Journal of Organometallic Chemistry, 144 (1978) 49—59

PREPARATION AND CHARACTERIZATION OF TRIALKYLSTANNYLAMIDOXIMES *

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(Received June 27th, 1977)

Summary

A series of tributyl- (or triphenyl-) stannylamidoximes, $R(H_2N)C{=}NO \cdot SnR'_3$ (where R = Me, Et, Pr or Ph and R′ = Bu or Ph) were prepared by the reaction of tributyl- or triphenyl-tin isopropoxide with amidoximes. Trimethylstannyl derivatives of amidoximes were prepared from the reaction of trimethyltin chloride with amidoximes in the presence of an equimolar amount of sodium or potassium alkoxide. Tributylstannyl amidoximes were prepared from a mixture of bis(tributyltin) oxide and amidoxime in a 1/2 molar ratio.

Introduction

The literature contains only one reference to alkyltin amidoximes , viz. a patent report [1] which described the use of the products of the reactions of alkyl- (or aryl-) tin oxides with amidoximes, $R(H_2N)C{=}NOH$, as stabilizers for polyvinyl chloride. However, the chemistry of alkyltin(IV) derivatives containing ligands with nitrogen and oxygen atoms has been a subject of much interest [2]. In view of the interesting features noted during studies of the alkylsilicon derivatives of amidoximes [3—6] extending the investigations to trialkyltin(IV) compounds seemed worthwhile.

Results and discussion

In the present investigation, some trialkyl- and triphenyl-tin derivatives of amidoximes have been prepared by the reactions of trialkyltin chlorides, trialkyl- and triphenyl-tin isopropoxides and bis(tributyltin) oxide with amidoximes. Reactions of tributyltin chloride, Bu_3SnCl, with amidoximes in equimolar

* Part of this work has been presented at the First International Symposium on Organic Chemistry of Ge, Sn and Pb, Marseille, France, 1974.

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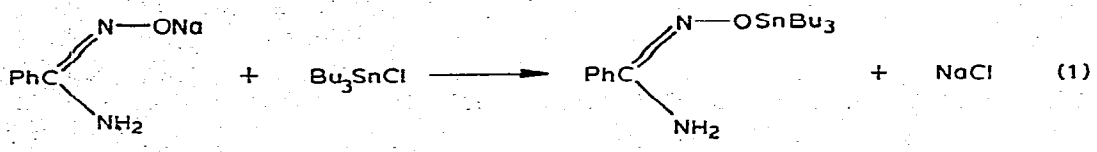
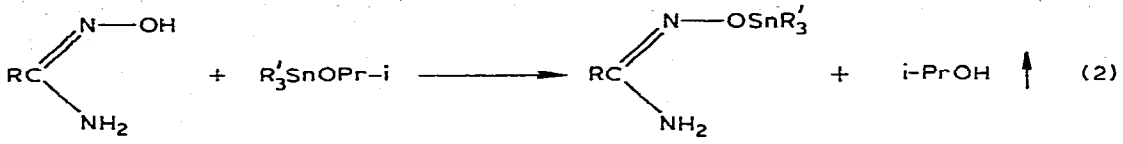
ratio in the presence of triethylamine were very slow, and were not complete even after 20 h refluxing. The sodium salt of benzamidoxime reacted readily with tributyltin chloride at room temperature to give *O*-tributylstannylbenzamidoxime, $Ph(H_2N)C{=}N{-}OSnBu_3$ (eq. 1).



(1)

Triphenyl- and tributyl-stannylamidoximes were mainly synthesized by the reactions of triphenyl- and tributyl-tin isopropoxides with amidoximes in equimolar proportion in refluxing benzene (eq. 2).



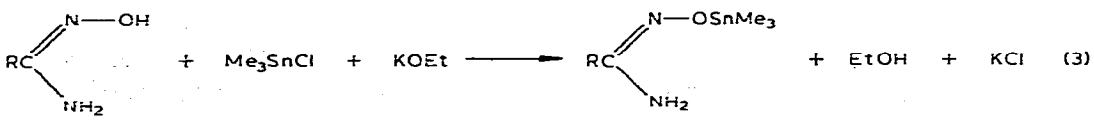
(2)

(I)

(R = Me, Et, Pr or Ph and R′ = Bu or Ph)

The progress of the reactions was followed by estimating isopropanol fractionated out azeotropically with benzene. The reactions are complete within 3—5 h.

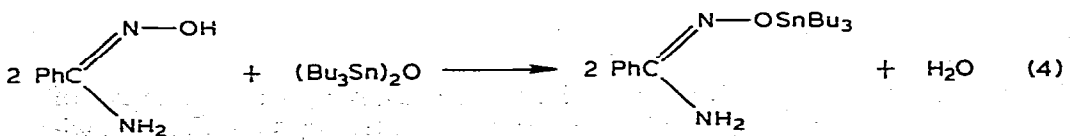
In view of the difficulties in isolating trimethyltin isopropoxide, *O*-trimethyl stannylamidoximes were prepared by treatment of trimethyltin chloride with amidoximes in the presence of an equimolar amount of sodium or potassium alkoxide.



(3)

(R = Me, Et, Pr or Ph)

The product of type I with R = Ph and R′ = Bu was also been prepared by azeotropic removal of water with benzene from a mixture of bis(tributyltin) oxide and benzamidoxide in 1/2 molar ratio (eq. 4). This method offers an



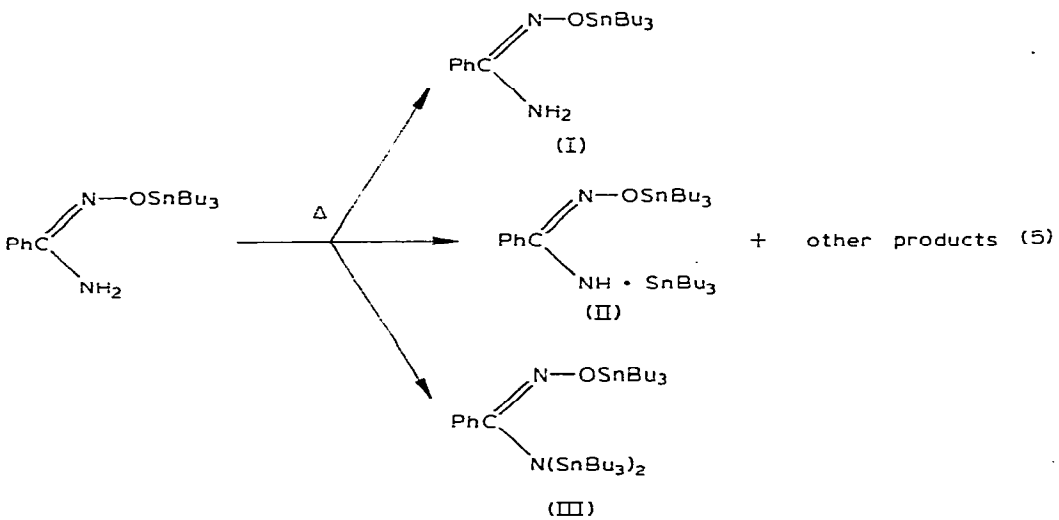
(4)

alternative route for the synthesis of *O*-trialkylstannylamidoximes.

The properties of *O*-trialkylstannylamidoximes are summarized in Table 1. *O*-Triphenylstannylamidoximes are white solids; they are insoluble in most of the common organic solvents such as benzene, chloroform, carbon tetrachloride, n-hexane, and cyclohexane etc., and so their molecular weights could not be determined. They could be sublimed under reduced pressure in poor yields

(≈30—40%). *O*-Trimethylstannylamidoximes are white solids, except for trimethylstannylbenzamidoxime, $Ph(H_2N)C{=}N{-}OSnMe_3$, which is a highly viscous liquid; they could be volatilised under reduced pressure. The molecular weights in refluxing benzene lie between those expected for the dimer and monomer.

Tributylstannylamidoximes, $R(H_2N)C{=}N{-}OSnBu_3$ are colourless liquids, monomeric in refluxing benzene. Except for the derivative of benzamidoxime, they decomposed on attempted distillation under reduced pressure (0.1 mmHg) at a bath temperature of ca. 180°C; the major product of the decomposition was identified as bis(tributyltin)oxide, $(Bu_3Sn)_2O$, and in some cases the nitrile and ammonia were also detected. With tributylstannylbenzamidoxime, $Ph(H_2N)C{=}N{-}OSnBu_3$, however, thermal disproportionation occurred and the fractions obtained by distillation were shown to be mono- (I), bis- (II), and tris(tributylstannyl)benzamidoximes (III) (eq. 5).



Similar thermal disproportionation was observed by Mehrotra et al. [7] in the case of tributyltin aminoalkoxide, $Bu_3SnOCH_2CH_2NH_2$.

Attempted preparations of *N,O*-bis(tributylstannyl)amidoximes, $R(Bu_3SnNH)C{=}N{-}OSnBu_3$, by treatment of tributyltin isopropoxide with amidoximes in 2/1 molar ratio always resulted in mono-products (I) even when the mixtures were refluxed for longer times (ca. 20 h) in benzene. Attempted preparation of bis-compound through the lithium derivative of the mono-product, $R(LiNH)C{=}N{-}OSnBu_3$, was frustrated by cleavage of the Sn—O bond, which led to Bu_4Sn and an unidentified product.

However, *N,O*-bis(tributylstannyl)benzamidoxime (II; R = Ph) was prepared by a transamination reaction of mono(tributylstannyl)benzamidoxime with (diethylamino)tributylstannane.

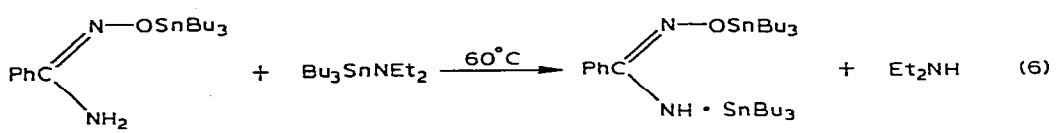
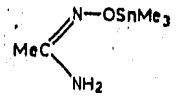
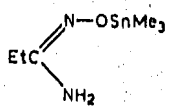
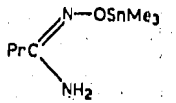
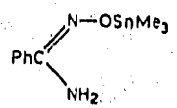
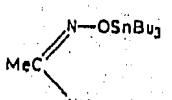
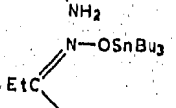
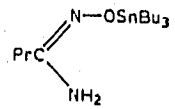
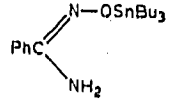
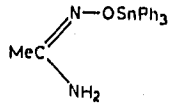
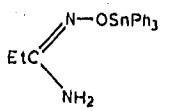
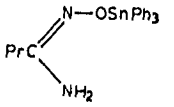
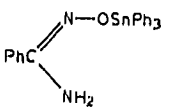
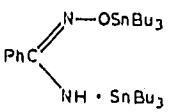


TABLE 1

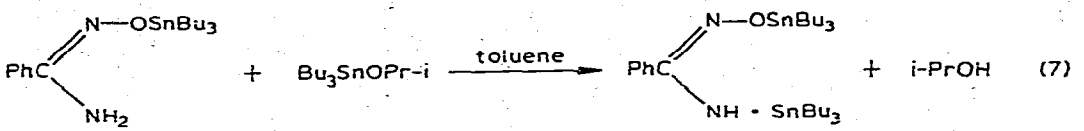
PROPERTIES OF TRIALKYLSTANNYLAMIDOXIMES

Product		B.p. (°C/mmHg) and nature	Mol. wt. Found (Calcd.)	n_D^{35}	IR bands (cm^{-1})
I		67—68/0.1, White crystalline solid	431 (236.8)	—	3460m, 3350s, 1660vs, 1630vs, 1592m, 925s, 770vs, 570—80m(br), 540s, 510w
II		76—78/0.4, colourless liquid, turned to white solid on keeping for ca. 3 days	412 (250.8)	—	3452m, 3310—30m(br), 1659vs, 1626vs, 1590m, 936s, 770vs, 528w, 540s, 512m
III		76—78/0.2, low melting solid	384 (264.9)	— —	3455s, 3300—30s(br), 1660vs, 1625s, 1590m, 935s, 770vs, 580w, 540s, 510m
IV		130—132/0.1, viscous liquid	317 (298.9)	—	3450s, 3300—20s(br), 305m, 1640vs, 1615vs, 1575s, 910vs, 770vs, 695vs, 580w, 540vs, 510m
V		—, colourless liquid [a]	379 (362.8)	1.4975	3460m, 3350m, 1650vs, 1590m, 925m, 690vs, 600m, 540w, 500m
VI		—, colourless liquid [a]	399 (376.8)	1.4910	3440m, 3300m, 3150m(br), 1650vs, 1595m, 928w, 680vs, 600m, 542w, 510m

VII		—, colourless liquid [a]	414 (390.8)	1.3920	3442m, 3303m, 3155m(br), 1649vs, 1597m, 925w, 682vs, 600m, 543w, 512m
VIII		—, colourless viscous liquid	421 (424.8)	1.5310	3450m, 3350m, 2140m(br), 3050w, 1630vs, 1580s, 900vs, 768m, 692vs, 600m, 555s, 505m
IX		s.190—195/0.1 [b] White solid insoluble in benzene	— (423.1)	—	3430m(br), 3350m, 3140w(br), 3050m, 1650m, 1570m, 1072s, 730vs, 700vs, 610w, 555s, 455vs, 445vs
X		s.120—130/0.1 White solid insoluble in benzene	— (437.1)	—	3440m(br), 3350vs, 3150m(br), 3050w, 1650s, 1560m, 1070m, 730vs, 700vs, 608w, 560m, 453s, 444s
XI		s. 140—145/0.1 White solid insoluble in benzene	— (451.1)	—	3440m(br), 3352vs, 3148m(br), 3050w, 1649s, 1562m, 1071m, 730vs, 700vs, 609w, 562m, 452s, 445s
XII		s. 180—190/0.1 White solid insoluble in benzene	— (485.0)	—	3450m, 3350—60m(br), 3145m(br), 3050m, 1640s, 1600m, 1072s, 770s, 730vs, 700vs, 615w, 550s, 455s, 445s
XIII		—, colourless viscous liquid [a]	695 (713.5)	1.5200	3350m, 3050m, 1625vs, 1600m, 1175m, 1072s, 910s, 875vs, 770vs, 695vs, 600m, 560m, 506m

[a] Compounds V—VII, XIII decompose into $(Bu_3Sn)_2O$ at a 180°C bath on attempted distillation under reduced pressure of 0.1 mm. [b] s., sublimation; v = very, s = strong, m = medium, w = weak, (br) = broad.

This bis-product (II) was also obtained by treatment of the mono-product (I) with tributyltin isopropoxide in 1/1 molar ratio in refluxing toluene for ca. 10 h.



(7)

The bis-product is a light yellowish liquid, monomeric in refluxing benzene. On attempted distillation at 0.1 mmHg and at a bath temperature of 180°C it decomposed to $(Bu_3Sn)_2O$ and other, unidentified, products.

Infrared spectra

For the mono(trialkylstannyl)amidoximes (I), the absence of the ν(OH) absorption band in the region 3570—3650 cm^{-1} (present in the parent amidoxime [8] suggests that the products are *O*-substituted. The two bands in the regions 3430—3460 and 3300—3360 cm^{-1} in the spectra of products I are due to $\nu(NH_2)$ [8] the deformation band of which appears at ca. 1580 cm^{-1}. Furthermore, in the bis-products, $Ph(Bu_3SnNH)C{=}N{-}OSnBu_3$, a single band in the region 3310—3350 cm^{-1} is obtained due to ν(>NH). A medium and slightly broader band in the region 3130—3160 cm^{-1} is probably due to hydrogen bonded >NH. In phenyl-containing products the aromatic ν(C—H) vibrations are observed at ca. 3050 cm^{-1}.

A strong band shown by the monomeric compounds, $R(H_2N)C{=}N{-}OSnR'_3$ (R′ = Bu or Ph), in the region 1630—1650 cm^{-1} is assigned to ν(C=N) of the oxime moiety. *O*-Trimethylstannylamidoximes (I, R′ = Me), which probably exist as an equilibrium mixture of monomer and dimer species, exhibit two bands assignable to ν(C=N) in the regions 1630—1660 and 1615—1630 cm^{-1}. The ν(N—O) stretching vibrations appear at 920 ± 10 cm^{-1} [9].

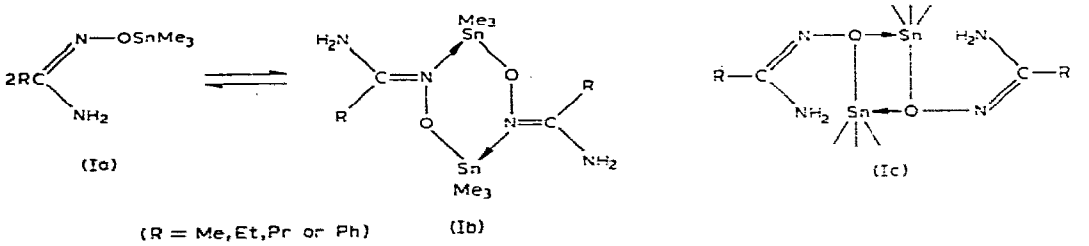
In trimethylstannylamidoximes the strong band at ca. 770 cm^{-1} is assigned to the methyltin rocking mode [10,11]. In triphenylstannylamidoximes a band appears at ca. 450 cm^{-1}; this was absent in the case of the trialkyltin benzamidoxime, and can be assigned to ν_{as}(Sn—Ph) [12,13]. This band has been interpreted by Pollen [14] as to be due to the phenyl ring vibration. The trialkylstannyl products show two bands in the lower region of the infrared spectra, the ν_{as}(Sn—C) frequency appears in the range 540—600 cm^{-1}, and the second band, at 500 ± 10 cm^{-1}, is due to ν_s(Sn—C) vibrations [15].

With *O*-trialkylstannylamidoximes, the ν(Sn—O) absorption bands [16] appear at 550 ± 10 cm^{-1}. Sometimes this band is associated with the ν(Sn—C) bands and cannot be distinguished from it.

The ν(Sn—N) absorption bands are reported to occur over a wide range of frequencies, and appear to be very sensitive to changes in the molecular environments of the Sn—N group. Recently, for $(Me_3Sn)_3N$ the $\nu_{as}(NSn_3)$ and $\nu_s(NSn_3)$ bands appear at 672 and 514 cm^{-1}, respectively [12]. The ν(Sn—N) stretching frequencies in *N*-trimethylstannylaniline appears at 843 cm^{-1} [17]. In (diethylamino)trialkylstannane, the vibrations due to $Sn{-}NEt_2$ group [18] appear at 1170s, 1010s, 880vs and 780vs cm^{-1}. In *N*,*O*-bis(tributylstannyl)benzamidoxime,

a strong band is observed at ca. 875 cm^{-1}, and may be assigned to ν(Sn—N).

The IR spectra of trimethylstannylamidoximes, which show molecular weights between the dimer and the monomer, show no appreciable change in the position of $\nu(NH_2)$ in comparison to parent amidoximes [3] indicating that amine nitrogen is not involved in coordination. The doublet in the regions 1630—1660 and 1615—1630 cm^{-1} due to ν(C=N) suggests the equilibrium [9] Ia ⇌ Ib, with the two bands corresponding to free and bridging C=N moieties.



On the basis of the evidence available, the possibility that the dimers have structures of type Ic cannot be eliminated.

Experimental

Moisture was rigorously excluded. Benzene was dried over sodium wire and azeotropically fractionated. Amidoximes were prepared by standard methods [19,20] and analyzed before use. Trimethyltin chloride (b.p. 152—154°C), tributyltin chloride (97°C/0.4 mmHg) and bis(tributyltin)oxide (157°C/0.05 mmHg) were distilled before use. Tributyltin isopropoxide (100°C/1 mmHg) was prepared from tributyltin chloride. $Bu_3Sn—NEt_2$ was prepared from Bu_3SnCl and Et_2NH in the presence of lithium and naphthalene in n-hexane under dry nitrogen [21].

The IR spectra were recorded neat or in nujol mulls using KBr optics (Perkin—Elmer 337) in the range 4000—400 cm^{-1}. An ebulliometer (Gallenkamp) was used for molecular weight determination.

Tin was estimated as oxide and nitrogen by the Kjeldahl method. Alcohol in the azeotrope was estimated by oxidation [22]. C and H analyses were carried out by CSIRO (Australia).

Preparation of Ph_3SnOPr-i

A solution of triphenyltin chloride (19.28 g) in benzene (120 ml) was added dropwise to sodium isopropoxide (5% excess than equimolar quantity) in benzene (90 ml). This mixture was refluxed for ca. 6 h, and the sodium chloride was then filtered off. The solvent was removed under reduced pressure and the residue was distilled (b.p. 172—175°C/0.1 mmHg) as a white solid (m.p. 68—69°C) in 85% yield. Found: Sn, 29.04. Ph_3SnOPr-i calcd.: 29.01%.

Reactions of R'_3SnOPr-i with amidoximes in 1/1 molar ratio in benzene

A mixture of the tributyl- and triphenyl-tin isopropoxides with the amidoxime (ca. 55 ml) was refluxed at bath temperatures of 115—120°C for about 6 h, and the binary azeotrope (benzene/isopropanol) was fractionated out slowly. The excess of the solvent was distilled off and the products, $R(H_2N)C=NOSnR'_3$,

TABLE 2

REACTIONS OF $R'_3SnOPr\text{-}i$ WITH AMIDOXIMES IN 1/1 MOLAR RATIOS

	Reactants (g)		Refluxed for (h)	Product [a]	Isopropanol in azeotrope (g)	Analysis (Found (calcd.) (%))			
	Alkoxide	Amidoxime $R(NH_2)C=NOH$		B.p. (°C/mmHg) (Yield, %)		Sn	N	C	H
I	$Bu_3SnOPr\text{-}i$	R = Me							
	4.98	1.06	8	$Me(NH_2)C=N-OSnBu_3$	0.82	32.67	7.65	47.53	8.62
				d. 180/0.1 (98)	(0.86)	(32.72)	(7.72)	(46.40)	(8.80)
		R = Et							
II	6.07	1.53	6	$Et(NH_2)C=N-OSnBu_3$	1.00	31.46	7.42	—	—
				d. 180/0.1 (97)	(1.05)	(31.51)	(7.44)	—	—
		R = Pr							
III	5.80	1.70	6	$Pr(NH_2)C=N-OSnBu_3$	0.97	30.25	7.13	—	—
				d. 180/0.1 (97)	(1.00)	(30.38)	(7.17)	—	—
		R = Ph							
IV	4.68	1.83	6	$Ph(NH_2)C=N-OSnBu_3$	0.77	27.50	6.42	54.10	8.20
				(96)	(0.80)	(27.95)	(6.60)	(53.75)	(8.07)
	$Ph_3SnOPr\text{-}i$	R = Me							
V	4.48	0.81	5.5	$Me(NH_2)C=N-OSnPh_3$	0.59	28.11	6.64	—	—
				s. 190—195/0.1 (36)	(0.66)	(28.06)	(6.62)	—	—
		R = Et							
VI	2.01	0.63	5	$Et(NH_2)C=N-OSnPh_3$	0.42	27.05	6.40	—	—
				s. 125—130/0.1 (35)	(0.43)	(27.16)	(6.41)	—	—
		R = Pr							
VII	3.81	0.95	5	$Pr(NH_2)C=N-OSnPh_3$	0.54	26.42	6.16	—	—
				s. 140—145/0.1 (30)	(0.56)	(26.31)	(6.21)	—	—
		R = Ph							
VIII	3.50	1.17	6	$Ph(NH_2)C=N-OSnPh_3$	0.49	24.71	5.55	—	—
				s. 180—190/0.1 (30)	(0.51)	(24.47)	(5.77)	—	—

[a] s. = Sublimation, d. = decomposition.

were dried at 0.5 mmHg and at 40—55°C. Details of the reactions are given in Table 2.

When tributyltin derivatives of amidoximes other than benzamidoxime were vacuum distilled, they decomposed at a bath temperature of 180°C and a colourless liquid distilled out (b.p. 155—158°C/0.1 mmHg). Found: Sn, 39.46. $(Bu_3Sn)_2O$ calcd.: Sn, 39.88%.

When tributyltin derivative of benzamidoxime was vacuum distilled, three fractions were obtained:

Fraction 1: Colourless liquid (yield 20%; b.p. 115—120°C/0.2 mmHg). Found: Sn, 27.82; N, 6.62. $Ph(H_2N)C{=}NOSnBu_3$ calcd.: Sn, 27.95; N, 6.60%.

Fraction 2: Colourless liquid (yield 25%; b.p. 125—128°C/0.2 mmHg). Found: Sn, 33.45; N, 3.96. $Ph[H(Bu_3Sn)]C{=}NOSnBu_3$ calcd.: Sn, 33.27; N, 3.92%.

Fraction 3: Yellowish coloured liquid (yield 45%; b.p. 155—157°C/0.2 mmHg). Found: Sn, 35.51; N, 2.85. $Ph[(Bu_3Sn)_2]C{=}NOSnBu_3$ calcd.: Sn, 35.53; N, 2.80%.

Preparations of O-trimethylstannylamidoximes

Potassium ethoxide was prepared by dissolving potassium in an excess of ethanol and removal of surplus under reduced pressure. The solid ethoxide was dissolved in benzene and the solution was added to a solution of trimethyltin chloride and amidoxime in benzene. The system was refluxed for about 4 h and KCl was filtered off. The product, $R(H_2N)C{=}N{-}OSnMe_3$ was obtained after removing the solvent under reduced pressure 35°C/2—4 mmHg. Details are given in Table 3.

Reaction of sodium salt of benzamidoxime with tributyltin chloride in 1/1 molar ratio

An exothermic reaction occurred when Bu_3SnCl (1.79 g) was added to a suspension of sodium salt of benzamidoxime (0.87 g) in benzene (ca. 30 ml). The mixture was refluxed for 2 h and sodium chloride was filtered off. The solvent was removed under reduced pressure and the colourless viscous product was dried at ca. 40°C/0.5 mmHg for ca. 1 h (yield 2.30 g, 98%). Found: Sn, 27.81; N, 6.55. $Ph(H_2N)C{=}NOSnBu_3$ calcd.: Sn, 27.95; N, 6.60%.

Reaction between bis(tributyltin)oxide and benzamidoxime in 1/2 molar ratio

$(Bu_3Sn)_2O$ (3.23 g) and benzamidoxime (1.48 g) were mixed in benzene (ca. 60 ml). The mixture was refluxed for ca. 6 h and the water liberated was fractionated out azeotropically. Remaining benzene was distilled off, and the colourless viscous product was finally dried at ca. 40°C/0.6 mmHg for ca. 1/2 h (yield 4.60 g, 99%). Found: Sn, 27.82; N, 6.50. $Ph(H_2N)C{=}NOSnBu_3$ calcd.: Sn, 27.95; N, 6.60%.

Attempted preparation of N,O-bis(tributylstannyl)benzamidoxime by the reaction of mono(tributylstannyl)benzamidoxime with Bu_3SnCl (butyllithium method)

Butyllithium was prepared from lithium (0.11 g) and BuCl (0.75 g) in n-hexane (ca. 30 ml) under dry nitrogen while stirring the mixture at ca. 30°C for ca.

TABLE 3

PREPARATIONS OF *O*-TRIMETHYLSTANNYLAMIDOXIMES

	Reactants (g)			Refluxed for (h)	Product	Analysis (Found (calcd.) (%))			
	Me_3SnCl	K	$R(H_2N)C{=}NOH$		B.p. (°C/mmHg) (Yield, %)	Sn	N	C	H
			R = Me		$Me(NH_2)C{=}N{-}OSnMe_3$	49.60	11.68	16.95	3.83
I	2.80	0.59	1.04	6	67–68/0.1 (75)	(50.13)	(11.83)	(16.54)	(3.86)
			R = Et		$Et(NH_2)C{=}N{-}OSnMe_3$	47.20	11.10	—	—
II	2.70	0.53	1.20	4.5	76–78/0.4 (67)	(47.33)	(11.19)	—	—
			R = Pr		$Pr(NH_2)C{=}N{-}OSnMe_3$	44.64	10.52	21.85	4.75
III	1.46	0.29	0.75	4	76–78/0.2 (70)	(44.81)	(10.58)	(21.49)	(4.61)
			R = Ph		$Ph(NH_2)C{=}N{-}OSnMe_3$	39.59	9.26	—	—
IV	2.55	0.51	1.75	5	130–132/0.1 (40)	(39.79)	(9.37)	—	—

12 h. Mono(tributylstannyl)benzamidoxime (3.44 g) was added, and the mixture was stirred for another 3 h. Addition of Bu_3SnCl (2.63 g) was followed by stirring for ca. 2 h. LiCl was filtered off and the solvent was removed. Distillation of the residue gave a colourless liquid at 98°C/0.1 mmHg. Found: Sn, 34.10. Bu_4Sn calcd.: Sn, 34.20%.

Preparation of N,O-bis(tributylstannyl)benzamidoxime from the reaction of mono(tributylstannyl)benzamidoxime with $Bu_3Sn—NEt_2$

(Diethylamino)tributylstannane (1.10 g) was added to *O*-tributylstannylbenzamidoxime (1.30 g). The reaction was exothermic. The mixture was dried at 60—65°C/0.2 mmHg for ca. 1 h to give a colourless liquid (yield 2.17 g, 99%). Found: Sn, 32.86; N, 3.94. $PH[H(Bu_3Sn)N]C{=}NOSnBu_3$ calcd.: Sn, 33.27; N, 3.92%.

Reaction of O-tributylstannylbenzamidoxime with Bu_3SnOPr-i in 1/1 molar ratio in toluene

O-Tributylstannylbenzamidoxime (2.18 g) and Bu_3SnOPr-i (1.79 g) were mixed in toluene (ca. 50 ml). The mixture was refluxed for ca. 10 h and isopropanol liberated was fractionated azeotropically out with toluene. The solvent was removed under reduced pressure and the product was dried for ca. 1 h at 50°C/0.2 mmHg (yield 3.60 g, 98%). Found: isopropanol in the azeotrope, 0.29 g; calcd.: for one mole, 0.31 g. Found: Sn, 33.35; N, 3.67. $Ph[H(Bu_3Sn)N]C{=}NOSnBu_3$ calcd.: Sn, 33.27; N, 3.92%.

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