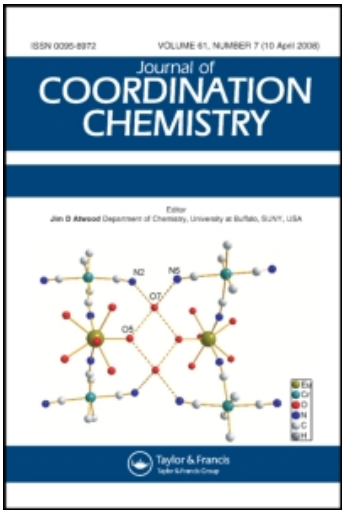

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Guanylurea derivatives of diorganotin(IV) dichloride

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Guanylurea derivatives of diorganotin(IV) dichloride

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A series of organotin complexes with guanylurea (H_2L) have been synthesized. All the complexes **1**–**8**, BuMeSnClHL, PhEtSnClHL, PhMeSnClHL, PhBuSnClHL, BuMeSnL, PhEtSnL, PhMeSnL, and PhBuSnL have been characterized by elemental analysis, FTIR, and multinuclear spectroscopic techniques. Complexes **1**–**4** are five-coordinate whereas **5**–**8** are four-coordinate.

Keywords: Diorganotin dichlorides; Diorganotin oxide; Guanylurea; Synthesis; Characterization

1. Introduction

Organotin complexes have potential industrial applications and biological activity [1]. Tetraorganotin compounds, tintetrahalides, and stannanes (SnR_nH_{4-n}, where $n = 1$–3) are of synthetic value for production of derivatives for industrial end values [2]. Industrial use of less-toxic organotin compounds (R_2SnX_2 and $RSnX_3$ types) accounts for almost two-third of the total world's consumption of tin. Several organotin compounds such as di(*n*-butyl)tin laurate are used as hydrochloric acid scavengers in PVC. Organotin carboxylate complexes are employed as catalysts for trans-esterification and polyurethane polymerization [2]. Many organotin compounds are used as antitumor drugs [3–6]; other biological applications include insecticides, fungicides, wood preservatives, and anti-inflammatory drugs [7, 8].

Synthesis and structural studies of neutral tetra-coordinated tin compounds derived from ligands with strategically placed donor atoms (O, N, and S) have received attention [9], and there has been resurgence of interest in metal-sulfur and

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metal-nitrogen chemistry because of the occurrence of complexes having metal-sulfur and metal-nitrogen bonds in biological systems [10–12].

Guanylurea compounds are nitrification inhibitors in agriculture and horticulture [13], used as fire retardant in plywood, textiles, timber, and other polymers [14–17] and in making fire-resistant double-sided adhesive tapes for fixing of electronic devices [18]. Its complexes of Cu, Zn, Co, and Fe are used in deodorants for removing NH_3, H_2S, and mercaptans from odorous air [19] and in preventing the cytostatically caused falling out of hair [20]. Guanylurea is a strong bidentate ligand [21]. Complexes of guanylurea with B, V, As, Cu, Ni, and Si are well known [22–24]. The ligand has been used extensively in coordination chemistry of transition metals but its potential in main group chemistry is limited.

We elected to study the reactions of phenylmethyltin dichloride, phenylethyltin dichloride, phenylbutyltin dichloride, and butylmethyltin dichloride with guanylurea in 1 : 1 molar ratio to synthesize complexes having the *N*,*O*′-bidentate coordination and *N*,*N*′-bidentate coordination.

2. Experimental

2.1. *Materials*

All manipulations were carried out using standard Schlenk techniques under a dry nitrogen atmosphere. Organotin compounds, R_4Sn (R = Me, Et, Bu), $RSnCl_3$ (R = Me, Bu, Ph), and guanylurea sulfate (Aldrich) were used as received. Methylphenyltin dichloride, ethylphenyltin dichloride, *n*-butylphenyltin dichloride, *n*-butylmethyltin dichloride [25], corresponding diorganotin oxides, and guanylurea were prepared as reported [26, 27].

2.2. *Measurements*

Tin was quantitatively determined by standard method [28]. Solvents were dried and distilled by reported methods. IR spectra were recorded using a Shimadzu FTIR 8700 spectrophotometer. ^{1}H-, ^{13}C-, and ^{119}Sn-NMR spectra were recorded on a Bruker 300 MHz spectrometer using $CDCl_3$ as solvent. Elemental analysis was carried out using a EURO EA 3000 elemental analyzer. The FAB mass spectra in 3-nitrobenzyl alcohol (NBA) were recorded at room temperature on a JEOL SX 102/DA-6000 Mass Spectrometer/Data System using argon/xenon (6 kV, 10 mA) as the FAB gas. The accelerating voltage was 10 kV.

3. Synthesis

Complexes **1**–**4** were obtained following a procedure, reported here for **1** and **5**–**8** were obtained following a procedure, reported for **5**.

3.1. *Synthesis of PhMeSnClHL (1)*

A mixture of ~2.5 mmol of KOH in 10 mL methanol and 0.25 g (2.5 mmol) of guanylurea was homogenized by shaking at room temperature and the pH was adjusted to 7 by addition of 2 N HCl. $PhMeSnCl_2$ (0.705 g, 2.5 mmol) was added and the mixture was stirred for 2 h at room temperature. The solution was filtered and the filtrate was evaporated under vacuum. The product was dissolved in a minimum of methanol, filtered and the solvent from the filtrate was removed *in vacuo* to obtain a pure light yellow viscous liquid. Yield, 71%: Anal. Calcd for $C_9H_{13}N_4OClSn$: C, 31.1; H, 3.8; N, 16.2; Cl, 10.2; Sn, 34.1. Found: C, 30.8; H, 3.6; N, 16.5; Cl, 9.9; Sn, 33.9%.

3.2. *Synthesis of PhMeSnL (5)*

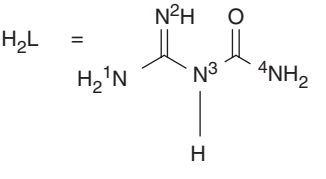
A solution of 0.705 g (2.5 mmol) of $PhMeSnCl_2$ in ~15 mL methanol was added to a mixture of 5.5 mmol of KOH and ~15 mL of methanol. PhMeSnO was separated immediately. It was washed twice with methanol and added to a solution of 0.25 g (2.5 mmol) guanylurea in methanol within 5 min and the resulting solution was stirred at room temperature. After 4 h small amounts of PhMeSnO were filtered off and clear colorless filtrate was concentrated *in vacuo*. During this operation a dark yellow viscous liquid was separated. Yield, 61%: Anal. Calcd for $C_9H_{12}N_4OSn$: C, 34.8; H, 3.9; N, 18.0; Sn, 38.2. Found: C, 34.6; H, 3.8; N, 18.3; Sn, 38.1%.

$$R^1R^2SnCl_2 + H_2L \xrightarrow[\text{room temp. 2 h}]{\text{KOH/Methanol}} R^1R^2SnClLH + KCl + H_2O$$

$$R^1R^2SnCl_2 + KOH \xrightarrow[\text{room temp.}]{\text{Methanol}} R^1R^2SnO + 2KCl + H_2O$$

$$R^1R^2SnO + H_2L \xrightarrow{\text{Methanol/4 h/r.t.}} R^1R^2SnL + H_2O$$

where $R^1 = Ph$, $R^2 = Me$ (**1**, **5**); $R^1 = Ph$, $R^2 = Et$ (**2**, **6**); $R^1 = Ph$, $R^2 = Bu$ (**3**, **7**); $R^1 = Bu$, $R^2 = Me$ (**4**, **8**).



4. Results and discussion

4.1. *Chemistry*

Dehydrochlorination occurs during the synthesis of **1**–**4** and dehydration during the synthesis of **5**–**8**. All complexes were yellow viscous liquids, soluble in

dimethylsulfoxide, methanol, and chloroform and insoluble in pentane, hexane, and heptane.

4.2. *IR spectra (Listing in Supplementary material)*

For **1–4**, shifting of the bands at 1638 cm^{-1} and 1310 cm^{-1} for C=N from 1604 cm^{-1} and 1350 cm^{-1}, respectively, may be explained by ligand tautomerization [27]. The strong band at 1740 cm^{-1} for guanylurea, assigned to C=O shifted to ~1711 cm^{-1}, indicating that carbonyl is involved in bond formation. Further absorption bands at ~664 cm^{-1} due to ν(Sn–O) [29] indicated carbonyl bonding with tin. The band at ~469 cm^{-1} showed formation of a Sn–N bond [27]. Stretching frequency due to $\nu(NH_2)$ mode was observed at ~3418 cm^{-1} both in the ligand and derivatives, indicating that both end amino groups are not involved in bond formation. The band due to $\nu(N^3H)$, observed at 3130 cm^{-1} in case of ligand, was absent in the complexes.

For **5–8**, the persistence of the strong band at 1740 cm^{-1} for C=O and 1604 cm^{-1} for C=N group at the same positions as for free ligand showed that neither C=O nor C=N were involved in bond formation. The band due to the $\nu(N^3H)$ at 3130 cm^{-1} for ligand was observed in **5–8** at the same position indicating non-involvement of N^3H in bond formation. Absence of stretching frequencies due to ν(Sn–O) and $\nu(NH_2)$ modes at ~561 and 3418 cm^{-1} [29], respectively, indicated that bond formation occurred through the $-NH_2$ of the ligand with loss of water.

Due to strong chelating nature of guanylurea [21], it is proposed that formation of a chelate ring had taken place, as for boron and other complexes [21, 27].

4.3. *NMR spectra*

^{1}H-NMR spectra of ligand showed a signal at δ5.7 due to N^4H; N^1H, N^2H, and N^3H were observed as a broad signal at δ5.3–4.7. In ^{1}H-NMR spectra of **1–4**, the N^4H protons were observed downfield at ~δ6.2 due to coordination of carbonyl with Sn. N^2H and N^1H protons were observed as a broad signal at δ5.5–4.9. In ^{1}H-NMR spectra of **5–8**, the N^4H proton was observed at ~δ4.8 indicating direct coordination of N^4H with Sn while all other protons of the ligand were a broad band at δ3.9–3.5. The other ^{1}H-NMR signals of all the complexes were observed at their usual positions (table 1). The proton integrations were consistent with the molecular formula.

^{13}C-NMR spectra of complexes showed a signal at δ182–171 due to C=O [30] and signals at δ10.5–10.2 and 18.7–18.3 were due to ethyl-carbons. Peaks in the range δ136.5–127.1 were due to phenyl carbons at their usual positions [31]; phenyl carbons coupled to tin could not be distinguished due to identical environment of *ortho* and *meta* carbon of the phenyl ring and assignments made by analogy [32]. The other ^{13}C-NMR signals for all complexes are shown in table 2.

^{119}Sn-NMR spectra of **1–4** (figure 1) showed singlets at $\delta -76$, $\delta -55$, $\delta -23$, and $\delta -67$, respectively, while ^{119}Sn-NMR spectra of **5–8** (figure 2) showed singlets at δ119, δ144, δ173, and δ139, respectively. The appearance of the singlets showed that there is only one isomer [33]. The signals in **1–4** and **5–8** are attributed to

Table 1. ^{1}H-NMR spectral data of derivatives.

No.	Phenyl protons	$N^{2}H$ and $N^{1}H/N^{3}H$ (figure 1)	$N^{4}H$ (figure 1)	Sn–$^{1}CH_2$ $^{2}CH_3$	Sn–CH_3	$^{1}CH_3$ $^{2}CH_2$ $^{3}CH_2$ $^{4}CH_2Sn$
1	7.7–7.5 (5 H, m)	5.2 (3 H, br)	6.2 (2 H, s)	–	1.1 (s) $^{2}J(^{119}Sn,^{1}H)$ 69.2	–
2	7.5–7.3 (5 H, m)	4.9 (3 H, br)	6.2 (2 H, s)	1.1 q (1), 1.3 t (2) $^{2}J(^{119}Sn,^{1}H)$ 72.4	–	–
3	7.6–7.4 (5 H, m)	5.1 (3 H, br)	6.1 (2 H, s)	–	–	0.8 t (4), 1.6-1.1 m (1, 2, 3) $^{2}J(^{119}Sn,^{1}H)$ 73.1
4	–	5.5 (3 H, br)	6.2 (2 H, s)		1.1 (s) $^{2}J(^{119}Sn,^{1}H)$ 70.4	0.9 t (4) 1.9–1.4 m (1, 2, 3) $^{2}J(^{119}Sn,^{1}H)$ 73.8
5	7.4–7.2 (5 H, m)	3.9 (3 H, br)	4.9 (1 H, s)	–	1.2 (s) $^{2}J(^{119}Sn,^{1}H)$ 62.7	–
6	7.4–7.2 (5 H, m)	3.6 (3 H, br)	4.8 (1 H, s)	1.5 q (1), 1.9 t (2) $^{2}J(^{119}Sn,^{1}H)$ 68.1	–	–
7	7.3–7.2 (5 H, m)	3.7 (3 H, br)	4.7 (1 H, s)	–	–	0.8 t (4), 1.6–1.1 m (1, 2, 3) $^{2}J(^{119}Sn,^{1}H)$ 58.9
8	–	3.5 (3 H, br)	4.8 (1 H, s)		1.3 (s) $^{2}J(^{119}Sn,^{1}H)$ 59.4	0.9 t (4) 1.9–1.4 m (1, 2, 3) $^{2}J(^{119}Sn,^{1}H)$ 57.1

m = multiplet, s = singlet, q = quartet, t = triplet, br = broad.

Table 2. ^{13}C-NMR and ^{119}Sn NMR spectral data of derivatives.

No.	Phenyl carbons	$^{1}CH_3$ $^{2}CH_2$ $^{3}CH_2$ $^{4}CH_2Sn$	C=N		C=O	CH_3	$^{1}CH_2$ $^{2}CH_3$	^{119}Sn NMR
1	135.8 (C_1), 134.2 (C_4), 128.7(C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$610.3 $^{2}J(^{119}Sn,^{13}C)$49.5 $^{3}J(^{119}Sn,^{13}C)$68.1	–	157.7	159.2	172	9.5 $^{1}J(^{119}Sn,^{13}C)$ 595.1	–	−23
2	136.5 (C_1), 134.9 (C_4), 128.6 (C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$618.2 $^{2}J(^{119}Sn,^{13}C)$52.4 $^{3}J(^{119}Sn,^{13}C)$65.3	–	157.2	158.5	171	–	10.2 (1), 18.7 (2) $^{1}J(^{119}Sn,^{13}C)$ 553.8 $^{2}J(^{119}Sn,^{13}C)$ 34.2	−55
3	136.1 (C_1), 135.0 (C_4), 127.1 (C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$615.7 $^{2}J(^{119}Sn,^{13}C)$51.3 $^{3}J(^{119}Sn,^{13}C)$67.3	δ 28.9(1), δ 27.0(2), δ 25.6(3), δ 13.5(4) $^{1}J(^{119}Sn,^{13}C)$589.5 $^{2}J(^{119}Sn,^{13}C)$40.6 $^{3}J(^{119}Sn,^{13}C)$73.1	158.7	157.3	173	–	–	−67
4	–	δ 30.1(1), δ 27.7(2), δ 25. 1(3), δ 13.2(4) $^{1}J(^{119}Sn,^{13}C)$583.4 $^{2}J(^{119}Sn,^{13}C)$37.2 $^{3}J(^{119}Sn,^{13}C)$71.8	158.1	156.7	172	8.6 $^{1}J(^{119}Sn,^{13}C)$ 587.2		−76
5	136.1 (C_1), 134.6 (C_4), 129.1(C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$531.6 $^{2}J(^{119}Sn,^{13}C)$35.9 $^{3}J(^{119}Sn,^{13}C)$53.4	–	155.8	154.1	181	9.7 $^{1}J(^{119}Sn,^{13}C)$ 322.9	–	173
6	136.2 (C_1), 134.5 (C_4), 128.9 (C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$538.1 $^{2}J(^{119}Sn,^{13}C)$37.5 $^{3}J(^{119}Sn,^{13}C)$55.2	–	155.6	154.2	180	–	10.5 (1), 18.3 (2) $^{1}J(^{119}Sn,^{13}C)$ 325.2 $^{2}J(^{119}Sn,^{13}C)$ 24.7	144
7	135.9 (C_1), 134.5 (C_4), 128.4 (C_2, C_3, C_5, C_6) $^{1}J(^{119}Sn,^{13}C)$535.4 $^{2}J(^{119}Sn,^{13}C)$36.8 $^{3}J(^{119}Sn,^{13}C)$54.7	δ 31.3(1), δ 27.9(2), δ 25.8(3), δ 13.3(4) $^{1}J(^{119}Sn,^{13}C)$ 327.1 $^{2}J(^{119}Sn,^{13}C)$27.3 $^{3}J(^{119}Sn,^{13}C)$61.5	155.3	153.9	182	–	–	139
8	–	1.8(1), δ 28.3(2), δ 26.4(3), δ 13.1(4) $^{1}J(^{119}Sn,^{13}C)$312.4 $^{2}J(^{119}Sn,^{13}C)$24.9 $^{3}J(^{119}Sn,^{13}C)$58.9	155.8	154.4	178.8	8.9 $^{1}J(^{119}Sn,^{13}C)$ 335.6		119

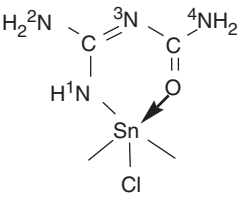


Figure 1. Structures of **1**–**4**.

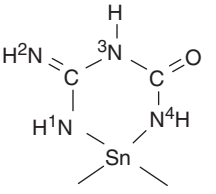


Figure 2. Structures of **5**–**8**.

penta-coordinate and tetra-coordinate Sn [34], respectively. The signals more upfield from that of **5**–**8** were due to increase in electron density on Sn due the oxygen donors of the ligand.

4.4. *Mass spectra*

Every effort to obtain crystals did not produce even a single crystal. Evidence in support of the structures of these compounds came from FAB mass spectral studies.

Under the FAB mass conditions, the compounds displayed very low-intensity molecular ions. These were observed at *m*/*z* 347, 361, 389, 327, 311, 325, 353, 291 for $[M]^+$ for monomeric structures. In all the products, major fragmentation was due to loss of the alkyl. Complete fragmentation of the ligand did not occur in a single event but a part underwent fragmentation and then the remaining part underwent fragmentation. Isotopic ions were observed corresponding to the different isotopes of tin. For instance, the isotopic peaks for PhMeSnL $[M - CH_3]^+$ were observed at 293 (44.0), 294 (27.5), 295 (75.2), 296 (33.2), 297 (100.0), 299 (14.1), and 301 (16.8). The mass fragmentation data are given in Supplementary material.

5. Conclusion

The stannylguanylureas reported herein are a new class of Sn–N/and O bonded compounds containing guanylurea in the framework. Elemental analysis (table 3) and spectroscopic and literature studies suggested the existence of penta-coordinate and tetra-coordinate structures.

Table 3. Analytical data.

No.	Reactants Compound : Ligand g(mmol) : g(mmol)	Yield (%)	Products	Analysis: Found (Calcd) (%) C	H	N	Sn	Cl
1	$PhMeSnCl_2 : H_2L$ 0.705 g (2.5) : 0.255 g (2.5)	71	PhMeSnClHL $C_7H_{14}N_4OSnCl$	30.8 (31.1)	3.6 (3.8)	16.5 (16.2)	33.9 (34.1)	9.9 (10.2)
2	$PhEtSnCl_2 : H_2L$ 0.740 g (2.5) : 0.255 g (2.5)	68	PhEtSnClHL $C_8H_{16}N_4OSnCl$	33.1 (33.2)	4.0 (4.2)	15.3 (15.5)	32.6 (32.8)	9.6 (9.8)
3	$PhBuSnCl_2 : H_2L$ 0.809 g (2.5) : 0.255 g (2.5)	74	PhBuSnClHL $C_{10}H_{20}N_4OSnCl$	36.8 (37.0)	4.6 (4.9)	14.7 (14.4)	30.3 (30.5)	8.9 (9.1)
4	$BuMeSnCl_2 : H_2L$ 0.654 g (2.5) : 0.255 g (2.5)	73	BuMeSnClHL $C_5H_{18}N_4OSnCl$	25.5 (25.7)	5.1 (5.2)	16.9 (17.1)	36.1 (36.2)	10.6 (10.8)
5	$PhMeSnCl_2 : H_2L$ 0.705 g (2.5) : 0.255 g (2.5)	61	PhMeSnL $C_7H_{12}N_4OSnCl$	34.6 (34.8)	3.8 (3.9)	18.3 (18.0)	38.1 (38.2)	–
6	$PhBuEtSnCl_2 : H_2L$ 0.740 g (2.5) : 0.255 g (2.5)	63	PhEtSnL $C_8H_{14}N_4OSnCl$	36.9 (37.0)	4.1 (4.3)	17.4 (17.2)	36.7 (36.5)	–
7	$PhBuSnCl_2 : H_2L$ 0.809 g (2.5) : 0.255 g (2.5)	62	PhBuSnL $C_{10}H_{18}N_4OSnCl$	40.9 (40.8)	4.9 (5.1)	15.7 (15.9)	33.3 (33.6)	–
8	$BuMeSnCl_2 : H_2L$ 0.654 g (2.5) : 0.255 g (2.5)	67	BuMeSnL $C_5H_{16}N_4OSnCl$	28.7 (28.9)	5.7 (5.5)	19.5 (19.3)	40.6 (40.8)	–

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