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Trirubidium cobalt tetrachloride nitrate(V), $Rb_3CoCl_4NO_3$

Rachid Ouarsal,[a] Mohammed Lachkar,[a] Michael Bolte[b] and Brahim El Bali[a]*

[a]Département de Chimie, Faculté des Sciences Dhar Mehraz, BP 1796 Atlas 30003, Fés, Morocco, and [b]Institut für Organische Chemie, J.-W.-Goethe-Universität, Marie-Curie-Straße 11, D-60439 Frankfurt/Main, Germany

Correspondence e-mail: belbali@fsdmfes.ac.ma

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(O–N) = 0.003 Å
R factor = 0.020
wR factor = 0.043
Data-to-parameter ratio = 27.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The title compound, $Rb_3CoCl_4NO_3$, is isostructural with $K_3ZnCl_4NO_3$. It is built up from Rb^+ cations, NO_3^- anions and $[CoCl_4]^{2-}$ complex ions which form a layer-like arrangement: one layer contains only rubidium cations while the other contains a mixture of rubidium, nitrate and tetrachlorocobaltate ions. Both rubidium cations are ninefold coordinated by three O atoms and six Cl ions. One Rb atom, the Co atom, two Cl atoms, the N atom and one O atom lie on a crystallographic mirror plane.

Comment

A survey of the literature indicated the existence of only a few compounds with the chemical formula $A_3TX_4NO_3$ (A = alkali metal, T = divalent transition metal or Mg and X = halogen), *viz*. $K_3ZnCl_4NO_3$ (Carter & Zompa, 1999) and $Cs_3TI_4NO_3$ (T = Co, Zn & Cd; Louer & Louer, 1986). In the course of our investigations of the H_3PO_4–$RbNO_3$–$CoCl_2$ system, we have isolated single crystals of the title compound, $Rb_3CoCl_4NO_3$, (I), which we describe here.

Compound (I) is isostructural with $K_3ZnCl_4NO_3$, both phases showing the same topology characterized by a double layer arrangement (Fig. 1). The two types of layers alternate parallel to (010): the first type, situated at y = *ca* 0.25, results from edge- and/or corner-sharing of [$Rb1Cl_6O_3$], [$CoCl_4$] and [NO_3] polyhedra; the second layer at y = 0 is built up from edge-sharing [$Rb_2Cl_6O_3$] polyhedra. The two layers are linked by way of Rb—O—Rb and Rb—Cl—Rb bonds (Fig. 2).

Both rubidium cations in (I) are ninefold coordinated by six chloride ions and three O atoms from NO_3 anions. The Rb—Cl distances are in the range 3.2903 (8)–3.5827 (8) Å (average 3.3944 Å) for Rb1 (site symmetry m) and 3.4434 (6)–3.6328 (6) Å for Rb2 (average 3.5036 Å). These average Rb—Cl distances are comparable to those found in $RbCoCl_3$ (3.5772 Å; Engberg & Soling, 1967) and Rb_2CoCl_4 (3.4695 Å; Novikova & Tamazyan, 1998), but longer than the 3.29 Å ionic separation in RbCl (Wang, 1970). The average Rb—O distances in (I) are 2.9336 and 2.9427 Å for Rb1 and Rb2, respectively, which are smaller than the corresponding value of 3.198 Å in $RbNO_3$ (Shamsuzzoha & Lucas, 1987).

In (I) the Co^{2+} cation (site symmetry m) forms a distorted tetrahedron, with a mean Co—Cl distance of 2.2707 Å, comparable to that for Co in a similar coordination in Rb_2CoCl_4 (2.2387 Å). The angular distortion is, however, more pronounced in $[CoCl_4]^{2-}$ anions than it is in $SrZnCl_4$, where Zn possesses the same basic coordination geometry (Wickleder *et al.*, 1999). The [$CoCl_4$] tetrahedra are isolated in the structure of (I); the shortest distance between two neighbouring Co^{2+} ions is more than 5.6 Å.

Experimental

Dark blue crystals of (I) arose as a side-product from a solution containing $RbNO_3$ (1 mmol), $CoCl_2{\cdot}6H_2O$ (1 mmol) and H_3PO_4 (1 mmol) rather than the desired mixed-metal phosphate. The crystals were filtered off and washed with a solution of 80% ethanol.

Crystal data

$Rb_3CoCl_4NO_3$
M_r = 519.15
Orthorhombic, *Pnma*
a = 9.3183 (7) Å
b = 10.0730 (9) Å
c = 12.4423 (9) Å
V = 1167.87 (16) Å^3
Z = 4
D_x = 2.953 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16196 reflections
θ = 3.8–32.3°
μ = 14.78 mm^{-1}
T = 173 (2) K
Block, blue
0.12 × 0.11 × 0.07 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.189, T_{max} = 0.361
18011 measured reflections
1793 independent reflections
1596 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 30.0°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.043
S = 1.02
1793 reflections
65 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0233P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0031 (3)

Table 1
Selected bond lengths (Å).

Rb1—O2^{i}	2.834 (2)	Rb2—Cl2^{v}	3.4434 (6)
Rb1—O1^{ii}	2.9835 (16)	Rb2—Cl2	3.4513 (6)
Rb1—O1	2.9835 (17)	Rb2—Cl3^{iii}	3.4681 (6)
Rb1—Cl1	3.2903 (8)	Rb2—Cl1^{iii}	3.5044 (6)
Rb1—Cl2^{iii}	3.3163 (6)	Rb2—Cl1^{v}	3.5221 (6)
Rb1—Cl2	3.4305 (6)	Rb2—Cl3^{vi}	3.6328 (6)
Rb1—Cl3	3.5827 (8)	Co1—Cl2	2.2583 (6)
Rb2—O1^{iv}	2.8679 (16)	Co1—Cl2^{ii}	2.2583 (6)
Rb2—O2	2.9031 (12)	Co1—Cl3^{vi}	2.2825 (8)
Rb2—O1	3.0572 (17)	Co1—Cl1^{vii}	2.2837 (8)

Symmetry codes: (i) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (ii) $x, \frac{3}{2}-y, z$; (iii) $1-x, 1-y, 1-z$; (iv) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (v) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; (vi) $x-1, y, z$; (vii) $x-\frac{1}{2}, y, \frac{3}{2}-z$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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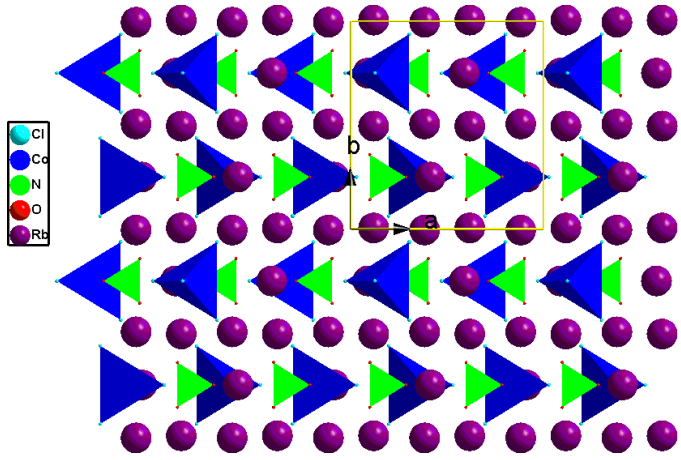

Figure 1
Projection along [001] of the crystal structure of $Rb_3CoCl_4NO_3$. Polyhedron colours: green NO_3 and blue $CoCl_4$.

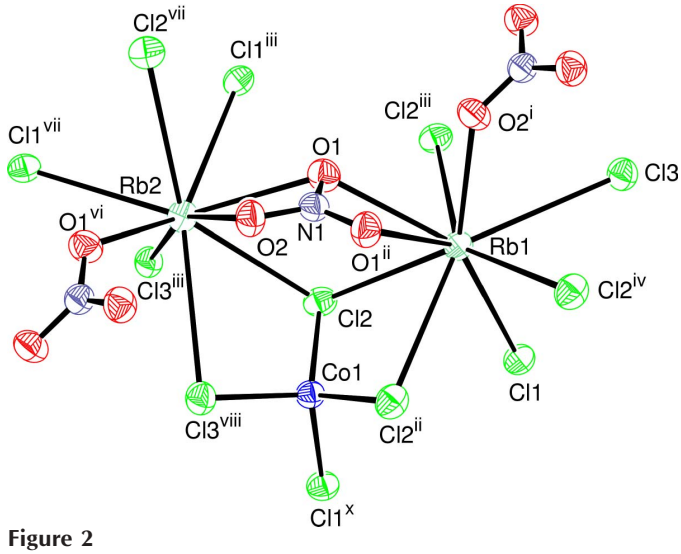

Figure 2
Coordination of Rb and Co in (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (ii) $x, \frac{3}{2}-y, z$; (iii) $1-x, 1-y, 1-z$; (iv) $1-x, \frac{1}{2}+y, 1-z$; (vi) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (vii) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; (viii) $x-1, y, z$; (x) $x-\frac{1}{2}, y, \frac{3}{2}-z$.]

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Brandenburg, K. (1999). *DIAMOND*. Version. 2.1c. Crystal Impact GbR, Bonn, Germany.
Carter, R. L. & Zompa, L. J. (1999). *Acta Cryst.* C**55**, 6–8.
Engberg, A. & Soling, H. (1967). *Acta Chem. Scand.* **21**, 168–174.
Louer, M. & Louer, D. (1986). *J.* Solid State *Chem.* **65**, 272–276.
Novikova, M. S. & Tamazyan, R. A. (1998). *Kristallografiya*, **43**, 210–223.
Shamsuzzoha, M. & Lucas, B. W. (1987). *Acta Cryst.* C**43**, 385–388.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Wang, F. F. Y. (1970). *Acta Cryst.* A**26**, 377–379.
Wickleder, C., Masselmann, S. & Meyer, G. (1999). *Z. Anorg. Allg. Chem.* **625**, 507–510.