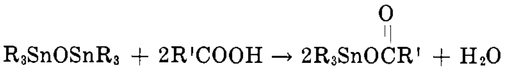
Synthesis of Five New Organotin Esters

JOHN SAMUEL ADAMS, JR.
Dayton Laboratory, Monsanto Research Corp., Dayton, Ohio 45407

Five new organotin esters were prepared and characterized. Per cent yields, melting points, elemental analyses, and infrared data are given for the reported compounds.

UNDER a program for the U. S. Army Biological Laboratories, five new organotin esters have been synthesized in these laboratories. The procedure used is well documented (*1, 2, 3*) in the literature.



$$R_3SnOSnR_3 + 2R'COOH \rightarrow 2R_3SnO\overset{\overset{O}{\|}}{C}R' + H_2O$$

Infrared spectra obtained on the purified products were consistent in all cases, showing aliphatic C—H stretch in the 3.4- to 3.5-μm. region, ester carbonyl absorption (Table I), and consistent aromatic substitution patterns for those containing a benzenoid fragment.

EXPERIMENTAL

All starting materials were obtained from Matheson, Coleman, and Bell and K&K Laboratories as reagent or practical grade, and were used without further purification. Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected. Infrared spectra were obtained on a Perkin-Elmer Model 137 spectrophotometer. Compounds were either examined in a KBr matrix or as a cast film from chloroform.

The reactants were combined in all cases without the use of solvent. In three cases, an immediate exothermic reaction took place and stirring was continued until a solid mass of material was obtained. In the synthesis of tributyltin *p*-iodobenzoate, the reaction mixture was heated with stirring to 95° C., where reaction was evident by an exotherm sustaining this temperature without external heating. In the case of tributyltin α-(2,4,5-trichlorophenoxy)propionate, it was necessary to heat the reactants to 150° C., where reaction was evident by vigorous evolution of water from the reaction mixture. Purification of all five esters was effected by solution in hot *n*-hexane, decolorization with charcoal, and subsequent crystallization in a refrigerator. Table I lists the compounds prepared, together with their melting points, yields, elemental analyses, and characteristic carbonyl absorption wave lengths in the infrared.

ACKNOWLEDGMENT

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LITERATURE CITED

(1) Anderson, H. H., *J. Org. Chem.* **22,** 147 (1957).
(2) Sasin, G. S., *Ibid.*, **18,** 1142 (1953).
(3) Sasin, G. S., Sasin, R., *Ibid.*, **20,** 387 (1955).

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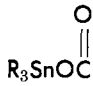
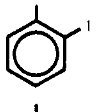
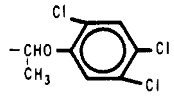
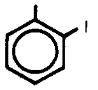
Table I. Characterization of Organotin Esters



$$R_3SnO\overset{\overset{O}{\|}}{C}R'$$

Compound	R	R'	Yield, %	M. P., ° C.	Ester Carbonyl Absorption, mμ	Analysis Calcd.	Analysis Found
Tributyltin γ-chlorobutyrate	$(C_4H_9)_3Sn$-	$-CH_2CH_2CH_2Cl$	54	64–66	5.92	C, 46.7 H, 8.1 Sn, 28.8	C, 46.3 H, 8.0 Sn, 28.5
Tributyltin o°-iodobenzoate	$(C_4H_9)_3Sn$-		37	45.5–46.5	6.3–6.5 (broad)	C, 42.5 H, 5.8 Sn, 22.1	C, 42.7 H, 5.8 Sn, 22.0
Tributyltin ρ-iodobenzoate	$(C_4H_9)_3Sn$-		76	~25	6.15	C, 42.5 H, 5.8 Sn, 22.1	C, 42.4 H, 6.0 Sn, 22.4
Tributyltin α-(2, 4, 5-trichlorophenoxy)-propionate	$(C_4H_9)_3Sn$-		46	65–67	6.3–6.4 (broad)	C, 45.2 H, 6.0 Sn, 21.2	C, 44.9 H, 6.0 Sn, 21.1
Tripropyltin o°-iodobenzoate	$(C_3H_7)_3Sn$-		80	50.5–52	6.4–6.6 (broad)	C, 38.8 H, 5.1 Sn, 24.0	C, 39.2 H, 5.2 Sn, 24.4