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Key indicators

Single-crystal X-ray study
 T = 100 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 R factor = 0.027
 wR factor = 0.050
 Data-to-parameter ratio = 28.6

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

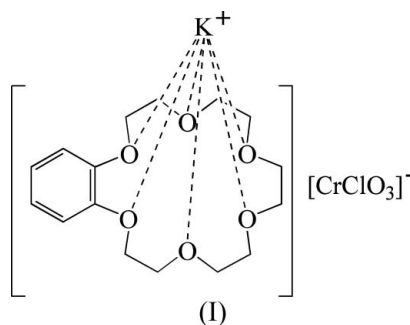
(Benzo-18-crown-6)potassium chlorochromate(VI)

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In the structure of the title compound, (2,3,5,6,8,9,11,12,14,15-decahydro-1,4,7,10,13,16-benzohexaoxacyclooctadecine)-potassium chlorochromate(VI), $[\text{K}(\text{C}_{16}\text{H}_{24}\text{O}_6)][\text{CrClO}_3]$, the K^+ cation is located approximately in the center of the crown ether cavity and is coordinated by two anions, one on each side of the macrocyclic ring, one anion being related to the other by the symmetry operator $(x, y - 1, z)$. One anion is bonded through O and Cl atoms and the other only by an O atom. As a result, in the crystal structure, polymeric chains, consisting of alternating anions and cations, are formed along the [010] direction.

Comment

Mixing ethyl acetate solutions of various aromatic crown ethers (CEs) and potassium chlorochromate (KCrClO_3) at room or increased temperature easily gives stable crystalline host-guest complexes (Kotlyar *et al.*, 2000). Their formation occurs without participation of the solvent. Interest has been focused on the comparison of structures of such complexes of benzo- and dibenzoCEs, containing different functional groups (bromo, nitro, amino, *etc.*). Recently, the crystal structures of 4-bromobenzo-18-crown-6 [(II); Kotlyar *et al.*, 2006a] and 4-nitrobenzo-18-crown-6 [(III); Kotlyar *et al.*, 2006b] with potassium chlorochromate 1:1 have been reported. In complex (II), the K^+ ion is coordinated equatorially by six O atoms from the macrocycle, and axially by two O atoms of symmetry-related anions, results in the formation of polymeric chains. In contrast, the presence of a nitro group in complex (III) results in formation of centrosymmetric dimers due to the axial coordination of the K^+ ion by the nitro group of a neighboring molecule.



We report here the crystal structure of the title complex, (I), obtained in the ratio 1:1 by mixing benzo-18-crown-6 and

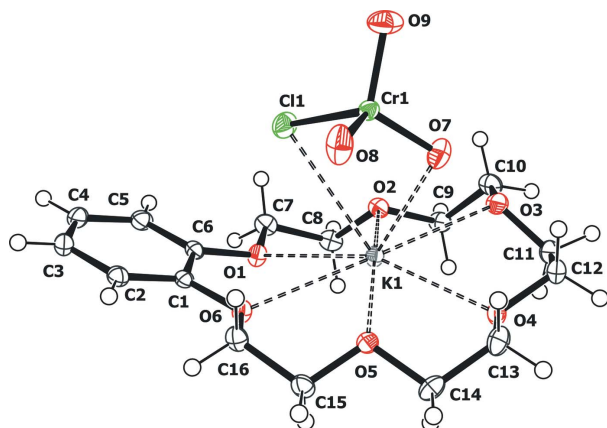


Figure 1
The asymmetric unit of the title structure with displacement ellipsoids drawn at the 50% probability level.

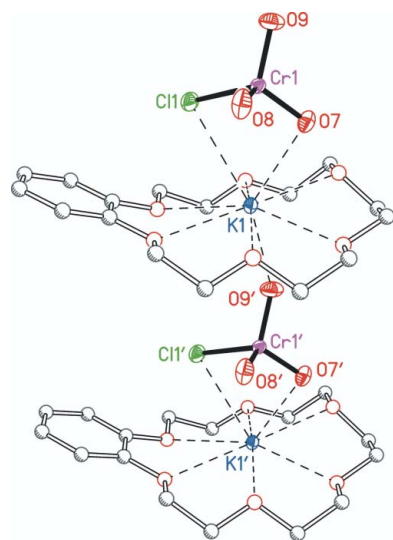


Figure 2
Part of the one-dimensional polymeric chain in the crystal structure of (I). H atoms have been omitted for clarity. Atoms having ' in the label are at the position $x, y - 1, z$.

KCrO_3Cl ethyl acetate solutions at room temperature. The title complex consists of a K^+ cation, a crown ether molecule and a chlorochromate anion (Fig. 1). The macrocycle possesses a crown-like conformation. Atoms O1, O2, O5 and O6 are approximately coplanar [r.m.s. deviation is 0.05 Å], with atoms O3 and O4 deviating from this plane by 0.532 (2) and -0.473 (2) Å, respectively. The K^+ cation is located approximately in the center of the CE cavity. The coordination bonds with ether O atoms are in the range 2.722 (1)–2.869 (1) Å. As in complex (II), the cation is bonded to two anions located on one on each side of the mean plane of the CE molecule, one being related to the other by the symmetry operation ($x, y - 1, z$). As a result, in the crystal structure, polymeric chains containing alternating cations and anions are formed (Fig. 2). The K^+ cation is bonded to atoms O7 and Cl1 of the symmetry-unique anion and to atom O9ⁱ [symmetry code: (i) $x, y - 1, z$] of the anion located on the other side of the macrocyclic ring. The participation of atoms O7 and O9ⁱ in the

coordination of K1 does not lead to elongation of the Cr1–O7 and Cr1–O9ⁱ bonds (Table 1).

Experimental

The title complex, (I), was prepared in 92% yield (4.29 g) according to a previously described procedure (Kotlyar *et al.*, 2006a) by mixing ethyl acetate solutions of benzo-18-crown-6 (3.0 g, 9.6 mmol in 20 ml of ethyl acetate) and potassium chlorochromate (1.68 g, 9.6 mmol in 69 ml of ethyl acetate) at room temperature. Transparent orange crystals of (I) suitable for X-ray diffraction study were obtained by evaporation of a dichloromethane solution [m.p. 444–446 K (decomposition)]. Analysis calculated for $\text{C}_{16}\text{H}_{24}\text{ClCrKO}_9$: C 39.47, H 4.97, Cl 7.28, Cr 10.68%; found: C 39.40, H 5.00, Cl 7.21, Cr 10.52%. The crystals are soluble at room temperature in dichloromethane, chloroform, dimethyl sulfoxide and dimethyl formamide, less soluble in acetone and 1,4-dioxane, and almost insoluble in diethyl ether, ethyl acetate, benzene and toluene.

Crystal data

$[\text{K}(\text{C}_{16}\text{H}_{24}\text{O}_6)][\text{CrClO}_3]$
 $M_r = 486.9$
Orthorhombic, $Pna2_1$
 $a = 15.8744$ (12) Å
 $b = 7.8637$ (10) Å
 $c = 16.562$ (2) Å
 $V = 2067.4$ (4) Å³

$Z = 4$
 $D_x = 1.564$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.93$ mm⁻¹
 $T = 100$ (2) K
Prism, pale orange
 $0.4 \times 0.3 \times 0.25$ mm

Data collection

Oxford Diffraction Xcalibur 3 diffractometer
 ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)
 $T_{\min} = 0.708, T_{\max} = 0.793$

15472 measured reflections
7230 independent reflections
5979 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\max} = 34.7^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.050$
 $S = 1.02$
7230 reflections
253 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.41$ e Å⁻³
 $\Delta\rho_{\min} = -0.37$ e Å⁻³
Absolute structure: Flack (1983),
2634 Friedel pairs
Flack parameter: 0.009 (11)

Table 1

Selected geometric parameters (Å, °).

Cr1–O7	1.6048 (11)	K1–O1	2.8215 (10)
Cr1–O9	1.6051 (12)	K1–O2	2.8217 (11)
Cr1–O8	1.6059 (11)	K1–O3	2.8371 (11)
Cr1–Cl1	2.2096 (5)	K1–O6	2.8686 (10)
K1–O4	2.7218 (10)	K1–O7	3.0953 (12)
K1–O9 ⁱ	2.8017 (13)	K1–Cl1	3.2394 (6)
K1–O5	2.8118 (10)		
C6–O1–C7	116.24 (11)	C12–O4–C13	112.40 (11)
C9–O2–C8	111.07 (11)	C15–O5–C14	110.54 (11)
C11–O3–C10	112.78 (11)	C1–O6–C16	116.97 (10)
O6–C1–C6–O1	−1.34 (17)	O3–C11–C12–O4	71.66 (16)
O1–C7–C8–O2	−69.91 (15)	O4–C13–C14–O5	−67.39 (16)
O2–C9–C10–O3	64.16 (15)	O5–C15–C16–O6	67.06 (15)

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

All H atoms were placed in calculated positions and included in the refinement in riding-model approximation, with C—H = 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WINGX* (Farrugia, 1999).

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