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Reduction of Organotellurium Trichlorides with Sodium Borohydride

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Summary: Organotellurium trichlorides are efficiently reduced with sodium borohydride. The intermediate organo tellurolates are transformed into diaryl ditellurides, alkyl aryl tellurides, tellurides containing a cyclic ether, and vinylic tellurides.

Introduction

Organotellurium trichlorides are easily obtained by a number of methods[1] and are convenient precursors of diorgano ditellurides, which are widely used in organotellurium chemistry.[1,2] The transformation of organotellurium trichlorides into the corresponding ditellurides has been performed using several reducing agents, the most common being sodium sulfide nonahydrate and hydrazine hydrate.[1,2] However, these reagents present drawbacks, such as the high temperature and the great excess of the reducing agent required in the case of sodium sulfide, which produces great amounts of malodorous aqueous byproducts. Hydrazine hydrate is toxic and a suspected carcinogen.

The widely used reducing agent sodium borohydride has not yet been used to reduce organotellurium trichlorides to the corresponding ditellurides.[3] We report here the transformation of organotellurium trichlorides into diorgano ditellurides and diorgano tellurides using sodium borohydride as the reducing agent.

Results and Discussion

The transformation of $ArTeCl_3$ (**1**), into ArTeTeAr (**2**), was effected by adding aqueous $NaBH_4$ to a solution of **1** in THF at 0 °C (eq 1). The color of the solution turned immediately from yellow to dark red and gas evolution was observed.

$$\underset{\mathbf{1}}{ArTeCl_3} \xrightarrow[\text{THF, 0 °C, air}]{NaBH_4\,/\,H_2O} \underset{\mathbf{2}}{ArTeTeAr} \qquad (1)$$

		Yield
Ar = p-$CH_3OC_6H_4$	(**2a**)	97%
p-$PhOC_6H_4$	(**2b**)	94%
p-$EtOC_6H_4$	(**2c**)	90%
m-FC_6H_4	(**2d**)	95%
Ph	(**2e**)	95%
2-Th	(**2f**)	89%

The products were obtained in high yield after column chromatography on silica gel eluting with hexane. In one case (compound **2a**) the reaction was performed in a 20 mmol scale and the product was obtained in 95% yield, demonstrating that the method is suitable for large-scale preparation of diaryl ditellurides.

To our knowledge the one-pot transformation of organotellurium trichlorides into diorgano tellurides has not yet been described. The $NaBH_4$ method serves nicely for this purpose. The aqueous $NaBH_4$ solution was added to a solution of the appropriate organotellurium trichloride and an alkyl halide in THF at 0 °C under nitrogen. After normal workup and column chromatography on silica gel the alkyl aryl telluride **3** was obtained in high yield (eq 2).

$$\underset{\mathbf{1}}{ArTeCl_3} \xrightarrow[\text{0 °C, N}_2]{\substack{NaBH_4\,/\,H_2O \\ RX\,/\,THF}} \underset{\mathbf{3}}{ArTeR} \qquad (2)$$

			Yield
Ar = Ph,	RX = EtBr	(**3a**)	91%
p-$CH_3OC_6H_4$,	RX = MeI	(**3b**)	95%
p-$CH_3OC_6H_4$,	RX = EtBr	(**3c**)	93%
p-$CH_3OC_6H_4$,	RX = $CH_3(CH_2)_2CH(CH_3)Br$	(**3d**)	90%
p-$PhOC_6H_4$,	RX = EtBr	(**3e**)	88%

Application of the above reaction sequence to the cyclization products of unsaturated alcohols **4**[4] with tellurium tetrachloride gave the tellurides containing a cyclic ether **6** in high yield (Scheme 1).

The sequence described in Scheme 1 constitutes a new and advantageous route to tellurides containing a cyclic ether since it occurs in higher yield, lower temperature, and shorter time if compared with the previous methods.[4] It was already demonstrated that tellurides containing a cyclic ether can be detellurated, to give the corresponding tetrahydrofurans and tetrahydropyrans.[4]

Another interesting extension of the transformation shown in eq 2 allowed the synthesis of tetrasubstituted olefins containing an organotellurium substituent (**9**) (Scheme 2). This is the first method developed to synthesize this class of vinyl tellurides. Mono- and disubstituted acetylenes react with tellurium tetrachlo-

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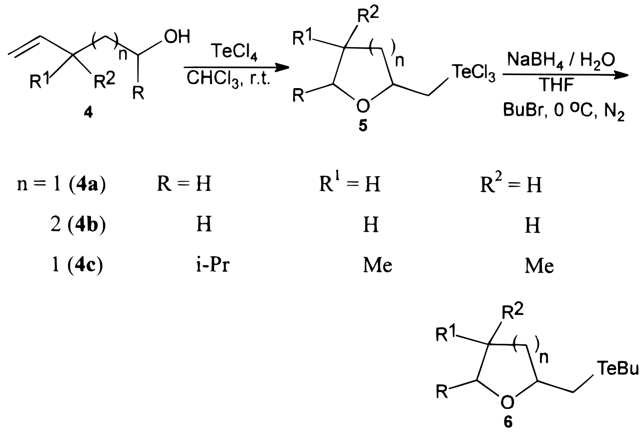
Scheme 1

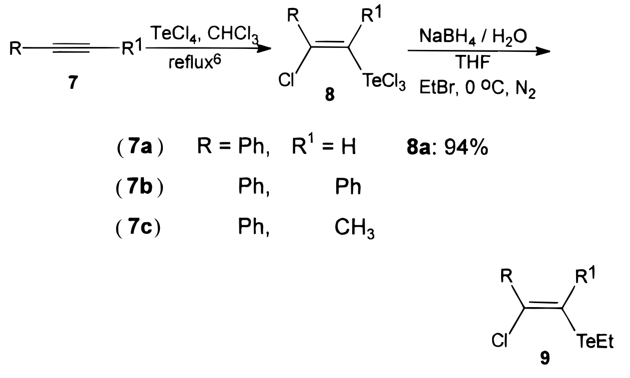


n	R	R^1	R^2
n = 1 (**4a**)	R = H	R^1 = H	R^2 = H
2 (**4b**)	H	H	H
1 (**4c**)	i-Pr	Me	Me

overall yield from **4**

(**6a**) 95%
(**6b**) 90%
(**6c**) 93%

Scheme 2



	R	R^1	
(**7a**)	R = Ph,	R^1 = H	**8a**: 94%
(**7b**)	Ph,	Ph	
(**7c**)	Ph,	CH_3	

overall yield from **7**

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(**9b**): 78%
(**9c**): 75%

ride leading to the (*Z*)-vinyltellurium trichlorides in good yield.[5] Attempts to reduce the vinylic tellurium trichloride **8a** with both sodium sulfide nonahydrate or with sodium borohydride led to decomposition of **8a** with deposition of elemental tellurium. Presumably, an elimination reaction involving a hydrogen and a chlorine *trans* to each other occurs under the reaction conditions, yielding unstable intermediates. However, the reduction of the trichlorides **8b**,**c** with sodium borohydride in the presence of ethyl bromide gave the vinylic tellurides **9b**,**c** in good yield (Scheme 2). Vinylic tellurides are useful precursors for vinyllithium and vinylcopper reagents.[6]

The intermediate (*Z*)-vinyltellurium trichlorides **8** need not be purified prior to the reduction/alkylation sequence, and the yields refer to the overall process starting from the acetylenes **7**. This method complements the previous methodology developed by us for the synthesis of (*Z*)-2-chloro-2-organoyl-1-ethenyl aryl tellurides from acetylenes and aryltellurium trichlorides since these reagents only react with terminal acetylenes.[7]

In conclusion, the reduction of organotellurium trichlorides with sodium borohydride followed by the appropriate manipulation of the intermediate organo tellurolate is an efficient method to prepare diaryl ditellurides, aryl alkyl tellurides, vinylic tellurides, and tellurides containing a cyclic ether.

Experimental Section

^1H (200 and 500 MHz) and ^{13}C (50 MHz and 125 MHz) NMR spectra were obtained on Bruker AC-200 and DRX-500 spectrometers in $CDCl_3$ with TMS as the internal reference. IR spectra were recorded on a Perkin Elmer 1600 spectrophotometer. Low-resolution mass spectra were obtained on a Finnigan 4021 spectrometer or on a GC/MS-Hewlett Packard 5988-8/5890 spectrometer, both operating at 70 eV. Elemental analyses were performed at the Microanalytical Laboratory of the Institute of Chemistry-USP. Column chromatography were carried out with Merck silica gel (230−400 mesh) according to the procedure by Still and co-workers.[8] Thin-layer chromatography (TLC) was performed on silica-gel 60 F-254 on aluminum. All solvents used were previously dried and distilled according to the usual methods.[9] Tellurium (200 mesh) was purchased from Aldrich. Aryltellurium trichlorides and tellurium tetrachloride were prepared according to literature procedures.[1,2] The remaining chemicals were obtained from commercial sources.

Typical Procedure for the Reduction of Aryltellurium Trichlorides with Sodium Borohydride. To a solution of the aryltellurium trichloride (**1**) (5 mmol) at 0 °C in THF (30 mL) was slowly added a solution of $NaBH_4$ (0.56 g, 15 mmol) in water (20 mL). The reaction mixture turned dark red. After the addition the mixture was stirred for 10 min at room temperature and then treated with saturated aqueous solution of NH_4Cl (100 mL) and extracted with ethyl acetate (3 × 20 mL). The extracts were washed with brine (30 mL) and dried with $MgSO_4$. The solvents were evaporated, and the residue was purified by column chromatography on silica gel, eluting with hexane, to give the diaryl ditellurides **2**.

Bis(4-methoxyphenyl) ditelluride (2a): Yield 1.14 g (97%); mp 58−60 °C (lit.[10] mp 58−59 °C).

Bis(4-phenoxyphenyl) ditelluride (2b): Yield 1.40 g (94%); mp 84−86 °C (lit.[13] mp 88−88.5 °C).

Bis(4-ethoxyphenyl) ditelluride (2c): Yield 1.12 g (90%); mp 108−110 °C (lit.[11] mp 107−108 °C).

Bis(3-fluorophenyl) ditelluride (2d): Yield 1.06 g (95%); oil (lit.[12] mp 44 °C).

Diphenyl ditelluride (2e): Yield 0.97 g (95%); mp 64−66 °C (lit.[13] mp 63.8−65 °C).

Bis(2-thienyl) ditelluride (2f): Yield 0.94 g (89%); mp 89−91 °C (lit.[14] mp 89−90 °C).

Typical Procedure for the Transformation of Aryltellurium Trichlorides into Alkyl Aryl Tellurides (3). To a solution of the aryltellurium trichloride (**1**) (5 mmol) and the

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appropriate alkyl halide (7.5 mmol) in THF (60 mL) under a nitrogen atmosphere was slowly added a solution of $NaBH_4$ (0.93 g, 25 mmol) in water (30 mL) at 0 °C. The color changed from yellow to red when the addition of $NaBH_4$ started and to yellow again after the addition was completed.[15] After the addition, the mixture was stirred for 20 min at room temperature and then treated with saturated aqueous solution of NH_4Cl (100 mL) and extracted with ethyl acetate (3 × 20 mL). The extracts were washed with brine (3 × 20 mL) and dried with $MgSO_4$, and the solvents were evaporated. The residue was purified by column chromatography on silica gel, eluting with hexane, to give the aryl alkyl tellurides **3**.

Ethyl phenyl telluride (3a):[16] Yield 1.06 g (91%).

4-Methoxyphenyl methyl telluride (3b): Yield 1.19 g (95%); ^{1}H NMR (200 MHz) δ 7.29 (d, J = 5.2 Hz, 2 H), 6.79 (d, J = 5.2 Hz, 2 H), 5.03 (s, 3 H), 4.07 (s, 3 H); ^{13}C NMR (50 MHz) δ 159.29, 139.26, 114.98, 100.87, 54.84, −16.09; LRMS *m/z* (relative intensity) 252 (M$^+$, 85), 237 (100); IR (KBr, film) (cm^{-1}) 1586, 1489, 1283, 1245, 1178, 820. Anal. Calcd for $C_8H_{10}OTe$: C, 38.47; H, 4.04. Found: C, 38.82; H, 3.84.

Ethyl 4-methoxyphenyl telluride (3c):[17] Yield 1.23 g (93%).

4-Methoxyphenyl 1-methylbutyl telluride (3d): Yield 1.43 g (90%); ^{1}H NMR (200 MHz) δ 7.71 (d, J = 8.8 Hz, 2 H), 6.77 (d, J = 8.8 Hz, 2 H), 3.80 (s, 3 H), 3.33 (sext, J = 7.3 Hz, 1 H), 1.72−1.33 (m, 7 H), 0.88 (t, J = 6.6 Hz, 3 H); ^{13}C NMR (50 MHz) δ 159.47, 142.25, 114.61, 100.33, 54.69, 41.38, 24.22, 22.94, 22.32, 13.43; LRMS *m/z* (relative intensity) 308 (M$^+$, 14), 108 (100); IR (KBr, film) (cm^{-1}) 2957, 2927, 2868, 1586, 1487, 1282, 1243, 1177, 1033, 822. Anal. Calcd for $C_{12}H_{18}OTe$: C, 47.12; H, 5.93. Found: C, 47.40; H, 5.96.

Ethyl 4-phenoxyphenyl telluride (3e): Yield 1.43 g (88%); ^{1}H NMR (200 MHz) δ 7.69 (d, J = 8.1 Hz; 2 H), 7.34 (t, J = 8.1 Hz, 2 H), 7.11 (t, J = 7.4 Hz, 1 H), 7.01 (d, J = 8.1 Hz, 2 H), 6.84 (d, J = 8.8 Hz, 2 H), 2.84 (quart, J = 7.4 Hz, 2 H), 1.63 (t, J = 7.4 Hz, 3 H); ^{13}C NMR (50 MHz) δ 157.34, 156.51, 140.60, 129.64, 123.43, 119.57, 119.15, 103.70, 29.31, 17.25, 0.71; LRMS *m/z* (relative intensity) 328 (M$^+$, 100); IR (KBr, film) (cm^{-1}) 3776, 3692, 3061, 3039, 1577, 1482, 1241, 1195, 1168, 752, 693. Anal. Calcd for $C_{14}H_{14}OTe$: C, 51.60; H, 4.33. Found: C, 51.94; H, 4.35.

Typical Procedure for the Cyclization of Unsaturated Alcohols with Tellurium Tetrachloride and the Reduction/Alkylation Sequence. To a flask containing a suspension of $TeCl_4$ (1.35 g, 5 mmol) in $CHCl_3$ (20 mL) under nitrogen was added the appropriate unsaturated alcohol (1.04 equiv, 5.2 mmol). After 5 min of stirring at room temperature the solvent was evaporated and the residual oil (the organotellurium trichloride **5**, Scheme 1), without purification, was treated as described above using 1-bromobutane (1.37 g, 10 mmol) as the alkylating agent to give the corresponding butyl telluride **6**.

Butyl (tetrahydro-2-furanyl)methyl telluride (6a): Yield 1.28 g (95%); ^{1}H NMR (500 MHz) δ 4.07 (quint, J = 6.6 Hz, 1 H), 3.92 (dd, J = 14.6, 7.1 Hz, 1 H), 3.77 (dd, J = 13.9, 7.9 Hz, 1 H), 2.82 (dd, J = 12.0, 5.8 Hz, 1 H), 2.78 (dd, J = 12.0, 7.3 Hz, 1 H), 2.67 (t, J = 7.6 Hz, 2 H), 2.12−2.07 (m, 1H), 1.96−1.88 (m, 2 H), 1.72 (quint, J = 7.4 Hz, 2 H), 1.59−1.52 (m, 1H), 1.38 (sext, J = 7.4 Hz, 2 H), 0.91 (t, J = 7.3 Hz, 3 H); ^{13}C NMR (125 MHz) δ 79.54, 67.66, 33.77, 32.33, 25.62, 24.47, 12.87, 8.50, 2.41; LRMS *m/z* (relative intensity) 272 (M$^+$, 34), 85 (100); IR (KBr, film) (cm^{-1}) 2957, 2927, 2858, 1459, 1414, 1377, 1351, 1316, 1248, 1184, 1164, 1094, 1054, 921, 867. Anal. Calcd for $C_9H_{18}OTe$: C, 40.06; H, 6.72. Found: C, 40.28; H, 6.57.

Butyl (tetrahydro-*2H*-2-pyranyl)methyl telluride (6b): Yield 1.28 g (90%); ^{1}H NMR (200 MHz) δ 4.01−3.93 (m, 1H), 3.50−3.35 (m, 2H), 2.81 (dd, J = 12.0, 6.7 Hz, 1 H), 2.69 (dd, J = 12.0, 6.0 Hz, 1H), 2.66 (t, J = 7.6 Hz, 2H), 1.87−1.76 (m, 2H), 1.72 (quint., J = 7.3 Hz), 1.50−1.30 (m, 4H), 1.38 (sext., J = 7.3, 2H), 0.91 (t, J = 7.3 Hz, 3 H); ^{13}C NMR (125 MHz) δ 78.46, 67.99, 33.75, 32.50, 25.22, 24.47, 22.93, 12.88, 8.94, 2.41; LRMS *m/z* (relative intensity) 286 (M$^+$, 19), 84 (100); IR (KBr, film) (cm^{-1}) 2954, 2933, 2842, 1463, 1439, 1086, 1049, 1017, 892. Anal. Calcd for $C_{10}H_{20}OTe$: C, 42.31; H, 7.10. Found: C, 42.64; H, 7.28.

Butyl (5-isopropyl-4,4-dimethyltetrahydro-2-furanyl)methyl telluride (6c): Yield 1.58 g (93%; 5:4 mixture of 2 diastereomers); ^{1}H NMR (500 MHz) δ 4.22−4.16 (m, 1 H, major isomer), 4.03−3.98 (m, 1H, minor isomer), 3.17 (d, J = 9.1 Hz, 1 H, major isomer), 3.02 (d, J = 8.4 Hz, 1H, minor isomer), 2.80−2.68 (m, 2 H), 2.66 (t, J = 7.3 Hz, 2H), 1.95 (dd, J = 12.2, 9.6 Hz, 1 H), 1.92 (dd, J = 12.4, 7.5 Hz, 1 H), 1.75−1.66 (m, 4 H), 1.54−1.34 (m, 4 H), 1.09 (d, J = 5.0 Hz, 6 H, major isomer), 0.98−1.00 (m, 6 H), 0.91 (t, J = 7.3 Hz, 3 H), 0.88 (d, J = 6.5 Hz, 6H, minor isomer); ^{13}C NMR (125 MHz) δ 93.23, 91.86, 77.21, 75.91, 51.35, 50.45, 41.70, 40.82, 34.10, 34.00, 29.70, 29.12, 28.21, 27.15, 26.19, 24.80, 24.69, 23.66, 21.55, 20.96, 20.86, 19.54, 18.85, 10.58, 8.22, 2.63, 2.49; LRMS *m/z* (relative intensity) 342 (M$^+$, 12), 97 (93); IR (KBr, film) (cm^{-1}) 2957, 2929, 2871, 1466, 1367, 1061, 1039, 1011, 942. Anal. Calcd for $C_{14}H_{28}OTe$: C, 49.46; H, 8.30. Found: C, 49.82; H, 8.36.

In one case the intermediate trichloride was isolated and identified. **(Tetrahydro-2-furanyl)methyl tellurium trichloride (5a):**[18] Yield 1.52 g (95%).

Typical Procedure for the Synthesis of the Trisubstituted Vinyl Tellurides (9). To a suspension of $TeCl_4$ (1.35 g, 5.0 mmol) in CCl4 (20 mL) under nitrogen was added acetylene (1.1 equiv). The mixture was heated under reflux for 2.5 h, and then the solvent was evaporated. The residue and ethyl bromide (0.82 g, 7.5 mmol) were dissolved in THF (60 mL). The mixture was cooled to 0 °C under nitrogen atmosphere, treated with $NaBH_4$ (0.93 g, 25 mmol) in water (30 mL), stirred at room temperature for 20 min, and then treated with a saturated aqueous solution of NH_4Cl (100 mL) and extracted with ethyl acetate (3 × 20 mL). The organic phase was washed with brine (3 × 50 mL) and dried with $MgSO_4$. The residue was purified by column chromatography on silica gel, eluting with hexane to give the telluride **9**.

(*Z*)-2-Chloro-1,2-diphenyl-1-ethenyl ethyl telluride (9b): Yield 1.44 g (78%); ^{1}H NMR (200 MHz) δ 7.05−7.19 (m, 10 H), 2.14 (q, J = 7.4 Hz, 2 H), 1.31 (t, J = 7.4 Hz, 3 H); ^{13}C NMR δ 139.80, 138.16, 131.80, 129.27, 128.05, 127.54, 127.01, 122.17, 16.54, 3.57; LRMS *m/z* (relative intensity) 372 (M$^+$, 4), 178 (100); IR (KBr, film) (cm^{-1}) 3060, 2955, 1596, 1492, 1440, 1198, 1030, 942, 911, 787, 761, 716, 694, 609, 582. Anal. Calcd for $C_{16}H_{15}ClTe$: C, 51.89; H, 4.08. Found: C, 51.92; H, 4.09.

(*Z*)-2-Chloro-1-methyl-2-phenyl-1-ethenyl ethyl telluride (9c): Yield 1.16 g (75%); ^{1}H NMR (200 MHz) δ 7.31−7.33 (m, 5 H), 2.77 (q, J = 7.4 Hz, 2 H), 2.11 (s, 3 H), 1.68 (t, J = 7.4 Hz, 3 H); ^{13}C NMR δ 138.04, 129.07, 128.14, 115.00, 23.26, 17.43, −0.29; LRMS *m/z* (relative intensity) 310 (M$^+$, 2), 115 (100); IR (KBr, film) (cm^{-1}) 3078, 3056, 2977, 2917, 1601, 1489, 1443, 1223, 885, 766, 698. Anal. Calcd for $C_{11}H_{13}ClTe$: C, 42.86; H, 4.25. Found: C, 43.28; H, 4.28.

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[15] The yellow color should persist until the end of the reaction. If it turns to red, a small amount of $NaBH_4$ must be added.

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