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Synthesis and Crystal Structures of Phenyltrimethylammonium Salts of Hexabromotellurate(IV), $[C_6H_5(CH_3)_3N]_2[TeBr_6]$, and *catena*-poly[(Di-μ-bromobis{tetrabromotellurate(IV)})-μ-bromine], $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10}\cdot Br_2]_n$

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The crystal structures of $[C_6H_5(CH_3)_3N]_2[TeBr_6]$ (**1**) and $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10}\cdot Br_2]_n$ (**2**) have been determined by X-ray methods and refined to $R=0.042$ and 0.044 for 1883 and 2793 observed reflections, respectively. Crystals of **1** are monoclinic, space group $P2_1/a$ with $Z=2$ and $a=14.314(1)$, $b=10.746(1)$, $c=8.947(1)$ Å, $\beta==103.89(1)°$. Crystals of **2** are triclinic, space group $P\bar{1}$ with $Z=1$ and $a=8.909(1)$, $b=8.989(1)$, $c=11.845(1)$ Å, $\alpha=95.39(1)°$, $\beta=101.88(1)°$, $\gamma=90.44(1)°$. The $[TeBr_6]^{2-}$ octahedron of **1** is centrosymmetric with Te–Br = 2.7054(6), 2.7056(6), 2.6931(5) Å and Br–Te—Br = 90.16(2), 90.15(2), 91.28(2)°. The dimeric, centrosymmetric $[Te_2Br_{10}]^{2-}$ unit of **2**, consisting of two edge-sharing distorted $TeBr_6$ octaheda, has Te–Br dimensions 2.5375(7) and 2.5419(8) Å for equatorial terminal, 2.9594(7) and 2.9666(7) Å for equatorial bridging, and 2.6915(8) and 2.6775(9) Å for axial bonds. One of the axial Br atoms in each $TeBr_6$ octahedron is linked to an equivalent Br atom by a Br_2 molecule, and so forming a polymeric chain, $[Te_2Br_{10}\cdot Br_2]_n^{2n-}$, with Br–$Br_2$ = 3.1217(11) Å, Br–Br′ = 2.3324(17) Å, Br–Br–Br′ = = 174.29(6)°.

Tellurium(IV) bromides are six-coordinated octahedral or distorted octahedral complexes. The monomeric anion $TeBr_6^{2-}$ is known from numerous structure determinations[1,2] and there is one known example of a monomeric $TeBr_5^-$ anion.[3] There are four known dimeric forms, one single Br-bridged trianion, $Te_2Br_{11}^{3-}$,[4] one double Br-bridged dianion, $Te_2Br_{10}^{2-}$,[4–7] one double Br-bridged anion $Te_2Br_9^-$, where one of the axial Br atoms of one octahedron is replaced by a phenyl group of a crown ether,[4] and one triple Br-bridged anion, $Te_2Br_9^-$ (quoted in Ref. 4). There is one trimeric anion, $Te_3Br_{13}^-$, consisting of three octahedra, each sharing one edge with each of the two others and so forming a trigonal unit with idealised C_{3v} symmetry,[2,4] one tetrameric neutral molecule, Te_4Br_{16}, consisting of four octahedra, each sharing one edge with each of the three others in a cubane-like structure,[8,9] and one polymeric anion, $[Te_2Br_9]_n^{n-}$, consisting of Te_2Br_{10} units with one axial Br atom of each octahedron situated on a twofold axis.[4] We report here the structure of a new salt of the monomeric anion, $[C_6H_5(CH_3)_3N]_2[TeBr_6]$ (**1**), and a new polymeric structure variant, $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10}\cdot Br_2]_n$ (**2**).

Experimental

Preparations. In the present syntheses elemental tellurium is oxidised to tellurium(IV) by bromine. By adding phenyltrimethylammonium bromide three different complexes have been isolated. If the amounts of reactants used are in accordance with the equation

$$Te + 2Br_2 + 2Br^- = TeBr_6^{2-}$$

orange bipyramidal prisms of $[C_6H_5(CH_3)_3N]_2[TeBr_6]$ can be isolated. Use of more bromide does not change the reaction. By using less bromide the dimeric complex

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can be isolated:

$2Te + 4Br_2 + 2Br^- = Te_2Br_{10}^{2-}$

The crystals of $[C_6H_5(CH_3)_3N]_2[Te_2Br_{10}]$ are orange–yellow prisms. Use of more bromine in combination with reduced amount of bromide will give a polymeric complex where the dimeric complex ions are held together by bromine molecules:

$2Te + 5Br_2 + 2Br^- = Te_2Br_{10} \cdot Br_2^{2-}$

The crystals of $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10} \cdot Br_2]_n$ are short orange prisms.

$[C_6H_5(CH_3)_3N]_2[TeBr_6]$ (**1**). To 4.0 mmol (0.64 g) of bromine in 5 g acetonitrile was added 2.0 mmol (0.255 g) tellurium followed by 4.0 mmol (0.86 g) of phenyltrimethylammonium bromide. The suspension was diluted with 35 g of acetonitrile, heated to boiling, stirred and set aside for 24 h in a refrigerator. Mostly small crystals but also some larger were isolated. Yield 1.55 g, 88% of theoretical value calculated from the equation above.

$[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10} \cdot Br_2]_n$ (**2**). To 15 mmol (2.4 g) of bromine in 5 g acetonitrile were added 2.0 mmol (0.255 g) tellurium and 2.0 mmol (0.43 g) phenyltrimethylammonium bromide. The reactants dissolve on heating and stirring. The solution was set aside for 24 h. The amount of bromine employed was three times the stoicheiometric amount. Yield 1.1 g or 74% of theoretical value.

X-Ray structure analyses. The summary of crystal data, intensity collections and refinements are given in Table 1. The determination of unit cell parameters and the data collections were carried out on an Enraf-Nonius CAD4 diffractometer, using monochromated Mo Kα radiation (λ = 0.710 69 Å). The cell parameters were based on a least-squares fit of accurate setting angles for 25 reflections. Intensities were corrected for Lorentz and polarization effects, decay and absorption. Reflections with $I > 2\sigma(I)$ were regarded as observed.

The structures were solved by using the coordinates of the heavy atoms in the isomorphous selenium bromine compounds.[10] C and N atoms were located from Fourier difference maps and the structures were refined by full-matrix least-squares calculations, using anisotropic displacement parameters for all non-hydrogen atoms. Hydrogen atom positions were calculated with bond lengths C–H = 0.95 Å, and held fixed with displacement parameters equal to $1.3B_{eq}$ for the atom to which they are attached. A secondary extinction coefficient, g, in $F_{corr} = F_c(1 + gI_c)^{-1}$, was included as a variable and

Table 1. Crystallographic data.

	1	2
Formula	$C_{18}H_{28}N_2Br_6Te$	$C_{18}H_{28}N_2Br_{12}Te_2$
Formula weight	879.5	1486.6
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/a$	$P\bar{1}$
a/Å	14.314(1)	8.909(1)
b/Å	10.746(1)	8.989(1)
c/Å	8.947(1)	11.845(1)
α/°	95.39(1)	95.39(1)
β/°	103.89(1)	101.88(1)
γ/°	90.44(1)	
Volume/Å^3	1335.9	923.7
Z	2	1
Temperature/K	293	293
D_x/g cm^{-3}	2.186	2.672
$F(000)$	824	674
Θ_{max}	28	28
Scan mode	ω	ω
Min scan width/°	1.00	1.00
Loss of intensity (%)	2.1	1.0
Absorption coefficient/mm^{-1}	10.01	14.46
Crystal size/mm	0.14 × 0.22 × 0.27	0.15 × 0.25 × 0.27
Crystal volume/mm^3	0.0086	0.0103
Correction for absorption	Numerical	Numerical
Transmission factors	0.11–0.30	0.06–0.16
Independent reflections	3216	4451
No. with $I > 2\sigma(I)$	1883	2793
No. of parameters refined	125	155
$R = \Sigma \lVert F_o \rvert - \lvert F_c \rVert / \Sigma \lvert F_o \rvert$	0.042	0.044
$R_w = [\Sigma w(F_o - F_c)^2 / \Sigma w F_o^2]^{1/2}$	0.044	0.048
$S = [\Sigma w(F_o - F_c)^2 / (n - m)]^{1/2}$	1.99	2.18
Max. $\Delta(\rho)/e$ Å^{-3}	1.90	1.79

refined to 3.75×10^{-7} for compound **1**, but was found to be negligible for **2**.

The programs used were the Enraf-Nonius 1987 Structure Determination Package. All refined atomic coordinates are given in Table 2. Lists of anisotropic displacement parameters and complete lists of bond lengths and angles are available from the authors.

Results and discussion

Selected bond lengths and angles are given in Table 3, and a view of the anion of compound **2** is shown in Fig. 1. The Te atom of compound **1** is situated on a centre of symmetry, and the $TeBr_6^{2-}$ anion has nearly ideal octahedral symmetry with largest deviation of angles 1.28°, and the three independent bond lengths are 2.6931(5), 2.7054(6) and 2.7056(6) Å. The average value 2.701 Å is about 0.20 Å longer than the sum of the covalent radii.[10,11] No obvious reason for the variation is found in the crystal structure. The only interionic contact is between the bromine atom with the shortest bond to tellurium and its symmetry equivalent. Variation of the bond lengths in 3*c*–4*e* systems is common. The results are in good agreement with the dimensions of the anion in the oxonium salt $[H_9O_4]_2[TeBr_6]$, with bond lengths in the range 2.671(4)–2.716(4) Å.[2]

The $[Te_2Br_{10} \cdot Br_2]^{2-}$ anions of compound **2** are double bridged centrosymmetric Te_2Br_{10} dimers that are polymerized by bonding contacts between axial Br atoms and Br_2 molecules situated on symmetry centres. The compound is isomorphous with $[C_6H_5(CH_3)_3N]_{2n}[Se_2Br_{10} \cdot Br_2]_n$,[12] and with $[C_6H_5(CH_3)_3N]_{2n}[Te_2Cl_{10} \cdot Br_2]_n$.[13] The bond lengths of the anions of these three compounds are given in Table 4. *Trans* situated terminal and bridging bonds are listed on the same line. The connection between the $Y_2X_{10}^{2-}$ ions (Y = Te or Se, X = Br or Cl) and the Br_2 molecules have Y–X–Br_2 angles in the range 112.17(3)–115.05(4)°. The axial bond lengths seem to be unaffected by the contacts to the Br_2 molecules, as there is no systematic difference between the one involved in this contact and the other one. They are close to the bond lengths found in the $[C_6H_5(CH_3)_3N]$ salts of the corresponding hexahalochalcogenate, 2.701 Å in $[TeBr_6]^{2-}$ (Table 3), 2.572 Å in $[SeBr_6]^{2-}$ (Ref. 12) and 2.540 Å in $[TeCl_6]^{2-}$ (Ref. 13). The dinuclear complexes are polymerised, probably by

Table 2. Fractional atomic coordinates with e.s.d.σ in parentheses.

Atom	*x*	*y*	*z*	B_{eq}/Å^2
(**1**) $[C_6H_5(CH_3)_3N]_2[TeBr_6]$				
Te	0	0	0	2.21(1)
Br(1)	−0.17049(6)	0.11778(9)	−0.02913(10)	3.78(2)
Br(2)	0.08959(6)	0.22201(9)	0.00302(10)	4.14(2)
Br(3)	0.02841(6)	0.00366(10)	0.30863(8)	3.99(2)
N	0.1990(4)	0.5381(6)	0.3222(7)	3.1(1)
C(1)	0.1335(7)	0.5392(9)	0.1632(10)	4.4(2)
C(2)	0.2359(7)	0.4073(9)	0.3537(10)	4.9(2)
C(3)	0.2834(6)	0.6219(10)	0.3226(11)	4.9(2)
C(4)	0.1498(5)	0.5820(8)	0.4433(9)	2.9(2)
C(5)	0.1945(6)	0.5631(10)	0.5955(10)	4.4(2)
C(6)	0.1529(7)	0.6082(11)	0.7091(10)	5.1(3)
C(7)	0.0688(6)	0.6698(9)	0.6728(10)	4.3(2)
C(8)	0.0259(7)	0.6896(10)	0.5221(12)	5.6(3)
C(9)	0.0668(6)	0.6466(9)	0.4059(10)	4.3(2)
(**2**) $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10} \cdot Br_2]_n$				
Te	0.37811(6)	0.48977(6)	0.13925(5)	2.30(1)
Br(1)	0.09992(10)	0.49398(13)	0.15912(10)	4.39(2)
Br(2)	0.47912(13)	0.49063(14)	0.35627(9)	4.66(3)
Br(3)	0.37628(12)	0.79029(11)	0.15251(9)	3.77(2)
Br(4)	0.36545(14)	0.19050(12)	0.11822(10)	4.82(3)
Br(5)	0.29735(10)	0.48905(11)	−0.11599(8)	3.23(2)
Br(6)	0.46292(13)	0.93550(14)	0.40812(10)	5.19(3)
N	0.1109(8)	0.9648(8)	0.7772(7)	3.4(2)
C(1)	0.1315(23)	0.0777(17)	0.8762(13)	10.6(6)
C(2)	0.2495(15)	1.0514(19)	0.7752(14)	11.0(4)
C(3)	−0.0029(19)	1.0791(18)	0.7931(15)	10.5(5)
C(4)	0.0582(10)	0.8651(10)	0.6658(8)	3.6(2)
C(5)	−0.0953(12)	0.8259(14)	0.6309(11)	5.7(3)
C(6)	−0.1413(14)	0.7336(16)	0.5294(13)	7.5(4)
C(7)	−0.0389(16)	0.6808(13)	0.4668(11)	6.8(4)
C(8)	0.1133(15)	0.7182(15)	0.5040(12)	6.9(4)
C(9)	0.1633(13)	0.8104(15)	0.6047(11)	6.0(3)

Table 3. Distances (in Å) and angles (in °) in the anionic tellurium–bromine species, with e.s.d.s in parentheses.

(**1**) $[TeBr_6]^{2-}$			
Te–Br(1)	2.7054(6)	Br(1)–Te–Br(2)	90.16(2)
Te–Br(2)	2.7056(6)	Br(1)–Te–Br(3)	90.15(2)
Te–Br(3)	2.6931(5)	Br(2)–Te–Br(3)	91.28(2)
Interionic contact:			
Br(3)···Br(3a)	3.706(1)	Te–Br(3)···Br(3a)	159.17(3)
Symmetry operations: (a) $-x$, $-y$, $1-z$			
(**2**) $[Te_2Br_{10}\cdot Br_2]^{2-}$			
Te–Br(1)	2.5375(7)	Te–Br(5a)	2.9666(7)
Te–Br(2)	2.5419(8)	Br(3)–Br(6)	3.1217(11)
Te–Br(3)	2.6915(8)	Br(6)–Br(6b)	2.3324(17)
Te–Br(4)	2.6775(9)	Te···Te(a)	4.3233(7)
Te–Br(5)	2.9594(7)		
Br(1)–Te–Br(2)	93.26(3)	Br(3)–Te–Br(4)	176.95(3)
Br(1)–Te–Br(3)	87.89(3)	Br(3)–Te–Br(5)	87.73(2)
Br(1)–Te–Br(4)	89.94(3)	Br(3)–Te–Br(5a)	87.58(2)
Br(1)–Te–Br(5)	93.25(3)	Br(4)–Te–Br(5)	90.25(3)
Br(1)–Te–Br(5a)	175.46(3)	Br(4)–Te–Br(5a)	94.57(3)
Br(2)–Te–Br(3)	92.36(3)	Br(5)–Te–Br(5a)	86.31(2)
Br(2)–Te–Br(4)	89.90(3)	Te–Br(5)–Te(a)	93.69(2)
Br(2)–Te–Br(5)	173.50(3)	Te–Br(3)–Br(6)	112.17(3)
Br(2)–Te–Br(5a)	87.20(2)	Br(3)–Br(6)–Br(6b)	174.29(6)
Interionic contacts:			
Br(1)···Br(1c)	3.835(2)	Te–Br(1)···Br(1c)	100.09(3)
Br(1)···Br(5c)	3.477(1)	Te–Br(1)···Br(5c)	166.26(4)
		Te–Br(5)···Br(1c)	100.38(2)
		Te(a)–Br(5)···Br(1c)	164.61(3)
Br(2)···Br(2d)	3.336(1)	Te–Br(2)···Br(2d)	165.67(5)
Br(3)···Br(4e)	3.658(1)	Te–Br(3)···Br(4e)	170.50(3)
		Te–Br(4)···Br(3f)	168.49(3)
Symmetry operations: (a) $1-x$, $1-y$, z; (b) $1-x$, $2-y$, $1-z$; (c) $-x$, $1-y$, $-z$; (d) $1-x$, $1-y$, $1-z$; (e) x, $1+y$, z; (f) x, $y-1$, z.			

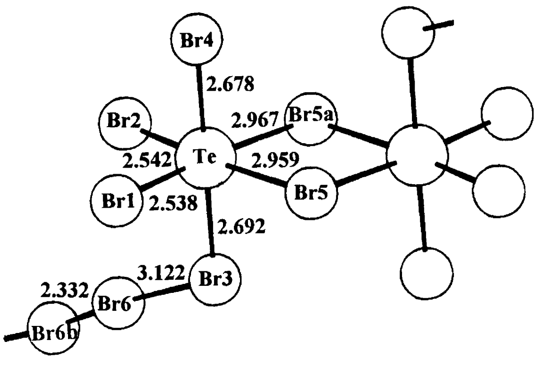

Fig. 1. View of the $Te_2Br_{10}\cdot Br_2^{2-}$ anion as found in the phenyltrimethylammonium salts.

charge transfer bonds where halogen ligands bonded to chalcogen are donors and the bromine molecules are acceptors. The X–Br_2–X sequences are almost linear with X–Br–Br angles of 173.00(19)–174.29(6)°. The Br–Br bonds of the Br_2 molecules, 2.302(1)–2.339(3) Å, are slightly longer than the covalent bond, 2.28 Å.[10] The Br–Br_2 bonds, 3.097(2) and 3.122(1) Å, and the Cl–Br_2 bond, 3.034(1) Å, are rather weak bonds.

In the equatorial planes of the dinuclear coordinations, each of the chalcogen atoms has a distorted square planar arrangement, being bonded to two terminal halogen atoms with rather strong 3*c*–4*e* bonds, the average bonds being about 0.15 Å shorter than the axial bonds. These bonds are close to or slightly longer than the sum of the covalent radii in the tellurium compounds, but about 0.10 Å longer than the sum of the covalent radii in the selenium compound.[10,11] The *trans* situated bridging bonds are correspondingly longer; the 3*c*–4*e* systems are asymmetric and the shortest terminal bonds have the longest *trans* bridging bond partners. The difference between the lengths of the two bridging bonds of one chalcogen atom is 0.008 Å for $[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10}\cdot Br_2]_n$, 0.061 Å for $[C_6H_5(CH_3)_3N]_{2n}[Te_2Cl_{10}\cdot Br_2]_n$, and 0.151 Å for $[C_6H_5(CH_3)_3N]_{2n}[Se_2Br_{10}\cdot Br_2]_n$. These three compounds are isomorphous, indicating a relation between variation of the bridging bond lengths and types of atoms involved. Table 5 lists selected data of the

Table 4. Selected bond lengths (in Å) and angles (in °) in $[Y_2X_{10} \cdot Br_2]^{2-}$ ions.

Compound	Equatorial terminal X-Y bonds	Equatorial bridging Y-X bonds	Axial Y-X bonds	X-Br_2 bonds	Br-Br bonds	Y-X-Br_2 angle	X-Br-Br angle	Ref.
$[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10} \cdot Br_2]_n$	2.538(1) 2.542(1)	2.967(1) 2.959(1)	2.678(1) 2.696(1)[a]	3.122(1)	2.332(2)	112.17(3)	174.229(6)	This work
$[C_6H_5(CH_3)_3N]_{2n}[Se_2Br_{10} \cdot Br_2]_n$	2.384(2) 2.423(2)	2.943(2) 2.792(2)	2.562(2) 2.573(2)[a]	3.097(2)	2.339(3)	114.23(8)	173.00(19)	12
$[C_6H_5(CH_3)_3N]_{2n}[Te_2Cl_{10} \cdot Br_2]_n$	2.357(1) 2.371(1)	2.882(1) 2.821(1)	2.518(1) 2.504(1)[a]	3.034(1)	2.302(1)	115.05(4)	173.73(4)	13

[a] X atom connected to Br_2.

Table 5. Bond lengths (in Å) in dimeric bromotellurate(IV) ions.

No.	Compound	Equatorial terminal Te-Br bonds	Equatorial bridging Te-Br bonds	Axial Te-Br bonds	Te···Te distance	Te-Br-Te angles	Ref.
(1)	$[(C_6H_5)_4P]_2[Te_2Br_{10}]$	2.579(2) 2.530(2)	2.876(2) 3.000(2)	2.648(2) 2.698(2)	4.213(2)	91.6(1)	5
(2)	$[H_3O(\text{Benzo-18-crown-6})]_2$ $[Te_2Br_{10}]$	2.548(3) 2.586(2) 2.585(2) 2.566(3)	3.019(3) 2.881(2) 2.891(2) 2.932(3)	2.664(2) 2.661(2) 2.685(2) 2.673(2)	4.256(2)	94.1(1) 92.1(1)	4
(3)	$[H_3O(\text{Dibenzo-18-crown-6})]_2$ $[Te_2Br_{10}] \cdot CH_3CN$	2.605(1) 2.563(2)	2.857(1) 2.952(2)	2.663(2) 2.701(2)	4.314(2)	95.9(1)	4
(4)	$[H_5O_2]_2[Te_2Br_{10}] \cdot 4C_4H_8O_2$	2.521(1) 2.571(1)	3.037(1) 2.914(1)	2.678(1) 2.673(1)	4.271(1)	91.7(1)	7
(5)	$[H_5O_2(\text{Benzo-24-crown-8})]_2$ $[Te_2Br_{10}]$	2.524(1) 2.606(1)	3.040(2) 2.811(1)	2.645(1) 2.706(1)	4.271(2)	93.7(1)	4
(6)	$[C_6H_5(CH_3)_3N]_{2n}[Te_2Br_{10} \cdot Br_2]_n$	2.538(1) 2.542(1)	2.967(1) 2.959(1)	2.678(1) 2.696(1)[a]	4.323(1)	93.7(1)	This work

[a] μ_2Br at 3.122(1) Å from Br_2.

known dimeric bromotellurate(IV) anions, including the present structure. From this table and a similar table given in the presentation of the structure of $[C_6H_5(CH_3)_3N]_{2n}[Te_2Cl_{10} \cdot Br_2]_n$,[13] it is seen that the difference between the lengths of the two bridging bonds of one tellurium atom varies within wide ranges. The data for the present structure are within the ranges for the other structures listed in the table. The presence of the charge transfer bond does not seem to have any certain influences on the crystal structure of the $Te_2Br_{10}^{2-}$ ion.

The dimensions of the phenyltrimethylammonium ion are within the following ranges: N–C = 1.503(7)–1.507(8) Å, C–C = 1.344(9)–1.390(9) Å, C–N–C = 107.2(5)–112.7(5)°, N–C–C = 118.7(5) and 121.4(5)°, C–C–C = 118.9(5)–121.3(6)° in compound **1** and N–C = 1.451(11)–1.511(8) Å, C–C = 1.352(14)–1.381(13) Å, C–N–C = 104.1(9)–111.8(8)°, N–C–C = 118.7(7) and 121.4(8)°, C–C–C = 118.0(8)–121.2(9)° in compound **2**.

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