to the University of British Columbia Computing Centre for assistance.

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Ruthenium Ketoxime Complexes.
II. Structure of the Hydrated Barium Salt of Tris(dihydrogenviolurato)ruthenate(II)

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Abstract

$Ba^{2+}[Ru^{II}(C_4H_2N_3O_4)_3^-]_2.9H_2O$, $C_{24}H_{12}BaN_{18}O_{24}Ru_2.9H_2O$, is triclinic, space group $P\bar{1}$, with $a = 12.81$ (1), $b = 14.12$ (1), $c = 15.61$ (1) Å, $\alpha = 96.5$ (1), $\beta = 99.7$ (1), $\gamma = 122.1$ (1)°, $Z = 2$. The structure was refined to $R = 0.091$ ($R_w = 0.102$). The chelation of the divalent Ru is similar to that in $H_3O^+[Ru^{II}(C_4H_2N_3O_3)_3]^-.3H_2O$. The Ba atom is surrounded by nine O atoms forming a deformed tricapped trigonal prism.

Introduction

The reaction between *trans*-$Na_2[Ru(OH)(NO)(NO_2)_4]$ and barbituric acid ($C_4H_4N_2O_3$) in aqueous solution gives a deep-red complex which has been characterized by IR, ^1H NMR, and magnetic measurements as sodium tris(dihydrogenviolurato)ruthenate(II)

(Brémard, Muller, Nowogrocki & Sueur, 1977). The present salt is isolated in high yield (95%) from reaction mixture by addition of the precipitating cation Ba^{2+}.
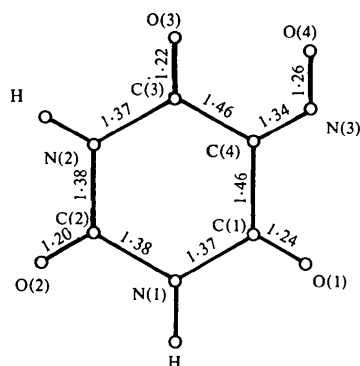

Fig. 1. Schematic structure and bond lengths (Å) of the dihydrogenviolurate ion in the hydrated potassium salt, with numbering scheme.

The dihydrogenviolurate anion, $C_4H_2N_3O_4^-$ (Fig. 1), may be represented as H_2vi.

In the first paper of this series (Abraham, Nowogrocki, Sueur & Brémard, 1978) the structure of the parent complex $H_3O^+[Ru(H_2vi)_3].3H_2O$, which was prepared from the barium salt, has been described. The X-ray analysis of the title compound was undertaken to provide information on the stereochemistry and more direct evidence about the nitrosation mechanism. The present work is part of an effort to explore the mechanism of nitrosation of organic substrates via nitrosyl complexes (Brémard, Nowogrocki & Sueur, 1979).

Experimental

$Ba[Ru(H_2vi)_3]_2.9H_2O$ was obtained as described previously (Brémard et al., 1977). When a saturated deep-red solution was cooled to 278 K the hydrated barium salt crystallized as black crystals. The crystals were separated from the solution by filtration, washed with cold water and air-dried. Elemental analyses of the crystals were undertaken by the Microanalytical Laboratory of CNRS, Villeurbanne, France. Ruthenium analysis was performed by a method described previously (Abraham et al., 1978).

|	Ru	C	N	H	Ba
Experimental (%)	13·9	20·0	17·4	2·4	9·9
Calculated (%)	14·06	20·02	17·54	2·09	9·55

A thermogravimetric analysis of the crystals was performed to check the water content. A weight loss of 11·5% in the temperature range 320–400 K was observed, which corresponded to a loss of about nine water molecules, so that the correct formula is $Ba[Ru(H_2vi)_3]_2.9H_2O$, confirmed by the structure determination. Crystals of the title compound are triclinic with $a = 12·81$ (1), $b = 14·12$ (1), $c = 15·61$ (1) Å, $\alpha = 96·5$ (1), $\beta = 99·7$ (1), $\gamma = 122·1$ (1)°, $V = 2287$ Å³, $Z = 2$, $d_c = 2·07$, and $d_m = 2·07$ (1) Mg m^{-3} (flotation method); the space group is $P\bar{1}$. The alternative space group $P1$ could be eliminated on the basis of the Patterson synthesis. The intensities of 6976 independent reflections were collected on a Philips PW 1100 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation; 6102 were classified as observed with $I > 2\sigma(I)$. Cell dimensions were determined by least-squares refinement from the 2θ, χ, φ angles of 25 reflections. Data were corrected for Lorentz and polarization effects and for absorption [μ(Mo $K\alpha$) = 1·7 mm^{-1}] (de Meulenaer & Tompa, 1965). The crystal used for data collection was bounded by the planes {001}, {00$\bar{1}$}, {1$\bar{1}$0}, {$\bar{1}$11}, {1$\bar{1}\bar{1}$}, {$\bar{1}$10}, {10$\bar{2}$}, {100}, {010}, {$\bar{1}$02}, {0$\bar{1}$2}, {0$\bar{1}$0}, and {$\bar{1}$00}, with perpendicular distances from an arbitrary point inside the crystal of 0·121, 0·086,

0·076, 0·136, 0·027, 0·140, 0·078, 0·195, 0·259, 0·115, 0·109, 0·113 and 0·160 mm respectively. The transmission factor ranged from 0·70 to 0·82.

A Patterson synthesis could be interpreted in terms of space group $P\bar{1}$ and yielded the positions of the Ba and Ru atoms. Least-squares refinement reduced R to 0·36. Subsequent difference syntheses and refinement located the remaining non-hydrogen atoms and revealed nine water O atoms. The structure was refined isotropically by full-matrix least squares (Prewitt, 1966). The last difference map indicated anisotropy around metallic atoms; accordingly refinement was continued with anisotropic temperature factors for Ba and Ru to give the final $R = 0·091$ ($R_w = 0·102$). The anisotropic refinement of all atoms was not possible in full-matrix mode because of the capacity of the computer used (CIIHB 10070), and a block-diagonal procedure does not lead to satisfactory convergence. So, H atoms were not included in the structure factor calculations and no attempt was made to locate them in the Fourier maps. Scattering factors were taken from Cromer & Waber (1965) and the anomalous-dispersion corrections from Cromer & Liberman (1970). The final atomic parameters and isotropic temperature factors are listed in Table 1.*

Description of the structure and discussion

The crystal structure consists of $[Ru(H_2vi)_3]^-$ anions, Ba^{2+} cations and water molecules (Fig. 2). Superficially, the two crystallographically independent $[Ru(H_2vi)_3]^-$ anions are very similar and are also very close to the corresponding anion of $H_3O^+[Ru(H_2vi)_3].3H_2O$, represented in Fig. 1 of the preceding paper (Abraham et al., 1978). The molecular structure of the two independent $[Ru(H_2vi)_3]^-$ moieties consists of a tris-chelate array around the Ru atoms, each of which has a distorted octahedral coordination geometry. Each H_2vi ligand is bidentate through O(1) and N(3), forming with C(1), C(4) and Ru a five-membered chelate ring. The octahedral coordination of the three H_2vi ligands to the Ru atom has facial (fac) geometry, as in related compounds which contain this chelating function (Abraham et al., 1978; Raston & White, 1976):

Table 1. *Atomic parameters*
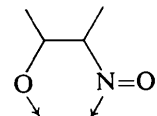
Water O atoms are labelled W. Positional parameters are fractional coordinates ($\times 10^4$). E.s.d.'s for positional and isotropic thermal parameters are given in parentheses and refer to the last significant digit.

	x	y	z	B (Å²)		x	y	z	B (Å²)
Ba	4482 (1)	1717 (1)	1461 (1)	2·9	Ru(2)	54 (1)	2341 (1)	3718 (1)	1·8
Ru(1)	3511 (1)	7377 (1)	1962 (1)	1·7					
Ligand A(1)					**Ligand A(2)**				
O(1)	3098 (8)	5676 (8)	1787 (7)	2·1 (2)	O(1)	−1069 (9)	2934 (8)	3176 (7)	2·7 (2)
C(1)	4037 (13)	5714 (11)	1622 (10)	2·1 (2)	C(1)	−433 (13)	4004 (12)	3456 (10)	2·2 (3)
N(1)	3971 (11)	4694 (9)	1465 (8)	2·1 (2)	N(1)	−885 (12)	4610 (11)	3123 (9)	2·8 (2)
C(2)	4904 (14)	4648 (12)	1213 (11)	2·5 (3)	C(2)	−221 (15)	5807 (13)	3378 (12)	3·0 (3)
O(2)	4773 (11)	3685 (10)	1027 (8)	3·6 (2)	O(2)	−623 (10)	6313 (9)	3018 (8)	3·4 (2)
N(2)	5941 (12)	5606 (11)	1118 (9)	3·2 (3)	N(2)	896 (13)	6349 (11)	4045 (10)	3·2 (3)
C(3)	6164 (14)	6719 (12)	1336 (11)	2·5 (3)	C(3)	1410 (15)	5836 (14)	4483 (12)	3·0 (3)
O(3)	7187 (11)	7538 (9)	1278 (8)	3·5 (2)	O(3)	2413 (12)	6443 (11)	5093 (9)	4·4 (3)
C(4)	5151 (13)	6715 (11)	1560 (10)	2·0 (2)	C(4)	716 (14)	4594 (12)	4162 (11)	2·4 (3)
N(3)	5103 (10)	7691 (9)	1756 (8)	1·8 (2)	N(3)	1075 (11)	3922 (10)	4445 (8)	2·3 (2)
O(4)	6088 (10)	8641 (9)	1759 (7)	2·9 (2)	O(4)	1985 (11)	4316 (10)	5134 (8)	3·9 (2)
Ligand B(1)					**Ligand B(2)**				
O(1)	4355 (8)	7824 (8)	3345 (7)	2·1 (2)	O(1)	−1190 (9)	747 (8)	2754 (7)	2·2 (2)
C(1)	4971 (13)	8917 (12)	3628 (10)	2·1 (2)	C(1)	−844 (12)	868 (11)	2035 (10)	1·9 (2)
N(1)	5712 (11)	9425 (10)	4491 (9)	2·5 (2)	N(1)	−1619 (11)	−29 (10)	1281 (8)	2·2 (2)
C(2)	6419 (13)	10602 (11)	4820 (10)	2·0 (2)	C(2)	−1390 (13)	41 (12)	470 (10)	2·1 (2)
O(2)	7076 (9)	11028 (8)	5610 (7)	2·8 (2)	O(2)	−2157 (10)	−708 (9)	−222 (7)	2·0 (2)
N(2)	6351 (11)	11237 (10)	4277 (9)	2·5 (2)	N(2)	−281 (11)	998 (10)	411 (9)	2·4 (2)
C(3)	5629 (13)	10834 (11)	3385 (10)	2·0 (2)	C(3)	647 (13)	1932 (11)	1165 (10)	2·0 (2)
O(3)	5710 (11)	11559 (10)	2961 (8)	3·6 (2)	O(3)	1620 (10)	2690 (9)	1017 (8)	3·1 (2)
C(4)	4917 (13)	9616 (12)	3056 (10)	2·1 (2)	C(4)	258 (13)	1840 (11)	1956 (10)	1·9 (2)
N(3)	4153 (11)	9013 (9)	2205 (8)	2·1 (2)	N(3)	910 (11)	2736 (10)	2759 (8)	2·3 (2)
O(4)	3955 (10)	9511 (9)	1642 (7)	2·9 (2)	O(4)	1907 (11)	3657 (9)	2766 (8)	3·5 (2)
Ligand C(1)					**Ligand C(2)**				
O(1)	1693 (9)	6844 (8)	2077 (7)	2·3 (2)	O(1)	−887 (9)	1687 (8)	4706 (7)	2·6 (2)
C(1)	961 (12)	6585 (11)	1316 (10)	1·9 (2)	C(1)	−234 (13)	1459 (11)	5233 (10)	2·1 (2)
N(1)	−306 (11)	6156 (10)	1241 (8)	2·3 (2)	N(1)	−680 (11)	1021 (10)	5932 (9)	2·6 (2)
C(2)	−1181 (12)	5827 (11)	428 (10)	1·8 (2)	C(2)	18 (14)	805 (13)	6562 (11)	2·8 (3)
O(2)	−2295 (10)	5445 (9)	383 (8)	3·1 (2)	O(2)	−423 (11)	406 (10)	7179 (8)	3·8 (2)
N(2)	−724 (11)	5941 (10)	−303 (9)	2·6 (2)	N(2)	1134 (12)	996 (11)	6461 (9)	2·9 (2)
C(3)	537 (13)	6413 (12)	−316 (10)	2·1 (2)	C(3)	1665 (14)	1385 (12)	5749 (11)	2·4 (3)
O(3)	822 (11)	6527 (10)	−1027 (9)	4·0 (2)	O(3)	2646 (12)	1465 (10)	5720 (9)	4·2 (2)
C(4)	1387 (12)	6707 (11)	536 (10)	1·8 (2)	C(4)	903 (13)	1599 (12)	5124 (10)	2·3 (3)
N(3)	2639 (10)	7080 (9)	716 (8)	1·6 (2)	N(3)	1199 (11)	1979 (10)	4351 (9)	2·5 (2)
O(4)	3157 (10)	7216 (9)	82 (8)	3·3 (2)	O(4)	2150 (11)	2116 (10)	4150 (8)	3·8 (2)
W(1)	4355 (13)	558 (12)	−166 (10)	5·0 (3)					
W(2)	9611 (11)	8342 (10)	1069 (8)	3·9 (2)					
W(3)	4339 (15)	2103 (13)	4521 (11)	6·2 (3)					
W(4)	6928 (28)	1804 (25)	1468 (20)	14·1 (8)					
W(5)	7053 (31)	3694 (27)	1644 (22)	15·6 (9)					
					W(6)	2916 (24)	1168 (22)	2535 (18)	11·8 (7)
					W(7)	5902 (27)	3628 (24)	3045 (20)	13·6 (8)
					W(8)	6325 (24)	5716 (21)	3322 (17)	11·3 (7)
					W(9)	4290 (29)	5793 (26)	3909 (21)	14·5 (9)

The causes of the predominance of the facial geometry in the solid state or in solution (Epps, Wiener, Stewart & Marzilli, 1977; Brémard *et al.*, 1979) are not known. It thus appears that some intrinsic stability is associated with the facial geometry in all octahedral systems thus far investigated. One might speculate that the $^\frown$N=O group might have a large *trans* effect and the mutually *trans* $^\frown$N=O group might also have a large *trans* effect and thus the mutually *trans* $^\frown$N=O groups in the meridional (*mer*) isomer would be unstable.

The deviations of the interatomic distances and angles from those of the free ligand H_2vi (Guillier, 1965) (Fig. 1) are caused by the coordination to the metallic atom. No significant differences are found for the two independent *fac* [Ru(H$_2$vi)$_3$]$^-$ moieties of the present crystal and the corresponding anion of H_3O^+-[Ru(H$_2$vi)$_3$].3H$_2$O. The interatomic distances are very similar for the six violurato ligands (Fig. 3) and close to those found in previous work on these ligands (Hamelin, 1972; Raston & White, 1976). The mean Ru—N(3) and Ru—O(1) values are 1·95 and 2·11 Å
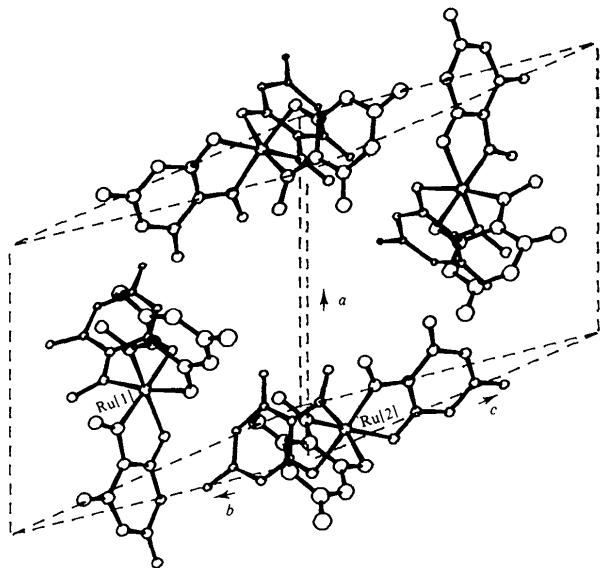
Fig. 2. View of the molecular packing in the unit cell of Ba[Ru(H$_2$vi)$_3$]$_2$.9H$_2$O omitting the H atoms. Ba and W atoms are excluded for clarity.

Table 2. *Mean-plane calculations for chelate rings and deviations* (Å) *of atoms from least-squares planes*

The mean-plane equations are calculated from O(1), C(1), C(4), and N(3) for each ring. E.s.d.'s in the distances are 0·010–0·019 Å for non-metallic and 0·001 Å for Ru atoms.

	O(1)	C(1)	C(4)	N(3)	O(4)	Ru
A(1)	0·008	−0·014	0·013	−0·007	−0·050	0·072
B(1)	0·002	−0·003	0·003	−0·001	−0·078	0·212
C(1)	−0·007	0·012	−0·011	0·006	0·009	0·014
A(2)	−0·009	0·017	−0·016	0·009	−0·178	0·313
B(2)	0·011	−0·020	0·022	0·011	0·008	−0·129
C(2)	−0·005	0·009	−0·009	0·004	0·025	−0·128

Interplanar angles (°) (e.s.d.'s ∼1°)

A(1)–B(1)	96·3	A(1)–C(1)	89·8	B(1)–C(1)	79·6
A(2)–B(2)	76·7	A(2)–C(2)	81·3	B(2)–C(2)	75·2

Equations of planes (X, Y, Z are coordinates in Å relative to an orthonormal basis)

$$A(1) \quad -0·2204X + 0·0725Y - 0·9727Z = 1·9934$$
$$B(1) \quad 0·9448X + 0·1752Y - 0·2770Z = 1·0637$$
$$C(1) \quad 0·3221X - 0·9353Y - 0·1464Z = 8·5272$$
$$A(2) \quad 0·6618X + 0·2997Y - 0·6872Z = 5·4744$$
$$B(2) \quad 0·8499X - 0·4390Y + 0·2916Z = 1·1535$$
$$C(2) \quad 0·0508X - 0·8413Y - 0·5382Z = 4·1965$$

reported in Table 2; Ru atoms are out of the mean planes of the rest of the atoms by at most 0·313 Å. The heterocyclic rings and the chelate rings are not rigorously coplanar; the most significant angle between the mean planes is 5–6° in the B(2) violurato ligand. The heterocyclic rings are almost planar. Around each water O atom there are O or N atoms of the violurato ligands at distances <3 Å. All the water molecules participate in their maximum number of hydrogen bonds. However, because H atoms could not be located an unequivocal assignment of most of the donors and acceptors involving the water molecules cannot be performed and an extensive network of hydrogen bonds cannot be proposed.

The coordination polyhedron around the Ba atom comprises nine O atoms, forming a deformed tricapped trigonal prism (Fig. 4). Five of these are water
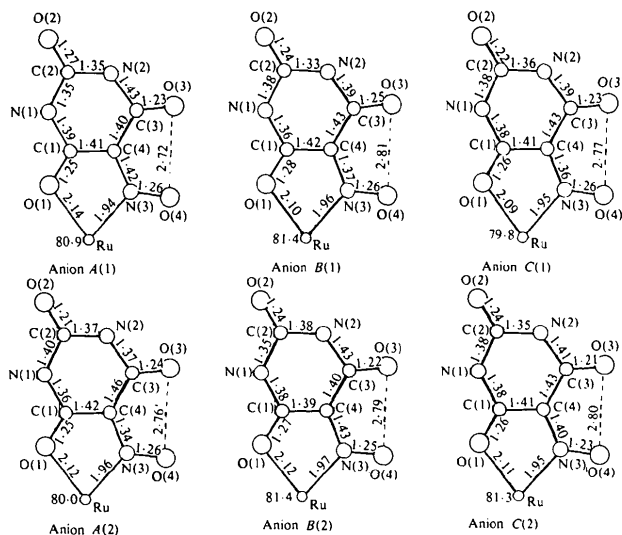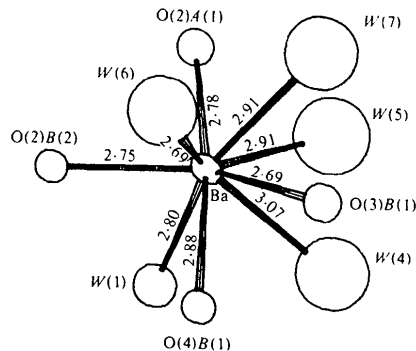

Fig. 3. Interatomic distances (Å) in the heterocyclic and chelate rings. The average e.s.d. is 0·01 Å on the distances and 0·1° on the angles.

respectively. In addition, the various non-bonded repulsions between the atoms in the crystal cause no significant differences in the O(1)–Ru–N(3) angles (Fig. 3). More significant differences appear in the chelate-ring conformations: although the configuration of the *fac* [Ru(H$_2$vi)$_3$]$^-$ moiety is $\Delta(\lambda\lambda\lambda)$ and $\Lambda(\delta\delta\delta)$ in H$_3$O$^+$[Ru(H$_2$vi)$_3$].3H$_2$O, the configurations of the two independent ones in the present salt are $\Delta(\delta\delta\lambda)$ and $\Lambda(\lambda\lambda\delta)$ because of the centrosymmetry of the space group. The mean planes of the chelate rings are

Fig. 4. The Ba coordination in Ba[Ru(H$_2$vi)$_3$]$_2$.9H$_2$O with the bond lengths (Å). The average e.s.d. is 0·01 Å.

O atoms, three belong to the same *fac* [Ru(1)(H$_2$vi)$_3$]$^-$ anion and the last to another independent [Ru(2)-(H$_2$vi)$_3$]$^-$ anion.

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The Structure of Antiferroelectric Tetramethylammonium Trichlorogermanate(II) at Room Temperature

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Abstract

Below 424 K [N(CH$_3$)$_4$][GeCl$_3$], C$_4$H$_{12}$N$^+$·Cl$_3$Ge$^-$, crystallizes in a strongly distorted perovskite structure, space group *Pna*2$_1$, $Z = 4$, with $a = 13 \cdot 069$ (10), $b = 8 \cdot 895$ (5), $c = 9 \cdot 115$ (5) Å. Refinement led to an R of 0·039 for 818 independent reflexions. The structure is characterized by the tendency of bivalent Ge to form discrete GeCl$_3$ groups with pseudo-trigonal-pyramidal geometry. At 424 K, [N(CH$_3$)$_4$][GeCl$_3$] undergoes a structural phase transition to an ionic conducting cubic phase with $a = 6 \cdot 552$ (2) Å. For this phase a model which demands dynamical disorder is proposed.

Introduction

[N(CH$_3$)$_4$][GeCl$_3$] shows unusual physical properties. At 424 K the colourless crystals undergo a phase transition to a cubic high-temperature modification with $a = 6 \cdot 552$ (2) Å and space group *Pm*3*m* (Möller & Felsche, 1979). At room temperature the electrical conductivity is of the order of 10^{-9} Ω^{-1} mm^{-1}. This value increases on approaching the transition point by several orders of magnitude to a value of about 10^{-3} Ω^{-1} mm^{-1}. A.c. conductivity, d.c. polarization and NMR investigations (Möller, 1978) confirm that the high conductivity in conjunction with a low value of E_A is due to ionic motion. The present structure determination should facilitate a better understanding of the unusual physical properties.

Experimental

Crystals of [N(CH$_3$)$_4$][GeCl$_3$], suitable for X-ray investigations and also for conductivity measurements, were grown by a convection method from an equimolar solution of [N(CH$_3$)$_4$]Cl and GeCl$_2$ in 5 *M* HCl. Preliminary Weissenberg precession photographs revealed the perovskite-type structure and the systematic extinctions (0*kl*: $k + l = 2n + 1$; *h0l*: $h = 2n + 1$) indicated the possible space groups *Pnam* and *Pna*2$_1$. A cube-shaped crystal 0·4 × 0·4 × 0·4 mm was used on a Syntex *P*2$_1$ autodiffractometer with Mo *K*α$_1$ radiation to determine the crystal data and to measure the intensities. The lattice constants were obtained by

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