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DIORGANOTIN(IV) DERIVATIVES OF *N*-PHTHALOYL AMINO ACIDS

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Summary

Twentyfour complexes of the general formulae (R_2SnL_2 and $R_2(L)SnOSn(L)R_2$ (L = *N*-phthaloyl derivative of L-leucine, DL-alanine and L-phenylalanine; R = CH_3, C_2H_5, n-C_4H_9 and n-C_8H_{17}) have been prepared by reacting ligand and dialkyltin(IV) oxide in 2/1 and 1/1 (ligand/metal) molar ratio. These complexes have been characterised by elemental analysis and structures assigned with the help of infrared, ^1H NMR and ^{119}Sn Mössbauer spectroscopy. These data support six-coordinated distorted octahedral structures with two alkyl groups in *trans* positions.

Introduction

Several triorganotin(IV) derivatives of amino acids, dipeptides and the X-ray structure of $Me_3Sn \cdot$ glycine have been reported [1–5]. The dimethyltin(IV) derivatives of glycine and β-alanine have been studied by using infrared and Mössbauer data [6]. Diorganotin(IV) derivatives of sulfur-containing amino acids have been studied by variable temperature Mössbauer data [7,8]. A distorted trigonal bipyramidal structure to chloro(ethyl-1-cysteinato-*N*,*S*)dimethyltin(IV) has been assigned on the basis of X-ray studies [9]. Recently, six- and five-coordinate (2/1 and 1/1, ligand/metal) diorganotin(IV) complexes of *N*-phthaloyl-glycine and *N*-benzoyl-glycine [10], *N*-acetylamino acids [11], have been prepared and characterized by infrared and Mössbauer data. The carboxylate group of the *N*-protected amino acids used in this communication acts as a bidentate group unlike its unidentate nature reported in the unprotected amino acid [6].

Experimental

N-Phthaloyl-DL-alanine, *N*-phthaloyl-L-leucine, *N*-phthaloyl-L-phenylalanine and diethyltin oxide were prepared by the reported methods [12,13]. Dimethyl, di-n-butyl,

and di-n-octyltin oxides (Alfa Products, U.S.A.) were used as such.

Melting points were determined in open capillaries and are uncorrected. Carbon, hydrogen and nitrogen analysis were carried out by the microanalytical service, Calcutta University. Tin was estimated gravimetrically as SnO_2. Molecular weights were determined cryoscopically in benzene as well as the Rast method in molten camphor. Infrared spectra (4000–200 cm^{-1}) (KBr) were recorded on a Perkin–Elmer 577 spectrophotometer. ^1H NMR spectra were recorded on Tesla BS 487 (80 MHz) in $CDCl_3$ with TMS as the internal standard. Mössbauer spectra were recorded with a Harwell 6000 series spectrometer with sample cooled by liquid nitrogen (ca. 80 K) and source (Pd–Sn) at room temperature. Isomer shifts are relative to SnO_2 measured at room temperature.

Preparation of the complexes

The title compounds in 2/1 and 1/1 (ligand/metal) molar ratio were prepared by refluxing the ligand and the diorganotin oxide in a mixed solvent (dry benzene (30 cm^3) and absolute ethanol (10 cm^3)) for 3–4 h and water formed during the reaction was removed azeotropically. The resulting solution was filtered and the solvent was removed under reduced pressure to obtain a white solid except in case of di-n-octyltin(IV) derivatives where a syrup was obtained which was kept under vacuum for 2–3 d when a white solid was obtained. All the complexes were recrystallized from methanol.

Results and discussion

In the present study, diorganotin(IV) complexes of *N*-phthaloyl-L-leucine (L_1), *N*-phthaloyl-DL-alanine (L_2) and *N*-phthaloyl-L-phenylalanine (L_3) have been prepared and 2/1 and 1/1 stoichiometries were assigned by elemental analysis (Table 1). All the complexes are colourless and soluble in common organic solvents such as benzene, alcohol, methanol and chloroform. Molecular weight determination in benzene shows various degrees of polymerization ($n = 1,2,3$ or 4 for different complexes) while all the complexes exist as monomers in molten camphor (175°C) (Table 2). Low degree of polymerization for the complexes is further supported by their solubility in common solvents.

Infrared data

Infrared spectra of the ligands and their complexes have been recorded in KBr (4000–200 cm^{-1}). The stretching frequencies for imido C=O, acid COO, Sn–C, Sn–O, Sn–N and Sn–O–Sn bonds are given in Table 3. The deprotonation of the carboxylic group in the complexes is evident from the disappearance of a broad band due to COOH group in the 3100–2500 cm^{-1} region. Presence of a broad band (3600–3400 cm^{-1}) in the complexes (Complexes **1**, **4**, **8**–**10**, **12**, **14**, **21**, Table 3) indicates water molecule. The remaining bands due to water are overlapped by ligand absorptions.

$\nu(C{=}O)_{imido}$ and $\nu(COO)_{acid}$. In the spectra of the ligands and the complexes, asymmetric and symmetric stretching modes of imido C=O are observed around 1720 and 1780–1770 cm^{-1} respectively, indicating non-participation of the imido C=O in complex formation. The $\Delta\nu$-value, $[\Delta\nu = \nu_{asym}(COO) - \nu_{asym}(COO)]$, 200 ±

10 cm^{-1} is lower in the spectra of all the complexes as compared to that observed in the spectra of the free ligands ($\Delta\nu$ 320 cm^{-1}) and is comparable to $\Delta\nu$-value (205 cm^{-1}) of sodium salts of the ligands which clearly indicates bidentate nature of the carboxylate [14]. However, in the case of 2/1 complexes a strong band present in the free carboxylate region (1710–1705 cm^{-1}) shows that in these complexes the second carboxylate group is bonded to tin(IV) in a unidentate manner [1,15].

ν(Sn–C), ν(Sn–O), ν(Sn–N) and ν(Sn–O–Sn). The presence of only one Sn–C absorption band in the spectra of the complexes in the 600–500 cm^{-1} region, indicates a linear configuration of the C–Sn–C moiety [6,14]. A broad band in the vicinity of 500 cm^{-1} is assigned to Sn–O bond [16] while a medium to weak intensity band around 470–430 cm^{-1} is attributed to Sn–N stretching mode [17]. In case of 1/1 complexes a strong broad band in 650–630 cm^{-1} region is assigned to Sn–O–Sn bond [18].

^1H NMR data

The ^1H NMR spectra of the ligands and complexes have been recorded in $CDCl_3$ (δ, ppm; Table 4). The absence of signal (9.1–8.5 ppm) in the spectra of all of the complexes indicates the replacement of the carboxyl proton by tin(IV). In the dimethyltin(IV) complexes the presence of a single methyl resonance show *trans* configuration of the two methyl groups. The coupling constant $J(^{119}Sn–C–^1H)$ for the dimethyltin(IV) derivatives with phthaloyl-DL-alanine and phthaloyl-L-leucine in 1/2 ratio are 67 and 90 Hz respectively which are consistent with a higher than four-coordinated structure in solution. In the diethyltin(IV) complexes, a single triplet due to the CH_3 protons further supports the *trans* position of the alkyl groups.

Di-n-butyl and di-n-octyltin(IV) complexes show two very broad signals due to the alkyl groups. The resonance due to the CH_3 protons of the two ligands, L-leucine and DL-alanine gets obscured by the alkyl protons in case of some of the complexes and become difficult to be identified. However, the integration area is equivalent to the number of protons calculated from the proposed structure in case of all the complexes.

Mössbauer data

In diorganotin(IV) complexes when the donor atoms are highly electronegative the *QS* is mainly determined by the C–Sn–C bond angle and distortion from a regular six-coordination gives values similar to those for five-coordination [19–29]. The Mössbauer data (*IS* 1.24–1.55 and *QS* 3.10–3.70 mm s^{-1}) for diorganotin(IV) carboxylates, di-n-butyltin(IV) diacetate (*IS* 1.36; *QS* 3.56 mm s^{-1}) and di-n-butyltin(IV) maleate (*IS* 1.38; *QS* 3.74 mm s^{-1}) support a distorted *trans* octahedral geometry around tin(IV) [30,31]. Me_2Sn(IV) Salen (*IS* 1.13; *QS* 3.46 mm s^{-1}) has been assigned a distorted octahedral structure with the two Sn–C bonds symmetrically bent towards the oxygen atoms [25]. In the present case the observed *QS* values while indicating a *trans* structure are considerably lower than expected. This is consistent with the distortion from a regular octahedral arrangement as discussed. This is further supported by the C–Sn–C bond angle values (172–138°) calculated using Sham and Bancroft method [32].

Interpretation of the Mössbauer data for the complexes **2,3** and **14** (Table 5) is a little different. For these three complexes the *IS* values are comparatively lower than

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TABLE 1

PHYSICAL AND ANALYTICAL DATA OF DIALKYLTIN(IV) COMPLEXES WITH *N*-PHTHALOYL AMINO ACIDS

Complex [a]	Yield (%)	M.p. (°C)	Analysis (Found (calcd.)(%))			
			C	H	N	Sn
$(L_1)_2Sn(CH_3)_2 \cdot H_2O$ (**1**)	75	170–175	52.09 (52.42)	5.16 (5.24)	–	17.89 (17.28)
$(L_2)_2Sn(CH_3)_2$ (**2**)	53	190–195	50.44 (49.26)	3.93 (3.76)	–	19.78 (20.30)
$(L_3)_2Sn(CH_3)_2$ (**3**)	80	257–260	58.41 (58.64)	4.25 (4.07)	4.11 (3.80)	15.64 (16.11)
$(L_1)_2Sn(C_2H_5)_2 \cdot 3H_2O$ (**4**)	70	265–266	51.21 (51.15)	6.18 (5.86)	3.91 (3.73)	16.32 (15.81)
$(L_2)_2Sn(C_2H_5)_2$ (**5**)	80	210	50.55 (50.92)	3.64 (4.24)	–	18.78 (19.37)
$(L_3)_2Sn(C_2H_5)_2$ (**6**)	60	200	60.22 (59.63)	4.35 (4.45)	4.35 (3.66)	16.03 (15.52)
$(L_1)_2Sn(n\text{-}C_4H_9)_2$ (**7**)	70	200–206	58.00 (57.39)	6.44 (6.11)	4.30 (3.72)	15.60 (15.77)
$(L_2)_2Sn(n\text{-}C_4H_9)_2 \cdot 3H_2O$ (**8**)	60	170–173	50.70 (49.81)	–	–.	16.70 (16.42)
$(L_3)_2Sn(n\text{-}C_4H_9)_2 \cdot 4H_2O$ (**9**)	75	208–210	56.82 (56.46)	5.98 (5.60)	3.40 (3.14)	–
$(L_1)_2Sn(n\text{-}C_8H_{17})_2 \cdot 3H_2O$ (**10**)	55	106–108	58.01 (57.47)	7.31 (7.40)	3.65 (3.05)	13.69 (12.92)
$(L_2)_2Sn(n\text{-}C_8H_{17})_2$ (**11**)	75	95–98	58.87 (58.72)	6.42 (6.40)	3.58 (3.59)	15.10 (15.20)

$(L_3)_2Sn(n\text{-}C_8H_{17})_2 \cdot 4H_2O$ (**12**)	70	55	60.00 (59.72)	–	3.12 (2.79)	–
$[L_1Sn(CH_3)_2]_2O$ (**13**)	70	255	45.71 (46.08)	4.91 (4.73)	3.43 (3.36)	29.14 (28.49)
$[L_2Sn(CH_3)_2]_2O \cdot H_2O$ (**14**)	75	260	40.50 (40.63)	4.85 (3.91)	4.26 (3.65)	31.80 (30.94)
$[L_3Sn(CH_3)_2]_2O$ (**15**)	57	223	51.26 (50.59)	4.65 (3.99)	3.16 (3.11)	26.01 (26.34)
$[L_1Sn(C_2H_5)_2]_2O$ (**16**)	80	260	48.44 (48.57)	5.00 (5.40)	3.85 (3.15)	27.14 (26.69)
$[L_2Sn(C_2H_5)_2]_2O$ (**17**)	94	245	45.07 (44.70)	5.27 (4.47)	3.75 (3.48)	29.00 (29-48)
$[L_3Sn(C_2H_5)_2]_2O$ (**18**)	80	230	52.12 (52.64)	4.47 (4.60)	3.39 (2.92)	–
$[L_1Sn(n\text{-}C_4H_9)_2]_2O$ (**19**)	70	106–108	52.01 (52.73)	7.02 (6.39)	–	24.23 (23.71)
$[L_2Sn(n\text{-}C_4H_9)_2]_2O$ (**20**)	85	175–180	50.60 (49.71)	5.90 (5.67)	3.85 (3.05)	25.24 (25.88)
$[L_3Sn(n\text{-}C_4H_9)_2]_2O \cdot H_2O$ (**21**)	60	206	55.87 (55.18)	5.89 (5.70)	3.25 (2.57)	–
$[L_1Sn(n\text{-}C_8H_{17})_2]_2O$ (**22**)	70	80–90	59.64 (58.76)	7.94 (7.83)	–	18.84 (19.37)
$[L_2Sn(n\text{-}C_8H_{17})_2]_2O$ (**23**)	80	50–56	57.04 (56.77)	7.49 (7.36)	3.07 (2.45)	–
$[L_3Sn(n\text{-}C_8H_{17})_2]_2O$ (**24**)	60	55	60.99 (61.23)	–	–	–

[a] L_1 = *N*-phthaloyl-L-leucine, L_2 = *N*-phthaloyl-DL-alanine, L_3 = *N*-phthaloyl-L-phenylalanine.

the other complexes, indicating four-coordinate tin(IV) while the *QS* values are in the range observed for other complexes. The ratio $\rho = QS/IS$, is > 3.2, which suggests higher coordination [8,31]. In case of diethyltin(IV) complexes (**4** and **5**, Table 5) the *QS* values are characteristic of a regular *trans* octahedral structure [33].

Thermogravimetric analysis (25–600°C)

In case of complexes **1**, **4**, **8–10**, **12**, **14** and **21** in Table 2, loss of weight between 25–120°C corresponds to the water molecule present in the crystal lattices. No definite conclusion could be drawn about the nature of the other decomposition products.

Structures

$R_2Sn(L)_2$[2/1] complexes. Mössbauer, ^{1}H NMR and infrared data support a six-coordinate distorted octahedral structure (I) with linear C–Sn–C moiety for the monomers. A weak molecular interaction present at low temperature in the case of di, tri and tetramers (complexes **1–4**, **7–9**, Table 2) may be visualized as in structure II.

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TABLE 2

MOLECULAR WEIGHT AND THERMOGRAVIMETRIC ANALYSIS (TGA) DATA

Complex [a]	Molecular weight [b]			n [c]	TGA		
	Found		Calcd.		T(°C)	%loss	Assignment
	C	R					
1	3012	756	686.7	Tetramer	20–120	2.8	$1H_2O$
2	1839	543	584.7	Trimer	–	–	–
3	2161	790	736.7	Trimer	–	–	–
4	2178	727	750.7	Trimer	90–120	7.2	$3H_2O$
5	659	613	612.7	Monomer	–	–	–
6	894	822	764.7	Monomer	–	–	–
7	1403	893	752.7	Dimer	–	–	–
8	1642	726	776	Dimer	80–120	5.6	$\sim 3H_2O$
9	1719	910	892.7	Dimer	100–140	7.5	$\sim 4H_2O$
10	865	1141	918.7	Monomer	80–110	5.8	$3H_2O$
11	835	825	780.7	Monomer	–	–	–
12	985	926	1004.7	Monomer	80–140	7.18	$4H_2O$
13	844	909	833.4	Monomer	–	–	–
14	816	689	767.4	Monomer	90–120	2.3	$1H_2O$
15	1014	931	901.4	Monomer	–	–	–
16	943	920	889.4	Monomer	–	–	–
17	1588	785	805.4	Dimer	–	–	–
18	1092	726	957.4	Monomer	–	–	–
19	2882	861	1001.4	Trimer	–	–	–
20	1800	930	917.4	Dimer	–	–	–
21	1055	1048	1087.4	Monomer	60–80	1.6	$1H_2O$
22	1800	1164	1225.4	Monomer	–	–	–
23	4469	1070	1141.4	Tetramer	–	–	–
24	4019	1060	1293.4	Trimer	–	–	–

[a] See for formulae of the complexes Table 1. [b] C = Cryoscopically; R = Rast method. [c] n = degree of polymerization in benzene.

TABLE 3

INFRARED SPECTRAL DATA (cm^{-1}; in KBr)

Complex [a]	$\nu_{sym}(C{=}O)_{imido}$	$\nu_{asym}(C{=}O)_{amido}$ $\nu_{asym}(COO)_{acid}$	$\nu_{sym}(COO)_{acid}$	ν(Sn–C)	ν(Sn–O)	ν(Sn–N)	ν(Sn–O–Sn)
N-Phthaloyl-L-leucine (L_1)	1775m	1640–1690s,b	1290s	–	–	–	–
N-Phthaloyl-DL-alanine (L_2)	1775s	1740–1690s,b	1390s,b	–	–	–	–
N-Phthaloyl-L-phenylalanine (L_3)	1770	1740–1700s,b	1390s,b	–	–	–	–
Sodium(*N*-pht-DL-alaninate)	1775m,b	1715s,b, 1600s,b	1395s	–	–	–	–
Sodium(*N*-pht-L-leucinate)	1775m,b	1710s,b,1600s,b	1395s,b	–	–	–	–
1	1770m	1720sh, 1710s, 1600s,b	1400s	500m	490m,b	470m,b	–
2	1775m,sh	1720sh, 1710s,b, 1595s,b	1390s	505w,b	495m	445m	–
3	1780s	1715s,b, 1675m, 1595s	1385s	570m	505m	430w,b	–
4	1770m	1715s, 1700sh, 1575s	1385s	540m	490m	460m	
5	1770s	1720sh, 1710s,b, 1600s,b	1390s,b	510w	490w	450s,b	–
6	1780m	1725sh, 1710s,b, 1600s,b	1390s,b	565m	490m	450m	–
7	1780m	1730s, 1710sh, 1600s	1392s	585m	490s	460m	–
8	1776s	1720sh, 1705s,b, 1605s,b	1380s,b	570w,b	490m,b	435m,b	–
9	1780m	1730sh, 1720s, 1600s	1390s	570m	495s	460sh	–
10	1755m	1700sh, 1690s, 1570s	1365s	610sh	520m	465m	–
11	1760s	1710s,b, 1685s, 1570s,b	1380s,b	545m	525s	498s,b	–
12	1775m	1720s, 1650m, 1595s	1385s	570m	485m	450sh	–
13	1780m	1715s, 1590s	1390s	530sh	505s	–	650s
14	1780m	1715s, 1590m	1390s	510w,b	490w,b	430w,b	630s,b
15 [b]	–	–	–	–	–	–	–
16	1770m	1715s, 1595s	1390s	540m	480m	445w	690m
17	1780m	1715s, 1595s	1390s	555m	490s	430w,b	635s
18 [b]	–	–	–	–	–	–	–
19	1775m	1715s, 1590s	1380s	575m	480m	445m	645s
20	1770m	1715s, 1590s	1380s	–	–	–	–
21	1775m	1715s, 1595s	1385s	570m	490m	450m	645sh
22	1775m	1705s,b, 1595s,b	1390s	600m,sh	495s	430w,b	635s
23 [b]	–	–	–	–	–	–	–
24 [b]	–	–	–	–	–	–	–

[a] See for formulae of the complexes Table 1. [b] Owing to their sticky nature, spectra could not be recorded in solid state.

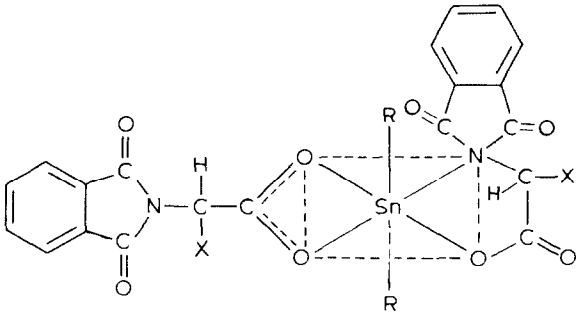
TABLE 4

^{1}H NMR SPECTRAL DATA (in $CDCl_3$; δ, ppm)

Complex [a]	$\delta(C_6H_4)/\delta(C_6H_5)$	δ(CH)	$\delta(CH_2)$	$\delta(CH_3)/\delta$(CH)	δ(Sn–CH_2)	δ(Sn–CH_3)
N-Phthaloyl-L-leucine (L_1)	7.75 (m,4H)	4.98 (q,1H)	2.15 (q,2H)	0.88 (d,6H)	–	–
N-Phthaloyl-DL-alanine (L_2)	7.76 (m,4H)	5.01 (q,1H)	–	1.75 (d,3H)	–	–
N-Phthaloyl-L-phenylalanine (L_3)	7.61 (m,4H)/ 7.11(s,5H)	5.15 (t,1H)	3.50 (d,2H)		–	–
1	7.75 (bm,8H)	4.65 (m,2H)	1.13–1.85 (m,4H)	0.75 (m,12H)/ 0.675(m,2H)	–	0.60 (s,6H)
2	7.62 (m,8H)	4.90 (q,2H)	–	1.63 (d,6H)	1.10 (s,6H)	–
3	7.50(m,8H)/ 7.00(s,10H)	4.80 (t,2H)	3.20 (d,4H)	–	0.70 (s,6H)	–
4	7.68 (m,8H)	4.95 (q,2H)	1.65 (t,4H)	0.90 (d,12H)	1.50–1.10 (bm,4H)	0.87 (t,6H)
5	7.68 (m,8H)	5.00 (q,2H)	–	1.75 (d,6H)	2.25–1.77 (bm,4H)	1.33 (t,6H)
6	7.60(m,8H) 7.11(s,10H)	5.00 (s,10H)	2.42 (d,4H)	–	1.66–1.00 (bm,4H)	0.89 (t,18H)
7	7.75 (m,8H)	4.45–4.90 (bq,2H)	–	–	1.5–1.0 (m,18H)	0.98 (bm,18H)
8	7.65 (m,8H)	4.65 (bq,2H)	–	–	1.70–1.00 (m,18H)	0.80 (t,6H)
9	7.75(m,8H)/ 7 10(s,10H)	4.67 (t,2H)	3.37 (d,4H)	–	1.66–1 10 (bm,12H)	0.85 (t,6H)

10 [b]	7.5–8.0 (m,8H)	4.42–4.80 (m2H)	1.55 (d,4H)	–	1.45–1.00 (m,28H)	0.88 (bm,18H)
11 [b]	7.58(m,8H)	4.80 (bq,2H)	–	1.62 (d,6H)	1.50–1.12 (m,28H)	0.90 (m,6H)
12	7.50(m,8H)/ 7.10(s,10H)	4.80 (bp,2H)	4.33 (bd,4H)	–	1.66–1.00 (bm,28H)	0.93 (bm,6H)
13 [d]	–	–	–	–	–	–
14	7.75 (m,8H)	4.67 (q,2H)	–	1.50 (d,6H)	0.70 (s,12H)	–
15	7.60(m,8H) 7.08(s,10H)	4.88 (t,2H)	3.33 (d,4H)	–	0.70 (s,12H)	–
16	7.75 (m,8H)	4.77 (q,2H)	1.65 (t,2H)	0.90 (d,12H)	1.23 (bm,8H)	0.80 (t,2H)
17	7.77 (m,8H)	4.85 (q,2H)	–	1.62 (d,2H)	2.25–1.75 (bm,8H)	1.25 (t,12H)
18	7.57(m,8H)/ 7.12(s,10H)	4.5–4.9 (m,2H)	–	–	0.5–2.4 (bm,38H)	–
19	7.62 (m,8H)	4.75 (bq,2H)	–	–	1.75–1.00 (bm,30H)	0.75 (m,12H)
20	7.62 (m,8H)	4.75 (bq,2H)	–	–	1.75–1.0 (bm,30H)	0.75 (m,12H)
21	7.55(m,8H)/ 7.08(s,10H)	4.67 (bd,2H)	3.37 (bd,4H)	–	1.50–1.0 (bm,24H)	0.85 (bd,24H)
22	7.60 (bm,8H)	4.5–4.87 (m,2H)	2.25 (bd,4H)	–	1.62–1.00 (bm,56H)	0.85 (bd,24H)
23	7.70 (m,8H)	4.75 (bq,2H)	–	1.62 (d,6H)	1.50–1.00 (bm,56H)	0.90 (bm,12H)
24 [d]	–	–	–	–	–	–

[a] See for formulae of the complexes Table 1. [b] Solvent CCl_4. [c] On a Varian analytical instrument, T-60A, 60 MHz. [d] Insoluble in $CDCl_3$ at low temperatures.



(I)

(X = $CH_2CH(CH_3)_2$, CH_3, $CH_2C_6H_5$)

$R_2(L)Sn–O–Sn(L)R_2$ [1 / 1] complexes. A six-coordinate distorted octahedral structure (III) with a Sn–O–Sn bridge and a linear configuration of C–Sn–C is supported by infrared, ^1H NMR and Mössbauer data for the monomers. Intermolecular association at low temperature for some complexes (**17**, **19**, **20**, **23**, **24**, Table 2) may be represented as in structure IV.

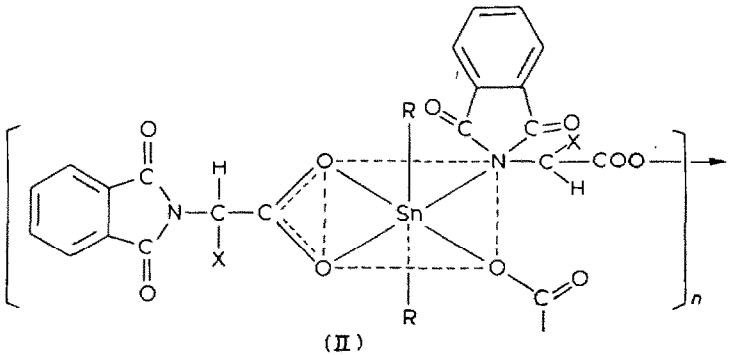
The assignment of these structures is tentative and based on spectroscopic data. It

TABLE 5

^{119m}Sn MÖSSBAUER DATA FOR *N*-PHTHALOYL AMINO ACID DIALKYLTIN(IV) COMPLEXES (at 80 K; *QS* and *IS* in mm s^{-1})

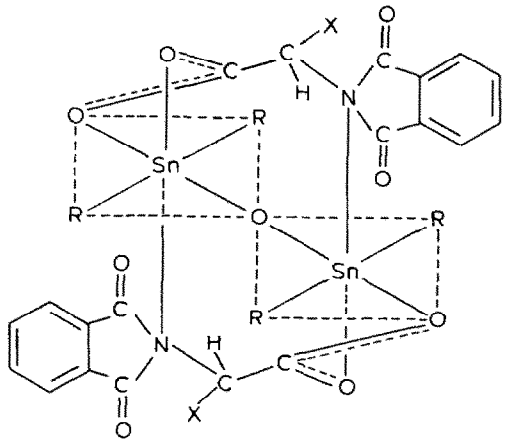
Complex [a]	*IS* (SnO_2)	*QS*	Line widths	$\rho = QS/IS$	C–Sn–C (°)	Probable structure
1 [b]	1.27	3.44	1.00; 0.99	2.70	141	II
2 [c]	1.06	3.41	1.02; 1.09	3.21	140	
3 [c]	1.03	3.56	1.04; 1.24	3.45	144	
4 [b]	1.67	4.09	0.84; 0.91	2.45	172	
5 [c]	1.57	3.91	0.90; 1.00	2.50	159	I
6 [c]	1.33	3.66	0.91; 0.92	2.70	148	
7 [c]	1.16	3.40	1.06; 1.06	2.93	139	II
8 [c]	1.37	3.52	1.01; 1.08	2.56	143	
9 [c]	1.33	3.45	1.02; 1.00	2.59	141	
10 [b]	1.38	3.43	0.88; 0.89	2.45	140	I
11	1.46	3.63	0.84; 1.10	3.00	147	
12 [c]	1.16	3.42	0.99; 0.96	2.94	140	
13 [b]	1.23	3.39	0.95; 0.95	2.75	140	III
14 [c]	1.03	3.35	1.00; 1.10	3.25	138	
15	1.25	3.37	0.87; 0.94	2.69	138	
16	1.45	3.64	1.06; 1.05	2.52	147	
17 [c]	1.15	3.43	1.15; 1.20	2.98	140	IV
18	1.39	3.46	0.93; 1.03	2.48	141	III
19	1.15	3.43	1.00; 1.08	2.98	140	IV
20 [c]	1.18	3.54	1.18; 1.19	3.00	144	
21	1.32	3.47	0.93; 0.91	2.62	141	III
22 [b]	1.38	3.38	1.00, 1.06	2.44	138	
23	1.38	3.50	0.93; 0.98	2.53	142	IV

[a] See for formulae of the complexes Table 1. [b] *IS* and *QS* ±0.03; line widths ±0.05. [c] *IS* and *QS* ±0.05; line widths ±0.08



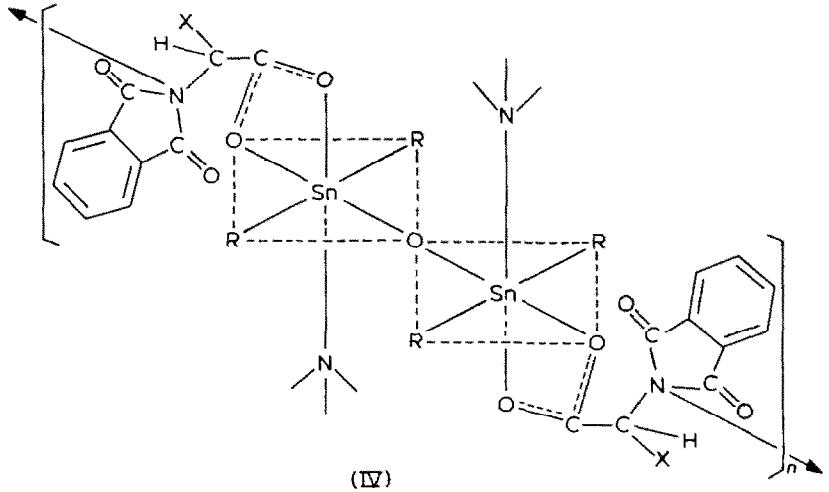
(II)

(X = $CH_2CH(CH_3)_2$, CH_3 , $CH_2C_6H_5$; n = 2,3 or 4)



(III)

(X = $CH_2CH(CH_3)_2$, CH_3 , $CH_2C_6H_5$)



(IV)

(X = $CH_2CH(CH_3)_2$, CH_3 , $CH_2C_6H_5$; n = 2,3 or 4)

needs further investigation by single crystal X-ray diffraction. However, these complexes are unique in the sense that they have a bidentate carboxylate group unlike the earlier reported organotin complexes with the unprotected amino acids which contain a unidentate carboxylate group [6]. Nevertheless cadmium(II) complexes with *N*-acetyl derivative of glycine [34], L-alanine and *β*-alanine [35] possess bidentate carboxylate groups.

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References

1 B.Y.K. Ho and J.J. Zuckerman, Inorg. Chem., 12 (1973) 1552.
2 G. Domazetis, R.J. Magee and B.D. James, J. Organomet. Chem., 173 (1979) 357.
3 B.Y.K. Ho, J.J. Zuckerman and J.A. Zubita, J. Chem. Soc., Chem. Commun., (1975) 88.
4 B.Y.K. Ho and J.J. Zuckerman, Inorg. Nucl. Chem. Lett., 9 (1973) 849.
5 B.K.H. Ho, K.C. Molloy, J.J. Zuckerman, F. Reidinger and Z.A. Zubieta, J. Organomet. Chem., 187 (1980) 213.
6 W.T. Hall and J.J. Zuckerman, Inorg. Chem., 16 (1977) 1239.
7 G. Domazetis, R.J. Magee and B.D. James, J. Organomet. Chem., 162 (1978) 239.
8 K.C. Molloy, J.J. Zuckerman, G. Domazetis and B.D. James, Inorg. Chim. Acta, 54 (1981) L217.
9 G. Domazetis, M.F. Mackay, R.J. Magee and B.D. James, Inorg. Chim. Acta, 34 (1979) L247.
10 S.S. Sandhu, Jr., Ph.D Thesis G.N.D.U. (1981).
11 G.K. Sandhu, R. Gupta, S.S. Sandhu and R.V. Parish, Polyhedron, in press.
12 A.K. Bose, F. Greer and C.C. Price, J. Org. Chem., 23 (1958) 1335; C.A., 53 (1959) 160081.
13 Organometallic compounds Micheal Dub (Ed.); Vol. II, Springer-Verlag, New York Inc., 1967, p. 368.
14 R. Okawara, M. Wada, Adv. Organomet. Chem., 5 (1967) 137.
15 N.W.G. Debye, D.E. Fenton and J.J. Zuckerman, J. Inorg. Nucl. Chem., 34 (1972) 352.
16 R.C. Poller, The chemistry of organotin compounds, Logos London, 1970, p. 231.
17 P.G. Shrivastava, Ind. J. Chem., 14A (1976) 708.
18 C.S.C. Wang and J.M. Shreeve, J. Organomet. Chem., 46 (1972) 271.
19 V.G. Kumar Das, N.S. Weng, P.J. Smith and R.H. Hill, J. Chem. Soc., Dalton Trans., (1981) 552.
20 W.D. Honnic and J.J. Zuckerman, J. Organomet. Chem., 178 (1979) 133.
21 S. Calogero, P. Furlani and V. Peruzzo, G. Tagliavini, J. Organomet. Chem., 128 (1977) 177.
22 L. Pellerito, G. Ruisi and R. Barbieri, Inorg. Chim. Acta, 32 (1979) 39.
23 E. Lindner and V. Ansorge, Z. Anorg. Chim., 442 (1978) 189.
24 D.V. Naik, L. May and C. Curran, J. Coord. Chem., 2 (1973) 309.
25 R. Barbieri and R.H. Herber, J Organomet. Chem., 42 (1972) 65
26 R.V. Parish, Prog. Inorg. Chem., 15 (1972) 101.
27 R.V. Parish and C.E. Johnson, J. Chem. Soc. A, (1971) 1906.
28 J.P. Ashmore, T. Chivers, K.A. Kere and J.H.G. Van Roode, Inorg. Chem., 16 (1977) 191
29 T.K. Sham, J.S. Tse, N. Wellington and G.M. Bancroft, Can. J. Chem., 55 (1977) 3487
30 J.J. Zuckerman, Adv. Organomet. Chem., 9 (1970) 21.
31 A.G. Maddock and R.H. Platt, J. Chem. Soc. A, (1971) 1191.
32 T.K. Sham and M.G. Bancroft, Inorg. Chem., 14 (1975) 2281.
33 S.S. Sandhu, S.S. Sandhu, Jr., G.K. Sandhu, R.V. Parish and O. Parry, Inorg. Chim. Acta, 58 (1982) 251.
34 G. Marcotrigiano, L. Menabue and G.C. Pellacani, J. Inorg. Nucl. Chem , 37 (1975) 2344.
35 G. Marcotrigiano, L. Menabue and G.C. Pellacani, J. Inorg. Nucl. Chem., 40 (1978) 753.