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Dipotassium hexachlorotantalate(IV), K_2TaCl_6

Liesbet Jongen and Gerd Meyer*

Institut für Anorganische Chemie, Universität zu Köln, Greinstraße 6, D-50939 Köln, Germany

Correspondence e-mail: gerd.meyer@uni-koeln.de

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Ta–Cl) = 0.003 Å
R factor = 0.020
wR factor = 0.042
Data-to-parameter ratio = 14.8

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

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K_2TaCl_6 was obtained as a by-product during the reaction of VCl_3 and KCl in a sealed tantalum container. The compound is isotypic with K_2PtCl_6 and contains octahedrally coordinated Ta atoms.

Comment

It is known from earlier studies that container materials can show undesired reactions, in the sense that tantalum reacts with one of the reagents, especially when unstable transition metal halides, such as VCl_3, are used (Böcker, 1996; Simon & Meyer, 1997). In an attempt to synthesize $KVCl_3$ from a mixture of VCl_3, V and KCl, black cubic single crystals of K_2TaCl_6 grew out of the wall of the tantalum container.

K_2TaCl_6 crystallizes isotypic with K_2PtCl_6 (Engel, 1935). Potassium and chloride ions form a cubic close packing of spheres. Tetravalent Ta^{IV} occupies half of the octahedral holes defined by Cl^- ions. Octahedral $[TaCl_6]^{2-}$ ions occupy the corners and face centres of the unit cell, and K^+ ions occupy the interstices formed by four such anions. The K^+ ions are cuboctahedrally coordinated by 12 Cl^- ions, with K^+—Cl^- distances of 3.5347 (6) Å. Ta^{4+}—Cl^- distances are 2.397 (3) Å.

Experimental

Black cubic crystals of K_2TaCl_6 were obtained as a by-product in an attempt to synthesize $KVCl_3$, starting from VCl_3, KCl and V powder (molar ratio 2:3:1) in a sealed tantalum container, jacketed by a silica ampoule. The reaction mixture was heated to 923 K for 10 d and then cooled slowly to room temperature. Single crystals of K_2TaCl_6 were selected under a microscope in an argon-filled dry-box.

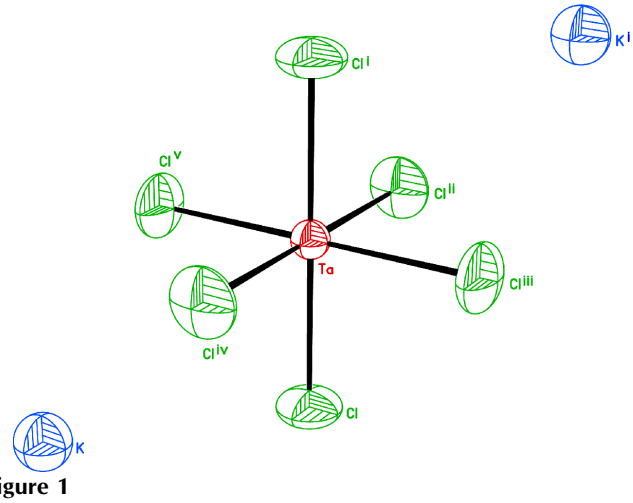

Figure 1
Octahedral coordination of the Ta atoms in K_2TaCl_6; displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x, -y, -z$; (ii) $y, -x, -z$; (iii) z, x, y; (iv) $-y, x, z$; (v) $-z, -x, -y$.]

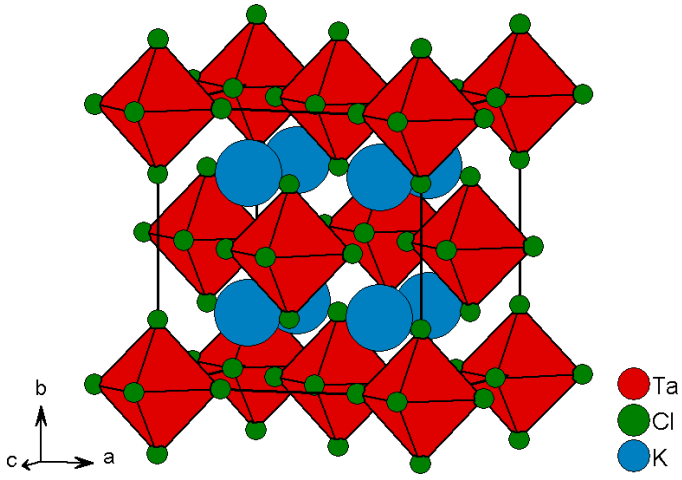

Figure 2
The unit-cell contents of K_2TaCl_6.

Crystal data

K_2TaCl_6
M_r = 471.85
Cubic, $Fm\bar{3}m$
a = 9.9935 (16) Å
V = 998.1 (3) Å^3
Z = 4
D_x = 3.140 Mg m^{-3}
Mo $K\alpha$ radiation

Cell parameters from 1314 reflections
θ = 1.9–28.2°
μ = 13.37 mm^{-1}
T = 293 (2) K
Cube, black
0.1 × 0.1 × 0.1 mm

Data collection

Stoe IPDS-I diffractometer
φ scans
Absorption correction: numerical (*X-RED*32; Stoe & Cie, 2002) after optimizing the crystal shape using *X-SHAPE* (Stoe & Cie, 1999)
T_{min} = 0.127, T_{max} = 0.240

2354 measured reflections
89 independent reflections
89 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 27.8°
$h = -13 \rightarrow 12$
$k = -13 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.042
S = 1.23
89 reflections
6 parameters

$w = 1/[\sigma^2(F_o^2) + 21.1812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ta—Cl^{iii}	2.397 (3)	K—Cl^{vi}	3.5347 (6)
Cl^{iii}—Ta—Cl^{ii}	90	Cl^{iii}—Ta—Cl^{v}	180

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999) and *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

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