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C,H,TeBr, C,H,TeX (X = Cl, Br) and (C,H,)$_2$TeX$_2$ (X = Br, I) have been prepared by the reaction of triphenyltin bromide, tetraphenyltin and tetraethyltin with appropriate tellurium compounds. Both tetraorganotin and triorganotin halides have shown the tendency to form diorganotin dihalide converting tellurium tetrahalide to diorganotellurium dihalide and organotellurium trihalide.

In an earlier communication$^1$ we reported the preparation of some phenylselenium and phenyltellurium chlorides from phenyltin compounds. We report here the preparation of phenyltellurium bromides and iodides along with some ethyl derivatives of tellurium.

Materials and Methods

All the reagents were purified and dried before use. Selenium tetrabromide, tellurium tetrabromide and tetraiodide,$^4$ tetraethyltin, tetraphenyltin$^5$ and triphenyltin bromide were prepared by the methods reported earlier. Tellurium tetrachloride (BDH, LR) was used as such. Tellurium was estimated gravimetrically as elemental tellurium,$^6$ Chlorine and bromine were estimated gravimetrically as silver chloride and bromide respectively.

Phenyltellurium tribromide—An equimolar mixture of triphenyltin bromide and tellurium tetrabromide was stirred for 4 hr in toluene-ether (5:1). The suspended impurities were filtered, and the solution concentrated to 15 ml followed by the addition of petroleum ether (40-60°). Pale yellow crystals thus obtained were filtered, washed, dried and recrystallized from dichloromethane to give phenyltellurium tribromide (yield 80%), m.p. 226° (d) (Found: C, 16·00; H, 1·19; Br, 53·95; Te, 27·95. (C$_6$H$_5$)$_3$TeBr$_3$ requires C, 16·20; H, 1·13; Br, 53·96; Te, 28·71%).

Diphenyltellurium dibromide—An equimolar mixture of triphenyltin bromide and tellurium tetraiodide was stirred for 4 hr in toluene-ether (5:1). The suspended impurities were filtered, and the solution concentrated to 15 ml followed by the addition of petroleum ether (40-60°). A pale yellow precipitate formed which was filtered, washed, dried and recrystallized from dichloromethane to give diphenyltellurium dibromide (yield 92%), m.p. 198-99° (d) (Found: C, 32·10; H, 1·93; Br, 36·73; Te, 27·95. (C$_6$H$_5$)$_2$TeBr$_2$ requires C, 32·63; H, 1·87; Br, 36·20; Te, 28·71%).

Diphenyltellurium diiodide—An equimolar mixture of triphenyltin bromide and tellurium tetraiodide was refluxed in toluene-ether (5:1) for 4 hr. The suspended particles were filtered and the filtrate was concentrated to 15 ml. The solution was treated with petroleum ether (40-60°) (yield 70%). A dark red precipitate was filtered, washed with toluene, dried and recrystallized from dichloromethane to give diphenyltellurium diiodide (yield 20%), m.p. 236° (Found: C, 26·58; H, 1·75; I, 47·22; Te, 23·19. (C$_6$H$_5$)$_2$TeI$_2$ requires C, 26·88; H, 1·87; I, 47·42; Te, 23·83%).

Results and Discussion

Phenyltellurium tribromide and diphenyltellurium dibromide have been prepared by the reaction of tellurium tetrabromide with triphenyltin bromide and tetraphenyltin respectively in accordance with Eqs. (1) and (2).

(C$_6$H$_5$)$_3$SnBr$_3$ + TeBr$_4$ → (C$_6$H$_5$)$_3$SnBr$_3$ + (C$_6$H$_5$)$_2$TeBr$_2$ + SnBr$_3$... (1)

(C$_6$H$_5$)$_2$Sn + TeBr$_4$ → (C$_6$H$_5$)$_2$SnBr$_3$ + (C$_6$H$_5$)$_2$TeBr$_2$... (2)

Corresponding diphenyltellurium diiodide have also been prepared from tetraphenyltin and tellurium...
tetraiodide. However, the yield is poor in this case. A suitable method for the preparation of phenyltellurium triiodide could not be worked out.

During the reactions involving phenyltellurium compounds appreciable quantity of heat is neither evolved nor absorbed but reactions involving phenylselenium compounds are exothermic. The reaction between tetraphenyltin and selenium tetrabromide proceeds even in the solid state and phenylselenium tribromide thus formed decomposes immediately. Since selenium tetrabromide decomposes in any solvent at room temperature, it is added slowly to a suspension of tetraphenyltin in dichloromethane chilled in liquid air. On raising the temperature to the melting point of dichloromethane, the blood red colouration of phenylselenium tribromide is imparted. At room temperature the products decompose immediately to give elemental selenium and pasty products. Ether extract of this product shows the presence of triphenyltin bromide, m.p. 121-22° (lit.4 m.p. 121-22°) suggesting thereby that though the reaction is possible it is difficult to obtain the desired selenium product. Ethyltellurium trichloride and tribromide have been prepared from triethyltin chloride/bromide and tellurium tetrachloride/bromide respectively in accordance with Eq. (3).

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(C_2H_5)_3SnX + TeX_4 \rightarrow (C_2H_5)_2SnX_2 + C_2H_5TeX_3
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(where X=Cl, Br) ...(3)

The reaction between triethyltin iodide or tetraethyltin and tellurium tetraiodide in non-polar solvents like toluene, etc., was unsuccessful, probably due to poor solubility of tellurium tetraiodide in these solvents. In polar solvents like methanol, tellurium tetraiodide dissolves but dissociates to a large extent and the resulting products could not be characterized.

The reactions between tetraethyltin with tellurium tetrachloride and tribromide could be carried out in any solvent but the oily products, diethyltellurium dichloride and dibromide, could not be separated owing to their low melting points (−10° and −12° respectively)11 and high solubility of these along with those of side products, viz. diethyltin dichloride and dibromide.

References