

Synthesis of mixed trialkyltin compounds via trialkylstannyl derivatives of sulphomaleic and 4-sulphophthalic anhydrides

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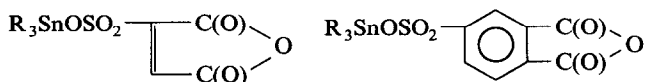
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Abstract

The mixed triorganotin compounds which combine two different trialkyltin moieties (R_3Sn and R'_3Sn) in the same molecule have been synthesized using trialkyltin derivatives of sulphomaleic and 4-sulphophthalic anhydrides as starting materials.

Introduction

Recently [1] we synthesized and characterized the trialkyltin derivatives of sulphomaleic and 4-sulphophthalic anhydrides of the following general formulas:



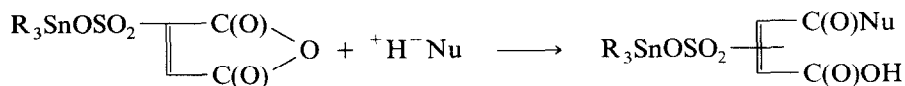
Easily opening under the action of nucleophilic agents, the anhydride cycle in these compounds makes it possible to obtain a large variety of new organotin compounds including mixed ones, i.e. combining R_3Sn - and R'_3Sn moieties in the same molecule. The range of their biological activity is greatly amplified by this process, such functions being combined as, for example, insecticidal and bactericidal (at $R = Me$, $R' = Et$), fungicidal and acaricidal (at $R = Bu$, $R' = \text{cyclo-Hex}$) etc., and attempts to synthesize such compounds have appeared [2].

In the present article, as a continuation of our work on studying the biological activity of organotin compounds and their synthesis [3–8], some reactions of organotin compounds of the above-mentioned general formulas have been studied. Using these reactions as the base, synthesis of mixed organotin compounds combining different trialkyltin groups in molecules of one and the same organic skeleton has been proposed.

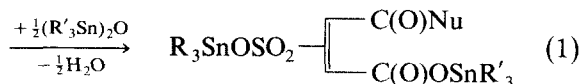
Results and discussion

A five-membered anhydride cycle is readily cleaved even by weak nucleophiles, to give derivatives of dicarboxylic acids. Similarly, a trialkylstannyloxysulphonyl-

maleic anhydride reacts with water, alcohols, ammonia, primary and secondary amines to give the acyclic products I and II:



I (R = Me), II (R = Bu)



III (R = Me), IV (R = Bu)

Nu = OH (a), OMe (b), NH₂ (c), NHPh (d), NEt₂ (e), N(CH₂)₅ (f).

The hydrolysis of trimethylstannyl- and tributylstannyl-oxysulphonylmaieic anhydrides takes place at 100°C with quantitative formation of trimethylstannyl-oxysulphonylmaieic acid (Ia) and tributylstannyl-oxysulphonylmaieic acid (IIa). The availability of two carboxylic groups is proved by quantitative determination of the active hydrogen atom by use of the Chugaev-Tserevetinov reaction.

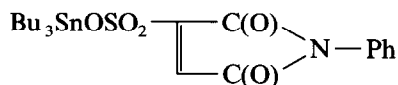
A monomethyl ester of tributylstannyl-oxysulphonylmaieic acid (IIb) has been obtained by alcoholysis of tributylstannyl-oxysulphonylmaieic anhydride in a solution of methanol at 100°C in a sealed vessel. Treatment with tributyltin oxide gave a tributylstannyl salt of the monomethyl ester of tributylstannyl-oxysulphonylmaieic acid (IVb).

In products I and II as well as in their derivatives (III and IV) where there are unlike substituents in carboxylic groups, conditions for isomerism appear that are connected with the possibility of various mutual dispositions of these substituents relative to the sulphonic acid group. It is possible, in our opinion, to form a mixture of isomers in this case.

The reaction of tributylstannyl-oxysulphonylmaieic anhydride with ammonia in acetonitrile under a dry atmosphere with an equimolar ratio of reagents, proceeds by splitting a heterocycle. This consumes half of the ammonia, and the other half neutralizes the resulting monoamide IIc to give its ammonium salt V. If, after adding the whole amount of ammonia, the reaction mixture is treated with tributyltin oxide, then the remaining part of the organotin sulphoanhydride (difficult to separate from the solution) gives the adduct IXc in accordance with equation 2. The yields of products V and IXc are close to the equimolar ratio with a good tin balance (89%).

Reactions with primary (aniline) or secondary (diethyl amine, piperidine) amines also run smoothly, yielding a monoanilide of tributylstannyl-oxysulphonylmaieic acid (IIId), *N,N*-diethyl amide of the trimethylstannyl-oxysulphonylmaieic acid (IIe), tributylstannyl analogue of the last (IIe) and *N,N*-(1,5-pentyl)amide of the tributylstannyl-oxysulphonylmaieic acid (IIIf). The acyclic product IIId, formed in the reaction with aniline, undergoes partial closing of the cycle under conditions of the

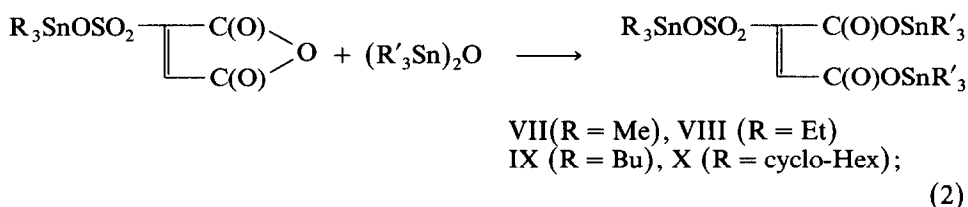
reaction. As a result, *N*-phenyl-(tributylstannyloxysulphonyl)maleimide (VI) of the general formula



separates out.

Compound Ie is very hygroscopic, and we failed to separate a sample that gave a satisfactory elementary analysis. However, the reaction of the compound Ie with a triethyltin oxide permitted its chemical identification and the production of a new mixed organotin compound IIIe ($\text{R}' = \text{Et}$) whose molecule contains both trimethyltin and triethyltin groups.

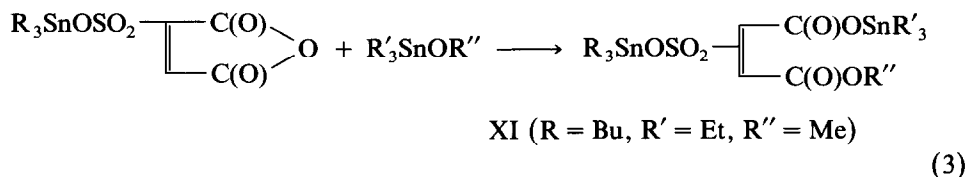
Another route to mixed trialkyltin compounds is the reaction of a trialkylstannyloxysulphonylmaelic anhydride with trialkyl(aryl)tin oxides (hydroxides). This is identical to what we have reported for 4-(trialkylstannyloxysulphonyl)phthalic anhydride [1]. This permits synthesis of an arbitrary combination of trialkyl(aryl)tin groups in sulpho- and carboxylic groups:

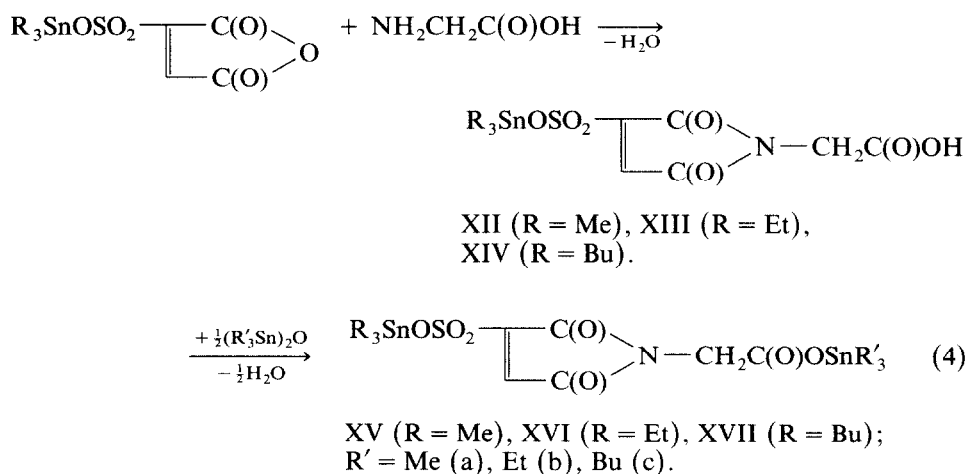


$\text{R}' = \text{Me}$ (a), Et (b), Bu (c), cyclo-Hex (d).

Using reaction 2, we have synthesized the following mixed organotin compounds: bis-trimethyltin (trimethylstannyloxysulphonyl)maleate (VIIa), bis-triethyltin (trimethylstannyloxysulphonyl)maleate (VIIb), bis-tributyltin (trimethylstannyloxysulphonyl)maleate (VIIc), bis-trimethyltin (triethylstannyloxysulphonyl)maleate (VIIIa), bis-triethyltin (triethylstannyloxysulphonyl)maleate (VIIIb), bis-tributyltin (triethylstannyloxysulphonyl)maleate (VIIIc), bis-trimethyltin (tributylstannyloxysulphonyl)maleate (IXa), bis-triethyltin (tributylstannyloxysulphonyl)maleate (IXb), bis-tributyltin (tributylstannyloxysulphonyl)maleate (IXc), bis-tricyclohexyltin (tributylstannyloxysulphonyl)maleate (IXd) and bis-tributyltin (tricyclohexylstannyloxysulphonyl)maleate (Xc).

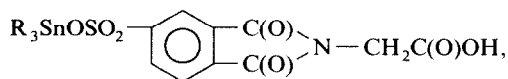
Unlike compound IIIe, the organotin compounds VII–X obtained in accordance with eq. 2 contain three trialkyltin groups, two of which are identical. This can be avoided by using a trialkyltin alkoxide instead of an oxide (eq. 3) or by another way which involves the initial step of an interaction of amino acetic acid and a trialkylstannyloxysulphonylmaelic anhydride and treatment of the obtained product, at the second stage, with a trialkyltin oxide (eq. 4).



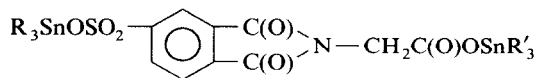


The reaction of cyclizing the organotin sulphonyl anhydride is easier with glycine than with aniline, and the yields of imido-products XII–XIV are very high. The availability of one carboxylic group is proved by the Chugaev–Tserevetinov reaction. In accordance with eq. 4, we have synthesized *N*-[(trimethylstannyloxysulphonyl)maleoyl]amino acetic acid (XII), *N*-[(triethylstannyloxysulphonyl)maleoyl]amino acetic acid (XIII), and *N*-[(tributylstannyloxysulphonyl)maleoyl]amino acetic acid (XIV) as well as mixed organotin compounds containing two different trialkyltin groups in a molecule: tributyltin *N*-[(trimethylstannyloxysulphonyl)maleoyl]amino acetate (XVb), tributyltin *N*-[(trimethylstannyloxysulphonyl)maleoyl]amino acetate (XVc), trimethyltin *N*-[(triethylstannyloxysulphonyl)maleoyl]amino acetate (XVIa) and trimethyltin *N*-[(tributylstannyloxysulphonyl)maleoyl]amino acetate (XVIIa).

Making use of 4-(trialkylstannyloxysulphonyl)phthalic analogues, it is possible, according to eq. 4, to synthesize the following organotin compounds:



XVIII (R = Me), XIX (R = Et), XX (R = Bu);



XXI (R = Me), XXII (R = Et), XXIII (R = Bu);
R' = Me (a), Et (b), Bu (c).

Thus we have obtained *N*-[(4-trimethylstannyloxysulphonyl)phthaloyl]amino acetic acid (XVIII), *N*-[(4-triethylstannyloxysulphonyl)phthaloyl]amino acetic acid (XIX) and *N*-[(4-tributylstannyloxysulphonyl)phthaloyl]amino acetic acid (XX) as well as triethyltin *N*-[(4-trimethylstannyloxysulphonyl)phthaloyl]amino acetate (XXIb), tributyltin *N*-[(4-trimethylstannyloxysulphonyl)phthaloyl]amino acetate (XXIc), trimethyltin *N*-[(4-triethylstannyloxysulphonyl)phthaloyl]amino acetate (XXIIa) and trimethyltin *N*-[(4-tributylstannyloxysulphonyl)phthaloyl]amino acetate (XXIIIa).

Some physico-chemical properties and data of the elemental analysis of all new organotins are presented in Table 1.

Table 1

Yields, melting points and analytical data for the organotin products

Com- pound	Yield (%)	M.p. (°C)	Formula	Anal. (Found (calcd.) (%))				
				C	H	N	S	Sn
Ia	98	95–100 dec.	C ₇ H ₁₂ O ₇ SSn	22.73 (23.42)	4.00 (3.35)		8.92 (8.23)	32.28 (33.09)
IIa	91	viscous	C ₁₆ H ₃₀ O ₇ SSn	38.98 (39.61)	7.00 (6.19)		5.33 (6.60)	24.23 (24.48)
IIb	87	oil	C ₁₇ H ₃₂ O ₇ SSn	41.50 (40.91)	6.97 (6.42)		6.95 (6.42)	24.53 (23.80)
IIc	48	160–165	C ₂₂ H ₃₅ NO ₆ SSn	46.70 (47.17)	6.37 (6.25)	3.00 (2.50)	5.17 (5.72)	20.87 (21.21)
IId	84	viscous	C ₂₀ H ₃₉ NO ₆ SSn	44.34 (44.47)	7.21 (7.22)	2.84 (2.59)	5.42 (5.92)	21.93 (21.99)
IIe	90	65–70	C ₂₁ H ₃₉ NO ₆ SSn	45.48 (45.68)	7.27 (7.07)	2.89 (2.54)	6.16 (5.80)	21.54 (21.51)
IIIe	69		C ₁₇ H ₃₅ NO ₆ SSn	32.04 (32.99)	5.73 (5.66)	2.81 (2.26)	4.87 (5.17)	37.89 (38.39)
IVb	91	viscous	C ₂₉ H ₅₈ O ₇ SSn ₂	45.79 (44.20)	8.07 (7.37)		4.16 (4.06)	30.97 (30.15)
V	43		C ₁₆ H ₃₄ N ₂ O ₆ SSn	38.24 (39.61)	6.58 (7.01)	4.49 (5.78)	7.60 (6.60)	24.34 (24.49)
VI	50	60 dec.	C ₂₂ H ₃₃ NO ₅ SSn	48.07 (48.74)	6.25 (6.09)	2.45 (2.58)	5.64 (5.91)	21.51 (21.91)
VIIa	88	110–115	C ₁₃ H ₂₈ O ₇ SSn ₃	22.77 (22.80)	3.95 (4.09)		4.26 (4.68)	52.32 (52.05)
VIIb	78	156–160	C ₁₉ H ₄₀ O ₇ SSn ₃	29.69 (29.68)	5.55 (5.21)		4.18 (4.17)	47.13 (46.36)
VIIc	90	90–95	C ₃₁ H ₆₄ O ₇ SSn ₃	39.64 (39.73)	7.25 (6.83)		3.26 (3.41)	38.00 (38.04)
VIIIa	88	95–100	C ₁₆ H ₃₄ O ₇ SSn ₃	26.44 (26.44)	4.68 (4.68)		4.49 (4.41)	50.37 (49.04)
VIIIb	84	100–105	C ₂₂ H ₄₆ O ₇ SSn ₃	32.55 (32.59)	5.58 (5.68)		3.71 (3.95)	43.12 (43.96)
VIIIc	95	viscous	C ₃₄ H ₇₀ O ₇ SSn ₃	42.03 (41.71)	7.42 (7.17)		3.05 (3.27)	37.07 (36.40)
IXa	90	115–118	C ₂₂ H ₄₆ O ₇ SSn ₃	32.40 (32.59)	5.70 (5.68)		3.13 (3.95)	43.48 (43.96)
IXb	90	90–95	C ₂₈ H ₅₈ O ₇ SSn ₃	36.99 (37.76)	6.78 (6.48)		3.64 (3.58)	40.10 (39.82)
IXc	91	viscous	C ₄₀ H ₈₂ O ₇ SSn ₃	45.00 (45.19)	7.71 (7.72)		2.82 (3.01)	33.53 (33.52)
IXd	81	55–58	C ₅₂ H ₉₄ O ₇ SSn ₃	50.81 (51.23)	7.75 (7.72)		2.35 (2.63)	30.50 (29.23)
Xc	67	42–45	C ₄₆ H ₈₈ O ₇ SSn ₃	48.10 (48.42)	7.83 (7.72)		2.94 (2.81)	32.00 (31.25)
XI	96	105–107	C ₂₃ H ₄₆ O ₇ SSn	39.02 (39.24)	6.70 (6.54)		4.03 (4.55)	33.72 (33.75)
XII	90	95 dec.	C ₉ H ₁₃ NO ₇ SSn	26.76 (27.16)	3.62 (3.27)	3.47 (3.52)	7.19 (8.05)	28.19 (29.85)
XIII	90	93 dec.	C ₁₂ H ₁₉ NO ₇ SSn	32.14 (32.75)	4.53 (4.32)	3.16 (3.18)	7.04 (7.28)	25.36 (26.99)
XIV	92	95 dec.	C ₁₈ H ₃₁ NO ₇ SSn	40.59 (41.24)	5.63 (5.92)	2.04 (2.67)	6.18 (6.11)	22.31 (22.67)

Table 1 (continued)

Compound	Yield (%)	M.p. (°C)	Formula	Anal. (Found (calcd.) (%))				
				C	H	N	S	Sn
XVb	93	65–67	$C_{15}H_{27}NO_7SSn_2$	30.29 (29.88)	4.98 (4.48)	2.16 (2.32)	4.51 (5.31)	38.18 (39.41)
XVc	82	60–70	$C_{21}H_{39}NO_7SSn_2$	36.62 (36.71)	4.92 (5.68)	1.89 (2.04)	4.93 (4.66)	33.80 (34.59)
XVIa	99	105–110	$C_{15}H_{27}NO_7SSn_2$	29.56 (29.88)	4.17 (4.48)	1.93 (2.32)	5.90 (5.31)	38.90 (39.41)
XVIIa	96	95	$C_{21}H_{39}NO_7SSn_2$	35.39 (36.71)	5.68 (5.68)	1.83 (2.04)	4.36 (4.66)	33.82 (34.59)
XVIII	89	150 dec.	$C_{13}H_{15}NO_7SSn$	33.96 (34.85)	3.39 (3.35)	3.62 (3.13)	6.83 (7.15)	26.43 (26.51)
XIX	90	120 dec.	$C_{16}H_{12}NO_7SSn$	40.08 (39.21)	3.82 (4.29)	3.02 (2.86)	5.26 (6.53)	24.18 (24.24)
XX	93		$C_{22}H_{33}NO_7SSn$	46.88 (46.02)	5.75 (5.75)	2.73 (2.44)	6.09 (5.58)	21.25 (20.69)
XXIb	88	55–65	$C_{19}H_{29}NO_7SSn_2$	35.20 (34.95)	5.01 (4.45)	2.42 (2.15)	4.63 (4.91)	35.65 (36.39)
XXIc	87	wax	$C_{25}H_{41}NO_7SSn_2$	41.34 (40.74)	5.97 (5.57)	2.20 (1.90)	3.95 (4.35)	31.99 (32.24)
XXIIa	92	52–54	$C_{19}H_{29}NO_7SSn_2$	35.62 (34.95)	4.34 (4.45)	2.80 (2.15)	4.04 (4.91)	36.43 (36.39)
XXIIIa	91	52–54	$C_{25}H_{41}NO_7SSn_2$	39.07 (40.74)	5.19 (5.57)	2.46 (1.90)	4.08 (4.35)	33.08 (32.24)

Experimental

The IR spectra of most of these new products have insufficiently good resolution to be informative.

The quantitative determination of active hydrogen atom in compounds Ia, IIa, IIc, VI, XII and XIX was carried out by action of methylmagnesium bromide in diisomyl ether in vacuum sealed ampoules at 100°C. It must be noted that the reaction at room temperature takes place on the surface of the substances only and is accompanied by their sticking together into indestructible grains. The quantity of released methane has been determined from the gas pressure resulting after the ampoule is unsealed in an evacuated system of known volume.

Hydrolysis of trialkylstannyloxysulphonylmaleic anhydride

A solution of trimethylstannyloxysulphonylmaleic anhydride (1.0 g) in 6 ml of water was boiled for 5 h. Then the solvent was removed under reduced pressure to obtain the compound Ia (1.02 g). Found: H_{act} , 0.59, calcd.: H_{act} , 0.56.

Similarly, hydrolysis of tributylstannyloxysulphonylmaleic anhydride (1.0 g) in 10 ml of aqueous alcohol (1:1) at 100°C gives the compound IIa (0.92 g) as a yellowish syrup. Found: H_{act} , 0.44, calcd.: H_{act} , 0.41.

Methanolysis of tributylstannyloxysulphonylmaleic anhydride

A solution of tributylstannyloxysulphonylmaleic anhydride (5.00 g) in 5 ml of absolute methanol was heated in an evacuated sealed ampoule at 100°C for 20 h. Then the solvent was removed under reduced pressure, and a non-volatile residue

was kept under vacuum for 3–5 h at 90°C. The compound IIb (4.65 g) was obtained as light-yellow oil.

To a solution of the compound IIb (4.00 g) in 50 ml of acetonitrile, tributyltin oxide (2.43 g) was added, the reaction mixture was heated at 90°C for 3–5 h, then the azeotrope and the solvent were removed almost completely, the residue was dissolved in 10 ml of alcohol and boiled with activated carbon. The filtrate was evaporated in vacuo at 90°C to dryness and the residue was kept in vacuo for some more hours to remove the solvent completely to give the compound IVb (5.75 g) as light-yellow viscous substance.

Ammonolysis of tributylstannyloxysulphonylmaleic anhydride

To a solution of tributylstannyloxysulphonylmaleic anhydride (2.3 g) in 50 ml of anhydrous acetonitrile cooled to 0°C, an equimolar amount of a solution of ammonia in acetonitrile (29.5 g, 0.165 mol) was added. After 3 h stirring at 0°C tributyltin oxide (1.46 g) was added and stirring continued at room temperature for 3 h. After the mixture was half concentrated, the precipitate was filtered, and washed with cooled acetone (2 × 3 ml) to yield compound V (1.02 g, 43%). The filtrate was evaporated under reduced pressure to yield the viscid compound IXc (2.39 g, 46%). Found: C, 45.17; H, 8.06; S, 2.75; Sn, 32.73. $C_{40}H_{82}O_7SSn_3$ calcd.: C, 45.19; H, 7.72; S, 3.01; Sn, 33.52%.

Aminolysis of trialkylstannyloxysulphonylmaleic anhydride by secondary amines

Diethyl amine (5 ml) was added dropwise under a dry atmosphere to a solution of trimethylstannyloxysulphonylmaleic anhydride (2.83 g) in 5 ml of anhydrous acetonitrile. A weak exothermic effect was observed. After 1 h stirring at room temperature the excess amine and solvent were removed in vacuo and the very hygroscopic residue was treated with a solution of triethyltin oxide (1.46 g) in 40 ml of anhydrous acetonitrile. The mixture was heated at 90°C for 1 h, azeotrope distilled off and solvent removed under reduced pressure at 90°C after which the crude product was boiled in alcohol with activated charcoal. The clear filtrate was evaporated to dryness to give the compound IIIe (2.91 g) as an amorphous hygroscopic light-brown solid with extended melting point.

A solution of diethyl amine (0.25 g) in 10 ml of anhydrous acetonitrile was added slowly to a solution of tributylstannyloxysulphonylmaleic anhydride (1.55 g) in 25 ml of anhydrous acetonitrile at room temperature. The mixture was heated at 55–60°C for 5 h, allowed to cool slowly to room temperature and filtered. The filtrate was evaporated under reduced pressure, the residue was kept in vacuo at 90°C for 3–5 h to give compound IIe (1.5 g) as a yellow syrup.

Similarly, compound IIf (1.64 g) was obtained as an amorphous light-yellow solid starting from tributylstannyloxysulphonylmaleic anhydride (1.54 g) and piperidine (0.293 g).

Reaction of tributylstannyloxysulphonylmaleic anhydride with aniline

A solution of freshly distilled aniline (0.56 g) in 10 ml of anhydrous acetonitrile was added slowly to a solution of tributylstannyloxysulphonylmaleic anhydride (2.81 g) in 30 ml of anhydrous acetonitrile. The mixture was stirred at room temperature for 20 h, the precipitate was separated and washed with acetonitrile (3 × 5 ml) to obtain compound IIId (1.6 g) as a colourless amorphous solid. Found:

$H_{\text{act.}}$ 0.38, calcd.: $H_{\text{act.}}$ 0.36%. The filtrate was evaporated under reduced pressure, the residue was kept in vacuo at 90 °C for 1 h to obtain compound VI (1.74 g) as an amorphous light-brown solid. The Chugaev–Tserevetinov test was negative.

Reaction of trialkylstannyloxysulphonylmaleic anhydride with trialkyltin oxides (hydroxides)

Typically, to a solution of trialkylstannyloxysulphonylmaleic anhydride (5–6 g) in 50 ml of acetonitrile a double molar quantity of trialkyltin hydroxide was added by portions, or the equimolar quantity of trialkyltin oxide dropwise was added dropwise. After 2–3 h stirring at 90 °C the solvent was removed in vacuo to dryness, the residue was purified by boiling in alcohol with activated carbon. Depending on the starting reagents, products VII–X were obtained as colourless amorphous solids, whereas compounds VIIIc and IXc proved to be viscid non-crystallizing substances.

Reaction of tributylstannyloxysulphonylmaleic anhydride with triethyltin methoxide

To a solution of tributylstannyloxysulphonylmaleic anhydride (1.57 g) in 20 ml of acetonitrile, triethyltin methoxide (0.8 g) was added under a dry atmosphere, the mixture was heated at 90 °C for 5 h, the solvent was removed in vacuum to dryness and the residue was evacuated at 90 °C for 3–5 h. The compound XI (2.3 g) was obtained as an amorphous light-yellow solid.

Reaction of trialkylstannyloxysulphonylmaleic and trialkylstannyloxysulphonylphthalic anhydrides with glycine

A test tube containing 2–3 g of trialkylstannyloxysulphonyl-maleic or -phthalic anhydride and the equimolar quantity of glycine was placed in an oil bath at 160 °C. Within 15 min, the temperature of the bath was raised to 175 °C. The released water was removed from the walls of the test tube with blotting-paper. The reaction mixture was treated with 20 ml of hot alcohol and filtered. The filtrate was evaporated in vacuum to dryness, and the residue was kept in vacuum at 90 °C for 2–3 h. Products were the compounds XII–XIV and XVIII–XX as amorphous slightly-coloured solids. For the compound XII: Found, %: $H_{\text{act.}}$ 0.28, calcd.: $H_{\text{act.}}$ 0.25%; for the compound XIX: Found: $H_{\text{act.}}$ 0.24, calcd.: $H_{\text{act.}}$ 0.22%.

Synthesis of mixed organotin compounds containing two different trialkyltin groups

Typically, to a solution of the compounds XII–XIV or XVIII–XX (1–2 g) in 20–30 ml of ethyl alcohol, an equimolar quantity of trialkyltin hydroxide or slightly less than double the quantity of trialkyltin oxide was added. The mixture was heated at 90 °C for 3 h, an azeotrope and the basic quantity of the solvent were distilled off under atmospheric pressure, and the residue was kept under reduced pressure at 90 °C for 2–3 h. Compounds XV–XVII and XXI–XXIII were obtained as amorphous slightly-coloured solids.

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