

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

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Summary

RTeCl_3 ($\text{R} = \text{C}_6\text{H}_5$, $p\text{-CH}_3\text{OC}_6\text{H}_4$ or $p\text{-C}_6\text{H}_5\text{OC}_6\text{H}_4$) reacts with $\text{Me}_3\text{SiNR}'_2$ ($\text{R}'_2 = \text{Et}_2$, C_4H_8) under dry nitrogen atmosphere to give $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$ and Me_3SiCl . The products readily decompose to give $(\text{R}'_2\text{NH}_2)^+(\text{RTeCl}_4)^-$. The products have been characterized by ^1H NMR, IR and mass spectra. R_2TeCl_2 does not react with $\text{Me}_3\text{SiNR}'_2$ even on refluxing for 6 h. Et_2NLi , however, reduces R_2TeCl_2 to R_2Te .

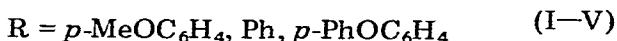
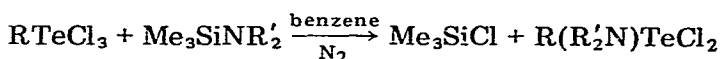
Introduction

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 R_2TeO with $\text{R}'\text{SO}_2\text{NH}_2$ [4] and 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 R_2NTeF_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 RTeCl_3 with silylated secondary amines. In addition to these reactions, we have also studied the reactions of R_2TeCl_2 and TeCl_4 with trimethyl(dialkylamino)silanes.

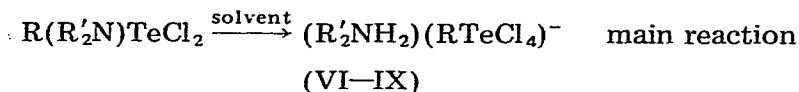
Results and discussion

Aryltellurium trichlorides (RTeCl_3) react vigorously with trimethyl(dialkylamino)silanes in benzene solution in a dry nitrogen atmosphere according to

the equation:



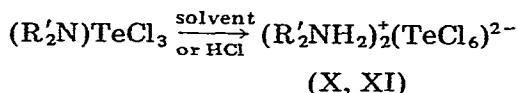
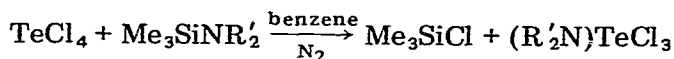
The addition of petroleum ether (30–60°C) to the reaction mixture results in the separation of species I–V, while on keeping the unseparated mixture as such for 4 or 5 days and then reducing the volume to half under vacuum yields VI–VIII. However, IX was obtained by the addition of petroleum ether prior 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 Me_3SiCl and moisture, or the interaction of $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$ or unreacted RTeCl_3 with the solvent. The reaction was monitored by ^1H 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 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 CH_2Cl_2 . However, an equimolar mixture of RTeCl_3 and $\text{Me}_3\text{SiNR}'_2$ in another aprotic solvent such as 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 TiCl_4 and FeCl_3 [8].

In the case of reaction with 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 1
ANALYTICAL DATA AND ¹H NMR SPECTRA OF THE COMPOUNDS I–XI

Compound		Analysis Found (calcd.)				Yield	M.p. (°C)	¹ H NMR ^a					
		C	H	N	Cl			H(1)	H(2)	—OCH ₃	H(A)	H(B)	NH
I	<i>p</i> -MeOC ₆ H ₄ (Et ₂ N)TeCl ₂	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 ₆ H ₄ (C ₄ H ₈ N)TeCl ₂	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 ₂ N)TeCl ₂	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 ₆ H ₄ (Et ₂ N)TeCl ₂	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 ₆ H ₄ (C ₄ H ₈ N)TeCl ₂	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 ₂ NH ₂) ⁺ (<i>p</i> -MeOC ₆ H ₄ TeCl ₄) [−]	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 ₄ H ₈ NH ₂) ⁺ (<i>p</i> -MeOC ₆ H ₄ TeCl ₄) [−]	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 ₂ NH ₂) ⁺ (PhTeCl ₄) [−]	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 ₂ NH ₂) ⁺ (<i>p</i> -PhOC ₆ H ₄ TeCl ₄) [−]	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 ₂ NH ₂) ₂ ⁺ (TeCl ₆) ^{2−}	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 ₄ H ₈ NH ₂) ₂ ⁺ (TeCl ₆) ^{2−}	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

^a Chemical shifts, in ppm from TMS, I–V in CDCl₃ and VI–XI in DMSO-*d*₆; 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 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 (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 ^a	XI	Assignment
2545w	2560m	2520w	2410m	2475m	2418w	N—H str.
2418w	2418m	2420w	2380m	2430m	2380w	
2335w	2345m			249w 228w	247w 230w	Te—Cl str. ν_3 $\nu_2(A_1)$
277s	275s	279w	270m			$\nu_7(E)$
266m		267m	260m			
254s	255(sh)		258(sh)			
248s	242s	247w				

^a Raman Spectrum shows bands at 340m, 286m and 249m for ν_1 and ν_2 .

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 R_2TeCl_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 $\text{Te}\text{--}\text{N}$ stretching frequency. The appearance of $\text{Te}\text{--}\text{Cl}$ stretching frequencies in the region where R_2TeCl_2 absorbs [13] suggests the structure of $\text{R}(\text{R}'_2\text{N})\text{TeCl}_2$ must be similar to R_2TeCl_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 ν_1 and ν_2 , respectively, Raman active) and 228 (for ν_3 , IR active) 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 $\text{Te}\text{--}\text{Cl}$ symmetric and asymmetric

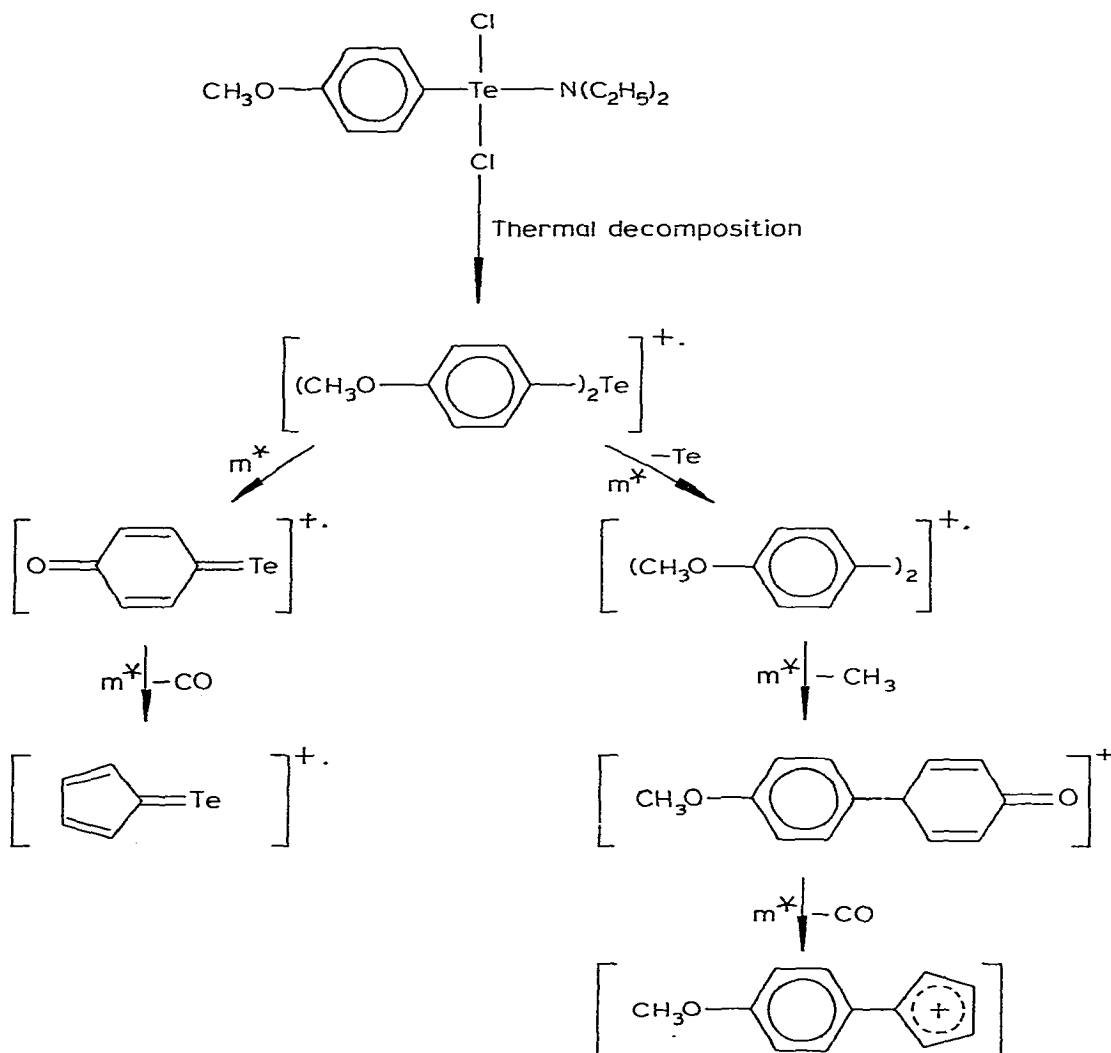


Fig. 1. Partial fragmentation scheme for I.

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 ^1H NMR spectra of the compounds. A linear relationship has been found between the chemical shift of the NH proton signal, (relative to the CH_2 signal), and temperature. An increase of temperature from -40 to 50°C , e.g., in VI, causes the NH proton signal to shift toward the 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 RTeCl_2^+ , RTeCl^+ , R_2Te^+ , RTe^+ , Te_2^+ , R_2^+ , TeCl^+ , Te^+ , RCl^+ , $\text{R}'_2\text{NH}^+$, $\text{R}'_2\text{N}^+$, $\text{C}_4\text{H}_{10}^+$, C_3H_7^+ , C_2H_6^+ and C_3H_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 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 RTeCl_2^+ and TeCl_3^+ , respectively, as the highest mass cluster detected other than Te_2^+ .

Experimental

The reactions were carried out in flame dried glassware under P_2O_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. $\text{Me}_3\text{-SiCl}$, Et_2NH and $\text{C}_4\text{H}_8\text{NH}$ were purchased from Alfa Inorganics and were used after drying and distillation. 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. ^1H 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 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. RTeCl_3 ($\sim 7\text{--}10\text{ mmol}$) and 50 ml of anhydrous benzene were placed in the flask and

$\text{Me}_3\text{SiNR}'_2$ (~ 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°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°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 CH_2Cl_2 /petroleum ether gave bright yellow needles of I, II, or III. Mass spectrum for a representative compound, with relative abundances for isotopes ^{130}Te and ^{35}Cl is given below.

Mass spectrum of I: M^+ (2.4%), R_2Te^{++} (13.0%), RTeCl_2^+ (3.0%), RTeCl^{++} (68.0%), $\text{C}_6\text{H}_4\text{OTeCl}^+$ (12.3%), RTe^+ (63.4%), $\text{C}_6\text{H}_4\text{OTe}^{++}$ (20.7%), 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%), TeCl^+ (20.1%), RCl^+ (36.6%), Te^{++} (9.8%), RTe^{++} (1.2%), 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: RTeCl_2^+ (0.6%), Te_2^+ (2.8%), RTeCl^{++} (5.0%), RTe^+ (13.4%), TeCl^+ (1.0%), R_2^{++} (2.2%), Te^{++} (5.0%), RCl^+ (5.0%), 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%), 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°C) (100 ml) was added to the filtrate, yellow needles of IV or V were separated which were recrystallized from CH_2Cl_2 /petroleum ether (30 – 60°C).

Mass spectrum of V: RTeCl_2^+ (0.2%), R_2^{++} (1.0%), RTeCl^{++} (1.0%), RTe^+ (4.2%), Te_2^+ (2.3%), $\text{C}_6\text{H}_4\text{OTe}^{++}$ (0.8%), RCl^+ (5.8%), $\text{C}_5\text{H}_4\text{Te}^{++}$ (1.6%), R^+ (4.7%), TeCl^+ (2.6%), 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%), C_4H_8^+ (100.0%), C_4H_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 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 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°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 CCl_4 also gave a 1 : 1 adduct, m.p. 132 – 135°C .

Reaction of 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 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/ CH_2Cl_2 to give X. A similar reaction of TeCl_4 with $\text{Me}_3\text{SiNC}_4\text{H}_9$ gave XI.

Reaction of Ph_2TeCl_2 with Et_2NLi

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 Et_2NH in 20 ml anhydrous ether at -10°C . BuLi (1.06 g, 16 mmol) was admitted slowly through septum so that a white precipitate of Et_2NLi was formed. The septum was then replaced with an addition funnel and Ph_2TeCl_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 LiCl . The filtrate was fractionally distilled to obtain yellow oil of Ph_2Te at $112^\circ\text{C}/1\text{ mmHg}$. Yield 85%.

A similar reaction of 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°C , literature [1] $53\text{--}54^\circ\text{C}$.

Acknowledgements

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