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LETTERS TO THE EDITOR

New Method for Preparing Aroxytetraaryl Derivatives of Antimony

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Aryl derivatives of antimony(V) of the general formula Ar_4SbX [X = Hlg, NO_3, SCN, OSO_2R, OC(O)R] can be prepared by the reaction of pentaarylantimony and a symmetrical antimony(V) derivative Ar_3SbX_2 in an yield of up to 98% [1–4]. Analogos reactions of pentaarylantimony with diaroxytriphenyl derivatives of antimony are unknown. We have found a new method for preparing tetraphenylantimony aroxides by the reaction of pentaphenylantimony with triphenylantimony diaroxides. By this method we prepared phenoxytetraphenyl antimony (**IIIa**) and (4-nitrophenoxy)tetraphenylantimony **IIIb** from pentaarylantimony **I** and triphenylantimony diaroxides **IIa, IIb** in yields of 99% and 93%, respectively.

$$\underset{\textbf{I}}{Ph_5Sb} + \underset{\textbf{IIa, IIb}}{Ph_3Sb(OAr)_2} \longrightarrow \underset{\textbf{IIIa, IIIb}}{2Ph_4SbOAr}$$

$$Ar = C_6H_5\ (\textbf{a}),\ C_6H_4(NO_2)\text{-}4\ (\textbf{b}).$$

The reactions were carried out by heating the starting materials in an aromatic hydrocarbon. The melting points and IR spectra of the tetraphenylantimony aroxides were consistent with those of samples prepared from pentaphenylantimony and the corresponding phenols [5–7]. The starting triphenylantimony diaroxides were obtained by a procedure previously used for the synthesis of triphenylantimony diacetate [8]. Like in the latter synthesis, the oxidative addition of phenol to triphenylstibine successfully occurred in the presence of hydrogen peroxide in an yield of up to 96%.

Diphenoxytriphenylantimony (IIa). To a mixture of 1.50 g of triphenylstibine and 0.80 g of phenol in 15 ml of ether, 0.42 ml of 30% aqueous hydrogen peroxide was added. After 24 h, the colorless crystals that formed were filtered off and dried to obtain 1.51 g (66%) of compound **IIa**, mp 149–150°C. Found, %: C 64.86; H 4.75; Sb 23.35. $C_{30}H_{25}O_2Sb$. Calculated, %: C 66.79; H 4.64, Sb 22.93.

Bis(*p*-nitrophenoxy)triphenylantimony (IIb). To a mixture of 1.00 g of triphenylstibine and 0.79 g of *p*-nitrophenol in 15 ml of ether, 0.28 ml of 30% aqueous hydrogen peroxide was added. After 24 h, yellow crystals formed and were filtered off and dried to obtain 1.72 g (96%) of compound **IIb**, mp 176–177°C. Found, %: C 57.58; H 4.40; Sb 17.82. $C_{30}H_{25}\cdot O_2Sb$. Calculated, %: C 57.23; H 3.64, Sb 19.40.

Phenoxytetraphenylantimony (IIIa). A mixture of 1.00 g of pentaphenylantimony and 1.06 g of diphenoxytriphenylantimony in 10 ml of toluene was heated at 90°C for 2 h. The solvent was removed, and the residue was crystallized from a 1 : 3 toluene–heptane mixture to obtain 2.05 g (99%) of compound **IIIa**, mp 153°C. Published data [6]: mp 152°C.

(*p*-Nitrophenoxy)tetraphenylantimony (IIIb). A mixture of 0.64 g of pentaphenylantimony and 0.79 g of bis(*p*-nitrophenoxy)triphenylantimony in 10 ml of toluene was heated for 2 h at 90°C. The solvent was removed, and the residue was crystallized from a 1 : 3 toluene–heptane mixture to obtain 1.33 g (93%) of compound **IIIb**, mp 148°C. Published data [7]: mp 148°C.

The IR spectra were recorded on a Hitachi-215 spectrometer in Vaseline oil.

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