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Addition of tellurium tetrabromides and alkyl and aryl tellurium tribromides to terminal acetylenes

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Abstract

The addition of tellurium tetrabromides and alkyl and aryl tellurium tribromides to terminal acetylenes gives rise to the corresponding bis(β-bromovinyl)tellurium dibromides and (β-bromovinyl)organyl tellurium dibromides which, by treatment with $NaBH_4$, leads to formation of the reduced tellurides.

Keywords: Acetylene; Aryl tellurium tribromide; Tellurium

The addition of $TeCl_4$ to phenyl- and diphenyl acetylene was described several years ago [1]. Under neat conditions the corresponding β-chlorovinyl-tellurium trichlorides [1] are formed. Treatment with HOAc converts **1** (R = H) into a mixture of $TeCl_4$ and bis(β-chlorovinyl)tellurium dichloride **2** (Eq. (1)).

$$PhC{\equiv}CR + TeCl_4 \rightarrow PhCCl{=}CRTeCl_3 \quad \textbf{(1)}$$

$$PhCCl{=}CRTeCl_3 \overset{HOAc}{\rightarrow} TeCl_4 + (PhCCl{=}CH)_2TeCl_2 \quad \textbf{(2)}$$

Some other examples of the addition of $TeCl_4$ to terminal and inner alkynes were described later [2] and the Z configuration was proposed for the adducts. A related reaction involves the addition of TeO_2 in HOAc to phenylacetylenes in the presence of LiCl, this leads to the formation of substituted benzo[β]tellurophene via the cyclization of the primary adduct [3].

In this paper, the addition of $TeBr_4$ to alkynes will be described. This reaction occurs in a 1:2 ratio giving bis(β-bromovinyl)tellurium dibromides **3**. By reduction of the dibromides with $NaBH_4$, the corresponding divinyl tellurides **4** are obtained (Table 1, Eq. (2)). Divinylic tellurides **4** are compounds of great synthetic potential, since they combine the reactivity of the double bond to form carbon–carbon bonds with the func-

Table 1
Stereoselective synthesis of (Z)-compounds **3** and **4**

R		Yield (%) **3** (Z:E)[b]		Yield (%)[a] **4**
C_6H_5	**3a**	70 (4.5:1)	**4a**	89
C_5H_{11}	**3b**	70 (3.4:1)	**4b**	70

[a] Isolated yields.
[b] Determined by ^{1}H-NMR.

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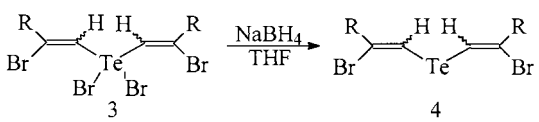
Table 2
Stereoselective synthesis of compounds **7** and **8**

R	R_1	Reaction time (h)		Yield (%) **7**	(Z:E) **7**		Yield (%) **8**
C_6H_5	C_6H_5	6.5	**7a**	75	5.0:1	**8a**	79
C_6H_5	C_4H_9	16	**7b**	74	4.9:1	**8b**	82
C_5H_{11}	C_6H_5	15	**7c**	71	5.7:1	**8c**	84
C_5H_{11}	C_4H_9	12	**7d**	71	4.0:1	**8d**	89
$HOCH_2$	C_6H_5	6	**7e**	65	3.5:1	**8e**	74
$HOCH_2$	C_4H_9	12	**7f**	60	1:1	**8f**	50

tional transformations achieved by the well known reactions of the organotellurium compounds [4].

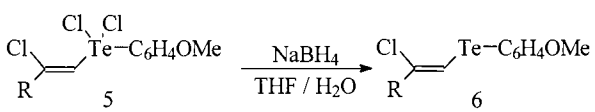
$$2RC{\equiv}CH + TeBr_4 \xrightarrow[\text{reflux}]{\text{benzene}}$$



3 4 (2)

Recently, we described the addition of an aryltellurium trichloride to terminal acetylenes in benzene at reflux temperature. This reaction gives rise to **5**, (β-chlorovinyl)aryl tellurium dichlorides [5,6] which can be converted to the corresponding tellurides by reduction with $NaBH_4$ (Eq. (3)).

$$RC{\equiv}CH + MeOC_6H_4TeCl_3 \xrightarrow[\text{reflux}]{\text{benzene}}$$



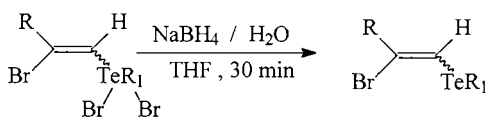
5 6 (3)

R = Ph, p-MeC_6H_4, p-$MeOC_6H_4$, p-$C_2H_5OC_6H_4$, p-ClC_6H_4, p-BrC_6H_4, CH_2OH, HOC_6H_{10}, C_6H_{11}.

A recent paper of Huang [7] describes the addition of aryltellurium tribromide to alkynes. This gives (β-bromovinyl)aryl tellurium dibromides which exhibit the Z or E configuration depending on the solvent—benzene or methanol—used in the reaction.

In view of our continued interest in this kind of reaction, we report the results obtained in our laboratory about this subject. Following preliminary results concerning the addition of $PhTeBr_3$ to terminal acetylenes [8], we have successfully extended the addition reaction to alkyl tellurium tribromides. The expected (β-bromovinyl)organyl tellurium dibromides **7** were obtained in good yields. The reduction of **7** with $NaBH_4$ gives the corresponding tellurides **8** (Table 2, Eq. (4)).

$$RC{\equiv}CH + R_1TeBr_3 \xrightarrow[\text{reflux}]{\text{benzene}}$$



7 8 (4)

1. X-ray structure analysis

Details of cell data, X-ray data collection and refinement are given in Table 3.

A complete crystallographic description of this and of the related Cl-compound will be published elsewhere [9]. The ORTEP drawing of Z-bis(β-bromovinyl)tellurium dibromide with the atom-numbering is shown in Fig. 1.

In accord with the literature, our NMR data support the view that Z isomers are the main products in both reactions. X-ray analysis of adduct **3a** agrees with the Z,Z geometry of the double bonds.

2. Experimental section

2.1. General

^{1}H-NMR spectra were recorded on a Bruker AC-200 spectrometer using tetramethylsilane as the standard ^{13}C-NMR spectra were obtained on a Bruker AC-200 using the central peak of $CDCl_3$ (77.0 ppm) as the standard. IR spectra were recorded on a Perkin Elmer 1600 spectrophotometer. Elemental analyses were performed at the Microanalytical Laboratory of the Institute of Chemistry, USP. Column chromatography was carried out using Merck silica-gel (230–400 mesh) according to the procedure of Still and coworkers [10]. All solvents were dried and distilled according to the established methods [11].

2.2. Addition of $TeBr_4$ to alkynes: bis(β-bromovinyl)tellurium dibromides **3**

2.2.1. Typical experiment

To a solution of tellurium tetrabromide (2 mmol) in benzene (20 ml) was added phenylacetylene (0.41 g, 4 mmol). The mixture was boiled under reflux for 15 h and the reaction was monitored by TLC. After this time the mixture was treated with methanol/water (1:1; 3×20 ml) and the organic phase was extracted with dichloromethane (2×20 ml). The organic phase was dried over magnesium sulfate and the solvents evaporated in a rotary evaporator. The resulting oil was

purified by column chromatography eluting first with hexane and then with a mixture of hexane/ethyl acetate (1:1).**3a**-Anal.Calcd. for $C_{16}H_{12}TeBr_4$: C, 29.50; H, 1.96; Found: C, 29.85; H, 1.93%. ^{1}H-NMR ($CDCl_3$) δ 7.38–7.65 (m, 10H); 8.24 (s, 1H); 8.44 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 121.57; 124.08; 127.59; 128.02; 128.76; 129.43; 131.24; 136.05; 140.16. [IR (neat) $\nu_{max/cm-1}$ 686; 710; 731; 876; 1210; 1442; 1486; 1549; 3026]cm^{-1}.**3b**-Anal.Calcd. for $C_{14}H_{24}TeBr_4$: C, 26.29; H, 3.52; Found: C, 26.49; H, 3.78%. ^{1}H-NMR ($CDCl_3$) δ 0.90 (t, 6H, $J = 6.6$ Hz); 1.32–1.48 (m, 4H); 1.62–180 (m, 8H); 2.67 (t, 6H, $J = 6.7$ Hz); 3.09 (t, 2H, $J = 6.7$ Hz); 7.67 (t, ^{1}H, $J = 1.0$ Hz); 7.85 (t, 3H, $J = 1.0$ Hz). ^{13}C-NMR ($CDCl_3$) δ 13.81; 22.13; 30.18; 30.78; 33.76; 42.35; 120.58; 124.87; 139.09; 145.21. [IR (neat) $\nu_{max/cm-1}$ 732; 911; 1111; 1164; 1206; 1378; 1463; 1595; 2928; 3013]cm^{-1}.

Table 3
Crystal data and structure refinement for bis(2-bromo-2-phenylethenyl)tellurodibromide

Compound	**3a**
Color/shape	Yellow/irregular
Formula	$Br_4C_{16}H_{12}Te$
Formula weight	651.50
Temperature (K)	293
Crystal system	Orthorhombic
Space group	$Pna2_1$
a (Å)	19.159(2)
b (Å)	5.781(1)
c (Å)	16.787(1)
α(°)	90
β(°)	90
γ(°)	90
V(Å^3)	1859.3(4)
D_c (g cm^{-3})	2.327
Z	4
μ (Mo–K$_\alpha$) (mm^{-1})	10.187
Diffractometer/scan	CAD4-Mach3/$\omega/2\theta$
Radiation/wavelength (Å)	Mo–K$_\alpha$/0.71073
F(000)	1200
Crystal size (mm)	0.35 × 0.20 (max./min.)
θ range data collection (°)	2.42–26
Index ranges (h, k, l)	−23/0, 0/7, 0/20
Reflections collected	1822
Independent/observed reflections [$I > 2\sigma(I)$]	1822/932
Absorption correction	psi-scans
Range of relative transmission factors	0.99 and 0.57
Refinement method	Full-matrix least-squares on F^2
Data/restraint/parameters	1822/1/190
Goodness-of-fit on F^2	1.104
SHELX-93 weight parameters	0.0440/3.8360
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0404$, $wR_2 = 0.0825$
R indices (all data)	$R_1 = 0.0825$, $wR_2 = 0.1150$
Largest difference peak and hole (e Å^{-3})$^{n\,a}$	1.025 and −0.824
Absolute structure parameter	0.05(8)

[a] The largest peaks have no chemical significance and are located between Te and Br_2.

2.3. Bis(β-bromovinyl)tellurides **4**

2.3.1. Typical experiment

A solution of bis(2-bromo-1-vinyl)dibromotelluride (2 mmol) in tetrahydrofuran (15 ml) was treated dropwise with a solution of sodium borohydride (0.075 g, 2 mmol) in water (10 ml). After 30 min of stirring at room temperature (r.t.) the mixture was diluted with ether (30 ml) and washed with water, a saturated solution of ammonium chloride, and brine. The organic phase was dried over magnesium sulfate and the solvent evaporated in a rotary evaporator. The residual oil was filtered through a column of silica gel, eluting with hexane.**4a**-Anal. Calcd. $C_{16}H_{12}TeBr_2$: C, 39.09; H, 2.46; Found: C, 38.74; H, 2.44%. ^{1}H-NMR ($CDCl_3$) δ 7.28–7.52 (m, 10H); 7.71 (s, 1H); 7.90 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 105.18; 110.92; 126.94; 127.93; 128.40; 128.59; 129.40; 131.40; 139.31. [IR (neat) $\nu_{max/cm-1}$ 698; 745; 788; 869; 1154; 1212; 1441; 1485; 1586; 3022; 3050]cm^{-1}.**4b**-^{1}H-NMR ($CDCl_3$) δ 0.90 (t, 6H, $J = 6.6$ Hz); 1.25–1.36 (m, 4H); 1.48–1.58 (m, 8H); 2.45–2.60 (m, 6H); 6.95 (t, 1H, $J = 1.0$ Hz); 7.07 (t, 3H, $J = 1.0$ Hz). ^{13}C-NMR ($CDCl_3$) δ 13.95; 22.33; 28.14; 30.61; 30.97; 39.05; 43.64; 103.57; 113.09; 129.10; 135.99. [IR (neat) $\nu_{max/cm-1}$ 761; 1046; 1114; 1202; 1418; 1449; 1574; 2856; 2931]cm^{-1}.

2.4. Addition of organoyltellurium tribromide to alkynes: (β-bromovinyl)organyl tellurium dibromides **7**

2.4.1. Typical procedure

Phenyltellurium tribromide (2 mmol) was added to phenylacetylene (0.204 g,2 mmol) in benzene (20 ml). The mixture was refluxed for 6.5 h, and the reaction was monitored by TLC (eluting with dichloromethane). After this time the mixture was washed with methanol/water (1:1; 3 × 20 ml) and the organic phase was extracted with dichloromethane (2 × 20 ml). The organic phase was dried over magnesium sulfate, and the solvent was evaporated in a rotary evaporator. The residual oil was filtered through a column of silica gel, eluting first with hexane and then with a mixture of hexane/ethyl acetate.**7a**-^{1}H-NMR ($CDCl_3$) δ 7.40–7.64 (m, 8H); 7.85 (s, 1H); 8.18 (s, 1H); 8.31 (d, 2H, $J = 6.5$ Hz); ^{13}C-NMR ($CDCl_3$) δ 124.18; 126.28; 127.93; 128.03; 128.69; 129.27; 130.20; 131.17; 131.39; 131.75; 134.22; 134.77; 135.28; 136.16; 139.05; 140.35. [IR (neat) $\nu_{max/cm-1}$ 680; 723; 771; 873; 994; 1192; 1437; 1474; 1549; 3046]cm^{-1}.**7b**-^{1}H-NMR ($CDCl_3$) δ 1.04 (t, 3H, $J = 6.8$ Hz); 1.50–1.63 (m, 4H); 2.12–2.33 (m, 2H); 3.78 (t, 2H, $J = 7.6$ Hz); 3.88 (t, 2H, $J = 7.6$ Hz); 7.39–764 (m, 5H); 7.75 (s, 1H); 8.07 (s, 1H). ^{13}C-NMR ($CDCl_3$) δ 13.46; 24,35; 27.40; 47.20; 48.53; 119.26;

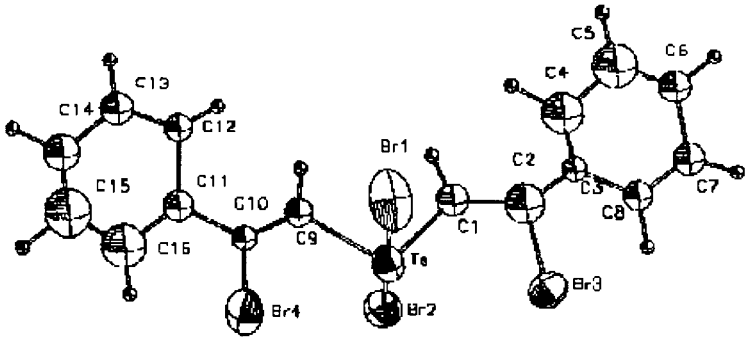


Fig. 1. ORTEP drawing of *Z*-bis(β-bromovinyl phenyl)telluride dibromide **3a.**

121.13; 127,91; 128.07; 129.15; 131.17; 136.36; 141.14. [IR (neat) $\nu_{max/cm^{-1}}$ 683; 746; 873; 1170; 1211; 1250; 1442; 1487; 1556; 2952; 3004]cm^{-1}.**7c**-^{1}H-NMR ($CDCl_3$) δ 0.92 (t, 3H, $J = 7.2$ Hz); 1,30–147 (m, 2H); 1.52–1.78(m, 4H); 2.69 (t, 2H, $J = 7.5$ Hz); 3.08 (t, 2H, $J = 7.5$ Hz); 6.99 (t, 1H, $J = 0.8$ Hz); 7.51–7.60 (m, 3H); 7.77 (t, 1H, $J = 1.2$ Hz); 8.25 (d, 2H, $J = 6.5$ Hz). ^{13}C-NMR ($CDCl_3$) δ 13.85; 22.21; 27.74; 30.23; 42.30; 121.39; 126,03; 130.23; 131.67; 132.07; 135.58; 141.51; 145.67. [IR (neat) $\nu_{max/cm^{-1}}$ 682; 733; 996; 1052; 1161; 1328; 1437; 1474; 1594; 2857; 2955]cm^{-1}.**7d**-^{1}H-NMR ($CDCl_3$) δ 0.89–1.10 (m, 6H); 1.29–1.74 (m, 6H); 2.18–2.30 (m, 2H); 2.68 (t, 2H, $J = 7.2$ Hz); 2.99 (t, 2H, $J = 7.6$ Hz); 3.79 (t, 2H, $J = 7.6$ Hz); 7.20 (t, 1H, $J = 0.8$ Hz); 7.63 (t, 1H, $J = 1.2$ Hz). ^{13}C-NMR ($CDCl_3$) δ 27.23; 27.76; 30.23; 30.97; 34.12; 42.40; 46.73; 51.09; 119.60; 120.79; 137.19; 146.34. [IR (neat) $\nu_{max/cm^{-1}}$ 756; 1048; 1170; 1454; 1594; 2924]cm^{-1}.**7e**-^{1}H-NMR ($CDCl_3$) δ 1.86 (s, 1H); 3.05 (s, 1H); 4.47 (s, 2H); 4.84 (s, 2H); 6.83 (t, 1H, $J = 1.2$ Hz); 7.46–7.61 (m, 3H); 7.97 (t, 1H, $J = 2.4$ Hz); 8.24 (d, 2H, $J = 7.2$ Hz). ^{13}C-NMR ($CDCl_3$) δ 67.27; 120.39; 124.72; 129.59; 130.27; 131.87; 134.81; 136.33; 142.35. [IR (neat) $\nu_{max/cm^{-1}}$ 682; 732; 995; 1043; 1247; 1373; 1437; 1474; 1605; 1705; 3054; 3408]cm^{-1}.**7f**-^{1}H-NMR ($CDCl_3$) δ 2.15–2.27 (m, 2H); 3.57 (s, 1H); 3.71 (t, 2H, $J = 7.6$ Hz); 3.86 (t, 2H, $J = 7.6$ Hz); 4.48 (t, 2H, $J = 7.0$ Hz); 4.75 (t, 2H, $J = 7.0$ Hz); 7.18 (t, 1H, $J = 1.2$ Hz); 8.15 (t, 1H, $J = 1.6$ Hz). ^{13}C-NMR ($CDCl_3$) δ 7.79; 11.62; 11.90; 37.19; 56.95; 63.90; 127.68; 132.57; 161,60; 163.29. [IR (neat) $\nu_{max/cm^{-1}}$ 693; 770, 1043; 1180; 1250; 1400; 1460; 1604; 2869; 2929; 2959; 3462]cm^{-1}.

2.5. (β-Bromovinyl)organyl tellurides **8**

2.5.1. Typical experiment

A solution of 1-(dibromoorganyltellurium)-2-bromo-2-organoethene (2 mmol) in tetrahydrofuran (15 ml) was treated dropwise with a solution of sodium borohydride (0.075 g, 2 mmol) in water (10 ml). After 30 min of stirring at r.t. the mixture was diluted with ether (30 ml) and washed with water, a saturated solution of ammonium chloride, and brine. The organic phase was dried over magnesium sulfate and the solvents evaporated in a rotary evaporator. The residual oil was filtered through a column of silica gel, eluting with hexane.**8a**-^{1}H-NMR ($CDCl_3$) δ 7.38 (s, 1H); 7.22–7.50 (m, 8H); 7.77 (s, 1H); 7.86 (d, 2H, $J = 6.5$ Hz). ^{13}C-NMR ($CDCl_3$) δ 113.74; 114.58; 126.91; 128.26; 128.48; 128.51; 129.24; 129.64; 129.93; 137.57; 138.28; 138.87; 139.42. [IR (neat) $\nu_{max/cm^{-1}}$ 691; 732; 867; 997; 1017; 1209; 1433; 1474; 1573; 3051]cm^{-1}.**8b**-^{1}H-NMR ($CDCl_3$) δ 0.93 (t, 3H, $J = 7.6$ Hz); 1.35–1.47 (m, 2H); 1.78–1.92 (m, 2H); 2.73 (t, 2H, $J = 7.6$ Hz); 2.80 (t, 2H, $J = 7.6$ Hz); 7.41 (s, 1H); 7.25–7.52 (m, 5H); 7.73 (s, 1H). ^{13}C-NMR ($CDCl_3$) δ 7.71; 13.30; 24.98; 34.32; 109.84; 126.84; 128.22; 128.94; 130.67; 139.78. [IR (neat) $\nu_{max/cm^{-1}}$ 700; 749; 866; 1161; 1441; 1486; 1549; 2925; 2957]cm^{-1}.**8c**-^{1}H-NMR ($CDCl_3$) δ 0.88 (t, 3H, $J = 7.2$ Hz); 1.22–1.33 (m, 2H); 1.47–1.58 (m, 4H); 2.43 (t, 2H, $J = 7.6$ Hz); 2.51 (t, 2H, $J = 7.6$ Hz); 6.55 (t, 1H, $J = 0.8$ Hz); 7.02 (t, 1H, $J = 1.2$ Hz); 7.19–7.30 (m, 3H); 7.77 (d, 2H, $J = 6.5$ Hz). ^{13}C-NMR ($CDCl_3$) δ 13.89; 22.03; 28.07; 30.44; 43.39; 108.19; 113.27; 128.00; 129.37; 134.96; 138.46. [IR (neat) $\nu_{max/cm^{-1}}$ 691; 732; 998; 1018; 1162; 1434; 1473; 1574; 2856; 2928]cm^{-1}.**8d**-^{1}H-NMR ($CDCl_3$) δ 0.88–0.96 (m, 6H); 1.25–1.60 (m, 6H); 1.70–1.86 (m, 2H); 2.52 (t, 2H, $J = 7.6$ Hz); 2.71 (t, 2H, $J = 7.6$ Hz); 3.11 (t, 2H, $J = 7.6$ Hz); 6.52 (t, 1H, $J = 0.8$ Hz); 6.95 (t, 1H, $J = 1.2$ Hz). ^{13}C-NMR ($CDCl_3$) δ 6.59; 13.38; 13.95; 22.32; 24.98; 28.03; 30.28; 34.33; 43,64; 108.90; 119.49; 135.89. [IR (neat) $\nu_{max/cm^{-1}}$ 761; 1160; 1377; 1418; 1452; 1573; 2956]cm^{-1}.**8e**-^{1}H-NMR ($CDCl_3$) δ 2.26 (s, 2H); 2.92 (s, 1H); 4.22 (s, 2H); 6.12 (t, 1H, $J = 1.6$ Hz); 7.21–7.33 (m, 3H); 7.49 (t, 1H, $J = 1.2$ Hz); 7.78 (d, 2H, $J = 6.5$ Hz). ^{13}C-NMR ($CDCl_3$) δ 65.72; 68.88; 103.56; 111.40; 112.27; 113.35; 125.41; 128.36; 129.49; 131.54; 138.66; 139.80. [IR (neat) $\nu_{max/cm^{-1}}$ 691; 734; 1017; 1067; 1157; 1434; 1473; 1573; 2919; 3051; 3335]cm^{-1}.**8f**-^{1}H-NMR ($CDCl_3$) δ 0.94 (t, 2H, $J = 7.6$ Hz); 1.36–1.45 (m, 2H); 1.80–1.87 (m, 2H); 2.50 (s, 1H); 2.76 (t, 2H, $J = 7.6$ Hz); 4.28 (s, 2H); 4.73 (s, 1H); 6.50 (t, 1H, $J = 1.2$ Hz); 7.48 (t, 1H, $J = 1.6$ Hz). ^{13}C-NMR ($CDCl_3$) δ 7.05; 13.34; 24.93;

34.28; 66.06; 69.18; 106.75; 107.36; 121.66; 132.42. [IR (neat) $\nu_{max/cm^{-1}}$ 836; 1072; 1160; 1248; 1376; 1450; 1729; 2923; 3409]cm^{-1}.

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