# Synthesis and Reactions of 1,6-Bis(2-butyltellurophenyl)-2,5-diazahexa-1,5-diene and Related Compounds. The Crystal and Molecular Structures of 2-(Butyldichlorotelluro)benzaldehyde and Bis[2-(hydroxyiminomethyl)phenyl] Ditelluride†

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The synthesis of 1,6-bis(2-butyltellurophenyl)-2,5-diazahexa-1,5-diene and related compounds is reported. Reaction with halogenating reagents leads to rupture of the Te–Bu bond and the isolation of new organyltellurium mono- and tri-halides stabilised *via* co-ordination to the imino-nitrogen atom. Reduction of the trihalides with sodium metabisulphite stops at the monohalide stage and no ditelluride is produced. Reduction of the tri- or mono-halides with hydrazine hydrate gives, depending on the molar ratio of reactants, either *N*-amino or *N*-hydroxo-compounds and in this case ditellurides are formed. Reduction of the trihalides with $NaBH_4$ affords a poly- or oligo-meric tritelluride stabilised by co-ordination of tellurium with the imino-nitrogen atom. By contrast the reaction of 2-(butyltelluro)benzaldehyde with chlorinating agents gives 2-(butyldichlorotelluro)benzaldehyde in which the Te–Bu linkage survives intact. Spectroscopic data, particularly $^{13}C$ n.m.r., are presented and briefly discussed. The crystal and molecular structures of bis[2-(hydroxyiminomethyl)phenyl] ditelluride and of 2-(butyldichlorotelluro)benzaldehyde have been determined. The ditelluride has Te–Te 2.746(1) Å with C–Te–Te angles of 100.0(2) and 100.3(2)°; Te–N distances of 2.822(5) Å and 2.876(5) Å indicate a weak interaction. In the benzaldehyde derivative the tellurium is bonded to two carbon atoms and two chlorine atoms; a weak Te ··· O interaction [2.839(4) Å (mean for two independent molecules)] and a lone pair of electrons complete a distorted pseudo-octahedral co-ordination. The molecule, apart from the two axial chlorine atoms, is essentially planar.

Over the past few years there has been a discernible growth in interest in the co-ordination chemistry of ligands containing selenium or tellurium as donor atoms.[1] The number of bi- or multi-dentate ligands containing tellurium donor atoms to have been investigated remains small, although we,[2,3] and others,[4] have recently investigated the chemistry of some bis-tellurides. Recent synthetic work in our group has targeted ligands containing both tellurium and nitrogen as potential donor atoms. Thus, for example, organotellurium compounds functionalised with a 2-pyridyl group have been prepared[5] and some preliminary results for 1,6-bis(2-butyltellurophenyl)-2,5-diazahexa-1,5-diene[6] have been reported. In this paper we present full preparative details for the latter material and also report on a number of reactions it undergoes, some products of which are of interest as ligands in their own right. Also included are two crystal structures, one of which was undertaken to verify the identification of an unexpected reaction product and the other to explore the preference of tellurium(IV) for the formation of inter- or intra-molecular secondary bonds.[7]

## Experimental

*Synthesis of* 1,6-*Bis*(2-*butyltellurophenyl*-)2,5-*diazahexa*-1,5-*diene* (**1**).—*o*-Bromobenzaldehyde (25 g, 0.14 mol) was treated with triethyl orthoformate (20 g, 0.14 mol) in refluxing ethanol (16 cm$^3$), containing potassium hydrogensulphate (0.5 g), with stirring for 3 h. The ethanol was removed under vacuum and the residue treated with water (100 cm$^3$). The aqueous medium was shaken with diethyl ether (100 cm$^3$); the organic layer was retained and dried over anhydrous sodium sulphate. Removal of the ether afforded the liquid acetal in 96% yield. The acetal (13 g, 0.05 mol) was dissolved in dry diethyl ether (50 cm$^3$) and to this solution was added, over 15 min under dinitrogen, a solution of butyl-lithium (3.2 g in 15 cm$^3$ diethyl ether–cyclohexane). The mixture was cooled to room temperature and tellurium powder (6.5 g, 0.05 mol) was added slowly, with stirring. After reflux for 30 min, the solution was cooled, poured onto ice, and extracted with diethyl ether; the ether solution was dried over anhydrous sodium sulphate. The ether was removed in a rotary evaporator to give 2-(butyltelluro)benzaldehyde diethyl acetal in 68% yield, b.p. 148—150 °C at 0.1 mmHg. The acetal (9 g) was hydrolysed by heating gently with concentrated HCl (10 cm$^3$). On cooling the mixture was extracted with diethyl ether and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum and the product, 2-(butyltelluro)benzaldehyde, vacuum distilled at 141—143 °C and 0.1 mmHg (lit.,[8] 140—142 °C) (Found: C, 43.2; H, 4.00. $C_{11}H_{14}OTe$ requires C, 43.5; H, 3.85%).

Finally, the above product (5.2 g, 0.018 mol) in absolute ethanol (10 cm$^3$) was treated with a solution of dry 1,2-diaminoethane (0.54 g, 0.009 mol) in absolute ethanol (10 cm$^3$). Following reflux for 30 min, the mixture was cooled to room temperature to afford a yellow crystalline *product*. The compound was separated and recrystallised from ethanol to give yellow needles (3.7 g, 68%), m.p. 83—85 °C (Found: C, 47.4; H, 5.50; N, 4.60. $C_{24}H_{32}N_2Te_2$ requires C, 47.7; H, 5.30; N, 4.65%).

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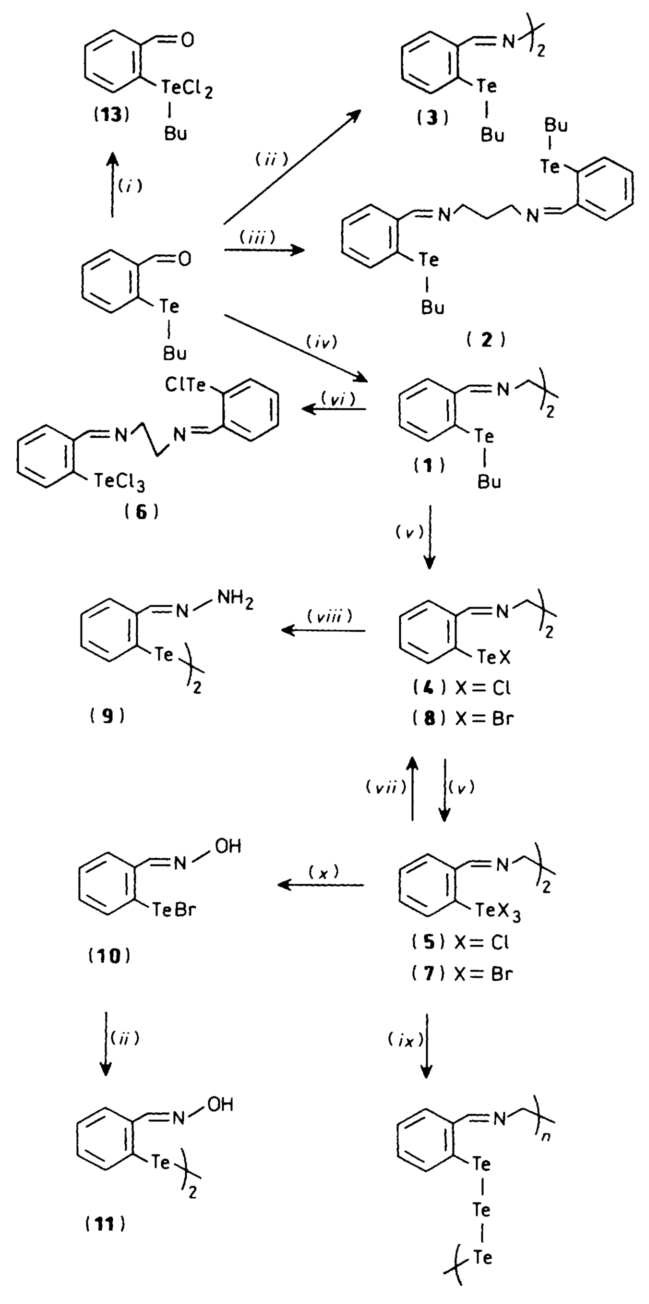
† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1989, Issue 1, pp. xvii—xx.

*Non-S.I. unit employed:* mmHg ≈ 133 Pa.



**Scheme.** Interconversions of new organyltellurium compounds. (*i*) $Cl_2$; (*ii*) $N_2H_4$; (*iii*) $H_2N(CH_2)_3NH_2$; (*iv*) $H_2N(CH_2)_2NH_2$; (*v*) $SOCl_2$ or $Br_2$; (*vi*) $SOCl_2$; (*vii*) $Na_2S_2O_5$; (*viii*) excess of $N_2H_4$; (*ix*) $NaBH_4$ and Te; (*x*) $N_2H_4$ (1:2)

*Synthesis of* 1,7-*Bis*(2-*butyltellurophenyl*)-2,6-*diazahepta*-1,6-*diene* (**2**).—1,3-Diaminopropane replaced 1,2-diaminoethane in the above preparation to give a yellow liquid product in 70% yield (Found: C, 48.4; H, 5.60; N, 4.50. $C_{25}H_{34}N_2Te_2$ requires C, 48.9; H, 5.50; N, 4.55%). Use of hydrazine hydrate gave compound (**3**), *i.e.* 1,4-*bis*(2-*butyltellurophenyl*)-2,3-*diazabuta*-1,3-*diene* (Found: C, 46.1; H, 4.75; N, 5.00. $C_{22}H_{28}N_2Te_2$ requires C, 45.9; H, 4.85; N, 4.85%).

*Chlorination of Compound* (**1**).—Three experiments were performed.

(*a*) *Synthesis of* 1,6-*bis*-(2-*chlorotellurophenyl*)-2,5-*diazahexa*-1,5-*diene* (**4**). Thionyl chloride (0.34 cm$^3$, 0.005 mol) in carbon tetrachloride (20 cm$^3$) was added slowly to a solution of compound (**1**) (1.25 g, 0.0025 mol) in carbon tetrachloride (30 cm$^3$). The mixture was stirred and gently warmed for 2 h. On cooling to room temperature a yellow solid was formed which was removed, washed several times with diethyl ether, and recrystallised from benzene. Yield 70%, m.p. 246—248 °C (Found: C, 34.3; H, 2.60; N, 4.90. $C_{16}H_{14}Cl_2N_2Te_2$ requires C, 34.3; H, 2.50; N, 5.00%).

(*b*) *Synthesis of* 1,6-*bis*(2-*trichlorotellurophenyl*)-2,5-*diazahexa*-1,5-*diene*, (**5**). Thionyl chloride (1.30 cm$^3$, 0.017 mol) in carbon tetrachloride (20 cm$^3$) was added to a solution of compound (**1**) (1.0 g, 0.0017 mol) in carbon tetrachloride (30 cm$^3$). The mixture was stirred and gently refluxed for 3 h. The yellow product was separated after cooling the mixture to room temperature, washed with carbon tetrachloride, and recrystallised from dimethylformamide (dmf)–1,4-dioxane (1:1) to give a yellow powder, m.p. 288—290 °C in 60% yield (Found: C, 28.1; H, 2.15; N, 3.95. $C_{16}H_{14}Cl_6N_2Te_2$ requires C, 27.4: H, 2.00; N, 4.00).

(*c*) *Attempted synthesis of* 1-(2-*chlorotellurophenyl*)-6-(2-*trichlorotellurophenyl*)-2,5-*diazahexa*-1,5-*diene*, (**6**). Thionyl chloride and compound (**1**) were allowed to react in a 4:1 ratio using similar conditions to those in synthesis (*b*). The yellow product was recrystallised from 1,4-dioxane to give a yellow amorphous powder of m.p. 226—228 °C in 60% yield (Found: C, 29.8; H, 2.55; N, 4.10. $C_{16}H_{14}Cl_4N_2Te_2$ required C, 30.5; H, 2.20; N, 4.45%).

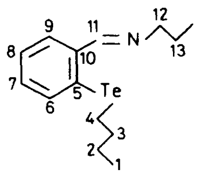
*Bromination of Compound* (**1**).—(*a*) *Synthesis of* 1,6-*bis*(2-*tribromotellurophenyl*)-2,5-*diazahexa*-1,5-*diene*, (**7**). Compound (**1**) (2.41 g, 0.004 mol) in carbon tetrachloride was treated with an excess of bromine in the same solvent. Following stirring and gentle warming over 2 h, the mixture was cooled and the yellow precipitate separated. After washing with carbon tetrachloride the *product* was recrystallised from 1,4-dioxane in 90% yield, m.p. 296—298 °C (Found: C, 20.2; H, 1.10; Br, 49.4; N, 2.70. $C_{16}H_{14}Br_6N_2Te_2$ requires C, 19.8; H, 1.40; Br, 49.5; N, 2.90%).

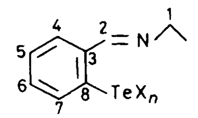
(*b*) *Synthesis of* 1,6-*bis*(2-*bromotellurophenyl*)-2,5-*diazahexa*-1,5-*diene*, (**8**). Compound (**1**) (4.0 g, 0.0066 mol) in carbon tetrachloride (30 cm$^3$) was treated with bromine [1.73 cm$^3$, 0.033 mol; *i.e.* $Br_2$: (**1**) = 5:1] in carbon tetrachloride after which procedure (*a*) above was followed. The yellow precipitate was filtered off, washed with carbon tetrachloride, and recrystallised from 1,4-dioxane; m.p. 259—261 °C (Found: C, 29.0; H, 2.10; N, 4.25. $C_{16}H_{14}Br_2N_2Te_2$ requires C, 29.6; H, 2.15; N, 4.30%). This compound may also be prepared by sodium disulphite ($Na_2S_2O_5$) reduction of the bis-tribromide, however use of an excess of reductant did not afford a ditelluride.

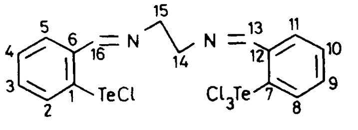
*Reductions with Hydrazine Hydrate.*—*Synthesis of bis*-(2-*hydrazonomethylphenyl*) *ditelluride* (**9**). 1,6-Bis(2-bromotellurophenyl)-2,5-diazahexa-1,5-diene (3.25 g, 0.0005 mol) was suspended in absolute ethanol (30 cm$^3$) and a 20-fold molar excess of hydrazine hydrate dissolved in absolute ethanol (20 cm$^3$) was added. The mixture was refluxed for 1 h, then cooled to room temperature and filtered. From the filtrate was isolated a bright yellow solid following evaporation of the solvent; this proved to be unreacted starting material (i.r. spectrum and m.p.). The residue consisted of a black solid (tellurium) and an orange-yellow material which recrystallised from 1,4-dioxane to give compound (**9**) in 44% yield, m.p. 159—161 °C (Found: C, 35.0; H, 2.80; N, 11.35. $C_{14}H_{14}N_4Te_2$ requires C, 34.1; H, 2.85; N, 11.35%).

*Synthesis of* [2-(*hydroxyiminomethyl*)*phenyl*]*tellurium*(II) *bromide*, (**10**). A suspension of 1,6-bis(2-tribromotellurophenyl)-2,5-diazahexa-1,5-diene (1.94 g, 0.002 mol) in ethanol (30 cm$^3$)

**Table 1.** Carbon-13 n.m.r. data (δ/p.p.m.)

| Compound | $C^1$ | $C^2$ | $C^3$ | $C^4$ | $C^5$ | $C^6$ | $C^7$ | $C^8$ | $C^9$ | $C^{10}$ | $C^{11}$ | $C^{12}$ | $C^{13}$ | $C^{14}$ | $C^{15}$ | $C^{16}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  | | | | | | | | | | | | | | | | |
| (**1**) | 13.5 | 25.5 | 31.9 | 8.2 | 121.2 | 132.3 | 133.1 | 124.9 | 129.7 | 136.2 | 162.9 | 60.4 | — | | | |
| (**2**) | 13.5 | 26.9 | 32.1 | 8.1 | 120.8 | 132.2 | 132.9 | 124.9 | 129.7 | 136.1 | 161.8 | 57.1 | 32.6 | | | |
| (**3**) | 13.5 | 25.5 | 32.3 | 9.2 | 120.2 | 133.3 | 133.7 | 125.6 | 130.4 | 135.2 | 162.6 | — | — | | | |
| (**4**)[a] | 56.8 | 167.1 | 135.5 | ← 126.7—133.0 → | | | | 140.8 | | | | | | | | |
| (**5**)[a] | 54.8 | 173.8 | 132.2 | ← 126.8—133.2 → | | | | 150.0 | | | | | | | | |
| (**6**)[b] | ← 126.7—137.3 → | | | | | | | | | | | | 173.7 | 55.0 | 56.8 | 167.6 |
| (**7**)[a] | 56.7 | 166.7 | 135.6 | 131.6 | 133.1 | 126.9 | 133.7 | 138.4 | | | | | | | | |
| (**8**)[a] | 56.7 | 166.8 | ← 126.8—133.7 → | | | | | | | | | | | | | |
| (**12**) | 54.6 | 140.8 | — | 128.6 | 129.7 | 126.9 | 131.7 | 132.0 | | | | | | | | |

[a] 

[b] 

was treated with hydrazine hydrate (0.004 mol) in ethanol (10 cm$^3$). The mixture was refluxed for 15 min and the solid material was filtered off. Recrystallisation from benzene gave a yellow crystalline material of m.p. 176—178 °C in 35% yield (Found: C, 24.7; H, 2.30; N, 4.55. $C_7H_6BrNOTe$ requires C, 25.6; H, 1.85; N, 4.25%).

*Synthesis of bis*[2-(*hydroxyiminomethyl*)*phenyl*] *ditelluride*, (**11**). 1,6-Bis(2-tribromotellurophenyl)-2,5-diazahexa-1,5-diene (1.94 g, 0.002 mol) was suspended in ethanol (30 cm$^3$) and treated with a six-fold molar excess of hydrazine hydrate in ethanol (15 cm$^3$). The mixture was refluxed for 20 min, cooled, and filtered. The resulting solid was recrystallised from benzene to give yellow-orange crystals in 75% yield, m.p. 276—278 °C (Found: C, 34.9; H, 2.50; N, 5.45. $C_{14}H_{12}N_2O_2Te_2$ requires C, 33.9; H, 2.40; N, 5.65%). This compound was characterised by *X*-ray crystallography, see below.

*Synthesis of poly*-{*bis*[2-(6-*phenyl*-2,5-*diazahexa*-1,5-*dienyl*)-*phenyl*] *tritelluride*}, (**12**). 1,6-Bis(2-tribromotellurophenyl)-2,5-diazahexa-1,5-diene (3.87 g, 0.004 mol) was suspended in ethanol (30 cm$^3$) and tellurium powder (0.26 g, 0.002 mol) was added. The mixture was stirred under argon whilst sodium tetrahydroborate (1.1 g, 0.03 mol) was added slowly. When the tellurium had reacted, the reddish solution was refluxed for 30 min, and filtered hot. On cooling the filtrate to room temperature, an orange precipitate appeared which was recrystallised from benzene in 65% yield, m.p. 127—128 °C (Found: C, 30.8; H, 2.50; N, 4.10. $C_{16}H_{14}N_2Te_3$ requires C, 31.1; H, 2.25; N, 4.55%).

*Synthesis of* 2-(*Butyldichlorotelluro*)*benzaldehyde*, (**13**).—2-(Butyltelluro)benzaldehyde (5.0 g, 0.014 mol) in carbon tetrachloride (30 cm$^3$) was treated with a stream of chlorine gas. On completion of the reaction, the solvent was removed and the solid residue was recrystallised from hexane–benzene (1:1). Bright white needles of the product were formed in 85% yield, m.p. 107—109 °C (lit.,[8] 105—110 °C) (Found: C, 36.6; H, 3.90. $C_{11}H_{14}Cl_2OTe$ requires C, 36.6; H, 3.90%).

*Physical Measurements.*—Infrared spectra were recorded for KBr disks with a Perkin-Elmer 1710 FTIR spectrophotometer (model PP-1), n.m.r. spectra ($^1H$, $^{125}Te$, and $^{13}C$) with a Bruker AC-300 instrument using tetramethylsilane as reference for $^1H$ and $^{13}C$ n.m.r. spectra. Conductivity measurements were made with a standard Mullard conductivity bridge and immersion-type platinum electrodes. Microelemental analyses were carried out by the Analytical Services section of the Department of Chemical Engineering and Applied Chemistry, and by Butterworth's Analytical Service Laboratories.

The Scheme gives the interconversions of the compounds whose syntheses are described above. Carbon-13 n.m.r. data are given in Table 1.

*Crystal and Molecular Structures of Compounds* (**11**) *and* (**13**).—After preliminary examination by photographic methods, the final cell dimensions and reflection intensities were measured with graphite-monochromated Mo-$K_\alpha$ radiation on an Enraf-Nonius CAD-4 diffractometer, operating in the ω—2θ scan mode. Three standard reflections were monitored at regular intervals and the orientation of the crystal was checked every 200 reflections. Compound (**13**) suffered severe decomposition when irradiated with *X*-rays, which necessitated the replacement of the original crystal once the intensity standards had dropped to 60% of their initial value. Scale factors were applied to correct for crystal deterioration and to equalise the data from the two crystals used. Lorentz and polarisation factors were applied. Details of the crystal structure determination and refinement parameters are given in Table 2.

The tellurium atoms of compounds (**11**) and (**13**) were located from Patterson syntheses. In both crystal structures the remaining non-hydrogen atoms were located by Fourier

**Table 2.** Crystal structure determination and refinement parameters *

| Compound | (**13**) | (**11**) |
|---|---|---|
| Formula | $C_{11}H_{14}Cl_2OTe$ | $C_{14}H_{12}N_2O_2Te_2$ |
| *M* | 360.7 | 495.5 |
| Crystal size (mm) | 0.575 × 0.275 × 0.025<br>(second crystal) | 0.475 × 0.625 × 0.300 |
| *a*/Å | 21.760 (9) | 15.790 (3) |
| *b*/Å | 8.178 (8) | 6.340 (8) |
| *c*/Å | 15.441 (5) | 15.903 (6) |
| β/° | 92.76 (3) | 99.50 (2) |
| *U*/Å$^3$ | 2 744.6 | 1 570.2 |
| *Z* | 8 | 4 |
| *F*(000) | 1 392 | 920 |
| $D_c$/g cm$^{-3}$ | 1.746 | 2.096 |
| μ/mm$^{-1}$ | 2.607 | 3.856 |
| Total data measured | 5 587 | 5 868 |
| Significant data [$I > 2.5\sigma(I)$] | 3 099 | 2 491 |
| Maximum shift/error in final least-squares cycle | 0.015 | 0.01 |
| *R* | 0.0626 | 0.0753 |
| *R′* | 0.0844 | 0.0906 |

* Details common to both compounds: space group $P2_1/n$; λ 0.710 69 Å; θ 2—25°; weighting scheme $w = [\sigma^2(F) + KF^2]^{-1}$ where $K = 0.002$.

**Table 3.** Fractional atomic co-ordinates ( × 10$^5$ for tellurium and × 10$^4$ for all other non-hydrogen atoms) with estimated standard deviations (e.s.d.s) in parentheses for compound (**13**)

| Atom | *x* | *y* | *z* | Atom | *x* | *y* | *z* |
|---|---|---|---|---|---|---|---|
| Te(1A) | 16 955(3) | 20 227(9) | 4 766(6) | Te(1B) | 83 608(3) | 64 597(9) | 45 051(6) |
| Cl(1A) | 2 408(2) | 2 386(4) | −734(3) | Cl(1B) | 7 639(2) | 6 789(4) | 5 708(3) |
| Cl(2A) | 989(2) | 2 064(6) | 1 708(3) | Cl(2B) | 9 092(2) | 6 538(5) | 3 295(3) |
| O(1A) | 789(5) | 794(13) | −747(9) | O(1B) | 9 236(5) | 5 149(12) | 5 730(8) |
| C(1A) | 1 161(5) | 3 946(13) | −92(8) | C(1B) | 8 897(5) | 8 350(14) | 5 108(8) |
| C(2A) | 1 281(6) | 5 536(17) | 173(9) | C(2B) | 8 785(6) | 9 972(13) | 4 873(8) |
| C(3(A) | 933(6) | 6 814(16) | −180(9) | C(3B) | 9 130(6) | 11 169(15) | 5 258(9) |
| C(4A) | 465(6) | 6 533(19) | −792(9) | C(4B) | 9 573(6) | 10 863(16) | 5 881(8) |
| C(5A) | 355(6) | 4 955(17) | −1 060(9) | C(5B) | 9 674(6) | 9 269(18) | 6 151(9) |
| C(6A) | 699(6) | 3 660(16) | −707(8) | C(6B) | 9 338(5) | 7 998(14) | 5 756(9) |
| C(7A) | 553(7) | 2 024(20) | −1 009(11) | C(7B) | 9 477(8) | 6 340(17) | 6 040(11) |
| C(8A) | 2 324(5) | 3 461(15) | 1 247(9) | C(8B) | 7 743(5) | 7 933(15) | 3 744(9) |
| C(9A) | 2 827(6) | 2 415(18) | 1 679(10) | C(9B) | 7 222(5) | 6 954(14) | 3 323(9) |
| C(10A) | 3 254(6) | 3 451(17) | 2 250(8) | C(10B) | 6 786(5) | 8 004(15) | 2 794(8) |
| C(11A) | 3 796(7) | 2 422(21) | 2 626(10) | C(11B) | 6 247(6) | 7 044(18) | 2 379(9) |

difference syntheses. With the exception of the O–H hydrogens of compound (**11**), which were found from a Fourier difference synthesis, hydrogen atoms were placed in calculated positions (C–H 1.08 Å) and were allowed to 'ride' on their respective carbon atoms during the subsequent least-squares refinements. Final atomic co-ordinates for compounds (**11**) and (**13**) are given in Tables 3 and 4, bond distances and angles in Tables 5 and 6. The molecular structures are illustrated in Figures 1 and 2.

Computations were carried out on the Birmingham University Honeywell computer with SHELX[9,10] and molecular diagrams were drawn with PLUTO.[11]

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates, thermal parameters, and remaining bond lengths and angles.

## Discussion

The crystal structure of 1,6-bis(2-butyltellurophenyl)-2,5-diazahexa-1,5-diene, (**1**) has been reported,[6] and full preparative details for the compound are now provided. One of the interesting features of the structure is the Te–N distance of 2.773 Å, well within the van der Waals distance of 3.61 Å following Bondi[12] or 3.7 Å following Pauling.[13] On complexing to mercury(II) the Te–N interactions remain almost unchanged[6] (2.752 and 2.786 Å) and the ligand co-ordinates to the metal *via* the tellurium atoms only, thus giving a complex with a 13-membered chelate ring. [Actually, if the Te–N interaction is accepted as a weak co-ordinate bond, then the mercury(II) ion 'sees' a seven-membered chelate ring.] Other bases may replace 1,2-diaminoethane in the synthetic method for (**1**), thus the related compounds (**2**) and (**3**) (see Scheme) may be prepared using 1,3-diaminopropane or hydrazine hydrate. These materials were characterised by analysis and n.m.r. spectroscopy (Table 2). In addition $^{125}$Te n.m.r. spectra were recorded for $CDCl_3$ solutions of (**1**)—(**3**) the observed chemical shifts with respect to $TeMe_2$ being 464.4, 464.9, and 464.1 p.p.m. respectively. These data confirm the equivalence of the two tellurium atoms in each of the molecules, and since the shieldings are identical within experimental error the data strongly suggest that a weak Te–N interaction occurs in (**2**) and (**3**) similar to that demonstrated in (**1**).

We were interested to ascertain the strength of the Te–N interaction if the oxidation state of the tellurium were increased to IV; thus the reaction of compound (**1**) with both $SOCl_2$ and $Br_2$ was investigated. The reaction of $SOCl_2$ with (**1**) in a 2:1 mol ratio gave a bis(tellurenyl chloride) (**4**) with loss of the butyl group (analysis, i.r. and $^{13}$C n.m.r. spectra; no resonances between 0 and 50 p.p.m.). Compound (**2**) undergoes a similar reaction with $Br_2$ to give similar products which it is not

**Table 4.** Fractional atomic co-ordinates ( × $10^5$ for tellurium and × $10^4$ for all other non-hydrogen atoms) with e.s.d.s in parentheses for compound (**11**)

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Te(1) | −5 300(3) | 5 060(9) | 9 620(3) |
| Te(2) | −12 014(3) | 39 820(9) | 588(3) |
| O(1) | 678(6) | −4 324(11) | 2 173(5) |
| O(2) | −1 901(7) | 8 600(15) | −1 650(7) |
| N(1) | 106(5) | −2 604(11) | 2 173(5) |
| N(2) | −2 090(5) | 6 804(13) | −1 204(5) |
| C(1) | −1 023(5) | 959(12) | 2 122(5) |
| C(2) | −1 523(5) | 2 726(13) | 2 238(5) |
| C(3) | −1 874(5) | 2 936(17) | 2 969(6) |
| C(4) | −1 777(6) | 1 406(15) | 3 580(5) |
| C(5) | −1 280(6) | −313(15) | 3 474(6) |
| C(6) | −900(5) | −620(13) | 2 773(5) |
| C(7) | −374(6) | −2 452(13) | 2 724(6) |
| C(8) | −2 499(5) | 2 954(12) | −328(4) |
| C(9) | −2 794(6) | 1 147(14) | −2(6) |
| C(10) | −3 661(6) | 501(19) | −199(6) |
| C(11) | −4 230(6) | 1 837(19) | −718(7) |
| C(12) | −3 929(6) | 3 552(20) | −1 064(7) |
| C(13) | −3 077(5) | 4 206(15) | −868(5) |
| C(14) | −2 845(6) | 6 136(15) | −1 300(6) |

**Table 5.** Selected bond lengths (Å) and angles (°) with e.s.d.s in parentheses for compound (**13**)

| | | | |
|---|---|---|---|
| Te(1A)–Cl(1A) | 2.504(4) | Te(1B)–Cl(1B) | 2.504(4) |
| Te(1A)–Cl(2A) | 2.503(4) | Te(1B)–Cl(2B) | 2.512(4) |
| Te(1A)···O(1A) | 2.848(4) | Te(1B)···O(1B) | 2.830(4) |
| Te(1A)–C(1A) | 2.121(11) | Te(1B)–C(1B) | 2.125(11) |
| Te(1A)–C(8A) | 2.124(12) | Te(1B)–C(8B) | 2.119(12) |
| O(1A)–C(7A) | 1.192(20) | O(1B)–C(7B) | 1.196(18) |
| Cl(1A)–Te(1A)–Cl(2A) | 172.4(1) | Cl(1B)–Te(1B)–Cl(2B) | 172.4(1) |
| Cl(1A)–Te(1A)–C(1A) | 87.1(3) | Cl(1B)–Te(1B)–C(1B) | 87.0(3) |
| Cl(2A)–Te(1A)–C(1A) | 87.7(3) | Cl(2B)–Te(1B)–C(1B) | 87.3(3) |
| Cl(1A)–Te(1A)–C(8A) | 87.0(4) | Cl(1B)–Te(1B)–C(8B) | 86.9(4) |
| Cl(2A)–Te(1A)–C(8A) | 88.2(4) | Cl(2B)–Te(1B)–C(8B) | 88.9(4) |
| O(1A)–Te(1A)–C(1A) | 68.8(4) | O(1B)–Te(1B)–C(1B) | 69.2(4) |
| O(1A)–Te(1A)–C(8A) | 166.8(4) | O(1B)–Te(1B)–C(8B) | 167.2(4) |
| C(1A)–Te(1A)–C(8A) | 98.4(4) | C(1B)–Te(1B)–C(8B) | 98.6(4) |

**Table 6.** Bond lengths (Å) and selected bond angles (°) with e.s.d.s in parentheses for compound (**11**)

| | | | |
|---|---|---|---|
| Te(1)–Te(2) | 2.746(1) | C(4)–C(5) | 1.370(14) |
| Te(1)–C(1) | 2.136(8) | C(5)–C(6) | 1.364(13) |
| Te(2)–C(8) | 2.142(8) | C(6)–C(7) | 1.438(12) |
| O(1)–N(1) | 1.415(10) | C(8)–C(9) | 1.370(12) |
| O(2)–N(2) | 1.399(12) | C(8)–C(13) | 1.393(11) |
| N(1)–C(7) | 1.253(12) | C(9)–C(10) | 1.414(12) |
| N(2)–C(14) | 1.252(12) | C(10)–C(11) | 1.401(16) |
| C(1)–C(2) | 1.400(11) | C(11)–C(12) | 1.340(16) |
| C(1)–C(6) | 1.431(10) | C(12)–C(13) | 1.393(13) |
| C(2)–C(3) | 1.375(12) | C(13)–C(14) | 1.478(13) |
| C(3)–C(4) | 1.363(13) | | |
| Te(2)–Te(1)–C(1) | 100.3(2) | Te(1)–Te(2)–C(8) | 100.0(2) |
| O(1)–N(1)–C(7) | 121.2(8) | O(2)–N(2)–C(14) | 119.4(8) |

necessary to discuss. This reaction is not without precedent since Cava and co-workers[14] have shown that the reaction of 1 mol of $Br_2$ with benzyl *o*-nitrophenyl telluride gives *o*-nitrophenyltellurium bromide. Cava speculates that the mechanism involves an ionic tellurium(IV) intermediate of the type $R(R')TeBr^+Br^-$, a view that gains support from our recent isolation of similar species.[5] Use of a moderate excess of bromine produced the corresponding tellurenyl bromide, (**8**). It is therefore no surprise that an excess of halogenating agent should produce bis(organotellurium trihalides), chloride (**5**) and bromide (**7**). These materials are adequately characterised by $^{13}C$ n.m.r. spectroscopy (Table 1) and elemental analysis. Although these substances could be recrystallised, they gave amorphous powders which were not suitable for crystallography, hence we are left to speculate as to their molecular complexity. It is however likely that the Lewis acidity of the tellurium will be satisfied by internal co-ordination with the imino-nitrogen in a similar fashion to the case of (phenylazophenyl-*C,N'*)tellurium (IV) trichloride,[15] rather than by the formation of bromo-bridged polymers or oligomers. The bis(monohalides) are also likely to be similar structurally to (phenylazophenyl-*C,N'*)tellurium(II) chloride.[16]

Since it was certain that the formation of the bis(trihalides) followed the formation of the bis(monohalide) *via* loss of the butyl group, we attempted to isolate the intermediate 'monohalide, trihalide' by treating compound (**1**) with a four molar excess of $SOCl_2$. The reaction did indeed produce a material, (**6**), which gave the correct analysis and sharp melting point [although this was below those of both the bis(trichloride) and the bis(monochloride)]. If the intermediate has been formed, the two halves of the molecule are chemically and magnetically inequivalent, hence this should be reflected in the $^1H$ n.m.r. spectrum. Indeed, two signals are seen for the imino-protons at δ 9.64 and 9.75 [*cf.* 9.63, bis(trichloride); 9.75, bis(monochloride)]; also for the methylene protons of the 1,5-diene backbone of the molecule a broad (4 H) signal is seen at δ 4.61 (poorly resolved AB multiplet?), rather than the sharp singlets at 4.60 and 4.61 for the pure bis(tri- and bis(mono-chlorides) respectively. The $^{13}C$ n.m.r. spectrum shows two imino-carbon resonances (173.7 and 167.6 p.p.m., *cf.* Table 1), also two methylene carbon resonances (55.0 and 56.8 p.p.m.). We feel that this evidence is too ambiguous to confirm the identity of the compound, although it would be remarkable if the alternative perfect 1:1 mixture was produced. In common with compounds (**4**) and (**5**), this material was amorphous.

The reduction of organyltellurium(IV) trihalides or of organyltellurium(II) halides may afford ditellurides. Reduction of compounds (**4**), (**5**), (**7**), or (**8**) may therefore lead to either polymeric or macrocyclic ditellurides, hence attempts were made to reduce these materials using $Na_2S_2O_5$ or hydrazine hydrate. The results obtained were not as initially expected.

Sodium disulphite reduced the trihalides to the monohalides, but attempts to carry the reduction further led to recovery of the monohalides. Use of 2 molar equivalents of hydrazine hydrate with the tribromide (**7**) also produced a monobromide (**10**). The material had a strong i.r. band at 3 260 cm$^{-1}$ [ν(OH)] and elemental analysis, together with i.r. and n.m.r. data, confirm the loss of the 1,2-diaminoethane residue. The product was a hydroxyimino-compound (see Scheme). Treatment of (**10**) with an excess of hydrazine hydrate [or of (**7**) with a 6:1 excess of $N_2H_4{\cdot}H_2O$] gave a ditelluride (**11**) which afforded crystals suitable for *X*-ray analysis, which indeed confirmed the presence of an hydroxyimino-group.

The bis(monobromide) (**8**) was treated with a large excess (20:1) of hydrazine hydrate to give a moderate (44%) yield of compound (**9**). The presence of the amino-group was supported by the observation of bands at 3 372 and 3 269 cm$^{-1}$ [ν($NH_2$)] in the i.r. spectrum. Also a $^1H$ n.m.r. resonance at δ 7.14 (2 H) was assignable to the amino-group. The above observations suggest that there is a base-promoted hydrolysis of the Schiff base and that hydrazine reacts with the released aldehyde [*cf.* the formation of compound (**3**)] to give an N-amino compound.

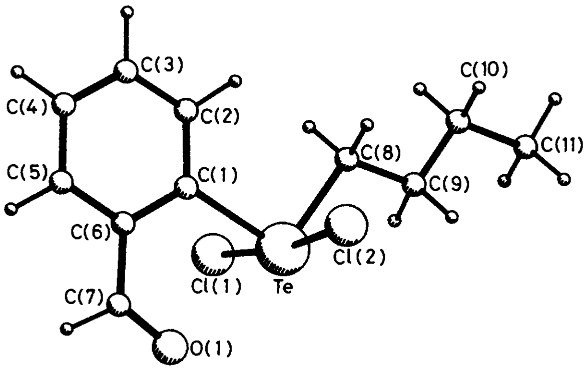


**Figure 1.** View of compound (**13**)

In the presence of an excess of hydrazine this compound survives to give, after reduction, compound (**9**). However if hydrazine is not in large excess, further hydrolysis occurs to give compound (**10**) and, following reduction, (**11**).

We recently demonstrated that reduction of co-ordinatively stabilised organyltellurium halides with sodium tetrahydroborate in the presence of tellurium gave tritellurides.[5,17] Accordingly, compound (**7**) was thus treated and gave a product which was analytically a tritelluride, (**12**). The use of $NaBH_4$ gave no problem with the hydrolytic stability of the Schiff-base linkage. No crystals suitable for *X*-ray analysis have yet been grown, although it seems probable that the material is poly- or oligomeric. The generality of the previously reported tritelluride synthesis[17] is however usefully illustrated.

The loss of the butyl group on treatment of compound (**1**) or (**2**) with halogen led to the discovery of a range of new compounds, but frustrated the initial objective to investigate the preference of tellurium(IV) in a diorganyltellurium dihalide for intra- or inter-molecular secondary interactions. Since 2-(butyltelluro)benzaldehyde was available as a synthetic intermediate in the formation of compounds (**1**)—(**3**), we elected to halogenate this material. The reaction went smoothly to give a well crystalline phase, (**13**), with no breakage of the Te–Bu linkage. The crystal structure of this material is discussed below.

$^{13}C$ *N.m.r. Data.*—Some representative data are given in Table 1; full discussions of the assignments are given in refs. 18 and 19. We confine further comment to the chemical shifts of the carbon atoms bonded directly to tellurium. For the butyl group this carbon has a high-field resonance (8.1—9.2 p.p.m. *vs.* $SiMe_4$) as expected.[2] In a discussion of the effect of tellurium on the directly attached carbon of a phenyl ring it has been assumed[20] that the 'heavy atom effect' is the major cause of the significant decrease in $\delta$ [112 p.p.m. in TeEt(Ph) relative to benzene, 128.7 p.p.m.]. Shielding effects of the relatively electropositive tellurium atom have been invoked to account for the *ipso* carbon shift (114.7 p.p.m.) of $TePh_2$.[21] The *ipso* carbon shifts reported in Table 1 ($C^5$ and $C^8$) are therefore amongst the highest such shifts for organyltellurium compounds. If the data in Table 1 are considered, it seems probable that the $C^5$ shift reflects the weak Te–N interaction revealed in the structure of compound (**1**).[6] Kalabin *et al.*[20] assumed that differences in *ipso* carbon chemical shifts for $PhER_n$ (E = element from Group 14—17, and $n$ = 3, 2, 1, or 0 respectively) could be related to the number of non-bonded electron pairs in the valence shell of E (more positive shift as the number of electron pairs increases). Simplistically, the Te–N bond introduces more electron density into the tellurium valence shell and renders the atom more halogen-like, *cf.* PhBr, *ipso* carbon 123.3 p.p.m. and compound (**1**), 121.2 p.p.m.

Consideration of the *ipso* carbon resonance in Table 1 reveals even more positive shifts. The tellurium substituent in these compounds is now significantly more electropositive and the order of shift *e.g.* $C^8(-TeCl_3-N) > C^8(-TeCl-N)$ is in accord with expectation for the relative shieldings of these groups, given that $-TeCl_3-N$ will be more electropositive than –TeCl–N.

*Crystal and Molecular Structures of Compounds* (**11**) *and* (**13**).—The structure of compound (**13**) is shown in Figure 1. The asymmetric unit consists of two discrete molecules approximately related by a non-crystallographic pseudo-two-fold screw axis. Their geometries are quite similar and only molecule B is depicted in Figure 1. The Te $\cdots$ O(1) distance (2.848 and 2.830 Å in the two molecules) is much longer than the sum of the covalent radii (2.03 Å)[13] but much less than the sum of the van der Waals radii[12] (3.60 Å), indicating a weak co-ordinate interaction. Similar long Te-O(carbonyl) contacts have been reported in (2,6-diacetylpyridine-C,N,O)tellurium(IV) trichloride[22] and in acetato(2-phenylazophenyl-*C,N'*)tellurium(II) (2.953 Å).[23] If the Te–O interaction is regarded as significant the co-ordination around the tellurium atom of compound (**3**) can be considered as essentially octahedral, with a lone pair of electrons occupying the fourth equatorial site. The molecule, apart from the apical chlorine atoms, Cl(1) and Cl(2), is practically planar (deviation from planarity is ±0.19 Å for molecule A and ±0.21 Å for B). The group of atoms forming the basal plane, Te,C(1),O(1),C(8), is coplanar within ±0.05 Å in molecule A and deviates by ±0.08 Å in molecule B; Cl(1) and Cl(2) lie 2.45 and 2.54 Å on opposite sides of the basal plane in molecule A, 2.45 and 2.56 Å in molecule B. The angles between the mean planes of the aromatic ligand and the butyl chain are 8.3 (molecule A) and 11.0° (molecule B).

Some of the distortions of the co-ordination geometry from ideal octahedral values (180 and 90°) can be explained in terms of a lone pair of electrons occupying an equatorial position between O(1) and C(8). The Cl–Te–C angles are slightly less than 90° so that the Te–Cl bonds are bent slightly away from the lone pair as might be expected from the valence-shell electron-pair repulsion (VSEPR) theory, resulting in a Cl–Te–Cl angle of 172.4° in both molecules. The small value of O(1)–Te–C(1) (68.8, A; 69.2°, B) and the O(1)–Te–C(8) angles of 166.8 (A) and 167.2° (B) are due to constraints arising from the five-membered Te–C(1)–C(6)–C(7)–O(1) ring system.

The distance Te–C(1) (2.121 and 2.125 Å) is in good agreement with the sum of the Pauling[13] single-bond covalent radii of Te (1.37 Å) and $sp^2$-hybridised carbon (0.74 A), and with the values in the crystal structures of the analogous compounds $C_{12}H_9N_2TeCl_3$,[15] $(C_7H_7O)_2TeCl_2$,[24] $Ph_2TeCl_2$,[25] and phenoxatellurium-10,10-dichloride[26] which fall within the range 2.087—2.114 Å. The distance Te–C(8) [2.124(12) and 2.119(12) Å] is somewhat shorter than the sum of the Pauling[13] single-bond covalent radii for a Te–$sp^3$-hybridised carbon single covalent bond, 2.142 Å, and may be compared with values in analogous structures[6,27,28] which fall in the range 2.115—2.185 Å. The Te–Cl distances, 2.503—2.512 Å, are in good agreement with the sum of covalent radii for Te(axial)[29] and Cl[13] of 2.52 Å and fall within the range of 2.45—2.58 Å (mean 2.51 Å) defined by the structural data for four other $R_2TeCl_2$ compounds.[24—26,30] These values may also be compared with mean lengths C(aromatic)–Te 2.116, C($sp^3$)–Te 2.158, and Cl–Te 2.520 Å given by Allen *et al.*[31] There is a weak secondary[7] intermolecular interaction between Te and Cl(1), 3.740(4) and 3.775(4) Å, approximately *trans* to the Te–C(1) bond. Although this feature is similar to secondary interactions observed in a number of diorganyltellurium dihalides,[25,26,30,31] in compound (**13**) the weak intramolecular interaction with O(1) is preferred to the formation of a second intermolecular contact. Since

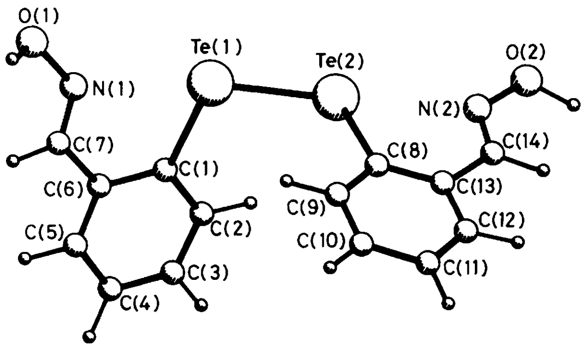


**Figure 2.** View of compound (**11**)

no other significant intermolecular interactions are noted, the molecule could be regarded as monomeric with weak interaction between Te and a chlorine in a neighbouring molecule.

The structure of complex (**11**) is shown in Figure 2. The formulation of the complex as an $N$-hydroxo-derivative is confirmed. The Te–Te bond length of the ditelluride, 2.746(1) Å, is in good agreement with the sum of the Pauling single-bond covalent radii[13] (2.74 Å) and may be compared to values in the crystal structures of a number of analogous compounds, range 2.697—2.715 Å,[32–35] and with a value in a tritelluride of 2.776 Å.[17] The distances Te(1)–C(1) 2.136(8) Å and Te(2)–C(8) 2.142(8) Å are slightly longer than the sum of the Pauling[13] single-bond covalent radii for a Te–C($sp^2$) single bond, 2.11 Å, but are similar to values found in other diorganyl ditellurides, range 2.09—2.16 Å,[32–35] and with the analogous bond in a tritelluride, 2.130(9) Å.[17] The Te–N distances, 2.822(5) and 2.876(5) Å, are much greater than the sum of the covalent radii,[13] but are within the van der Waals distance, 3.61 Å,[12] and therefore can be considered to represent a weak interaction. In fact the Te–N distances in this ditelluride, in which the formal oxidation state of tellurium is I, are shorter than those recently reported[36] for the tellurium(IV) compound $Me_2Te(OC_6H_4N{=}CHC_6H_4NO_2\text{-}4)_2$, 2.962 and 2.923 Å. The aldoxime and phenyl groups of the compound are each planar, with the aldoxime group oriented at 11.2(3) and 4.1(2)° to the phenyl ring. The bond lengths and angles of the aldoxime groups are in reasonable agreement with previously published results.[37]

Examination of packing distances shows that the shortest distances between tellurium atoms are between Te(1) and Te(1) ($-x$, $-y$, $-z$), 3.777(4) Å, and between Te(2) and Te(2) ($-x$, $-y$, $-z$), 4.041(4) Å. These distances are close to the van der Waals distance of 4.12[12] or 4.40 Å[13] and may thus represent a very weak interaction between the molecules. In the crystal there is parallel stacking of the organic rings, but the rings do not directly overlie one another.

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