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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.

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$<$, 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:

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

$$Me_2SnCl_2 + 2\,NaOEt + 2\,HON{=}CR^2R^3 \xrightarrow[8-10\text{ h}]{\text{benzene, reflux}} Me_2Sn(ON{=}CMe_2)_2 + 2\,NaCl + 2\,EtOH \qquad (2)$$

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

$$R_2Sn(OR^1)_2 + n\,HON{=}CR^2R^3 \xrightarrow[5-7\text{ h}]{\text{benzene, reflux}} Bu_2Sn(OR^1)_{2-n}(ON{=}CR^2R^3)_n + n\,R^1OH \qquad (3)$$

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TABLE 1

PMR SPECTRAL DATA FOR SOME DIORGANOTIN OXIMATES

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

[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, $OCH_2\underline{CH_3}$) and 5.98 (q, *J* 8 Hz, $O\underline{CH_2}CH_3$). [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:

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

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]	90/10 [c]	—	40.50	9.48	298
(90)			(40.51)	(9.56)	(293)
$Me_2Sn(ON{=}CMePh)_2$	110/1.5	—	28.43	6.64	422
(88)			(28.46)	(6.71)	(417)
$Et_2Sn(ON{=}CHMe)_2$ [b]	90/6 [c]	—	40.48	9.48	390
(83)			(40.52)	(9.56)	(293)
$Et_2Sn(ON{=}CMe_2)_2$	86/0.4	1.4858	36.93	8.64	326
(82)			(36.98)	(8.73)	(321)
$Et_2Sn(ON{=}CMeEt)_2$	124/0.4	1.4930	33.96	7.90	352
(75)			(34.00)	(8.03)	(349)
$Et_2Sn(ON{=}CMePh)_2$	128/0.2	1.5520	26,63	6.18	440
(65)			(26.66)	(6.29)	(445)
$Bu_2Sn(ON{=}CHMe)_2$	102/0.2	1.4845	33.96	7.96	408
(72)			(34.00)	(8.03)	(349)
$Bu_2Sn(ON{=}CMe_2)_2$	108/0.2	1.4738	31.40	7.35	380
(78)			(31.47)	(7.43)	(377)
$Bu_2Sn(ON{=}CMeEt)_2$	148/1.5	1.4762	29.27	6.85	410
(72)			(29.29)	(6.92)	(405)
$Bu_2Sn(ON{=}CHPh)_2$	169/0.7	1.5250	25.06	5.87	476
(68)			(25.08)	(5.92)	(473)
$Bu_2Sn(ON{=}CMePh)_2$	191/2.5	1.5220	23.66	5.49	506
(70)			(23.68)	(5.59)	(501)
$Bu_2Sn[ON{=}C(Me)CH_2Cl]_2$	110/0.3	—	26.58	6.20	449
(58)			(26.61)	(6.28)	(446)
$Bu_2Sn[ON{=}C(Me)C_6H_4Cl]_2$	164/0.3	1.5478	20.80	4.82	574
(56)			(20.82)	(4.91)	(570)
$Bu_2Sn[ON{=}C(Me)C_6H_4Br]_2$	182/0.5	1.5430	17.95	4.18	653
(60)			(18.01)	(4.25)	(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:

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

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

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

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

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

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

(A = B = CCl_3CHO, PhNCO, NpNCO; n = 1 or 2)

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

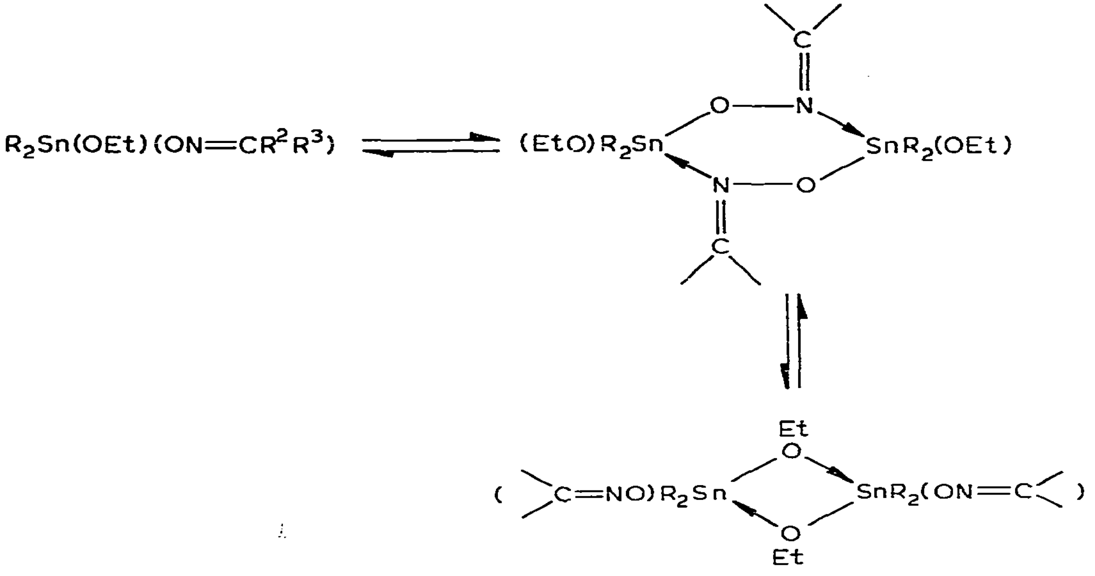
Infrared spectra

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

In the derivatives, $R_2Sn(OR^1)(ON{=}CR^2R^3)$, $Bu_2Sn(Cl)(ON{=}CMe_2)$, $Bu_2Sn(acac)(ON{=}CMe_2)$, $Bu_2Sn(OEt)(SPh)$, $Bu_2Sn(O\text{-}i\text{-}Pr)(COOMe)$, 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}, ν(C=N) and 965—910 cm^{-1}, ν(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} (ν(C=N) bridging) and another at 1660—1640 cm^{-1} (ν(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 (τ (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 $\mathrm{Bu_2Sn(ON{=}CMe_2)_2}$ (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 $\mathrm{Me_2Sn(ON{=}CMe_2)_2}$ (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

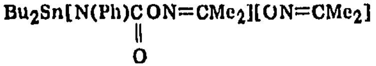
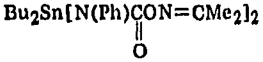
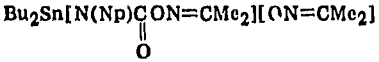
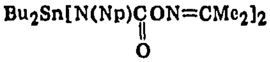
SOME DIORGANOTIN (MONO)OXIMATES

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

[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	ν(C=N)	ν(N–O)	ν(C=O)
$Bu_2Sn[OCH(CCl_3)ON{=}CMe_2][ON{=}CMe_2]$	Yellow-red viscous liquid	22.60 (22.63)	5.28 (5.34)	1640w	928s	—
$Bu_2Sn[OCH(CCl_3)ON{=}CMe_2]_2$	Yellow-red viscous liquid	17.64 (17.67)	4.07 (4.17)	1648m	925s	—
 $Bu_2Sn[N(Ph)\underset{\underset{O}{\Vert}}{C}ON{=}CMe_2][ON{=}CMe_2]$	Red-viscous liquid	23.89 (23.92)	8.40 (8.47)	1640w	932m	1720s
 $Bu_2Sn[N(Ph)\underset{\underset{O}{\Vert}}{C}ON{=}CMe_2]_2$	Red-viscous liquid	19.27 (19.30)	9.00 (9.11)	1638w(sh)	9.28s	1710–1680s(br)
 $Bu_2Sn[N(Np)\underset{\underset{O}{\Vert}}{C}ON{=}CMe_2][ON{=}CMe_2]$	Yellow-viscous liquid	21.70 (21.73)	7.58 (7.69)	1642w	932m	1685s
 $Bu_2Sn[N(Np)\underset{\underset{O}{\Vert}}{C}ON{=}CMe_2]_2$	Yellow-coloured semi-solid	16.58 (16.60)	7.78 (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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