Communications TO THE EDITOR

Synthesis of Tetrakistriphenylstannoxytitanium

Sir:

In recent years several reports have appeared on the isolation of trialkylsiloxy and triarylsiloxy derivatives of titanium.[1-4] However, tetrakistriphenylstannoxytitanium (I) prepared herein appears to be the first compound containing a tin-oxygen-titanium linkage. This was synthesized by an ester interchange between tetra-*n*-butyl titanate and triphenyltin hydroxide:

$$4(C_6H_5)_3SnOH + (C_4H_9O)_4Ti \longrightarrow [(C_6H_5)_3SnO]_4Ti + 4C_4H_9OH$$

I

A 20% yield was obtained. Side reactions may occur in the following manner:

$$2(C_6H_5)_3SnOH \longrightarrow (C_6H_5)_3SnOSn(C_6H_5)_3 + H_2O$$

$$(C_4H_9O)_4Ti + H_2O \longrightarrow (C_4H_9O)_3TiOH + C_4H_9OH$$

$$2(C_4H_9O)_3TiOH \longrightarrow (C_4H_9O)_3TiOTi(OC_4H_9)_3 + H_2O$$

No attempt was made to increase the yield of final product by azeotropic distillation of the water and alcohol or by purification of the triphenyltin hydroxide.

Tetrakistriphenylstannoxytitanium melts at 215–216° and is quite soluble in benzene. In comparison tetrakistriphenylsiloxytitanium melted[2] at 460–470° and was very slightly soluble in benzene.

Tetra-*n*-butyl titanate was obtained from Matheson, Coleman & Bell and purified by fractional distillation (b.p. 156–157° at 2.5 mm.). Triphenyltin hydroxide (Practical Grade) was obtained from Anderson Chemical Co.

Preparation of tetrakistriphenylstannoxytitanium, I. A 0.0125-mol. sample of triphenyltin hydroxide was added to 100 ml. of hot benzene and filtered; 0.0030 mol. of tetra-*n*-butyl titanate in 50 ml. benzene was added slowly with stirring to the triphenyltin hydroxide solution. A small amount of light brown residue was obtained on filtration. The supernatant liquid was evaporated by use of a Rinco evaporator leaving a light yellow solid residue. Upon recrystallization from benzene and then washing with petroleum ether a white solid was isolated, m.p. 215–216° (hot stage melting point apparatus). *Anal.* Calcd. for $[(C_6H_5)_3SnO]_4Ti$: C, 57.20; H, 4.00; Sn, 31.40; Ti, 3.17. Found: C, 57.43, 57.55; H, 4.25, 4.35; Sn, 31.10; Ti, 2.95, 3.03.

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