2)	90.9(2)
O^{b}-M-Cl(1)^{b}	89.4(2)	89.1(2)
O^{b}-M-Cl(2)	89.4(2)	89.9(2)
O^{b}-M-Cl(2)^{b}	90.6(2)	90.1(2)
Cl(1)-M-Cl(1)^{b}	180	180
Cl(1)-M-Cl(2)	90.55(8)	90.3(1)
Cl(1)-M-Cl(2)^{b}	89.45(8)	89.7(1)
Cl(1)^{b}-M-Cl(2)	89.45(8)	89.7(1)
Cl(1)^{b}-M-Cl(2)^{b}	90.55(8)	90.3(1)
Cl(2)-M-Cl(2)^{b}	180	180
M-O-H(1)	134.9(4)	126.4(6)
M-O-H(2)	98.6(3)	94.9(5)
H(1)-O-H(2)	118.5(5)	133.7(9)

[a] Estimated standard deviations are given in parentheses. Symmetry codes; (b) $-x, -y, -z$; (c) $x, -1+y, z$; (d) $1+x, -1+y, z$; (e) $-x, -y, -1-z$; (f) $1-x, -y, -1-z$; (g) $-x, 1-y, -z$; (h) $1-x, -y, -z$.

SUPPLEMENTARY MATERIAL

A listing of anisotropic thermal parameters for non-H atoms and tables of calculated and observed structure factors are available from the authors on request.

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Table IV Comparison of interatomic distances in octahedral Co^{2+} and Ni^{2+} complexes.[a]

Complex	Octahedral unit	Average interatomic distance (Å)	
		M—Cl	M—O
$2RbCl \cdot CoCl_2 \cdot 2H_2O^b$	$[CoCl_4(OH_2)_2]^{2-}$ (*trans*)	2.479(3)	2.107(5)
$CoCl_2 \cdot 6H_2O^c$	$[CoCl_2(OH_2)_4]$ (*trans*)	2.445(2)	2.081(5)
$CoCl_2 \cdot 4H_2O^c$	$[CoCl_2(OH_2)_4]$ (*cis*)	2.414(8)	2.110(11)
$2RbCl \cdot NiCl_2 \cdot 2H_2O^b$	$[NiCl_4(OH_2)_2]^{2-}$ (*trans*)	2.437(3)	2.070(8)
$NiCl_2 \cdot 6H_2O^d$	$[NiCl_2(OH_2)_4]$ (*trans*)	2.38	2.10
$NiCl_2 \cdot 4H_2O^e$	$[NiCl_2(OH_2)_4]$ (*cis*)	2.386(12)	2.071(8)
$2RbCl \cdot MnCl_2 \cdot 2H_2O^f$	$[MnCl_4(OH_2)_2]^{2-}$ (*trans*)	2.56(2)	2.08(4)
$MnCl_2 \cdot 4H_2O^g$	$[MnCl_2(OH_2)_4]$ (*cis*)	2.488(13)	2.206(14)

[a] Numbers in parentheses are e.s.d.s. [b] This work. [c] Ref. 10(a). [d] Ref. 10(b). [e] Ref. 10(a). [f] Ref. 6. [g] Ref. 15.

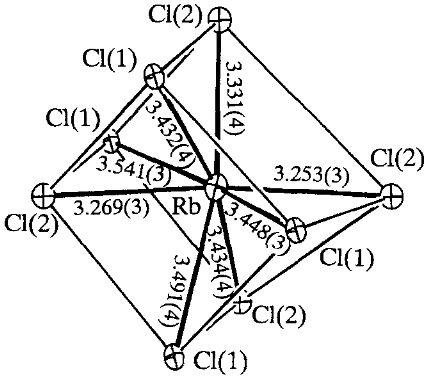

Figure 3 An ORTEP drawing[14] of the surrounding of the Rb^+ ion with thermal ellipsoids scaled at the 30% probability level. Figure 3 is viewed from the same direction as Figure 2 and refers to the Rb^+ located at the upper right in Figure 2(a). The bond lengths are in Å (see Table III).

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