Asian Journal of Chemistry Vol. 6, No. 3 (1994), 498–508

Monophenyltin Derivatives of Some N-[*o*-Hydroxy Substituted (or H) Benzyl] Glycines

PRASANTHAN SIVADASAN and MADHUP CHANDRA*
Department of Chemistry,
DSB Campus, Kumaun Univeristy,
Nainital 263 002, India.

Several monophenyltin derivatives of N-[*o*-hydroxy substituted (or H) benzyl] glycines have been prepared by the interaction of monophenyltin triisopropoxide with the latter in 1 : 1, 1 : 2 and 1 : 3 molar ratios in benzene medium. The various compounds thus prepared were obtained as coloured solids with some of them being hygroscopic. All these compounds were characterized by azeotrope and elemental analyses, as well as by IR and PMR spectral measurements.

INTRODUCTION

The donor sites in N-[*o*-hydroxy substituted (or H) benzyl] glycines provide a favourable geometry for stable metal chelate ring formation which evoked considerable interest towards examining their ligating properties. However, such studies have largely been performed in solution[1,2]. No attempt has so far been made to prepare their metallo derivatives *via* the reactivity of the corresponding metal alkoxide, especially in view of the fact that these complexones contain replaceable hydrogen(s). A systematic programme of work in this direction has recently been undertaken in these laboratories.

The work described here, thus deals with the preparation and characterization of monophenyltin derivatives of some N-[*o*-hydroxy substituted (or H) benzyl] glycines(I) *viz.*. (i) N-(2-hydroxy benzyl) glycine, (H_2hbg), (ii) N-(2-hydroxy-3-methyl benzyl) glycine (H_2hmbg–3), (iii) N-(2-hydroxyl-6-methyl benzyl) glycine (H_2hmbg–6), and (iv) N-(2-hydroxy-5-methyl benzyl) glycine (H_2hmbg–5). These derivatives were prepared by alcoholysis reactions involving the interaction of monophenyltin triisopropoxide with (I) in appropriate stoichiometric ratios *viz.* 1:1, 1:2 and 1:3 in benzene medium. The various compounds thus prepared were obtained as coloured solids and amongst them those containing isopropoxy group were found to be hygroscopic. All these compounds were characterized by azeotrope and elemental analysis, as well as by IR and PMR spectral measurements. However, the PMR spectra could be recorded only in case of 1:2 derivatives. It was not possible to have these measurements in case of 1:1 and 1:3 derivatives owing to their insolubility in the required solvents.

EXPERIMENTAL

Owing to highly hygroscopic nature of organotin alkoxides stringent precautions were taken to perform the experiments under strictly anhydrous conditions, using glasswares with standard interchangeable joints. Special weighing tubes and measuring pipettes were used for the analytical sampling of the compounds. For filtration purposes, a specially designed G_4 sintered glass filtering funnel fitted with a guard take at the upper end and filled with fused calcium chloride, was used.

The details of other glass apparatus are described as reported earlier.[3a]

Benzene (BDH, AR), isopropanol (BDH, Glaxo AnalaR) and solvent ether (E. Merck) were dried by standard procedures.[3b] Monophenyltin triisopropoxide was prepared by sodium method,[4] using monophenyltin trichloride, which in turn was prepared by a known method[5]. Tin was estimated by direct ignition of the sample after digestion with nitric and sulphuric acids followed by neutralization and precipitation by ammonia solution[6]. The other spectral studied was as performed in previous studies.[3a]

Preparation of N-[*o*-Hydroxy Substituted (or H) Benzyl] Glycines

The various N-[*o*-hydroxy substituted (or H) benzyl] glycines were prepared adopting procedures almost similar to those described by Mehrotra *et al*[7]. by condensing phenol, *o*-cresol, *m*-cresol or *p*-cresol with formaldehyde and glycine in the presence of sodium acetate and acetic acid at 60–80°. The free acid in case of phenol and *o*-cresol were obtained by pouring the condensed product into crushed ice, while those in case of *m*- and *p*-cresol by pouring the condensed product into ice cold water and tap water, respectively. The crude product in each case thus obtained was purified by dissolving it in a requisite quantity of concentrated sodium hydroxide solution followed by its reprecipitation by 50% hydrochloric acid. The relevant analytical details of the various derivatives thus prepared are recorded in Table 1.

Reaction between $PhSn(OPr^i)_3$ and H_2hbg; 1:1 Molar Ratio

A mixture of $PhSn(OPr^i)_3$ (3.3313 g; 8.94 m mole) and H_2hbg (1.6102 g; 8.88 m mole) in dry benzene (60 ml) taken in a R.B. flask was refluxed on a wax bath (90–100°), using a fractionating column. After *ca.* 6h of reflux, the isopropanol liberated during the course of reaction was removed azeotropically and determined by an oxidimetric method[8,9]. On completion of the reaction the excess of solvent from the reaction mixture was removed in *vacuo*, when the product, $PhSn(OPr^i)hbg$ isolated as a pink coloured solid which was washed with dry benzene (3-4 times) and then by dry ether (3-4 times) to remove excess of $PhSn(OPr^i)_3$ and finally dried under suction. The product was found to be insoluble in common organic solvents like ethanol, benzene, toluene, chloroform, dimethylformamide and dimethylsulphoxide etc.

TABLE 1

ANALYTICAL DATA OF VARIOUS N-[*o*-HYDROXY SUBSTITUTED(OR H) BENZYL] GLYCINES AND THEIR MONOPHENYLTIN DERIVATIVES

S. No.	Compound/ molar ratio/ Colour	Reflux Time (h)	Yield (%)	M.p. (°C)	Azeotropic analysis	Analysis % found (calcd)			
					Pr^iOH(g) found (calcd)	C	H	N	Sn
1.	H_2hbg (off white)	—	61.2	50	—	59.33 (59.54)	6.15 (6.18)	7.70 (7.72)	—
2.	H_2hmbg–3 (off white)	—	63.9	190	—	61.33 (61.45)	6.63 (6.66)	7.14 (7.17)	—
3.	H_2hmbg–6 (off white)	—	67.2	88	—	61.35 (61.45)	6.70 (6.66)	7.15 (7.17)	—
4.	H_2hmbg–5 (off white)	—	69.1	148	—	61.32 (61.45)	6.63 (6.66)	7.14 (7.17)	—
5.	PhSn(Opr^i)(hbg) (1:1) (Pink)	6	92.9	220	0.96 (1.06)	50.08 (49.75)	4.81 (4.84)	3.21 (3.22)	27.45 (27.34)
6.	PhSn(Opr^i)(Hhbg)$_2$ (1:2) (Pink)	8	91.2	213	0.75 (0.76)	52.89 (52.64)	4.37 (4.39)	4.57 (4.55)	19.20 (19.29)
7.	PhSn(Hhbg)$_3$ (1:3) (Light pink)	12	90.6	235	1.05 (1.17)	54.08 (53.76)	4.77 (4.75)	5.73 (5.70)	16.20 (16.12)
8.	PhSn(Opr^i)(hmbg–3) (1:1)(Yellowish brown)	5	92.1	205	0.09 (1.05)	51.07 (50.87)	5.16 (5.13)	3.11 (3.12)	26.66 (26.48)
9.	PhSn(Opr^i)(Hhmbg–3)$_2$ (1:2) (Light pink)	7	88.6	195	0.79 (0.80)	54.31 (54.08)	5.56 (5.59)	4.38 (4.35)	18.50 (18.44)
10.	PhSn(Hhmbg–3)$_3$ (1:3) (Pink)	10	90.1	223	1.00 (1.12)	55.69 (55.48)	5.29 (5.27)	5.36 (5.39)	15.30 (15.24)
11.	PhSn(OPr^i)(hmbg–6) (1:1) (Light pink)	6	89.8	195	0.94 (0.97)	51.68 (50.87)	5.15 (5.13)	3.13 (3.12)	26.60 (26.48)
12	PhSn(OPr^i)(Hhmbg–6)$_2$ (1:2) (Pink)	9	90.0	215	0.75 (0.77)	54.33 (54.08)	5.70 (5.59)	4.38 (4.35)	18.51 (18.44)
13.	PhSn(Hhmbg–6)$_3$ (1:3) (Pale-yellow)	12	91.2	225	0.96 (1.07)	55.71 (55.48)	5.45 (5.27)	5.36 (5.39)	15.16 (15.24)
14.	PhSn(OPr^i)(hmbg–5) (1:1) (Pink)	4	89.0	230	0.96 (1.06)	50.69 (50.87)	5.11 (5.13)	3.28 (3.12)	26.63 (26.48)
15.	PhSn(OPr^i)(Hhmbg–5)$_2$ (1:2) (Yellow)	6	91.3	218	0.84 (0.87)	54.34 (54.08)	5.69 (5.59)	4.38 (4.35)	18.34 (18.44)
16.	PhSn(Hhmbg–5)$_3$ (Light yellow)	10	89.6	143	1.09 (1.17)	55.68 (55.48)	5.30 (5.27)	5.42 (5.39)	15.30 (15.24)

Abbreviations**:** Ph = C_6H_5, $OPr^i = OC_3H_7$; $H_2hbg = HOC_6H_4CH_2\overset{+}{N}H_2CH_2COO^-$ and $H_2hmbg–3$(or –6 or –5) = $HOC_6H_3(CH_3)CH_2\overset{+}{N}H_2CH_2COO^-$

It may be mentioned here that since $PhSn(OPr^i)_3$ is soluble in benzene, while H_2hbg is insoluble, the latter was taken in slightly less than the required stoichiometric amount in each case (as evident from the weights given before) in order to avoid contamination of impurities likely to occur by the unreacted H_2hbg. The amount of isopropanol liberated was, therefore, calculated according to the amount of I taken in each case. The analytical and spectral data are recorded in Tables 1–3.

RESULTS AND DISCUSSION

The various reactions occurring between monophenyltin triisopropoxide and H_2hbg may be illustrated as:

$$PhSn(OPr^i)_3 + H_2hbg \rightarrow PhSn(OPr^i)(hbg) + 2Pr^iOH \qquad (1)$$

$$PhSn(OPr^i)_3 + 2H_2hbg \rightarrow PhSn(OPr^i)(Hhbg)_2 + 2Pr^iOH \qquad (2)$$

$$PhSn(OPr^i)_3 + 3H_2hbg \rightarrow PhSn(Hhbg)_3 + 3Pr^iOH \qquad (3)$$

Identical course of reactions followed in case of H_2hmbg–3, H_2hmbg–6 and H_2hmbg–5.

Spectral studies

N-[*o*-Hydroxy Substituted (or H) Benzyl] Glycines

The constitution of these derivatives were earlier reported[7] by their alkylation followed by oxidation and then identifying the resultant acid in each case. Further characterization of these derivatives has now been achieved by IR and PMR spectral measurements, which indicate that these derivatives exist in zwitter ionic form (I). The characteristic IR frequencies and the PMR spectral data (in DMSO d_6) of these compounds are summarized in Tables 2 and 3, respectively.

Monophenyltin Derivatives of N-[*o*-Hydroxy Substituted (or H) Benzyl] Glycines

1:1 Derivatives

As mention before, only the IR spectral studies could be made in case of these derivatives.

The derivative, $PhSn(OPr^i)(hbg)$ displays a broad band between 3200–3000 cm^{-1} which may be attributed to the overlapping of aromatic νC–H and νN–H[10,11]. The band due to νOH (phenolic) is observed to be absent here, indicating the participation of phenolate oxygen in bonding with tin. The appearance of νN–H in the lower region together with the absence of the band due to the $>\overset{+}{N}H_2$ group (as observed in H_2hbg) suggests possible coordination of nitrogen to tin[12]. A very strong band at 1630 cm^{-1} may be assigned to the overlapping of $\nu_{as}COO$[13], aromatic νC=C and N-H deformation. A weak broad band at 1380 cm^{-1} appears because of ν_sCOO and C-H bending due to the *gem* dimethyl structure of the isopropoxy group[11,13]. A shift of 20 cm^{-1} in ν_sCOO, as compared to H_2hbg, indicates the bonding of carboxylate oxygen with tin. Further,

TABLE 2
CHARACTERISTIC INFRARED BAND (cm^{-1}) OF N-[*o*-HYDROXY SUBSTITUTED (OR H) BENZYL] GLYCINES AND THEIR MONOPHENYLTIN DERIVATIVES

S. No.	Compound	νOH/and aromatic νC–H	νN–H and aromatic νC–H	νC–H of the –CH_2– and –CH_3– groups	νN–H of the NH_2 group	ν_{as}COO	ν_sCOO	ΔνCOO	νSn–C	νSn–O'	νSn–N
1.	H_2hbg	3450– 3000 (vb)	—	2940 (w), 2850 (w)	2600 (wb)	1630 (vsb)	1400 (s)	—	—	—	—
2.	H_2hmbg–3	3600– 3000 (vb)	—	2950 (vb), 2855 (wb)	2390 (wb)	1635 (vsbsh)	1400 (m)	—	—	—	—
3.	H_2hmbg–6	3500– 3000 (vb)	—	2910 (mb), 2860 (wb)	2375 (wb)	1640 (vsbsh)	1405 (m)	—	—	—	—
4.	H_2hmbg–5	3500– 3000 (vb)	—	2910 (mb) 2870 (w)	2380 (w)	1635 (vsbsh)	1400 (s)	—	—	—	—
5.	PhSn(OPr^i)(hbg)	—	3200– 3000 (b)	2950 (w), 2850(w)	—	1630 (vs)	1380 (w)	250	1040 (mb)	575 (s) 490 (w)	450 (s)
6.	PhSn(OPr^i)(Hhbg)$_2$	3450– 3300 (b)	3200– 3000 (wb)	2940 (w), 2850 (w)	—	1630 (vs)	1390 (m)	240	1060 (m)	570 (w) 530 (w)	450 (s)
7.	PhSn(Hhbg)$_3$	3500– 3300 (b)	3200– 3000 (wb)	2960 (w), 2840 (w)	—	1630 (vs)	1390 (mb)	240	1050 (s)	580 (mb)	460 (s)
8.	PhSn(OPr^i)(hmbg–3)	—	3200– 3000 (b)	2950 (w), 2860 (w)	—	1625 (vs)	1390(w)	235	1040 (mb)	560 (mb)	450 (s)
9.	PhSn(OPr^i)(Hhmbg–3)$_2$	3450– 3300 (b)	3200– 3050 (wb)	2940 (w), 2850 (w)	—	1625 (vs)	1390 (m)	235	1070 (s)	570 (w) 530 (w)	450 (s)
10.	PhSn(Hhmbg–3)$_3$	3450– 3300 (b)	3200– 3000 (wb)	2940 (w), 2850 (w)	—	1625 (vs)	1380 (mb)	245	1070 (s)	560 (mb)	460 (s)

11. PhSn(OPr^i)(hmbg–6)	—	3200– 3050 (b)	2960 (w), 2840 (w)	—	1630 (vs)	1380 (w)	250	1050 (m)	560 (mb) 490 (w)	450 (s)
12. PhSn(OPr^i)(Hhmbg–6)$_2$	3500– 3300 (b)	3200– 3000 (wb)	2950 (w), 2840 (w)	—	1630 (vs)	1380 (w)	250	1070 (s)	570 (w) 530 (w)	450 (s)
13. PhSn(Hhmbg–6)$_3$	3500– 3300 (b)	3200– 3000 (wb)	2940 (w), 2850 (w)	—	1630 (vs)	1395 (mb)	235	1060 (s)	570 (mb)	450 (s)
14. PhSn(OPr^i)(hmbg–5)	—	3200– 3000 (b)	2950 (w), 2850 (w)	—	1630 (vs)	1390 (w)	240	1050 (m)	570 (mb) 500 (w)	460 (s)
15. PhSn(OPr^i)(Hhmbg–5)$_2$	3600– 3350 (b)	3220– 3000 (b)	2960 (w), 2850 (w)	—	1635 (vs)	1385 (m)	250	1060 (s)	570 (m) 530 (w)	450 (s)
16. PhSn(Hhmbg–5)$_3$	3600– 3300 (b)	3200– 3030 (wb)	2960 (w), 2840 (w)	—	1630 (vs)	1380 (mb)	250	1070 (s)	560 (mb)	450 (s)

Abbreviations: w = weak, m = medium, wb = weak broad, mb = medium broad, b = broad, vb = very broad, s = sharp, vs = very sharp, vsb = very strong broad and vsbsh = very strong broad with a shoulder.

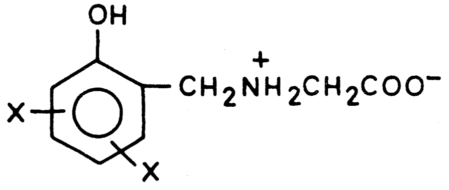
TABLE 3

PROTON MAGNETIC RESONANCE SPECTRAL DATA (δ value) OF N-[*o*-HYDROXY SUBSTITUTED (OR H) BENZYL] GLYCINES AND THEIR 1 : 2 MONOPHENYLTIN DERIVATIVES.

S. No.	Compound	Aromatic ring	Phenolic OH	$>\overset{+}{N}H_2$	>NH	$-CH_3$ attached with benzene ring	$-CH_2$–attached with		Isopropoxy (*gem* dimethyl)
							benzene ring	glycine part	
1.	H_2hbg	6.50–7.30 (q)	4.50–5.90 (h)	3.60 (s)	—	—	3.25 (s)	2.45 (s)	—
2.	H_2hmbg–3	6.50–7.10 (t)	4.50–5.70 (h)	3.55 (s)	—	3.15 (s)	3.00 (s)	2.15 (s)	—
3.	H_2hmbg–5	6.40–7.10 (t)	4.40–5.60 (h)	3.65 (s)	—	3.20 (s)	3.10 (s)	2.15 (s)	—
4.	H_2hmbg–6	6.50–7.00 (t)	4.50–5.50 (h)	3.70 (s)	—	3.25 (s)	3.00 (s)	2.15 (s)	—
5.	$PhSn(OPr^i)(Hhbg)_2$	6.70–7.30 (m)	4.00–4.40 (h)	—	3.80 (bs)	—	2.80 (s)	2.15 (s)	0.98–1.10 (d)
6.	$PhSn(OPr^i)(Hhmbg–3)_2$	6.50–7.20 (m)	4.20–4.80 (h)	—	3.90 (h)	3.20 (s)	3.00 (s)	2.15 (s)	0.90 (d)
7.	$PhSn(OPr^i)(Hhmbg–6)_2$	6.70–7.50 (m)	4.20–4.70 (h)	—	3.90 (h)	3.25 (s)	2.90 (s)	2.16 (s)	1.10 (d)
8.	$PhSn(OPr^i)(Hhmbg–5)_2$	6.80–7.20 (m)	4.50–5.20 (h)	—	3.90 (h)	3.20 (s)	3.00 (s)	2.15 (s)	1.00 (d)

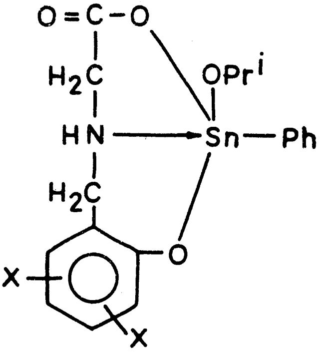
Abbreviations: s = singlet, bs = broad singlet, d = doublet, m = multiplet and h = hmp the separation value, $\Delta\nu COO$ ($\nu_{as}COO-\nu_{s}COO$) of 250 cm^{-1}, rules out the possibility of a bridged or coordinated carboxylate group[12,14]. A medium broad band between 1270–1240 cm^{-1} may be attributed to the possible overlapping of $\nu C-N$ and $\nu C-O$[11]. A weak band at 1160 cm^{-1} indicates $\nu C-O$ of the isopropoxy group, while the absorptions at 1120 cm^{-1}, 1110 cm^{-1} and 1100 cm^{-1} correspond to the aromatic C-H-in-plane bending modes[21]. The medium broad band at 1040 cm^{-1} occurs because of $\nu Sn-C$[15,16], while the strong absorptions at 900 cm^{-1} and 700 cm^{-1} may be assigned to the aromatic C-H out of plane bendings[10]. A strong peak at 760 cm^{-1} shows one of the characteristic absorptions for an *ortho* disubstituted benzene nucleus[11]. A medium band at 575 cm^{-1} and a weak band at 490 cm^{-1} may be attributed to $\nu_{as}Sn-O$ and $\nu_{s}Sn-O$[16], respectively, while a strong band at 450 cm^{-1} corresponds to $\nu Sn-N$[16]. On the basis of the above observations, it appears that the tin atom in PhSn(hbg)(OPr^i) shows penta-coordination as a result of bonding with one of the oxygens from the carboxylate group, the nitrogen from the imino group and the phenolate oxygen, along with an isopropoxy and one phenyl group.

The IR spectra of other 1:1 derivatives viz. PhSn(OPr^i) (hmbg-3), PhSn(OPr^i) (hmbg-6) and PhSn(Opr^i) (hmbg-5) were interpreted similarly which indicate identical modes of bonding, as those observed in PhSn(OPr^i)(hbg) (II).



Where X=-H Or -CH3

(I)



Where X=-H Or -CH3

(II)

1:2 Derivative

In respect of these derivatives it was possible to have both the infrared and proton magnetic resonance spectral measurements.

Infrared Spectra

A borad band in the region 3450–3300 cm^{-1} in PhSn(OPr^i)($Hhbg)_2$ indicates νOH of the unbounded phenolic group. The absorption due to the $>\overset{+}{N}H_2$ group, as observed in H_2hbg, is found to disappear here. The band between

3200–3000 cm^{-1} shows possible overlapping of aromatic νC–H and νN–H. The appearance of νN–H in the lower region suggests possible coordination of nitrogen to tin. The weak absorptions at 2940 cm^{-1} and 2850 cm^{-1} may be ascribed to νC–H of the $-CH_3$ and $-CH_2$ groups. A very strong band at 1630 cm^{-1} may be attributed to the overlapping of $\nu_{as}COO$, aromatic νC=C and N-H deformation, while the weak bands at 1505 cm^{-1} and 1475 cm^{-1} occur because of the aromatic skeletal modes. A medium band at 1390 cm^{-1} corresponds to ν_sCOO and C-H bending of the *gem* dimethyl structure of the isopropoxy group. A shift of 10 cm^{-1} in ν_sCOO, as compared to H_2hbg, suggests the bonding of the carboxylate oxygen with tin. Further, the separation value, ΔνCOO of 240 cm^{-1} shows the absence of bridged or coordinated carboxylate groups. The bands at 1260 cm^{-1} and 1230 cm^{-1} may be assigned to νC–N and νC–O, respectively. The weak bands at 1150 cm^{-1} and 1110 cm^{-1} indicate the aromatic C-H-in-plane bending, while a medium peak at 1060 cm^{-1} corresponds to νSn–C. The weak absorptions at 860 cm^{-1}, 820 cm^{-1}, 730 cm^{-1} and 700 cm^{-1} may be attributed to the aromatic C-H out of plane bending, while a medium band at 750 cm^{-1} shows one of the characteristic absorption for an *ortho* disubstituted benzene ring. The weak bands at 570 cm^{-1} and 530 cm^{-1} occur due to ν_{as}Sn–O and ν_sSn–O, respectively, while a strong band at 450 cm^{-1} corresponds to νSn–N. It is thus evident that the tin atom in $PhSn(OPr^i)(Hhbg)_2$ displays hexa-coördination by way of bonding with one of the oxygens from each of the two carboxylate groups, the nitrogen from each of the two imino groups available from two moles of H_2hbg, along with an isopropoxy and one phenyl group.

Proton magnetic resonance spectra

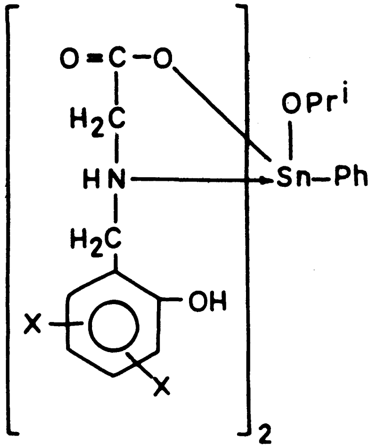
The PMR spectrum of $PhSn(OPr^i)(Hhbg)_2$ shows a multiplet in the region δ 6.70–7.30, which may be attributed to the aromatic protons.[17,18] The hump between δ 4.00–4.40 corresponds to the proton of the unbounded phenolic groups[11]. A singlet at δ 3.60 due to the $>\overset{+}{N}H_2$ protons, as observed in H_2hbg, disappears here and a new broad signal observed at δ 3.80 corresponding to the >NH group proton (obtained as a result of deprotonation of the $>\overset{+}{N}H_2$ group) suggests possible coordination of nitrogen to tin. The singlets at δ2.80 and δ2.15 may be assigned to the protons of the $-CH_2-$ groups attached to the benzene nucleus and the glycine part of H_2hbg, respectively[11,17], while a doublet between δ0.98–1.10 corresponds to the gem dimethyl protons of the isopropoxy group[19]. Thus, the inferences drawn here are in conformity to those derived from the IR spectral measurements before.

The other 1:2 derivatives *viz.* $PhSn(OPr^i)(Hhmbg–3)_2$, $PhSn(OPr^i)(Hhmbg–6)_2$ and $PhSn(OPr^i)(Hhmbg–5)_2$ show identical modes of bonding, as those observed in $PhSn(OPr^i)(Hhbg)_2$ (III).

1:3 Derivatives

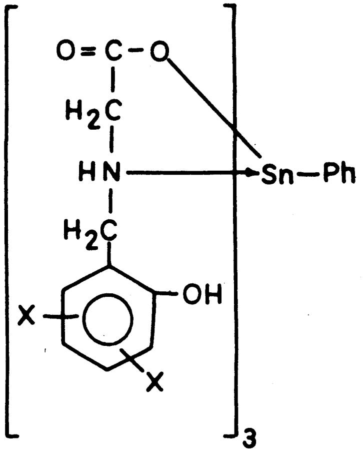
Here, again only the ir spectral studies could be made and the main findings relating to the structures of the various 1:3 derivatives are as under:

The derivatives, $PhSn(Hhbg)_3$, $PhSn(Hhmbg–3)_3$, $PhSn(Hhmbg–6)_3$ and



Where X=-H Or-CH_3

(III)



Where X=-H Or-CH_3

(IV)

PhSn(Hhmbg–5)$_3$(IV) contain a heptacoordinated tin atom in each case as a consequence of bonding with one of the oxygens from each of three carboxylate groups and the nitrogen from each of the three imino groups available from three moles of I, along with one phenyl group.

RERERENCES

1. M. Chandra, *Transition Metal Chem.*, **6**, 17, 25, 276 (1983).
2. M. Chandra and A.K. Dey, *Chim Acta Hung.*, **124** 259 (1987).
3. (a) Yashmani Gupta, Tripti Gangwar, Robina Aman and M. Chandra *Asian J. Chem.* **6**, 484 (1994)
   (b) A.I. Vogel, A Text Book of Practical Organic Chemistry, 4th Edn., Longmans, London (1960).
4. C.K. Narula and V.D. Gupta, *Synth. React. Inorg. Met. Org. Chem.*, **11**, 133 (1981).
5. R.C. Poller, The Chemistry of Organotin Compound, Logos Press, London (1978).
6. C.L. Wilson and D.W. Wilson, Comprehensive analytical Chemistry Elsevier, London (1960).
7. K. Kumar, N.D. Pandey and J.K. Mehrotra, *J. Indian Chem. Soc.*, **51**, 944 (1974).
8. D.C. Bradley, F.M.A. Halim and W. Wardlaw, *J. Chem. Soc.*, 3450 (1950).
9. R.C. Mehrotra, *J. Indian Chem. Soc.*, **31**, 904 (1954).
10. L.J. Bellamy, The Infrared Spectra of Complex Molecules, Methuen, London (1962).
11. R.M. Silverstein, G.C. Bassler and T.C. Morrill, Spectroscopic Identification of Organic Compounds, John Wiley, New York (1981).
12. B.Y.K. Ho and J.J. Zuckerman, *Inorg. Chem.*, **12**, 1552 (1973).
13. G.K. Sandhu, N.S. Bopari and S.S. Sandhu, *Synth. React. Inorg. Met. Org. Chem.*, **10**, 535 (1980).

14. K. Nakamoto, Infrared and Raman Spectra of Inorganic and Coordination Compounds, John Wiley, New York (1978).
15. R.C. Poller, *J. Inorg. Nucl. Chem.*, **24**, 593 (1962).
16. J. Wang, F.Q. Liu, Y. Zhang, R. Wang, H.G. Wang and X.K. Yao, *J. Organometal Chem.*, **375**, 173 (1989).
17. Asahi Research Centre Co. Ltd. Tokyo (Edited by), Hand Book of Proton NMR and Data, Vols. 1–3, Academic press, Japan (1985).
18. T.N. Srivastava and J.D. Singh, *Indian J. Chem.*, **24A**, 433 (1982).
19. R. Ramchandran, B. Singh, A.K. Narula, P.N. Kapoor, P.K. Gupta and R.N. Kapoor *Polyhedron*, **4**, 1007 (1985).

(*Received:* 9 January 1993; *Accepted:* 23 September 1993) AJC-684