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Treating $[K_4(H_2O)_{0.5}][SnTe_4]$ with PPh_4^+ or $AsPh_4^+$: Formation of a Highly Air-Stable Tellurostannate Salt, a Tellurolate with One-Coordinate Te Atoms, and a Polytelluride Salt with ${}^{1}_{\infty}[Te_4(H_2O)_2]^{2-}$ Chains

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Dedicated to Professor Dieter Fenske on the occasion of his 65th birthday

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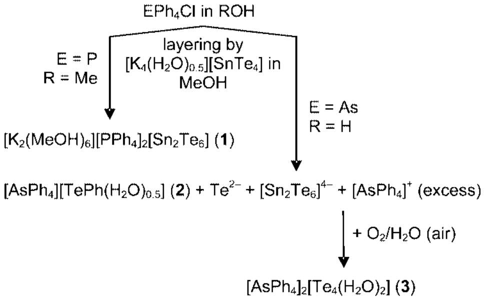
In reactions of potassium orthotellurostannates with EPh_4Cl (E = P, As), different single-crystalline products are obtained. An air-stable dimeric tellurostannate forms by partial replacement of strongly interacting K^+ cations by weakly interacting or noninteracting $[PPh_4]^+$ ions. For E = As, an equally air-stable tellurophenolate, in which Te has the rare coordination number 1, results upon As–C bond cleavage at a tetraphenylarsonium cation. Oxidation of the air-sensitive $[Sn_2Te_6]^{4-}$ anion and Te^{2-} by O_2 (air) affords a tetratelluride salt with strands of H_2O-bridged $[Te_4]^{2-}$ anions.

Introduction

The influence of counterions on both structure and reactivity of complex anions and anionic networks is subtle and therefore actively investigated.[1] Ion-exchange reactions in solution may lead to either crystallization of the anionic structure with another cation or the crystallization of another anionic species than originally used due to a lower solubility of further co-existing components of the equilibrium in solution, or even upon a chemical reaction. Being interested in the effect of bulky cations on the properties of chalcogenostannate anions, we have performed reactions of the highly reactive potassium salt of $[SnTe_4]^{4-}$ with EPh_4Cl (E = P, As). For E = P, the phosphonium salt of dimeric $[Sn_2Te_6]^{4-}$ crystallized – similar to observations that have been made in the presence of NR_4^+ cations.[2] However, this salt shows an unexpected air-stability which is unique for tellurostannate compounds. A completely different behavior was observed for E = As, where the only crystalline product evinced to be a tellurolate salt with Te in its rare coordination number 1. If air is allowed to diffuse into the reaction mixture, the tellurolate salt remains unaffected, whereas unreacted telluride species are oxidized to form a novel tetratelluride compound.

Results and Discussion

$[K_2(MeOH)_6][PPh_4]_2[Sn_2Te_6]$ (**1**), $[AsPh_4][TePh(H_2O)_{0.5}]$-(**2**), and $[AsPh_4]_2[Te_4(H_2O)_2]$ (**3**) were obtained by reactions of $[K_4(H_2O)_{0.5}][SnTe_4]$[3] with PPh_4Cl or $AsPh_4Cl$ (Scheme 1). All compounds have been structurally characterized by means of single-crystal X-ray diffraction.[4,5] Table 1 summarizes the crystallographic data. The anion of compound **1** is the product of a dimerization reaction [Equation (1)] that is based on an aggregation equilibrium [Equation (2)], which is known to occur in protic solutions of $[SnQ_4]^{4-}$ anions (Q = S, Se, Te).[6] However, the reported shift of the respective equilibrium when decreasing the pH value upon addition of acid or dilution of the solution cannot be the reason for the crystallization of the dimeric anion here, since the addition of PPh_4Cl does not affect the pH value. Thus, the observation is rather the result of better crystallization due to better matching sizes of cations and anions when the anionic diameter is increased. Figure 1 shows fragments of the crystal packing in **1**.



Scheme 1. Syntheses of compounds **1**, **2**, and **3**.

The asymmetric unit in **1** comprises half a formula unit, i.e. one half of the hexatellurostannate dimer, “$SnTe_3$”, one potassium cation, one tetraphenylphosphonium cation, and

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$$2\,[SnTe_4]^{4-} + 2\,[PPh_4]^+ + 6\,MeOH \rightarrow [K_2(MeOH)_6][PPh_4]_2[Sn_2Te_6]\ (\mathbf{1}) + 2\,Te^{2-} \tag{1}$$

$$2\,[SnQ_4]^{4-} \rightleftarrows [Sn_2Q_6]^{4-} + 2\,Q^{2-}\ (Q = S, Se, Te) \tag{2}$$

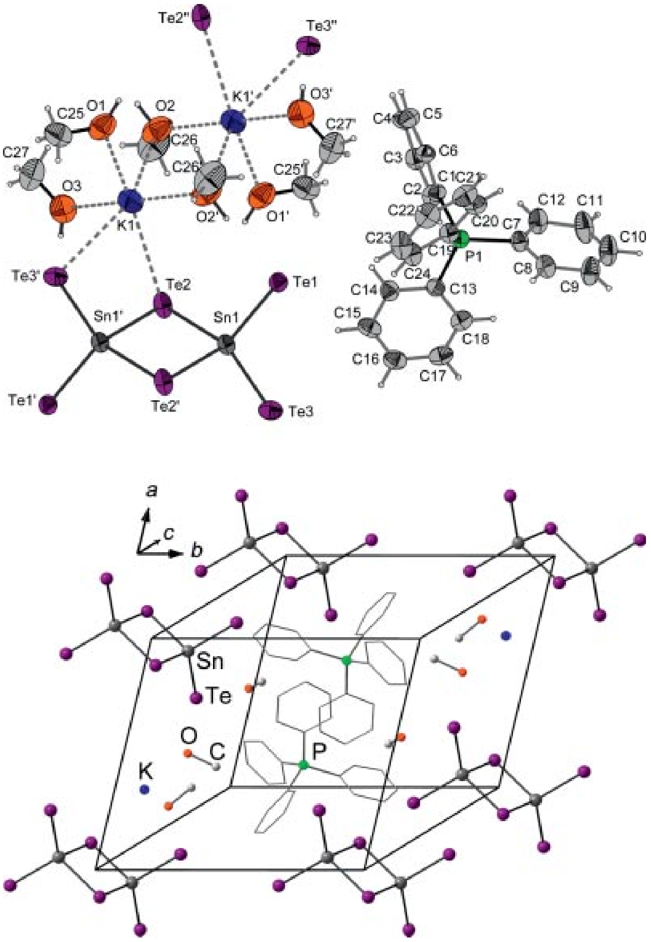

Figure 1. Top: relative orientation of the tetraphenylphosphonium cation, potassium cations within their coordination sphere, and an $[Sn_2Te_6]^{4-}$ anion in **1**. Thermal ellipsoids are drawn to 50% probability. Selected interatomic distances [Å] and angles [°]: Sn–Te$_t$ 2.682(1)–2.806(1), Sn–(μ-Te) 2.794(8)–2.806(1), Sn1···Sn1′ 3.799(1), P–C 1.783(8)–1.793(9), Te···(H–)O 3.481(6)–3.578(10), Te···K 3.383(4)–3.766(4), Te···H(–C) 3.147(5)–3.577(2) K···O$_t$ 2.753(11)–2.789(11), K···(μ-O) 2.670(13)–2.728(11); Te$_t$–Sn–μ-Te 108.41(3)–115.05(3), Te1–Sn–Te3 114.37(3), Te2–Sn–Te2 94.55(3), C–P–C 109.5(3). The subscript "t" denotes terminal atoms. Bottom: fragment of the crystal packing in **1**. H atoms are omitted for clarity.

three MeOH molecules. The latter coordinate to K$^+$ ions to form a coordination aggregate of two edge-sharing octahedra, $[(MeOH)_2Te_2K(\mu\text{-}MeOH)]_2$, the Te donor atoms arising from two adjacent $[Sn_2Te_6]^{4-}$ anions that are linked this way. In addition, all MeOH molecules form hydrogen bonds to terminal tellurium atoms. PPh_4^+ cations do not seem to interact significantly with any of the further components. The shortest interatomic distance Te···H–C around the phosphonium group is 3.147(5) Å. Compound **1** is unexpectedly air-stable: although the starting compound, $[K_4(H_2O)_{0.5}][SnTe_4]$, decomposes within seconds upon contact with air, **1** remains unchanged for several days at room temperature. This is in contrast to the air-sensitivity of recently reported $[Mn(en)_3]_2[Sn_2Te_6]\cdot n H_2O$[7–9] and $[K_2(2,2,2\text{-crypt-}K)_2][Sn_2Te_6]$.[10] Although all of these salts contain bulky cations, the noninteracting PPh_4^+ ion seems to protect the $[Sn_2Te_6]^{4-}$ groups most effectively. This might be due to its own distinct indifference toward air, i.e. its insensitivity toward hydration or oxidation.

The synthesis of compound **2** provides feasible access to the very rare tellurophenolate anion, that could so far only be obtained by using a multistage synthesis in liquid ammonia:[11] free telluride anions, which were obtained according to Equation (2), combine with a phenyl rest of a tetraphenylarsonium cation [Equation (3)]. This observation is in agreement with similar bond strengths of As–C bonds (E_{Diss} = 252 kJ/mol)[12a] and Te–C bonds (E_{Diss} ≈ 250 kJ/mol)[12b,c] in comparison with somewhat stronger P–C bonds (E_{Diss} = 264 kJ/mol)[12d] that are less likely to break.

Compound **2** contains two crystallographically independent cations, two crystallographically independent anions, and one water molecule per asymmetric unit. The latter is situated between two tellurium atoms, forming two hydrogen bonds (Figure 2). Most of the crystal structures of known compounds containing RTe$^-$ exhibit direct Te···A interactions involving the metal counterion A$^+$. For instance, even in the structures of mesitylene analogs with large counterions $[Na(tmeda)_2]^+$ or $[K(18\text{-crown-}6)]^+$, one observes direct Te···A contacts (Te···Na 3.494 Å, Te···K 3.499 Å).[13] Compound **2** is thus one of the very few examples in which Te exhibits the rare coordination number 1. To the best of our knowledge, these are up to now the tellurocyanate $[Ph_3P{=}N{=}PPh_3][TeCN]$,[14] two 2-thienyl analogs, $[PPh_4][C_4H_3STe]$ and its 5-methyl derivative, tetraphenylphosphonium 2-tellurido-5-methylthiophene,[15] and $[Li(THF)_4][(PhMe_2Si)_3C{-}Te]$,[16] all four with bulky, weakly interacting cations. However, the low coordination number does not seem to affect the Te–C distances: in **2**, the Te–C distances (2.130 Å on average) range between the extremely short value in the tellurocyanate (2.020 Å) and that in the tris(phenyldimethyl)silyl tellurolate (2.270 Å), all of which do not differ significantly from the Te–C distances of the metal coordinating tellurolates [2.135(5) or 2.150(3) Å].[13]

$$2\,[SnTe_4]^{4-} + 2\,[AsPh_4]^+ + 0.5H_2O \rightarrow [AsPh_4][TePh(H_2O)_{0.5}]\ (\mathbf{2}) + AsPh_3 + Te^{2-} + [Sn_2Te_6]^{4-} \tag{3}$$

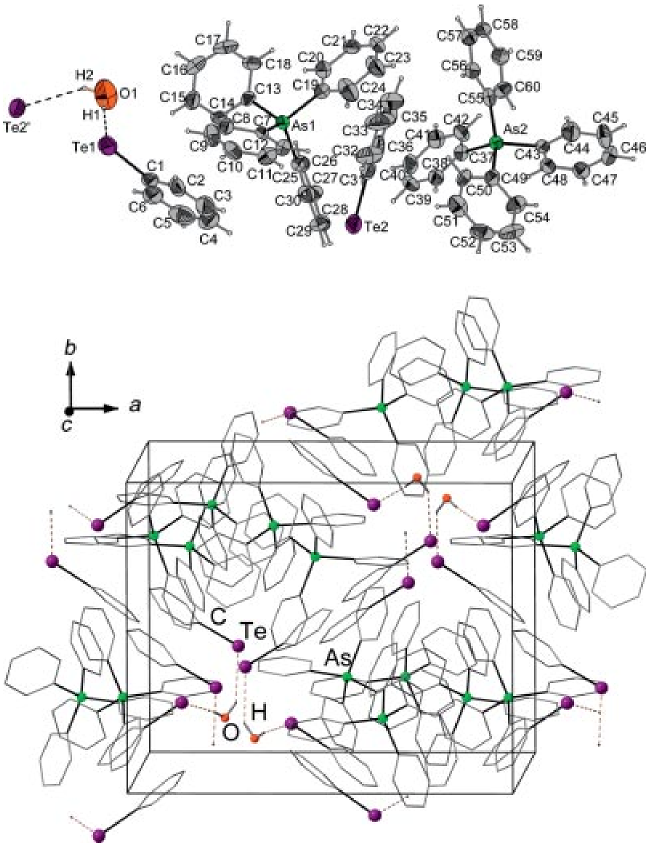

Figure 2. Top: relative orientation of independent cations, anions, and one water molecule in **2**, showing the two independent formula units within one asymmetric unit. Adjacent Te···H distances are indicated by dashed lines. Thermal ellipsoids are drawn to 50% probability. Selected interatomic distances [Å] and angles [°]: As–C 1.899(9)–1.941(8), Te–C 2.123(9)–2.136(10), Te···(H–)O 3.654(9)–3.736(8), Te···H(–C) 3.469(1)–3.553(1); C–As–C 109.3(1). Bottom: fragment of the crystal packing in **2**, viewed down the crystallographic *a* axis. H atoms at C are omitted for clarity.

Being interested in the air-stability of **2** within the mother liquor, we allowed air to enter into the reaction mixture for a second. After three months, compound **3** was obtained in addition to unchanged **2**. Upon slow oxidation of the $[Sn_2Te_6]^{4-}$ and Te^{2-} ions [cf. Equation (3)] by air according to Equation (4), the reaction solution turned from red to transparent – an unambiguous hint that oxidation has taken place [Equation (4)].

By the same reaction that led to the synthesis of **1** here [cf. Equation (1)], Haushalter and co-workers obtained a mixture of $[PPh_4]_2[Te_4(MeOH)_2]$ and another, not further specified, tin-containing product.[17] Similar to the process observed here [Equation (4)], intrusion of air caused the formation of the polytelluride. The crystal structure of **3** comprises isolated tetraphenylarsonium cations and water-bridged tetratelluride anions. The relative orientation of the ions are shown as fragments of the crystal packing in **3** (Figure 3). Te1 and Te4 lie one on top of each other in the direction of the crystallographic *a* axis. Thus, the anionic substructure resembles the arrangement of the infinite chains $^1_\infty Te_n$ in elemental α-tellurium,[18] which have been formally cleaved by addition of two extra electrons after every fourth atom, thereby enlarging the Te–Te–Te angles [109.24(1)° and 109.01(1)° in **3**, 103.2° in α-Te]. The dihedral angle Te1–Te2–Te3–Te4 [103.94(1)°] is slightly larger than those in other tetratellurides, such as $[PPh_4]_2[Te_4(MeOH)_2]$ [99.85(12)°] or $[Sr(15\text{-crown-}5)_2][Te_4(H_2O)]$ [102.47(1)°].[19] A significant structural difference between **3**

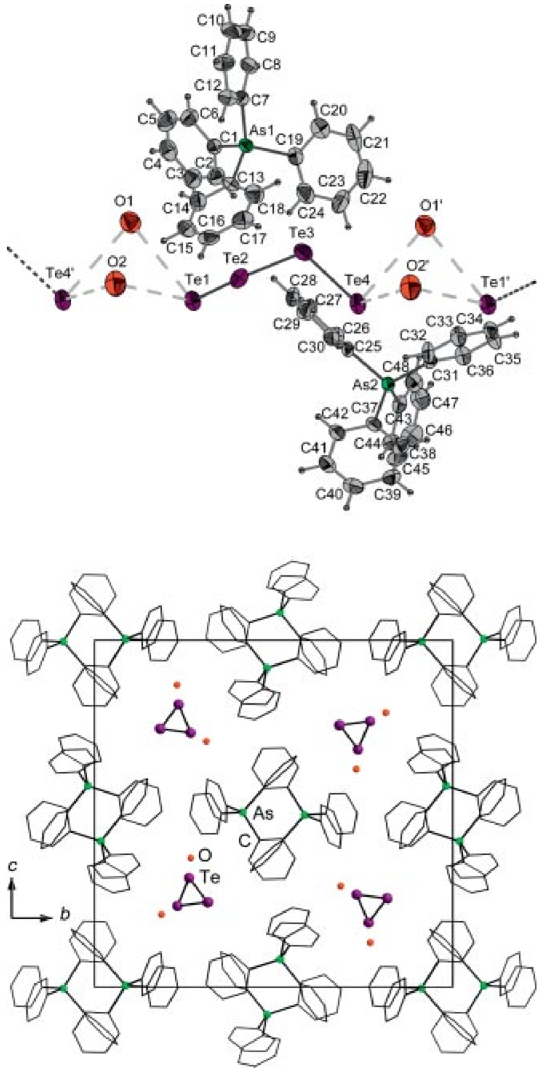

Figure 3. Top: relative orientation of the two independent cations, one tetratelluride anion and bridging water molecules in **3**. Adjacent Te···(H–)O distances are indicated by dashed lines. Thermal ellipsoids are drawn to 50% probability. Selected interatomic distances [Å] and angles [°]: As–C 1.882(10)–1.930(4), Te···(H–)O 3.628(47)–3.703(14), Te1–Te2 2.690(34), Te2–Te3 2.731(36), Te3–Te4 2.718(5), Te1···Te4′ 4.749(1), Te···H(–C) 3.103(1)–3.406(11); C–As–C 109.7(7), Te1–Te2–Te3 109.24(1), Te2–Te3–Te4 109.01(1). Bottom: fragment of the crystal packing in **3**. H atoms are omitted for clarity.

$$[Sn_2Te_6]^{4-} + 2\ Te^{2-} + 3\ O_2 + 8\ H_2O + 4\ [AsPh_4]^+ \rightarrow 2\ [AsPh_4]_2[Te_4(H_2O)_2]\ (\mathbf{3}) + 2\ [Sn(OH)_6]^{2-} \qquad (4)$$

and known $[Te_4]^{2-}$ compounds is the coordination sphere around the polytelluride anion: while two methanol molecules each form one hydrogen bond to the terminal tellurium atoms of one polytelluride anion in $[PPh_4]_2[Te_4(MeOH)_2]$,[17] two bridging water molecules lead to the formation of a one-dimensional coordination polymer $^{1}_{\infty}[Te_4(H_2O)_2]^{2-}$ in **3**. In other known hydrates of alkali(ne earth) metal polytellurides, H_2O molecules either coordinate to the metal counterion, like in {[BaCl(18-crown-6)(dmf)$_2$]$_2$[BaCl(18-crown-6)(dmf)(H$_2$O)]$_2$[Te$_4$]$_2$},[16] or one molecule is connected to both ends of one Te_4^{2-} unit through hydrogen bonds, like in [Sr(15-crown-5)$_2$][Te$_4$(H$_2$O)]. Te···(H–)O hydrogen bonds to water {e.g. in **2**, **3**, and [Sr(15-crown-5)$_2$][Te$_4$(H$_2$O)]} are by 11.1 pm longer on average than Te···(H–)O contacts to methanol molecules {e.g. in **1** or (PPh$_4$)$_2$[Te$_4$(MeOH)$_2$]}. Beside the supramolecular arrangement, the structural parameters [see Figure 3 (bottom)] are very similar to those in the cited tetratellurides and show the same characteristic distribution of bond lengths along the polytelluride chain as reported for all structurally characterized polychalcogenides.[20]

Conclusions

An uncommon synthesis route into a single-crystalline, surprisingly air-stable tellurostannate salt (**1**), a tellurolate salt (**2**), and a tetratelluride salt (**3**) provided high-yield access to these homologues of silicate, thiophenolate, or polysulfide compounds. The presented crystal structures extend the yet limited number of structural reports of $[Sn_2Te_6]^{4-}$, $[TePh]^{-}$, and $[Te_n]^{2-}$ salts. The synthesis of compound **2** represents a remarkably simple alternative to known syntheses of chalcogenophenolate compounds under drastic conditions. The investigations showed that the nature of cations (and solvent molecules) significantly affect the nature of the stabilized anion and the resulting crystal packing.

Experimental Section

General: All synthesis steps were performed under exclusion of air and external moisture (Ar atmosphere at a high-vacuum, double-manifold Schlenk line or N_2 atmosphere in a glove box), unless otherwise noted. MeOH was dried and freshly distilled prior to use; water was degassed by applying dynamic vacuum (10^{-3} Torr) for 1 h.

[K$_2$(MeOH)$_6$][PPh$_4$]$_2$[Sn$_2$Te$_6$] (1): PPh$_4$Cl (375 mg, 1 mmol) was drained in MeOH (5 mL). The solution was layered by a solution of [K$_4$(H$_2$O)$_{0.5}$][SnTe$_4$] (159 mg, 0.2 mmol) in MeOH (5 mL). Dark red blocks of **1** crystallized after two days. Yield: 275 mg [0.161 mmol, 80.5% according to Equation (1)]. $C_{54}H_{58}K_2O_6P_2Sn_2Te_6$ (1946.20): calcd. C 33.33, H 3.00; found C 33.62, H 2.98.

[AsPh$_4$][TePh(H$_2$O)$_{0.5}$] (2): AsPh$_4$Cl (419 mg, 1 mmol) was drained in H$_2$O (5 mL). The solution was layered by a solution of [K$_4$(H$_2$O)$_{0.5}$][SnTe$_4$] (159 mg, 0.2 mmol) in MeOH (5 mL). Large orange parallelepipeds of **2** crystallized after one hour. Yield: 49 mg [0.084 mmol, 83.3% according to Equation (3)]. $C_{30}H_{26}AsO_{0.5}Te$ (597.03): calcd. C 60.35, H 4.39; found C 59.99, H 4.42.

[AsPh$_4$]$_2$[Te$_4$(H$_2$O)$_2$] (3): Big black blocks of **3** crystallized after three months from the same solution as **2** upon opening the Schlenk tube for one second. Yield: 171 mg [0.130 mmol, 80.7% according to Equation (4)]. $C_{48}H_{44}As_2O_2Te_4$ (1313.07): calcd. C 43.91, H 3.38; found C 44.19, H 3.29.

Table 1. Crystal data and experimental details of the single-crystal X-ray analyses of compounds **1**, **2**, and **3**.

	1	**2**	**3**
Empirical formula	$C_{54}H_{58}O_6K_2P_2Sn_2Te_6$	$C_{30}H_{26}O_{0.5}AsTe$	$C_{48}H_{44}O_2As_2Te_4$
Formula weight [g mol^{-1}]	1946.20	597.03	1313.07
Crystal color and shape	dark red block	orange parallelepiped	black block
Crystal size [mm^3]	0.30 × 0.14 × 0.14	0.41 × 0.15 × 0.11	0.26 × 0.18 × 0.11
Diffractometer	Stoe IPDS	Stoe IPDS	Stoe IPDS
Radiation (λ [Å])	Mo-K_α (0.71073)	Mo-K_α (0.71073)	Mo-K_α (0.71073)
Temperature [K]	120	193	193
Crystal system	triclinic	orthorhombic	monoclinic
Space group	$P\bar{1}$ (no. 2)	$Pca2_1$ (no. 29)	$P2_1/c$ (no. 14)
a [Å]	11.0590(7)	17.5966(11)	10.790(2)
b [Å]	12.7370(7)	14.2255(14)	21.173(4)
c [Å]	13.3224(7)	20.5170(17)	20.521(4)
α [°]	75.252(4)	90	90
β [°]	67.782(5)	90	93.48(3)
γ [°]	76.021(5)	90	90
V [Å^3]	1657.73(16)	5135.8(7)	4679.4(16)
Z	1	8	4
ρ_{calc} [g cm^{-3}]	1.955	1.544	1.864
μ(Mo-K_a) [mm^{-1}]	3.556	2.45	3.909
Reflections measured	24634	29441	34351
Unique reflections	9072	9526	8572
No. of parameters	325	590	506
wR_2 (all data)	0.2183	0.0885	0.1458
R_1 [$F_o > 4\sigma(F_o)$]	0.0772	0.0425	0.0572
S (all data)	1.008	0.858	1.042
Largest diff. peak/hole [e$^-$ 10^{-6} pm^{-3}]	4.548/–3.238	1.086/–0.757	1.12/–1.74
Flack parameter[5]	–	0.008(15)	–

Optical Absorption Spectrum of 1: For the investigation of the optical absorption behavior of compound **1**, a UV/vis spectrum of the single crystalline material was recorded. As expected and in accordance with the visible color of the crystals, the onset of absorption – i.e. the lowest electronic excitation energy E_g – of compound **1** (1.4 eV) is small. It is even smaller than that in $[Mn(en)_3]_2$-$[Sn_2Te_6]\cdot 4H_2O$ (1.6 eV).[5]

NMR Spectroscopy of 1: The ^{125}Te-NMR and ^{119}Sn-NMR spectra of **1** served to prove the purity of this compound in D_2O solution, according to the known value for $[SnTe_4]^{4-}$.[3] ^{125}Te: two strong signals at δ = –199.9 ppm (terminal) and 553.0 ppm (bridged) and a very weak signal at –372.7 ppm arising from the $[SnTe_4]^{4-}$ monomer. ^{119}Sn: δ = –1680.5 ppm; slight shift in comparison to the signal of the monomer at –1670.2 ppm, which was not observed here.

Crystal Structure Analyses: All X-ray diffraction measurements were performed with Stoe Imaging Plate Systems (IPDS). The crystal data and experimental conditions are summarized in Table 1. For the data sets of **1** and **2** a numerical, for that of **3** a semiempirical absorption correction has been applied. The structures were solved by direct methods of SHELXS-97[4] and refined by full-matrix least-squares methods of SHELXL-97[4] with use of anisotropic displacement parameters for all non-hydrogen atoms. The H-atoms were treated as riding on idealized positions with isotropic displacement parameters set 1.2 U_{eq} (1.5 U_{eq} for methyl groups) of their bonding partners. For **1**, only crystals of poor quality could be quarried out of conglomerates. Thus, the precision of the results is reduced. In the structure of **2**, the H atomic positions of the water molecule were calculated for the orientation of possible H bonds toward the neighboring Te1 and Te2 atoms. The subsequent refinement as rigid groups with free rotation and free common isotropic displacement parameters for the H atoms converged well. The refined Flack parameter of 0.008(15) confirms the correct absolute structure. In the structure of **3**, the crystal showed a small contribution of a (001) reflection twinning. The 24 most affected reflections 4*kl* and 5*kl* were therefore eliminated. The hydrogen atoms of the two water molecules could not be localized.

CCDC 652251 (**1**), -652252 (**2**), and -652253 (**3**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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