Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Dicaesium hexachlorotantalate(IV), Cs_2TaCl_6

Hoseop Yun* and Gyung-Joo Jang

Division of Energy Systems Research and Department of Chemistry, Ajou University, Suwon 443-749, Republic of Korea

Correspondence e-mail: hsyun@ajou.ac.kr

Received 4 December 2006
Accepted 16 December 2006

Dicaesium hexachlorotantalate(IV), Cs_2TaCl_6, is a new member of the K_2PtCl_6 structure type. The structure consists of a face-centered cubic (fcc) $[TaCl_6]^{2-}$ anion lattice with Cs^+ cations occupying the tetrahedral holes. The Ta^{4+} ion is octahedrally coordinated by Cl^- ions and the Cs^+ ion is surrounded by Cl^- ions in a cubooctahedral fashion. The site symmetries of the Cs, Ta, and Cl atoms are $\bar{4}3m$, $m\bar{3}m$, and $4m.m$ respectively.

Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(Ta–Cl) = 0.003 Å
R factor = 0.028
wR factor = 0.056
Data-to-parameter ratio = 12.7

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

Comment

During an effort to expand the range of compounds within the quaternary Cs–Ta–P–S system by substituting alkali metals, single crystals of Cs_2TaCl_6 were obtained. The compound is isostructural with the previously reported K_2TaCl_6 (Jongen & Meyer, 2004). These phases adopt the K_2PtCl_6 structure type (Williams *et al.*, 1973). However, the synthesis and crystal structures of other phases in this $A_2M^{IV}Cl_6$ family (A = alkali metals, M = V, Nb, Ta) have not yet been reported (ICSD, 2006).

The structure of Cs_2TaCl_6 can be described in terms of a packing of Cs^+ and complex $[TaCl_6]^{2-}$ ions. The Ta atom is octahedrally coordinated by Cl^- ions with a Ta—Cl distance of 2.394 (4) Å, which corresponds to the usual Ta—Cl bond lengths for Ta^{4+} with an octahedral coordination of Cl^- (Bajan & Meyer, 1996). The structure consists of a face-centered cubic (fcc) $[TaCl_6]^{2-}$ anion lattice with Cs^+ cations occupying

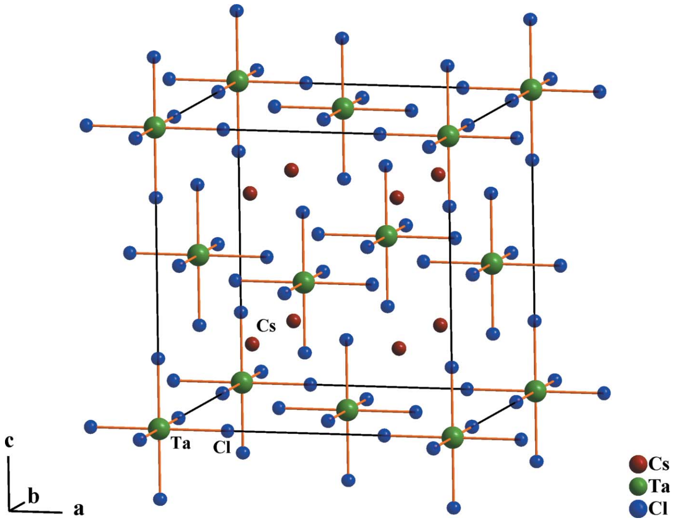

Figure 1
View of the unit-cell contents of Cs_2TaCl_6, showing displacement ellipsoids drawn at the 60% probability level.

the two types of tetrahedral voids (Fig. 1). The Cs—Cl distance [3.6354 (14) Å] is comparable with the sum of the ionic radii of Cs^+ (1.81 Å) and Cl^- ions (1.81 Å; Shannon, 1976).

Experimental

Cs_2TaCl_6 was prepared by the reaction of elemental Ta, P and S by the halide-flux technique. A combination of the pure elements, Ta powder (CERAC 99.8%), P powder (CERAC 99.5%) and S (CERAC 99.95%) were mixed in silica tubes in an atomic ratio Ta:P:S = 1:1:6, and then CsCl was added in a weight ratio of $TaPSe_6$:CsCl = 1:2. The tubes were evacuated to 10^{-2} Torr (1 Torr = 133.322 Pa), sealed, and heated gradually (5 K h^{-1}) to 973 K in a tube furnace, where they were kept for 96 h. The tubes were slowly cooled to room temperature at the rate of 5 K h^{-1}. The excess halide was removed with distilled water and black chunky crystals up to 0.5 mm in length were obtained. The crystals were stable in air. Qualitative analysis of the crystals with an EDAX-equipped scanning electron microscope indicated the presence of Cs, Ta, and Cl. No other element was detected.

Crystal data

Cs_2TaCl_6
$M_r = 659.47$
Cubic, $Fm\bar{3}m$
$a = 10.271$ (4) Å
$V = 1083.4$ (7) Å^3
$Z = 4$
$D_x = 4.043$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 18.18$ mm^{-1}
$T = 200$ (2) K
Block, black
0.30 × 0.25 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: numerical (*NUMABS*; Higashi, 2000) $T_{\min} = 0.008$, $T_{\max} = 0.025$
2146 measured reflections
89 independent reflections
85 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.056$
$S = 1.18$
89 reflections
7 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0012P)^2 + 79.0954P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.58$ e Å^{-3}
$\Delta\rho_{\min} = -1.44$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0032 (3)

The program *STRUCTURE TIDY* (Gelato & Parthé, 1987) was used to standardize the positional parameters. The highest residual electron density and the deepest hole were 0.81 and 0.91 Å, respectively, from the Ta site.

Data collection: *RAPID-AUTO* (Rigaku, 2005); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: locally modified version of *ORTEP* (Johnson, 1965); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This research was supported by the Korean Research Foundation (KRF-2006–521-C00088). Use was made of the X-ray facilities supported by Ajou University.

References

Bajan, B. & Meyer, H. J. (1996). *Z. Kristallogr.* **211**, 818.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gelato, L. M. & Parthé, E. (1987). *J. Appl. Cryst.* **20**, 139–143.
Higashi, T. (2000). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
ICSD (2006). *Inorganic Crystal Structure Database.* Version 2006-01. FIZ-Karlsruhe, Germany.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Jongen, L. & Meyer, G. (2004). *Acta Cryst.* E**60**, i91–i92.
Rigaku (2005). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Shannon, R. D. (1976). *Acta Cryst.* A**32**, 751–767.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Williams, R. J., Dillin, D. R. & Milligan, W. O. (1973). *Acta Cryst.* B**29**, 1369–1372.