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Novel Tellurium Oxide Fluorides: *cis*- and *trans*-$F_4Te(OTeF_5)_2$, *cis*- and *trans*-$F_2Te(OTeF_5)_4$, $FTe(OTeF_5)_5$, and $Te(OTeF_5)_6$[1]

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Received October 18, 1977

Preparation and structure of *cis*- and *trans*-$F_4Te(OTeF_5)_2$, *cis*- and *trans*-$F_2Te(OTeF_5)_4$, $FTe(OTeF_5)_5$, and $Te(OTeF_5)_6$ are described. All of them are generated by selective reactions starting with TeF_4 or $Te(OTeF_5)_4$; F_2, XeF_2, and $Xe(OTeF_5)_2$ have been used as oxidizers. These new tellurium oxide fluorides are examples of the rules that the environments around tellurium have to be octahedral and that Te–O double bonds do not occur. All compounds are, thermally and hydrolytically, surprisingly stable species with high vapor pressures. $Te(OTeF_5)_6$ crystallizes in two forms: I, triclinic, space group $P\bar{1}$, $a = 9.096$ (2) Å, $b = 9.132$ (2) Å, $c = 8.878$ (2) Å, $\alpha = 100.22$ (2)°, $\beta = 99.96$ (2)°, $\gamma = 115.41$ (2)°, $Z = 1$; II, trigonal, space group $R\bar{3}$, $a = 8.909$ (1) Å, $c = 27.543$ (3) Å, $Z = 3$ (hexagonal axes). The structures were solved with diffractometer data and refined by least-squares methods to $R = 0.029$ (I) and $R = 0.049$ (II). Both compounds are built up by discrete $Te(OTeF_5)_6$ molecules, whose molecular dimensions and angles are almost identical. The central Te atom is bonded octahedrally via the oxygens to six octahedral $OTeF_5$ groups. The average distances are Te–F = 1.817 Å and Te–O = 1.896 Å. The globular molecules are packed in a slightly different manner with 8 + 6 (I) and 6 + 6 (II) nearest neighbors.

Introduction

In 1956 Campbell and Robinson described tellurium oxide fluorides of the formulas $Te_3O_2F_{14}$ and $Te_6O_5F_{26}$ along with a species Te_2F_{10} which was later recognized as Te_2OF_{10}.[2-4] Only the last one has been structurally investigated.[4-7] The recent discovery of *trans*-$F_2Te(OTeF_5)_4$ by fluorination of $As(OTeF_5)_3$[8] and its structural investigations[9,10] raised the question if other tellurium oxide fluorides can be made in a more systematic manner. As already described in a short communication,[11] the chemistry of the Te–O–F system can be developed to a large extent.

Experimental Section

General Information. Fluorine NMR spectra were taken on a JEOL 60 HL instrument, using $CFCl_3$ as internal reference, 56.4 MHz. Infrared spectra were recorded on a Perkin-Elmer 457 spectrometer, Raman spectra were taken on a Coderc PH1 instrument, using a He–Ne laser for excitation, 190 mW. Mass spectra were recorded on a Varian CH7 instrument with 70 eV excitation energy. Analytical data caused problems, as tellurium and fluorine disturb each other. Fluorine was analyzed by Beller Co., Göttingen, West Germany, and tellurium was analyzed by reductive hydrolysis with N_2H_4/NH_3OH/HCl and weight of the elemental tellurium.

Conventional glass and metal vacuum lines were used for handling the materials. Moisture-sensitive compounds were handled in an automatic drybox of the Braun Co., West Germany.

Materials. Xenon difluoride was prepared from xenon and fluorine in a 4-L Pyrex glass vessel by irradiation with a high-pressure mercury lamp, 150 W; reaction time was 7 days.[12]

Xenon bis[pentafluorotellurate(VI)] was prepared from XeF_2 and $HOTeF_5$.[13,14]

Tellurium Tetrafluoride. As the other methods of preparation of TeF_4 were found to be rather ineffective and/or inconvenient, a new synthesis was developed. A total of 20 g (0.125 mol) of tellurium dioxide (commercial grade) was placed into a 100-mL stainless steel cylinder, and under vacuum and liquid-nitrogen cooling 63 g (0.585 mol) of sulfur tetrafluoride (commercial grade) was condensed on it. Heating to 130 °C for 48 h and pumping off the SF_4/SOF_2 volatiles afforded 22.1 g of crude TeF_4 that after sublimation at 100 °C (0.01 mbar) was a colorless, crystalline solid, yield 20 g (78%).

$B(OTeF_5)_3$ was obtained from BCl_3 and $HOTeF_5$.[15]

Tellurium(IV) Tetrakis[pentafluorotellurate(VI)]. In the drybox 69.7 g (95.9 mmol) of $B(OTeF_5)_3$ and 14.6 g (71.9 mmol) of TeF_4 were added in a quartz vessel. At reduced pressure of ca. 500 mbar the mixture is heated slowly to 80 °C. BF_3 evolved and was pumped off occasionally. The reaction was finished when the mixture was completely liquid at 100 °C. After cooling to room temperature the solid was sublimed at 90 °C (0.01 mbar), yield 77.4 g (99.5%), mp 89–91 °C. Anal. Calcd for $Te_5O_4F_{20}$: Te, 59.01; F, 35.1. Found: Te, 60.0; F, 35.7. Raman spectrum: 894 (m), 796 (s), 745 (sh), 722 (s), 703 (s), 670 (vs), 606 (s), 450 (sh), 415 (s), 348 (s), 316 (s), 240 cm^{-1} (s). ^{19}F NMR in $CFCl_3$ solution: ab_4 pattern δ_a 44.1 ppm, δ_b 34.3 ppm, $J_{ab} = 183$ Hz. The mass spectrum gave evidence for the ions $Te_5O_4F_{19}^+$, $Te_4O_3F_{15}^+$, $Te_3O_2F_{11}^+$, $Te_3O_3F_9^+$, $Te_2OF_7^+$, $Te_2O_2F_5^+$, and smaller fragments. Metastable ions at *m/e* 467, 265, and 85.5 arise from the transitions $Te_4O_3F_{15}^+ \rightarrow Te_3O_2F_{11}^+ + TeOF_4$, $Te_3O_2F_{11}^+ \rightarrow Te_2OF_7^+ + TeOF_4$, and $Te_2OF_7^+ \rightarrow TeF_3^+ + TeOF_4$, respectively.

cis- **and** *trans*-**$F_4Te(OTeF_5)_2$.** In the drybox 3.7 g (18.2 mmol) of TeF_4 and 11.5 g (18.9 mmol) of $Xe(OTeF_5)_2$ were placed into a 50-mL quartz vessel. The mixture was heated to 110 °C; gas evolution (Xe) was observed. The disappearance of the yellow color of $Xe(OTeF_5)_2$ was a sign for the termination of the reaction. The cis, trans mixture was washed with 50% sulfuric acid and dried over P_2O_5 and then distilled in a dynamic vacuum into a −78 °C cold trap, yield 11.4 g (92%), colorless liquid, not moisture sensitive.

Separation of the Cis and Trans Isomers of $F_4Te(OTeF_5)_2$. The mixture was cooled to −30 °C and colorless crystals of *trans*-$F_4Te(OTeF_5)_2$ crystallized. Dilution of the liquid with *n*-C_5F_{12} at −50 °C afforded another, impure fraction of the trans isomer, whereas the pure cis isomer stays in solution and can be isolated by distillation.

trans-**$F_4Te(OTeF_5)_2$:** mp 19 °C, bp 150 °C, colorless liquid, not moisture sensitive. Anal. Calcd for $Te_3O_2F_{14}$: Te, 56.2; F, 39.1. Found: Te, 55.6; F, 39.2. IR spectrum (gas): 920 (m), 852 (sh), 848 (s), 747 (s), 718 (s), 705 (m), 691 (m), 662 (w), 635 cm^{-1} (w). Raman spectrum (liquid): 828 (w), 722 (s, p), 704 (m, p), 673 (vs, p), 663 (sh), 651 (sh, p), 479 (s, p), 451 (s, p), 356 (w), 318 (m, p), 275 (w, dp), 252 (m, p), 239 (sh), 197 (sh, p), 180 (m, p), 149 (w, dp), 122 (m, p), 109 cm^{-1} (sh, p). ^{19}F NMR ($CFCl_3$ external): ab_4 pattern δ_a 51.7 ppm, δ_b 40.3 ppm, $J_{ab} = 182$ Hz, and a single line at δ 20.7 ppm. ^{125}Te isotope lines are observed as well. The mass spectrum gave evidence for the ions $Te_3O_2F_{13}^+$, $Te_2OF_9^+$, $Te_2O_2F_7^+$, $Te_2OF_7^+$, and smaller fragments.

cis-**$F_4Te(OTeF_4)_2$:** mp −26.5 °C, bp 127 °C. Anal. Calcd for $Te_3O_2F_{14}$: Te, 56.2; F, 39.1. Found: Te, 53.7; F, 39.9. IR (gas): 900 (m, sh), 888 (s), 851 (s), 747 (vs), 712 (s), 709 (s), 698 (m, sh), 661 (w), 638 cm^{-1} (w). Raman spectrum (liquid): 726 (s, p), 674 (vs, p), 663 (sh), 475 (m, p), 461 (m, p), 357 (w), 318 (m, dp), 277 (w, dp), 253 (m, p), 173 (m, p), 132 (w, p), 114 cm^{-1} (m, p). ^{19}F NMR ($CFCl_3$ external standard): ab_4 pattern δ_a 51.8 ppm, δ_b 40.7 ppm, $J_{ab} = 181$ Hz; and a c_2d_2 pattern with δ_c 27.7 ppm, δ_d 35.9 ppm, $J_{cd} = 174$ Hz, ^{125}Te isotope lines as well. Mass spectrum: $Te_3O_2F_{13}^+$, $Te_2OF_9^+$, $Te_2O_2F_7^+$, $Te_2OF_7^+$.

cis- **and** *trans*-**$F_2Te(OTeF_5)_4$.** $Te(OTeF_5)_4$ (53.9 g, 49.9 mmol) was placed under dry conditions into a 250-mL glass vessel and suspended in 180 mL of 1,1,2-trichlorotrifluoroethane (Freon 113). A slow, undiluted stream of fluorine was bubbled through the suspension at −10 °C. The reaction was finished when the starting material had completely dissolved. The solvent was pumped off in vacuo and the residue was dissolved in *n*-C_6F_{14}. Cooling to −30 °C afforded crystallization of *trans*-$F_2Te(OTeF_5)_4$ along with $FTe(OTeF_5)_5$; see below. The remaining solution was distilled in vacuo with a spaltrohr column: 2 g of *cis*-$F_2Te(OTeF_5)_4$, 3.6% yield, colorless liquid, not moisture sensitive. One of the more volatile products among others was *asym*-$F_3Te(OTeF_5)_3$ but was only characterized by the ^{19}F NMR spectrum.

0020-1669/78/1317-1926\$01.00/0

cis-**$F_2Te(OTeF_5)_4$**: mp 12 °C, bp 63 °C (2 mbar). Anal. Calcd for $Te_5O_4F_{22}$: Te, 57.0; F, 37.3. Found: Te, 58.4; F, 37.3. Raman spectrum (liquid): 746 (m), 716 (s, p), 693 (s, p), 674 (vs, p), 662 (s, sh), 474 (s, p), 439 (s, p), 422 (s, p, sh), 328 (m, dp, sh), 315 (s, dp), 280 (w, dp), 249 (s, dp), 197 (m, p), 180 (m, p), 133 (s, p), 110 (s, p), 90 (s, p), 71 cm^{-1} (s). ^{19}F NMR ($CFCl_3$ external): two overlapping ab_4 patterns δ_a 51.5 ppm, $\delta_{a'}$, 51.6 ppm, δ_b 39.7 ppm, $\delta_{b'}$, 39.7 ppm, $J_{ab} = J_{a'b'} = 174$ Hz and a single line at δ 3.3 ppm. Mass spectrum: $Te_5O_4F_{21}^+$, $Te_5O_4F_{19}^+$, $Te_4O_3F_{17}^+$, $Te_4O_3F_{15}^+$, $Te_3O_2F_{13}^+$, $Te_3O_3F_{11}^+$, $Te_3O_2F_{11}^+$, $Te_2OF_9^+$, $Te_2O_2F_7^+$, $Te_2OF_7^+$, $Te_2O_2F_5^+$, $Te_2OF_5^+$, and smaller fragments. Metastable ions at *m/e* 389.4, 269.9, 202.7, and 82.2 prove the decompositions

$$Te_5O_4F_{19}^+ \rightarrow Te_3O_3F_{11}^+ + Te_2OF_8$$

$$Te_3O_2F_{13}^+ \rightarrow Te_2O_2F_7^+ + TeF_6$$

$$Te_4O_3F_{17}^+ \rightarrow Te_2O_2F_7^+ + Te_2OF_{10}$$

$$Te_2O_2F_7^+ \rightarrow TeF_3^+ + TeO_2F_4$$

trans-**$F_2Te(OTeF_5)_4$**.[7–10] The crystalline material was distilled slowly through a 25-cm column at 33 mbar. The distillate boiling not higher than 115 °C was collected and redistilled and then sublimed at 0.1 mbar onto a −10 °C cooled finger. Less volatile materials consisted mainly of $FTe(OTeF_5)_5$.

trans-**$F_2Te(OTeF_5)_4$**: mp 76 °C, bp 107–115 °C (33 mbar) colorless crystalline solid, not moisture sensitive. Anal. Calcd for $Te_5O_4F_{22}$: Te, 56.9; F, 37.3. Found: Te, 55.8; F, 37.9. Raman spectrum (solid): 830 (w), 755 (sh), 717 (s), 676 (vs), 661 (sh), 489 (m), 430 (m), 430 (sh), 419 (s), 321 (s), 244 cm^{-1} (m). ^{19}F NMR spectrum ($CFCl_3$ external): ab_4 pattern δ_a 50.6 ppm, δ_b 38.2 ppm, $J_{ab} = 177$ Hz, and a single line at δ −3.9 ppm. Mass spectrum: $Te_5O_4F_{21}^+$, $Te_5O_4F_{19}^+$, $Te_4O_3F_{17}^+$, $Te_4O_3F_{15}^+$, $Te_3O_2F_{13}^+$, $Te_3O_3F_{11}^+$, $Te_3O_2F_{11}^+$, $Te_2OF_9^+$, $Te_2O_2F_7^+$, $Te_2OF_7^+$, and smaller fragments. Metastable peaks at *m/e* 269.9 and 203.0 have the same origin as in the *cis*-$F_2Te(OTeF_5)_4$.

$FTe(OTeF_5)_5$. $Te(OTeF_5)_4$ (5 g, 4.6 mmol) was placed into a quartz trap under dry conditions; 0.84 g (5.0 mmol) of xenon difluoride was added. An exothermic reaction with gas evolution took place. After cooling, the mixture crystallized in part. Recrystallization at −30 °C from *n*-C_6F_{14} gave pure $FTe(OTeF_5)_5$, the liquid reaction products are *cis*-$F_2Te(OTeF_5)_4$ and *trans*-$F_4Te(OTeF_5)_2$.

$FTe(OTeF_5)_5$: colorless crystals, mp 48 °C, bp 90–100 °C (5 mbar). Anal. Calcd for $Te_6O_5F_{26}$: Te, 57.2; F, 36.9. Found: Te, 57.7; F, 35.3. Raman spectrum (solid): 755 (m), 733 (m), 716 (s), 690 (s), 676 (vs), 663 (s), 478 (m), 429 (s), 420 (s), 374 (w), 354 (w), 326 (s), 314 (s), 248 (s), 229 (s), 215 (m), 195 (s), 184 (m), 162 cm^{-1} (s). ^{19}F NMR: two overlapping ab_4 patterns of the intensity ratio 1:4 with δ_a 48.0 ppm, $\delta_{a'}$ 48.05 ppm, $\delta_b = \delta_{b'}$, 36.9 ppm, and a single line at δ −14.2 ppm. Mass spectrum: $Te_6O_5F_{25}^+$, $Te_6O_5F_{23}^+$, $Te_5O_4F_{21}^+$, $Te_5O_4F_{19}^+$, $Te_4O_3F_{17}^+$, $Te_4O_3F_{16}^+$, $Te_4O_3F_{15}^+$, $Te_3O_2F_{16}^+$, $Te_3O_2F_{13}^+$, and smaller fragments.

Attempted Reaction of $Te(OTeF_5)_4$ with Ozone. $Te(OTeF_5)_4$, solved in $C_2F_3Cl_3$, does not react with O_3/O_2 mixtures between −20 and +48 °C.

Tellurium Hexakis[pentafluorotellurate(VI)]. (a) A total of 2.8 g (4.56 mmol) of $Xe(OTeF_5)_2$ and 4.92 g (4.55 mmol) of $Te(OTeF_5)_4$ were added to a 50-mL quartz vessel under dry conditions. Slow heating to 100 °C resulted in a gas evolution and solidifying of the reaction mixture. The product sublimed at 110 °C (0.01 mbar), yield 6.1 g (86%) of pure $Te(OTeF_5)_6$.

(b) A total of 6.8 g (11.2 mmol) of $Xe(OTeF_5)_2$ and 11 g (10.2 mmol) of $Te(OTeF_5)_4$ were added to a 50-mL quartz vessel and dissolved in 40 mL of $C_2F_3Cl_3$. At room temperature the solution was irradiated by a 500-W high-pressure mercury lamp. Xenon evolution occurred along with precipitation of almost pure $Te(OTeF_5)_6$. Filtration and sublimation at 110 °C (0.01 mbar) afforded 13.9 g (88%) of pure $Te(OTeF_5)_6$.

$Te(OTeF_5)_6$: colorless, crystalline solid, mp 242 °C, hydrolytically stable, almost unsoluble at room temperature in $CFCl_3$, $C_2F_3Cl_3$, acetone, or acetonitrile. Anal. Calcd for $Te_7O_6F_{30}$: Te, 57.3; F, 36.6. Found: Te, 57.2; F, 36.6. Raman spectrum (solid): 827 (w), 795 (w), 756 (m), 745 (m), 733 (sh), 715 (s), 677 (vs), 662 (s), 468 (m), 432 (sh), 412 (s), 372 (m), 324 (s), 239 (sh), 211 cm^{-1}. ^{19}F NMR spectrum (solution in $C_2F_3Cl_3$, 120 °C): ab_4 pattern, δ_a 49.4 ppm, δ_b 37.3 ppm, $J_{ab} = 189$ Hz. Mass spectrum: $Te_7O_6F_{29}^+$, $Te_7O_6F_{27}^+$, $Te_6O_5F_{25}^+$, $Te_6O_5F_{23}^+$, $Te_5O_4F_{21}^+$, $Te_5O_4F_{19}^+$, $Te_4O_3F_{17}^+$, $Te_4O_4F_{15}^+$, $Te_4O_{315}^+$, and smaller fragments.

Table I. Crystal Data of Tellurium(VI) Hexakis[pentafluorotellurate(VI)]

	Form I	Form II
Formula	$Te(OTeF_5)_6$	$Te(OTeF_5)_6$
Mol wt	1559.13	1559.13
Crystal systems	Triclinic	Trigonal
Space groups	$P\bar{1}$	$R\bar{3}$
Extinctions	None	None (for rhombohedral axes)
Cell parameters, Å and deg	$a = 9.096$ (2) $b = 9.132$ (2) $c = 8.878$ (2) $\alpha = 100.22$ (2) $\beta = 99.96$ (2) $\gamma = 115.41$ (2)	Hexagonal axes $a = 8.909$ (1) $c = 27.543$ (3) Rhombohedral axes $a = 10.524$ (2) $\alpha = 50.09$ (2)
Cell vol Å^3	629.3	631.1 (rhomb axes) 1893.3 (hex axes)
Z (no. of formula units per cell)	1	1 (rhomb axes) 3 (hex axes)
Density (calcd), g cm^{-3}	4.114	4.102
Density (measd), g cm^{-3}	4.03	
Linear absorption factor (Mo Kα), cm^{-1}	82.6	82.4
Size of crystal, mm^3	0.2 × 0.1 × 0.8	0.2 × 0.2 × 0.8
Transmission factor	0.14–0.39	0.11–0.18
Rotation axis	*c*	*c*
Scan rate, deg min^{-1}	2.4	1.2
Time for background measurements, s	25	30
$2\theta_{max}$, deg	56	56
$2\theta_{min}$, deg	6.4	6.4
Measd layers	*hk*0–*hk*11	*hk*0–*hk*35
No. of measd reflections	2953	1890, yielding 982 unique

Crystal Structure of $Te(OTeF_5)_6$. The compound was recrystallized from $C_2F_3Cl_3$ (Freon 113). Two different forms of crystals were found: hexagonal columns (I) that were triclinic and bicapped rhombohedra (II) which were trigonal. The compound can be handled in the open air, but the crystal faces get dim after some hours; therefore all X-ray measurements were done with crystals sealed in glass capillaries. The cell parameters were determined from precession photographs and refined with the indexed lines of Guinier films (Guinier-Hägg XDC 700 camera, Cu Kα_1 radiation, λ 1.54051 Å, quartz monochromator, calibration substance $Pb(NO_3)_2$, $a = 7.856$ (1) Å). For the Guinier photographs the two forms were separated under the microscope. The density was measured pycnometrically for a sublimed sample, which contained only the triclinic form I. Intensity data were collected with a STOE Weissenberg diffractometer using the ω-scan technique (graphite monochromator, Mo Kα radiation, λ 0.7107 Å). The scan range was varied because of the elongation of reflections at low θ' angles on higher layers and was calculated by the formula $\Delta\omega = A + B \sin \mu / \tan \theta'$. The background was measured on both sides of the scan. Some equator reflections, which were measured daily, showed no decomposition of the crystals. *Lp* and absorption corrections were applied to the data. Reflections with σ_I greater than the intensity I were considered unobserved and not included in the refinement. ($\sigma_I = [Z + (U_1 + U_2)q^2 + (0.03I)^2]^{1/2}$, where Z is the peak count, U_1 and U_2 are the background counts, q is the ratio of the peak scan time to the time for both background measurements.) Crystal data and details of the measurements are given in Table I.

Structure Determination and Refinement. The structures were solved by the heavy-atom method and refined by least-squares techniques minimizing $\sum w(|F_o| - |F_c|)^2$. The weight w was set equal to $1/\sigma_F^2$, where $\sigma_F = 0.5\sigma_I[(Lp)I]^{-1/2}$. The R values are defined by

$$R_1 = \frac{\Sigma |F_o| - |F_c|}{\Sigma |F_o|}$$

for all reflections and

$$R_2 = \left[\frac{\Sigma w(|F_o| - |F_c|)^2}{\Sigma w F_o^{\,2}} \right]^{1/2}$$

only for reflections with $w \neq 0$. The scattering factors used were

Table II. Atomic Parameters and Anisotropic Temperature Factors[a] for $Te(OTeF_5)_6$ (Standard Deviations in Parentheses)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
(A) Modification I									
Te(1)	0	0	0	1.13 (1)	1.19 (1)	1.37 (1)	0.42 (1)	0.15 (1)	0.30 (1)
Te(2A)	0.39498 (3)	0.08431 (3)	0.24852 (3)	1.36 (1)	2.10 (1)	1.82 (1)	0.71 (1)	0.05 (1)	0.51 (1)
Te(2B)	0.24245 (3)	0.23962 (3)	−0.22658 (3)	1.63 (1)	1.68 (1)	1.86 (1)	0.48 (1)	0.46 (1)	0.66 (1)
Te(2C)	0.07535 (3)	0.39336 (3)	0.24229 (3)	1.84 (1)	1.45 (1)	1.87 (1)	0.70 (1)	0.26 (1)	0.12 (1)
O(A)	0.2199 (3)	0.0179 (3)	0.0607 (3)	1.4 (1)	2.3 (1)	1.8 (1)	1.0 (1)	0.2 (1)	0.5 (1)
O(B)	0.0764 (3)	0.1615 (3)	−0.1156 (3)	1.9 (1)	1.9 (1)	2.1 (1)	0.9 (1)	0.8 (1)	0.9 (1)
O(C)	0.0542 (4)	0.1738 (3)	0.1861 (3)	2.1 (1)	1.5 (1)	1.5 (1)	0.7 (1)	0.2 (1)	0.2 (1)
F(1A)	0.5457 (3)	0.1400 (4)	0.1317 (3)	1.8 (1)	4.3 (1)	3.4 (1)	1.2 (1)	0.8 (1)	1.2 (1)
F(2A)	0.4269 (4)	0.3000 (3)	0.2963 (3)	3.1 (1)	2.1 (1)	3.7 (1)	1.0 (1)	−0.2 (1)	−0.2 (1)
F(3A)	0.2469 (4)	0.0296 (4)	0.3693 (3)	2.6 (1)	5.4 (1)	2.2 (1)	1.3 (1)	0.8 (1)	1.1 (1)
F(4A)	0.3712 (4)	−0.1277 (4)	0.2054 (4)	3.5 (1)	2.7 (1)	4.5 (1)	1.7 (1)	0.1 (1)	1.2 (1)
F(5A)	0.5669 (4)	0.1462 (4)	0.4244 (3)	2.5 (1)	4.3 (1)	2.9 (1)	1.3 (1)	−0.4 (1)	0.9 (1)
F(1B)	0.2439 (4)	0.4422 (3)	−0.1807 (3)	3.3 (1)	2.0 (1)	3.9 (1)	1.1 (1)	1.1 (1)	1.2 (1)
F(2B)	0.4143 (3)	0.3138 (4)	−0.0457 (3)	1.9 (1)	3.4 (1)	3.0 (1)	0.5 (1)	−0.1 (1)	1.0 (1)
F(3B)	0.2448 (4)	0.0380 (3)	−0.2749 (3)	3.7 (1)	2.5 (1)	4.2 (1)	1.8 (1)	2.0 (1)	1.0 (1)
F(4B)	0.0761 (4)	0.1705 (4)	−0.4091 (3)	2.8 (1)	3.8 (1)	2.0 (1)	0.8 (1)	0.1 (1)	0.9 (1)
F(5B)	0.3980 (4)	0.3194 (4)	−0.3346 (3)	2.9 (1)	3.6 (1)	3.6 (1)	0.8 (1)	1.6 (1)	1.5 (1)
F(1C)	0.2267 (4)	0.4392 (3)	0.4286 (3)	3.8 (1)	2.8 (1)	2.3 (1)	1.3 (1)	−0.7 (1)	−0.3 (1)
F(2C)	0.2464 (4)	0.4748 (3)	0.1504 (3)	3.3 (1)	2.3 (1)	3.9 (1)	0.7 (1)	1.8 (1)	0.9 (1)
F(3C)	−0.0776 (4)	0.3503 (3)	0.0570 (4)	3.6 (1)	2.7 (1)	3.0 (1)	1.8 (1)	−0.6 (1)	0.4 (1)
F(4C)	−0.0947 (4)	0.3197 (4)	0.3372 (4)	3.5 (1)	3.5 (1)	4.6 (1)	1.5 (1)	2.4 (1)	0.7 (1)
F(5C)	0.0991 (4)	0.6049 (3)	0.3029 (4)	4.4 (1)	1.7 (1)	4.1 (1)	1.6 (1)	0.7 (1)	−0.1 (1)
(B) Modification II									
Te(1)	0	0	0	2.71 (4)	2.71	0.84 (2)	1.36	0.0	0.0
Te(2)	0.68732 (5)	−0.00051 (4)	0.08022 (1)	2.02 (1)	2.30 (1)	1.48 (2)	1.16 (1)	0.29 (1)	−0.10 (1)
O	0.8828 (5)	0.0855 (5)	0.0391 (1)	2.1 (2)	2.0 (2)	1.6 (1)	1.0 (1)	0.4 (1)	0.0 (1)
F(1)	0.7950 (5)	0.2018 (5)	0.1135 (1)	3.6 (2)	3.3 (2)	2.7 (1)	1.6 (1)	0.2 (1)	−0.9 (1)
F(2)	0.7799 (5)	−0.0917 (5)	0.1216 (1)	4.4 (2)	4.6 (2)	2.3 (1)	2.9 (2)	0.6 (1)	1.1 (1)
F(3)	0.5758 (5)	−0.2044 (5)	0.0470 (1)	3.2 (2)	2.8 (2)	3.7 (2)	0.6 (1)	0.8 (1)	−0.9 (1)
F(4)	0.5934 (5)	0.0931 (6)	0.0406 (1)	3.6 (2)	5.3 (2)	3.0 (1)	3.3 (2)	0.1 (1)	0.3 (1)
F(5)	0.5004 (5)	−0.0781 (6)	0.1201 (1)	3.1 (2)	4.9 (2)	3.0 (2)	1.9 (2)	1.3 (1)	0.1 (2)

[a] The form of the temperature factor is $\exp[-0.25(h^2a^{*2}B_{11} + k^2b^{*2}B_{22} + l^2c^{*2}B_{33} + 2hka^*b^*B_{12} + 2hla^*c^*B_{13} + 2klb^*c^*B_{23})]$.

those of Cromer and Mann.[16] The dispersion correction for tellurium was included.[17]

The refinement with anisotropic temperature factors for all atoms led to $R_1 = 0.031$ and $R_2 = 0.040$ for I and $R_1 = 0.051$ and $R_2 = 0.059$ for II, which decreased after a refinement of the interscale factors to $R_1 = 0.029$ and $R_2 = 0.039$ for I and $R_1 = 0.050$ and $R_2 = 0.056$ for II. For some of the strongest reflections the F_c value was much higher than the F_o value. These reflections (10 for form I, 17 for form II) were assigned zero weight and excluded from the last refinement. The final R values are $R_1 = 0.029$ and $R_2 = 0.029$ for form I (206 parameters refined) and $R_1 = 0.049$ and $R_2 = 0.042$ for form II (76 parameters refined). Final positional and thermal parameters are listed in Table II.

Results

The reaction of TeF_4 with $Xe(OTeF_5)_2$ gives in almost quantitative yield a mixture of the *cis*- and *trans*-$F_4Te(OTeF_5)_2$. It can be assumed that this reaction goes via the formation of $OTeF_5$ free radicals[14] that oxidize TeF_4 to the hexavalent state.

$$TeF_4 + Xe(OTeF_5)_2 \rightarrow Xe + cis\text{- and } trans\text{-}F_4Te(OTeF_5)_2$$

Due to the large differences in the molecular shape—the trans species is a chain type molecule, the cis species a more bulky one—the separation of the isomers is surprisingly easy. Their characterization is made without doubt by their very different ^{19}F NMR spectra; see Figures 1 and 2. The cis isomer shows as expected nonequivalence of the four central fluorine atoms in terms of a well-resolved a_2b_2 pattern. Historically it is of interest that the material $Te_3O_2F_{14}$, prepared by Campbell and Robinson in 1956[2] is *cis*-$F_4Te(OTeF_5)_2$, as can be derived from melting point and boiling point data. Even on heating to 150 °C no isomerization could be observed.

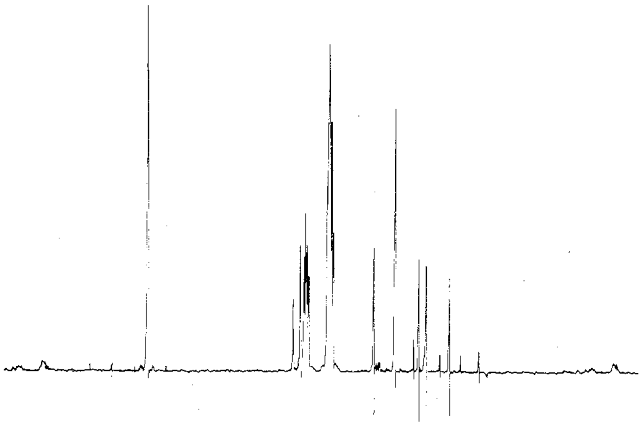


Figure 1. ^{19}F NMR spectrum of *trans*-$F_4Te(OTeF_5)_2$: ab_4 spectrum with a single line of the central equivalent fluorine atoms.

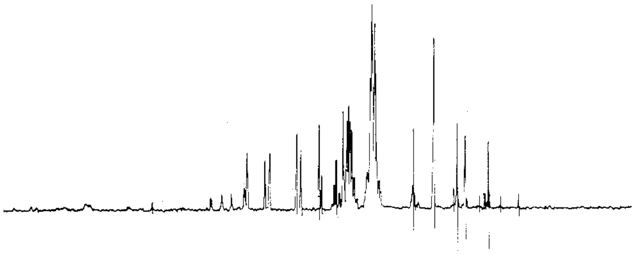


Figure 2. ^{19}F NMR spectrum of *cis*-$F_4Te(OTeF_5)_2$: partly overlapping ab_4 and a_2b_2 spectrum.

A similar pair of isomers exists naturally in *cis*- and *trans*-$F_2Te(OTeF_5)_4$. This trans isomer has been known recently including its molecular and crystal structure.[8-10] The good packing of the molecules resulted in a high melting point of 72 °C. Its first synthesis was based on the fluorination of $As(OTeF_5)_3$[8] but no reasonable mechanism could be given for its formation. Fluorination of $Te(OTeF_5)_4$ was expected to be an understandable route, but here a mixture of many compounds $F_xTe(OTeF_5)_{6-x}$ is observed. Yet *trans*-F_2Te-

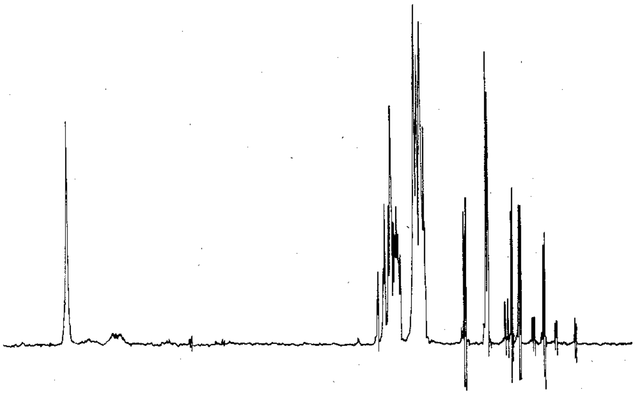


Figure 3. ^{19}F NMR spectrum of *cis*-$F_2Te(OTeF_5)_4$: nonequivalency of the $OTeF_5$ groups, visible in the a part on the right-hand side.

$(OTeF_5)_4$ can be purified from the mixture as well as *cis*-$F_2Te(OTeF_5)_4$.

$Te(OTeF_5)_4 + F_2 \rightarrow$ *trans*- and *cis*-$F_2Te(OTeF_5)_4$

The latter is a liquid, and its structure is proved by the existence of different $OTeF_5$ groups in the ^{19}F NMR spectrum; see Figure 3. Isomerization between *cis*- and *trans*-$F_2Te(OTeF_5)_4$ is not observed. The reaction mixture of the fluorination contains a lot of other species, from which an *asym*-$F_3Te(OTeF_5)_3$ could be detected by its characteristic ab_2 pattern in the ^{19}F NMR spectrum. It remains unexplainable how on the reaction of $Te(OTeF_5)_4$ with F_2 or even XeF_2 a major product is $FTe(OTeF_5)_5$. In the case of fluorination with XeF_2 it is even the main product besides some *cis*-$F_2Te(OTeF_5)_4$, no *trans*-$F_2Te(OTeF_5)_4$, but some *trans*-$F_4Te(OTeF_5)_2$.

$Te(OTeF_5)_4 + XeF_2 \rightarrow Xe + FTe(OTeF_5)_5$
cis-$F_2Te(OTeF_5)_4$
trans-$F_4Te(OTeF_5)_2$

The proposed structure of $FTe(OTeF_5)_5$ is again in accord with the NMR spectra: two different $OTeF_5$ groups with an intensity ratio of 1:4 and a single fluorine. The largest molecule of this series is readily prepared according the equation

$Te(OTeF_5)_4 + Xe(OTeF_5)_2 \rightarrow Xe + Te(OTeF_5)_6$

No by-products are observed. The extremely high melting point of 242.5 °C is already a strong indication for the spherical shape of this molecule.

Structure Description of $Te(OTeF_5)_6$

The atomic numbering is given in Figures 4 and 5. The distances and angles are listed in Table III. Both structures are built up by discrete $Te(OTeF_5)_6$ molecules. The central tellurium atom is bonded octahedrally to the six oxygen atoms of the $OTeF_5$ groups, which are also almost octahedral. In form II the central tellurium lies on the rotary inversion center and the molecule has $\bar{3}$ symmetry imposed by the lattice. In form I the required crystallographic symmetry of the molecule is $\bar{1}$ with the central tellurium atom on the inversion center, but the actual symmetry is almost $\bar{3}$. The molecule projected on a plane through Te(2A), Te(2B), and Te(2C) (Figure 4a) shows a very close relationship to the molecule in form II (Figure 4b). The parameters of I transformed into a trigonal system (the $\bar{3}$ axis perpendicular to the plane through Te(2A), Te(2B), and Te(2C)) deviate only by a maximum of 0.04 Å from the trigonal symmetry.

The molecular dimensions are very similar in both forms. The angles around the central and outer tellurium atoms deviate by a maximum of 3° from the octahedron angles. The angles at the oxygen atoms in I and II have the same value of about 139° which agrees very well with the value found in *trans*-$F_2Te(OTeF_5)_4$.[9,10] The Te–F distances range from 1.810 to 1.825 Å with an average distance of 1.817 Å. The Te–O distances to the central tellurium atoms are slightly longer (1.903 Å) than those to the outer tellurium atoms (1.893 Å). The difference lies in the range of the standard deviations and is not significant. The corresponding values found in *trans*-$F_2Te(OTeF_5)_4$[9,10] are somewhat shorter (Te–F 1.806 Å average value for the $OTeF_5$ group, Te–O 1.875 Å). As the

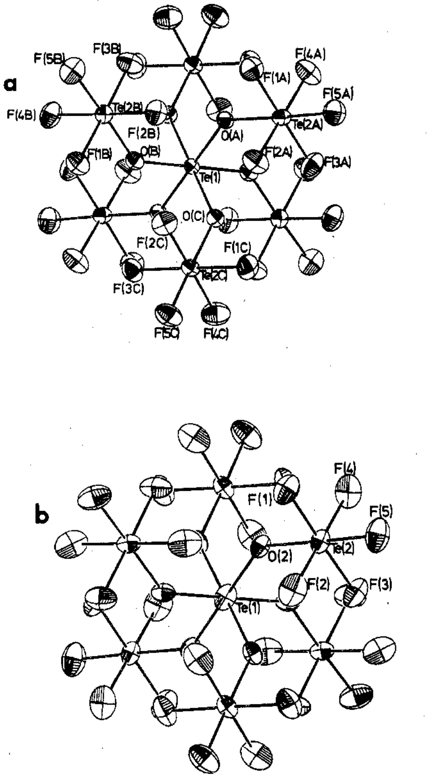


Figure 4. $Te(OTeF_5)_6$ molecule, projected along the $\bar{3}$ axis: (a) form I, (b) form II. The thermal ellipsoids are scaled to 50% probability.

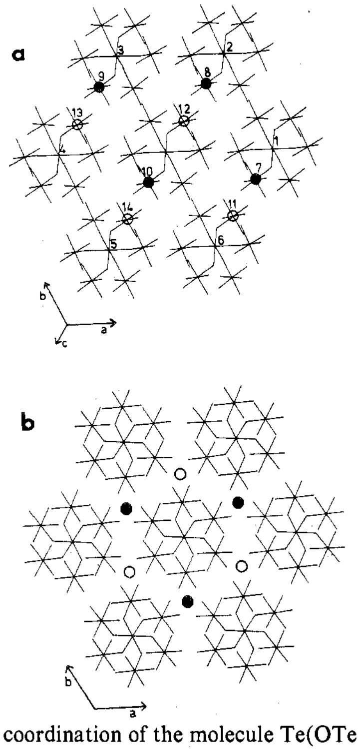


Figure 5. The coordination of the molecule $Te(OTeF_5)_6$: (a) form I, (b) form II. Projection on the *ab* plane. Only the molecules in the plane are shown. Molecules on the plane above and below are represented by solid and open circles, respectively.

Table III. Bond Distances (Å) and Angles (deg) (Standard Deviations in Parentheses)

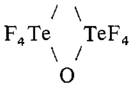
	Modification I			
	A	B	C	Modification II
Te(1)-O	1.906 (3)	1.901 (3)	1.900 (3)	1.903 (4)
Te(2)-O	1.892 (3)	1.898 (3)	1.894 (3)	1.890 (3)
Te(2)-F(1)	1.815 (3)	1.816 (3)	1.810 (3)	1.811 (4)
Te(2)-F(2)	1.820 (3)	1.821 (3)	1.816 (3)	1.819 (4)
Te(2)-F(3)	1.824 (3)	1.825 (3)	1.818 (3)	1.821 (4)
Te(2)-F(4)	1.814 (3)	1.812 (3)	1.821 (3)	1.813 (4)
Te(2)-F(5)	1.813 (3)	1.812 (3)	1.817 (3)	1.817 (4)
Te(1)-O-Te(2)	139.0 (2)	138.9 (2)	139.1 (2)	139.1 (2)
F(1)-Te(2)-F(2)	89.8 (2)	90.1 (2)	90.4 (2)	89.6 (2)
F(1)-Te(2)-F(3)	178.9 (2)	178.7 (2)	178.9 (2)	179.1 (2)
F(1)-Te(2)-F(4)	89.4 (2)	89.2 (2)	89.4 (2)	89.1 (2)
F(1)-Te(2)-F(5)	89.8 (2)	89.8 (2)	89.6 (2)	90.1 (2)
F(1)-Te(2)-O	88.5 (2)	88.4 (2)	88.5 (2)	88.7 (2)
F(2)-Te(2)-F(3)	90.2 (2)	89.8 (2)	90.0 (2)	90.7 (2)
F(2)-Te(2)-F(4)	177.9 (2)	178.2 (2)	177.9 (2)	178.2 (2)
F(2)-Te(2)-F(5)	89.0 (2)	89.3 (2)	88.8 (2)	89.2 (2)
F(2)-Te(2)-O	91.9 (2)	91.8 (2)	91.7 (2)	91.5 (2)
F(3)-Te(2)-F(4)	90.6 (2)	90.9 (2)	90.1 (2)	90.5 (2)
F(3)-Te(2)-F(5)	89.1 (2)	88.9 (2)	89.4 (2)	89.1 (2)
F(3)-Te(2)-O	92.6 (2)	92.9 (2)	92.5 (2)	92.1 (2)
F(4)-Te(2)-F(5)	89.0 (2)	89.0 (2)	89.1 (2)	89.6 (2)
F(4)-Te(2)-O	90.1 (2)	89.9 (2)	90.4 (2)	89.8 (2)
F(5)-Te(2)-O	178.0 (2)	177.9 (2)	178.1 (2)	178.7 (2)
O(A)-Te(1)-O(B)	90.9 (2)			
O(A)-Te(1)-O(C)	91.2 (2)			
O(B)-Te(1)-O(C)	90.8 (2)			
O-Te(1)-O				91.2 (2)
Torsion angles				
Te(1)-O-Te(2)-F(1)	−152.5 (3)	−152.7 (3)	−152.4 (3)	−151.8 (3)
Te(1)-O-Te(2)-F(2)	−62.8 (3)	−62.6 (3)	−62.1 (3)	−62.2 (3)
Te(1)-O-Te(2)-F(3)	27.5 (3)	27.2 (3)	28.0 (3)	28.6 (3)
Te(1)-O-Te(2)-F(4)	118.1 (3)	118.1 (3)	118.2 (3)	119.1 (3)
O′-Te(1)-O-Te(2)	26.9 (3)	28.4 (3)	26.6 (3)	25.7 (3)
O″-Te(1)-O-Te(2)	117.7 (3)	119.6 (3)	117.5 (3)	116.9 (3)

given distances in all structure determinations are not corrected for thermal motion, the differences may be caused by the higher temperature factors of *trans*-$F_2Te(OTeF_5)_4$.

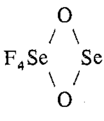
The arrangement of the $OTeF_5$ groups around the central tellurium atom is the same in both structures as can be seen from the torsion angles O′–Te(1)–O–Te(2) and Te(1)–O–Te(2)–F(n) [n = 1, 4]. The $OTeF_5$ groups are rotated around the O–Te(2) bond by ca. 27° out of the eclipsed form. Obviously this is sterically most favored. In *trans*-$F_2Te(OTeF_5)_4$ this angle is somewhat smaller (17°). Most distances between atoms of neighboring molecules are longer than the sum of the corresponding van der Waals radii. There are a few distances between fluorine atoms which are slightly shorter (2.82 Å, 2.88 Å) than twice the van der Waals radius for fluorine (1.47 Å),[18] but they do not correspond to any intermolecular interaction.

Discussion

It is obvious from Table IV, that all oxide fluorides of Te(VI) are based on the octahedral building principle. Tellurium–oxygen double bonds have not been observed. This includes even compounds like

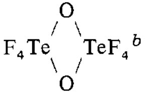


where no monomeric $TeOF_4$ can be observed,[19,20] quite in contrast to



$O{=}SeF_4$[19] and $O{=}SF_4$. The perfect shielding of the tellurium by six atoms is the reason for the rather high hydrolytical stability and the high volatility.

Table IV. The Known Oxide Fluorides of Tellurium

	Mp, °C	Bp, °C (mbar)
F_5Te-O-O-TeF_5[a]	−39	81.5
 [b]	28	77.5
F_5Te-O-TeF_5[c]	−36.6	59.8
trans-F_5TeO-TeF_4-$OTeF_5$[d]	19	150
cis-$F_4Te(OTeF_5)_2$[d,e]	−26.5	127
trans-$F_2Te(OTeF_5)_4$[d,f]	76	107-115 (33)
cis-$F_2Te(OTeF_5)_4$[d]	−12	63 (2)
$FTe(OTeF_5)_5$[d]	48	90-100 (5)
$Te(OTeF_5)_6$[d]	242.5	100 (0.01)
$(TeOF_4)_n$[g]		>250
$Te_6O_5F_{26}$[e]		>300
$Te(OTeF_5)_4$[d]	89-91	90 (0.01)

[a] Reference 12. [b] K. Seppelt, *Angew. Chem., Int. Ed. Engl.*, 13, 92 (1974); ref 19; [c] References 3-6. [d] This work. [e] Reference 2. [f] References 8-10. [g] References 8, 15.

Most of these new oxide fluorides have the general formula $F_xTe(OTeF_5)_{6-x}$, that means a central tellurium is surrounded by either fluorine or $OTeF_5$ groups. In that series only *sym*- and *asym*-$F_3Te(OTeF_5)_3$ are missing, but the latter one was already observed in the ^{19}F NMR spectra.

Isomers do not exchange as no ligand transfer takes place, either at room temperature or above. This is quite a contrast to the system $F_xU(OTeF_5)_{6-x}$, where all members were observed but interchanged rapidly, so that only $U(OTeF_5)_6$ was isolated in the pure state.[21,22] $Te(OTeF_5)_6$ and $U(OTeF_5)_6$ are quite similar in their physical data and are good examples for a study of the packing of huge spherically shaped molecules with only negligible intermolecular forces. The two crys-

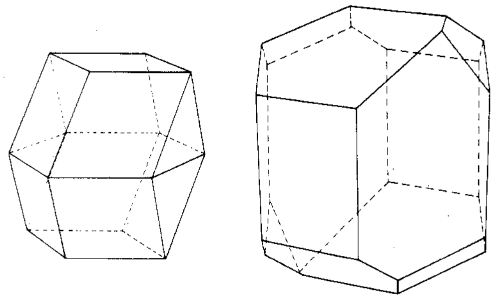


Figure 6. Coordination polyhedron (left) and corresponding polyhedral domain (right) of $Te(OTeF_5)_6$ form I.

tallographic forms of $Te(OTeF_5)_6$ differ only in the packing of the molecular units. The globular molecules are arranged in close packed triangular arrays in planes parallel to the *ab* plane. In form II the coordination is an exact hexagon with the six molecules at 8.91 Å, while in form I it is somewhat distorted with pairs of two neighbors at 9.10, 9.13, and 9.74 Å (Figure 5a, b). In II these layers are stacked in a way that the molecules of adjacent layers lie exactly in the middle of three molecules of the first layer. Besides its six neighbors in the plane each molecule gains three further neighbors above and three below at a distance of 10.05 Å. This arrangement corresponds to a cubic close packing, where only the distances between the layers are elongated.

In I the $\bar{3}$ axis of the molecule does not stand perpendicular on the plane as in II. The adjacent layers are shifted in the same direction and their molecules come to lie on the line between two molecules of the first layer, very close to one of them (Figure 5a). In that way each molecule has two more neighbors at a very short distance (8.88 Å) and two at a longer distance (10.82 Å). A similar coordination is found in $U(OTeF_5)_6$.[22] For $Te(OTeF_5)_6$ (I) a pseudomonoclinic, *C*-centered cell can be chosen with the cell parameters $a = 9.74$ Å, $b = 15.41$ Å, $c = 8.88$ Å, $\alpha = 89.8°$, $\beta = 109.1°$, $\gamma = 90.3°$, which shows the relation to $U(OTeF_5)_6$ (cell parameters: $a = 10.30$ Å, $b = 16.61$ Å, $c = 9.98$ Å, $\beta = 114.14°$). Besides these ten nearest neighbors the molecules have four more neighbors at distances of 11.55 and 11.56 Å in $Te(OTeF_5)_6$ (I) and of 12.35 Å in $U(OTeF_5)_6$. A comparison shows, that the distances in $Te(OTeF_5)_6$ scatter in a wider range:

$Te(OTeF_5)_6$ (I): 2 × 9.10 Å (1, 4), 2 × 9.13 Å (3, 6), 2 × 9.74 Å (2, 5), all in the plane; 2 × 8.88 Å (10, 12), 2 × 10.82 Å (8, 14), 2 × 11.55 Å (9, 11), 2 × 11.56 Å (7, 13), all in adjacent planes. (The numbering of Figure 5a is given in parentheses.)

$U(OTeF_5)_6$: 4 × 9.77 Å, 2 × 10.30 Å, in the plane; 2 × 9.88 Å, 2 × 11.02 Å, 4 × 12.35 Å, in adjacent planes.

The question of which molecules belong to the coordination sphere can be answered by constructing the polyhedral domain,[23] where the number of faces is the coordination number. The polyhedral domain of $Te(OTeF_5)_6$ (I) (Figure 6)—and that of $U(OTeF_5)_6$, which is similar, too—shows 14 faces. Therefore these molecules should be included in the coordination sphere and the coordination number is 8 + 6.

Conclusion

The octahedral, oxygen bridge building principle of tellurium(VI) oxide fluorides allows an infinite number of compounds. Besides those described in this work, long chains, cages, and ring species are possible. The polymer $(TeOF_4)_n$, a viscous liquid, may well belong in one of these categories.

Registry No. $Te(OTeF_5)_4$, 63599-48-4; *cis*-$F_4Te(OTeF_5)_2$, 63598-94-7; *trans*-$F_4Te(OTeF_5)_2$, 63569-53-9; *cis*-$F_2Te(OTeF_5)_4$, 63598-93-6; *trans*-$F_2Te(OTeF_5)_4$, 60788-81-0; $FTe(OTeF_5)_5$, 63599-47-3; $Te(OTeF_5)_6$, 63569-52-8; TeF_4, 15192-26-4; tellurium dioxide, 7446-07-3; SF_4, 7783-60-0; $B(OTeF_5)_3$, 40934-88-1; $Xe(OTeF_5)_2$, 25005-56-5; XeF_2, 13709-36-9.

Supplementary Material Available: A listing of structure factor amplitudes (19 pages). Ordering information is given on any current masthead page.

References and Notes

(1) Work supported by Fonds der Chemischen Industrie and by Deutsche Forschungsgemeinschaft. We are indebted to Dr. R. Geist for the mass spectra and Mr. D. Günauer for the tellurium analyses.
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