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Synthesis and characterization of bis(aryltelluro)methanes

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Abstract

The reaction of diarylditellurides with diazomethane was used to prepare 7 new bis(aryltelluro)methanes, $ArTeCH_2TeAr$. These compounds were characterized by their ^1H, ^{13}C NMR and IR spectra and elemental analysis. The ^{13}C NMR spectra of the corresponding ditellurides are published for the first time. The influence of the tellurium atoms upon the chemical shifts of the methylene group correlate for ^1H and ^{13}C NMR spectra.

Introduction

In recent years selenium acetals and mixed acetals containing selenium and other main-group elements have been shown to be very promising in the field of organic chemistry [1–3]. A group of compounds closely related to these are the bis(aryltelluro)methanes, $(ArTe)_2CH_2$. While the synthesis of these telluroacetals was first reported in 1970 [4] and subsequently synthetic developments have been described [5–10], little attempt has been made to spectroscopically characterize these compounds. There have been previous reports on the preparation of some derivatives: anisyl-, phenethyl- [4]; phenyl- [5,8,10]; benzo[*b*]-thien-2-yl-, *N*-tosyl-2-indolyl- [6]; phenethyl- [7]; and ferrocenyl- [9].

Bis(aryltelluro) methanes are also important reagents in organic synthesis [5,11,12], and constitute starting materials for the preparation of other kinds of organotellurium compounds [2]. Systematic studies for the synthesis and spectroscopic characterization (^{125}Te, ^{13}C, ^1H NMR and Mössbauer) of bis(alkyltelluro)methanes [8], di- and poly-telluroethers [10,13,14], and symmetric and unsymmetric dialkylditellurides [15] have appeared recently The lack of information and our interest in this field [16] led us to synthesize some of the title compounds and to study some of their spectroscopic properties.

Seebach [5] obtained bis(phenyltelluro)methane from lithium phenyltellurolate and diiodomethane in 6% yield. Herberhold obtained bis(ferrocenyltelluro)methane from lithium ferrocenyltellurolate and dichloromethane in 12% yield [9], Engman [6] used lithium aryltellurolates and dichloromethane, obtaining acceptable yields (68%).

Hope [10,13] used frozen mixtures of lithium alkyl- and aryl-tellurolates with diiodomethane in liquid nitrogen baths to produce bis(alkyl- and bis(aryltelluro)methanes in 66% yield. Karnika De Silva [7] using sodium phenyltellurolate and diiodomethane or dibromomethane, obtaining the bis(phenyltelluro)methanes that contain one mole of dihalogenomethane in each case. In this work we used the original method described by Petragnani [4] and used also by Jones [8], treating diarylditellurides dissolved in benzene with a solution of diazomethane prepared from *N*-nitroso-*N*-methylurea in ether at 0 ° C.

Results and discussion

Synthesis of bis(aryltelluro)methanes

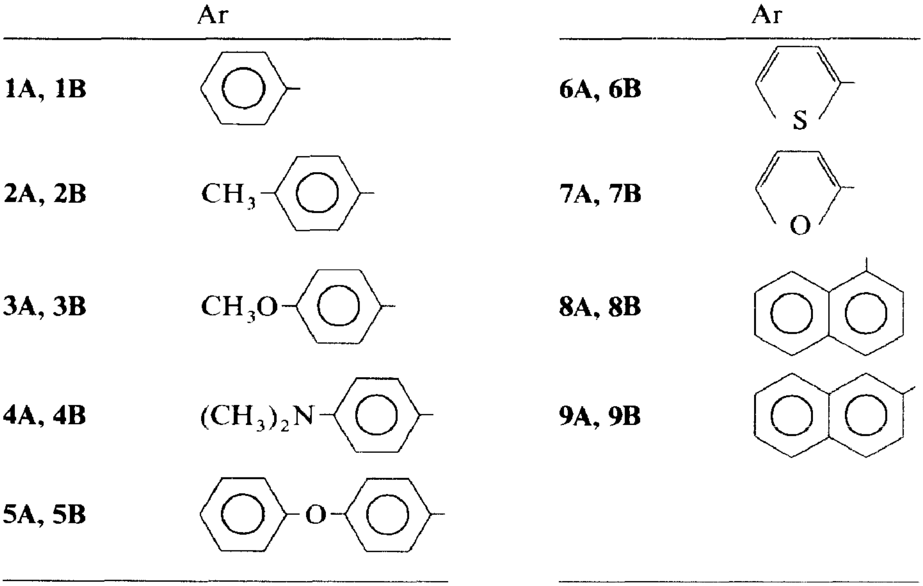
The insertion reaction of methylene (carbene), from diazomethane into the Te–Te bond at 0 ° C has previously been described and a carbene mechanism proposed [4,17]. This reaction is illustrated in Scheme 1; compounds **2B**, **4B–9B** are new.

In this work, some reactions were run at −78 ° C in ethyl ether, however, the yields were not improved. In the reaction at 0 ° C, in all cases a small quantity of liquid material ca. 3–4% is also produced. The malodorous smell suggests that a dialkyl- or arylalkyl-telluride is formed, and ^{13}C and ^{1}H indicate its presence. These minor products are under further investigation.

^{1}H NMR Spectra

The proton NMR spectra of bis(aryltelluro)methanes (Table 1) prepared can be considered in two ways: first, the aromatic protons: the *para*-substituted rings

$$\underset{\textbf{(A)}}{\text{ArTeTeAr}} + \text{CH}_2\text{N}_2 \xrightarrow[\text{Benzene, 0 °C}]{\text{Ether}} \underset{\textbf{(B)}}{\text{ArTeCH}_2\text{TeAr}} + \text{N}_2$$



Scheme 1.

Table 1

Proton NMR data for bis(aryltelluro)methanes and corresponding diaryl ditellurides (δ ppm)

1B	7.78 (m,4H); 7.28(m,6H), 3.85 (S,2H,^{2}J(Te–H) 21 Hz)
1A	7.70–7.95(m,4H); 7.08–7.50(m,6H)
2B	7.75(d,4H,J 9 Hz); 7.01(d,4H,J 9 Hz); 3.84(S,2H ^{2}J(Te–H) 14Hz); 2.34(S,3H)
2A	7.80(d,4H,J 9 Hz); 7.07(d,4H,J 9 Hz); 2.40(S,3H)
3B	7.72(d,4H,J 9 Hz); 6.70(d,4H,J 9 Hz); 3.90(S,2H, ^{2}J(Te–H) 27 Hz); 3.80(S,3H)
3A	7.56(d,4H,J 9 Hz); 6.62(d,4H,J 9 Hz); 3.70(S,3H)
4B	7.64(d,4H,J 9.6 Hz); 6.52(d,4H,J 9.6 Hz); 3.71(S,2H, ^{2}J(Te–H) 19 Hz)
4A	7.68(d,4H,J 9.6 Hz); 6.54(d,4H,J 9.6 Hz); 2.95(S,3H)
5B	7.85(d,4H,J 9.6 Hz); 7.02(d,4H,J 9.6 Hz); 7.17–7.60 (m,10H); 3.80(S,2H, ^{2}J(Te–H) 19 Hz)
5A	7.88(d,4H,J 9.6 Hz); 6.86(d,4H,J 9.6 Hz); 7.00–7.60 (m,10H)
6B	H(3):7.27(d,2H, J(H(3)–H(4)) 3.44 Hz, d,J(H(3)–H(5) 1.02 Hz); H(4); 6.59(2H,d,J(H(4)–H(5)) 5.16 Hz,d,J(H(4)–H(3)) 3.44 Hz); H(5): 6.96(d,2H,J(H(5)–H(3)) 1.02 Hz,d,J(H(5)–H(4)) 5.13 Hz); 3.36 (S, 2H) (^{2}J(Te–H) 24 Hz)
6A	H(3):7.47(d,2H,J(H(3)–H(4)) 3.48 Hz, d,J(H(3)–H(5)) 1.03 Hz); H(4):6.95(2H,d J(H(4)–H(5)) 5.13Hz, d,J(H(5)–H(4)) 5.13 Hz)
7B	H(3): 6.82(d,2H); H(4): 6.39(t,2H); H(5): 7.57(d,2H); 3.66(S,2H, ^{2}J(Te–H) 24 Hz)
7A	H(3): 6.59(d,2H; H(4):5.92(t,2H) H(5):7.08(d,2H)
8B	(6.97–8.20,m 14H) 3.85(S,2H, ^{2}J(Te–H) 18 Hz);
8A	7.65–8.15(m,6H) 7.05–7.55(m,8H)
9B	8.20(broad, S2H); 7.57–7.88(m,8H), 734–7.57(m, 4H); 4.01(S,2H, ^{2}J(Te–H) 20 Hz
9A	8.30(broad S,2H); 7.20–8.10m, 12H.

present a typical AA′BB′ spectral pattern, with the protons *ortho* to tellurium at lower field (deshielded), and the *meta*-protons at higher field. The chemical shifts for the tolyl- [18–20], anisyl- [20], and phenyl- [21] derivatives are close to values in the literature. The values for **4B–9B** are from this work. In **1A** and **1B** the *ortho*-^{1}H resonances show different δ from reported [24] but show the same trend within the series of compounds prepared in this work. In general, there are no significant differences in the chemical shifts of the bis(aryltelluromethanes) **B** compared to the corresponding diarylditellurides **A**. However, for the bis(thienyltelluro)methane **6B**, the H(3) and H(4) protons are at 0.20 ppm and for H(5) at 0.30 ppm higher than for **6A** δ values. For bis(furyltelluro)methane **7B** the H(3) (0.23), H(4) (0.47) and H(5) (0.49) are at higher δ vaues than for the corresponding ditelluride **7A**.

In the second place, the chemical shift of the methylene protons inserted in the Te–Te bond is at δ 3.36–4.01, in agreement with literature, as shown by the following known examples: **1B** δ 3.80 [5]; δ 3.75 [10]; **3B** δ 3.77 [4]; bis(*p*-ethoxyphenyltelluro)methane δ 3.81 [4.7]; (ferrocenyltelluro)methane δ 3.51 [9]; bis(trichlorotelluro)methane δ 4.08 [22]; bis(benzo[*b*]-thien-2-yltelluro)methane δ 3.93 [6]; and bis((*N*-tosyl-2-indolyl)telluro)methane δ 3.91 [6]. The proton signals for the methylene group between the two tellurium atoms occur as a sharp singlet flanked by well defined satellites with coupling constants $^{2}J(^{125}\mathrm{Te}-^{1}\mathrm{H})$ 18–27 Hz that correlate well for Te–CH_2 [10,21]. The methylene protons are deshielded by two tellurium atoms and by the aromatic substituent, as can be concluded from the δ 3.4 for bis(methyltelluro)methane [10] where only the two tellurium atoms deshield the protons, and from CH_3Te, δ 1.8; $CH_3TeTeCH_3$, δ 2.6 [10] and CH_2Te δ 2.85 [13] where only one tellurium atom deshields the protons. In our work, the effect is less for the thienyltelluro-substituent δ 3.36 and larger for the β-naphthyltelluro substituent δ 4.01.

$^{13}C\{^1H\}$ NMR spectra

The literature of ^{13}C NMR chemical shifts in organotellurium compounds is rather sparse. Although the synthesis of aryl- and alkyl-telluromethanes has been published and the $^{125}Te\{^1H\}$ NMR studied, the $^{13}C\{^1H\}$ NMR has not been reported [8]. $^{13}C\{^1H\}$ chemical shifts of bis(phenyltelluro)methane are reported [10] and the chemical shift of methylene assigned at δ −6.2. It is remarkable that in several measurements of ^{13}C spectra of this compound we obtain δ −37.4 for $TeCH_2Te$. The first reported ^{13}C NMR for this kind of methylene appeared in 1987 [9], in bis(ferrocenyltelluro)-methane, with δ −35.1. In this work we obtain for the substituted benzene derivatives values of δ −35.29 to −37.40 ppm, for other derivatives: furyl −33.66, thienyl −29.24, β-naphthyl −21.16 and α-naphthyl −18.72 ppm. The ^{13}C NMR chemical shifts have been assigned for some ditellurides: phenyl- [21,23,24]; *p*-anisyl- [23] in other related compounds: phenyl- [10,13,18], anisyl- [9], tolyl- [18].

Owing to the lack of the ^{13}C NMR chemical shift data for the other diaryl ditellurides used in this work, we report for the first time (as far as we know) these spectra for the compounds **4A** to **9A** (Table 2). We also report the ^{13}C NMR data for the bis(aryltelluro)methanes (Table 3), from which the 7 compounds **2B, 4B–9B** are new. The assignments of the signals of the ring carbon atoms of Tables 2 and 3, were made by the use of closely related aromatic compounds [10,13,23–25] for phenyl derivatives; [9,23,25–27] for anisyl derivatives; [26] for tolyl-, *p*-*N*,*N*-dimethylaminophenyl- and phenoxyphenyl derivatives; [25] for furyl- and thienyl derivatives; [25,28] for naphthyl derivatives.

The assignments of peak frequencies to the phenyl and *p*-substituted rings are relatively easy by the fact that signals for C(1) and C(2) contain satellites, due to spin–spin coupling between ^{125}Te and ^{13}C atoms, while C(3) and C(4) do not; however, sometimes these are not observed [23]. For all the compounds the C(1) signal is the smallest because carbon atoms not bonded to any hydrogen atoms generally relax one order of magnitude slower than hydrogen-bearing carbon atoms [24]. The C(4) in mono-substituted benzenes relaxes much faster than quaternary carbon atoms, and faster than C(2) and C(3) [25]. Following the same methods, we made the assignments for naphthalene derivatives. In the case of furyl- and thienyl derivatives the assignment of signals is somewhat more complicated because C(2) does not contain satellite signals, while C(3), C(4) and C(5) contain the same number and form of satellite signals. The ^{13}C-chemical shift of some ditellurides has been qualitatively rationalized [18,24].

IR spectra

The IR spectra are known for some of the diarylditellurides synthesized [36,37] in this work. The important feature is that all of the **B** compounds exhibit new bands

(*continued on p. 75*)

Table 2

Carbon-13 NMR data [a] and substituent shifts [b] of diaryl ditellurides

Compound	Solvent	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	C(10)	CH_3	^1J(C–Te)	^2J(C–Te)
$(C_6H_5Te)_2$	C_6H_6	108.3	137.8	129.4	128.1								56.1	21.0
(1A)		(−20.4)	(+9.1)	(+0.7)	(−0.6)									
$(p\text{-}MeC_6H_4Te)_2$	$CDCl_3$	104.1	138.0	130.0	138.1							21.0		22.0
(2A)		(−21.5)	(+9.3)	(+0.7)	(+0.1)									
$(p\text{-}MeOC_6H_4Te)_2$	$CDCl_3$	98.8	140.0	115.2	160.0							55.5	57.4	20.6
(3A)		(−22.0)	(+10.4)	(+1.1)	(0.0)									
$(p\text{-}Me_2NC_6H_4Te)_2$	$CDCl_3$	92.3	140.5	113.6	150.9							39.9		20.0
(4A)		(−24.6)	(+11.0)	(+0.6)	(−0.2)									
$(p\text{-}C_6H_5OC_6H_4Te)_2$	$CDCl_3$	100.4	140.0	129.8	156.6	158.2	119.4	123.7	119.4					
(5A)		(−23.0)	(+11.0)	(+10.6)	(+1.2)	(+0.4)	(+0.2)	(−5.3)	(−4.0)					
(2-ThienylTe)$_2$	$CDCl_3$		93.8	141.3	128.4	134.8								
(6A)			(−31.8)	(+13.9)	(+1.0)	(+9.2)								
(2-FurylTe)$_2$	C_6D_6		112.7	126.6	112.2	149.1								
(7A)			(−35.5)	(+16.8)	(+2.4)	(+6.3)								
$(1\text{-}C_{10}H_7Te)_2$	$CDCl_3$	111.5	140.6	128.7	126.7	130.1	126.2	126.2	132.8	133.4	136.2			
(8A)		(−16.8)	(+14.5)	(+2.6)	(−1.6)	(−1.8)	(−0.1)	(−0.1)	(−4.5)	(+0.5)	(−2.3)			
$(2\text{-}C_{10}H_7Te)_2$	$CDCl_3$	137.6	105.8	134.9	127.7	127.4	126.5	126.3	128.6	133.0	134.0			
(9A)		(+9.3)	(−20.3)	(+8.4)	(−0.6)	(−0.9)	(−0.4)	(−0.2)	(−0.3)	(−0.9)	(−0.1)			

[a] Chemical shift values in ppm, coupling constants in Hz. [b] Given in parentheses.

Table 3

Carbon-13 NMR data [a] and substituent shifts [b] of bis(aryltelluro)methanes

Compound	Solvent	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	C(10)	CH_2	CH_3	^1J(C–Te)	^2J(C–Te)
$(C_6H_5Te)_2CH_2$	$CDCl_3$	103.5	137.9	128.0	129.3							−37.4		166.0	20.5
(1B)		(−25.2)	(+9.2)	(−0.7)	(+0.6)										
$(p\text{-}MeC_6H_4Te)_2CH_2$	$CDCl_3$	100.2	138.3	130.1	137.9							−36.7	21.1		
(2B)		(−25.4)	(+9.6)	(+0.8)	(−0.1)										
$(p\text{-}MeOC_6H_4Te)_2CH_2$	$CDCl_3$	105.1	140.7	115.3	160.0							−35.6	55.2	156.0	20.3
(3b)		(−15.7)	(11.1)	(+1.2)	(0.0)										
$(p\text{-}Me_2NC_6H_4Te)_2CH_2$	$CDCl_3$	98.9	139.7	112.8	149.8							−35.3	39.5	212.0	25.0
(4B)		(−18.0)	(+10.2)	(−0.2)	(−1.3)										
$(p\text{-}C_6H_5OC_6H_4Te)_2CH_2$	$CDCl_3$	108.1	140.4	129.8	156.6	157.9	119.3	123.7	119.6			−36.1			
(5B)		(−15.3)	(+11.4)	(+10.6)	(−1.3)	(+0.1)	(+0.1)	(−5.3)	(−3.8)						
(2-ThienylTe)$_2CH_2$	C_6D_6		102.7	141.7	129.1	134.7						−29.2		219.0	27.5
(6B)			(−22.5)	(+14.3)	(+1.7)	(+8.9)									
(2-FurylTe)$_2CH_2$	C_6D_6		123.5	126.4	112.0	148.7						−33.7		216.0	26.9
(7B)			(−24.7)	(−17.1)	(+2.2)	(+5.9)									
$(1\text{-}C_{10}H_7Te)_2CH_2$	$CDCl_3$	119.3	140.6	131.8	129.5	126.8	126.1	128.8	131.9	138.5	133.6	−18.7			
(8B)		(−9.0)	(+14.5)	(+5.7)	(+1.2)	(−1.5)	(0.0)	(+2.7)	(+3.6)	(+1.4)	(+0.3)				
$(2\text{-}C_{10}H_7Te)_2CH_2$	$CDCl_3$	137.9	113.1	134.2	127.8	127.4	126.4	126.3	128.4	132.8	134.2	−21.2			
(9B)		(+9.6)	(−13.0)	(+8.1)	(−0.9)	(−0.9)	(−0.3)	(+0.2)	(+0.1)	(+1.1)	(−0.3)				

[a] Chemical shift values in ppm, coupling constants in Hz. [b] Given in parentheses.

Table 4

Infrared data for bis(aryltelluro)methanes and corresponding diarylditellurides (cm^{-1}) [a]

1B	1570, 1470, 1430, 1380, 1065, 1040*, 1020, 1000, 735, 695, 660, 465, 375, 368, 338*
1A	1565, 1430, 1380, 1320, 1060, 1015, 998, 745, 695, 670, 660, 466, 355
2B	1585, 1480, 1435, 1410, 1390, 1310, 1270, 1240, 1185, 1045, 1010*, 1000*, 890, 795, 770, 640
2A	1585, 1480, 1440, 1385, 1298, 1205, 1178, 1105, 1030, 1005, 795
3B	1580, 1560, 1480, 1450, 1430, 1390, 1300, 1290, 1285, 1180, 1100*, 1070*, 1060, 1020, 995, 830, 800, 785, 665*, 660, 530, 340*, 310
3A	1580, 1560, 1480, 1455, 1430, 1280, 1245, 1175, 1115, 1055, 1030, 1000, 830, 810, 790, 600, 520, 300
4B	1580, 1490, 1430, 1350, 1310, 1220, 1200, 1165, 1130, 1070, 1020*, 1000, 950, 805, 750, 670*, 525, 340*
4A	1580, 1500, 1480, 1355, 1230, 1200, 1170, 1070, 995, 950, 810, 755, 525
5B	1605, 1595, 1580, 1480, 1300, 1275, 1240, 1200, 1170, 1010*, 870, 830, 790, 750, 690, 510, 490
5A	1580, 1495, 1485, 1275, 1240, 1180, 995, 870, 825, 810, 795, 750, 690, 505, 490
6B	1415, 1380, 1320, 1310, 1245, 1210, 1070, 1040, 930, 890, 840, (830–800)*, (750–680), 610
6A	1410, 1385, 1320, 1245, 1210, 1075, 1040, 935, 890, 840, 825, (760–675), 610
7B	1440, 1415, 1360, 1250, 1205, 1145, 1085, 1045, 995, 890, 880, (770–670)
8B	1650, 1640, 1590, 1560, 1500, 1450, 1380, 1330, 1250, 1130, 1060, 1040*, 1020, 950, 790, 770, 640, 520, 410
8A	1650, 1645, 1580, 1560, 1500, 1450, 1380, 1340, 1260, 1130, 1060, 1020, 790, 770, 640, 510, 410
9B	1625, 1585, 1560, 1500, 1345, 1330, 1270, 1200, 1130, 1060, 1040*, 1020, 960, 940, 890, 845, 830, 815, 740, 690, 620, 480
9A	1620, 1585, 1560, 1505, 1345, 1340, 1270, 1205, 1135, 1060, 1020, 960, 950, 890, 840, 830, 810, 740, 690, 620, 480

[a] See text.

relative to the **A** compounds. These are assigned to the CH$_2$ groups and are indicated in Table 4 by means of an asterisk.

Experimental Section

Melting points were determined on an electrically heated hot stage (Koffler type, Bristol-line) and are uncorrected. In the mass spectra all tellurium compounds mass peaks, are reported for ^{130}Te. ^{1}H NMR spectra for the compounds synthesized were obtained using: a Varian T-60 instrument for **1A** to **5A** and **1B** to **5B**; Varian XL-200 for **6A–7A** and **6B–7B**; Perkin Elmer R-24 for **8A–9A** and **8B–9B**. ^{13}C NMR spectra of the compounds were obtained using: a Bruker WP80 instrument for **2A–5A** and **8A–9A, 1B, 3B–5B, 8B–9B**; Varian XL-200 for compounds **1A, 6A–7A, 2B, 6B–7B**. All the spectra were recorded for ca. 10% solutions in CDCl$_3$ or C$_6$D$_6$ containing Me$_4$Si as an internal standard and are reported in ppm (δ) units. IR spectra for the compounds except **2B, 6A–7A, 6B–7B**, were recorded with a Perkin Elmer 467 Grating instrument for KBr disks, principal bands are reported in cm^{-1}. Tellurium was determined gravimetrically [29]. Carbon and hydrogen were estimated microanalytically. Mass spectra for **3B**: Varian MATCH 7 (ionization energy 70 eV).

The syntheses of diaryl ditellurides for this work were made using the following routes: **1A, 2A, 4A, 8A** and **9A** from the corresponding amines and through the diazonium salt gave the arylmercury chlorides [30], which were treated with tellurium tetrachloride in dioxane to obtain the aryltellurium trichlorides [29,31]; for

3A [32] and **5A** [33], we directly treated anisol and diphenyl ether with tellurium tetrachloride in carbon tetrachloride to get the corresponding trichlorides. All these aryltellurium trichlorides were reduced with sodium sulfide to get the diaryl ditellurides [34]. **6A** and **7A** were synthesized using a transmetalation reaction [6]. Tellurium tetrachloride was prepared from Te^0 and $Cl_{2(g)}$ [29].

Diazomethane was prepared by decomposition of *N*-nitroso-*N*-methylurea with a 50% solution of potassium hydroxide and trapped in ethyl ether and dried over potassium hydroxide pellets [35].

Synthesis of bis(aryltelluro)methanes

All preparations were performed in a hood. 0.005 mole of diaryl ditelluride dissolved in 50 ml of dry benzene was placed in a three necked 250 ml round bottom flask fitted with a gas inlet and an overpressure mercury valve and magnetic stirring. At 0 ° C a solution of standardized diazomethane in ether, 0.006 mole (10% excess) was added with a pipette while a brisk stream of nitrogen was passed through the 3rd open neck. In all cases the red color of the ditelluride disappeared almost completely in ca. 10 minutes. The reaction mixture was warmed up to room temperature and stirred two more hours to eliminate the unreacted diazomethane, the solvent was removed and the product recrystallized.

*Bis(phenyltelluro)methane (**1B**)*

The reaction product, an oily clear greenish material, was treated with hexane and allowed to stand overnight at -20 ° C, forming beige crystals which by filtering give 1.95 g (89.4% yield), m.p. 33–34 ° C (Lit. [5] 35–36 ° C. IR 1040 cm^{-1}. Found: C, 36.58; H,2.00; Te, 60.55. $C_{13}H_{12}Te_2$ calcd.: C, 36.88; H, 2.1; Te, 60.27%.

*Bis(p-methylphenyltelluro)methane (**2B**)*

The reaction product was an oily clear greenish semisolid (all the spectra were measured with this material) which didn't crystallize in different solvents. When trying to distill this product at 0.1 Torr. it was transformed into a dark red oil at ca. 105 ° C without distillation, owing probably to decomposition by heat to form the ditelluride. In a subsequent preparation, the product was dissolved in CCl_4 and an equimolar quantity of freshly distilled $SOCl_2$, was added at 0 ° C (ice-water bath). After removing the solvent, the crystalline product was dissolved in benzene and hexane added, resulting in crystals (100% yield). This product was shown to be the tetrachloride p-$CH_3C_6H_4Te(Cl_2)CH_2Te(Cl_2)C_6H_4CH_3$ by ^1H and ^{13}C NMR spectra, m.p. 163–165 ° C. Found: C, 29.96; H, 2.56; Te. 43.55. $C_{15}H_{16}Te_2Cl_4$ calcd.: C, 30.36; H, 2.72; Te, 43.01%.

*Bis(p-methoxyphenyltelluro)methane (**3B**)*

The reaction product was an off-white crystalline solid. It was redisolved in benzene and precipitated with hexane to produce brilliant needles; filtration gave 2.05 g (93.5% yield). m.p. 102–103 ° C (Lit. [4] 98–99 ° C). Mass spectrum: *m/e* (relative intensity) $[M]^+$ 488 (4.5), 476 (2.5%) 344 (4.2%) 251 (3.8%), 237 (7.2%), 214 (8.0%), 199 (5.2%), 121 (100%). IR: 1070 cm^{-1}.

*Bis(p-N,N-dimethylaminophenyltelluro)methane (**4B**)*

The reaction product was a yellowish powder with an oily appearance. It was washed with hexane and filtrated, redissolved in benzene and precipitated with hexane to give 2.4 g (94.2%) of a white yellowish powder m.p. 74–76 °C. IR: 1020 cm^{-1}. Found: C, 41.00; H, 4.17; Te, 49.50. $C_{17}H_{22}N_2Te_2$ calcd.: C, 40.07: H, 4.35; Te, 50.08%.

*Bis(p-phenoxyphenyltelluro)methane (**5B**)*

In this case the color of the ditelluride dissappeared but the solution became a different red color. After the work up and removal of the solvent, a reddish semisolid was obtained. Redissolving in benzene, precipitation with hexane and filtration gave 2.25 g of a white powder (74.1% yield), m.p. 114 °C, IR: 1010 cm^{-1}. Found: C, 49.57; H, 3.39; Te, 41.36. $C_{25}H_{20}O_2Te_2$ calcd.: C, 49.41; H, 3.32: Te, 42.00%.

*Bis(2-thienyltelluro)methane (**6B**)*

The reaction product was a red semisolid which gave 3 spots on TLC (alumina/hexane). Purification was accomplished by column chromatography on alumina. The principal fraction was separated as a greenish liquid that decomposed at −76 °C and 0.3 Torr, before it began to distill. Using a similar procedure as in **2B**, was obtained 2-thienylTe(Cl_2)CH_2Te(Cl_2)thienyl, which was characterized by ^1H and ^{13}C NMR spectra, m.p. 205 °C (dec). Found: C, 19.25; H, 1.46. $C_9H_8Te_2Cl_4S_2$ calcd.: C, 18.72; H, 1.40%.

*Bis(2-furyltelluro)methane (**7B**)*

The oily red product was passed through a chromatographic column (alumina, pentane), and the 2nd fraction was collected. The solvent was partially removed, the concentrate refrigerated overnight at −20 °C to give a yellow crystals, m.p. −15 to −10 °C. Found: C, 27.03, H, 2.09. $C_9H_8O_2Te_2$ calcd.: C, 26.80; H, 2.00%.

*Bis(1-naphthyltelluro)methane (**8B**)*

The reaction product was a red semisolid. Redissolving in benzene and precipitation with hexane gave 2.00 g (76.4% yield) of a white crystalline powder, m.p. 89–90 °C, IR: 1040 cm^{-1}. Found: C, 48.50; H, 3.01; Te, 49.05. $C_{21}H_{16}Te_2$ calcd.: C, 48.17; H, 3.08; Te, 48.75%.

*Bis(2-naphthyltelluro)methane (**9B**)*

The reaction product was a very fine beige colored powder. Redissolving in benzene and precipitation with petroleum ether (40–60 °C) gave 2.59 g (99.00% yield) of a beige powder, m.p. 107–109 °C, IR: 1040 cm^{-1}. Found: C, 48.02; H, 3.02; Te, 48.60. $C_{21}H_{16}Te_2$ calcd.: C, 48.17; H, 3.08: Te, 48.75%.

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