Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Dipotassium di-μ-hydroxo-bis[(nitrilotriacetato-κ^4*N*,*O*,*O*′,*O*″)chromium(III)] hexahydrate

Jong-Ha Choi,[a] Takayoshi Suzuki[b] and Sumio Kaizaki[b]*

[a]Department of Chemistry, Andong National University, Andong 760-749, South Korea, and [b]Department of Chemistry, Graduate School of Science, Osaka University, Toyonaka 560-0043, Japan

Correspondence e-mail:
kaizaki@chem.sci.osaka-u.ac.jp

Received 11 August 2003
Accepted 26 August 2003
Online 30 August 2003

Key indicators

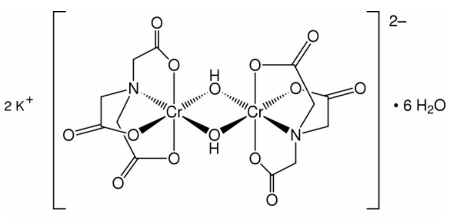
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
H-atom completeness 54%
R factor = 0.056
wR factor = 0.179
Data-to-parameter ratio = 18.9

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

The crystal structure of the title compound, $K_2[Cr_2(C_6H_6NO_6)_2(OH)_2]\cdot 6H_2O$ or $K_2[Cr_2(\mu\text{-}OH)_2(nta)_2]\cdot 6H_2O$ (nta = nitrilotriacetate), is composed of a dinuclear hydroxo-bridged chromium(III) complex anion, potassium cation and water molecules. Each Cr^{III} ion is in a distorted octahedral environment and coordinated by one nitrogen and three carboxylate O atoms of the nta^{3-} ligand plus two bridging hydroxo O atoms. The dinuclear complex anion possesses 2/*m* crystallographic symmetry. The Cr—O(μ-OH) bond lengths are 1.937 (4) and 1.951 (4) Å. The Cr—N and Cr—O bond distances of the nta^{3-} ligand are 2.054 (4) and 1.968 (3)–1.983 (3) Å, while the HO—Cr—OH and Cr—OH—Cr angles are 80.46 (16) and 99.54 (16)°, respectively.

Comment

The nitrilotriacetate ion (nta^{3-}) may coordinate to a metal ion as a tridentate or tetradentate ligand *via* the one N and three carboxylate O atoms. The crystal structure of di(μ-hydroxo)-bis[(nitrilotriacetato)chromium(III)] with the Cs^+ cation has been reported previously (Visser *et al.*, 1999). The present structure is another example of a $[Cr_2(\mu\text{-}OH)_2(nta)_2]^{2-}$ complex anion with a different cation, K^+. The sharp-line electronic transitions and their splittings in chromium(III) complexes are very sensitive to the exact bond angles around the metal. Thus, it may be possible to extract structural information from the electronic spectroscopy without a full X-ray structure determination (Hoggard, 1986; Choi, 1994); the relationship between environmental changes and geometric distortion may be found in the chromium(III) complexes. In order to examine the influence of cations and water contents of the crystal on the configurations of the Cr^{III} complexes, the title complex, (I), was prepared and its crystal structure was determined.



(I)

The structure analysis showed that the crystal of (I) consists of a $[Cr_2(\mu\text{-}OH)_2(nta)_2]^{2-}$ anion, two K^+ cations, and six water molecules. Each Cr^{III} ion displays a distorted octahedral environment, formed by two bridging hydroxo O atoms and one N and three O atoms of the nta^{3-} ligand. It is also found that with decreasing ionic radius of the cations the water content increases from four for Cs^+ to six for K^+ (Golič & Bulc, 1988).

Experimental

The starting material, $K_3[Cr(nta)_2]\cdot 2H_2O$, was synthesized according to the literature (Visser *et al.*, 1999). This complex was suspended in water and a blue–black precipitate was deposited after several days. Recrystallization of the precipitate from water afforded brown crystals of (I) suitable for X-ray analysis.

Crystal data

$K_2[Cr_2(C_6H_6NO_6)_2(OH)_2]\cdot 6H_2O$
$M_r = 700.55$
Monoclinic, $C2/m$
$a = 7.311$ (2) Å
$b = 13.321$ (2) Å
$c = 12.862$ (2) Å
$\beta = 94.45$ (2)°
$V = 1248.8$ (5) Å^3
$Z = 2$
$D_x = 1.863$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.7$–$15.0°$
$\mu = 1.30$ mm^{-1}
$T = 296$ (2) K
Block, brown
0.30 × 0.22 × 0.20 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.755$, $T_{\max} = 0.790$
2000 measured reflections
1869 independent reflections
1277 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 30.0°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = -17 \rightarrow 17$
3 standard reflections every 150 reflections intensity decay: 2.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.179$
$S = 1.04$
1869 reflections
99 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1161P)^2 + 0.9272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.81$ e Å^{-3}
$\Delta\rho_{\min} = -0.71$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr—O7	1.937 (4)	K···O1^{ii}	2.815 (2)
Cr—O7^i	1.951 (4)	K···O3	2.868 (3)
Cr—O1	1.968 (3)	K···O4	2.926 (3)
Cr—O3	1.983 (3)	K···O3^{iii}	3.033 (3)
Cr—N1	2.054 (4)		
O7—Cr—O7^i	80.46 (16)	O3—Cr—O3^{iv}	165.32 (14)
O7—Cr—O1	96.56 (15)	O7—Cr—N1	178.84 (16)
O7^i—Cr—O1	177.02 (14)	O7^i—Cr—N1	98.38 (15)
O7—Cr—O3	97.34 (7)	O1—Cr—N1	84.60 (15)
O7^i—Cr—O3	91.38 (8)	O3—Cr—N1	82.67 (7)
O1—Cr—O3	89.00 (8)	Cr—O7—Cr^i	99.54 (16)

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

Table 2
Contact distance (Å).

Cr···Cr^i	2.9687 (15)

Symmetry code: (i) $-x, -y, 1-z$.

The H atoms of nta^{3-} and bridging OH^- were placed geometrically, with O—H distances of 0.91 Å and C—H distances of 0.97 Å, and these atoms were refined using a riding model, $U_{\rm iso}(H) = U_{\rm eq}$(O or C). Other H atoms of water molecules were not included. In the final difference synthesis map, four rather large peaks (more than 0.65 e Å^{-3}) were found at (*a*) (0.0331, 0.0000, 0.4198), (*b*) (0.1122, 0.3315, 0.6691), (*c*) (0.3252, 0.1676, 0.4900), and (*d*) (0.0425, 0.3444, 0.4254). Peaks *a* and *b* were close to O7 and O4 at distances of 0.666 and 1.142 Å, respectively, while peaks *c* and *d* were located around the K atom at distances of 1.283 and 1.075 Å, respectively. These peaks are therefore not related to the H atoms of water molecules.

Data collection: *Rigaku/AFC Diffractometer Control Software* (Rigaku, 1985); cell refinement: *Rigaku/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation and Rigaku, 2000); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1970); software used to prepare material for publication: *SHELXL*97.

The authors gratefully acknowledge the support of this research by a Grant-in-Aid for Scientific Research (No. 10304056) from the Ministry of Education, Science and Culture, Japan.

References

Choi, J. H. (1994). *Bull. Korean Chem. Soc.* **15**, 145–150.
Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.
Golič, L. & Bulc, N. (1988). *Acta Cryst.* C**44**, 2065–2068.
Hoggard, P. E. (1986). *Coord. Chem. Rev.* **70**, 85–120.
Johnson, C. K. (1970). *ORTEP*. Report ORNL-3794, 2nd revision. Oak Ridge National Laboratory, Tennessee, USA.
Molecular Structure Corporation & Rigaku (2000). *TEXSAN*. Version 1.11. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo 196–8666, Japan.
Rigaku (1985). *Rigaku/AFC Diffractometer Control Software*. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196–8666, Japan.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Visser, H. G., Purcell, W. & Basson, S. S. (1999). *Polyhedron*, **18**, 2795–2801.

Acta Cryst. (2003). E**59**, m812–m813 Jong-Ha Choi *et al.* • $K_2[Cr_2(C_6H_6NO_6)_2(OH)_2]\cdot 6H_2O$ **m813**