

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their parent C atoms. The H atoms of the methyl C atoms and of C2 were refined for a few cycles and then a riding model was used for all H atoms, with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C})$.

Data collection and cell parameters: *CAD-4 Express* (Enraf–Nonius, 1994). Data reduction, structure solution and refinement, molecular graphics and preparation of material for publication: *MolEN* (Fair, 1990). Other programs include *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1153). Services for accessing these data are described at the back of the journal.

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Calcium Potassium Tris(oxalato-*O*¹,*O*²)-chromate(III) Pentahydrate

VANESSA MASTERS, LAWRENCE R. GAHAN AND COLIN H. L. KENNARD

Department of Chemistry, The University of Queensland, Brisbane, Q 4072, Australia. E-mail: c.kennard@mailbox.uq.edu.au

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Abstract

The Cr^{III} atom in CaK[Cr(C₂O₄)₃].5H₂O, has a regular octahedral geometry with three oxalato groups completing the coordination. Both the calcium and potassium

cations are coordinated to the O atom of the oxalate group.

Comment

This is another example of a [Cr(ox)₃]³⁻ complex (ox is oxalato) but with different cations (Bulc, Golic & Siftar, 1982, 1985; Merrachi, Mentzen & Chassagneux, 1986, 1987; van Niekerk & Schoening, 1952; Taylor, 1978). This compound is a complex existing as a double salt of monovalent potassium and divalent calcium cations and the chromium(III) cation.

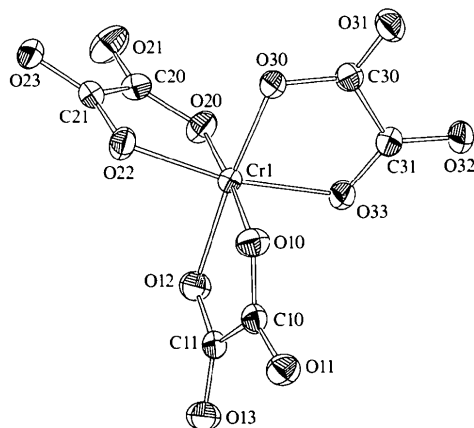
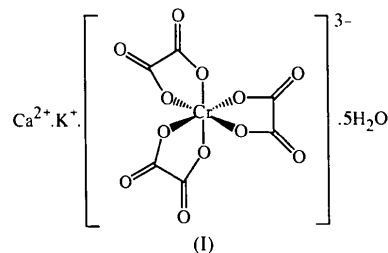


Fig. 1. *PLATON96* (Spek, 1996) view of [Cr(ox)₃]³⁻ with displacement ellipsoids drawn at the 30% probability level.

Experimental

Crystals of (I) were obtained by slow evaporation of an aqueous solution of the complex.

Crystal data

CaK[Cr(C₂O₄)₃].5H₂O

$M_r = 485.32$

Monoclinic

$P2_1/n$

$a = 10.923(4) \text{ \AA}$

$b = 11.114(1) \text{ \AA}$

$c = 13.548(4) \text{ \AA}$

$\beta = 99.52(1)^\circ$

$V = 1622.1(8) \text{ \AA}^3$

$Z = 4$

$D_x = 1.996 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 10\text{--}13^\circ$

$\mu = 1.368 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Well formed needle

$0.53 \times 0.33 \times 0.27 \text{ mm}$

Purple

Data collection

CAD-4 MicroVAX-
controlled diffractometer
 θ/ω scans
Absorption correction:
empirical ψ scan (North,
Phillips & Mathews,
1968)
 $T_{\min} = 0.62$, $T_{\max} = 0.69$
3006 measured reflections
2849 independent reflections

2497 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 13$
 $l = -16 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.087$
 $S = 1.100$
2849 reflections
235 parameters
H atoms not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 2.1859P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.662 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.641 \text{ e } \text{\AA}^{-3}$
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Cr1—O20	1.959 (2)	Ca1—O23 ⁱⁱⁱ	2.481 (2)
Cr1—O30	1.960 (2)	Ca1—O32 ⁱ	2.513 (2)
Cr1—O10	1.968 (2)	Ca1—O23 ⁱⁱ	2.701 (2)
Cr1—O33	1.977 (2)	K1—O5	2.696 (3)
Cr1—O22	1.977 (2)	K1—O4	2.774 (3)
Cr1—O12	1.978 (2)	K1—O2 ^{iv}	2.776 (3)
Ca1—O2	2.445 (2)	K1—O13 ^{iv}	2.944 (2)
Ca1—O31 ⁱ	2.456 (2)	K1—O30 ^v	3.043 (2)
Ca1—O1	2.468 (2)	K1—O22 ^v	3.301 (2)
Ca1—O21 ⁱⁱ	2.470 (2)		

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

The structure was solved using the *SHELXS86* (Sheldrick, 1990) Patterson heavy-atom method and refined using *SHELXL93* (Sheldrick, 1993). H atoms were found in difference maps.

Data collection: *SDP* (Frenz, 1985). Cell refinement: *SDP*. Data reduction: *Xtal3.0* (Hall & Stewart, 1990). Molecular graphics: *PLATON96* (Spek, 1996). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1140). Services for accessing these data are described at the back of the journal.

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[(1,2,5,6- η)-1,5-Cyclooctadiene][(11R,12R)-9,10-dihydro-9,10-ethanoanthracene-11,12-bis(diphenylphosphino-*P*)]rhodium(I) Tetrafluoroborate

TAI Y. FU, ZHAOQING LIU, STEVEN J. RETTIG, JOHN R. SCHEFFER AND JAMES TROTTER

Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1. E-mail: jtrt@xray4.chem.ubc.ca

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Abstract

The title compound, $[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_{40}\text{H}_{32}\text{P}_2)]\text{BF}_4$, which functions as an asymmetric catalyst, contains a five-membered chelate ring, and has molecular geometry and dimensions similar to those in related materials.

Comment

The Rh–anthraphos title complex, (I), was prepared for use as an asymmetric catalyst in hydrogenation reactions (Fu, Liu, Scheffer & Trotter, 1994).

