

Synthesis and Structures of Bis(pentafluorophenyl) Selenoxide / Telluroxide

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Bis(pentafluorophenyl) Selenoxide, Bis(pentafluorophenyl) Telluroxide, Solvate Structure

Bis(pentafluorophenyl) selenoxide and telluroxide were prepared by oxidation of the corresponding selenide and telluride with *m*-chloroperbenzoic acid. $(\text{C}_6\text{F}_5)_2\text{SeO}$ crystallizes in the triclinic space group $P\bar{1}$ with $a = 9.9452(9)$, $b = 14.871(2)$, $c = 15.279(2)$ Å, $\alpha = 117.17(1)$, $\beta = 101.02(1)$, $\gamma = 95.39(1)^\circ$. The telluroxide $(\text{C}_6\text{F}_5)_2\text{TeO}$ crystallizes as a dichloromethane or benzene solvate. The crystallographic data are for $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \text{CH}_2\text{Cl}_2$: monoclinic space group $P2_1/n$ with $a = 18.1918(2)$, $b = 7.7545(1)$, $c = 23.1719(3)$ Å, $\beta = 97.9523(4)^\circ$; for $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \frac{1}{2}\text{C}_6\text{H}_6$: triclinic space group $P\bar{1}$ with $a = 5.7064(1)$, $b = 10.9473(1)$, $c = 12.1476(1)$ Å, $\alpha = 81.5366(8)$, $\beta = 86.8528(8)$, $\gamma = 88.0700(5)^\circ$. The selenoxide forms hexamers, the telluroxide forms strong dimers with chalcogen-oxygen interactions.

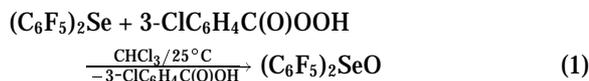
Introduction

As a result from our recent studies on the chemistry of polyfluorophenylselenium and -tellurium compounds we, and others in the past, have been able to determine a variety of crystal structures of tellurium(IV) compounds with $\text{Te} \cdots \text{Hal}$ ($\text{Hal} = \text{F}, \text{Cl}, \text{Br}$) [1–4] and $\text{Te} \cdots \text{N}$ interactions [5]. In contrast, only one structural example is known of fluorophenyl-substituted selenium(IV) compounds, the seleninic acid $\text{C}_6\text{F}_5\text{Se}(\text{O})\text{OH}$ [6]; and moreover, no data are available regarding fluorophenyl-tellurium oxides. In this contribution, we report on the direct synthesis and crystal structures of the bis(pentafluorophenyl) chalcogenoxides of selenium and tellurium.

Results and Discussion

Synthesis and properties

The selenoxide $(\text{C}_6\text{F}_5)_2\text{SeO}$ was originally obtained as a hydrolysis product of the corresponding, rather sensitive selenium(IV) difluoride, $(\text{C}_6\text{F}_5)_2\text{SeF}_2$ [7]. A selective and straightforward synthesis involves the oxidation of $(\text{C}_6\text{F}_5)_2\text{Se}$ with *m*-chloroperbenzoic acid at ambient temperature (eq. (1)).



The oxidation of the tellurium analogue with *m*-chloroperbenzoic acid does not proceed as straightforward. A reaction occurred immediately, faster as in eq. (1), but at 25 °C considerable amounts of pentafluorophenol were detected by ^{19}F NMR, indicating a cleavage of the Te–C bond. Lowering the reaction temperature to –20 °C avoids unwanted formation of $\text{C}_6\text{F}_5\text{OH}$. Further decrease in temperature to –60 °C did not increase the yield (36%). The isolation of $(\text{C}_6\text{F}_5)_2\text{TeO}$ ($\delta^{125}\text{Te} = 1010$) from a mixture with two further $\text{C}_6\text{F}_5\text{Te}$ compounds ($\delta^{125}\text{Te} = 1442$ and 809) is possible, because of its relatively low solubility in chloroform (higher solubility in acetone). The exact identity of the other two compounds is not known at this time. One of them could possibly be the mixed anhydride $(\text{C}_6\text{F}_5)_2\text{Te}(\text{OH})\text{OC}(\text{O})\text{C}_6\text{H}_4\text{Cl}$, similar to the product reported for the reaction of $(\text{C}_6\text{F}_5)_2\text{Te}$ with HNO_3 , which resulted in the formation of $(\text{C}_6\text{F}_5)_2\text{Te}(\text{OH})\text{ONO}_2$ [8]. Treatment of this material with water at *ca.* 50 °C yielded an insoluble material and *m*-chlorobenzoic acid.

The telluroxide $(\text{C}_6\text{F}_5)_2\text{TeO}$ is thermally not stable; in a solution in CDCl_3 at 60 °C the resonances of

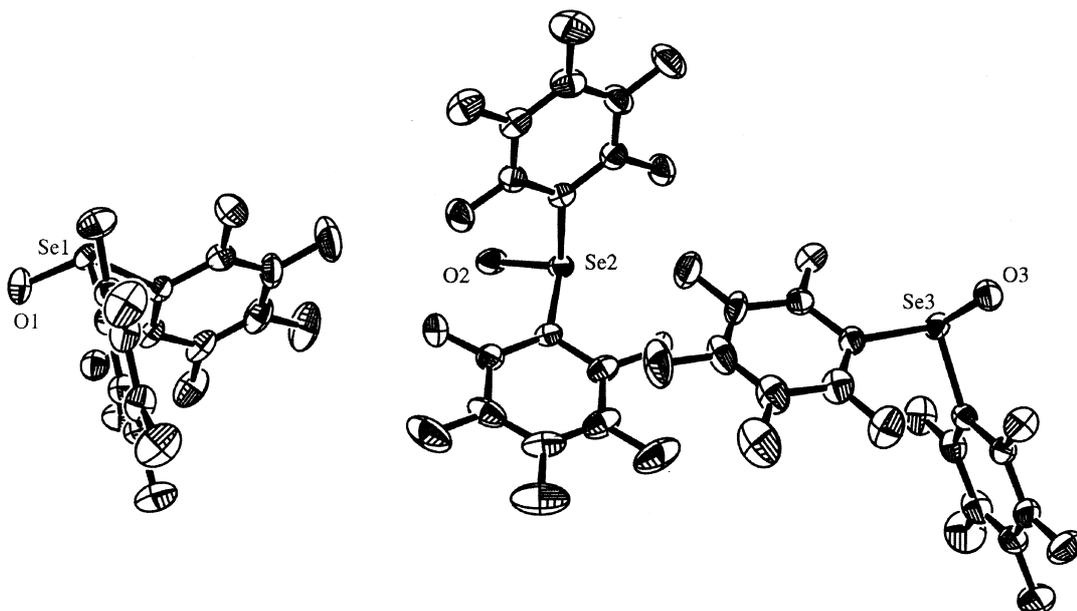


Fig. 1. Molecular structure of the three independent $(\text{C}_6\text{F}_5)_2\text{SeO}$ molecules with thermal ellipsoids at the 50% probability level (only Se and O labeled for clarity). Selected bond lengths and angles (\AA , $^\circ$): Se(1)-O(1) 1.643(3), Se(1)-C(1) 1.954(3), Se(1)-C(7) 1.964(3), Se(2)-O(2) 1.645(3), Se(2)-C(13) 1.974(3), Se(2)-C(19) 1.951(3), Se(3)-O(3) 1.641(3), Se(3)-C(25) 1.956(3), Se(3)-C(31) 1.969(3); O(1)-Se(1)-C(1) 102.5(1), O(1)-Se(1)-C(7) 105.8(1), C(1)-Se(1)-C(7) 95.0(1), O(2)-Se(2)-C(13) 103.8(1), O(2)-Se(2)-C(19) 102.0(1), C(19)-Se(2)-C(13) 97.2(1), O(3)-Se(3)-C(25) 103.6(1), O(3)-Se(3)-C(31) 104.4(1), C(25)-Se(3)-C(31) 97.4(1).

pentafluorobenzene, $\text{C}_6\text{F}_5\text{H}$, were identified in the ^{19}F NMR spectrum after short periods (*ca.* 30 min).

Assignments of the chalcogen oxygen stretching vibrations ν_{SeO} (*ca.* 850 cm^{-1}) and ν_{TeO} (703 cm^{-1}) were made tentatively by comparing the Raman spectra with those of the corresponding precursors, the selenide, the telluride, and in case of $(\text{C}_6\text{F}_5)_2\text{TeO}$, also chloroform.

The oxidation to the chalcogenoxides causes the expected low-field shift of the $^{77}\text{Se}/^{125}\text{Te}$ NMR resonances (Table 1). The multiplicity of the selenide and telluride resonances changes from quintets to multiplets or to broadened resonances at $25\text{ }^\circ\text{C}$ due to inequivalent pentafluorophenyl groups in the chalcogenoxides.

Crystal structures

Single crystals of $(\text{C}_6\text{F}_5)_2\text{SeO}$ (Figs. 1 and 2) were obtained by evaporation of ethereal solutions. Crystals of $(\text{C}_6\text{F}_5)_2\text{TeO}$ formed slowly by hydrolysis out of $(\text{C}_6\text{F}_5)_2\text{Te}(\text{CN})_2$ [4] solutions in either dichloromethane (Fig. 3) or benzene (Fig. 4). The chloroform solvate is also crystalline and suitable

Table 1. ^{77}Se and ^{125}Te NMR shifts of bis(pentafluorophenyl) chalcogenides and chalcogenoxides (CDCl_3 , $25\text{ }^\circ\text{C}$).

	$\delta\ ^{77}\text{Se}$	$\delta\ ^{125}\text{Te}$
$(\text{C}_6\text{F}_5)_2\text{Ch}$	122 [6]	305 [3]
$(\text{C}_6\text{F}_5)_2\text{ChO}$	916	1010

for an X-ray study as well, but was not pursued further, since its structure will likely be similar to that of the dichloromethane solvate. Quite recently, and simultaneous to our studies, $(\text{C}_6\text{F}_5)_2\text{TeO}$ was reported to be also formed as a crystalline material in the partial hydrolysis of $(\text{C}_6\text{F}_5)_3\text{TeBr}$ in toluene [9].

The molecular structure and selected structural parameters of bis(pentafluorophenyl) selenoxide is shown in Fig. 1. Because of the three independent molecules in the unit cell, the values are averaged. The Se-O distance with an average of $1.643(3)\text{ \AA}$ indicates double-bond character, comparable to $1.638(3)\text{ \AA}$ found in $\text{C}_6\text{F}_5\text{SeOOH}\cdot\text{H}_2\text{O}$ (Se-OH $1.741(3)$, Se-C $1.962(4)\text{ \AA}$ [6]). The Se-C distances are on average 1.96 \AA and similar to data for $\text{C}_6\text{F}_5\text{SeOOH}\cdot\text{H}_2\text{O}$. One pentafluorophenyl sub-

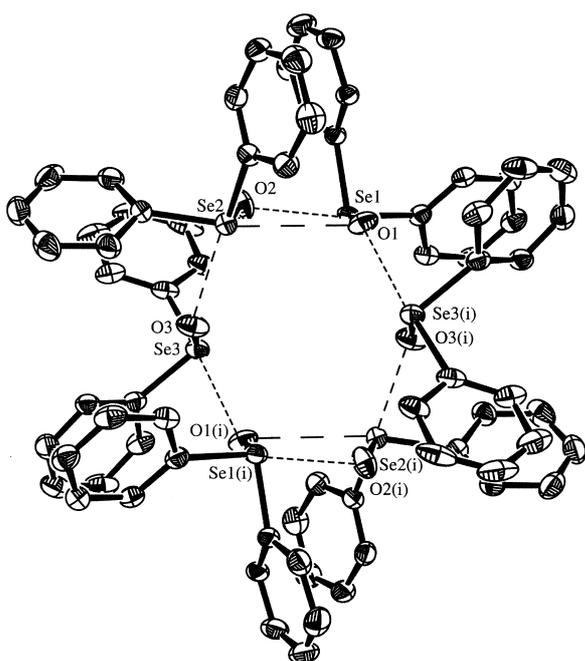


Fig. 2. Hexameric units of $(\text{C}_6\text{F}_5)_2\text{SeO}$ (fluorine atoms omitted and only Se and O labeled for clarity). Selected distances and angles (\AA , $^\circ$): Se(1) \cdots O(2) 2.595(2), O(1) \cdots Se(2) 3.201(3), Se(2) \cdots O(3) 2.639(2), O(1) \cdots Se(3*i*) 2.595(2); O(1)-Se(1) \cdots O(2) 87.3(1), Se(1)-O(1) \cdots Se(3*i*) 123.6(1), C(1)-Se(1) \cdots O(2) 77.1(1), Se(1)-O(1) \cdots Se(2) 89.51(9), C(7)-Se(1) \cdots O(2) 166.0(1), with $i = -x, -y, -z$.

stituent is located in the axial and one in the equatorial position. The axial distances ($\text{Se}-\text{C}_{\text{ax}} = 1.97 \text{ \AA}$) are slightly longer than the equatorial distances ($\text{Se}-\text{C}_{\text{eq}} = 1.95 \text{ \AA}$).

Six molecules of $(\text{C}_6\text{F}_5)_2\text{SeO}$, shown in Fig. 2, form hexamers (star-shaped) linked by three different types of Se \cdots O interactions (2.595(3), 2.639(2) and 3.201(3) \AA), which are shorter than the sum of the van der Waals radii of selenium and oxygen (3.42 \AA [10]). This leads to either penta- (Se1, Se3) or hexa-coordination (Se2) of selenium. The coordination around the Se^{IV} centers can be described as either a distorted trigonal bipyramid or a pseudo-octahedron, with the selenium lone pair of electrons occupying the fifth or sixth (axial) position, respectively.

The structures of bis(pentafluorophenyl) telluroxide, obtained as a solvate with dichloromethane or benzene, are shown in Figs. 3 and 4. The structures are similar and consist of isolated Te_2O_2 dimers

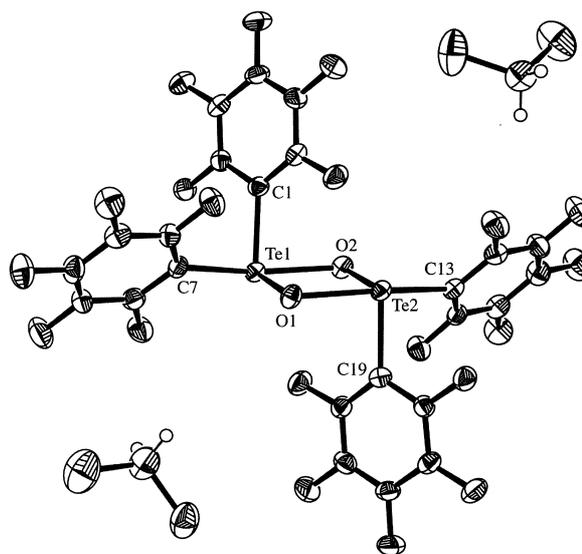


Fig. 3. Molecular structure of $(\text{C}_6\text{F}_5)_2\text{TeO}\cdot\text{CH}_2\text{Cl}_2$ with thermal ellipsoids at the 50% probability level (only Te, O and TeC labeled for clarity). Selected bond lengths and angles (\AA , $^\circ$): Te(1)-O(1) 1.872(2), Te(1)-O(2) 2.223(2), Te(1)-C(1) 2.135(4), Te(1)-C(7) 2.194(4), Te(2)-O(1) 2.244(2), Te(2)-O(2) 1.872(2), Te(2)-C(13) 2.195(4), Te(2)-C(19) 2.127(4), Te(1) \cdots Te(2) 3.2471(3); O(1)-Te(1)-O(2) 76.1(1), O(1)-Te(1)-C(1) 106.3(1), O(1)-Te(1)-C(7) 87.8(1), O(1)-Te(2)-O(2) 75.5(1), O(1)-Te(2)-C(13) 160.9(1), O(1)-Te(2)-C(19) 86.9(1), C(1)-Te(1)-C(7) 88.2(1), C(1)-Te(1)-O(2) 84.9(1), C(7)-Te(1)-O(2) 159.9(1), C(13)-Te(2)-C(19) 89.4(1), C(13)-Te(2)-O(2) 87.4(1), C(19)-Te(2)-O(2) 104.7(1), Te(1)-O(1)-Te(2) 103.8(1), Te(1)-O(2)-Te(2) 104.6(1).

with an approximate trigonal bipyramidal coordination of tellurium (including the nonbonding electron pair at Te), with no further Te \cdots O interactions shorter than the sum of the van der Waals radii of tellurium and oxygen (3.58 \AA [10]). As already found in $(\text{C}_6\text{F}_5)_2\text{SeO}$, one pentafluorophenyl substituent is located in an axial and one in an equatorial position. Each tellurium atom has one shorter (*ca.* 1.87 \AA) and one longer (*ca.* 2.22 \AA) bond distance to oxygen. Rather short Te \cdots Te distances are found in both structures, 3.2471(3) and 3.2238(4) \AA . Additional short Te \cdots F interactions (sum of the van der Waals radii of tellurium and fluorine 3.53 \AA [10]) exist between a tellurium atom and one *ortho*-fluorine atom of the pentafluorophenyl group of the second molecule, which are located in *trans* positions to the Te_2O_2 planes (3.015(2)/3.029(2) and 3.007(2) \AA). In the dichloromethane solvate structure one of the two molecules of CH_2Cl_2 is disordered.

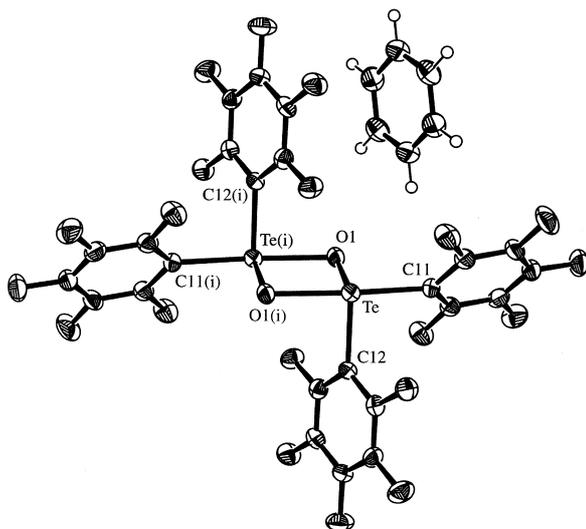


Fig. 4. Molecular structure of $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \frac{1}{2}\text{C}_6\text{H}_6$ with thermal ellipsoids at the 50% probability level (only Te, O and TeC labeled for clarity). Selected bond lengths and angles (\AA , $^\circ$): Te-O(1) 1.875(3), Te-O(1(i)) 2.214(2), Te-C(11) 2.201(4), Te-C(12) 2.136(4), Te...Te(i) 3.2238(4); O(1)-Te-O(1(i)) 76.2(1), O(1)-Te-C(11) 86.8(1), O(1)-Te-C(12) 105.5(1), C(11)-Te-C(12) 91.2(1), O(1(i))-Te-C(11) 161.6(1), O(1(i))-Te-C(12) 86.5(1), Te-O(1)-Te(i) 103.8(1), with $i = 2 - x, 1 - y, -z$.

A similar structural type is reported for the toluene solvate of bis(pentafluorophenyl) telluroxide [9] and diphenyl telluroxide [11], which are dimers. The influence of the less strongly electron-withdrawing nature of phenyl compared to pentafluorophenyl is documented in weaker secondary bonds of tellurium to oxygen (2.55 \AA in $(\text{C}_6\text{H}_5)_2\text{TeO}$ vs. 2.22 \AA). A comparison of the Te-C distances in $(\text{C}_6\text{F}_5)_2\text{TeO}$ and $(\text{C}_6\text{H}_5)_2\text{TeO}$ shows a larger difference in bond lengths of Te- C_{ax} and Te- C_{eq} . Whereas for $(\text{C}_6\text{F}_5)_2\text{TeO}$ a larger distance for Te- $C_{\text{ax}} = 2.20 \text{ \AA}$ (Te- $C_{\text{eq}} = 2.13 \text{ \AA}$) is observed, for $(\text{C}_6\text{H}_5)_2\text{TeO}$ almost identical distances (Te- $C_{\text{ax}} = 2.14 \text{ \AA}$; Te- $C_{\text{eq}} = 2.13 \text{ \AA}$) were reported.

The structure of the lighter homologue diphenyl sulfoxide, $(\text{C}_6\text{H}_5)_2\text{SO}$, is reported, but apparently no sulfur oxygen interactions exist [12].

Experimental Section

Bis(pentafluorophenyl) selenide [6] / telluride [3] and bis(pentafluorophenyl)tellurium(IV) dicyanide [4] were obtained by reported procedures. *m*-Chloroperbenzoic acid (77% max.) is commercially available (Aldrich).

Infrared spectra were recorded as KBr pellets on a Nicolet 520 FT-IR spectrometer, Raman spectra on a Perkin-Elmer Spectrum 2000 NIR FT-Raman spectrometer (100 mW). The NMR spectra were obtained on a Jeol 400 Eclipse instrument using CDCl_3 as solvent at 25 $^\circ\text{C}$. The chemical shifts are with respect to $(\text{CH}_3)_4\text{Si}$ (^1H , 400.18 MHz; ^{13}C , 100.63 MHz), CFCl_3 (^{19}F , 376.55 MHz), $(\text{CH}_3)_2\text{Se}$ (^{77}Se , 76.36 MHz) and $(\text{CH}_3)_2\text{Te}$ (^{125}Te , 126.26 MHz). The mass spectrum was recorded on a Finnigan MAT 95Q spectrometer.

For $(\text{C}_6\text{F}_5)_2\text{SeO}$ a Stoe IPDS area detector was employed for data collection with Mo- K_α radiation, for $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \text{CH}_2\text{Cl}_2$ and $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \frac{1}{2}\text{C}_6\text{H}_6$ a Nonius Kappa CCD was used. The structures were solved by direct methods SIR97 [13] and were refined by means of the full-matrix least squares procedures using SHELXL97 [14] (Table 2). All non-hydrogen atoms were refined anisotropically. Further details are available under the depository numbers CCDC-173658, 173659, 173660 from the Cambridge Crystallographic Data Centre.

Bis(pentafluorophenyl) selenoxide $(\text{C}_6\text{F}_5)_2\text{SeO}$. A solution of 1.5 mmol of *m*-chloroperbenzoic acid in 5 ml of chloroform was added to a solution of 1.0 mmol of $(\text{C}_6\text{F}_5)_2\text{Se}$ in 3 ml of chloroform and the mixture stirred for 24 h at 25 $^\circ\text{C}$. After removal of the solvent the by-product *m*-chlorobenzoic acid was separated by sublimation at 40 $^\circ\text{C}/0.01$ mbar. The residue was purified by repeated slow sublimation at 60 - 70 $^\circ\text{C} / 0.01$ mbar, and the crude product recrystallized from diethyl ether to give the selenoxide in 70% yield as colorless crystals.

M. p. 152 - 154 $^\circ\text{C}$. - IR (KBr): $\nu = 1639$ m, 1590 w, 1520 s, 1499 s, 1422 w, 1400 m, 1388 m, 1337 w, 1287 m, 1264 w, 1150 w, 1088 s, 1049 w, 1013 m, 1000 m, 982 s, 977 s, 859 s/ 849 m (ν_{SeO}), 798 w, 750 w, 724 w, 626 w, 612 w, 583 w, 492 w, 395 w, 369 w, 355 w, 324 w, 312 w, 272 m cm^{-1} . - Raman: $\nu = 1638$ (26), 1517 (4), 1441 (3), 1400 (4), 1283 (5), 1145 (7), 1093 (12), 981 (3), 851 (52)/844 (74, ν_{SeO}), 800 (14), 720 (4), 616 (9), 586 (58), 493 (100), 443 (48), 390 (50), 349 (35), 327 (17), 282 (30), 262 (30), 242 (33), 224 (68), 195 (38), 180 (25), 151 (17), 118 (20) cm^{-1} . - ^{19}F NMR: $\delta = -137.3$ (m, 2F, 2-F), -145.0 (tt, 1F, 4-F, $^3J_{\text{F-F}} = 19.9$ Hz, $^4J_{\text{F-F}} = 4.3$ Hz), -157.3 (m, 2F, 3-F). - $^{13}\text{C}\{^{19}\text{F}\}$ NMR: $\delta = 145.8$ (C-2, $J_{\text{C-Se}} = 12.7$ Hz), 144.2 (C-4), 137.8 (C-3), 113.9 (C-1, $J_{\text{C-Se}} = 159.5$ Hz). - ^{77}Se NMR: $\delta = 916$ (m). - $\text{C}_{12}\text{F}_{10}\text{OSe}$ (429.1): calcd C 33.6; found C 33.7. Mass spectrum and ^{19}F NMR spectrum in CH_3CN see [7].

Bis(pentafluorophenyl) telluroxide chloroformate $(\text{C}_6\text{F}_5)_2\text{TeO} \cdot \text{CHCl}_3$. Into a solution of 3.0 mmol of $(\text{C}_6\text{F}_5)_2\text{Te}$ in 10 ml of chloroform was added a solution of 3.0 mmol of *m*-chloroperbenzoic acid at -20 $^\circ\text{C}$ within 45 min. Immediate decolorization from pale yellow occurred and a colorless precipitate formed. The precipi-

Table 2. Crystal data and structure refinements.

	(C ₆ F ₅) ₂ SeO	(C ₆ F ₅) ₂ TeO · CH ₂ Cl ₂	(C ₆ F ₅) ₂ TeO · 1/2 C ₆ H ₆
Empirical formula	C ₇₂ F ₆₀ O ₆ Se ₆	C ₂₆ H ₄ Cl ₄ F ₂₀ O ₂ Te ₂	C ₃₀ H ₆ F ₂₀ O ₂ Te ₂
Formula weight	2574.48	1125.29	1033.54
Temperature [K]	200(3)	200(2)	200(2)
Crystal size [mm]	0.48 × 0.35 × 0.22	0.23 × 0.11 × 0.04	0.44 × 0.05 × 0.05
Crystal system	triclinic	monoclinic	triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> $\bar{1}$
<i>a</i> [Å]	9.9452(9)	18.1918(2)	5.7064(1)
<i>b</i> [Å]	14.871(2)	7.7545(1)	10.9473(1)
<i>c</i> [Å]	15.279(2)	23.1719(3)	12.1476(1)
α [°]	117.17(1)		81.5366(8)
β [°]	101.02(1)	97.9523(4)	86.8528(8)
γ [°]	95.39(1)		88.0700(5)
<i>V</i> [Å ³]	1930.4(3)	3237.39(7)	749.21(2)
<i>Z</i>	1	4	1
ρ (calcd.) [g/cm ³]	2.215	2.309	2.291
μ_{Mo} [mm ⁻¹]	3.045	2.277	2.104
<i>F</i> (000)	1224	2112	486
θ range [°]	2.28 - 25.92	2.75 - 27.47	1.70 - 27.51
Index ranges	-12 ≤ <i>h</i> ≤ 12 -18 ≤ <i>k</i> ≤ 18 -18 ≤ <i>l</i> ≤ 18	-23 ≤ <i>h</i> ≤ 23 -10 ≤ <i>k</i> ≤ 9 -30 ≤ <i>l</i> ≤ 30	-7 ≤ <i>h</i> ≤ 7 -14 ≤ <i>k</i> ≤ 12 -15 ≤ <i>l</i> ≤ 15
Reflections collected	15165	68010	15182
Independent reflections	6981 (<i>R</i> _{int} = 0.0539)	7400 (<i>R</i> _{int} = 0.0660)	3399 (<i>R</i> _{int} = 0.0435)
Observed reflections	5544	5924	3039
Data/restraints/parameters	6981/0/649	7400/0/497	3399/0/244
Goodness-of-fit on <i>F</i> ²	0.972	1.059	1.214
<i>R</i> 1, <i>wR</i> 2 (<i>I</i> > 2σ(<i>I</i>))	0.0326, 0.0779	0.0358, 0.0807	0.0290, 0.0376
<i>R</i> 1, <i>wR</i> 2 (all data)	0.0448, 0.0816	0.0523, 0.0878	0.0376, 0.1011
Larg. diff. peak/hole [e/Å ³]	0.643/-0.580	0.858/-1.138	1.024/-1.543

tate was separated from the cold solution and washed with small amounts of cold chloroform. The precipitate is moderately soluble in chloroform and was shown to be pure (C₆F₅)₂TeO. The residue of the filtrate is a mixture of two further unidentified C₆F₅Te compounds. The yield of (C₆F₅)₂TeO as a chloroform solvate (colorless crystals) is 36% (dec. ~60 °C).

IR (KBr): ν = 2998 w, 1637 m, 1612 w, 1582 w, 1553 w, 1517 s, 1487 s, 1476 s, 1388 m, 1370 m, 1280 m, 1145 w, 1082 s, 1009 m, 971 s, 797 m, 782 w, 756 m, 710 m, 619 m, 583 w, 486 w, 398 m, 374 m, 350 w, 339 w cm⁻¹. – Raman: ν = 3001 (12), 1639 (16), 1512 (4), 1279 (9), 1135 (5), 1082 (18), 781 (16), 760 (10), 703 (72, ν_{TeO}), 667 (24), 648 (10), 620 (9), 584 (56), 489 (100), 444 (47), 393 (30), 380 (23), 367 (27), 348 (28), 280 (23), 262 (28), 233 (25), 204 (32), 166 (41), 124 (11) cm⁻¹. – ¹H NMR: δ = 7.24 (CHCl₃). – ¹⁹F NMR: δ = -131.8 (br, 2F, 2-F),

-147.4 (t, 1F, 4-F, ³*J*_{F-F} = 19.9 Hz), -157.5 (m, 2F, 3-F). – ¹³C {¹⁹F} NMR: δ = 146.8 (C-2, *J*_{C-Te} = 32.3 Hz), 143.4 (C-4), 137.6 (C-3), 113.0 (C-1, *J*_{C-Te} = 393 Hz), 77.2 (CHCl₃, ¹*J*_{C-H} = 209.1 Hz). – ¹²⁵Te NMR: δ = 1010 (br). – EI MS (70 eV) *m/z* (refers to [(C₆F₅)₂TeO]₂ and ¹³⁰Te, %): 793 (2) [M⁺-C₆F₅], 777 (1) [M⁺-C₆F₅-O], 631 (71) [(C₆F₅)₃Te⁺], 483 (4) [(C₆F₅)₂TeF⁺], 464 (83) [(C₆F₅)₂Te⁺], 334 (21) [(C₆F₅)₂Te⁺], 297 (100) [C₆F₅Te⁺], 167 (7) [C₆F₅⁺], 130 (12) [Te⁺]. – C₁₃HCl₃F₁₀OTe (597.1): calcd C 26.1, H 0.2, Cl 17.8; found C 26.5, H 0.3, Cl 14.8.

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