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Potassium *trans*-diaquabis[oxalato(2−)-κ^2O,O']chromate(III)

Michel Bélombé,[a] Justin Nenwa,[a] Boniface P. T. Fokwa[b] and Richard Dronskowski[b]*

[a]Department of Inorganic Chemistry, University of Yaounde I, POB 812 Yaounde, Cameroon, and [b]Institut für Anorganische Chemie, RWTH Aachen, D-52056 Aachen, Germany

Correspondence e-mail: belombe2000@yahoo.fr

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The title compound, $K[Cr(C_2O_4)_2(H_2O)_2]$, was crystallized from an aqueous solution. It is the first member without uncoordinated water molecules of formula $A_m[M(C_2O_4)_2(H_2O)_2]\cdot xH_2O$, where A = monovalent cation, $m = 1$ or 2, M = metal(II) or metal(III), $x \geq 0$. The crystal packing is governed by electrostatic and coordinative interactions, and intermolecular hydrogen bonds help to stabilize the packing. The coordination of the Cr sites is quasi-octahedral, and the K ions are eight-coordinated by O atoms of oxalate ligands, forming strongly distorted square antiprisms.

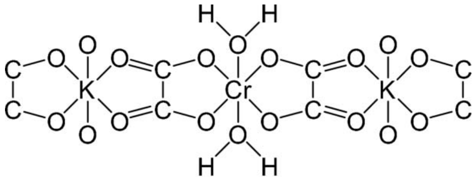
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.137
Data-to-parameter ratio = 13.8

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

Comment

The attempt to prepare the elusive tris(oxamide dioxime)chromium(III) tris(oxalato)chromate(III), $[Cr(H_2oxado)_3][Cr(C_2O_4)_3]\cdot nH_2O$ (H_2oxado is oxamide dioxime), similar to $[Co(H_2oxado)_3][Cr(C_2O_4)_3]\cdot 5H_2O$ (Nenwa, 2004; Bélombé *et al.*, 2006), led to isolation of compound (I), which appears to be the first member without uncoordinated water molecules of transition metal complex salts of general formula $A_m[M(C_2O_4)_2(H_2O)_2]\cdot xH_2O$, where A = monovalent cation, M = metal(II) or metal(III), m = 1 or 2, $x \geq 0$, characterized earlier (Román *et al.*, 1993; Grey *et al.*, 1985; van Niekerk & Schoening, 1951).



(I)

It seems relevant to compare the structural features of (I) with those of the hydrated homologues, particularly with those of $K[Cr(C_2O_4)_2(H_2O)_2]\cdot 3H_2O$, (II) (van Niekerk & Schoening, 1951). The structural feature of focal interest in this series obviously is the complex anion, $[M(C_2O_4)_2(H_2O)_2]^{n-}$ (n = 1 or 2), typified by Fig. 1. It shows the only slightly distorted (4 + 2)-octahedral coordination of the central metal (M) by four equatorial O atoms (O1) of two coplanar oxalate and two axial O atoms (O3) of water ligands. In $[V(C_2O_4)_2(H_2O)_2]^-$ (Grey *et al.*, 1985), the (4 + 2)-octahedron experiences an oblate distortion. In (II) and in $K_2[Ni(C_2O_4)_2(H_2O)_2]\cdot 0.25H_2O$ (Román *et al.*, 1993), a prolate distortion is realized. In (I), the six Cr–O bond lengths (Table 1) are comparable within experimental error. Thus, they produce almost perfect octahedral coordination around the Cr atom. The coordination geometry around the K sites is shown in Fig. 2. It is seen that

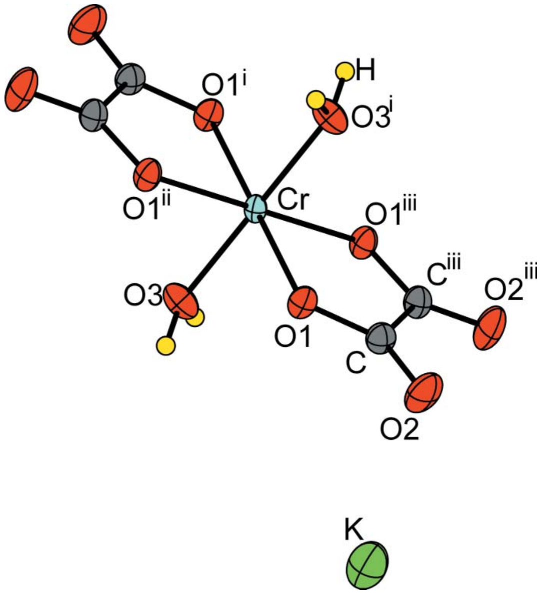


Figure 1
The structure of the constituent ions of (I), showing the atom numbering. Displacement ellipsoids are drawn at the 70% probability level. Symmetry codes as in Table 1.

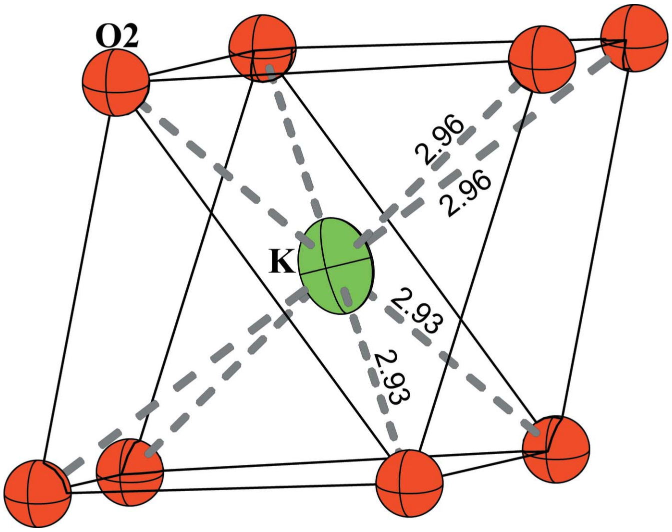


Figure 2
The strongly distorted square antiprismatic coordination around K sites.

each K^+ ion is surrounded by eight terminal oxalate O atoms of neighboring complex anions, yielding strongly distorted square antiprisms. Structural differences between (I) and (II) ought to be linked to the sole fact that water of crystallization is absent in the former, but is present in the latter material. In (II), the crystal packing is rationalized in terms of electrostatic, coordinative and intermolecular H-bonding, the latter bonding being induced by water of crystallization. In (I), however, intermolecular hydrogen bonds, depicted in Fig. 3, link neighboring complex anions into layers parallel to (002) crystallographic planes, with an intermolecular separation O1$\cdots$O3 of 2.637 Å.

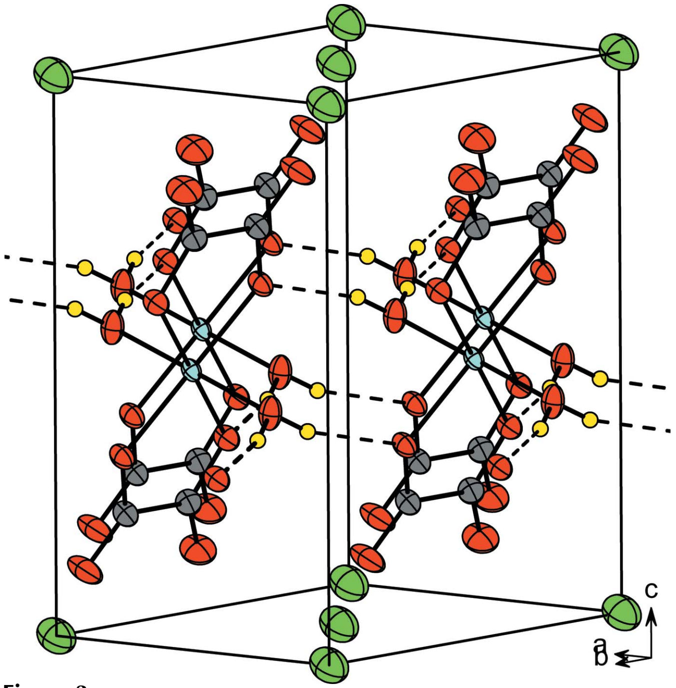


Figure 3
A projection of the crystal structure of (I), showing the intermolecular hydrogen bonds (dashed lines).

Experimental

Compound (I) was obtained from a mixture of $K_3[Cr(C_2O_4)_3]\cdot 3H_2O$ (Bailar & Jones, 1939; 98 mg, 2 mmol), $CrCl_3\cdot 6H_2O$ (540 mg, 2 mmol; Riedel-de Haën, 97%) and freshly prepared oxamide dioxime (Ephraim, 1889; 720 mg, 6 mmol) in H_2O (100 ml) at 333 K. The filtered solution was adjusted to pH $\simeq$ 1 with concentrated HNO_3, then evaporated at ambient temperature over *ca* 4 months. The crystals were separated by filtration, washed with H_2O (5 ml) and dried in air (yield 410 mg).

Crystal data

$K[Cr(C_2O_4)_2(H_2O)_2]$
$M_r = 303.17$
Monoclinic, $C2/m$
$a = 6.6012\ (10)$ Å
$b = 7.2585\ (11)$ Å
$c = 9.8539\ (16)$ Å
$\beta = 92.228\ (3)^\circ$
$V = 471.79\ (13)$ Å^3
$Z = 2$
$D_x = 2.134$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.70$ mm^{-1}
$T = 298$ K
Needle, pink
0.32 × 0.10 × 0.08 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.772$, $T_{\max} = 0.873$
3283 measured reflections
636 independent reflections
625 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.064$
$\theta_{\max} = 28.3^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.137$
$S = 1.15$
636 reflections
46 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 1.7264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -1.51$ e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

Cr—O1	1.9575 (19)	O1—C	1.302 (3)
Cr—O3	1.976 (3)	O2—C	1.200 (4)
K—O2	2.929 (3)	C—C^{ii}	1.561 (6)
K—O2^{i}	2.956 (3)		
O1—Cr—O1^{iii}	180	O2—K—O2^{vii}	67.07 (7)
O1—Cr—O1^{iv}	97.13 (12)	O2^{viii}—K—O2^{vii}	70.64 (4)
O1—Cr—O1^{ii}	82.87 (12)	O2^{vi}—K—O2^{vii}	109.36 (4)
O2^{v}—K—O2	180	O2—C—O1	125.5 (3)
O2^{v}—K—O2^{vi}	80.93 (11)	O2—C—C^{ii}	121.17 (19)
O2—K—O2^{vi}	99.07 (11)	O1—C—C^{ii}	113.31 (15)
O2^{v}—K—O2^{vii}	112.93 (7)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H···O1^{ix}	0.78 (4)	1.88 (4)	2.636 (3)	163 (4)

Symmetry code: (ix) $x-\frac{1}{2}, y+\frac{1}{2}, z$.

The H atoms were located in a difference Fourier map and were refined isotropically. The deepest hole is located 0.39 Å from atom K.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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: *WinGX* publication routines (Farrugia, 1999).

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