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# SYNTHESIS, REACTIONS AND SPECTROSCOPIC PROPERTIES OF SOME DIORGANOTIN OXIMATES

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## Summary

Several tin derivatives of the types, $R_2Sn(OR^1)(ON{=}CR^2R^3)$ and $R_2Sn(ON{=}CR^2R^3)_2$ have been synthesized and a study made of their spectra and reactions.

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## Introduction

Trialkyltin oximates have been studied [1—4] extensively, but there have been only two reports [5,6] on diorganotin dioximates. We describe below the first systematic studies on diorganotin dioximates with a view, (i) to establishing the influence of the nature of organic groups attached to the imino carbon atom on the degree of the association of the oximate derivatives, (ii) to comparing the strength of $\geqslant Sn{-}O$ bond in $\geqslant Sn{-}O{-}R$ with that in $\geqslant Sn{-}O{-}N{=}C\leqslant$, and (iii) making a comparative study of the reactivity of multiple bonded acceptors towards dibutyltin diacetoximate.

## Results and discussion

The following reactions were used for the synthesis of diorganotin oximates:

$$Bu_2SnO + 2\ HON{=}CMe_2 \xrightarrow[10-12\ h]{benzene,\ reflux} Bu_2Sn(ON{=}CMe_2)_2 + H_2O \qquad (1)$$

$$Me_2SnCl_2 + 2\ NaOEt + 2\ HON{=}CR^2R^3 \xrightarrow[8-10\ h]{benzene,\ reflux}$$

$$Me_2Sn(ON{=}CMe_2)_2 + 2\ NaCl + 2\ EtOH \qquad (2)$$

($R^2$, $R^3$ = Me, Me; Me, Ph)

$$R_2Sn(OR^1)_2 + n\ HON{=}CR^2R^3 \xrightarrow[5-7\ h]{benzene,\ reflux}$$

$$Bu_2Sn(OR^1)_{2-n}(ON{=}CR^2R^3)_n + n\ R^1OH \qquad (3)$$

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TABLE 1

PMR SPECTRAL DATA FOR SOME DIORGANOTIN OXIMATES

| Compound | Chemical shifts[a] (τ, ppm) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $\mathrm{Sn{-}CH_3}$ | $\mathrm{Sn{-}C_2H_5}$ | $\mathrm{Sn{-}C_4H_9}$ | $\mathrm{N{=}C}\diagdown^{\diagup \mathrm{CH_3}}$ | $\mathrm{-N{=}C}\diagdown^{\diagup \mathrm{CH_2\underline{CH_3}}}$ | $\mathrm{-N{=}C}\diagdown^{\diagup \mathrm{(CH_2)_n}}$ | $\mathrm{-N{=}C}\diagdown^{\diagup \mathrm{H}}$ | $\mathrm{-N{=}C}\diagdown^{\diagup \mathrm{C_6H_5}}$ |
| $\mathrm{Me_2Sn(ON{=}CMe_2)_2}$ | 9.25s | — | — | 8.17s | — | — | — | — |
| $\mathrm{Me_2Sn(ON{=}CMePh)_2}$ | 9.47s | — | — | 7.86s | — | — | — | 2.67—2.33m [b] |
| $\mathrm{Et_2Sn(ON{=}CMe_2)_2}$ | — | 8.70s(br) | — | 7.98s | — | — | — | 2.33—2.00m [c] |
| $\mathrm{Et_2Sn(OEt)(ON{=}CMe_2)}$ [d] | — | 8.54s(br) | — | 8.10s | — | — | — | — |
| $\mathrm{Et_2Sn(ON{=}OMeEt)_2}$ | — | 8.70s(br) | — | 8.17s | 8.90t [e] | 8.36—7.10m | — | — |
| $\mathrm{Et_2Sn(ON{=}OMePh)_2}$ | — | 8.67s(br) | — | 7.73s | — | — | — | 3.00—2.60m [b]<br>2.60—2.10m [c] |
| $\mathrm{Bu_2Sn(OH{=}CMeH)_2}$ | — | — | 9.30—8.30m | 8.22d [f] | — | — | 3.50—3.00q [g]<br>2.90—2.40q [h] | — |
| $\mathrm{Bu_2Sn(ON{=}CMe_2)_2}$ | — | — | 9.33—8.18m | 8.04s | — | — | — | — |
| $\mathrm{Bu_2Sn(ON{=}CMeEt)_2}$ | — | — | 9.70—8.27m | 8.20s | 8.80t [e] | 8.05—7.50m | — | — |
| $\mathrm{Bu_2Sn(ON{=}CMePh)_2}$ | — | — | 9.37—8.21m | 7.78s | — | — | — | 2.87—2.48m [b]<br>2.48—2.12m [c] |

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[a] Downfield TMS; s, singlet, d, doublet; t, triplet; q, quartet; m, multiplet; (br) broad. [b] *meta*- and *praa*-Phenyl protons. [c] *ortho*-Phenyl protons. [d] Ethoxy group protons at 8.17 (t, *J* 7 Hz, $\mathrm{OCH_2\underline{CH_3}}$) and 5.98 (q, *J* 8 Hz, $\mathrm{\underline{OCH_2}CH_3}$). [e] *J* 7 Hz. [f] *J* 6 Hz. [g] *J* 6 Hz, *anti*-azomethine proton. [h] *J* 6 Hz, *syn*-azomethine proton.

## Properties

The diorganotin dioximates (Table 2) and diorganotin (mono)oximate products (Table 3) are generally colourless (sometimes with reddish or greenish tinge) distillable liquids or sublimable solids. All these new compounds are soluble in common organic solvents and are highly susceptible to hydrolysis. Most of them are monomeric in benzene except for $R_2Sn(OEt)(ON{=}CR^2R^3)$ and $R_2Sn(ON{=}CHMe)_2)$, which are partly associated (molecular complexity: 1.2—1.4).

## Chemical reactions

The diorganotin diacetoximate were found to undergo co-disproportionation with dibutyltin dichloride readily:

$$Bu_2Sn(ON{=}CMe_2)_2 + Bu_2SnCl_2 \xrightarrow[4\ h]{benzene,\ 110^\circ C} 2\ Bu_2Sn(Cl)(ON{=}CMe_2) \qquad (4)$$

The oximate group can be replaced readily by a number of reagents as

TABLE 2

SOME DIORGANOTIN DIOXIMATES

| Compound[a] (yield (%))[d] | B.p. (°C/mmHg) | $n_D^{26}$ | Analysis (Found (calcd.) (%)) | | Mol. wt. (Found (calcd.)) |
|---|---|---|---|---|---|
| | | | Sn | N | |
| $Me_2Sn(ON{=}CMe_2)_2$[b]<br>(90) | 90/10[c] | — | 40.50<br>(40.51) | 9.48<br>(9.56) | 298<br>(293) |
| $Me_2Sn(ON{=}CMePh)_2$<br>(88) | 110/1.5 | — | 28.43<br>(28.46) | 6.64<br>(6.71) | 422<br>(417) |
| $Et_2Sn(ON{=}CHMe)_2$[b]<br>(83) | 90/6[c] | — | 40.48<br>(40.52) | 9.48<br>(9.56) | 390<br>(293) |
| $Et_2Sn(ON{=}CMe_2)_2$<br>(82) | 86/0.4 | 1.4858 | 36.93<br>(36.98) | 8.64<br>(8.73) | 326<br>(321) |
| $Et_2Sn(ON{=}CMeEt)_2$<br>(75) | 124/0.4 | 1.4930 | 33.96<br>(34.00) | 7.90<br>(8.03) | 352<br>(349) |
| $Et_2Sn(ON{=}CMePh)_2$<br>(65) | 128/0.2 | 1.5520 | 26.63<br>(26.66) | 6.18<br>(6.29) | 440<br>(445) |
| $Bu_2Sn(ON{=}CHMe)_2$<br>(72) | 102/0.2 | 1.4845 | 33.96<br>(34.00) | 7.96<br>(8.03) | 408<br>(349) |
| $Bu_2Sn(ON{=}CMe_2)_2$<br>(78) | 108/0.2 | 1.4738 | 31.40<br>(31.47) | 7.35<br>(7.43) | 380<br>(377) |
| $Bu_2Sn(ON{=}CMeEt)_2$<br>(72) | 148/1.5 | 1.4762 | 29.27<br>(29.29) | 6.85<br>(6.92) | 410<br>(405) |
| $Bu_2Sn(ON{=}CHPh)_2$<br>(68) | 169/0.7 | 1.5250 | 25.06<br>(25.08) | 5.87<br>(5.92) | 476<br>(473) |
| $Bu_2Sn(ON{=}CMePh)_2$<br>(70) | 191/2.5 | 1.5220 | 23.66<br>(23.68) | 5.49<br>(5.59) | 506<br>(501) |
| $Bu_2Sn[ON{=}C(Me)CH_2Cl]_2$<br>(58) | 110/0.3 | — | 26.58<br>(26.61) | 6.20<br>(6.28) | 449<br>(446) |
| $Bu_2Sn[ON{=}C(Me)C_6H_4Cl]_2$<br>(56) | 164/0.3 | 1.5478 | 20.80<br>(20.82) | 4.82<br>(4.91) | 574<br>(570) |
| $Bu_2Sn[ON{=}C(Me)C_6H_4Br]_2$<br>(60) | 182/0.5 | 1.5430 | 17.95<br>(18.01) | 4.18<br>(4.25) | 653<br>(659) |

[a] All compounds are liquids unless otherwise indicated. [b] White solids. [c] Sublimation temperature. [d] Yields refere to distilled or sublimed products.

depicted below:

$$\mathrm{Bu_2Sn(ON{=}CMe_2)_2 + Hacac \xrightarrow[6\ h]{neat,\ 86^\circ C} Bu_2Sn(ON{=}CMe_2)(acac) + HON{=}CMe_2} \tag{5}$$

$$\mathrm{Bu_2Sn(OEt)(ON{=}CMe_2) + HSPh \xrightarrow[7\ h]{CCl_4,\ 110^\circ C} Bu_2Sn(OEt)(SPh) + HON{=}CMe_2} \tag{6}$$

$$\mathrm{Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CMe_2) + (MeCO)_2O \xrightarrow[12\ h]{CCl_4,\ 100^\circ C} Bu_2Sn(O\text{-}i\text{-}Pr)(OCOMe) + MeCOON{=}CMe_2} \tag{7}$$

Reactions 6 and 7 indicate that the iminoxy group is replaced more readily than the alkoxy group.

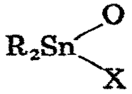
The tin—oxygen bond of the compound $\mathrm{Bu_2Sn(ON{=}CMe_2)_2}$ undergoes addition reactions with suitable multiply bonded acceptor molecules (A=B):

$$\mathrm{Bu_2Sn(ON{=}CMe_2)_2} + n\ \mathrm{A{=}B} \xrightarrow{\text{neat, room temp.}} \mathrm{Bu_2Sn(ABON{=}CMe_2)}_n\mathrm{(ON{=}CMe_2)}_{2-n} \tag{8}$$

(A = B = $\mathrm{CCl_3CHO}$, PhNCO, NpNCO; $n$ = 1 or 2)

The reactivity of the multiply-bonded electrophiles decreases in the order $\mathrm{CCl_3CHO}$ > PhNCO > NpNCO > PhNCS, which parallels the order reported by earlier workers [7].

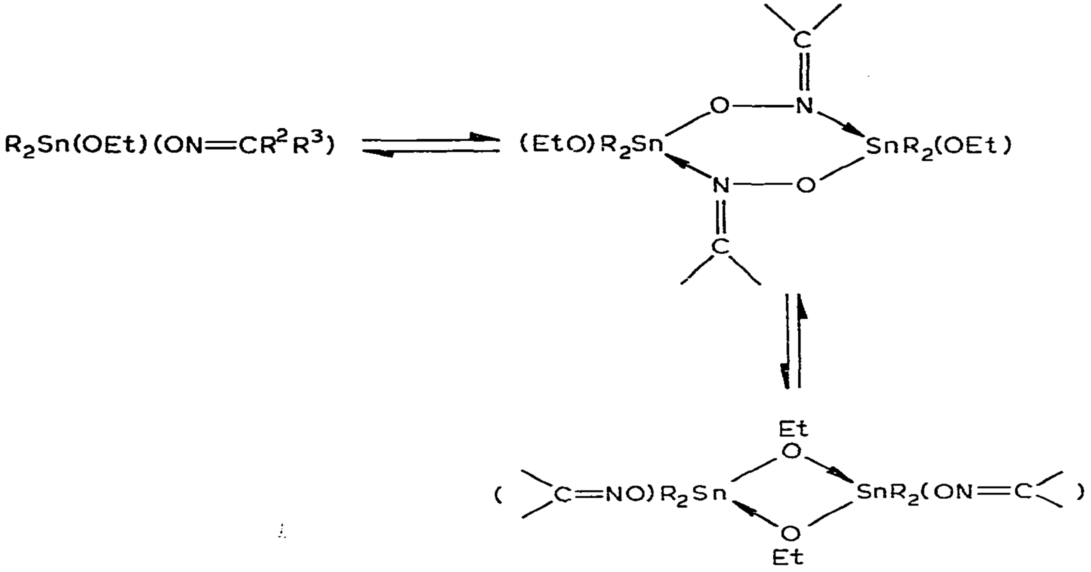
## Infrared spectra

The assignments in the spectra of diorganotin dioximates and (mono)oximate products $\mathrm{R_2Sn(X)(ON{=}CR^2R^3)}$ (where X = $\mathrm{OR^1}$, Cl, MeCOO or SPh), can be made by dividing the molecule into separate parts, i.e., the  $\mathrm{R_2Sn\langle^{O}_{X}}$ frame-work, 'X' and $\mathrm{ON{=}CR^2R^3}$ systems. Dimethyltin diacetoximate shows absorptions at 592, $\nu_{as}$(Sn—O); 570, $\nu_{as}$(Sn—C); 530, $\nu_s$(Sn—C) and 512 cm$^{-1}$, $\nu_s$(Sn—O), which can be ascribed [8—10] to the vibrations of the $\mathrm{Me_2SnO_2}$ moiety. The bands characteristic of $\mathrm{Et_2SnO_2}$ moiety [9—12] were observed at 675 ± 5, $\rho$(Sn—Et); 602—565, $\nu_{as}$(Sn—O); 535—520, $\nu_{as}$(Sn—C); 490 ± 5, $\nu_s$(Sn—C) and 468 ± 3, $\nu_s$(Sn—O); and those characteristic of $\mathrm{Bu_2SnO_2}$ moiety [9—14] in the regions: 715—660, (Sn—$\mathrm{CH_2}$ *gauche*); 675—640, (Sn—$\mathrm{CH_2}$ *trans*); 608—570, $\nu_{as}$(Sn—O) + $\nu_{as}$(Sn—C); 520—5.04, $\nu_s$(Sn—C) and 490—465 cm$^{-1}$, $\nu_s$(Sn—O).

In the derivatives, $\mathrm{R_2Sn(OR^1)(ON{=}CR^2R^3)}$, $\mathrm{Bu_2Sn(Cl)(ON{=}CMe_2)}$, $\mathrm{Bu_2Sn(acac)(ON{=}CMe_2)}$, $\mathrm{Bu_2Sn(OEt)(SPh)}$, $\mathrm{Bu_2Sn(O\text{-}i\text{-}Pr)(COOMe)}$, absorptions characteristic [10,14—17] of C—O(alkoxy) Sn—Cl, C=O, Sn—S, C=O (acetoxy) appear at 1065—1050, 330, 1610—1580, 340, 1590—1550 cm$^{-1}$, respectively.

The presence of oximate groups in the derivatives is characterized [18—20] by bands in the region: 1660—1600 cm$^{-1}$, $\nu$(C=N) and 965—910 cm$^{-1}$, $\nu$(N—O) [20].

Some significant features of the infrared spectra are: (i) the appearance of two peaks in the C=N region, one at 1620—1600 cm$^{-1}$ ($\nu$(C=N) bridging) and another at 1660—1640 cm$^{-1}$ ($\nu$(C=N) terminal) in the derivatives of the type $R_2Sn(ON{=}CHMe)_2$, is indicative of the dimeric nature of derivatives having SnONSnON bridges. It is noteworthy that although the derivatives $R_2Sn(ON{=}CMe_2)_2$ are monomeric in refluxing benzene, they exhibit two peaks in the C=N stretching region which is indicative of a tendency of association in liquid or solid state, (ii) the appearance of two peaks assignable to the C=N stretching frequency in derivatives of the type $R_2Sn(OEt)(ON{=}CR^2R^3)$ points to an equilibrium of the type:



## Proton magnetic resonance spectra

PMR spectral data ($\tau$ (ppm), TMS) of these new derivatives exhibited the expected signals characteristic of organotin and oximate moieties (Table 1), and some significant features are described below:

(i) The $^{119}Sn-CH_3$ coupling constant of ~72 Hz observed in the derivative $Me_2Sn(ON{=}CMe_2)_2$ is in the expected range for trigonal-bipyramidal systems [21,22]. However, in all the other dioximate derivatives the observed coupling constant ~59 Hz is indicative of a tetrahedral structure [23].

(ii) Two doublets centered at 7.85 ($J$ 6 Hz) and 7.72 ($J$ 6 Hz) in the derivative $Et_2Sn(ON{=}CHMe)_2$ are presumably due to terminal and bridging iminoxy groups, resulting from the dimeric structure. The azomethine protons appear as two poorly resolved quartets in the regions 3.30—2.80 and 2.80—2.30 due to *syn* and *anti* isomers.

## Experimental

Precautions were taken to exclude moisture during the reactions. Solvents and starting materials were dried by conventional techniques. Diorganotin dialkoxides [24,25] and oximes [26] were prepared by literature methods. Analytical and insrtumental procedures were as described previously [27,28].

*Preparation of dialkyltin dioximates*

Representative examples of preparative procedures (eq. 1—3) are given below: Further details are in Tables 2 and 3.

(1) A mixture of dibutyltin oxide (6.70 g, 26.92 mmol) and acetoxime (3.94 g, 26.95 mmol) in benzene (ca. 40 ml) was refluxed and the binary azeotrope of benzene/water was slowly fractionated out (12 h). After removal of benzene, the resultant colourless liquid on distillation under reduced pressure gave $Bu_2Sn(ON{=}CMe_2)_2$ (Yield: 70%) (Table 2).

(2) To a benzene solution of sodium ethoxide, prepared by dissolving metallic sodium (0.57 g, 24.78 mmol) in the minimum quantity of ethanol, acetoxime (1.80 g, 24.62 mmol) was added, followed by a benzene solution of dimethyltin dichloride (2.71 g, 12.33 mmol). The mixture was refluxed for 10 h. After removal of solvents under reduced pressure, the residual mass on sublimation yielded $Me_2Sn(ON{=}CMe_2)_2$ (Table 2).

(3) Methylethylketoxime (0.51 g, 5.86 mmol) was added to dibutyltin diisopropoxide (1.03 g, 2.93 mmol) in benzene (80 ml). The mixture was refluxed

TABLE 3

SOME DIORGANOTIN (MONO)OXIMATES

| Compound [a] (Yield (%)) [b] | B.p. (°C/mmHg) | $n_D^{26}$ | Analysis (Found (calcd.) (%)) | | Mol. wt. (Found (calcd.) |
|---|---|---|---|---|---|
| | | | Sn | N | |
| $Et_2Sn(OEt)(ON{=}CMeH)$<br>(80) | 86/0.6 | 1.4982 | 42.36<br>(42.40) | 4.90<br>(5.00) | 397<br>(280) |
| $Et_2Sn(OEt)(ON{=}CMe_2)$<br>(78) | 116/1.2 | 1.5172 | 40.36<br>(40.38) | 4.68<br>(4.76) | 372<br>(294) |
| $Bu_2Sn(OEt)(ON{=}CMeH)$<br>(75) | 130/3.0 | 1.4782 | 35.30<br>(35.32) | 3.96<br>(4.17) | 445<br>(336) |
| $Bu_2Sn(OEt)(ON{=}CMe_2)$<br>(70) | 128/2.0 | 1.4834 | 33.89<br>(33.90) | 3.91<br>(4.00) | 423<br>(350) |
| $Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CMe_2)$<br>(80) | 103/0.4 | 1.4750 | 32.58<br>(32.60) | 3.75<br>(3.85) | 369<br>(364) |
| $Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CMeEt)$<br>(75) | 135/1.5 | — | 31.36<br>(31.39) | 3.59<br>(3.70) | 389<br>(378) |
| $Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CHPh)$<br>(60) | 164/0.6 | 1.5250 | 28.76<br>(28.80) | 3.32<br>(3.40) | 424<br>(412) |
| $Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CMePH)$<br>(72) | 182/1.0 | 1.5220 | 27.82<br>(27.85) | 3.21<br>(3.29) | 434<br>(426) |
| $Bu_2Sn(Cl)(ON{=}CMe_2)$ [c] | 102/0.2 | — | 34.82<br>(34.86) | 3.96<br>(4.11) | — |
| $Bu_2Sn(acac)(ON{=}CMe_2)$ | 118/0.3 | — | 29.34<br>(29.38) | 3.38<br>(3.47) | — |
| $Bu_2Sn(OEt)(SPh)$ [d] | 160/1.0 | — | 30.64<br>(30.66) | — | — |
| $Bu_2Sn(O\text{-}i\text{-}Pr)(OCOMe)$ [e] | 105/0.6 | — | 33.79<br>(33.81) | 0.43<br>— | — |

[a] All compounds are liquids. [b] Yields refer to distilled products. [c] % Cl in the compound, Found: 10.36; calcd. 10.41. [d] % S in the compound, Found: 8.22; calcd.: 8.28. [e] White solid.

TABLE 4

PHYSICAL PROPERTIES AND RELEVANT IR DATA FOR ORGANOTIN INSERTION PRODUCTS

| Compound | Nature and colour | Analysis (found (calcd.) (%)) | | IR (cm$^{-1}$) | | |
|---|---|---|---|---|---|---|
| | | Sn | N | $\nu$(C=N) | $\nu$(N–O) | $\nu$(C=O) |
| $\mathrm{Bu_2Sn[OCH(CCl_3)ON{=}CMe_2][ON{=}CMe_2]}$ | Yellow-red viscous liquid | 22.60<br>(22.63) | 5.28<br>(5.34) | 1640w | 928s | — |
| $\mathrm{Bu_2Sn[OCH(CCl_3)ON{=}CMe_2]_2}$ | Yellow-red viscous liquid | 17.64<br>(17.67) | 4.07<br>(4.17) | 1648m | 925s | — |
| $\mathrm{Bu_2Sn[N(Ph)\underset{O}{\underset{\|}{C}}ON{=}CMe_2][ON{=}CMe_2]}$ | Red-viscous liquid | 23.89<br>(23.92) | 8.40<br>(8.47) | 1640w | 932m | 1720s |
| $\mathrm{Bu_2Sn[N(Ph)\underset{O}{\underset{\|}{C}}ON{=}CMe_2]_2}$ | Red-viscous liquid | 19.27<br>(19.30) | 9.00<br>(9.11) | 1638w(sh) | 9.28s | 1710–1680s(br) |
| $\mathrm{Bu_2Sn[N(Np)\underset{O}{\underset{\|}{C}}ON{=}CMe_2][ON{=}CMe_2]}$ | Yellow-viscous liquid | 21.70<br>(21.73) | 7.58<br>(7.69) | 1642w | 932m | 1685s |
| $\mathrm{Bu_2Sn[N(Np)\underset{O}{\underset{\|}{C}}ON{=}CMe_2]_2}$ | Yellow-coloured semi-solid | 16.58<br>(16.60) | 7.78<br>(7.84) | 1635w | 930s | 1680s |

with continuous fractionation out of the benzene-isopropanol azeotrope (i-PrOH found: 0.33 g, calcd. for two moles: 0.35 g) for about 5 h. Removal of the solvent, followed by distillation under reduced pressure, gave $Bu_2Sn(ON{=}CMeEt)_2$ (Table 2).

When the above reactions, were carried out with 1/1 molar ratios, the mono-alkoxy (mono)oximate products were obtained (Table 3).

### *Reactions of dibutyltin diacetoximate with (i) dibutyltin dichloride and (ii) acetylacetone*

(i) A mixture of $Bu_2Sn(ON{=}CMe_2)_2$ (1.09 g, 2.89 mmol) dibutyltin dichloride (0.88 g, 2.90 mmol) and benzene (ca. 10 ml) was heated for 4 h at 110°C. After removal of the solvent, the residual liquid was distilled under reduced pressure to yield $Bu_2Sn(Cl)(ON{=}CMe_2)$ (Table 3).

(ii) Dibutyltin diacetoximate (1.04 g, 2.76 mmol) and acetylacetone (0.27 g, 2.70 mmol) were heated together at 80—90°C for 6 h. Distillation afforded acetoxime, b.p. 135°C (identified by IR and PMR spectroscopy), and $Bu_2Sn$(acac) $(ON{=}CMe_2)$ (Table 3).

### *Reactions of (i) dibutyltin (ethoxy)acetoximate with thiophenol and of (ii) dibutyltin (isopropoxy)acetoximate with acetic anhydride*

(i) Thiophenol (1.00 g, 9.09 mmol) was added to $Bu_2Sn(OEt)(ON{=}CMe_2)$ (3.30 g, 9.06 mmol) in carbon tetrachloride (ca. 10 ml). The mixture was refluxed for about 7 h. Removal of the solvent followed by distillation under reduced pressure afforded the acetoxime (identified by IR and PMR spectra) and $Bu_2Sn(OEt)(SPh)$ (Table 3).

(ii) An exothermic reaction was observed when acetic anhydride (0.61 g, 5.98 mmol) was added to a carbon tetrachloride solution of $Bu_2Sn(O\text{-}i\text{-}Pr)(ON{=}CMe_2)$ (2.20 g, 6.04 mmol). The mixture was refluxed at 100°C for 12 h. Removal of the solvent followed by distillation under reduced pressure gave a colourless liquid, *O*-oxime ester (identified by elemental analyses and IR spectroscopy) and a white solid $Bu_2Sn(O\text{-}i\text{-}Pr)(OCOMe)$ (Table 3).

### *Preparation of insertion products*

On mixing dibutyltin diacetoximate and an organic electrophile in equimolar quantitites at room temperature reaction took place immediately and exothermally with $CCl_3CHO$, PhNCO and NpNCO to give 1/1 insertion products (monitored by IR spectroscopy). Formation of 1/2 adducts (see Table 4) required varying periods of time ($CCl_3CHO$, 5 min; PhNCO, 24 h; NpNCO, 5 days). However, PhNCS did not react (in 1/1 or 1/2 ratios) with dibutyltin diacetoximate during one week at room temperature.

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