

Synthesis and polycondensation of some new organotellurium compounds containing hydroxymethyl groups

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A new series of organotellurium compounds [*i.e.* 2-HOCH₂C₆H₄TeBr₃ (1), (2-HOCH₂C₆H₄)₂TeBr₂ (2), (2-HOCH₂C₆H₄)₂Te (3), (2-HOCH₂C₆H₄Te—)₂ (4), 4-HOCH₂C₆H₄TeBr₃ (5), (4-HOCH₂C₆H₄)₂TeBr₂ (6), (4-HOCH₂C₆H₄)₂Te (7), and (4-HOCH₂C₆H₄Te—)₂ (8)] were prepared by reacting hydroxymethylphenylmercury chlorides with tellurium tetrabromide in dry dioxane. Bis(2-hydroxymethylphenyl) telluride (3), bis(2-hydroxymethylphenyl) ditelluride (4) and bis(4-hydroxymethylphenyl) telluride (7) were polymerized by a solution polycondensation technique with toluene diisocyanate and terephthaloyl chloride, leading to new organic tellurium polyurethanes and polyesters. All the new compounds were characterized by elemental analysis and spectroscopic data. Copyright © 2002 John Wiley & Sons, Ltd.

KEYWORDS: organotellurium compounds; polycondensation; polyester; polyurethane; telluration; hydroxymethyl groups

INTRODUCTION

Organotellurium compounds containing a hydroxy group within the same organic radical are well documented in the literature.^{1–3} Phenols reacted with tellurium tetrachloride under relatively mild conditions in chloroform or carbon tetrachloride solution gave insoluble aryltellurium trichlorides, even when a twofold quantity of phenols was present.^{4–7}

There are few examples in the literature involving aromatic tellurides bearing hydroxymethyl groups. Such compounds were prepared by indirect methods, either by reduction of an aldehyde group by sodium borohydride in alkaline solution⁸ or by reacting Grignard reagent with 2-carbaldehydophenyl methyltelluride.⁹ On the other hand, treatment of tellurophene-2-carbaldehyde or methyltellurophene-2-carbaldehyde with lithium aluminum hydride gave tellurophene-2-yl methanol in good yield.⁹

The synthesis of organic polymers containing tellurium is very rare in the literature. Our recent work¹⁰ described the synthesis of amine-substituted organic tellurium com-

pounds from which new organic tellurium polyamides and polyurea were prepared. To the best of our knowledge, there is no direct method to prepare organotellurium compounds containing hydroxymethyl groups *ortho* or *para* to the tellurium atom in the organic radical. Thus, the objective of the present work was to synthesize hydroxymethylphenyltellurium compounds and to use them to prepare polymers via solution polycondensation techniques.

EXPERIMENTAL

Synthesis

4-Hydroxy- and 2-hydroxy-methylphenylmercury chlorides were prepared from benzyl alcohol according to a literature method.¹¹

2-Hydroxymethylphenyltellurium tribromide (1)

A mixture of 2-hydroxymethylphenylmercury chloride (2.06 g; 6 mmol) and tellurium tetrabromide (2.69 g; 6 mmol) in 50 cm³ of dry dioxane was refluxed for 5 h under nitrogen atmosphere. The solution was filtered hot, and on cooling to room temperature deposited a 2:1 complex of 2diox·HgClBr as white plates, which was filtered off. The filtrate was evaporated to dryness on a rotary evaporator. An orange-yellow precipitate was obtained. Recrystallization from

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ethanol:hexane (9:1) gave yellow crystals in 68% yield (1.94 g), m.p. 208–210 °C.

Found: C, 17.90; H, 1.10. Calc. for $C_7H_7Br_3OTe$: C, 17.72; H, 1.49%.

IR (cm^{-1}): 3280s, 3040w, 2960m, 2870m, 1490s, 1420s, 1215m, 1015s, 730s, 700m.

^1H NMR (CDCl_3): 1.68 (s, br, 2H, OH); 5.32 (s, 2H, CH_2); 7.25–7.27 (m, 1H, Ar-H); 7.38–7.44 (m, 2H, Ar-H); 7.90 (m, 1H, Ar-H). ^{13}C NMR (CDCl_3): 71.8, 124.5, 127.8, 128.2, 130.6, 130.8, 148.1.

Bis(2-hydroxymethylphenyl)tellurium dibromide (2)

A mixture of 2-hydroxymethylphenylmercury chloride (2.75 g; 8 mmol) and tellurium tetrabromide (1.79 g; 4 mmol) in dry dioxane (60 cm^3) was refluxed for 4 h under nitrogen. The solution was filtered hot, and upon cooling to room temperature gave a complex of 2diox-HgClBr as white plates. The filtrate was added in small portions to ice-water, during which a brown-yellow precipitate was formed. The residue was recrystallized from methanol to afford a yellow precipitate in 60% yield (1.20 g), m.p. 287–288 °C.

Found: C, 33.75; H, 2.67. Calc. for $C_{14}H_{14}Br_2O_2Te$: C, 33.52; H, 2.81%.

IR (cm^{-1}): 3300s, 2960m, 2880m, 1470s, 1210m, 1010s, 750s, 710m.

^1H NMR (CDCl_3): 1.65 (s, br, 2H, OH); 4.67 (s, 4H, CH_2); 7.26–7.29 (m, 2H, Ar-H); 7.35–7.39 (m, 4H, Ar-H); 7.82–7.88 (m, 2H, Ar-H). ^{13}C NMR (CDCl_3): 72.5, 125.0, 127.8, 128.3, 130.5, 131.2, 148.1.

Bis(2-hydroxymethylphenyl) telluride (3)

A suspension of bis(2-hydroxymethylphenyl)tellurium dibromide (2.51 g; 5 mmol) in a mixture of ethanol (70 cm^3) and water (30 cm^3) was heated under reflux. To this solution, a solution of hydrazine hydrate (1.80 g in 50 cm^3 of ethanol) was added slowly with stirring until evolution of nitrogen ceased. The solution was then filtered. The filtrate was evaporated under reduced pressure to dryness to afford a red-brown precipitate. The residue was dissolved in hot chloroform; hexane was then added dropwise until the hot solution became turbid and the solution left overnight in a refrigerator. White crystals were obtained in 77% yield (1.32 g), m.p. 161–162 °C.

Found: C, 48.83; H, 3.97. Calc. for $C_{14}H_{14}O_2Te$: C, 49.19; H, 4.13%.

IR (cm^{-1}): 3350s, 3220s, 3030w, 2940m, 2900m, 1480s, 1430s, 1205m, 1020s, 740s, 690m.

^1H NMR (CDCl_3): 1.61 (s, 2H, OH); 5.41 (d, $J = 1.1 \text{ Hz}$, 4H, CH_2); 7.24–7.27 (m, 2H, Ar-H); 7.34–7.43 (m, 4H, Ar-H); 7.86–7.89 (m, 2H, Ar-H). ^{13}C NMR (CDCl_3): 72.1, 124.1, 128.0, 128.2, 130.7, 131.1, 148.2.

Bis(2-hydroxymethylphenyl) ditelluride (4)

To a suspension of 2-hydroxymethylphenyltellurium tribromide (0.95 g; 2 mmol) in water (50 cm^3) was added sodium

sulfide nonahydrate (2.95 g; 16 mmol). The reaction mixture was heated to 100 °C in a water bath with stirring for 30 min, then cooled to room temperature and extracted with ethyl acetate ($4 \times 30 \text{ cm}^3$). The combined extracts were dried by anhydrous magnesium sulfate (MgSO_4). Evaporation of the solvent in a rotary evaporator at room temperature gave a deep-red oil. Attempts to solidify the product were unsuccessful and led to the formation of a semi-solid deep-red compound in 65% yield (0.16 g¹).

Found: C, 35.67; H, 2.93. Calc. for $C_{14}H_{14}O_2Te_2$: C, 35.82; H, 3.01%.

IR (cm^{-1}): 3360sb, 3038w, 2960m, 2880m, 1490s, 1450s, 1215m, 1025s, 750s, 710m.

^1H NMR (CDCl_3): 1.75 (sb, 2H, OH); 4.83 (s, 4H, CH_2); 7.30–7.33 (m, 2H, Ar-H); 7.37–7.41 (m, 4H, Ar-H); 7.85–7.90 (m, 2H, Ar-H). ^{13}C NMR (CDCl_3): 71.8, 124.3, 127.8, 128.1, 130.1, 131.2, 147.7.

4-Hydroxymethylphenyltellurium tribromide (5)

4-Hydroxymethylphenylmercuric chloride (2.75 g; 8 mmol) and tellurium tetrabromide (2.69 g; 8 mmol) in dry dioxane were treated by a procedure similar to that used for the preparation of **1**, providing **5** (75% yield, 2.14 g) as yellow crystals, m.p. 189–190 °C.

Found: C, 17.60; H, 1.32. Calc. for $C_7H_7Br_3OTe$: C, 17.72; H, 1.49%.

IR (cm^{-1}): 3340sb, 3060w, 2960m, 2860m, 1460s, 1440s, 1210m, 1015s, 750s, 700m.

^1H NMR (CDCl_3): 2.25 (s, br, 1H, OH); 4.35 (s, 2H, CH_2); 7.38 (d, 2H, Ar-H); 7.72 (d, 2H, Ar-H). ^{13}C NMR (CDCl_3): 63.5, 107.6, 127.9, 135.2, 141.6.

Bis(4-hydroxymethylphenyl)tellurium dibromide (6)

4-Hydroxymethylphenylmercuric chloride (2.75 g; 8 mmol) and tellurium tetrabromide (1.79 g; 4 mmol) were refluxed in dry dioxane (60 cm^3) for 4 h. The reaction mixture was treated as described for the preparation of **2**, and give yellow crystals in 65% yield (1.31 g), m.p. 265–266 °C (dec.).

Found: C, 33.41; H, 2.70. Calc. for $C_{14}H_{14}Br_3OTe$: C, 33.52; H, 2.81%.

IR (cm^{-1}): 3350sb, 3040w, 2920m, 2870m, 1480s, 1455s, 1205m, 1010s, 740s, 710m.

^1H NMR (CDCl_3): 2.15 (s, 2H, OH); 4.48 (s, 4H, CH_2); 7.26 (d, 4H, Ar-H); 7.78 (d, 4H, Ar-H). ^{13}C NMR (CDCl_3): 67.2, 108.6, 127.6, 135.2, 139.8.

Bis(4-hydroxymethylphenyl) telluride (7)

Compound **6** (0.95 g; 2 mmol) was converted to compound **7** by reducing it with ethanolic hydrazine hydrate (1.80 g in 50 cm^3 ethanol) and as described for the preparation of **3**. Yellow crystals were obtained in 80% yield (1.37 g), m.p. 170–171 °C.

Found: C, 48.96; H, 4.01. Calc. for $C_{14}H_{14}O_2Te$: C, 49.19; H, 4.13%.

IR (cm^{-1}): 3380sb, 3060m, 2940m, 2860m, 1590s, 1450s, 1200s, 1020s, 755s, 720m.

^1H NMR (CDCl_3): 2.25 (s, br, 2H, OH); 4.54 (s, 4H, CH_2); 7.45 (d, 4H, CH_2); 7.80 (d, 4H, Ar-H). ^{13}C NMR (CDCl_3): 67.2, 107.8, 128.1, 136.3, 141.7

Bis(4-hydroxymethylphenyl) ditelluride (8)

4-Hydroxymethylphenyltellurium tribromide (2.15 g; 16 mmol) was treated with sodium sulfide nonahydrate (2.95 g; 16 mmol) as described for the preparation of **4**. Orange crystals were obtained in 68% (0.64 g), m.p. 159–160°C.

Found: C, 35.73; H, 2.87. Calc. for $\text{C}_{14}\text{H}_{14}\text{O}_2\text{Te}_2$: C, 35.82; H, 3.01%.

IR (cm^{-1}): 3400s, 3080w, 2940m, 2860m, 1490s, 1440s, 1210m, 1025s, 760s, 710m.

^1H NMR (CDCl_3): 2.32 (s, 2H, OH); 4.38 (s, 4H, CH_2); 7.51 (d, 4H, Ar-H); 7.86 (d, 4H, Ar-H). ^{13}C NMR (CDCl_3): 64.3, 107.9, 127.8, 136.8, 142.3.

Polycondensation of bis(2-hydroxymethylphenyl)telluride and toluene diisocyanate (TDI) (9)

A solution of bis(2-hydroxymethylphenyl) telluride (1.03 g; 3 mmol) in 50 cm^3 of dry chloroform was added in one portion to a well stirred solution of TDI (80% 2,4- and 20% 2,6-isomer) in dry chloroform (80 cm^3) at room temperature. The reaction mixture was refluxed for 6 h. A yellow precipitate was obtained, collected, washed several times with hot chloroform and dried *in vacuo*. The resulting polyurethane was crushed to a fine powder, refluxed in toluene, and filtered. The yellow precipitate was washed several times with boiled diethyl ether, to remove any impurities and dried *in vacuo* at 100°C for 72 h to give a yellow solid in 81% yield (1.26 g), m.p. 292–296°C.

Found: C, 53.19; H, 3.52; N, 5.18. Calc. for $(\text{C}_{23}\text{H}_{19}\text{N}_2\text{O}_4\text{Te})_n$: C, 53.53; H, 3.90; N, 5.43%.

IR (cm^{-1}): 3340sb, 3040w, 2940w, 2860m, 2280sb, 1720s, 1670s, 1540s, 1510s, 1480s, 1420s, 1215s.

Polycondensation of bis(4-hydroxymethylphenyl) telluride and TDI (10)

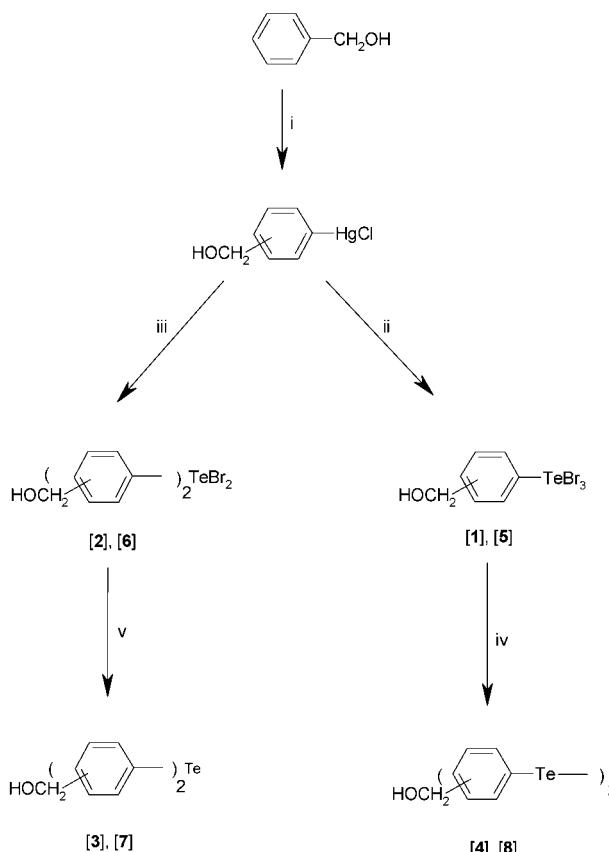
Bis(4-hydroxymethylphenyl) telluride (1.71 g; 5 mmol) was treated with TDI (0.87 g; 5 mmol) in a similar way as described for the preparation of **9**. A yellow solid was obtained in 90% yield (2.32 g), m.p. 276–280°C.

Found: C, 53.07; H, 3.85; N, 5.4. Calc. for $(\text{C}_{23}\text{H}_{19}\text{N}_2\text{O}_4\text{Te})_n$: C, 53.53; H, 3.90; N, 5.43%.

IR (cm^{-1}): 3300sb, 3080w, 2940m, 2860m, 2280s, 1710s, 1660s, 1600s, 1540s, 1410s, 1440m, 1280s, 1210s.

Polycondensation of bis(4-hydroxymethylphenyl) ditelluride and TDI (11)

Bis(4-hydroxymethylphenyl) ditelluride (1.17 g; 2.5 mmol) was treated with TDI by the same procedure as described for



Scheme 1. Reagents: i) $\text{Hg}(\text{OAc})_2/\text{LiCl}$. ii) $\text{TeBr}_4/\text{dioxane}$. iii) $1/2\text{TeBr}_4/\text{dioxane}$. iv) $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$. v) N_2H_4 .

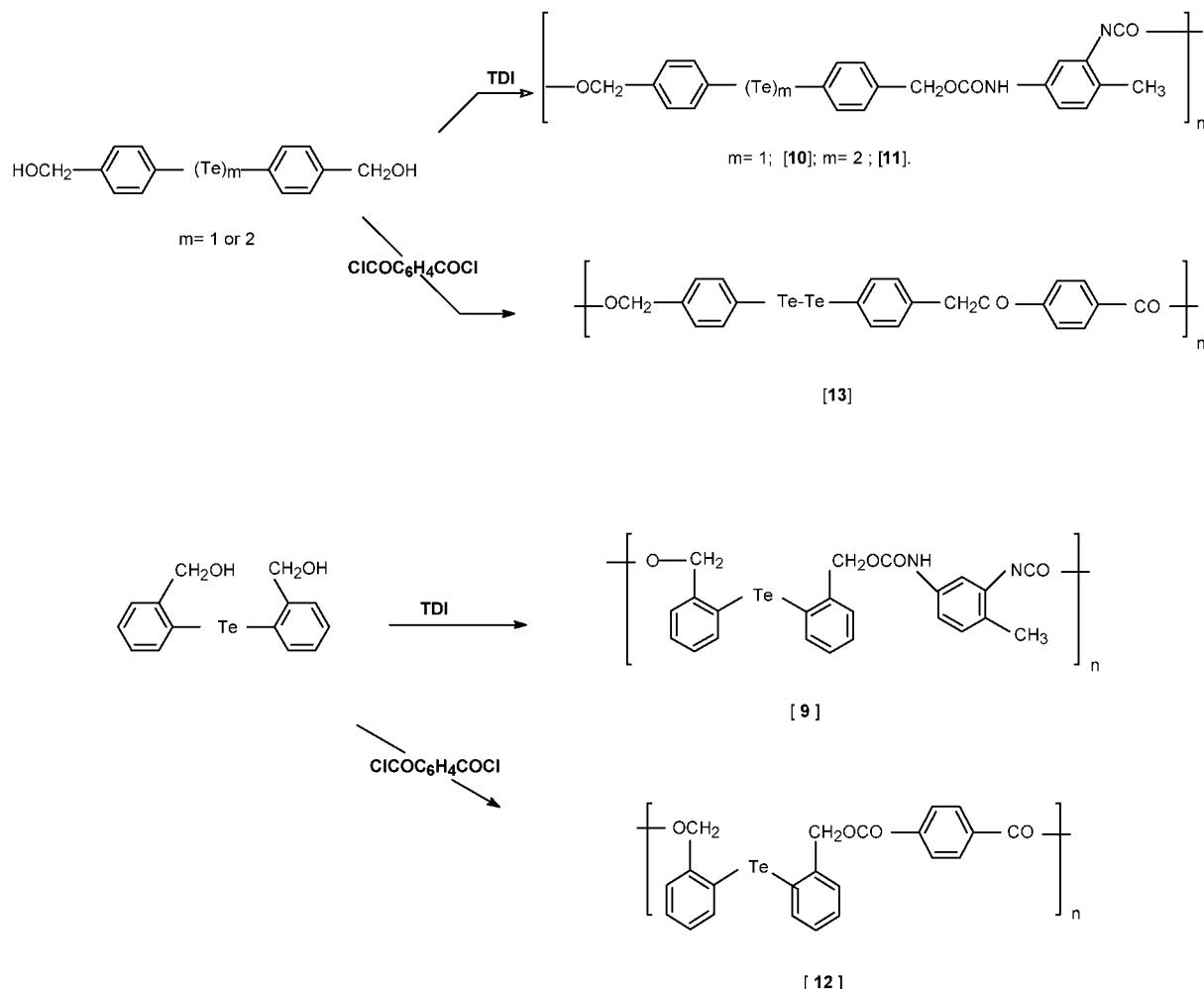
9. An orange solid was obtained in 85% yield (1.36 g), m.p. 300–305°C.

Found: C, 42.18; H, 3.61; N, 4.86. Calc. for $(\text{C}_{23}\text{H}_{19}\text{N}_2\text{O}_4\text{Te})_n$: C, 42.92; H, 3.13; N, 4.35%.

IR (cm^{-1}): 3320sb, 3100w, 2940w, 2880m, 2290s, 1715s, 1655s, 1605, 1550s, 1515s, 1480s, 1430m, 1300s, 1230s.

Polycondensation of bis(2-hydroxymethylphenyl) telluride and terephthaloyl chloride (12)

A solution of bis(2-hydroxymethylphenyl) telluride (0.85 g; 2.5 mmol) and triethylamine (0.64 g; 6.3 mmol) in dry dichloromethane (20 cm^3) was added with stirring to a solution of terephthaloyl chloride (0.51 g; 2.5 mmol) in 20 cm^3 of dry dichloromethane. The stirring was continued vigorously for 1 h, during which some precipitate was observed. The contents of the flask were poured into dry hexane (500 cm^3). The resulting pale-yellow precipitate was washed with water and then acetone, and then dried. The product was refluxed in toluene for 1 h. The precipitate was washed several times with diethyl ether and dried *in vacuo* at 50°C for 72 h. A red-brown solid was obtained in 82% yield (1.12 g), m.p. 218–222°C.

**Scheme 2.**

Found: C, 55.83; H, 3.91. Calc. for $(\text{C}_{22}\text{H}_{16}\text{O}_4\text{Te})_n$: C, 55.99; H, 4.42%.

IR (cm^{-1}): 3060w, 2930m, 2860m, 1720s, 1600s, 1440s, 1290sb, 1130s.

Polycondensation of bis(4-hydroxymethylphenyl) ditelluride and terephthaloyl chloride (**13**)

Bis(4-hydroxymethylphenyl) ditelluride (1.64 g; 3.5 mmol) was treated with terephthaloyl chloride (0.71 g, 3.5 mmol) in the presence of triethylamine (0.88 g; 8.8 mmol) in a similar way to that described for preparation of **12**. A yellow solid was obtained in 77% yield (1.81 g), m.p. 153–157°C.

Found: C, 43.47; H, 2.31. Calc. for $(\text{C}_{22}\text{H}_{16}\text{O}_4\text{Te}_2)_n$: C, 43.07; H, 2.69%.

IR (cm^{-1}): 3020w, 2930m, 2850m, 1725s, 1590s, 1480s, 1280sb, 1120s.

Physical measurements

^1H and ^{13}C NMR spectra were obtained on Brucker 250 MHz

and on Jeol EX-90FT spectrometers with tetramethylsilane as an internal reference. IR spectra were recorded as KBr discs in the range 4000–200 cm^{-1} on a Pye–Unicam SP-300s infrared spectrophotometer. Microanalyses for carbon, hydrogen, and nitrogen were obtained on a Carlo-Ebra EA1-108 elemental analyzer. All melting points were determined using a Gallenkamp melting point apparatus and are uncorrected.

RESULTS AND DISCUSSION

In the present study, a series of new organotellurium compounds based on benzyl alcohol were prepared by a direct and convenient method. The preparation of compounds **1–8** was based on *trans*-telluration of 2- and 4-hydroxymethylphenylmercury chlorides¹¹ by tellurium tetrabromide. Thus, 2- and 4-hydroxymethylphenyltellurium tribromide (**1** and **5**) were readily prepared, in 80% and 75% yields respectively, by reaction of equimolar

amounts of tellurium tetrabromide with 2- and 4-hydroxyphenylmercury chlorides in refluxing dioxane, (Scheme 1).

When 1 mol of tellurium tetrabromide and 2 mol of 2- and 4-hydroxymethylphenylmercury chloride were refluxed in dry dioxane for 4 h, compounds **2** and **4** were obtained in 64% and 65% yields respectively. The dibromides **2** and **4** were reduced by ethanolic hydrazine hydrate to afford the corresponding tellurides (**3** and **7**). Reduction of **1** and **5** with sodium sulfide nonahydrate in aqueous medium at 100 °C afforded the ditellurides **4** and **8** in 65% and 68% yields respectively (Scheme 1). It is well known that most diaryl ditellurides containing *ortho*-substituted groups are oils or semi-solid compounds.^{1–3} Thus, compound **4** was obtained as a semi-solid compound, whereas **8** was obtained as an orange solid compound (see Experimental section).

The IR spectra of compounds **1–8** give characteristic bands that are quite similar to those of 2- and 4-hydroxymethylphenylmercury chlorides, confirming that telluration has occurred at the position initially occupied by HgCl. The spectra of all new compounds show strong bands around 3300 cm^{−1} due to OH stretching vibrations. The IR spectrum of **3** shows two strong broad bands centered at 3350 and 3220 cm^{−1} due to ν(OH). This is probably due to a possible interaction between the tellurium atom and the oxygen atom of one of the –CH₂OH groups.

The ¹H NMR spectra of the new compounds **1–8** exhibit the expected resonance of the *para*- and *ortho*-disubstituted benzene (see Experimental section). The ¹H NMR signal due to the methylene groups in **3** was observed as a doublet. The two resonances are due to the two non-equivalent hydrogen atoms, bound to the same carbon atom. Thus, the inequivalence is due to the incorporation of this carbon atom into a five-membered ring. This means that both hydroxymethyl groups are involved in intramolecular coordination. It is worth noting that Singh and coworkers¹² provide many similar examples. Compound **4** shows only one resonance at 4.83 ppm.

The ¹³C NMR spectra of compounds **1–8** are in agreement with their structures. The large variation for carbon atoms bearing tellurium atoms can be attributed to the polarity of the Te–C bond.^{13,14}

Solution polycondensation of **3**, **4**, and **7** with terephthaloyl chloride and TDI was the general method^{15–17} used for the preparation of the new tellurium-containing polyester and polyurethane (Scheme 2). The polymerization reactions were carried out at ambient temperature and as described in our previous work.¹⁰

The CHN analyses of the new polymers (**9–13**) are in good agreement with calculated values (see Experimental section).

The IR spectra of **9**, **10**, and **11** exhibited broad intense N–H stretching vibrations in the range 3300–3400 cm^{−1}. The ν(CO) of the urethane linkage was observed in the range 1710–1720 cm^{−1} as a strong band. The N–H bending and N–H deformation bands were observed as strong bands in the range 1655–1670 cm^{−1} and 1600–1605 cm^{−1} respectively. The asymmetrical stretching of ν(C–O–O) appeared as a

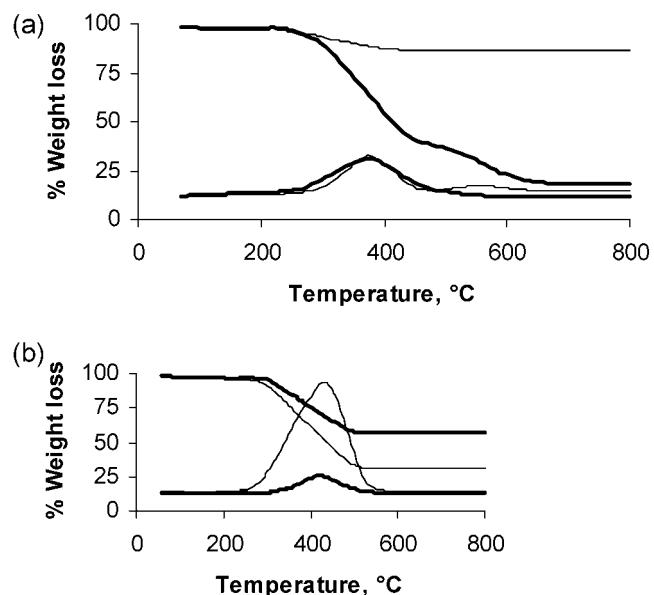


Figure 1. Thermograms for: (a) **9** (thin line) and **10** (thick line); (b) **11** (thin line) and **12** (thick line).

strong band in the range 1215–1230 cm^{−1}. Furthermore, the spectra showed a strong band in the range 1280–1300 cm^{−1} which may be assigned to aromatic ν(CN) stretching.^{17,18} The IR spectra of **12** and **13** showed strong bands attributed to carbonyl group stretching at 1720 cm^{−1} and 1725 cm^{−1} respectively. The symmetrical C–O–C absorption appeared at 1130 (**12**) and at 1120 cm^{−1} (**13**) as a strong band.^{18,19}

It was not possible to determine the molecular mass of these new polymers by viscometric, osmotic, and light-scattering techniques, due to their low solubilities in any common organic solvents.

The solid conductivities of polymers **9**, **11**, and **13** were measured at room temperature using a type 417 current amplifier with HP-K80. The values of conductivity for **9**, **11**, and **13** were found to be $1.21 \times 10^{-13} \Omega^{-1} \text{cm}^{-1}$, $1.07 \times 10^{-13} \Omega^{-1} \text{cm}^{-1}$, and $5.1 \times 10^{-14} \Omega^{-1} \text{cm}^{-1}$ respectively. This means that these polymers are insulator materials at room temperature.

The thermal stabilities of polymers **9–12** were studied by thermogravimetric analysis. The thermogram of **9** shows a total loss of 65% at 405 °C, then a loss of 15% at 625 °C. The maximum loss may be attributed to the elimination of a tellurium atom, forming TeO(NCOH) as a residual product (calculated mass loss 64.9%); Fig. 1a. The proposed product is not unique, because it is known that tellurium is capable of forming stable compounds of this type.²⁰ The polymer **10** loss of 11.25% at 415 °C is due to the elimination of CO and N₂ molecules and the residue exhibited a high thermal stability even when the temperature reached 800 °C (Fig. 1a). For polymer **11**, a total weight loss of 68.7% was observed at

Table 1. Thermal stability characteristics of new polymers containing tellurium

Polymer	Decomposition temperature ^a (°C)	Char content (%)	
	<i>T_i</i>	<i>T_{max}</i>	<i>T_f</i>
9	285 ^b	360 ^b	425 ^a
	455 ^c	510 ^c	625 ^b
10	265	375	445
11	255	405	485
12	320	400	480

^a Decomposition temperature; *T_i*: intial; *T_{max}*: maximum; *T_f*: final.

^b First decomposition temperature.

^c Second decomposition temperature.

465 °C (Fig. 1b). This can be associated with the extrusion of an extra tellurium atom per formula unit compared with polymer **10** and formation of TeO(NCOH) as a residual product (calculated mass loss 67.7%). The formation of TeO(NCOH) may be explained as a result of the presence of a water molecule, as indicated in the thermogram (Fig. 1b). For polymer **12**, a total weight loss of 42.5% was observed at 480 °C in one step (Fig. 1b).

The char content was determined at 800 °C. The thermal stability characteristics of polymers **9–12** are shown in Table 1. In general, the new polymers show good thermal stabilities compared with other polymers of the same type.^{21,22}

In conclusion, this paper described the synthesis of a new series of organotellurium compounds containing hydroxymethyl groups and their use as precursors for preparing new polymers containing tellurium.

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