Synthesis, Structures and Properties of CF_3S-Substituted Tellurium Compounds

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New preparations for the following compounds are described: $Te(SCF_3)_2$, $Te(NSO)_2$, and $Cl_2Te(NSO)_2$. The catalytic or thermal SO_2 elimination from $Cl_2Te(NSO)_2$ affords a coordinated or non-coordinated thiachalcogenadiazole. Conversions of these compounds are reported. Evidence for the proposed mechanism for the reaction between $Te(NSO)_2$ and $SbCl_5$ is provided. $Cl_4Te_2N_2(SCF_3)_2$, prepared from $TeCl_4$ and $CF_3SN(SiMe_3)_2$, reacts with $LiN(SiMe_3)_2$ to form $[(Me_3Si)_2N]_4Te_2N_2(SCF_3)_2$. An X-ray structural analysis of $Cl_4Te_2N_2(SCF_3)_2$ is presented.

N-Sulfinyl compounds are useful building blocks for directed synthesis of sulfur-nitrogen heterocycles in organic[1,2] and inorganic[3–6] chemistry. Bis(sulfinylamido)tellurium[7] (**1**), a highly promising synthon, has so far only been subjected to reaction with $SbCl_5$ to form an oligomeric ring system $[\overline{\text{SNSNTe}}\text{Cl} \cdot \text{Cl} \cdot \text{SbCl}_3]_n$ in about 1% yield[5]. The potential of $Te(NSO)_2$ as a starting material has not been widely investigated, as it is very sensitive to hydrolysis, thermally rather unstable and not easily accessible. Therefore, a more efficient method [see eq. (1)[7]] for preparing **1** was necessary.

$$(tBuMe_2Si)_2Te + 2\,ClNSO \longrightarrow \underset{\mathbf{1}}{Te(NSO)_2} + S(NSO)_2 + TeCl_4 + 2\,tBuMe_2SiCl \qquad (1)$$

A synthon of high potential for the synthesis of **1** is bis(trifluoromethylthio)tellurium (**2**). So far, it has only poorly been characterized[8] and prepared on a milligram scale. Higher yields and preparative amounts are obtained according to the reactions demonstrated in eq. (2–4). The yellow liquid has an unpleasant smell, decomposes in the air and under the influence of light with deposition of tellurium. It is soluble in CH_2Cl_2, $CHCl_3$, THF, CS_2 as well as in aliphatic and aromatic hydrocarbons. In SO_2 and alcohols decomposition takes place. Dissolved in benzene or CS_2 with the exlusion of light **2** is stable at 60°C during several weeks and a suitable starting material for the preparation of **1**.

According to the element displacement principle[9] the CF_3S group can be considered a parachlorine (pCl) and NSO a parafluorine (pF), so that $Te(SCF_3)_2$ and $Hg(NSO)_2$ should react according to $Te(pCl)_2 + Hg(pF)_2 \rightarrow Te(pF)_2 + Hg(pCl)_2$.

When $Te(SCF_3)_2$ and $Hg(NSO)_2$ are allowed to react in CS_2 metathesis takes place as postulated, and **1** is formed in 60% yield according to eq. (5). It is slightly soluble in CH_2Cl_2 and CS_2 but sparing in CH_3NO_2. An oxidative halogenation takes place when **2** is treated with ClNSO(ClpF) to yield bis(sulfinylamido)tellurium(IV) chloride (**3**) as shown in eq. (6). It is also accessible directly from tellurium and ClNSO [see eq. (7)].

$$TeCl_4 + 4\,Hg(SCF_3)_2 \xrightarrow{\text{no solvent}} 4\,CF_3SHgCl + (CF_3S)_2 + \underset{\mathbf{2}}{Te(SCF_3)_2} \qquad (2)$$

$$Na_2Te + 2\,CF_3SCl \xrightarrow{\text{ether}} \mathbf{2} + 2\,NaCl \qquad (3)$$

$$Na_2Te + 2(C_6H_5)_3SnCl \longrightarrow Te[Sn(C_6H_5)_3]_2 \xrightarrow{2\,CF_3SCl} \mathbf{2} + 2\,(C_6H_5)_3SnCl \qquad (4)$$

$$\mathbf{2} + Hg(NSO)_2 \xrightarrow{CS_2} \underset{\mathbf{1}}{Te(NSO)_2} + Hg(SCF_3)_2 \qquad (5)$$

$$\mathbf{2} + 2\,ClNSO \xrightarrow{CS_2} \underset{\mathbf{3}}{Cl_2Te(NSO)_2} + (CF_3S)_2 \qquad (6)$$

$$Te + 2\,ClNSO \xrightarrow{CS_2} \mathbf{3} \qquad (7)$$

With traces of water hydrolysis of **3** to a yellow-coloured, not identified material occurs. Spontaneous deposition of tellurium is observed when **3** comes in contact with air. In toluene it is slightly soluble with slow evolution of SO_2 at 20°C. A close relationship between **1**, **2** and **3** has been found. It is possible to chlorinate **1** in CS_2 under mild conditions without breaking the Te–N bond to **3** which can be

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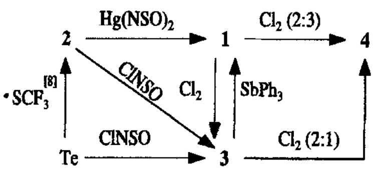
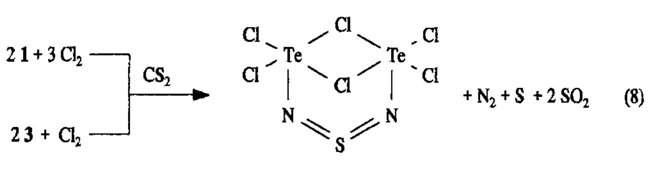
dehalogenated with $Sb(C_6H_5)_3$ to **1** as illustrated in Scheme 1.

Scheme 1



When **1** or **3** are chlorinated with stoichiometrical amounts of Cl_2 according to eq. (8) di-μ-chlorotetrachloro-1κ²*Cl*,2κ²*Cl*-μ-sulfurdiimidato(2−)-1κ*N*,2κ-*N'*-ditellurium(IV) (**4**), SO_2, N_2, and sulfur are obtained. Compound **4** was also obtained by metathesis of $TeCl_4$ and $(CH_3)_3SiNSO$ in CH_2Cl_2[10]. This reaction is solvent-dependent. If this procedure is carried out in $(CH_3)_3SiOSi(CH_3)_3$ besides **4** less than 10% of **3**[11] are formed as a byproduct.



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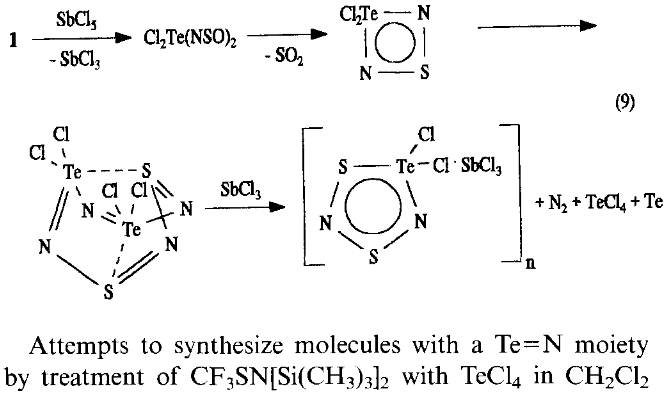
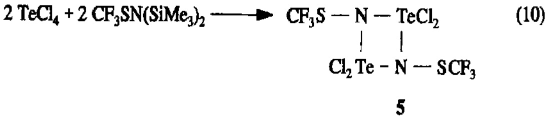
The formation of $[\overline{SNSNTe}Cl \cdot Cl \cdot SbCl_3]_n$[5] from **1** and $SbCl_5$ is postulated to occur by chlorination of **1** with $SbCl_5$ and elimination of SO_2 to provide an unstable intermediate $Cl_2\overline{TeNSN}$, dimerizing to another, cage-shaped intermediate, which decomposes in the presence of $SbCl_3$ to afford the oligomer $[\overline{SNSNTe}Cl \cdot Cl \cdot SbCl_3]_n$, N_2, $TeCl_4$, and Te[5]. With the isolation and characterization of **3** and its reaction with $SbCl_3$ to the oligomer, it can be stated that chlorination of **1** is the first reaction step followed by elimination of SO_2 according to eq. (9). The byproducts SO_2 and N_2 were detected by IR and mass spectrometry. Additionally, $TeCl_4$ was extracted with CH_2Cl_2 from the solid residue and characterized by its mass spectrum. These results are summarized in eq. (9). Pyrolysis of **3** at 95°C yielded $Cl_2\overline{TeNSNS}$[12], SO_2, N_2, and $TeCl_4$, but formation of $[\overline{SNSNTe}Cl \cdot Cl \cdot SbCl_3]_n$ from $Cl_2\overline{TeNSNS}$ and $SbCl_3$ could not be accomplished.

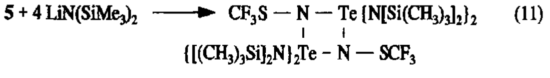


Attempts to synthesize molecules with a Te=N moiety by treatment of $CF_3SN[Si(CH_3)_3]_2$ with $TeCl_4$ in CH_2Cl_2 failed. The yellow-coloured solid 1,1,3,3-tetrachloro-2,4-bis(trifluoromethylthio)-1λ⁴,3λ⁴,2λ³,4λ³-ditelluradiazetidine (**5**), soluble in benzene, toluene and tetrahydrofuran, is very sensitive to water. The mass spectrum exhibits the fragment $CF_3SNTeCl_2^+$ (*m/z* = 315) as the largest ion, but an X-ray single crystal structure analysis reveals a dimeric Te_2N_2 ring arrangement according to eq. (10). With $LiN[Si(CH_3)_3]_2$ a total replacement of chlorine is observed yielding 1,1,3,3-tetrakis[bis(trimethylsilyl)amido]-2,4-bis(trifluoromethylthio)-1λ⁴,3λ⁴,2λ³,4λ³-ditelluradiazetidine(**6**). The first $[(CH_3)_3Si]_2N$ substituted Te(IV) compound is a dark yellow viscous oil, soluble in benzene, hexane, CH_2Cl_2 and $CHCl_3$ which decomposes spontaneously in air with elimination of tellurium.



$$2\,TeCl_4 + 2\,CF_3SN(SiMe_3)_2 \longrightarrow \begin{array}{c} CF_3S-N-TeCl_2 \\ |\quad\quad| \\ Cl_2Te-N-SCF_3 \end{array} \qquad (10)$$

5



$$\mathbf{5} + 4\,LiN(SiMe_3)_2 \longrightarrow \begin{array}{c} CF_3S-N-Te\{N[Si(CH_3)_3]_2\}_2 \\ |\quad\quad| \\ \{[(CH_3)_3Si]_2N\}_2Te-N-SCF_3 \end{array} \qquad (11)$$

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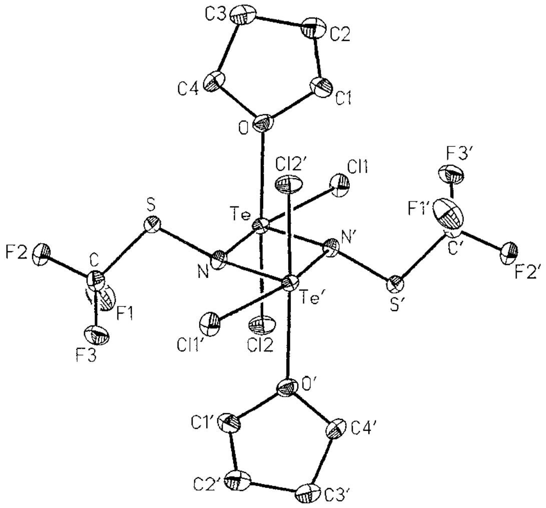
A single crystal of **5**, solvated with 2 THF, was examined by X-ray crystallography and its structure (C_i symmetry) is shown in Figure 1. It consists of a planar, four-membered Te_2N_2 ring with a distorted pseudooctahedral geometry of the form OCl_2NTeE (E = free electron pair) at the tellurium atom. The ligands Cl1, Cl2, N, and O occupy equatorial, N' and E apical positions of the octahedron. Apical *d*(Te−N') = 2.017(3) Å is slightly shorter than equatorial *d*(Te−N) = 2.130(2) Å, probably due to a *trans* effect. The nitrogen atoms exhibit a distorted pyramidal structure (sum of angles 346.1°). A π-delocalization of the free electron pairs of Te and N can be ruled out. Different Te−Cl bond lengths *d*(Te−Cl1) = 2.511(1) Å and *d*(Te−Cl2) = 2.373(1) Å may also be explained by the *trans* effect. But both are significantly shorter than *d*(Te−Cl) of bridging chloro atoms in $(TeCl_4)_4$[21] and can be considered typical Te−Cl single bond lengths. The distance *d*(Te−O) = 2.448(3) Å is characteristic of a coordinative Te−O interaction.

Figure 1. Ellipsoid plot (50%) of **5**



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Experimental

All reactions were carried out on a vacuum line or in a glove box (Fa. M. Braun GmbH, München) under argon (dried with Sicapent); solvents were dried according to published procedures[13]. All solvents were dried and transferred from activated 4-Å molecular sieves. – Microanalyses were performed with a Carlo-Erba Elemental Analyser, Model 1106. – IR: Bruker IFS 66 FT, solids as KBr discs, liquids as capillary films and gases in a 10 cm cell with KBr windows. Very weak bands and shoulders are not recorded. – Ra: Bruker IFS 66 FT with Raman divice FRA 106 (Neodym-YAG-Laser 1064 nm), solids in sealed glass cappillaries. – MS: Varian MAT CH7, 70 eV, emission 100 μA. – NMR: Bruker WP 80 (^1H, ^{19}F), WM 250 (^{13}C, ^{29}Si, ^{125}Te), AM 400 (^{17}O). – Standards: ^1H, ^{13}C, ^{29}Si: TMS, ^{13}C: $CDCl_3$ $\delta = 77.0$, ^{17}O: D_2O (ext.) $\delta = 0$, ^{19}F: $CFCl_3$, ^{125}Te: $(CH_3)_2Te$ (ext.) $\delta = 0$.

The compounds $Hg(SCF_3)_2$[14], CF_3SCl[15], Na_2Te[16], $Hg(NSO)_2$[17], ClNSO[18], $CF_3SN[Si(CH_3)_3]_2$[19], $LiN[Si(CH_3)_3]_2$[20] were prepared according to literature methods. All other substances are commercially available and were used without further treatment.

X-Ray Crystal-Structure Determination[22] *of* **5** · 2 THF *at −145°C:* $C_{10}H_{16}Cl_4F_6N_2O_2S_2Te_2$, yellow crystals. 0.34 × 0.24 × 0.19 mm, triclinic, space group $P\bar{1}$, $a = 7.820(2)$, $b = 7.838(2)$, $c = 9.610(2)$ Å, $\alpha = 83.98(2)°$, $\beta = 79.18(2)°$, $\gamma = 83.14(2)°$, $V = 572.3(2)$ Å^3, $Z = 1$, $\rho_{calcd.} = 2.238$ g · cm^{-3}, $\mu(Mo\text{-}K_\alpha) = 32.6$ cm^{-1}, $\lambda = 0.71069$ Å, Nicolet-R3 m/V diffractometer, 2034 unique intensities, 1984 considered observed. $[F_o \geq 4.0\sigma(F)]$, solution by direct methods, empirical absorption correction, 128 parameters refined, $R = 0.0232$, $R_w = 0.0280$, $[w^{-1} = \sigma^2(F) + 0.0005\ F^2]$, largest difference peak 0.60 e · Å^{-3}, program SHELXTL PLUS.

Bis(triphenylstannyl)tellurium: To a suspension of 8.68 g (50 mmol) of Na_2Te in 30 ml of THF a solution of 38.5 g (100 mmol) of $(C_6H_5)_3SnCl$ in 150 ml THF was dropped with vigorous stirring at boiling temp. Refluxing and stirring were continued for another 2 h. After addition of 200 ml of benzene NaCl precipitated which was separated by filtration by use of a sintered glass frit. The filtrate was evaporated in vacuo to dryness and the colourless solid residue was dried at 70°C (24 h) in vacuo and was analytically pure. Yield 38.7 g (93.5%), m.p. 151–152°C (ref.[29] 149–150°C). – MS (110°C), *m/z* (%): 828 (6) [M^+], 751 (4) [M^+ – Ph], 597 (2) [M^+ – 3 Ph], 477 (7) [$TeSnPh_3^+$], 351 (100) [$SnPh_3^+$], 274 (16) [$SnPh_2^+$], 197 (50) [$SnPh^+$], 154 (55) [Ph_2^+], 120 (32) [Sn^+], 77 (43) [Ph^+]. – $C_{36}H_{30}Sn_2Te$ (827.7): calcd. C 52.2, H 3.7; found C 52.2, H 3.5.

Bis(trifluoromethylthio)tellurium (**2**). – a) *According to eq. (1):* 1.0 g (3.7 mmol) of $TeCl_4$ and 6.0 g (14.9 mmol) of $Hg(SCF_3)_2$ were placed in a 50-ml Carius tube by means of a Young valve and Teflon cock under dry argon. The mixture was melted and kept at the melting temp. for 1 h. Volatile components were removed in vacuo and condensed in a trap cooled to −196°C. The condensate was warmed to −10°C and purified by fractional condensation in vacuo into traps kept at −45 and −78°C. Compound **2** was retained at −45°C. Yield: 1.0 g (83%).

b) *According to eq. (2):* 1.32 g (7.6 mmol) of Na_2Te was placed in a 500-ml Carius tube and dried for 12 h (20°C) in vacuo. Afterwards, 300 ml of ether and 2.07 g (15.2 mmol) of CF_3SCl were condensed. With vigorous stirring and exclusion of light, the suspension was warmed to 20°C and stirred for another 4 h. The volatile components were separated by fractional condensation into traps kept at −50 and −196°C. Compound **2** is kept back at −50°C. Yield: 1.25 g (50%).

c) *According to eq. (3):* 8.24 g (10.0 mmol) of $[(C_6H_5)_3Sn]_2Te$ was placed in a 500-ml Carius tube which was evacuated. At −196°C 350 ml of 2-methylbutane and 2.71 g (19.8 mmol) of CF_3SCl were condensed. Reaction and separation were carried out as described under b). Yield: 3.1 g (95%), m.p. −54°C. – IR: $\tilde{\nu} =$ 1166 cm^{-1} (vs), 1112 (s), 1095 (vs), 752 (m), 730 (m). – MS (50°C), *m/z* (%): 332 (13) [M^+], 231 (31) [$TeSCF_3^+$], 162 (25) [TeS^+], 130 (24) [Te^+], 101 (21) [CF_3S^+], 82 (62) [F_2CS^+], 69 (100) [CF_3^+], 63 (63) [FCS^+], 32 (34) [S^+]. – ^{13}C NMR ($CDCl_3$): $\delta = 130.5$ [q, $^1J(C\text{-}F) = 310.9$ Hz, 1 C, CF_3]. – ^{19}F NMR: $\delta(CDCl_3) = -38.1$, $\delta(C_6D_6) = -37.7$ (s, 3 F, CF_3). – ^{125}Te NMR ($CDCl_3$): $\delta = 1219$ [sept, $^3J(Te\text{-}F) = 7.4$ Hz, 1 Te, $TeSCF_3$]. – Ra: $\tilde{\nu} = 991$ cm^{-1} (w), 756 (s), 554 (w), 452 (m), 408 (s), 262 (vvs), 129 (s), 101 (s). – $C_2F_6S_2Te$ (329.8): calcd. C 7.3, S 19.4; found C 7.2, S 19.3.

Bis(sulfinylamido)tellurium (**1**). – a) *From* **2** *and Hg(NSO)₂:* To a suspension of 1.97 g (6.1 mmol) of $Hg(NSO)_2$ in 15 ml of CS_2 2.0 g (6.1 mmol) of **2** was condensed in a 50-ml Carius tube. The mixture was kept at 50°C (5 d) with exclusion of light, then cooled to −30°C and the clear solvent was decanted in an evacuated Schlenk flask. To the residue 15 ml of CS_2 was condensed and the liquid phase was once again decanted at −30°C. The moist residue was dried in vacuo at 20°C (24 h) and then sublimed at 55°C/10^{-3} Torr. Yield: 0.95 g (62%).

b) *From* **3** *and Sb(C₆H₅)₃:* In an apparatus consisting of two bulb-shaped glass vessels equipped with a medium-sintered frit and Teflon-stemmed glass Young valves a mixture of 0.25 g (0.8 mmol) of **3** and 0.27 g (0.8 mmol) of $Sb(C_6H_5)_3$ were placed in one of the bulbs. Into this mixture 30 ml of CS_2 was condensed and the suspension was stirred at 20°C (24 h). The yellow-coloured mixture was filtered in the second bulb and the residue washed 3 times with 20 ml of CS_2. The solvent was removed at 20°C in vacuo and **1** was purified by sublimation at 55°C/10^{-3} Torr. Yield: 0.13 g (65%), m.p. 131–132°C (decomp.), ref.[7] 132°C (decomp.). IR and mass spectra are in complete agreement with those reported in ref.[7] – Ra: $\tilde{\nu} =$ 1194 cm^{-1} (w), 1036 (vvs), 593 (w), 406 (w), 151 (m), 72 (w).

Bis(sulfinylamido)tellurium(IV) Chloride (**3**). – a) *From* **2** *and ClNSO:* In a 50-ml Carius tube 0.92 g (2.8 mmol) of **2**, 15 to 20 ml of CS_2 and 0.60 g (6.2 mmol) of ClNSO were condensed. The reaction vessel was kept at −78°C (12 h) with stirring of the reaction mixture. Volatile components of the slightly yellow-coloured suspension were removed and the solid residue was dried at 20°C (24 h) in vacuo. Yield: 0.88 g (98%).

b) *From Tellurium and ClNSO:* As described under a) 0.76 g (6.0 mmol) of Te and 1.5 g (15.0 mmol) of ClNSO were allowed to react in 15 to 20 ml of CS_2 at 0°C (48 h) with stirring. Yield: 1.92 g (100%).

c) *From* **1** *and Chlorine:* To a stirred suspension of 0.29 g (1.2 mmol) of **2** in 50 ml of CS_2 81.0 mg (1.1 mmol) of Cl_2 was added. The reaction was carried out in a 100-ml Carius tube at 0°C and chlorine diffused from the gaseous into the liquid phase during stirring for 3 d. The solvent was removed in vacuo and the residue dried at 20°C (24 h). Yield: 0.35 g (94%), m.p. 96 to 98°C (decomp.). IR and MS data are in agreement with those reported in ref.[11]. – Ra: $\tilde{\nu} =$ 1214 cm^{-1} (w), 1083 (s), 1070 (m), 606 (s), 407 (vvs), 367 (w), 343 (w), 304 (vs), 263 (vs), 162 (s), 141 (s), 121 (s), 91 (s). – ^{17}O NMR (C_6D_6, toluene): $\delta = 106$ (s, br, 2 O, NSO). – ^{125}Te NMR (C_6D_6, toluene): $\delta = 1456$ (s, 1 Te, Cl_2Te). – $Cl_2N_2S_2O_2Te$ (322.6): calcd. N 8.7, S 19.9; Cl 22.0, found N 8.8, S 19.1, Cl 22.2.

Chlorination of **1** *or* **3** *to* **4**: To 0.20 g (0.8 mmol) of **1** or 0.32 g (1.0 mmol) of **3** 10 ml of CS_2 and 85.0 mg (1.2 mmol) or 46.0 mg (0.65 mmol) of Cl_2 were condensed by using the apparatus described for the preparation of **1** according to b). The mixtures were warmed up to 20°C and stirred for 12 h. After filtration and washing of the residue with CS_2 several times it was dried in vacuo during 24 h. Yields: 0.20 g (95%) or 0.22 g (84%). IR and mass spectra are consistent with those reported in ref.[10].

Pyrolysis of **3**: In the apparatus described above 0.70 g (2.17 mmol) of **3** was heated to 95°C for 12 h. Volatile compounds were removed in vacuo and the residue was powdered and then extracted three times with 20 ml each of CH_2Cl_2. The solvent was removed from the combined extracts and the residue dried in vacuo. Yield: 0.27 g (86%) of $Cl_2\overline{TeNSNS}$[12] and 0.22 g (84%) of $TeCl_4$ recovered from the eluent. Yields were calculated from: $4\ Cl_2Te(NSO)_2 \rightarrow 2\ Cl_2\overline{TeNSNS} + 4\ SO_2 + 2\ N_2 + TeCl_4 + Te$.

Reaction of **3** *with* $SbCl_3$: As already mentioned 0.45 g (1.40 mmol) of **3** and 0.33 g (1.45 mmol) of $SbCl_3$ were stirred in 20 ml of CH_2Cl_2 at 40°C for 10 d. The yellow suspension was filtered and the residue washed twice with 10 ml of CH_2Cl_2 each. The solvent was evaporated from the combined filtrates to afford a dark yellow oil which crystallized to yellow-coloured $[\overline{SNSNTe}Cl \cdot Cl \cdot SbCl_3]_n$. Yield: 0.15 g (21%). Physical and spectroscopical data are in agreement with those presented in ref.[5]

1,1,3,3-Tetrachloro-2,4-bis(trifluoromethylthio)-1λ⁴,3λ⁴,2λ³,4λ³-ditelluradiazetidine (**5**): In the apparatus described already 18.0 g (66.8 mmol) of $TeCl_4$ and 17.5 g (67.0 mmol) of $CF_3SN[Si(CH_3)_3]_2$ were filled in each bulb. Into the one containing the aminosilane 50 ml of CH_2Cl_2 was condensed at −196°C and after warming to 20°C transferred to $TeCl_4$. The mixture was vigorously stirred at 20°C (24 h) and then filtered. The yellow solid was extracted several times with 30 ml of CH_2Cl_2 and the residue obtained by evaporation of the solvent from the combined filtrates was dried in vacuo for 48 h. Yield: 20.3 g (97%). m.p. 274−277°C (decomp.). − IR: $\tilde{\nu}$ = 1160 cm^{-1} (vs), 1105 (vs), 835 (s), 756 (s), 621 (s), 466 (s), 404 (s). − MS (160°C), *m/z* (%): 315 (4) $[Cl_2TeNSCF_3^+]$, 280 (14) $[ClTeNSCF_3^+]$, 266 (7) $[ClTeSCF_3^+]$, 202 (44) $[(CF_3S)_2^+]$, 200 (22) $[TeCl_2^+]$, 176 (4) $[TeNS^+]$, 165 (30) $[TeCl^+]$, 130 (21) $[Te^+]$, 115 (12) $[CF_3SN^+]$, 101 (14) $[CF_3S^+]$, 69 (100) $[CF_3^+]$, 46 (33) $[NS^+]$, 35 (15) $[Cl^+]$. − Ra: $\tilde{\nu}$ = 1888 cm^{-1} (vvs), 923 (m), 757 (w), 613 (w), 427 (w), 328 (vs), 249 (vs), 212 (s), 158 (s), 137 (s), 120 (s), 105 (s). − ^{19}F NMR (C_6D_6, THF): δ = −51.6 (s, 3 F, $NSCF_3$). − ^{13}C NMR (C_6D_6, THF): δ = 134.2 [q, ^1J(C-F) = 316.6 Hz, 1 C, $NSCF_3$]. − ^{125}Te NMR (C_6D_6, THF): δ = 1704 (s, 1 Te, $TeNSCF_3$). − $C_2Cl_4F_6N_2S_2Te_2$ (627.2): calcd. C 3.8, Cl 22.6, N 4.5, S 10.2; found C 3.5, Cl 23.7, N 4.9, S 9.0.

1,1,3,3-Tetrakis[bis(trimethylsilyl)amido]-2,4-bis(trifluoromethylthio)-1λ⁴,3λ⁴,2λ³,4λ³-ditelluradiazetidine (**6**): As mentioned above 2.03 g (3.2 mmol) of **5** and 2.17 g (13.0 mmol) of $LiN[Si(CH_3)_3]_2$ were suspended in one bulb with 100 ml of *n*-hexane. The mixture was warmed from −196°C to 20°C and heated with stirring to 50°C, then kept at this temp. for 24 h. The suspension was filtered and the remaining LiCl washed with *n*-hexane several times. The combined filtrates were evaporated to dryness in vacuo and **6** was dried for 12 h. Yield: 2.95 g (80%). − IR: $\tilde{\nu}$ = 2954 cm^{-1} (s), 2898 (m), 1406 (m), 1250 (vs), 1158 (s), 1112 (s), 844 (br), 779 (s), 758 (s), 703 (s), 674 (s), 631 (m), 619 (m), 520 (s). − Ra: $\tilde{\nu}$ = 2951 cm^{-1} (vvs), 2899 (vvs), 1410 (m), 1262 (m), 1140 (w), 838 (w), 749 (w), 680 (s), 632 (vvs), 555 (w), 518 (w), 395 (s), 352 (w), 324 (w), 190 (vs), 119 (s). − MS (120°C), *m/z* (%): 579 (1) $\{CF_3SN_2Te[N(SiMe_3)_2]_2^+\}$, 478 (3) $\{N_2Te[N(SiMe_3)_2]_2^+\}$, 450 (9) $\{Te[N(SiMe_3)_2]_2^+\}$, 290 (36) $[TeN(SiMe_3)_2^+]$, 275 (50) $[CF_3SNTeN_2^+]$, 160 (4) $[N(SiMe_3)_2^+]$, 147 (21) $[Me_3SiNSiMe_2^+]$, 130 (100) $[Te^+]$, 115 (10) $[CF_3SN^+]$, 101 (20) $[CF_3S^+]$, 73 (100) $[SiMe_3^+]$, 69 (4) $[CF_3^+]$, 59 (17) $[HSiMe_2^+]$, 45 (20) $[SN^+]$, 28 (7) $[Si^+]$. − ^1H NMR (C_6D_6]: δ = 0.23 (s, 72 H, $SiCH_3$). − ^{19}F NMR (C_6D_6): δ = −55.2 (s, 1 F, $NSCF_3$). − ^{13}C NMR (C_6D_6): δ = 5.4 [q, ^1J(C-H) = 118 Hz, 24 C, $SiCH_3$], 131.1 [q, ^1J(C-F) = 314.7 Hz, 1 C, $NSCF_3$]. − ^{29}Si NMR (C_6D_6): δ = 15.7 [m, ^2J(Si-H) = 7.0 Hz, 4 Si, $SiCH_3$]. − ^{125}Te NMR (C_6D_6): δ = 1917 (s, 1 Te, $TeNSCF_3$). − $C_{26}H_{72}F_6N_6S_2Si_8Te_2$ (1126.9): calcd. C 27.7, H 6.4, N 7.5; found C 27.7, H 6.4, N 7.5.

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[22] Further details of the crystal structure investigation are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, on quoting the depository number CSD-401283, the names of the authors, and the journal citation.

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