Table 2. *Selected geometric parameters (Å, °) for $CuCr_2O_4$*

Cu—O^{i}	1.957 (5) × 4	Cr—O^{ii}	1.999 (4) × 4
Cr—O	1.973 (5) × 2		
O^{i}—Cu—O^{iii}	121.4 (1)	O—Cr—O^{ii}	86.1 (3)
O^{i}—Cu—O^{iv}	103.9 (5)	O—Cr—O^{v}	93.9 (3)
O^{v}—Cr—O^{vi}	82.0 (2)	O^{ii}—Cr—O^{vi}	98.0 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{3}{4}-z$; (iii) $x, y-\frac{1}{2}, 1-z$; (iv) $y-\frac{3}{4}, \frac{1}{4}+x, \frac{3}{4}+z$; (v) $\frac{1}{4}-y, \frac{3}{4}+x, \frac{1}{4}+z$; (vi) $y-\frac{1}{4}, \frac{3}{4}+x, \frac{1}{4}+z$.

Table 3. *Contact distances (Å) for $CuCr_2O_4$*

Cr···Cr^{i}	2.8884 (2)	Cr···Cr^{ii}	3.0170 (2)

Symmetry codes: (i) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{3}{4}-z$; (ii) $x, y-\frac{1}{2}, 1-z$.

$CuRh_2O_4$

Crystal data

$CuRh_2O_4$
$M_r = 333.36$
Tetragonal
$I4_1/amd$
$a = 6.1756$ (5) Å
$c = 7.9006$ (6) Å
$V = 301.31$ (4) Å^3
$Z = 4$
$D_x = 7.35$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
$\theta = 9$–$70°$
$\mu = 96.8$ mm^{-1}
$T = 295$ K
$15 \times 12 \times 0.1$ mm
Black

Data collection

Philips diffractometer
$\theta/2\theta$ scans
Specimen mounted in reflection mode

$2\theta_{\min} = 17°$
$2\theta_{\max} = 140°$
Increment in $2\theta = 0.025°$

Refinement

$R_p = 0.044$
$R_{wp} = 0.060$
$R_{\exp} = 0.028$
$R_B = 0.047$
Excluded region(s): none
Profile function: pseudo-Voigt

20 parameters
$(\Delta/\sigma)_{\max} = 0.10$
Preferred orientation correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 4. *Fractional atomic coordinates and isotropic displacement parameters (Å^2) for $CuRh_2O_4$*

	x	y	z	$U_{\rm iso}$
Cu	0	1/4	7/8	0.016 (2)
Rh	0	1/2	1/2	0.010 (2)
O	0	0.4693 (15)	0.2413 (13)	0.015 (2)

Table 5. *Selected geometric parameters (Å, °) for $CuRh_2O_4$*

Cu—O^{i}	1.962 (8) × 4	Rh—O^{ii}	2.055 (7) × 4
Rh—O	2.053 (8) × 2		
O^{i}—Cu—O^{iii}	124.2 (2)	O—Rh—O^{ii}	87.9 (5)
O^{i}—Cu—O^{iv}	102.7 (8)	O—Rh—O^{v}	92.1 (3)
O^{v}—Rh—O^{vi}	82.5 (3)	O^{ii}—Rh—O^{vi}	97.5 (3)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{3}{4}-z$; (iii) $x, y-\frac{1}{2}, 1-z$; (iv) $y-\frac{3}{4}, \frac{1}{4}+x, \frac{3}{4}+z$; (v) $\frac{1}{4}-y, \frac{3}{4}+x, \frac{1}{4}+z$; (vi) $y-\frac{1}{4}, \frac{3}{4}+x, \frac{1}{4}+z$.

Table 6. *Contact distances (Å) for $CuRh_2O_4$*

Rh···Rh^{i}	2.9442 (3)	Rh···Rh^{ii}	3.0878 (3)

Symmetry codes: (i) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{3}{4}-z$; (ii) $x, y-\frac{1}{2}, 1-z$.

For both compounds, program(s) used to refine structures: *DBWS*-9411 (Young, Sakthivel, Moss & Paiva-Santos, 1994); molecular graphics: *ATOMS* (Dowty, 1989).

Lists of powder diffraction intensity data have been deposited with the IUCr (Reference: BR1169). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Armbruster, T., Lager, G. A., Ihringer, J., Rotella, F. J. & Jorgensen, J. D. (1983). *Z. Kristallogr.* **162**, 8–10.
Bérar, J.-F. & Lelann, P. (1991). *J. Appl. Cryst.* **24**, 1–5.
Bergerhoff, G., Hundt, R., Sievers, R. & Brown, I. D. (1983). *J. Chem. Inf. Comput. Sci.* **23**, 66–69.
Dowty, E. (1989). *ATOMS. A Computer Program for Displaying Atomic Structures.* Shape Software, Kingsport, Tennessee, USA.
Dulac, J. (1969). *Bull. Soc. Fr. Mineral. Cristallogr.* **92**, 25–29.
Ivanov, V. V. & Talanov, V. M. (1995). *Inorg. Mater.* **31**, 107–110.
Morris, M. C., McMurdie, H. F., Evans, E. H., Paretzkin, B., Parker, H. S. & Pyrros, N. P. (1984). *Nat. Bur. Stand. Monogr.* **25**(20), 46.
Powder Diffraction File. International Centre for Diffraction Data, 12 Campus Boulevard, Newtown Square, Pennsylvania 19073-3273, USA.
Prince, E. (1957). *Acta Cryst.* **10**, 554–556.
Young, R. A., Sakthivel, A., Moss, T. S. & Paiva-Santos, C. O. (1994). *User's Guide to Program DBWS*-9411. School of Physics, Georgia Institute of Technology, Atlanta, GA 30332, USA.

Acta Cryst. (1997). **C53**, 659–661

Pentaamminechlorocobalt(III) Hydroxotetranitronitrosylruthenate(III)

VITALY K. BELSKY[a] AND VERA N. KOKUNOVA[b]

[a]*L. Karpov Institute of Physical Chemistry, 10 Vorontsovo pole, 103064 Moscow, Russia, and* [b]*Moscow Academy of Fine Chemical Technology, 86 Prospekt Vernadskogo, 117571 Moscow, Russia. E-mail: belsky@cc.nifhi.ac.ru*

(*Received 25 June 1996; accepted 26 November 1996*)

Abstract

The structure of $[CoCl(NH_3)_5][Ru(NO_2)_4(OH)(NO)]$ consists of octahedral $[CoCl(NH_3)_5]^{2+}$ and $[Ru(NO_2)_4(OH)(NO)]^{2-}$ ions. These units are held together *via* a three-dimensional network of intermolecular O—H···O, N—H···O and N—H···Cl hydrogen bonds.

Comment

The structure analysis of the title compound was undertaken during research into the formation of metallic solid solutions of Co and Ru for microelectronics.

Both structural units are practically undistorted octahedra, with the nitrosyl and hydroxo groups in axial positions in the anion. All metal–ligand bond lengths in the Ru anion correspond to those found in earlier studies (Butman, Khodashova, Minacheva & Tayukin, 1964; Simonsen & Müller, 1965; Kokunova & Stash, 1994; Salomov *et al.*, 1984). The bond distances in the cation are within the limits indicated by Orpen *et al.* (1989).

The ions in the crystal are held together *via* a three-dimensional network of intermolecular hydrogen bonds between ions of the same type and between ions of different types (Table 2).

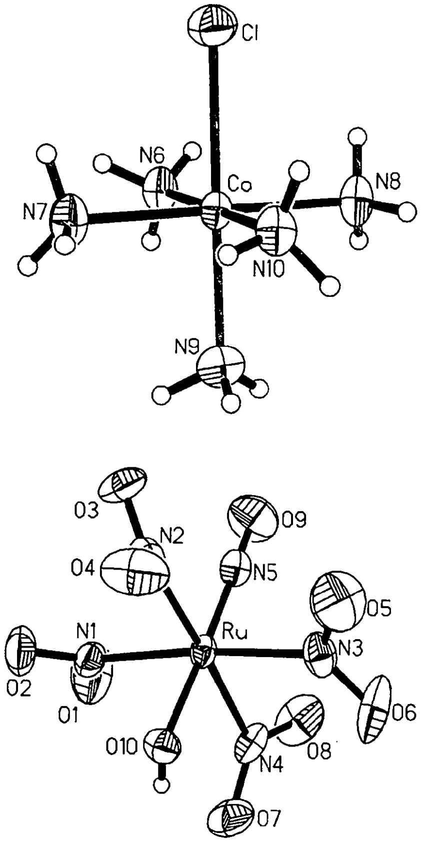


Fig. 1. View of the cation and anion showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level for non-H atoms and H atoms are drawn as small circles of arbitrary radii.

Experimental

The crystals were obtained from an aqueous solution containing $[Co(NH_3)_5]Cl_2$ and $Na_2[Ru(NO_2)_4(OH)(NO)].2H_2O$ in a stoichiometric ratio.

Crystal data

$[CoCl(NH_3)_5]$-
$[Ru(NO_2)_4(OH)(NO)]$
$M_r = 511.68$
Monoclinic
$P2_1/c$
$a = 6.107\,(1)$ Å
$b = 17.653\,(4)$ Å
$c = 14.448\,(3)$ Å
$\beta = 94.80\,(3)°$
$V = 1552.1\,(5)$ Å^3
$Z = 4$
$D_x = 2.190$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 12 reflections
$\theta = 10$–$12.5°$
$\mu = 2.283$ mm^{-1}
$T = 293\,(2)$ K
Rod
$0.44 \times 0.16 \times 0.16$ mm
Crimson

Data collection

Nicolet *P*3 diffractometer
θ–2θ scans
Absorption correction: numerical (*SHELXTL*; Sheldrick, 1981)
$T_{\min} = 0.640$, $T_{\max} = 0.694$
2313 measured reflections
2313 independent reflections

2303 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.06°$
$h = -7 \rightarrow 0$
$k = -20 \rightarrow 0$
$l = -17 \rightarrow 17$
3 standard reflections every 100 reflections intensity decay: 0.4%

Refinement

Refinement on F^2
$R(F) = 0.020$
$wR(F^2) = 0.056$
$S = 1.088$
2303 reflections
273 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 1.6445P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.275$

$\Delta\rho_{\max} = 0.556$ e Å^{-3}
$\Delta\rho_{\min} = -0.362$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0044 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

Ru—N5	1.767 (3)	Co—Cl	2.2668 (10)
Ru—O10	1.935 (2)	O1—N1	1.228 (4)
Ru—N1	2.078 (3)	O2—N1	1.231 (4)
Ru—N2	2.080 (3)	O3—N2	1.260 (4)
Ru—N4	2.083 (3)	O4—N2	1.200 (4)
Ru—N3	2.105 (3)	O5—N3	1.205 (4)
Co—N9	1.939 (3)	O6—N3	1.220 (4)
Co—N8	1.955 (3)	O7—N4	1.229 (4)
Co—N10	1.964 (3)	O8—N4	1.239 (4)
Co—N7	1.967 (3)	O9—N5	1.132 (4)
Co—N6	1.978 (3)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O(10)—H(10)$\cdots$O(5$^{\rm i}$)	0.67	2.54	3.151 (4)	152
N(6)—H(61)$\cdots$O(6$^{\rm ii}$)	0.82	2.55	3.099 (4)	125
N(6)—H(62)$\cdots$O(7$^{\rm iii}$)	0.79	2.37	3.115 (4)	157
N(6)—H(63)$\cdots$O(8$^{\rm iv}$)	0.83	2.22	3.030 (4)	163
N(7)—H(71)$\cdots$O(10$^{\rm v}$)	0.89	2.04	2.908 (4)	165
N(7)—H(72)$\cdots$O(8$^{\rm vi}$)	0.84	2.71	3.063 (4)	167
N(7)—H(73)$\cdots$Cl$^{\rm i}$	0.88	2.88	3.698 (4)	154
N(8)—H(81)$\cdots$O(5$^{\rm ii}$)	0.86	2.53	3.345 (4)	160
N(8)—H(81)$\cdots$O(6$^{\rm ii}$)	0.86	2.52	3.295 (4)	151
N(8)—H(82)$\cdots$O(7$^{\rm iii}$)	0.86	2.46	3.049 (4)	126
N(8)—H(83)$\cdots$O(2$^{\rm vii}$)	0.81	2.28	3.063 (4)	160
N(9)—H(91)$\cdots$Cl$^{\rm i}$	0.88	2.49	3.347 (4)	166
N(9)—H(92)$\cdots$O(4$^{\rm v}$)	0.76	2.25	2.785 (4)	128

N(9)—H(93)···O(6^{ii})	0.92	2.08	2.874 (4)	145
N(10)—H(101)···O(10^{v})	0.82	2.17	2.965 (4)	163
N(10)—H(102)···O(3^{viii})	0.90	2.21	3.066 (4)	160
N(10)—H(103)···O(4^{ix})	0.83	2.40	3.190 (4)	160

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

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

One of us (VKB) greatly appreciates the financial support of the Russian Foundation for Basic Research (grant 95-03-09030).

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BR1156). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Butman, L. A., Khodashova, T. S., Minacheva, L. Kh. & Tayukin, V. I. (1964). *Zh. Strukt. Khim.* **5**, 250–254.
Kokunova, V. N. & Stash, A. I. (1994). *Russ. J. Coord. Chem.* **20**, 815–818.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–S83.
Salomov, A. S., Parpiev, N. A., Sharipov, Kh. T., Kokunova, V. N., Sinitsin, N. M. & Porai-Koshits, M. A. (1984). *Russ. J. Inorg. Chem.* **29**, 2853–2864.
Sheldrick, G. M. (1981). *Nicolet SHELXTL Operations Manual.* Revision 3. Nicolet XRD Corporation, Cupertino, California, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Simonsen, S. H. & Müller, M. H. (1965). *J. Inorg. Nucl. Chem.* **26**, 7–12.

Acta Cryst. (1997). C**53**, 661-663

Hexaamminecobalt(III) Thiosulfate Chloride Monohydrate

ALEXANDER N. SOBOLEV AND BRIAN N. FIGGIS

Department of Chemistry, The University of Western Australia, Nedlands, WA 6907, Australia. E-mail: ans@crystal.uwa.edu.au

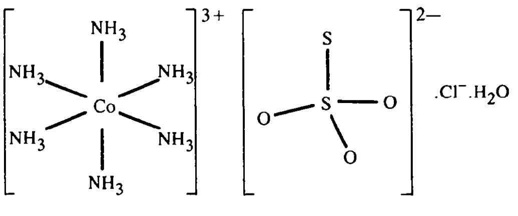
(*Received 17 December 1996; accepted 11 February 1997*)

Abstract

The crystal structure of the title compound, $[Co(NH_3)_6](S_2O_3)Cl.H_2O$, consists of discrete ions and water molecules of hydration. The $[Co(NH_3)_6]^{3+}$ cation in the asymmetric unit is distributed between two different symmetry centres. The water molecule and the ions are linked in the crystal by weak hydrogen bonds and interionic contacts.

Comment

The title structure, (I) (Fig. 1), is a member of the family of hexaammine-M^{III} salts, where M = Ru or Co, synthesized and studied by us using different methods (Engelhardt, Reynolds & Sobolev, 1994; Reynolds, Engelhardt & Sobolev, 1994).



(I)

The $[Co(NH_3)_6]^{3+}$ cation is composed of an octahedrally coordinated Co atom bonded to six ammonia ligands with *cis*-N—Co—N angles in the range 88.1 (2)–91.9 (2)°. The Co—N distances vary from 1.955 (2) to 1.965 (3) Å [average value 1.960 (4) Å] and are in good agreement with those in the literature (Restivo, Ferguson & Balahura, 1977; Reynolds, Engelhardt & Sobolev, 1994). The S—S bond length of 1.982 (1) Å and the average S—O distance of 1.467 (2) Å in the $(S_2O_3)^{2-}$ anion also agree with normal values. The hydrogen-bonding network found in (I) comprises O—H···O, N—H···O, N—H···S and N—H···Cl contacts (Table 3) linking water molecules and ions.

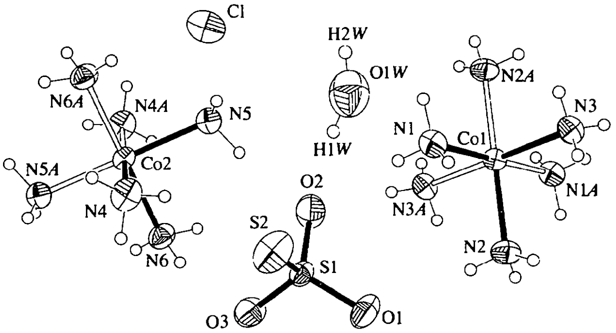


Fig. 1. A general view of the title compound with the atom-numbering scheme and 50% probability ellipsoids. The unshaded bonds correspond to atoms symmetry transformed from those with shaded bonds.

Experimental

Crystals of the title compound were prepared by room-temperature evaporation of a solution of $[Co(NH_3)_6]Cl_3$ to which concentrated $Na_2S_2O_3$ had been added. Crystals (dark-red needles and plates) appeared after about 30 min. One of the needles was cut and prepared for X-ray study.