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CLEAVAGE REACTIONS OF THE LEAD–CARBON BOND

IV*. SYNTHESIS OF SOME ARYLTELLURIUM HALIDES USING ORGANOLEAD COMPOUNDS

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SUMMARY

Aryltellurium chlorides have been prepared in excellent yields by the reaction between triaryllead chlorides and tellurium tetrachloride or aryltellurium trichlorides in benzene at room temperature.

INTRODUCTION

The cleavage of lead–carbon bond by a variety of reagents has been shown to provide a suitable route to several organolead salts[1-3]. This paper describes the synthesis of aryltellurium chlorides by treatment of various aryllead compounds with tellurium tetrachloride or aryltellurium trichlorides.

RESULTS AND DISCUSSION

The cleavage of organolead compounds by tellurium halides in hydrocarbon solvents (benzene or toluene) at room temperature can be represented by the following equations:

$$(C_6H_5)_4Pb + TeCl_4 \rightarrow (C_6H_5)_2TeCl_2 + (C_6H_5)_2PbCl_2$$

$$(C_6H_5)_3PbCl + TeCl_4 \xrightarrow{\text{dioxane}} C_6H_5TeCl_3 + (C_6H_5)_2PbCl_2$$

$$(C_6H_5)_3PbCl + RArTeCl_3 \rightarrow Ar(C_6H_5)TeCl_2 + (C_6H_5)_2PbCl_2$$

$$(Ar = C_6H_5 \text{ or } p\text{-}C_2H_5OC_6H_4)$$

$$(C_6H_5)_2Pb(CH_3)_2 + TeCl_4 \rightarrow (C_6H_5)_2TeCl_2 + (CH_3)_2PbCl_2$$

The noteworthy features of the reaction are:

1. The fact that reaction goes to completion at room temperature reduces the possibility of decomposition of the tellurium compounds formed.

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[*] For Parts I–III see refs. 5 and 6.

TABLE 1

M.P's, YIELDS AND ANALYSES OF ORGANOTELLURIUM CHLORIDES

Reactants	*Organotellurium chloride obtained*	*M.p. (°C) found (lit. value)*	*Yield (%)*	*Analytical data (%) found (calcd.)*		
				C	*H*	*Cl*
$(C_6H_5)_4Pb + TeCl_4$	$(C_6H_5)_2TeCl_2$	159 (160)	97	40.58 (40.86)	2.77 (2.86)	19.99 (20.10)
$(p\text{-}CH_3C_6H_4)_4Pb + TeCl_4$	$(p\text{-}CH_3C_6H_4)_2TeCl_2$	165–167 (166–167)	90	44.11 (44.16)	3.69 (3.70)	18.59 (18.62)
$(C_6H_5)_3PbCl + TeCl_4$	$C_6H_5TeCl_3$	214–216 (215–218)	98	23.13 (23.17)	1.63 (1.62)	33.9 (34.19)
$(C_6H_5)_3PbCl + C_6H_5TeCl_3$	$(C_6H_5)_2TeCl_2$	159 (160)	96	40.61 (40.86)	2.81 (2.86)	20.12 (20.10)
$(C_6H_5)_3PbCl + p\text{-}C_2H_5OC_6H_4TeCl_3$	$p\text{-}C_2H_5OC_6H_4TeCl_2C_6H_5$	113–115	90	42.20 (42.38)	3.57 (3.55)	17.78 (17.87)
$(C_6H_5)_2Pb(CH_3)_2 + TeCl_4$	$(C_6H_5)_2TeCl_2$	160 (160)	97	40.69 (40.86)	2.85 (2.86)	20.11 (20.10)

2. The yields of the organotellurium chlorides are excellent, and their isolation presents no problem because of their solubility in the solvent used for the reaction, whereas diorganolead dichloride precipitates out.

3. The procedure can be used for the synthesis of a variety of organotellurium halides because of the wide range of available organolead derivatives.

The reaction between tetraphenyllead and tellurium tetrachloride was monitored by thin-layer chromatography on silica-gel-coated plates using benzene as the solvent and dithizone spray as the developer. The reaction was thus found to proceed in two stages, firstly the exchange of one phenyl group giving phenyltellurium trichloride:

$$(C_6H_5)_4Pb + TeCl_4 \xrightarrow{\text{dioxane}} C_6H_5TeCl_3 + (C_6H_5)_3PbCl$$

and secondly its reaction with triphenyllead chloride to give diphenyltellurium dichloride:

$$(C_6H_5)_3PbCl + C_6H_5TeCl_3 \rightarrow (C_6H_5)_2TeCl_2 + (C_6H_5)_2PbCl_2$$

In a separate experiment, the reaction of triphenyllead chloride with phenyltellurium trichloride has been shown to give diphenyltellurium dichloride. The physical properties and analytical data for various organotellurium chlorides obtained are summarized in Table 1.

EXPERIMENTAL

Organolead derivatives were prepared by conventional methods[4]. Tellurium tetrachloride (BDH) was used without any further purification. The solvent used for the reaction (benzene, toluene or dioxane) was refluxed over sodium for 4 h and fractionated. As similar experimental procedures were followed for all the reactions only a few are described as typical examples.

Reaction between tetraphenyllead and tellurium tetrachloride

A solution of tellurium tetrachloride (3 g in 25 ml of toluene) was added to a stirred suspension of tetraphenyllead (5.2 g) in toluene (30 ml) during 30 min at room temperature. An exothermic reaction occurred with the formation of a white precipitate. The mixture was stirred for an additional 4 h. Filtration followed by evaporation of toluene gave a white powder. Recrystallization from boiling methanol or a mixture of benzene/hexane (10 parts/90 parts) gave pure diphenyltellurium dichloride (3.4 g 97% yield).

Reaction between triphenyllead chloride and tellurium tetrachloride

A mixture of tellurium tetrachloride (3.4 g) and triphenyllead chloride (5.7 g) in dioxane (50 ml) was stirred at room temperature for 4 h. The precipitated diphenyllead dichloride was filtered and the solvent removed under vacuum. The solid obtained was recrystallized from boiling benzene/hexane mixture to give pure phenyltellurium trichloride (3.7 g, 98% yield).

Reaction between triphenyllead chloride and (p-ethoxyphenyl)tellurium trichloride

Triphenyllead chloride (2.4 g) was added in small portions during 20 min to a stirred suspension of (*p*-ethoxyphenyl)tellurium trichloride (1.8 g) in toluene (50 ml) at room temperature. An exothermic reaction occurred. The mixture was stirred for 6 h. Evaporation of toluene from the filtrate gave a white powder, which was recrystallized from boiling methanol (1.8 g, 90% yield).

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