Azido and Dimethylamino Derivatives of Tellurium Hexafluoride

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It has been established by ^{19}F NMR spectroscopy that TeF_6 and Me_3SiX ($X = N_3$ or NMe_2) react to form compounds TeF_nX_{6-n} ($n = 1$–5). Reaction of TeF_6 with Me_3SiNMe_2 in a 1:2 ratio gave a mixture of $TeF_5(NMe_2)$, *cis*-$TeF_4(NMe_2)_2$ and *fac*-$TeF_3(NMe_2)_3$. With a 1:4 ratio, $TeF_4(NMe_2)_2$ (both isomers), $TeF_2(NMe_2)_4$ (both isomers) and $TeF(NMe_2)_5$ were obtained. Reaction of TeF_6 with Me_3SiN_3 in a 1:1 ratio gave $TeF_5(N_3)$ and *cis*-$TeF_4(N_3)_2$ as the major components with minute quantities of *trans*-$TeF_4(N_3)_2$ and *fac*-$TeF_3(N_3)_3$. A 1:2 reaction initially led to all members of the series except *mer*-$TeF_3(N_3)_3$, with $TeF_5(N_3)$ and *trans*-$TeF_4(N_3)_2$ gradually disappearing. An unidentified tellurium(IV) species appeared in both of the azide reactions and often obscured the signal due to *cis*-$TeF_2(N_3)_4$. With azide ion, TeF_6 gave $[TeF_5]^-$, $[TeF_7]^-$ and nitrogen.

Compounds of general formula TeF_nX_{6-n} ($n = 1$–5) are known only where X is OH[1] or OMe,[2,3] and are prepared either from TeF_6 and water or from TeX_6 and anhydrous HF. Other highly substituted alkoxy species, $TeF_n(OR)_{6-n}$, have also been prepared: *e.g.* R = $MeOCH_2CH_2$ ($n = 1$–4),[3,4] $EtOCH_2CH_2$ ($n = 3$–4),[4] $ClCH_2CH_2$ ($n = 1$–3),[3] $BrCH_2CH_2$ ($n = 1$–4),[3] CF_3CH_2 ($n = 2$–3)[3] or $NCCH_2CH_2$ ($n = 3$–4)[4] and can be obtained from TeF_6 and alcohols, ROH. Introduction of amino ligands (using silyl reagents) only gave the mono- and di-substituted products.[5,6] Identification of all these compounds was made using NMR spectroscopy (^{19}F or ^{125}Te).

In this work we use Me_3SiX ($X = N_3$ or NMe_2) to obtain TeF_nX_{6-n} ($n = 1$–5) in NMR-tube reactions and species were identified using ^{19}F NMR spectroscopy. Only $TeF_5(NMe_2)$ and *cis*-$TeF_4(NMe_2)_2$ have been reported previously.[5] The analysis and assignment of the spectra were accomplished by observing the appearance and disappearance of products, by the multiplet patterns and by the consistency of the data. Unfortunately, ^{125}Te NMR spectra, which would have aided assignment and allowed identification of TeX_6 ($X = N_3$ or NMe_2), were not useful. They showed very broad, low-intensity features, presumably due to the proximity of quadrupolar ^{14}N nuclei. A signal at δ −159, due to Me_3SiF, was observed in all ^{19}F NMR spectra.[7]

Results and Discussion

Of the eight members of the series TeF_nX_{6-n} ($n = 1$–5) only three can be unambiguously identified from their ^{19}F signal multiplicities *viz.* TeF_5X, *cis*-TeF_4X_2 and *mer*-TeF_3X_3. The remaining species all give singlets. Linear functions of the type $\delta_F = pC + qT$, first applied to compounds of tin,[8] have been used to predict the fluorine chemical shifts in octahedral and pseudo-octahedral species from empirical correlations of the shifts with certain substitution parameters. Constants C and T are the *cis* and *trans* substituent chemical shifts (s.c.s), characteristic of a ligand, and p and q are the number of fluorines *cis* and *trans* to the test nucleus, *i.e.* in the case of TeF_nX_{6-n}, $q = 0$ or 1, whilst $p = 0$–4. Such methods have been successfully applied to TeF_nX_{6-n} ($X = OH$,[1a] OMe[2] or OR[3]) and the results for data obtained from the reaction between TeF_6 and Me_3SiNMe_2 in CD_2Cl_2 are shown in Fig. 1. The data are given in Table 1 and a typical spectrum is shown in Fig. 2.

Central to Fig. 1 is the data from the unequivocal assignment of the AB_4 spectrum of $TeF_5(NMe_2)$ and the A_2X_2 spectrum of *cis*-$TeF_4(NMe_2)_2$ where the A_2 shift completes the left hand side parallelogram. Extrapolation of this parallelogram indicated that both the *trans* disubstituted and the *fac* trisubstituted compound should appear at around δ −70. They were found at δ −71 and −66, respectively. Predicted signals (a triplet at δ −60 and a doublet at δ −92) due to *mer*-$TeF_3(NMe_2)_3$ were not observed. The signals for *cis*- and *trans*-$TeF_2(NMe_2)_4$ are predicted to appear at around δ −84 and −100, respectively; they occurred at δ −86 and −99. The $TeF(NMe_2)_5$ signal should occur near δ −92 and was found at δ −93. The assignments here are relatively straightforward because there are substantial, although equal, s.c.s. values of opposite sign. The parallelograms are not perfectly symmetric and show some variation in the underlying s.c.s. values.

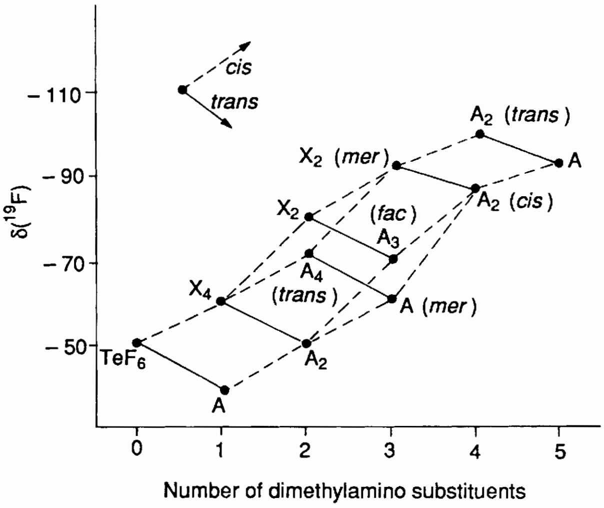

Fig. 1 Plot of ^{19}F chemical shifts against number of dimethylamino substituents. The result of substitution *cis* or *trans* to the test fluorine is shown by broken (shielding s.c.s.) or solid (deshielding s.c.s.) lines, respectively. The *mer* chemical shifts are estimated values

The value of n in the series $TeF_n(NMe_2)_{6-n}$ depended critically on the TeF_6:Me_3SiNMe_2 ratio. A 1:1 mixture showed only TeF_6 (δ −54[1a]) and $TeF_5(NMe_2)$ after 3 d. Table 2 shows the integrated intensities (normalised to 100%) from spectra of the reactions of TeF_6 and Me_3SiNMe_2 in 1:2 and 1:4 ratios. For the 1:2 mixture, only TeF_6, $TeF_5(NMe_2)$ and *cis*-$TeF_4(NMe_2)_2$ were present initially, but after 1 d a low concentration of *fac*-

Table 1 Fluorine-19 NMR data (chemical shifts δ, coupling constants *J*/Hz) for $TeF_{6-n}(NMe_2)_n$ (n.o. = not observed)

Compound		δ_F	$^2J(FF)$	$^1J(^{125}Te^{19}F)$	$^1J(^{123}Te^{19}F)$
1 $TeF_5(NMe_2)$	A	−39.22	170	3139	
	X_4	−63.47		3902	
2 *cis*-$TeF_4(NMe_2)_2$	A_2	−50.05	139	3590	2984
	X_2	−81.10		3465	2870
3 *trans*-$TeF_4(NMe_2)_2$	A_4	−71.50			
4 *fac*-$TeF_3(NMe_2)_3$	A_3	−66.29		3746	
5 *mer*-$TeF_3(NMe_2)_3$	AX_2	n.o.			
6 *cis*-$TeF_2(NMe_2)_4$	A_2	−86.27			
7 *trans*-$TeF_2(NMe_2)_4$	A_2	−99.17			
8 $TeF(NMe_2)_5$	A	−92.95			

Table 2 Relative intensities of $TeF_{6-n}(NMe_2)_n$ signals (n.o. = not observed)

	TeF_6:Me_3SiNMe_2 1:2				1:4			
Compound	0	1	5	12 d	0	1	5	12 d
1 $TeF_5(NMe_2)$	84	71	41	31	0	0	0	0
2 *cis*-$TeF_4(NMe_2)_2$	16	26	47	53	62	80	84	96
3 *trans*-$TeF_4(NMe_2)_2$	0	0	0	0	3	7	6	4
4 *fac*-$TeF_3(NMe_2)_3$	0	3	12	16	0	0	0	0
5 *mer*-$TeF_3(NMe_2)_3$	n.o.	n.o.	n.o.	n.o.	n.o.	n.o.	n.o.	n.o.
6 *cis*-$TeF_2(NMe_2)_4$	0	0	0	0	6	1	0.7	0
7 *trans*-$TeF_2(NMe_2)_4$	0	0	0	0	5	2	1.3	0
8 $TeF(NMe_2)_5$	0	0	0	0	24	10	8	0

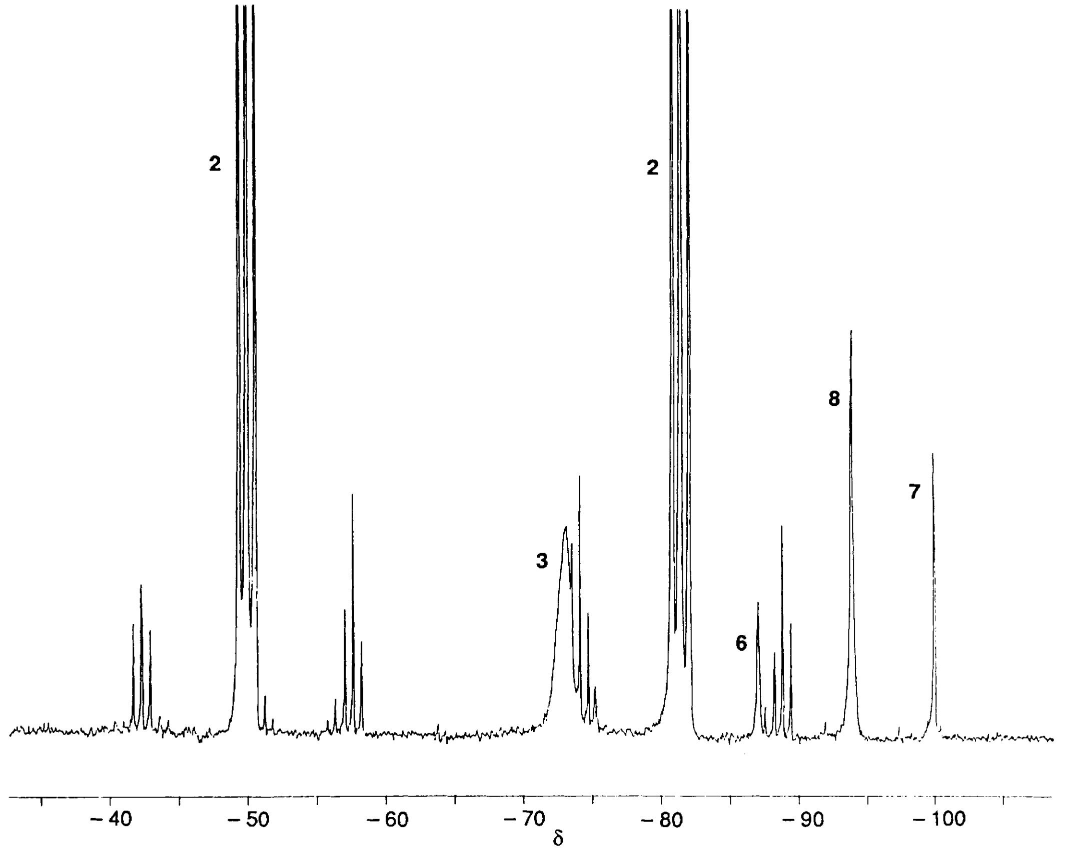

Fig. 2 Fluorine-19 NMR spectrum of a TeF_6–Me_3SiNMe_2 mixture (1:4) in CD_2Cl_2 after 1 d. The unlabelled signals are satellites

$TeF_3(NMe_2)_3$ was also observed. After 5 d, the amount of *cis*-$TeF_4(NMe_2)_2$ and *fac*-$TeF_3(NMe_2)_3$ had increased relative to that of $TeF_5(NMe_2)$ and after 12 d *cis*-$TeF_4(NMe_2)_2$ was the major component. In the 1:4 mixture, *cis*-$TeF_4(NMe_2)_2$ was always the dominant species with minor amounts of the *trans* isomer present as well as *cis*- and *trans*-$TeF_2(NMe_2)_4$ and $TeF(NMe_2)_5$; TeF_6 was absent. These minor species gradually disappeared and were absent after 12 d. Since the species $TeF_n(NMe_2)_{6-n}$ (n = 1–3) were present in only very small quantities, isolation is likely to be difficult. These solutions had to be stored at low temperature since decomposition to a grey powder, presumably tellurium, occurred above 0 °C after several h.

Unfortunately, for the series $TeF_n(N_3)_{6-n}$, the s.c.s. values are

Table 3 Fluorine-19 NMR data (chemical shifts δ, coupling constants J/Hz) for $TeF_{6-n}(N_3)_n$ (n.o. = not observed)

Compound		δ_F	$^2J(FF)$	$^1J(^{125}Te^{19}F)$	$^1J(^{123}Te^{19}F)$
9 $TeF_5(N_3)$	A_4	−40.55	168	3856	3205
	B	−41.80		3789	3200
10 *cis*-$TeF_4(N_3)_2$	A_2	−32.60	148.5	3697	
	X_2	−36.05		3562	
11 *trans*-$TeF_4(N_3)_2$	A_4	−31.14			
12 *fac*-$TeF_3(N_3)_3$	A_3	−30.98		3419	2836
13 *mer*-$TeF_3(N_3)_3$	A_2B	n.o.			
14 *cis*-$TeF_2(N_3)_4$	A_2	−26.90			
15 *trans*-$TeF_2(N_3)_4$	A_2	−25.24		3316	
16 $TeF(N_3)_5$	A	−29.14		3180	

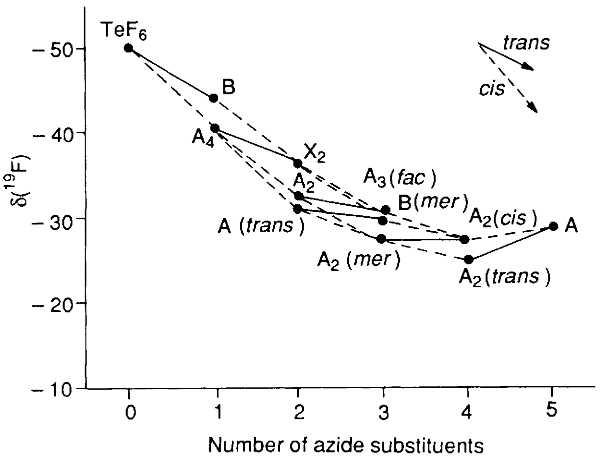

Fig. 3 Plot of ^{19}F chemical shifts against number of azido substituents. The result of substitution *cis* or *trans* to the test fluorine is shown by broken (shielding s.c.s) or solid (deshielding s.c.s) lines, respectively. The *mer* chemical shifts are estimated values

very similar and positive, leading to parallelograms which are too compressed for each assignment as shown in Fig. 3. The data are given in Table 3 and a typical spectrum is shown in Fig. 4.

First, the AB_4 and A_2B_2 signals of $TeF_5(N_3)$ and *cis*-$TeF_4(N_3)_2$, respectively, were unambiguously assigned. Assignment of the remaining singlets was based on Fig. 3 and was supported by the ^1J(TeF) values, which decreased monotonically as the number of fluorine atoms decreased. A signal for *mer*-$TeF_3(N_3)_3$ was not observed. Again, the parallelograms are not symmetric and appear to lie on a curve.

Table 4 gives intensity data (normalised to 100%) for the various members of the series. These data are only approximate since the signal for *cis*-$TeF_2(N_3)_4$ was often obscured by the resonance due to the tellurium(IV) species. Also, a solid was precipitated as the reaction progressed. However, the data show that in the 1:1 reaction *cis*-$TeF_4(N_3)_2$ was always the major component in solution with $TeF_5(N_3)$ also present, in about half the amount. Minor signals due to *trans*-$TeF_4(N_3)_2$ and *fac*-$TeF_3(N_3)_3$ also appeared and a signal for TeF_6 was always present. In the 1:2 reaction, *cis*-$TeF_4(N_3)_2$ was again the principal component, with signals for *fac*-$TeF_3(N_3)_3$, *cis*- and *trans*-$TeF_2(N_3)_4$ and $TeF(N_3)_5$ also observed. In addition, $TeF_5(N_3)$ and *trans*-$TeF_4(N_3)_2$ were present initially but had disappeared after 7 d. The tellurium(IV) compound was also a major component and TeF_6 was absent. After 23 d, only *fac*-$TeF_3(N_3)_3$ and *trans*-$TeF_2(N_3)_4$ (3:1) remained in the spectrum and the solution contained solid material. Ligand exchange and the probable explosive nature[9] of the azido species may frustrate isolation. The compound $TeF_5(N_3)$ might be preparable in the presence of a large excess of TeF_6; reaction with Me_3SiN_3 in a 2:1 ratio gave a 1:1 mixture of $TeF_5(N_3)$ and *cis*-$TeF_4(N_3)_2$ after 1 d.

Interestingly, the low concentrations of *trans*-substituted compounds and the failure to observe *mer*-substituted species in both systems is in agreement with previous work where *cis* substitution dominated the reaction pathways.[10] Random statistics give *cis*:*trans* and *fac*:*mer* ratios of 4:1 and 1.5:1, respectively.

Additional sets of resonances were observed in both systems. In the azido system an AB signal was observed at δ −26.75 with J(FF) = 29 Hz and J(TeF) = 3582 Hz on both arms of the AB. This is attributed to a tellurium(IV) species, *cis*-$TeF_2(N_3)_2X$ (X = MeCN?), because of the small FF coupling constant. Unidentified tellurium(IV) species have been obtained previously[11] in reactions of TeF_5Cl. The dimethylamino system yielded a minor A_2MX signal. This most probably arises from an unsymmetrically *cis*-disubstituted tetrafluorotellurium(VI) compound,[12] $TeF_4(NMe_2)Y$ (Y = OH?): A_2, dd, δ −70.36, J = 132.15 Hz; M, q, δ −50.6, J = 148 Hz; X, dt, δ −42.4, J = 163.1 Hz.

Attempts to prepare the known compounds TeF_5X (X = Cl,[11,13,14] Br,[13,15] or NCO[6b,16]) from TeF_6 and Me_3SiX failed. Earlier work[17] reported the formation of a complex mixture in the reaction of TeF_6 with $[NMe_4][N_3]$ in liquid sulfur dioxide. Reaction of TeF_6 with $[NMe_4][N_3]$ in acetonitrile did not give $TeF_5(N_3)$ {*cf.* preparation[17] of $WF_5(N_3)$ from WF_6 and $[NMe_4][N_3]$} but rather a mixture of $[NMe_4][TeF_5]$, $[NMe_4][TeF_7]$ and nitrogen according to equation (1).

$$2TeF_6 + 2[NMe_4][N_3] \longrightarrow [NMe_4][TeF_7] + [NMe_4][TeF_5] + 3N_2 \quad (1)$$

The salts were identified by ^{19}F and ^{125}Te NMR spectra which were compared with published data and the spectra of an independently prepared sample of $[NBu_4][TeF_5]$.[18] The pentafluorotellurate was prepared since the two previous reports[18] differ in their ^{19}F chemical shift values (when those in ref. 18*a* are corrected for $CFCl_3$). The results obtained here agree with those given in ref. 18*b*. The ^{125}Te chemical shift is close to that already reported[19] as are the data for the heptafluorotellurate.[20]

Conclusion

The reaction of TeF_6 with Me_3SiX (X = N_3 or NMe_2) gave all members of the series TeF_nX_{6-n} (n = 1–5) except *mer*-TeF_3X_3. Reduction also occurred to give a tellurium(IV) species with X = N_3 and elemental tellurium with X = NMe_2. Azide ion reduced TeF_6 to $[TeF_5]^-$ with $[TeF_7]^-$ and nitrogen as co-products.

Experimental

Manipulation of volatile compounds was carried out on an all-glass vacuum line fitted with a spiral gauge for measurement of tellurium hexafluoride. Hydrofluoric acid was handled in Teflon beakers and measured with a Polythene pipette. Tellurium

Table 4 Relative intensities of $TeF_{6-n}(N_3)_n$ (n.o. = not observed)

	$TeF_6:Me_3SiN_3$ 1:1			1:2		
Compound	1	2	14 d	1	7	14 d
9 $TeF_5(N_3)$	34	33	33	34	0	0
10 *cis*-$TeF_4(N_3)_2$	60	60	61	52	49	25
11 *trans*-$TeF_4(N_3)_2$	0.3	0.3	0.6	0.2	0	0
12 *fac*-$TeF_3(N_3)_3$	0.7	1.7	1.2	0.8	9	18
13 *mer*-$TeF_3(N_3)_3$	n.o.	n.o.	n.o.	n.o.	n.o.	n.o.
14 *cis*-$TeF_2(N_3)_4$	0	0	1?	1	?	?
15 *trans*-$TeF_2(N_3)_4$	0	0	0	1	14	18
16 $TeF(N_3)_5$	0	0	0	7	7	12
17 Te^{IV}	5	5	3.2	4	21	27

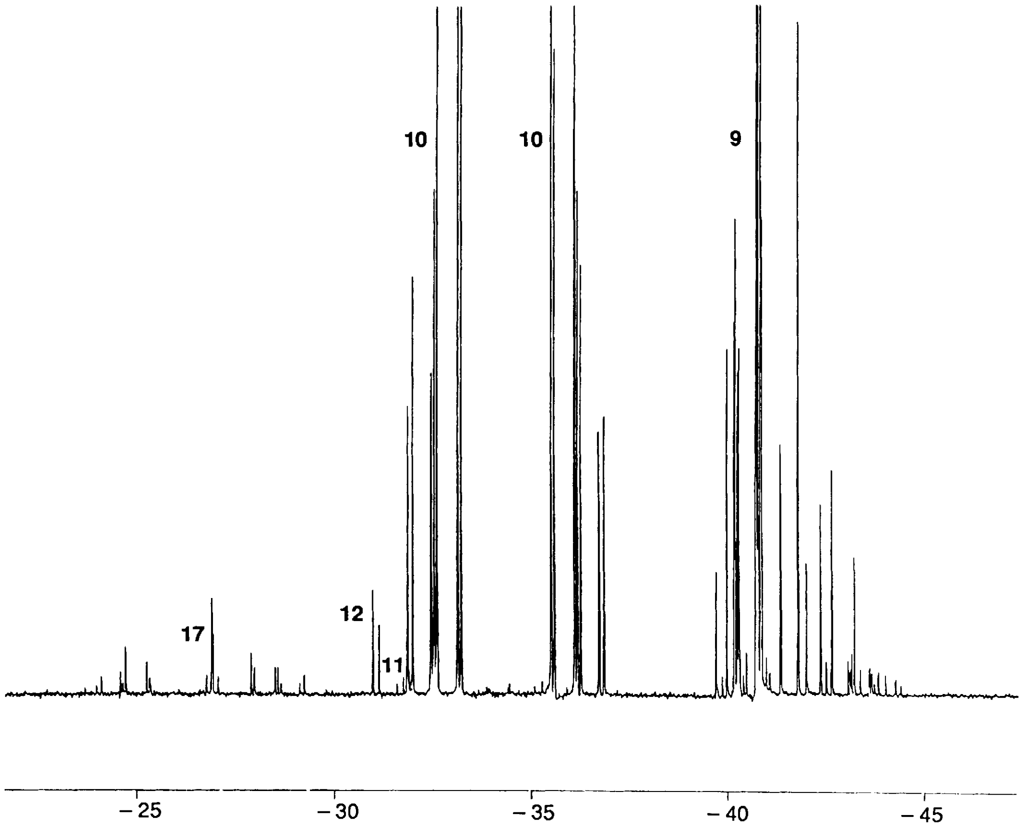

Fig. 4 Fluorine-19 NMR spectrum of a TeF_6–Me_3SiN_3 mixture (1:1) in CD_3CN after 14 d. The unlabelled signals are satellites

hexafluoride was supplied by Ozark-Mahoning. Azidotrimethylsilane, *N*,*N*-dimethylaminotrimethylsilane, $[^2H_3]$acetonitrile and $[^2H_2]$dichloromethane were supplied by Aldrich. Tellurium dioxide (99%) and concentrated hydrofluoric acid (Aristar, 40%) were supplied by BDH. The preparations of $[NBu_4]F \cdot xH_2O$ ($x \approx 29$) and $[NMe_4][N_3]$ will be reported elsewhere.[21] All reagents and solvents were used as supplied except $[^2H_3]$acetonitrile and $[^2H_2]$dichloromethane which were dried by storing over P_4O_{10}. Carbon, hydrogen and nitrogen were determined by microcombustion using a Perkin Elmer 240 elemental analyser. Fluorine was determined by fusion with potassium followed by passage down an ion-exchange column to give hydrofluoric acid which was titrated against sodium hydroxide solution. NMR spectra were recorded at 21 °C in sealed 5 mm tubes at 84.675 MHz on a Bruker HX90E spectrometer and at 23 °C and 235.42 MHz (^{19}F) or 78.935 MHz (^{125}Te) on a Bruker AC250 spectrometer. Trichlorofluoromethane in CD_3CN or CD_2Cl_2 or neat dimethyltellurium was used as the external reference. The estimated accuracy of the ^{19}F chemical shifts was ±0.3 ppm, allowing for concentration/susceptibility effects. An accuracy of ±1 Hz was obtained using 8K free induction decays, zero filling to 16K channels and expansion down to 1200 Hz. Pulse widths were 10 μs, pulse angles 60°, relaxation delays 1–3 s, acquisition times 0.13 s and the number of transients typically *ca.* 400.

Spectra were recorded at intervals determined by the reaction rate. The reactions were studied using a number of mixtures of TeF_6 and Me_3SiX in different molar ratios. In a typical experiment *ca.* 0.1 g Me_3SiX ($X = N_3$ or NMe_2) was condensed into the NMR tube at 77 K, followed by deuteriated solvent (*ca.* 1 cm^3). The correct amount of TeF_6, determined tensimetrically, was then added and the tube sealed off. Tubes containing $TeF_6:Me_3SiNMe_2$ mixtures (very pale yellow) was stored in a freezer at 243 K; those containing $TeF_6:Me_3SiN_3$ mixtures (initially colourless but becoming yellow with excess of Me_3SiN_3 present) were stored at room temperature.

Preparation of $[NBu_4][TeF_5]$.—A solution of TeO_2 (0.56 g, 3.23 mmol) in 40% Aristar HF (2 cm^3) was added to a (two-layered) solution of $[NBu_4]F \cdot 29H_2O$ (2.75 g, 3.5 mmol) also in

40% Aristar HF (2 cm^3) to give a two-layered mixture. After 12 h, two layers were still present and more HF (20 cm^3) was slowly added until a homogeneous solution was obtained. This was heated on a steam-bath for several hours until solid material was observed. After overnight storage in a plastic desiccator over $Mg(ClO_4)_2$–NaOH a white solid mass was obtained which was broken up with a spatula and dried *in vacuo* for 3 h. Yield 1.41 g, 93.8% (Found: C, 41.6; H, 8.1; F, 20.2; N, 3.0. Calc. for $C_{16}H_{36}F_5NTe$: C, 41.3; H, 7.8; F, 20.4; N, 3.0%). $\delta_F(CD_2Cl_2)$ -29.4 [$1F_{ax}(A)$, q] and -36.9 [$4F_{eq}(B_4)$, d, J(FF) 50 Hz]; $\delta_{Te}(CD_2Cl_2)$ 1142.25 [dq, $J(TeF_{ax})$ 2909, $J(TeF_{eq})$ 1385 Hz].

Reaction of TeF_6 *with* $[NMe_4][N_3]$.—In a typical experiment, $[NMe_4][N_3]$ (*ca.* 2 mg, 0.017 mmol) was transferred to an NMR tube in the glove-box. The required amounts of [$^2H_3$]-acetonitrile (*ca.* 1 cm^3) and TeF_6 were then condensed in on the vacuum line and the tube was sealed off. $\delta_F(CD_3CN)$ 16.72 [7F, s, $J(^{125}TeF)$ 2871, $J(^{123}TeF)$ 2383], -29.4 [$1F_{ac}(A)$, q] and -37.1 [$4F_{eq}(B_4)$, d, J(FF) 50 Hz].

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