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Structure of Potassium (2,2′-Bipyridine)tetrachlororuthenate(III)

BY B. DURHAM, D. I. COX, A. W. CORDES AND S. BARSOUM

Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR 72701, *USA*

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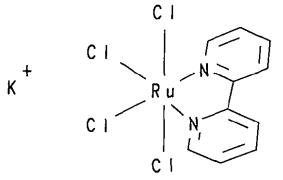
Abstract. $K[RuCl_4(C_{10}H_8N_2)]$, $M_r = 438{\cdot}2$, orthorhombic, *Pbcn*, $a = 14{\cdot}382\ (7)$, $b = 13{\cdot}980\ (4)$, $c = 7{\cdot}491\ (3)$ Å, $V = 1506\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}93$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 20{\cdot}0$ cm^{-1}, $F(000) = 852$, $T = 293$ K, $R = 0{\cdot}051$ for 686 reflections with $F_o^2 > 3\sigma(F_o^2)$. The $[Ru(bpy)Cl_4]^-$ anion lies on a crystallographic twofold axis. The Ru—Cl distances are 2·376 (3) Å for the Cl perpendicular to the bpy plane and 2·414 (3) Å for the Cl atom in the bpy plane. The axial Cl—Ru—Cl bond angle is 175·8 (1)° with the Cl atoms bending toward the bpy unit. The organic rings of neighboring anions overlap with spacings between planes of 3·27 and 3·36 Å.

Experimental. Title compound (I) obtained by the method given by James & McMillan (1975). Red crystals obtained by slow cooling of a 6 *M* solution.



(I)

Data crystal 0·08 × 0·08 × 0·78 mm (needle axis along *c*) mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 17 reflections with $16 < 2\theta < 20°$. Analytical absorption correction based on crystal-face measurements varied from 0·76 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $0 \le h \le 17$, $-16 \le k \le 0$, $0 \le l \le 8$. Three standard reflections (152;224;312) indicated crystal decomposition of 2·0% over 12·1 h of data collection; a simple correction was made. 1556 reflections measured, 1325 unique, 639 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma^2_{CS}(I) + (0{\cdot}06I)^2$; $\sigma_{CS}(I)$ is standard deviation of I based on counting statistics. No extinction correction was made. Solved by Patterson and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms were constrained to idealized posi-

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

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

	x	*y*	*z*	B_{eq}(Å^2)
Ru	0·000	0·23232 (8)	0·250	2·55 (2)
K	0·500	0·4209 (3)	0·250	5·3 (1)
Cl(1)	0·0868 (2)	0·2386 (2)	0·5196 (4)	3·00 (6)
Cl(2)	0·0995 (2)	0·1087 (2)	0·1308 (5)	3·59 (7)
N	0·0779 (6)	0·3443 (6)	0·168 (1)	2·4 (2)
C(1)	0·0442 (8)	0·4322 (7)	0·202 (1)	2·2 (2)
C(2)	0·0915 (8)	0·5142 (8)	0·155 (2)	3·5 (3)
C(3)	0·1776 (9)	0·5069 (9)	0·068 (2)	4·1 (3)
C(4)	0·2135 (9)	0·4180 (9)	0·032 (2)	4·3 (3)
C(5)	0·1627 (9)	0·3380 (8)	0·083 (2)	3·7 (3)

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ru	Cl(1)	2·376 (3)	C(1)	C(1′)	1·46 (2)	
Ru	Cl(2)	2·414 (3)	C(1)	C(2)	1·380 (13)	
Ru	N	2·021 (7)	C(2)	C(3)	1·402 (15)	
N	C(1)	1·347 (11)	C(3)	C(4)	1·37 (2)	
N	C(5)	1·377 (14)	C(4)	C(5)	1·389 (15)	

Cl(1)	Ru	Cl(1′)	175·8 (1)	C(1)	N	C(5)	117·8 (8)
Cl(1)	Ru	Cl(2)	91·7 (1)	N	C(1)	C(1′)	114·1 (5)
Cl(1′)	Ru	Cl(2)	91·3 (1)	N	C(1)	C(2)	122·0 (9)
Cl(2)	Ru	Cl(2′)	88·6 (1)	Cl(1′)	C(1)	C(2)	123·8 (6)
Cl(1)	Ru	N	86·5 (2)	C(1)	C(2)	C(3)	120· (1)
Cl(1′)	Ru	N	90·2 (2)	C(2)	C(3)	C(4)	119· (1)
Cl(2)	Ru	N	96·5 (2)	C(3)	C(4)	C(5)	119· (1)
Cl(2′)	Ru	N	174·5 (2)	N	C(5)	C(4)	123· (1)
N	Ru	N′	78·5 (5)				

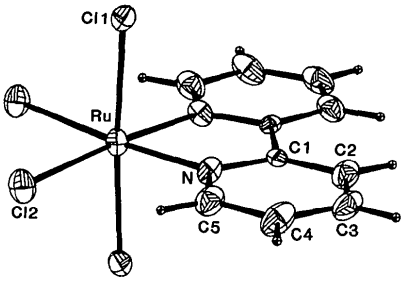


Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme of the anion. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

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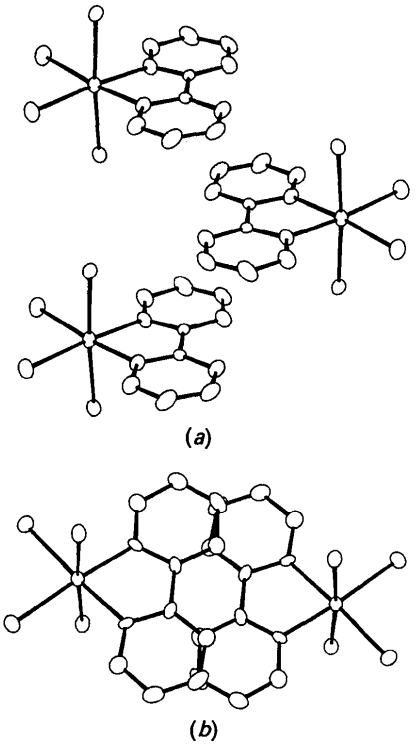

Fig. 2. The anion stacking as viewed (*a*) normal to the c direction and (*b*) parallel to the c direction.

tions (C—H = 0·95 Å) with fixed isotropic B values of 1·2 times the B value of the attached C atoms. All non-H atoms were refined anisotropically for a total of 83 parameters. $R = 0{\cdot}051$, $wR = 0{\cdot}062$, GOF = 1·3, for reflections with $I > 3\sigma(I)$ where non-Poisson $w^{-1} = [\sigma^2(I) + (0{\cdot}06I)^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0{\cdot}01$, $\Delta\rho_{max} = 1{\cdot}4\,(1)$ (two peaks within 1 Å of the Ru atom) and $\Delta\rho_{min} = -0{\cdot}7\,(1)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the anion with the numbering scheme, and Fig. 2 shows the overlap of the anions parallel to the c axis of the crystal.

Related literature. The utility of this anion has been extensively studied by Dwyer, Goodwin & Gyarfas (1963). Structural aspects of ruthenium complexes with bipyridine ligands have been discussed by Durham, Wilson, Hodgson & Meyer (1980).

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* Tables of H-atom coordinates, anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52239 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $(H_4cyclam)^{4+}.ReCl_6^{2-}.2Cl^-.4(CH_3)_2SO$

BY ALEXANDER J. BLAKE,* JOHN A. GREIG AND MARTIN SCHRÖDER

Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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Abstract. 1,4,8,11-Tetraazoniacyclotetradecanehexachlororhenate(IV) dichloride–dimethyl sulfoxide (1/4), $[C_{10}H_{28}N_4][Cl_6Re]Cl_2.4C_2H_6OS$, $M_r = 986{\cdot}7$, triclinic, $P\bar{1}$, $a = 9{\cdot}6478\,(9)$, $b = 9{\cdot}9716\,(12)$, $c = 10{\cdot}9887\,(9)$ Å, $\alpha = 66{\cdot}470\,(7)$, $\beta = 89{\cdot}033\,(4)$, $\gamma = 84{\cdot}813\,(4)°$, $V = 965{\cdot}1$ Å^3, $Z = 1$, $D_x = 1{\cdot}697$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 3{\cdot}987$ mm^{-1}, $F(000) = 495$, $T = 295$ K, $R = 0{\cdot}0228$ for 2480 unique observed reflections. The quadruply protonated cyclam cation and the $ReCl_6^{2-}$ anion lie

* Author for correspondence.

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