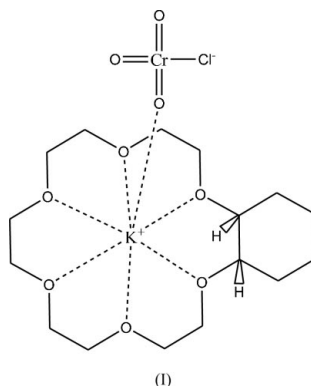


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roman@xray.isc.kharkov.com**Key indicators**Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.035
 wR factor = 0.084
Data-to-parameter ratio = 8.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(*cis*-Cyclohexano-18-crown-6)potassium chlorochromate(VI)**

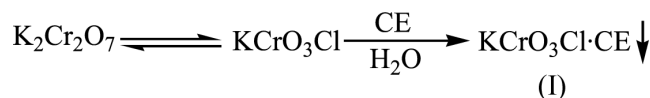
In the crystal structure of the title compound, (*cis*-hexadeca-hydro-1,4,7,10,13,16-benzohexaoxacyclooctadecin)potassium chlorochromate(VI), $[\text{K}(\text{C}_{16}\text{H}_{30}\text{O}_6)][\text{CrClO}_3]$, the cation forms almost equivalent bonds with the ether O atoms and with the O atom of the anion. The complex is chiral, but the crystal structure is racemic.

Received 14 June 2004
Accepted 21 June 2004
Online 30 June 2004**Comment**

The present report continues a series of X-ray structure investigations of new chromium(VI) complexes with aliphatic, cycloaliphatic, benzo- and dibenzocrown ethers (CEs). This investigation is aimed at the determination of the complex stoichiometry and details of the cation coordination, depending on the structure of the CE cavity and type of anion.



The title complex, (I) (Fig. 1), was prepared by addition of *cis*-cyclohexano-18-crown-6 to an equilibrium mixture of potassium dichromate and chlorochromate which forms in aqueous HCl solution. By the same method, we have previously obtained the sandwich-type complex of *cis*-cyclohexano-12-crown-4 with potassium chlorochromate, KCrO_3Cl , in a 2:1 molar ratio (Kotlyar *et al.*, 2004).



We have shown that, in changing from the 12-membered to the 18-membered ligand, the selectivity of the CEs to potassium chlorochromate is retained, but the complex stoichiometry and the interaction between the complex cation and anion are changed.

The cation in (I) is situated above the mean plane of the CE O atoms at a distance of 0.51 Å towards the anion. It forms almost equivalent bonds both with ether O atoms and with atom O9 of the anion (Table 1). The macrocycle has a crown-

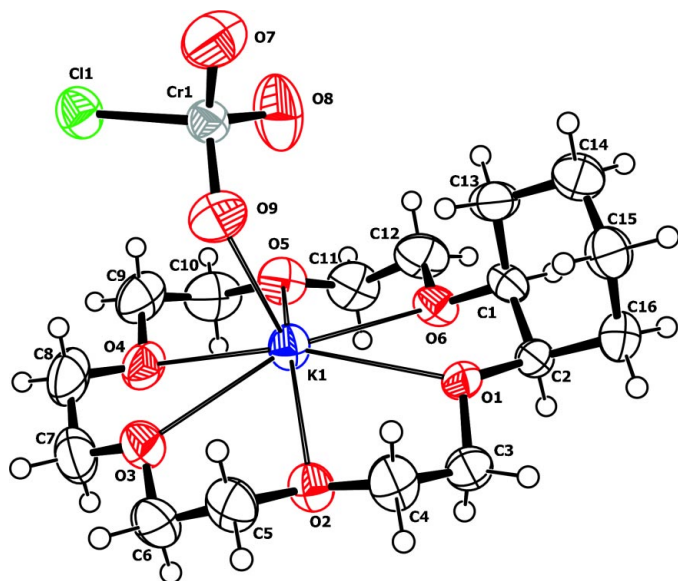


Figure 1
View of (I), with 50% probability displacement ellipsoids.

like conformation, with the O atoms oriented towards the cation. The O—C—C—O fragments have alternating *+cs* and *-sc* conformations. The rings are *cis*-fused and the H1—C1—C2—H2 torsion angle is -57° . The molecules of (I) are chiral, with two chiral centers, C1 and C6, having absolute configurations (*R* and *S* in the molecule depicted in Fig. 1). The crystal structure, with glide planes, is racemic.

Experimental

The title complex, (I), was prepared according to the procedure of Kotlyar *et al.* (2004). Suitable transparent orange crystals [m.p. 417–420 K (decomposition)] were obtained by the evaporation of an acetone solution. Analysis calculated for $C_{16}H_{30}ClCrO_9$: C 38.98, H 6.13, Cl 7.19, Cr 10.55%; found: C 39.15, H 6.03, Cl 7.10, Cr 10.44%. The crystals are soluble in methylene chloride, dimethyl sulfoxide and dimethylformamide, moderately soluble in chloroform, acetone and ethyl acetate, and slightly soluble in benzene.

Crystal data

$[K(C_{16}H_{30}O_6)][CrClO_3]$
 $M_r = 492.95$
 Orthorhombic, $Pca2_1$
 $a = 14.693$ (4) Å
 $b = 9.221$ (3) Å
 $c = 16.515$ (5) Å
 $V = 2237.5$ (12) Å³
 $Z = 4$
 $D_x = 1.463$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 24 reflections
 $\theta = 12$ – 18°
 $\mu = 0.86$ mm⁻¹
 $T = 293$ (2) K
 Prism, orange
 $0.30 \times 0.20 \times 0.15$ mm

Data collection

Siemens P3/PC diffractometer
 θ - 2θ scans
 2040 measured reflections
 2040 independent reflections
 1405 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 25.1^\circ$

$h = 0 \rightarrow 17$
 $k = 0 \rightarrow 10$
 $l = -19 \rightarrow 0$
 2 standard reflections
 every 98 reflections
 intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.084$
 $S = 0.94$
 2040 reflections
 253 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³
 Absolute structure: Flack (1983),
 no Friedel pairs
 Flack parameter = 0.00 (4)

Table 1

Selected geometric parameters (Å, °).

K1—O1	2.886 (3)	K1—O5	2.778 (4)
K1—O2	2.798 (4)	K1—O6	2.789 (4)
K1—O3	2.823 (4)	K1—O9	2.797 (5)
K1—O4	2.876 (4)		
O1—C3—C4—O2	70.3 (6)	O4—C9—C10—O5	-66.2 (7)
O2—C5—C6—O3	-62.4 (6)	O5—C11—C12—O6	63.4 (7)
O3—C7—C8—O4	64.8 (7)	O6—C1—C2—O1	-58.7 (5)

All H atoms were placed in calculated positions and included in the refinement in the riding-model approximation; C—H = 0.97–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: P3 (Siemens, 1989); cell refinement: P3; data reduction: XDISK (Siemens, 1991); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1991); software used to prepare material for publication: SHELXL97.

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