bonding environment of the 2,4,6-(t-Bu)$_3$C$_6$H$_2$P moiety in I is anticipated.

This work, together with earlier studies in our laboratory,[9] and elsewhere,[13,14] suggests that the reactivity of the Na$^+$(THF)-Li$^+$[2-(SiMe$_3$)-3-(R)-2,3-C$_2$B$_4$H$_4$]$^{2-}$ double salts (R = SiMe$_3$, Me, or H) toward a variety of dihalo compounds of the main-group elements and transition metals should lead to several new carborane compounds. Efforts to incorporate other group 15 elements into the C$_2$B$_4$ carborane system are currently underway in our laboratories.

Acknowledgment. This work was supported by grants from the National Science Foundation (Grant (CHE-8800328 to N.S.H. and Grant CHE-9005967 to A.H.C.), the Robert A. Welch Foundation (Grant N-1016 to N.S.H. and Grant F-135 to A. H.C.), and the donors of the Petroleum Research Fund, administered by the American Chemical Society.

---

(13) Davis, J. H., Jr.; Sinn, E.; Grimes, R. N. *J. Am. Chem. Soc.* **1989**, *111*, 4776.

(14) Beck, J. S.; Sneddon, L. G. *Inorg. Chem.* **1990**, *29*, 295.

Contribution from the Department of Chemistry,
Northwestern University, Evanston, Illinois 60208

Synthesis of K$_4$M$_3$Te$_{17}$ (M = Zr, Hf) and the Structure of K$_4$Hf$_3$Te$_{17}$, a New One-Dimensional Solid-State Ternary Polytelluride

Patricia M. Keane and James A. Ibers*

Received September 26, 1990

The compounds K$_4$Zr$_3$Te$_{17}$ and K$_4$Hf$_3$Te$_{17}$ have been synthesized through the reaction at 900 °C of Zr or Hf with a K$_2$Te/Te melt as a reactive flux. The compounds crystallize in space group C^5_{2h}-$P2_1/c$ of the monoclinic system with four formula units in cells: a = 10.148 (6) Å, b = 28.889 (17) Å, c = 11.626 (7) Å, β = 115.21 (2)° (T = 107 K) for K$_4$Hf$_3$Te$_{17}$; a = 10.146(2) Å, b = 29.98 (1) Å, c = 11.669 (4) Å, β = 115.01 (3)° (T = 153 K) for K$_4$Zr$_3$Te$_{17}$. The structure of K$_4$Hf$_3$Te$_{17}$ has been determined from single-crystal X-ray data. The structure comprises infinite, one-dimensional chains of Hf-centered polyhedra that extend along [101] and are separated from each other by K$^+$ ions. Each Hf atom is eight-coordinate. If the Te–Te maximum bond length is taken to be 2.94 Å, then there are six Te$_2$$^{2-}$ ligands, one μ_2-η^1-Te$_2$$^{2-}$ ligand, and a μ-Te$_3$$^{2-}$ ligand. The composition of the infinite chain is $^1_\infty$[Hf$_3$(Te$_3$)(Te$_2$)$_7$$^{4-}$] with the Hf atoms present in the +4 oxidation state.

Introduction

Recently, the use of fluxes or molten salts in the synthesis of new ternary polychalcogenides was described.[1] This technique exploits molten salts of the type A$_2$Q/Q (A = alkali metal, Q = chalcogenide (S, Se, Te)) not only as a flux or crystallizing agent but also as a reactant. The resultant compounds typically exhibit unusual chalcogen–chalcogen bonding and novel structure types. Although there have been several ternary sulfides[1-3] and selenides[2,4,5] reported that make use of this synthetic procedure, no tellurides have been synthesized. In general, the well-characterized intercalation compounds of the type A$_x$M$_y$Q$_z$ (M = group IV–VI metal; Q = S, Se, Te) are pervasive in this system.[6,7] The extreme thermodynamic stability of the binary metal tellurides MTe$_3$[8] and MTe$_5$[9] (M = Zr, Hf) hinders the formation of the new ternary polytellurides. We demonstrate here that through utilization of a K$_2$Te/Te flux, the technique, as predicted,[1] can be extended to the tellurides.

Experimental Section

In order to facilitate the reaction, elemental K (Alfa, 99%) and elemental Te (AESAR, 99.5%) in the stoichiometric ratio of 1:2 were prereacted at 650 °C for 3 days. In a drybox under an Ar atmosphere, 0.157 g of this mixture, 0.025 g of Hf powder (AESAR, 99.6%), and 0.068 g of Te powder were ground together and then loaded into a silica tube. The tube was subsequently evacuated (~10^{-4} Torr) and sealed. It was heated at 650 °C for 6 days and then ramped to 900 °C to heat for 4 days. The tube was then cooled at a rate of 3 °C/h to 450 °C and then to room temperature at 90 °C/h. The product contained air-stable, dull black needle-shaped crystals at the surface and within the melt. Single crystals of what proved to be K$_4$Hf$_3$Te$_{17}$ suitable for X-ray diffraction studies were manually extracted from the melt. K$_4$Zr$_3$Te$_{17}$ was prepared by the same route with 0.165 g of the K/Te mixture, 0.0135 g of Zr powder (AESAR, 99%), and 0.071 g of Te powder. Similarly, the product consisted of dull black needles within the melt.

Table I. Data Collection and Refinement Details for K$_4$Hf$_3$Te$_{17}$

formula	K$_4$Hf$_3$Te$_{17}$	density (calcd), g cm^{-3}	5.96
fw	2861.06		
space group	C^5_{2h}-$P2_1/c$	radiation	graphite-monochromated Mo Kα (λ(Kα_1) = 0.7093 Å)
a, Å	10.148 (6)		
b, Å	29.889 (17)	linear abs coeff, cm^{-1}	225
c, Å	11.626 (7)		
β, deg	115.21 (2)	transm factors	0.43–0.52[b]
vol, Å^3	3190 (1)	$R_w(F_o)$	0.094
Z	4	$R(F)$ for $F_o^2 > 3\sigma(F_o^2)$	0.084
T, K	107[a]		

[a] The low-temperature system is based on a design by: Huffman, J. C. Ph.D. Thesis, Indiana University, 1974. [b] The analytical method was used for the absorption correction (de Meulenaer, J.; Tompa, H. *Acta Crystallogr.* **1965**, *19*, 1014–1018).

Analysis of these compounds with an EDAX-equipped Hitachi S570 scanning electron microscope confirmed the presence of (K, Hf, Te) and (K, Zr, Te) in the approximate ratio of (3.8:3.0:14.4). The exact composition of the Hf compound was established from the X-ray structure determination.

X-ray Structure Analysis

K$_4$Zr$_3$Te$_{17}$. The cell parameters (a = 10.146 (2), b = 29.98 (1), c = 11.669 (4) Å; β = 115.01 (3)°) of a single crystal of K$_4$Zr$_3$Te$_{17}$ were determined by a least-squares analysis of 25 reflections centered on an Enraf-Nonius CAD4 diffractometer at 153 K. Systematic absences were

---

(1) Sunshine, S. A.; Kang, D.; Ibers, J. A. *J. Am. Chem. Soc.* **1987**, *109*, 6202–6204.

(2) Schreiner, S.; Aleandri, L. E.; Kang, D.; Ibers, J. A. *Inorg. Chem.* **1989**, *28*, 392–393.

(3) Kanatzidis, M. G.; Park, Y. J. *J. Am. Chem. Soc.* **1989**, *111*, 3767–3769.

(4) Kang, D.; Ibers, J. A. *Inorg. Chem.* **1988**, *27*, 549–551.

(5) Park, Y.; Kanatzidis, M. G. *Angew. Chem., Int. Ed. Engl.* **1990**, *29*, 914–915.

(6) Lēvy, F. *Crystallography and Crystal Chemistry of Materials with Layered Structures*; Reidel Publishing Co.: Dordrecht, Holland/Boston, MA, 1976; pp 93–127.

(7) Huan, G.; Greenblatt, M. *Mater. Res. Bull.* **1987**, *22*, 505–512, 943–949.

(8) Brattås, L.; Kjekshus, A. *Acta Chem. Scand.* **1972**, *26*, 3441–3449.

(9) Furuseth, S.; Brattås, L.; Kjekshus, A. *Acta Chem. Scand.* **1973**, *27*, 2367–2374.

0020-1669/91/1330-1327$02.50/0

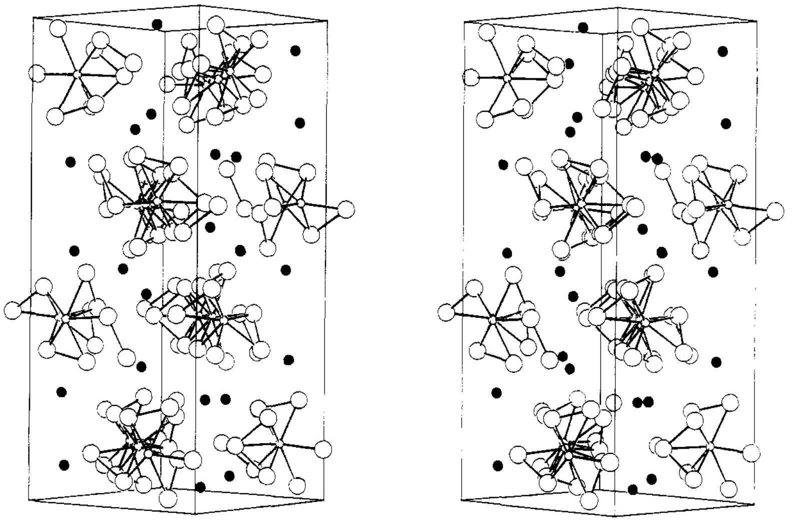

Figure 1. Unit cell of $K_4Hf_3Te_{17}$. The small open circles are Hf^{4+} ions; the medium solid circles are K^+ ions; the large open circles are Te atoms. With the origin at the bottom left corner, the *b* axis is the long axis and the *c* axis extends out of the plane of the paper toward the viewer.

Table II. Positional and Isotropic Thermal Parameters ($Å^2$) for $K_4Hf_3Te_{17}$

atom	*x*	*y*	*z*	B^a
Hf(1)	0.786 66 (26)	0.618 991 (76)	0.993 61 (23)	1.02 (4)
Hf(2)	0.447 62 (26)	0.616 674 (77)	0.663 63 (23)	1.07 (4)
Hf(3)	0.117 41 (28)	0.618 173 (83)	0.324 15 (25)	1.47 (4)
Te(1)	1.025 26 (38)	0.555 12 (11)	1.103 15 (33)	0.62 (5)
Te(2)	1.013 26 (38)	0.684 65 (10)	1.118 74 (33)	0.62 (5)
Te(3)	0.798 56 (38)	0.597 63 (11)	1.243 00 (33)	0.63 (5)
Te(4)	0.992 39 (38)	0.606 65 (10)	0.877 74 (34)	0.66 (6)
Te(5)	0.622 23 (39)	0.679 06 (11)	1.085 83 (35)	0.80 (6)
Te(6)	0.695 64 (38)	0.552 76 (10)	0.782 05 (33)	0.59 (5)
Te(7)	0.690 42 (38)	0.679 57 (11)	0.766 96 (33)	0.60 (5)
Te(8)	0.474 69 (38)	0.607 47 (10)	0.927 47 (33)	0.60 (5)
Te(9)	0.406 46 (39)	0.522 13 (11)	0.718 30 (34)	0.71 (6)
Te(10)	0.398 95 (39)	0.709 59 (11)	0.709 91 (34)	0.67 (6)
Te(11)	0.384 41 (38)	0.558 13 (11)	0.446 25 (33)	0.67 (6)
Te(12)	0.378 26 (39)	0.677 73 (11)	0.448 87 (34)	0.72 (6)
Te(13)	0.130 42 (37)	0.611 74 (11)	0.587 60 (33)	0.65 (5)
Te(14)	0.382 72 (40)	0.616 79 (12)	0.255 98 (35)	1.05 (6)
Te(15)	0.055 29 (40)	0.695 32 (11)	0.640 31 (35)	0.91 (6)
Te(16)	−0.002 68 (40)	0.526 02 (11)	0.341 71 (34)	0.77 (6)
Te(17)	−0.008 47 (40)	0.707 42 (11)	0.344 94 (35)	0.71 (6)
K(1)	0.731 5 (15)	0.480 65 (40)	1.038 9 (13)	1.5 (2)
K(2)	0.695 2 (15)	0.720 33 (43)	0.435 3 (13)	1.9 (2)
K(3)	0.244 1 (13)	0.704 00 (37)	0.966 8 (11)	1.1 (2)
K(4)	0.228 2 (15)	0.430 48 (42)	0.478 3 (13)	1.7 (2)

[a] $B = 8\pi^2\langle u^2\rangle$, where $\langle u^2\rangle$ is the mean-square displacement.

consistent with the space group $P2_1/c$.

$K_4Hf_3Te_{17}$. Preliminary studies established monoclinic symmetry, and the cell constants were determined from 16 reflections centered on a Picker FACS-1 diffractometer. Intensity data of 6459 unique reflections were recorded. The refined cell constants and some experimental details are listed in Table I; more information is available in Table IS (supplementary material). Six standards from diverse regions of reciprocal space were monitored every 100 reflections and exhibited no significant variation in intensity throughout data collection.

All calculations were performed on a Stellar GS2000 computer with programs standard to this laboratory.[10] Conventional atomic scattering factors were used. Analysis of the data for systematic absences indicated space group $P2_1/c$. The initial positions for the K, Hf, and Te atoms were determined with the direct-methods program SHELX-86.[11] A suitable model was difficult to find owing to the unusual nature of the structure, including the uncommon bonding modes of atoms Te(14) and Te(15) and the intense dependence of the *R* index on these two atoms. The structure was refined in a straightforward manner. Thermal motion was kept isotropic. The final refinement on F_o^2 involved 97 variables and 6459

Table III. Selected Distances (Å) and Angles (deg) in $K_4Hf_3Te_{17}$

Hf(1)–Te(1)	2.916 (4)	K(1)···Te(8)	3.49 (1)
Hf(1)–Te(2)	2.900 (4)	K(1)···Te(1)	3.54 (1)
Hf(1)–Te(3)	2.921 (4)	K(1)···Te(6)	3.57 (1)
Hf(1)–Te(4)	2.950 (4)	K(1)···Te(4)	3.64 (1)
Hf(1)–Te(5)	2.946 (4)	K(1)···Te(9)	3.65 (1)
Hf(1)–Te(6)	2.981 (4)	K(1)···Te(16)	3.66 (1)
Hf(1)–Te(7)	2.997 (4)	K(1)···Te(1)	3.67 (1)
Hf(1)–Te(8)	2.943 (4)	K(1)···Te(9)	3.98 (1)
Hf(2)–Te(6)	2.988 (4)	K(2)···Te(15)	3.49 (1)
Hf(2)–Te(7)	2.920 (4)	K(2)···Te(12)	3.53 (1)
Hf(2)–Te(8)	2.974 (4)	K(2)···Te(7)	3.56 (1)
Hf(2)–Te(9)	2.965 (4)	K(2)···Te(17)	3.60 (1)
Hf(2)–Te(10)	2.911 (4)	K(2)···Te(10)	3.69 (1)
Hf(2)–Te(11)	2.912 (4)	K(2)···Te(5)	3.71 (1)
Hf(2)–Te(12)	2.926 (4)	K(2)···Te(5)	4.00 (1)
Hf(2)–Te(13)	2.957 (4)	K(2)···Te(7)	4.06 (1)
Hf(3)–Te(11)	3.050 (4)	K(3)···Te(15)	3.46 (1)
Hf(3)–Te(12)	3.002 (4)	K(3)···Te(2)	3.53 (1)
Hf(3)–Te(13)	3.014 (4)	K(3)···Te(17)	3.53 (1)
Hf(3)–Te(16)	3.054 (4)	K(3)···Te(5)	3.56 (1)
Hf(3)–Te(17)	3.011 (4)	K(3)···Te(10)	3.65 (1)
Hf(3)–Te(1)	2.996 (4)	K(3)···Te(4)	3.72 (1)
Hf(3)–Te(2)	2.936 (4)	K(3)···Te(12)	3.83 (1)
Hf(3)–Te(3)	3.023 (5)	K(3)···Te(8)	3.86 (1)
Te(1)–Te(4)	2.933 (4)	K(3)···Te(10)	3.93 (1)
Te(2)–Te(17)	2.814 (5)	K(3)···Te(14)	4.01 (1)
Te(3)–Te(16)	2.828 (5)	K(4)···Te(6)	3.46 (1)
Te(5)–Te(8)	2.804 (5)	K(4)···Te(3)	3.47 (1)
Te(6)–Te(9)	2.854 (5)	K(4)···Te(16)	3.61 (1)
Te(7)–Te(10)	2.885 (5)	K(4)···Te(13)	3.61 (1)
Te(11)–Te(14)	2.816 (5)	K(4)···Te(11)	3.66 (1)
Te(12)–Te(14)	2.904 (5)	K(4)···Te(9)	3.78 (1)
Te(13)–Te(15)	2.756 (5)	K(4)···Te(16)	3.92 (1)
Hf(1)–Hf(2)	3.914 (4)	K(4)···Te(4)	3.94 (1)
Hf(1)–Hf(3)	3.879 (4)	K(4)···Te(14)	4.09 (1)
Hf(2)–Hf(3)	3.942 (4)		

Hf(2)–Hf(1)–Hf(3)	178.19 (8)	Te(11)–Hf(2)–Te(12)	75.5 (1)
Hf(3)–Hf(2)–Hf(1)	177.05 (9)	Te(12)–Hf(2)–Te(13)	86.6 (1)
Hf(1)–Hf(3)–Hf(2)	178.71 (8)	Te(11)–Hf(2)–Te(13)	84.3 (1)
Te(1)–Hf(1)–Te(2)	83.7 (1)	Te(10)–Hf(2)–Te(9)	145.0 (1)
Te(2)–Hf(1)–Te(3)	87.8 (1)	Te(10)–Hf(2)–Te(7)	59.3 (1)
Te(1)–Hf(1)–Te(3)	75.4 (1)	Te(10)–Hf(2)–Te(6)	134.5 (1)
Te(6)–Hf(1)–Te(7)	78.8 (1)	Te(9)–Hf(2)–Te(8)	69.5 (1)
Te(7)–Hf(1)–Te(8)	85.9 (1)	Te(11)–Hf(3)–Te(12)	72.4 (1)
Te(6)–Hf(1)–Te(8)	77.6 (1)	Te(12)–Hf(3)–Te(13)	84.2 (1)
Te(5)–Hf(1)–Te(7)	85.9 (1)	Te(11)–Hf(3)–Te(13)	81.0 (1)
Te(5)–Hf(1)–Te(6)	132.9 (1)	Te(1)–Hf(3)–Te(2)	81.7 (1)
Te(4)–Hf(1)–Te(6)	64.6 (1)	Te(2)–Hf(3)–Te(3)	85.2 (1)
Te(4)–Hf(1)–Te(7)	72.7 (1)	Te(1)–Hf(3)–Te(3)	72.7 (1)
Te(4)–Hf(1)–Te(8)	139.3 (1)	Te(17)–Hf(3)–Te(3)	76.7 (1)
Te(6)–Hf(2)–Te(7)	78.9 (1)	Te(17)–Hf(3)–Te(1)	129.6 (1)
Te(7)–Hf(2)–Te(8)	86.8 (1)	Te(17)–Hf(3)–Te(12)	75.7 (1)
Te(6)–Hf(2)–Te(8)	77.0 (1)		

---

(10) Waters, J. M.; Ibers, J. A. *Inorg. Chem.* **1977**, *16*, 3273–3277.

(11) Sheldrick, G. M. In *Crystallographic Computing 3*; Sheldrick, G. M., Kruger, C., Goddard, R., Eds.; Oxford University Press: London/New York, 1985; pp 175–189.

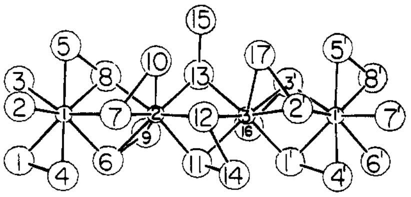

Figure 2. The ${}^{1}_{\infty}[\mathrm{Hf_3(Te_3)(Te_2)_7^{4-}}]$ chain in $K_4Hf_3Te_{17}$. The large circles are Te atoms; the small are circles Hf atoms. Atoms labeled with primed numbers are related by translation.

unique data, including those for which $F_o^2 \leq 0$. The final difference electron density map showed no peak heights greater than 25% of a K atom. There are no unusual trends in an analysis of $\sum w(F_o^2 - F_c^2)^2$ as a function of F_o^2, setting angles, or Miller indices. Final values of the positional parameters and isotropic thermal parameters are presented in Table II.

Results and Discussion

A stereoscopic view of the $K_4Hf_3Te_{17}$ structure is shown in Figure 1, and important distances and angles are given in Table III. The structure consists of one-dimensional Hf/Te chains that extend along the [101] direction. The K^+ cations that separate these chains exhibit asymmetric coordination: atoms K(1) and K(2) are coordinated by eight Te atoms, atom K(3) by ten Te atoms, and atom K(4) by nine Te atoms. K···Te distances range from 3.457 (1) to 4.092 (1) Å.

The tellurides have a greater propensity than do the selenides or sulfides to exhibit Q–Q interactions intermediate between a Q–Q single bond and a $Q^{2-}\cdots Q^{2-}$ interaction (about 2.76[8] and 4.10 Å[12] for Te). MTe_3[9] (M = Zr, Hf), $CuTe$,[13] UTe_2,[14] Rb_2Te_5,[15] and Cs_2Te_5[16] are just a few of many compounds that exhibit these and intermediate Te–Te distances. But, even in the selenides, intermediate Se–Se bonding is found, for example in Nb_2Se_9,[17,18] where there are four Se–Se single bonds 2.316 (2), 2.319 (2), 2.364 (2), and 2.365 (2) Å in length and also two longer Se–Se bonds at 2.643 (2) and 2.663 (2) Å.

In the $K_4Hf_3Te_{17}$ structure, which contains 17 crystallographically independent Te atoms, there are a multitude of Te–Te interactions within the 2.76–4.10 Å range. What length to take as a maximum for a Te–Te single bond is somewhat arbitrary. Hence, one must refer to descriptions of similar structures as well as consider credible oxidation states and coordination geometries of the metal atoms. In the $K_4Hf_3Te_{17}$ structure, if a Te–Te single-bond cutoff were taken to be 3.26 Å, then the compound would contain the unlikely Te_{14}^{2-} ring composed of atoms Te(13), Te(15), Te(10), Te(12), Te(14), Te(11), Te(9), Te(6), Te(4), Te(1), Te(16), Te(3), Te(5), and Te(6), the formulation of the compound would be $K_4Hf_3(Te_{14})(Te_2)(Te)$, and the average oxidation state of Hf would be $+{}^{2}/_{3}$! In the description that follows, we take the maximum Te–Te single bond length to be 2.94 Å. Then the Hf/Te chain can be considered to contain one Te_3^{2-} and seven Te_2^{2-} ligands. Figure 2 shows the one-dimensional chain with the appropriate Te–Te bonding modes and the numbering scheme. The composition of the chain is ${}^{1}_{\infty}[\mathrm{Hf_3(Te_3)(Te_2)_7^{4-}}]$, with each of the three Hf atoms in the +4 oxidation state. There are two unique types of Te_2^{2-} ligands and one Te_3^{2-} ligand. A μ_2-η^1-Te_2^{2-} ligand bridges atoms Hf(2) and Hf(3) through atom Te(13) and contains the terminal atom Te(15). The remaining six Te_2^{2-} ligands are composed of one Te atom that is shared by two neighboring Hf atoms and a second Te atom that is bonded to only one Hf atom. The Te_3^{2-} ligand bridges atoms Hf(2) and Hf(3) through the two terminal atoms Te(11) and Te(12).

The structure contains three crystallographically distinct Hf atoms that are bridged together in a similar way. Atom Hf(1) is bridged to atom Hf(2) through atoms Te(6), Te(7), and Te(8); atom Hf(2) is bridged to atom Hf(3) through atoms Te(11), Te(12), and Te(13); and atom Hf(3′) is bridged to atom Hf(1) through atoms Te(1), Te(2), and Te(3). If we take the maximum Hf–Te distance as 3.06 Å, then all three atoms are eight-coordinate with Hf–Te distances within a narrow range of 2.900 (4)–3.054 (4) Å.

The $K_4Hf_3Te_{17}$ structure bears some resemblance to that of $HfTe_5$.[8] In both structures, the Hf atoms are eight-coordinate. The Te–Te bond distances are very similar, ranging from 2.763 (4) to 2.908 (3) Å in $HfTe_5$ and from 2.756 (5) to 2.933 (5) Å in $K_4Hf_3Te_{17}$. Similarly, the Hf–Te distances (2.954 (3)–2.960 (2) Å) in $HfTe_5$ resemble those in $K_4Hf_3Te_{17}$ (2.900 (4)–3.054 (4) Å). In the $HfTe_5$ structure the Hf atom is at the center of a distorted bicapped trigonal prism, one edge of which contains a full Te–Te single bond of 2.763 (4) Å. These polyhedra are linked together by zigzag Te chains to form a basic bicapped trigonal-prismatic framework that extends along the [100] direction. In the ${}^{1}_{\infty}[\mathrm{Hf_3(Te_3)(Te_2)_7^{4-}}]$ chain, atoms Te(1), Te(2), and Te(3), atoms Te(6), Te(7), and Te(8), and atoms Te(11), Te(12), and Te(13) make up a trigonal-prismatic skeleton with "capping" atoms of Te(4), Te(5), Te(9), Te(10), Te(16), and Te(17). In the ternary structure, the Hf centers are nearly collinear and the trigonal prisms are slightly distorted.

While reactive metal polychalcogenide fluxes are certainly useful and may indeed be necessary for the preparation of low-dimensional compounds incorporating polychalcogenide ligands, the use of low temperatures is not. The present compounds, as well as $K_4Ta_2S_{11}$[2] and $K_3CuNb_2Se_{12}$,[19] are representative of low-dimensional polychalcogenide compounds prepared at 800 °C or higher. Nor are low temperatures necessary for the formation of Q_n^{2-} ligands with $n > 2$, although this has been suggested.[20] The present compounds as well as Nb_2Se_9[17,18] contain such ligands (Te_3^{2-} and Se_5^{2-}) and were prepared at high temperatures.

The extension of the reactive flux method to tellurides greatly increases the potential applicability of the technique.

Acknowledgment. This research was supported by the U.S. National Science Foundation through Grant DMR88-13623. Use was made of the SEM facilities of the Materials Research Center at Northwestern University (U.S. National Science Foundation Grant DMR88-21571).

Supplementary Material Available: For $K_4Hf_3Te_{17}$ complete crystallographic details (Table IS) (2 pages); a listing of observed and calculated structure amplitudes (25 pages). Ordering information is given on any current masthead page.

---

(12) Shannon, R. D. *Acta Crystallogr.* **1976**, *A32*, 751–767.
(13) Hulliger, F. *Phys. Chem. Mater. Layered Struct.* **1976**, *5*, 134.
(14) Haneveld, A. J. K.; Jellinek, F. *J. Less-Common Met.* **1970**, *21*, 45–49.
(15) Böttcher, R.; Kretschmann, U. *J. Less-Common Met.* **1983**, 95, 81–91.
(16) Böttcher, R.; Kretschmann, U. *Z. Anorg. Allg. Chem.* **1982**, 491, 39–46.
(17) Meerschaut, A.; Guémas, L.; Berger, R.; Rouxel, J. *Acta Crystallogr.* **1979**, *B35*, 1747–1750.
(18) Sunshine, S. A.; Ibers, J. A. *Acta Crystallogr.* **1987**, *C43*, 1019–1022.
(19) Lu, Y. J.; Ibers, J. A. Submitted for publication in *Inorg. Chem.*
(20) Kanstzidis, M. G. *Chem. Mater.* **1990**, *2*, 353–363.