

(expected, 4·10%), R factors for the integrated intensity and structure factors, R_i and R_F , were 7·36 and 5·34%, respectively, for 151 reflections. From the scale factors of the impurities, volume ratios $\text{Er}_2\text{O}_3:\text{Er}_2\text{BaNiO}_5 = 0\cdot032:1$ and $\text{NiO}:\text{Er}_2\text{BaNiO}_5 = 0\cdot015:1$ were calculated.

The results obtained from the neutron powder refinement are consistent with those previously determined by single-crystal X-ray diffraction (Schiffler & Müller-Buschbaum, 1987). Standard deviations of the oxygen positions are about six times smaller in the present study. No extra lines have been observed in the powder pattern, which confirms the symmetry and space group. Small irregularities in the background are thought to be due to the presence of small amounts of a glassy phase, probably related to BaO, remaining from the incomplete reaction between the starting reagents.

Bonding distances and angles for the polyhedra around the metallic atoms are given in Table 2. The most surprising feature in this structural type is the abnormal coordination of nickel, which is in a flattened octahedron of O atoms, $\text{Ni}(\text{O}1)_4(\text{O}2)_2$. In the erbium compound, nickel is coordinated to four O atoms at 2·180 Å and two more at shorter distances, 1·877 Å. Nickel octahedra form independent chains along the a axis via O2, as shown in Fig. 2. O1 and O2 positions are fully occupied within one and two standard deviations, respectively, which confirms the

oxygen stoichiometry and sixfold coordination of nickel.

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Structure of $\text{MgCl}_2\cdot\text{RbCl}\cdot 6\text{H}_2\text{O}$

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Abstract. Magnesium rubidium trichloride hexahydrate, $M_r = 324\cdot22$, triclinic, $P1$, $a = 6\cdot672$ (5), $b = 13\cdot282$ (15), $c = 6\cdot639$ (5) Å, $\alpha = 89\cdot83$ (8), $\beta = 91\cdot72$ (6), $\gamma = 90\cdot41$ (8)°, $V = 588\cdot0$ (14) Å³, $Z = 2$,

$D_x = 1\cdot831$ Mg m⁻³, $\bar{\lambda}(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 5\cdot17$ mm⁻¹, $F(000) = 320$, $T = 253$ K, $R(F) = 0\cdot078$ for 2604 independent reflections with $|F_o| > 3\sigma(|F_o|)$. The crystal consists of a network of top-sharing $[\text{RbCl}_6]$ octahedra and isolated $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$ octahedra occupying holes in the

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[RbCl₆] network. Rb⁺ ions are coordinated with six Cl⁻ ions and each Cl⁻ ion is surrounded by two Rb⁺ ions and four O atoms of water molecules. The Mg—O bond length varies from 1.982 (14) to 2.115 (13) Å in the [Mg(H₂O)₆]²⁺ octahedron. Each H₂O molecule is hydrogen bonded to two Cl⁻ ions.

Introduction. Andress & Saffe (1939) determined the crystal parameters of ammonium carnallite (MgCl₂·NH₄Cl·6H₂O) and rubidium carnallite (MgCl₂·RbCl·6H₂O) and reported that both crystals are of the same crystal system, monoclinic: $a = 13.30$, $b = 6.66$, $c = 6.68$ Å, $\beta \approx 90^\circ$, and $Z = 2$ for MgCl₂·NH₄Cl·6H₂O and $a = 13.30$, $b = 6.65$, $c = 6.62$ Å, $\beta \approx 90^\circ$, and $Z = 2$ for MgCl₂·RbCl·6H₂O. However, Nakayasu, Suzukawa & Kobayashi (1983) recently reinvestigated the crystal structure of MgCl₂·NH₄Cl·6H₂O and found that the crystal system is triclinic, $P1$, $a = 6.665$ (2), $b = 13.301$ (3), $c = 6.665$ (2) Å, $\alpha = 89.92$ (3), $\beta = 91.52$ (3), $\gamma = 89.99$ (3)°, $V = 590.6$ Å³ and $Z = 2$. In their study, Mg—O bond lengths in the crystal are different: two Mg—O bonds are shorter than the other four. They suggested that the structural change during the dehydration of MgCl₂·NH₄Cl·6H₂O to MgCl₂·NH₄Cl·2H₂O was probably caused by the loss of the four water molecules having the longer Mg—O bonds. It has also been known from the thermoanalytical investigations that MgCl₂·RbCl·6H₂O transforms into MgCl₂·RbCl·2H₂O through decomposition (Busch, 1959; Emons, Voigt, Pohl & Naumann, 1987), and a similar mechanism may be expected for MgCl₂·RbCl·6H₂O. The crystal structure of MgCl₂·RbCl·6H₂O has therefore been reinvestigated in order to check the previous result by Andress & Saffe (1939), especially from the viewpoint of the Mg—O bond lengths.

Experimental. The title double salt crystals were prepared from a solution of the ternary system MgCl₂·RbCl·H₂O by slow evaporation of the solvent. A colourless lumpy crystal with dimensions 0.3 × 0.4 × 0.4 mm was used for the data collection. The crystal was coated with paraffin and cooled to 253 K to avoid decomposition in air during the measurement. Diffraction intensities were measured on an Enraf-Nonius CAD-4 automated four-circle diffractometer with graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å). The unit-cell parameters were determined from setting angles of 25 reflections with $22.92 \leq 2\theta \leq 24.34^\circ$. Intensities of 3484 reflections ($-9 \leq h \leq 9$, $0 \leq k \leq 18$, $-9 \leq l \leq 9$) within the range $\theta < 30^\circ$ were collected using an ω - 2θ scan mode with a scan width of $(1.1 + 0.35 \tan \theta)^\circ$. Intensity statistics indicated the non-centrosymmetric space group $P1$. Reflections were monitored every two hours by three standard reflections, showing no

Table 1. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters for non-H atoms

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>B</i> _{eq} (Å ²) |
|-------|-----------|----------|-----------|--|
| Rb(1) | 0 | 0 | 0 | 2.7 |
| Rb(2) | -84 (2) | 4999 (1) | 77 (3) | 2.5 |
| Mg(1) | 4830 (12) | 2500 (7) | 5008 (15) | 1.4 |
| Mg(2) | 4904 (12) | 7482 (7) | 5060 (14) | 1.4 |
| Cl(1) | 4970 (6) | 71 (3) | -108 (7) | 3.0 |
| Cl(2) | 4772 (5) | 5001 (3) | 157 (5) | 1.9 |
| Cl(3) | 29 (6) | 16 (3) | 4921 (6) | 2.5 |
| Cl(4) | -262 (5) | 5076 (3) | 5121 (6) | 2.1 |
| Cl(5) | -212 (9) | 2501 (6) | 10 (12) | 2.2 |
| Cl(6) | -17 (10) | 7562 (6) | 23 (12) | 2.3 |
| O(1) | 5688 (17) | 3890 (9) | 6125 (15) | 3.3 |
| O(2) | 2066 (15) | 3008 (9) | 4185 (14) | 3.0 |
| O(3) | 5872 (15) | 2898 (7) | 2206 (16) | 2.8 |
| O(4) | 3913 (14) | 2119 (8) | 7761 (14) | 2.7 |
| O(5) | 7686 (14) | 1990 (8) | 5792 (17) | 2.8 |
| O(6) | 4151 (13) | 1092 (7) | 4072 (15) | 2.3 |
| O(7) | 7641 (13) | 7165 (7) | 4016 (12) | 2.0 |
| O(8) | 5662 (16) | 7093 (7) | 7862 (17) | 3.0 |
| O(9) | 4089 (14) | 8118 (7) | 2231 (13) | 2.1 |
| O(10) | 2067 (15) | 7929 (8) | 5941 (18) | 3.2 |
| O(11) | 3837 (14) | 6136 (7) | 4237 (15) | 2.4 |
| O(12) | 5911 (17) | 8941 (8) | 5766 (17) | 3.5 |

significant variation in the intensities. No absorption correction was applied [$\mu(\text{Mo K}\alpha) = 51.7 \text{ cm}^{-1}$]. The standard deviations, $\sigma(F_o)$, were estimated from counting statistics, and 2604 independent reflections with $|F_o| > 3\sigma(|F_o|)$ were used for the structure determination. The heavy-atom method was applied and the structure was refined by the full-matrix least-squares method. Several cycles of refinement (on F) including anisotropic temperature factors of non-H atoms were carried out with the weighting scheme $w = \{[\sigma(|F_o|)]^2 + (0.035|F_o|)^2\}^{-1}$. Positions of all H atoms were estimated by Fourier difference synthesis, and an isotropic temperature factor was assumed. The final values are: $R = 0.078$, $wR = 0.087$, $S = 1.918$, $(\Delta/\sigma)_{\text{max}} = 0.36$ for positional parameters and 0.16 for thermal parameters, $(\Delta\rho)_{\text{max}} = 3.5$, $(\Delta\rho)_{\text{min}} = -2.9 \text{ e } \text{Å}^{-3}$. Scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a HITAC M-680H computer at the Computer Center in the Institute for Molecular Science, using the program system UNICSIII (Sakurai & Kobayashi, 1979).

Discussion. The final atomic parameters are listed in Table 1.* Bond lengths and bond angles are summarized in Table 2. An ORTEP (Johnson, 1965) drawing of the crystal structure together with the atomic numbering is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53400 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

| | | | |
|-------------------|------------|------------------|------------|
| Rb(1)—Cl(1) | 3.319 (5) | Rb(1)—Cl(3) | 3.267 (5) |
| Rb(1)—Cl(5) | 3.326 (9) | Rb(1)—Cl(6) | 3.238 (9) |
| Rb(2)—Cl(2) | 3.242 (5) | Rb(2)—Cl(4) | 3.290 (5) |
| Rb(2)—Cl(5) | 3.319 (9) | Rb(2)—Cl(6) | 3.404 (9) |
| Mg(1)—O(1) | 2.062 (15) | Mg(1)—O(2) | 2.027 (13) |
| Mg(1)—O(3) | 2.071 (14) | Mg(1)—O(4) | 2.007 (13) |
| Mg(1)—O(5) | 2.078 (13) | Mg(1)—O(6) | 2.016 (13) |
| Mg(2)—O(7) | 2.020 (12) | Mg(2)—O(8) | 1.982 (12) |
| Mg(2)—O(9) | 2.115 (13) | Mg(2)—O(10) | 2.088 (14) |
| Mg(2)—O(11) | 1.991 (13) | Mg(2)—O(12) | 2.097 (15) |
| Cl(1)—O(4) | 3.138 (12) | Cl(1)—O(6) | 2.156 (11) |
| Cl(1)—O(9) | 3.081 (10) | Cl(1)—O(12) | 3.208 (12) |
| Cl(2)—O(1) | 3.138 (11) | Cl(2)—O(3) | 3.184 (11) |
| Cl(2)—O(8) | 3.225 (11) | Cl(2)—O(11) | 3.185 (11) |
| Cl(3)—O(5) | 3.125 (11) | Cl(3)—O(6) | 3.155 (10) |
| Cl(3)—O(10) | 3.159 (12) | Cl(3)—O(12) | 3.152 (12) |
| Cl(4)—O(1) | 3.204 (12) | Cl(4)—O(2) | 3.235 (13) |
| Cl(4)—O(7) | 3.190 (10) | Cl(4)—O(11) | 3.138 (10) |
| Cl(5)—O(2) | 3.193 (12) | Cl(5)—O(3) | 3.080 (12) |
| Cl(5)—O(4) | 3.214 (12) | Cl(5)—O(5) | 3.167 (13) |
| Cl(6)—O(7) | 3.159 (12) | Cl(6)—O(8) | 3.238 (13) |
| Cl(6)—O(9) | 3.152 (12) | Cl(6)—O(10) | 3.118 (14) |
| O(1)—Mg(1)—O(2) | 92.0 (6) | O(1)—Mg(1)—O(3) | 90.1 (5) |
| O(1)—Mg(1)—O(4) | 89.0 (6) | O(1)—Mg(1)—O(5) | 87.9 (5) |
| O(1)—Mg(1)—O(6) | 175.4 (6) | O(2)—Mg(1)—O(3) | 90.0 (5) |
| O(2)—Mg(1)—O(4) | 91.7 (5) | O(2)—Mg(1)—O(5) | 178.8 (7) |
| O(2)—Mg(1)—O(6) | 92.1 (6) | O(3)—Mg(1)—O(4) | 178.1 (6) |
| O(3)—Mg(1)—O(5) | 88.7 (5) | O(3)—Mg(1)—O(6) | 91.9 (6) |
| O(4)—Mg(1)—O(5) | 89.5 (6) | O(4)—Mg(1)—O(6) | 88.9 (6) |
| O(5)—Mg(1)—O(6) | 88.0 (5) | O(7)—Mg(2)—O(9) | 89.6 (5) |
| O(7)—Mg(2)—O(8) | 93.3 (5) | O(7)—Mg(2)—O(11) | 91.8 (5) |
| O(7)—Mg(2)—O(10) | 174.4 (7) | O(8)—Mg(2)—O(9) | 171.5 (7) |
| O(7)—Mg(2)—O(12) | 89.4 (5) | O(8)—Mg(2)—O(11) | 96.0 (6) |
| O(8)—Mg(2)—O(10) | 91.2 (6) | O(9)—Mg(2)—O(10) | 85.5 (5) |
| O(8)—Mg(2)—O(12) | 87.6 (6) | O(9)—Mg(2)—O(11) | 84.4 (5) |
| O(9)—Mg(2)—O(11) | 91.9 (5) | O(9)—Mg(2)—O(12) | 87.5 (6) |
| O(10)—Mg(2)—O(11) | 91.1 (5) | | |
| O(11)—Mg(2)—O(12) | 176.2 (7) | | |

Crystals of $\text{MgCl}_2 \cdot \text{RbCl} \cdot 6\text{H}_2\text{O}$ consist of a network of top-sharing $[\text{RbCl}_6]$ octahedra and isolated $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$ octahedra occupying holes in the $[\text{RbCl}_6]$ network as shown in Fig. 2. Each Rb^+ ion is octahedrally coordinated with six Cl^- ions such as those in RbCl (Pies & Weiss, 1973). The distance between Rb^+ and Cl^- ions varies from 3.238 to 3.404 Å. The average distance of 3.301 Å is in good agreement with that in RbCl , 3.295 (2) Å (Pies & Weiss, 1973), and the sum of each ionic radius, 3.30 Å (Shannon & Prewitt, 1969, 1970). Each Cl^- ion is surrounded by two Rb^+ ions and four water molecules of four $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$ moieties (see Table 2 and Fig. 1). The coordination around the Cl^- ions is different from those in the RbCl and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ crystals; in RbCl , each Cl^- ion is octahedrally coordinated with six Rb^+ ions, and in $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, a Cl^- ion is surrounded by eight O atoms of the water molecules of six $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$ moieties.

Each water molecule is hydrogen bonded to two Cl^- ions (see Fig. 1). The hydrogen-bonding $\text{Cl} \cdots \text{O}$ distances, 3.080 (12) ~ 3.235 (13) Å, are comparable with the average $\text{Cl} \cdots \text{O}$ hydrogen-bonding distance, 3.12 Å, reported previously (Pimentel & McClellan, 1960). The $\text{Cl} \cdots \text{O}$ hydrogen bond may play an

important role in the stabilization of the crystal structure of the double salt.

The two independent Mg^{2+} ions are both octahedrally coordinated with six water molecules. The $\text{Mg}-\text{O}$ bond length varies significantly from 2.007 (13) to 2.078 (13) Å for $\text{Mg}(1)$, and from 1.982 (14) to 2.115 (13) Å for $\text{Mg}(2)$. The $\text{Mg}-\text{O}$ bonds can be classified into two groups by the bond lengths: $[\text{Mg}(1)-\text{O}(1), \text{Mg}(1)-\text{O}(3), \text{Mg}(1)-\text{O}(5)]$ and $[\text{Mg}(1)-\text{O}(2), \text{Mg}(1)-\text{O}(4), \text{Mg}(1)-\text{O}(6)]$ for $\text{Mg}(1)$, and $[\text{Mg}(2)-\text{O}(9), \text{Mg}(2)-\text{O}(10), \text{Mg}(2)-\text{O}(12)]$ and $[\text{Mg}(2)-\text{O}(7), \text{Mg}(2)-\text{O}(8), \text{Mg}(2)-\text{O}(11)]$ for $\text{Mg}(2)$. The two $\text{Mg}-\text{O}$ bonds at the *trans* position belong to different groups, and the $\text{Mg}-\text{O}$ bonds of the long-distance group are longer than the $\text{Mg}-\text{O}$ bonds, 2.057 (1) and 2.062 (1) Å, in $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. A similar bond-length variation was also seen in $\text{MgCl}_2 \cdot \text{NH}_4\text{Cl} \cdot 6\text{H}_2\text{O}$, 1.96 (1)–2.14 (1) Å (Nakayasu *et al.*, 1983).

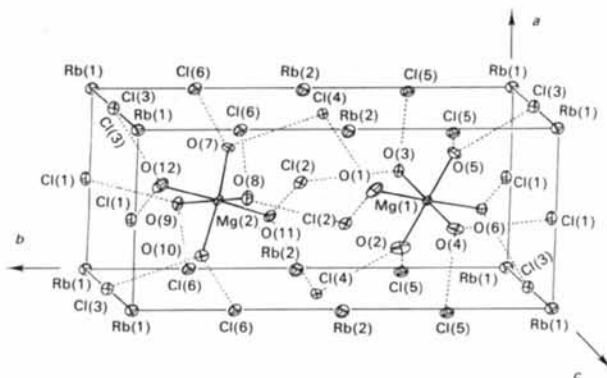


Fig. 1. An ORTEP drawing of $\text{MgCl}_2 \cdot \text{RbCl} \cdot 6\text{H}_2\text{O}$ with thermal ellipsoids scaled at the 30% probability level. Hydrogen bonds are represented by dashed lines.

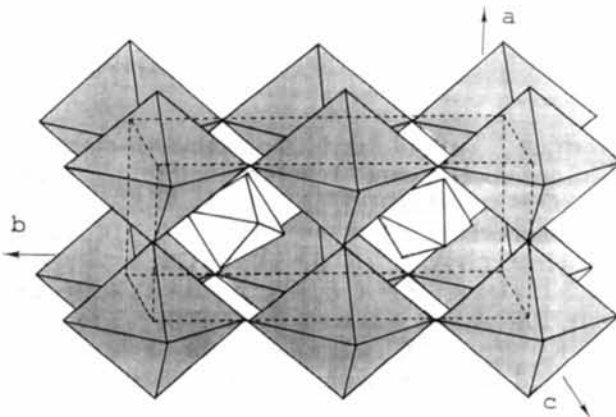


Fig. 2. Network of top-sharing $[\text{RbCl}_6]$ octahedra (large and dark shaded) and isolated $[\text{Mg}(\text{H}_2\text{O})_6]^{2+}$ octahedra (small).

Generally, the Mg^{II} ion prefers octahedral coordination. In MgCl₂·6H₂O, the Mg^{II} ion forms an almost regular octahedron (Agron & Busing, 1985). However, the Mg—O bond lengths in the double salts of MgCl₂·RbCl·6H₂O and MgCl₂·NH₄Cl·6H₂O vary widely due to the effect of the surrounding Cl⁻ ions. The different bond lengths in the coordination sphere of the Mg^{II} ion may be responsible for the dehydration process of the double salts.

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Structure of a Neutral *N*-Alkylated Diaminedithiol (dadt) ⁹⁹Tc^V Complex: *syn*[⁹⁹TcO(NEt-tmdadt)]

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Abstract. *syn*-(4-Ethyl-2,9-dimethyl-4,7-diaza-2,9-decanedithiolato)oxo(⁹⁹Tc)technetium(V), *syn*-[⁹⁹TcO(NEt-tmdadt)], [Tc(O)(C₁₂H₂₅N₂S₂)], *M_r* = 375, monoclinic, *P*2₁/*n*, *a* = 9.638 (2), *b* = 14.371 (5), *c* = 11.893 (3) Å, β = 100.79 (2)°, *V* = 1618.15 (75) Å³, *Z* = 4, *D_x* = 1.54 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 11.0 cm⁻¹, *F*(000) = 776, *T* = 293 K, *R* = 0.028, *wR* = 0.042 for 2788 unique observed reflections with *I* > σ(*I*). This study is the

first which conclusively establishes the structure of the major complex derived from the reaction of an *N*-substituted diaminedithiol ligand with reduced pertechnetate. An intramolecular repulsion exists between the ethyl substituent and the oxo-metal core, resulting in a larger-than-expected angle for N(2)—Tc—O, 104.7 (1)°.

Introduction. The metastable isotope of technetium, technetium-99m (^{99m}Tc, γ = 140 keV, *t*_{1/2} = 6.02 h), is the radionuclide chosen for performing the majority of diagnostic nuclear medicine procedures

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