

## REACTIONS OF TELLURIUM(IV) COMPOUNDS WITH TRIMETHYL(DIALKYLAMINO)SILANES

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### Summary

$R\text{TeCl}_3$  ( $R = \text{C}_6\text{H}_5$ ,  $p\text{-CH}_3\text{OC}_6\text{H}_4$  or  $p\text{-C}_6\text{H}_5\text{OC}_6\text{H}_4$ ) reacts with  $\text{Me}_3\text{SiNR}'_2$  ( $R'_2 = \text{Et}_2$ ,  $\text{C}_4\text{H}_8$ ) under dry nitrogen atmosphere to give  $R(\text{R}'_2\text{N})\text{TeCl}_2$  and  $\text{Me}_3\text{SiCl}$ . The products readily decompose to give  $(\text{R}'_2\text{NH}_2)^+(\text{R}\text{TeCl}_4)^-$ . The products have been characterized by  $^1\text{H}$  NMR, IR and mass spectra.  $\text{R}_2\text{TeCl}_2$  does not react with  $\text{Me}_3\text{SiNR}'_2$  even on refluxing for 6 h.  $\text{Et}_2\text{NLi}$ , however, reduces  $\text{R}_2\text{TeCl}_2$  to  $\text{R}_2\text{Te}$ .

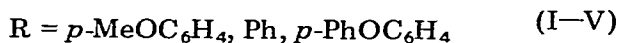
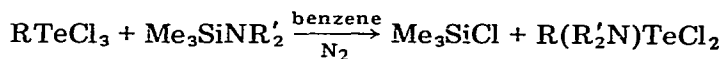
### Introduction

Organotellurium compounds containing a tellurium–metal or tellurium–metalloid bond have been the subject of a number of studies [1–3], though very little work has been done on compounds containing a tellurium–nitrogen bond. Recently compounds with such a Te–N double bond have been reported. The imido derivatives of tellurium have been prepared by the reaction of  $\text{R}_2\text{TeO}$  with  $\text{R}'\text{SO}_2\text{NH}_2$  [4] and  $\text{TeX}_4$  with  $\text{RSO}_2\text{N}(\text{SiMe}_3)_2$  or  $\text{RC}(\text{O})\text{N}(\text{SiMe}_3)_2$  [5]. The compounds of the type  $(\text{R}_2\text{N})_2\text{TeX}_2$  [6] and  $\text{R}_2\text{NTeF}_5$  and  $(\text{R}_2\text{N})_2\text{TeF}_4$  [7] are also known. We now report our results for some organotellurium compounds containing a tellurium–nitrogen single bond. We have prepared several aryl(dialkylamino)tellurium compounds by the reaction of  $\text{R}\text{TeCl}_3$  with silylated secondary amines. In addition to these reactions, we have also studied the reactions of  $\text{R}_2\text{TeCl}_2$  and  $\text{TeCl}_4$  with trimethyl(dialkylamino)silanes.

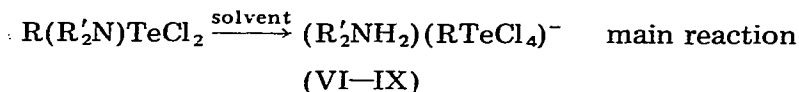
### Results and discussion

Aryltellurium trichlorides ( $\text{R}\text{TeCl}_3$ ) react vigorously with trimethyl(dialkylamino)silanes in benzene solution in a dry nitrogen atmosphere according to

the equation:



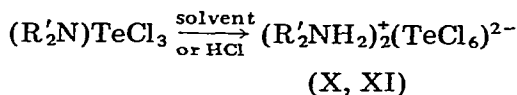
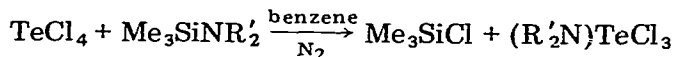
The addition of petroleum ether (30–60°C) to the reaction mixture results in the separation of species I–V, while on keeping the unseparated mixture as such for 4 or 5 days and then reducing the volume to half under vacuum yields VI–VIII. However, IX was obtained by the addition of petroleum ether prior to the recovery of IV.



The disiloxane present in the reaction mixture has been detected mass spectrometrically. The yields of I–V or VI–IX are never more than 40%. The products I–V are highly sensitive to moisture and air. Since the conversion of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  to  $(\text{R}'_2\text{NH}_2)^+(\text{RTeCl}_4)^-$  requires two molecules of HCl, this can be formed either from the interaction of  $\text{Me}_3\text{SiCl}$  and moisture, or the interaction of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  or unreacted  $\text{RTeCl}_3$  with the solvent. The reaction was monitored by  $^1\text{H}$  NMR spectroscopy and with passage of time, only a trace of disiloxane was observed. Thus we can conclude that the conversion of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  to  $(\text{R}'_2\text{NH}_2)^+(\text{RTeCl}_4)^-$  is mainly due to the interaction of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  or  $\text{RTeCl}_3$  with the solvent.

The formation of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  has been observed not only in benzene, but also in toluene or  $\text{CH}_2\text{Cl}_2$ . However, an equimolar mixture of  $\text{RTeCl}_3$  and  $\text{Me}_3\text{SiNR}'_2$  in another aprotic solvent such as  $\text{CCl}_4$  does not proceed with Si–N cleavage even at reflux temperature, but provides an addition product of 1 : 1 stoichiometry. This suggests that the solvent may play a significant role in this reaction. Similar observation was made by Vasishat and Goyal in case of reaction of  $\text{M}_3\text{SiNC}_4\text{H}_8$  with  $\text{TiCl}_4$  and  $\text{FeCl}_3$  [8].

In the case of reaction with  $\text{TeCl}_4$ , it has not been possible to isolate  $\text{R}'_2\text{N}-\text{TeCl}_3$ , as such; instead  $(\text{R}'_2\text{NH}_2)_2^+(\text{TeCl}_6)^{2-}$  was always obtained, the yield being 40% when  $\text{R}'_2 = \text{Et}_2$  and 15% when  $\text{R}'_2 = \text{C}_4\text{H}_8$ .



Another major reaction product is elemental tellurium, thus additionally accounting for the chlorine in the HCl required for the formation of X or XI. However, since some  $\text{Me}_6\text{Si}_2\text{O}$  has been detected in the reaction mixture, the

TABLE I  
ANALYTICAL DATA AND <sup>1</sup>H NMR SPECTRA OF THE COMPOUNDS I-XI

Compound	Analysis Found (calcd.)				Yield	M.p. (°C)	<sup>1</sup> H NMR <sup>a</sup>					
	C	H	N	Cl			H(1)	H(2)	-OCH <sub>3</sub>	H(A)	H(B)	NH
I <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> (Et <sub>2</sub> N)TeCl <sub>2</sub>	34.96 (34.67)	4.50 (4.77)	3.71 (3.52)	18.80 (18.83)	30	103	1.21	3.44	3.87	7.07	8.23	
II <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> (C <sub>4</sub> H <sub>8</sub> N)TeCl <sub>2</sub>	35.14 (35.09)	3.99 (3.72)	3.73 (3.44)	18.90 (19.04)	35	60	1.87	3.51	3.86	7.06	8.25	
III Ph(Et <sub>2</sub> N)TeCl <sub>2</sub>	34.44 (34.52)	4.52 (4.31)	3.94 (4.03)	20.29 (20.43)	31	105	1.16	2.90		7.38		
IV <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> (Et <sub>2</sub> N)TeCl <sub>2</sub>	43.51 (43.68)	4.38 (4.32)	3.30 (3.18)	16.30 (16.15)	30	135-138	1.20	2.96		7.28	8.47	
V <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> (C <sub>4</sub> H <sub>8</sub> N)TeCl <sub>2</sub>	43.84 (43.88)	4.04 (3.88)	3.28 (3.20)	16.62 (16.22)	40	166-168	1.85	3.11		7.01	8.36	
VI (Et <sub>2</sub> NH <sub>2</sub> ) <sup>+</sup> ( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> TeCl <sub>4</sub> ) <sup>-</sup>	29.24 (29.29)	4.49 (4.22)	3.00 (3.11)	31.23 (31.51)	25	90-92	1.24	3.05	3.84	6.99	8.46	2.07
VII (C <sub>4</sub> H <sub>8</sub> NH <sub>2</sub> ) <sup>+</sup> ( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> TeCl <sub>4</sub> ) <sup>-</sup>	29.71 (29.42)	3.75 (3.79)	3.25 (3.12)	31.30 (31.65)	23	170	1.83	3.09	3.81	7.04	8.35	1.95
VIII (Et <sub>2</sub> NH <sub>2</sub> ) <sup>+</sup> (PhTeCl <sub>4</sub> ) <sup>-</sup>	28.53 (28.54)	4.04 (4.27)	3.33 (3.36)	33.76 (33.36)	21	100-101	1.14	2.90		7.48	8.52	1.90
IX (Et <sub>2</sub> NH <sub>2</sub> ) <sup>+</sup> ( <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> TeCl <sub>4</sub> ) <sup>-</sup>	37.40 (37.46)	4.26 (4.10)	2.71 (2.73)	27.45 (27.70)	40	148-150	1.17	2.92		7.04	8.43	2.09
X (Et <sub>2</sub> NH <sub>2</sub> ) <sup>+</sup> <sub>2</sub> (TeCl <sub>6</sub> ) <sup>2-</sup>	19.49 (19.65)	5.08 (4.91)	5.62 (5.73)	43.63 (43.59)	40	215-217	1.17	2.91				8.60
XI (C <sub>4</sub> H <sub>8</sub> NH <sub>2</sub> ) <sup>+</sup> <sub>2</sub> (TeCl <sub>6</sub> ) <sup>2-</sup>	19.71 (19.81)	4.32 (4.13)	5.80 (5.78)	43.76 (43.95)	15	200-202	1.84	3.10				8.83

<sup>a</sup> Chemical shifts, in ppm from TMS, I-V in CDCl<sub>3</sub> and VI-XI in DMSO-*d*<sub>6</sub>; H(1) = high field methyl or methylene, H(2) = low field methylene, H(A) = meta aromatic protons, H(B) = *ortho* aromatic protons, *J*(1-2) ~ 7 Hz, *J*(A-B) = 9 Hz.

possibility of the interaction with moisture cannot be ruled out.

The reaction of  $R_2TeCl_2$  with  $Me_3SiNR'_2$  was not successful, even on refluxing the solution in benzene for 6 h. This is consistent with the fact that  $TeCl_4$  and  $RTeCl_3$  are electrophilic reagents, while  $R_2TeCl_2$  is insensitive to nucleophilic attack [9]. Another possible reason for the failure of this reaction may be steric hinderance in  $R_2TeCl_2$ .

Another reagent which produces metal–nitrogen bonds in inorganic compounds is  $N,N'$ -lithium diethylamide [10]. In the reaction of  $R_2TeCl_2$  ( $R = Ph, p\text{-MeOC}_6\text{H}_4$ ) with  $Et_2NLi$ , it was found that instead of forming a  $Te-N$  bond, this reagent reduces tellurium(IV) to tellurium(II). Thus  $R_2Te$  was obtained. However, it is also possible that  $Et_2NLi$  reacts with  $R_2TeCl_2$  to give  $R_2Te(NEt)_2$  which then decomposes to  $R_2Te$  and  $Et_2NNEt_2$ . The yield of  $R_2Te$  from the dichloride was excellent, but the highly air- and moisture-sensitive  $Et_2NLi$  cannot be recommended as a routine reducing agent because more convenient reducing agents are available [1].

Compounds successfully prepared are listed in Table 1, along with their melting points, analytical and  $^1H$  NMR data. The compounds of type  $R(R'_2N)TeCl_2$  are yellow needles and are recrystallisable from  $CH_2Cl_2$ /petroleum ether ( $30\text{--}60^\circ C$ ). The compounds decompose slowly at room temperature during 2–3 weeks. Integration of peaks in  $^1H$  NMR spectra served to confirm the stoichiometry indicated by the analytical data. The aromatic signals in  $p$ -methoxy substituted products show four-line AB signals with  $J(A-B)$  of 9 Hz. No cross ring couplings of the AA'BB' variety are detected. The aromatic protons in the *ortho* position to Te appears at a lower field than *meta* protons [11].

Selenium compounds containing an  $R_2N-Se$  function exhibit an IR band at  $540\text{ cm}^{-1}$  which has been assigned to the  $Se-N$  stretching frequency [12]. The corresponding  $Te-N$  stretching frequency is, thus expected to be below 540

TABLE 2  
IR SPECTRA ( $\text{cm}^{-1}$ ) OF THE COMPOUNDS I–XI

I	II	III	IV	V	Assignment	
468m	468w	473(sh) 455s	460w	475s	Te–N str.	
280s	291(sh)	292s	203s	280m	Te–Cl sym. str.	
255m	261w	250s	259s	255m	Te–Cl asy. str.	
269w	270w	275s	278s	267w	Te–C(phenyl) str.	
248s	249w		253m	247s	or t,t'(phenyl)	
VI	VII	VIII	IX	X <sup>a</sup>	XI	Assignment
2545w	2560m	2520w	2410m	2475m	2418w	N–H str.
2418w	2418m	2420w	2380m	2430m	2380w	
2335w	2345m			249w	247w	Te–Cl str.
				228w	230w	$\nu_3$
277s	275s	279w	270m			$\nu_2(A_1)$
266m		267m	260m			
254s	255(sh)		258(sh)			$\nu_7(E)$
248s	242s	247w				

<sup>a</sup> Raman Spectrum shows bands at 340m, 286m and 249m for  $\nu_1$  and  $\nu_2$ .

$\text{cm}^{-1}$ . Careful examination of the IR spectra of the compounds of the type  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  and comparison with the IR spectra of  $\text{R}_2\text{TeCl}_2$  and  $\text{R}'_2\text{NH}$  indicated that the absorption peaks in the region  $460\text{--}475\text{ cm}^{-1}$  (Table 2) would seem a reasonable choice for Te—N stretching frequency. The appearance of Te—Cl stretching frequencies in the region where  $\text{R}_2\text{TeCl}_2$  absorbs [13] suggests the structure of  $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$  must be similar to  $\text{R}_2\text{TeCl}_2$ . Thus the chlorine atoms occupy axial positions in a pseudotrigonal bipyramid.

The ionic products VI—XI exhibit association between alkyl ammonium cation and anion.  $(\text{Et}_4\text{N})_2^+(\text{TeCl}_6)^{2-}$  showed bands at 280 and 243 (for  $\nu_1$  and  $\nu_2$ , respectively, Raman active) and 228 (for  $\nu_3$ , IR active)  $\text{cm}^{-1}$  [14], whereas  $(\text{Et}_2\text{NH}_2)_2^+(\text{TeCl}_6)^{2-}$  (X) shows three bands in the Raman spectrum and 2 bands in the IR spectrum (Table 2). The IR spectra of VI—IX also shows three or four bands, instead of two, for the IR active Te—Cl symmetric and asymmetric

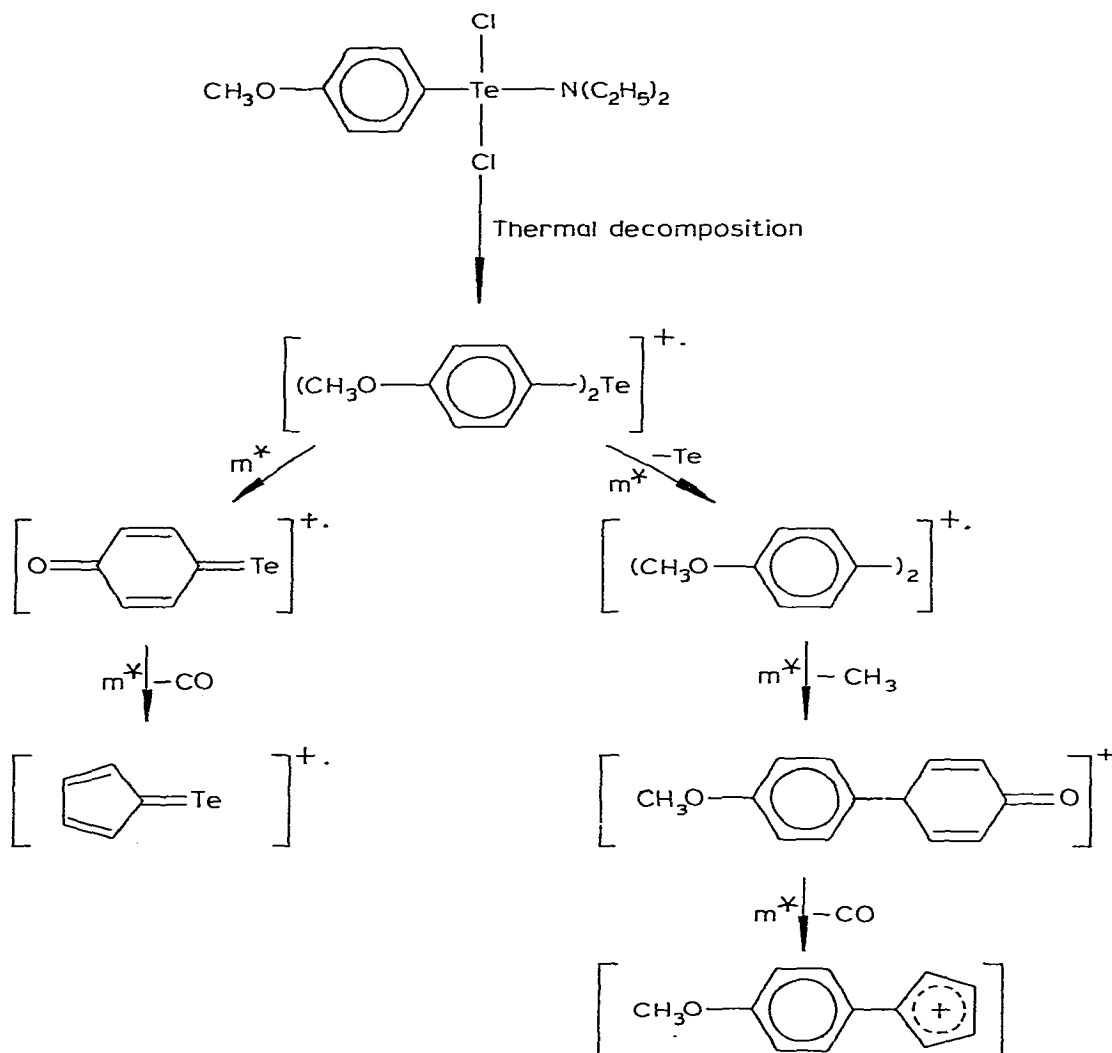


Fig. 1. Partial fragmentation scheme for I.

stretches [15]. This is consistent with a lower symmetry, probably due to hydrogen bonding to the chlorine. Further evidence for the hydrogen bonding comes from the  $^1\text{H}$  NMR spectra of the compounds. A linear relationship has been found between the chemical shift of the NH proton signal, (relative to the  $\text{CH}_2$  signal), and temperature. An increase of temperature from  $-40$  to  $50^\circ\text{C}$ , e.g., in VI, causes the NH proton signal to shift toward the  $\text{CH}_2$  signal, i.e. to higher field, by approximately 1.6 ppm. It was further observed that dilution of the compounds by the solvent had an effect similar to raising the temperature.

Tellurium is a polyisotopic element, with mass varying between 130–120, and in the mass spectra of tellurium compounds the various fragments containing tellurium atoms are very easily recognisable because of their typical isotopic cluster [16]. In the mass spectra of I–V, the principal fragments formed are  $\text{RTeCl}_2^+$ ,  $\text{RTeCl}^+$ ,  $\text{R}_2\text{Te}^{++}$ ,  $\text{RTe}^+$ ,  $\text{Te}_2^+$ ,  $\text{R}_2^{++}$ ,  $\text{TeCl}^+$ ,  $\text{Te}^{++}$ ,  $\text{RCl}^+$ ,  $\text{R}'\text{NH}^{++}$ ,  $\text{R}'\text{N}^+$ ,  $\text{C}_4\text{H}_{10}^+$ ,  $\text{C}_3\text{H}_7^{++}$ ,  $\text{C}_2\text{H}_6^+$  and  $\text{C}_3\text{H}_3^{++}$ . The parent or molecular ion was clearly shown by only I. Two other compounds, II and III show only a trace of molecular ion. The formation of  $(p\text{-MeOC}_6\text{H}_4)_2\text{Te}^{++}$  may be the result of facile thermal decomposition of the compound within the inlet of ion source of the mass spectrometer. This ion is one of the few for which adequate metastable ion data can be obtained, consequently, a partial metastable ion-supported fragmentation scheme for I is proposed in Fig. 1. The detection of  $\text{RCl}^+$  species (in I–IX) suggests the transfer of a chlorine atom from the tellurium to the hydrocarbon part of the molecule. The ionic products VI–IX and X and XI show  $\text{RTeCl}_2^+$  and  $\text{TeCl}_3^+$ , respectively, as the highest mass cluster detected other than  $\text{Te}_2^+$ .

## Experimental

The reactions were carried out in flame dried glassware under  $\text{P}_2\text{O}_5$ -dried nitrogen gas. All other manipulations were done in a dry box occasionally flushed with nitrogen gas. The solvents were distilled and sodium dried.  $\text{Me}_3\text{-SiCl}$ ,  $\text{Et}_2\text{NH}$  and  $\text{C}_4\text{H}_8\text{NH}$  were purchased from Alfa Inorganics and were used after drying and distillation.  $\text{TeCl}_4$  was used as such.  $\text{Me}_3\text{SiNR}'_2$  [17,18] and organotellurium compounds [1,19] were prepared by literature methods.

The IR spectra ( $4000\text{--}200\text{ cm}^{-1}$ ) were recorded on a Perkin-Elmer 225 grating spectrophotometer using KBr and polyethylene windows. Mass spectra were recorded on AEI-MS/30 double beam spectrometer at 70 eV using PFK as a reference and a direct probe inlet. The Fortran program BMASROS was used to calculate theoretical isotope clusters of tellurium- and chlorine-containing fragments.  $^1\text{H}$  NMR spectra were recorded on a Bruker WP-60 multinuclear magnetic resonance, with an internal deuterium lock. Raman spectra were obtained with a Jarrell Ash-based instrument built at Brock University. Microanalyses were performed by Guelph Chemical Laboratories Ltd.

### *General procedure for reactions of $\text{RTeCl}_3$ and $\text{Me}_3\text{SiNR}'_2$*

A 200 ml round bottom flask was set up with a magnetic stirrer, reflux condenser and addition funnel, flushed with nitrogen, and flame dried.  $\text{RTeCl}_3$  ( $\sim 7\text{--}10$  mmol) and 50 ml of anhydrous benzene were placed in the flask and

$\text{Me}_3\text{SiNR}'_2$  ( $\sim 7$ – $10$  mmol) was added dropwise from the addition funnel. After stirring for 3 h, the contents were filtered in a Schlenk-type apparatus unit to remove any insoluble impurity present.

Products were obtained from this filtrate A in the following ways:

*Preparation of I, II and III.* To the filtrate A, 50 ml petroleum ether ( $30$ – $60^\circ\text{C}$ ) were added. A slurry formed slowly and after being allowed to settle for 2–3 h, a clear solution was decanted. More petroleum ether ( $30$ – $60^\circ\text{C}$ ) was added to the clear solution, which, on standing overnight, deposited yellow needle-like crystals, which were separated and dried in vacuum. Recrystallization from  $\text{CH}_2\text{Cl}_2$ /petroleum ether gave bright yellow needles of I, II, or III. Mass spectrum for a representative compound, with relative abundances for isotopes  $^{130}\text{Te}$  and  $^{35}\text{Cl}$  is given below.

Mass spectrum of I:  $M^+$  (2.4%),  $\text{R}_2\text{Te}^{++}$  (13.0%),  $\text{RTeCl}_2^+$  (3.0%),  $\text{RTeCl}^{+*}$  (68.0%),  $\text{C}_6\text{H}_4\text{OTeCl}^+$  (12.3%),  $\text{RTe}^+$  (63.4%),  $\text{C}_6\text{H}_4\text{OTe}^{++}$  (20.7%),  $\text{R}_2^+$  (32.9%),  $\text{C}_{13}\text{H}_{11}\text{O}_2^+$  (39.0%),  $\text{C}_5\text{H}_4\text{Te}^{++}$  (11.0%),  $\text{C}_{12}\text{H}_{11}\text{O}^+$  (7.2%),  $\text{TeCl}^+$  (20.1%),  $\text{RCl}^+$  (36.6%),  $\text{Te}^{++}$  (9.8%),  $\text{RTe}^{++}$  (1.2%),  $\text{R}^{++}$  (4.9%),  $\text{C}_6\text{H}_4\text{O}^{++}$  (28.0%),  $\text{R}'_2\text{NH}^{+*}$  (67.1%),  $\text{R}'_2\text{N}^+$  (61.0%),  $\text{C}_5\text{H}_4^{++}$  (23.2%),  $\text{C}_4\text{H}_{10}^+$  (100.0%).

*Preparation of VI, VII and VIII.* The filtrate A was allowed to stand for 4–5 days and then on reducing the volume to half under vacuum, colourless crystals of VI, VII or VIII were obtained, which were filtered, washed twice with 2–3 ml benzene and dried in vacuum.

Mass spectrum of VIII:  $\text{RTeCl}_2^+$  (0.6%),  $\text{Te}_2^+$  (2.8%),  $\text{RTeCl}^{+*}$  (5.0%),  $\text{RTe}^+$  (13.4%),  $\text{TeCl}^+$  (1.0%),  $\text{R}_2^{++}$  (2.2%),  $\text{Te}^{++}$  (5.0%),  $\text{RCl}^+$  (5.0%),  $\text{R}^+$  (46.9%),  $\text{R}'_2\text{NH}^{+*}$  (20.6%),  $\text{R}'_2\text{N}^+$  (15.0%),  $\text{C}_4\text{H}_{10}^+$  (100.0%),  $\text{C}_4\text{H}_3^{++}$  (25.0%),  $\text{C}_2\text{H}_6\text{N}^+$  (43.7%),  $\text{HCl}^{+*}$  (69%).

*Preparation of IV, V and IX.* The addition of 50 ml of petroleum ether to the filtrate A gave a dull white precipitate, which upon recrystallization from benzene gave IX.

When more petroleum ether ( $30$ – $60^\circ\text{C}$ ) (100 ml) was added to the filtrate, yellow needles of IV or V were separated which were recrystallized from  $\text{CH}_2\text{Cl}_2$ /petroleum ether ( $30$ – $60^\circ\text{C}$ ).

Mass spectrum of V:  $\text{RTeCl}_2^+$  (0.2%),  $\text{R}_2^{++}$  (1.0%),  $\text{RTeCl}^{+*}$  (1.0%),  $\text{RTe}^+$  (4.2%),  $\text{Te}_2^+$  (2.3%),  $\text{C}_6\text{H}_4\text{OTe}^{++}$  (0.8%),  $\text{RCl}^+$  (5.8%),  $\text{C}_5\text{H}_4\text{Te}^{++}$  (1.6%),  $\text{R}^+$  (4.7%),  $\text{TeCl}^+$  (2.6%),  $\text{Te}^{++}$  (5.8%),  $\text{C}_6\text{H}_4\text{O}^{++}$  (4.2%),  $\text{R}'_2\text{NH}^{+*}$  (7.4%),  $\text{R}'_2\text{N}^+$  (8.9%),  $\text{C}_4\text{H}_8^+$  (100.0%),  $\text{C}_4\text{H}_3^+$  (63.2%),  $\text{C}_3\text{H}_8^{++}$  (42.1%).

#### *Reaction of $p\text{-MeOC}_6\text{H}_4\text{TeCl}_3$ with $\text{Me}_3\text{SiNC}_4\text{H}_8$ in $\text{CCl}_4$*

In a similar reaction set up,  $p\text{-MeOC}_6\text{H}_4\text{TeCl}_3$  (1.14 g, 3.3 mmol) was treated with (0.5 g, 3.3 mmol)  $\text{Me}_3\text{SiNR}'_2$  in 50 ml  $\text{CCl}_4$ . The reaction was stirred for 12 hours and was subsequently filtered to remove any insoluble impurity. Ether (50 ml) was then added causing a white precipitate which was separated immediately by filtration, washed with ether and dried in vacuum to yield 0.7 g (45%) of a highly moisture- and air-sensitive adduct,  $p\text{-MeOC}_6\text{H}_4\text{TeCl}_3 \cdot \text{Me}_3\text{SiNC}_4\text{H}_8$  (m.p.  $75$ – $77^\circ\text{C}$ ), which could not be recrystallized satisfactorily. Found: C, 34.00; H, 4.43; N, 2.75; Cl, 21.60. Calcd. for  $\text{C}_{14}\text{H}_{29}\text{ONSiTe}$ : C, 34.70; H, 4.96; N, 2.89; Cl, 22.00%.

A similar reaction with  $\text{Me}_3\text{SiNEt}_2$  in  $\text{CCl}_4$  also gave a 1 : 1 adduct, m.p.  $132$ – $135^\circ\text{C}$ .

### Reaction of $\text{TeCl}_4$ and $\text{Me}_3\text{SiNEt}_2$

In a 500 ml round bottom flask equipped with a magnetic stirrer were placed 3.02 g  $\text{TeCl}_4$  (11.2 mmol) and 100 ml benzene.  $\text{Me}_3\text{SiNEt}_2$  (1.62 g, 11.2 mmol) in 100 ml benzene was added very slowly over a period of 6 h. The colour of the reaction mixture changed from yellow to green and a black muddy substance (tellurium) separated. The reaction mixture was stirred for another 6 h and filtered in a Schlenk-type filtration unit. The volume of filtrate was reduced to half by applying vacuum. Fine yellowish green crystals separated slowly from the filtrate. The crystals were washed with ether and dried in vacuum. Extraction of the black muddy residue with acetone gave another crop of yellowish green crystals. Both the crops were combined and recrystallized from acetone/ $\text{CH}_2\text{Cl}_2$  to give X. A similar reaction of  $\text{TeCl}_4$  with  $\text{Me}_3\text{SiNC}_4\text{H}_8$  gave XI.

### Reaction of $\text{Ph}_2\text{TeCl}_2$ with $\text{Et}_2\text{NLi}$

In a 200 ml three-necked flask equipped with a magnetic stirrer, nitrogen gas inlet, a reflux condenser and septum were placed 1.73 ml (16 mmol) of  $\text{Et}_2\text{NH}$  in 20 ml anhydrous ether at  $-10^\circ\text{C}$ .  $\text{BuLi}$  (1.06 g, 16 mmol) was admitted slowly through septum so that a white precipitate of  $\text{Et}_2\text{NLi}$  was formed. The septum was then replaced with an addition funnel and  $\text{Ph}_2\text{TeCl}_2$  (2.94, 8 mmol) in 40 ml benzene was added dropwise with constant stirring in 2 h. The contents were refluxed for an additional hour and were filtered on cooling to remove  $\text{LiCl}$ . The filtrate was fractionally distilled to obtain yellow oil of  $\text{Ph}_2\text{Te}$  at  $112^\circ\text{C}/1$  mmHg. Yield 85%.

A similar reaction of  $\text{LiNEt}_2$  with  $(p\text{-MeOC}_6\text{H}_4)_2\text{TeCl}_2$  gave  $(p\text{-MeOC}_6\text{H}_4)_2\text{Te}$ . Yield 80%, m.p.  $54^\circ\text{C}$ , literature [1]  $53\text{--}54^\circ\text{C}$ .

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