

Infinite chain of $[\text{Ba}\{\text{Cr}(\text{ox})_2(\text{phen})\}_2]_n$

Krister Larsson

Department of Inorganic Chemistry, Chalmers
University of Technology, SE-412 96 Göteborg,
Sweden

Correspondence e-mail: kla@inoc.chalmers.se

Key indicators

Single-crystal X-ray study

$T = 293 \text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$

H-atom completeness 58%

R factor = 0.044

wR factor = 0.125

Data-to-parameter ratio = 15.2

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

The title compound, *catena*-poly[[diaquabarium(II)-bis[di- μ -oxalato-[(1,10-phenanthroline)chromate(III)]]] tetrahydrate, $[\text{Ba}\{\text{Cr}(\text{C}_2\text{O}_4)_2(\text{C}_{12}\text{H}_8\text{N}_2)\}_2(\text{H}_2\text{O})_6]_n$, with ten-coordinate barium(II) and six-coordinate chromium(III), consists of infinite chains along the b axis and exhibits stacking with π -interactions between the chains. Even though the crystal is racemic, only one of the enantiomers of the chromium anion is present in the single chain due to the twofold axis passing through the barium, making the chains chiral.

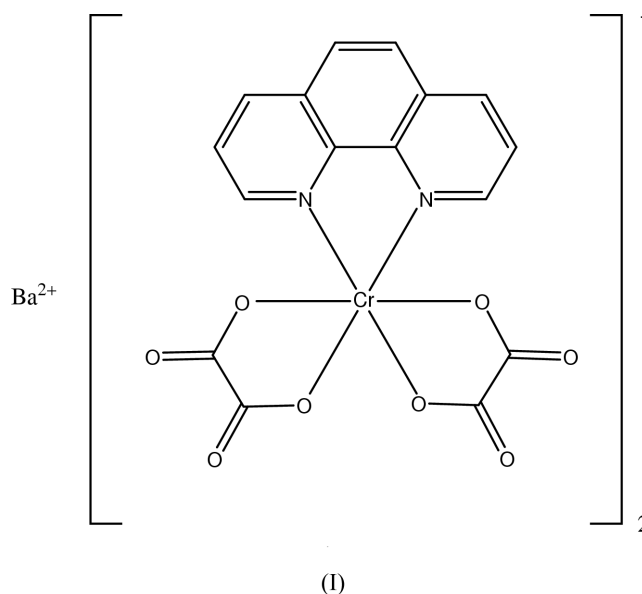
Received 21 March 2001

Accepted 4 April 2001

Online 12 April 2001

Comment

The structure of several compounds with the $[\text{Cr}(\text{ox})_2(\text{L}-\text{L})]^-$ ($\text{ox} = \text{oxalate}$, $\text{L}-\text{L} = \text{phen}$ or bipy , $\text{phen} = 1,10\text{-phenanthroline}$, $\text{bipy} = \text{bipyridine}$) anion have been published. By using different counter-ions, such as Na^+ , K^+ , Mn^{2+} and Ba^{2+} , one-dimensional chains as well as two- and three-dimensional nets have been obtained (Rochon & Massarweh, 1999; Marinescu *et al.*, 2000). In $\text{Ba}[\text{Cr}(\text{ox})_2(\text{bipy})]_2$, the complex forms a three-dimensional network. This is, however, not the case for the title compound, (I), which forms an infinite one-dimensional structure similar to $[\text{Mn}\{\text{Cr}(\text{ox})_2(\text{bipy})\}_2]_n$ (Rochon *et al.*, 1996).



The title compound has an asymmetric unit containing one barium and one chromium with two oxalates and one 1,10-phenanthroline (Fig. 1). Barium exhibits a ten-coordinate environment with four oxalates and two water molecules and chromium coordinates two oxalates and one phenanthroline in a distorted octahedral environment (Table 1). The asym-

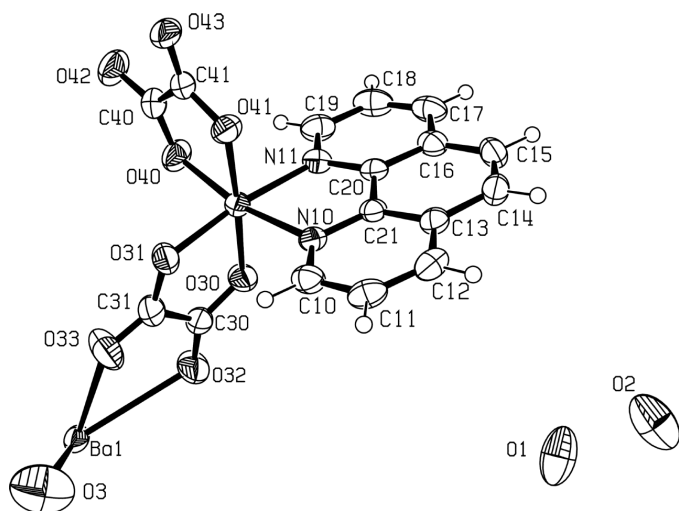


Figure 1
View of (I) shown with 50% probability displacement ellipsoids.

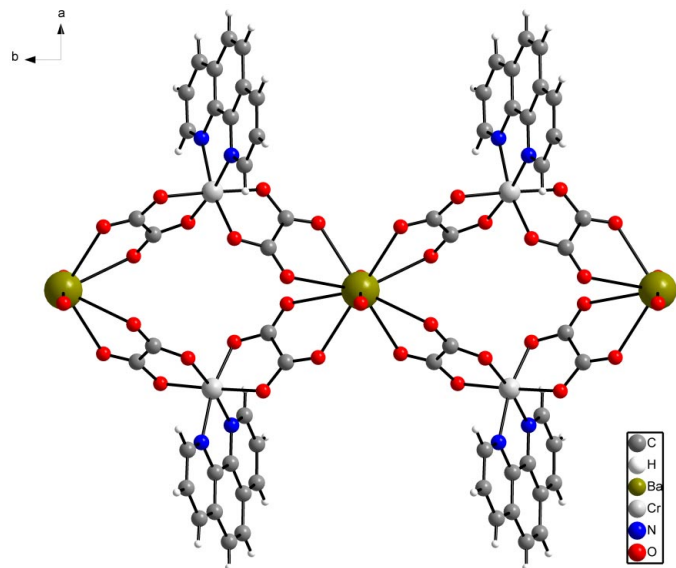


Figure 2
View of the chain in (I) along the twofold axis.

metric unit forms infinite chains along the b axis and has a twofold symmetry (Fig. 2). The two chromium complex anions acts as a bridge between the barium ions with solvent water located in the cavities around barium. Within the chains, the Ba...Ba distance is 10.154 (1) Å and the bridging Cr atoms are 6.892 (12) Å apart. The four chains in the unit cell are related to each other by a twofold screw axis and a c -glide plane normal to the b axis. This results in the enantiomers forming layers along the c axis which are stacked together by π -interactions. These interactions result in the phenanthrolines being 3.4 Å apart with a parallel displacement of 3 Å, not uncommon in aromatic compounds containing heteroatoms (Janiak, 2000). Even though the structure contains both enantiomers of the octahedral Cr anion, only one of the enantiomers, Δ or Λ , is found in the individual chains. This makes the chains chiral as in the [Mn(Cr(bi-

py)(ox)₂]_n analog (Rochon *et al.*, 1996).

The structure, which differs from its bipyridine analog, shows the importance of π -interactions in crystal design which, in this case, plays a key role in the topology of the framework.

Experimental

The compound was synthesized according to the method described by Broomhead (1962).

Crystal data

[Ba{Cr(C₂O₄)₂(C₁₂H₈N₂)₂(H₂O)₆]
 $M_r = 1061.92$
 Monoclinic, $C2/c$
 $a = 26.1127$ (18) Å
 $b = 10.1535$ (6) Å
 $c = 14.6165$ (9) Å
 $\beta = 92.367$ (7)°
 $V = 3872.0$ (4) Å³
 $Z = 4$

$D_x = 1.822$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 3.1$ – 27°
 $\mu = 1.65$ mm⁻¹
 $T = 293$ (2) K
 Prism, red-violet
 0.3 × 0.2 × 0.2 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.593$, $T_{\max} = 0.760$
 4442 measured reflections
 4203 independent reflections
 2749 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 27.0^\circ$
 $h = 0 \rightarrow 33$
 $k = 0 \rightarrow 12$
 $l = -18 \rightarrow 18$
 4 standard reflections
 frequency: 120 min
 intensity decay: 21%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.125$
 $S = 1.01$
 4203 reflections
 276 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 13.8292P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 2.09$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.15$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ba1—O43 ⁱ	2.729 (4)	Cr1—O41	1.946 (3)
Ba1—O32	2.861 (4)	Cr1—O30	1.959 (3)
Ba1—O3	2.869 (7)	Cr1—O31	1.959 (3)
Ba1—O33	2.883 (4)	Cr1—N11	2.062 (4)
Ba1—O42 ⁱ	2.955 (4)	Cr1—N10	2.065 (4)
Cr1—O40	1.946 (3)		
O32—Ba1—O33	57.29 (11)	O30—Cr1—O31	82.70 (14)
O43 ⁱ —Ba1—O42 ⁱ	58.33 (11)	N11—Cr1—N10	80.11 (15)
O40—Cr1—O41	83.52 (14)		

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

No H atoms were assigned to the water molecules. A correction for intensity decay was applied by the default method in *XCAD4* (Harms & Wocadlo, 1995)

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997).

The author would like to thank Drs G. Svensson and L. Öhrström for fruitful discussions.

References

Broomhead, J. A. (1962). *Aust. J. Chem.* **15**, 228–231.

- Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Janiak, C. (2000). *J. Chem. Soc. Dalton. Trans.* pp. 3885–3896.
- Marinescu, G., Andruh, M., Lascouëzec, R., Muñoz, M. C., Cano, J., Lloret, F. & Julve, M. (2000). *New J. Chem.* **24**, 527–536.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Rochon, F. D. & Massarweh, G. (1999). *Can. J. Chem.* **77**, 2059–2068.
- Rochon, F. D., Melanson, R. & Andruh, M. (1996). *Inorg. Chem.* **35**, 6086–6092.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.