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A synchrotron study of $Ba_5Ta_2Cl_2O_9$

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The structure of pentabarium ditantalum dichloride nonaoxide, $Ba_5Ta_2Cl_2O_9$, is isotypic with $Ba_5Ru_{1.6}W_{0.4}Cl_2O_9$ and with one polymorph of $Ba_5Ru_2Cl_2O_9$. It is related to the perovskite structure and shows a ten-layer stacking of BaO_3 and BaCl blocks along the *c* axis. The Ta cations occupy octahedral interstices, forming Ta_2O_9 dimers of distorted face-shared TaO_6 octahedra. Except for one O atom, all atoms are situated on special positions: Ba1 (Wyckoff position $\overline{6}m2$), Ba2 and Ba3 ($3m.$), Ta ($3m.$), Cl ($3m.$) and O1 ($mm2$).

Key indicators

Single-crystal synchrotron study
T = 150 K
Mean σ(a–O) = 0.014 Å
R factor = 0.035
wR factor = 0.088
Data-to-parameter ratio = 17.5

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

Comment

Compounds showing perovskite-related structures are of great interest as they show a range of properties, such as ionic conductivity, superconductivity, colossal magnetoresistance and catalytic properties (Cava, 2001; Ishihara *et al.*, 1994; Rao & Raveau, 1998). Mixed-anion compounds are not as common as oxides showing perovskite-related structures, but these

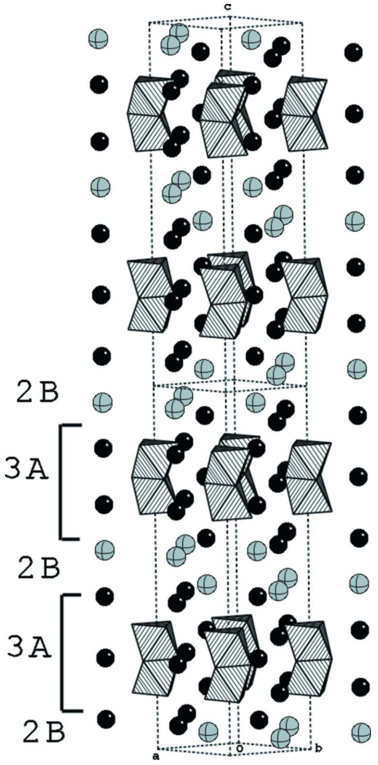

Figure 1
The structure of $Ba_5Ta_2Cl_2O_9$. The polyhedra represent the Ta_2O_9 dimers of face-shared TaO_6 octahedra. Grey and black spheres represent Cl^- and Ba^{2+}, respectively. The ten-layer stacking sequence, $(BAAAB)_2$, with B = BaCl and A = BaO_3, is indicated.

offer a means of tuning physical properties by variation of the anion composition.

We have now prepared the chloride oxide $Ba_5Ta_2Cl_2O_9$, which is isotypic with $Ba_5Ru_{1.6}W_{0.4}Cl_2O_9$ (Hong & Sleight, 1997) and with one polymorph of $Ba_5Ru_2Cl_2O_9$ (Tancret *et al.*, 2004). $Ba_5Ta_2Cl_2O_9$ is also structurally related to $Ba_5RuTaCl_2O_9$ (Wilkens & Müller-Buschbaum, 1991), which was refined in the non-centrosymmetric space group $P6_322$ due to differences in the O2 atomic coordinates. Although the reaction mixture contained Ca_3N_2, there was no evidence for the presence of Ca or N within the structure.

The structure of $Ba_5Ta_2Cl_2O_9$ is shown in Fig. 1 and can be described as a ten-layer stacking of BaO_3 and BaCl blocks along the *c* axis, in the sequence $(BAAAB)_2$, where $A = BaO_3$ and B = BaCl. The Ta cations occupy the octahedral sites created by the three BaO_3 layers, forming Ta_2O_9 dimers of distorted face-shared TaO_6 octahedra. Therefore, the structure can be described as an intergrowth of hexagonal perovskite blocks $(Ba_3Ta_2O_9)^{2-}$ and of NaCl-type double layers $(Ba_2Cl_2)^{2+}$.

The six symmetry-equivalent terminal Ta—O bonds are 1.873 (13) Å in length, while the remaining six Ta—O bond lengths in the dimer are considerably longer, at 2.139 (11) Å. Ba1 is twelve-coordinated by six O2 [2.949 (12) Å] and six O1 [2.996 (11) Å]. Ba2 is ten-coordinated by three O1 [2.734 (11) Å], six O2 [2.949 (12) Å] and one Cl [3.113 (6) Å]. Ba3 is seven-coordinated by three O2 [2.562 (11) Å], one Cl [3.159 (7) Å] and three additional Cl atoms [3.557 (2) Å].

Experimental

All operations had to be carried out under an inert atmosphere due to the extreme air- and moisture-sensitivity of the reagents. A mixture of polycrystalline $BaCl_2$, BaO and Ca_3N_2 (1:1:1 molar ratio) was placed in a crucible formed from tantalum metal foil and, subsequently, sealed inside a silica tube, with a valve head to allow the reaction to take place under flowing nitrogen. The mixture was heated for 36 h at 1273 K, then cooled to room temperature at 285 K h^{-1}. A large number of yellow and orange crystals were found to have grown on the Ta foil. None of the orange crystals proved suitable for X-ray diffraction. A yellow block-like crystal was analysed at station 16.2SMX of the UK Synchrotron Radiation Source, Daresbury Laboratory.

Crystal data

$Ba_5Ta_2Cl_2O_9$	$Z = 2$
$M_r = 1263.50$	Synchrotron radiation
Hexagonal, $P6_3/mmc$	$\lambda = 0.7848$ Å
$a = 5.9787$ (3) Å	$\mu = 27.18$ mm^{-1}
$c = 24.796$ (3) Å	$T = 150$ (2) K
$V = 767.59$ (10) Å^3	0.04 × 0.02 × 0.02 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	5409 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	351 independent reflections
$T_{min} = 0.337$, $T_{max} = 0.665$	317 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.093$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	20 parameters
$wR(F^2) = 0.088$	$\Delta\rho_{max} = 1.93$ e Å^{-3}
$S = 1.12$	$\Delta\rho_{min} = -2.88$ e Å^{-3}
351 reflections	

When refined with anisotropic displacement parameters, the O atoms gave physically unreasonable values. Therefore, the O atoms were refined with isotropic displacement parameters in the final model. The highest peak in the final difference Fourier map is located 0.67 Å from atom O2 and the deepest hole is located 1.46 Å from atom Ba1.

Data collection: *APEXII* (Bruker, 2004); cell refinement: *APEXII*; data reduction: *APEXII*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

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