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**Rb₄Zr₃Te₁₆, a one-dimensional zirconium telluride synthesized from molten salt**

ASHLEY B. ANDERSON, RU-JI WANG AND JING LI

**Synopsis**

The crystal structure of Rb₄Zr₃Te₁₆ consists of infinite one-dimensional chains of [Zr₃Te₁₆]₄⁻ separated from each other by Rb⁺ cations. Within these chains, each Zr atom is surrounded by eight Te atoms to give a distorted bicapped trigonal prism polyhedron.

**Subject index**

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Tetrarubidiumtrizirconiumhexadecatelluride

**Inorganic formula index**

Note that for coordination complexes, the ligands are listed in alphabetic order. This means that the indexing term may differ from the IUPAC formula used elsewhere in the paper.

Rb₄Te₁₆Zr₃
Acta Cryst. (1999). C55, 000–000

Rb₄Zr₃Te₁₆, a one-dimensional zirconium telluride synthesized from molten salt

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Abstract
A new ternary metal telluride, tetrarubidiumtrizirconiumhexadecatelluride, Rb₄Zr₃Te₁₆, has been synthesized through reactions at 698 K using elemental Zr and Rb₂Te/Te melt as a reactive flux, and characterized by single-crystal X-ray diffraction. Although the structure of this compound is very similar to its analogue, Cs₄Zr₃Te₁₆, they crystallize in different space groups, the former in $C_{2h}^6-C2/c$ and the latter in $C_{2h}^5-P2_1/n$. Both compounds consist of infinite one-dimensional chains of $[Zr_3Te_{16}]^{8-}$ separated from each other by Rb⁺ or Cs⁺ cations. Within the chain, each Zr atom is surrounded by eight Te atoms to give a distorted bicapped trigonal prism polyhedron. There are two unambiguous Te—Te single bonds of 2.758 (2) and 2.765 (2) Å and four longer Te···Te interatomic distances in the range of 2.928–3.045 Å that indicate weak interactions between the adjacent Te atoms. Because of the wide range of Te···Te interactions, simple formalisms cannot be used to describe the bonding within the chain.

Comment
The reactive flux technique has proved to be an effective method of preparing new ternary polychalcogenides. A series of compounds with the general formula $A_xM_yQ_z$ ($A$ = alkali metal, $M$ = Ti, Zr or Hf, and $Q$ = S, Se or Te) have been reported, such as K₄Ti₃S₁₄ (Sunshine et al., 1987), Na₂Ti₂Se₈ (Kang & Ibers, 1988), K₄M₄Te₁₇ ($M$ = Zr or Hf) (Keane & Ibers, 1991) and Cs₄Zr₃Te₁₆ (Cody & Ibers, 1994). A survey of the reactions of alkali-metal polychalcogenide molten salts to yield new materials with Ti, Cu, Au, Hg and Sn is given by Kanatzidis (1990). Although in general substitutions of elements in the same group lead to isostructural compounds, it is found that substitutions in ternary or quaternary chalcogenides containing group IV metals do not just involve simple replacement of one atom for another. For example, substitution of Na for K in the quaternary $A/Cu/Zr/Q$ ($A$ = alkali metal and $Q$ = S, Se or Te) system (Mansuetto et al., 1992, 1993) results
in subtle differences in structure, while substitution of Cs for K in the ternary system $A/M/Te$ ($A$ = alkali metal and $M$ = Zr or Hf) even leads to a change in composition from $K_4M_3Te_{17}$ ($M$ = Zr or Hf; Keane & Ibers, 1991) to $Cs_4Zr_3Te_{16}$ (Cody & Ibers, 1994). In the present work, the substitution of Rb for Cs in the above-mentioned ternary system gives the title new compound, $Rb_4Zr_3Te_{16}$, with the same composition but a different space group, $C_{2h}-C2/c$.

As shown in Fig. 1, the crystal structure of the title compound is very similar to that of $Cs_4Zr_3Te_{16}$ (space group $C_{2h}-P2_1/n$). Both crystals have similar cell parameters and contain one-dimensional Zr/Te chains extended along the $a$ direction and separated by alkali metal cations. The $M/Te$ chains of $K_4Hf_3Te_{17}$ (Keane & Ibers, 1991), $Cs_4Zr_3Te_{16}$ (Cody & Ibers, 1994) and $Rb_4Zr_3Te_{16}$ are compared in Fig. 2. With the higher symmetry, there are only two crystallographically unique Zr atoms in $Rb_4Zr_3Te_{16}$. One of them, Zr2, is located on a twofold axis (Wyckoff position 4$e$) and the other, Zr1, on a general position. Each Zr atom is eight-coordinate and at the center of a bicapped trigonal prism of Te atoms. The Zr—Te bond lengths are in the range 2.890 (2) to 3.079 (2) Å (Table 1), which are comparable with those found in $Cs_4Zr_3Te_{16}$ (Cody & Ibers, 1994). Each coordination polyhedron of a Zr atom shares opposite triangular faces with the adjacent Zr polyhedron to form a one-dimensional chain. Zr1 is bridged to Zr2 through atoms Te1, Te3$^\prime$ and Te5, while Zr1 is bridged to Zr1$^{ii}$ through atoms Te7, Te7$^{ii}$ and Te8$^{ii}$ [symmetry codes: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; (ii) $1-x$, $y$, $\frac{1}{2}+z$]. A Zr atom coordinated by eight Te atoms in a bicapped trigonal prism has been found not only in $A/Al/Te$ ($A$ = alkali metal) ternary systems but also in Zr/Te binary compounds, such as $ZrTe_3$ (Furuseth & Fjellveg, 1991) and ZrTe5 (Furuseth et al., 1973).

FIGS. 1 and 2

As is well known, the tellurides have a greater propensity than do the selenides or sulfides to exhibit $Q-Q$ interactions of intermediate-strength between a $Q-Q$ single bond and a $Q^2$ van der Waals type interaction (about 2.76 and 4.10 Å for Te, respectively; Shannon, 1976). While an arbitrary maximum for a Te—Te single bond of 2.94 Å gives $[Hf_2(Te_2)(Te_2)]^{3−}$ for the Hf/Te chain in $K_4Hf_3Te_{17}$, where each Hf is in the +4 oxidation state, it is somewhat difficult to describe the Te—Te interactions in the Zr/Te chains of $A_4Zr_3Te_{16}$ ($A$ = Cs and Rb) and to arrive at a reasonable formal oxidation state assignment for the elements. For Rb$_4$Zr$_3$Te$_{16}$, there are two unambiguous Te—Te single bonds with bond lengths of 2.758 (2) and 2.765 (2) Å and four somewhat longer Te—Te distances in the range of 2.928–3.045 Å, which indicates some weak interaction between adjacent Te atoms. The Te—
Te single bonds are shown in Fig. 2 as solid lines and other longer Te–Te interactions of 3.2 Å or less are shown as broken lines.

The obvious differences between the structures of Rb$_4$Zr$_3$Te$_{16}$ and Cs$_4$Zr$_3$Te$_{16}$ (Cody & Ibers, 1994) are the coordination environments of the cations. Two unique Rb$^+$ cations in the former exhibit coordination numbers 12 (Rb1) and 11 (Rb2), with Rb–Te distances from 3.614 (2) to 4.316 (3) Å, while the four Cs$^+$ cations in the latter exhibit coordination numbers 12, 11, 11 and 9, with Cs–Te distances ranging from 3.629 to 4.456 Å (Cody & Ibers, 1994).

Experimental

Rb$_2$Te was prepared by reactions of rubidium metal (99.5%; Aldrich Chemical Company) and elemental tellurium (99.8%; Strem Chemicals, Inc.) in a 2:1 ratio in liquid ammonia. Rb$_2$Te (0.0746 g, 0.25 mmol), Zr (98%; Aldrich Chemical Company; 0.0228 g, 0.25 mmol) and Te (0.1595 g, 1.25 mmol) were weighed in an inert argon-filled glove box. After thorough mixing the reactants were transferred to a thin-walled Pyrex reaction tube (9 mm outside diameter). The sample was immediately sealed under a vacuum of approximately 10$^{-3}$ torr (1 torr = 133.32 Pa). The reaction vessel was then placed in a furnace and brought up to 698 K within 8 h. After heating at 698 K for 3 d, the container was slowly cooled to 423 K (2 K h$^{-1}$) followed by natural cooling to room temperature. Upon removal from the furnace, the sample was treated by an isolation procedure. The reaction mixture consisted of the final products embedded in the excess alkali-metal polychalcogenide melt. The remaining flux was removed after several washes with $N,N$-dimethylformamide in a nitrogen atmosphere. The sample was then washed twice with 95% ethanol and dried with diethyl ether. Black prism-like crystals were isolated after this procedure. Microprobe analysis was performed on selected single crystals using a Jeol JXA-8600 Superprobe and gave an approximate elemental ratio of Rb, Zr and Te in agreement with the crystal data.

Crystal data

Rb$_4$Zr$_3$Te$_{16}$
$M_r$ = 2657.14
Monoclinic
$C2/c$

$a$ = 11.982 (2) Å
$b$ = 18.613 (4) Å
$c$ = 15.078 (3) Å
$\beta$ = 102.79 (3)$^\circ$
$V$ = 3279.3 (11) Å$^3$
$Z$ = 4
$D_r$ = 5.382 Mg m$^{-3}$
$D_m$ not measured

Mo K$\alpha$ radiation
$\lambda$ = 0.71073 Å
Cell parameters from 25 reflections
$\theta$ = 7.6–12.5$^\circ$
$\mu$ = 20.781 mm$^{-1}$
$T$ = 293 (2) K
Prism
0.12 × 0.10 × 0.10 mm
Black

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$\mu$ = 20.781 mm$^{-1}$
$T$ = 293 (2) K
Prism
0.12 × 0.10 × 0.10 mm
Black
Data collection

Enraf–Nonius CAD-4 diffractometer
ω-scan
Absorption correction: ψ-scan (North et al. 1968)

 Tmin = 0.094, Tmax = 0.126
2991 measured reflections
2874 independent reflections
2335 reflections with I > 2σ(I)

Refinement

Refinement on F2
R(F) = 0.041
wR(F2) = 0.086
S = 1.940
2874 reflections
106 parameters

w = 1/[σ2(F2) + (0.001P)2 + 5P]
where P = (F2 + 2F2)/3

(Δ/σ)max < 0.001

Table 1. Selected geometric parameters (Å, °)

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Symmetry codes: (i) -x, y, 1/2 - z; (ii) 1-x, y, 1/2 - z; (iii) 1+x, y, z; (iv) =x, -y, 1-z; (v) 1/2 +x, y, -1/2 - z; (vi) 1/2-x, y, 1/2 + z; (vii) 1-x, y, 1/2 - z; (viii) 1-x, -y, 1-z; (ix) 1/2 -x, y, 1/2 + z.

Direct phase determination yielded the positions of Rb, Zr and Te atoms, and all were subjected to anisotropic refinement. The largest residual electronic density peaks were located around Te and Zr atoms.

Financial support from the National Science Foundation (Grant DMR-9553066 and supplement) is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1265). Services for accessing these data are described at the back of the journal.

References


Fig. 1 The crystal structure of Rb$_4$Zr$_3$Te$_{16}$ along the $a$ direction, with double shaded circles for Rb, single shaded circles for Zr and open circles for Te atoms. The atoms are of arbitrary size.

Fig. 2 Comparison of the one-dimensional M/Te chains of K$_4$Hf$_3$Te$_{17}$ (top, A), Cs$_4$Zr$_3$Te$_{16}$ (middle, B) and Rb$_4$Zr$_3$Te$_{16}$ (bottom, C) with shaded circles for Zr and open circles for Te atoms, black lines for Te—Te single bonds and broken lines for longer Te···Te interactions of less than 3.2 Å. The atom-numbering scheme for Rb$_4$Zr$_3$Te$_{16}$ is given; symmetry codes are as in Table 1.

ASHLEY B. ANDERSON, RU-JI WANG AND JING LI

Rb$_4$Zr$_3$Te$_{16}$

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Rb$_4$Zr$_3$Te$_{16}$

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Rb$_4$Zr$_3$Te$_{16}$
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The data shown below are not normally printed in *Acta Cryst. Section C* but the data will be available electronically via the online contents pages of the journal at http://www.iucr.org/journals/acta/tocs/actac/actac.html

Table S1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

\[ U_{eq} = \left( \frac{1}{3} \sum_j U_{ij} a_i a_j \right)^{1/2} \]

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