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 $U_{iso}(H) = 1.3U_{eq}(C)$.

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).

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

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.

## References

Basu Baul, T. S. & Tiekink, E. R. T. (1996). *Acta Cryst.* C**52**, 1428–1430.

Danish, M., Ali, S., Mazhar, M., Badshah, A., Masood, T. & Tiekink, E. R. T. (1995). *Main Group Met. Chem.* **18**, 27–34.

Enraf–Nonius (1994). *CAD-4 Express*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Ng, S. W. & Kumar Das, V. G. (1996). *Acta Cryst.* C**52**, 1371–1373.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Stocco, G., Gulì, G., Giraslo, M. A., Bruno, G., Nicolò, F. & Scopelliti, R. (1996). *Acta Cryst.* C**52**, 829–832.

Tahir, M. N., Ülkü, D., Danish, M., Ali, S., Badshah, A. & Mazhar, M. (1997). *Acta Cryst.* C**53**, 183–185.

Tiekink, E. R. T. (1994). *Trends Organomet. Chem.* **1**, 71–116.

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# Calcium Potassium Tris(oxalato-$O^1$,$O^2$)-chromate(III) Pentahydrate

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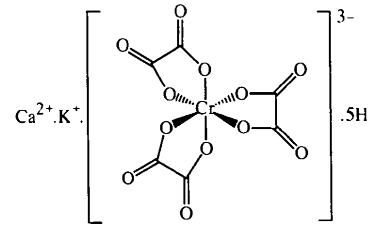
## Abstract

The $Cr^{III}$ atom in $CaK[Cr(C_2O_4)_3].5H_2O$, 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.



(I)

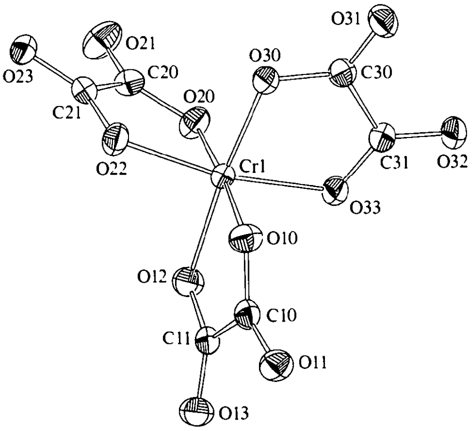


Fig. 1. *PLATON*96 (Spek, 1996) view of $[Cr(ox)_3]^{3-}$ with displacement ellipsoids drawn at the 30% probability level.

## Experimental

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

*Crystal data*

$CaK[Cr(C_2O_4)_3].5H_2O$
$M_r = 485.32$
Monoclinic
$P2_1/n$
$a = 10.923\ (4)$ Å
$b = 11.114\ (1)$ Å
$c = 13.548\ (4)$ Å
$\beta = 99.52\ (1)°$
$V = 1622.1\ (8)$ Å$^3$
$Z = 4$
$D_x = 1.996$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–13°$
$\mu = 1.368$ mm$^{-1}$
$T = 293\ (2)$ K
Well formed needle
$0.53 \times 0.33 \times 0.27$ mm
Purple

*Data collection*

CAD-4 MicroVAX-controlled diffractometer
$\theta/\omega$ scans
Absorption correction: empirical $\psi$ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.62$, $T_{\max} = 0.69$
3006 measured reflections
2849 independent reflections
2497 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -16 \rightarrow 15$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.662$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.641$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cr1—O20 | 1.959 (2) | Ca1—O23$^{\rm iii}$ | 2.481 (2) |
| Cr1—O30 | 1.960 (2) | Ca1—O32$^{\rm i}$ | 2.513 (2) |
| Cr1—O10 | 1.968 (2) | Ca1—O23$^{\rm ii}$ | 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$^{\rm iv}$ | 2.776 (3) |
| Ca1—O2 | 2.445 (2) | K1—O13$^{\rm iv}$ | 2.944 (2) |
| Ca1—O31$^{\rm i}$ | 2.456 (2) | K1—O30$^{\rm v}$ | 3.043 (2) |
| Ca1 O1 | 2.468 (2) | K1—O22$^{\rm v}$ | 3.301 (2) |
| Ca1—O21$^{\rm ii}$ | 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 *SHELXS*86 (Sheldrick, 1990) Patterson heavy-atom method and refined using *SHELXL*93 (Sheldrick, 1993). H atoms were found in difference maps.

Data collection: *SDP* (Frenz, 1985). Cell refinement: *SDP*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Molecular graphics: *PLATON*96 (Spek, 1996). Software used to prepare material for publication: *SHELXL*93.

The authors wish to thank the Australian Research Council, the University of Queensland and Griffith University for financial support for the purchase of the CAD-4.

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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.

## References

Bulc, N., Golic, L. & Siftar, J. (1982). *Vestn. Slov. Kem. Drus.* **29**, 211–225.
Bulc, N., Golic, L. & Siftar, J. (1985). *Vestn. Slov. Kem. Drus.* **32**, 221–229.
Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.
Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual*. Universities of Western Australia, Australia, and Maryland, USA.
Merrachi, E., Mentzen, B. & Chassagneux, F. (1986). *Rev. Chim. Miner.* **23**, 329–342.
Merrachi, E., Mentzen, B. & Chassagneux, F. (1987). *Rev. Chim. Miner.* **24**, 427–445.
Niekerk, J. van & Schoening, F. (1952). *Acta Cryst.* **5**, 499–505.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Spek, A. L. (1996). *PLATON*96. *Molecular Geometry Program*. University of Utrecht, The Netherlands.
Taylor, D. (1978). *Aust. J. Chem.* **31**, 1455–1462.

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

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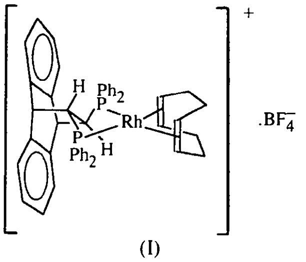
## Abstract

The title compound, $[\mathrm{Rh(C_8H_{12})(C_{40}H_{32}P_2)}]\mathrm{BF_4}$, 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).



(I)