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Preparation, IR, ^{13}C and ^{119m}Sn Mössbauer spectral studies of organotin(IV) derivatives of ethylenediaminetetraacetic acid

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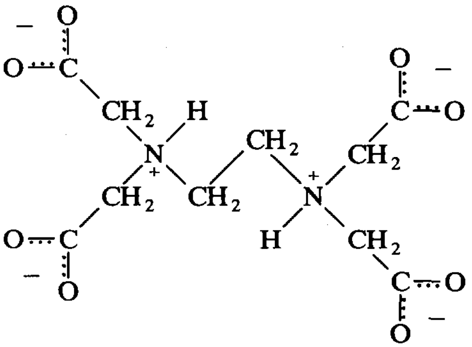
Abstract

New diorganotin(IV) and triorganotin(IV) derivatives $(R_2Sn)_n$EDTA (R = Me, Et, ^{n}Pr, ^{n}Bu, ^{n}Oct; $n = 1, 2$); $(R_2Sn)_3(EDTAH)_2$ (R = ^{n}Bu, ^{n}Oct); and $(R_3Sn)_3$EDTAH (R = ME, ^{n}Bu, Ph, Cyh) have been prepared by reacting diorganotindichloride and triorganothinchloride with di-, tri- and tetra-silver salts of ethylenediaminetetraacetic acid ($EDTAH_4$) in 1:1, 2:1, 3:2 and 3:1 (metal/ligand) molar ratio, respectively. The 3:2 diorganotin(IV) compounds possess an octahedral geometry around the Sn atom whereas the diorganotin (1:1, 2:1) and triorganotin (3:1) have five coordinate trigonal bipyramidal geometry around the Sn atom. One of the organotin moieties in $(R_2Sn)_3(EDTAH)_2$ acts as a bridge between two EDTA molecules, resulting in a novel type of complex.

Introduction

Disodiumethylenediaminetetraacetic acid ($EDTAH_2Na_2$) is one of the most versatile analytical and chelating reagents [1,2]. Only a few diorganotin [3] and triorganotin [4] compounds with EDTA have been reported. In continuation of our earlier work with triorganotin(IV) derivatives of EDTA [5], we report here on some new diorganotin(IV) and triorganotin(IV) complexes with EDTA prepared in different stoichiometries and with interesting bonding sites available to tin(IV).



$EDTAH_2Na_2$

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Results and discussion

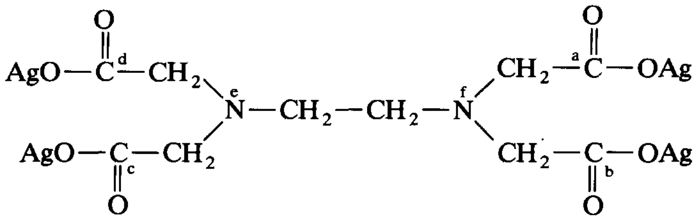
Diorganotin(IV) and triorganotin(IV) chlorides react with di-, tri- and tetra-silver salts of ethylenediaminetetraacetic acid in 1:1, 2:1, 3:2 (diorganotin) and 3:1 (triorganotin) molar ratios, respectively.

$R_2SnCl_2 + EDTAH_2Ag_2 \rightarrow R_2SnEDTAH_2 + 2AgCl$ (1:1)

$2R_2SnCl_2 + EDTAAg_4 \rightarrow (R_2Sn)_2EDTA + 4AgCl$ (2:1)

$3R_2SnCl_2 + 2EDTAHAg_3 \rightarrow (R_2Sn)_3(EDTAH)_2 + 6AgCl$ (3:2)

$3R_3SnCl + EDTAHAg_3 \rightarrow (R_3Sn)_3EDTAH + 3AgCl$ (3:1)



($EDTAAg_4$)

and characterized by elemental analysis, IR, ^1H NMR, ^{13}C NMR and Mössbauer spectroscopy. Ethylenediaminetetraacetic acid has six binding sites, four carboxylates (a, b, c, d) and two nitrogens (e, f) available for bonding to tin(IV). All the complexes are white, insoluble in common organic solvents such as chloroform, benzene, methanol, carbontetrachloride and are soluble in ethanol. Physical and analytical data are given in Table 1.

Infrared data

Infrared spectra of the silver, sodium salts and complexes of ethylenediaminetetraacetic acid were recorded in KBr discs in the range 4000–200 cm^{-1}. The stretching frequencies of interest are ν(N–H), ν(COO), ν(Sn–C), ν(Sn–O) (Table 2). In complexes **1–4** the ν(N–H) shifts to a higher value compared to the sodium salt of ethylenediaminetetraacetic acid ($EDTAH_2Na_2$), which reveals the non-participation of nitrogen atoms in coordination to tin(IV) [5]. In complexes **10–15** the lowering of ν(N–H) compared to $EDTAHNa_3$ is due to intramolecular hydrogen bonding NH...O=C–; the coordination of nitrogen to tin is ruled out because nitrogen atoms bind to metal only when nitrogen is deprotonated on complex formation. Absence of a ν(N–H) band in complexes **5–9** shows complete deprotonation of all the carboxylate groups. In 1:1 complexes **1–4**, a medium-intensity band in the region 1745–1730 cm^{-1} is due to one of the free C=O of the carboxylate group bonded to tin(IV). The second tin(IV)-bonded carboxylate C=O, which is intermolecularly bonded to tin(IV), C=O → Sn, falls in the range 1670–1645, which is also the region of the hydrogen-bonded two free carboxylates ...H–O–C=O...H–O... present in these complexes. Similarly, in the 1:2 complexes **5–9** free C=O of carboxylate bonded to tin(IV) appears in the range 1740–1730 cm^{-1}, whereas the intermolecularly bonded carboxylate C=O, (C=O → Sn) absorbs in the region 1650–1630 cm^{-1}. In the remaining 3:2 and 3:1

Table 1

Physical and analytical data of diorganotin and triorganotin(IV) complexes of EDTA

	Compound [a]		Yield (%)	m.p. (°C)	Analysis (%) found (calc.)			
					C	H	N	Sn
1	$(C_2H_5)_2Sn\ EDTAH_2$	1:1	88	103–104	35.89 (36.00)	4.87 (5.15)	4.82 (6.00)	27.02 (25.40)
2	$(n\text{-}C_3H_7)_2Sn\ EDTAH_2$	1:1	87	113–115	37.89 (38.80)	5.93 (5.65)	4.59 (5.65)	23.03 (23.99)
3	$(n\text{-}C_4H_9)_2Sn\ EDTAH_2$	1:1	92	117–120	41.17 (41.32)	6.54 (6.12)	4.11 (5.35)	25.07 (22.70)
4	$(n\text{-}C_8H_{17})_2Sn\ EDTAH_2$	1:1	89	103–105	48.94 (47.08)	7.12 (7.56)	4.12 (4.41)	19.43 (18.62)
5	$[(CH_3)_2Sn]_2EDTA$	2:1	69	143–145	27.90 (28.60)	4.10 (4.09)	4.39 (4.49)	41.27 (40.55)
6	$[(C_2H_5)_2Sn]_2EDTA$	2:1	87	120–121	34.3 (33.15)	5.10 (4.91)	4.05 (4.29)	35.98 (36.44)
7	$[(n\text{-}C_3H_7)_2Sn]_2EDTA$	2:1	75	116–118	37.80 (37.80)	6.00 (5.79)	3.81 (4.01)	37.98 (34.03)
8	$[(n\text{-}C_4H_9)_2Sn]_2EDTA$	2:1	92	98–100	40.64 (41.40)	6.59 (6.39)	2.78 (3.71)	32.48 (31.51)
9	$[(n\text{-}C_8H_{17})_2Sn]_2EDTA$	2:1	65	137–139	50.60 (51.04)	7.28 (8.17)	1.99 (2.86)	23.89 (24.28)
10	$[(n\text{-}C_4H_9)_2Sn]_3[EDTAH]_2$	3:2	73	147–150	40.60 (40.50)	6.61 (5.63)	3.73 (4.50)	27.92 (28.66)
11	$[n\text{-}(C_8H_{17})_2Sn]_3[EDTAH]_2$	3:2	76	134–137	50.06 (50.60)	8.75 (7.93)	2.47 (3.47)	22.90 (22.08)
12	$[(CH_3)_3Sn]_3EDTAH$	3:1	71	195–196	28.30 (29.20)	4.92 (5.12)	2.11 (3.58)	49.76 (50.36)
13	$[(n\text{-}C_4H_9)_3Sn]_3EDTAH$	3:1	67	1	–	–	–	31.23 (30.74)
14	$[(C_6H_5)_3Sn]_3EDTAH$	3:1	75	173–175	56.24 (57.30)	4.05 (4.30)	1.86 (2.00)	28.02 (26.60)
15	$[(C_6H_{11})_3Sn]_3EDTAH$	3:1	73	115–117	54.80 (55.16)	7.87 (8.04)	1.43 (2.01)	24.82 (25.58)

[a] All complexes are white.

Table 2

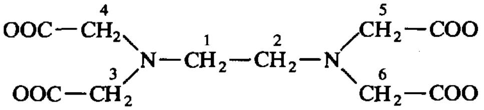
Infrared spectral data (4000–200 cm^{-1}) of diorgano and triorganotin(IV) complexes of EDTA.

	Compound	ν(N–H)	$\nu(COO)_{asym}$	$\nu(COO)_{sym}$	$\Delta\nu$	ν(Sn–C)	ν(Sn–O)
	$EDTANa_4$	–	1605s	1400s	205	–	–
			1660sh	1370m			
	$EDTAAg_4$	–	1580bs	1400s	180	–	–
	EDTAH Na_3	3395m	1630s	1390s	240	–	–
			1680sh	1320s	360		
	$EDTAHAg_3$	3420m	1580s	1410s	170	–	–
	$EDTAH_2Na_2$	3260m	1610s	1410s	200	–	–
			1680s	1300s	380		
	$EDTAH_2Ag_2$	3400m	1620s	1400s	220	–	–
			1660s	1350s	310		
1	$(C_2H_5)_2SnEDTAH_2$	3440m	1660sb	1400s	260	610m	360m
			1730m	1300s	430	540m	
2	$(n\text{-}C_3H_7)_2SnEDTAH_2$	3420m	1645sb	1380s	265	590m	440w
			1730m	1300s	430		
3	$(n\text{-}C_4H_9)_2Sn\ EDTAH_2$	3440m	1660sb	1385s	275	605m	490w
			1740m	1300s	440	550w	
4	$(n\text{-}C_8H_{17})_2SnEDTAH_2$	3440m	1670sb	1385s	285	610m	480w
			1745m	1300s	445	540m	

5	$[(CH_3)_2Sn]_2EDTA$	–	1630sb	1380s	250	600m	400w
			1730m	1300s	430	540m	
6	$[(C_2H_5)_2Sn]_2EDTA$	–	1650s	1380s	270	605m	400w
			1740m	1310s	430	530m	
7	$[(n\text{-}C_3H_7)_2Sn]_2EDTA$	–	1630s	1370s	260	600m	490w
			1730m	1300s	430	540m	
8	$[(n\text{-}C_4H_9)_2Sn]_2EDTA$	–	1640s	1370s	270	620m	400m
			1730m	1300s	430	540m	
9	$[(n\text{-}C_8H_{17})_2Sn]_2EDTA$	–	1620s	1370s	250	595m	480m
			1730m	1300s	430	545m	
10	$[(n\text{-}C_4H_9)_2Sn]_3[EDTAH]_2$	3340m	1640sb	1400s	240	540w	490w
			1680s	1280s	400	600w	
11	$[(n\text{-}C_8H_{17})_2Sn]_3[EDTAH]_2$	3350m	1640sb	1410s	230	540w	480w
			1685m	1310s	375		
12	$[(CH_3)_3Sn]_3EDTAH$	3340m	1600sb	1390s	210	550s	475m
			1660s	1250s	410		
13	$[(n\text{-}C_4H_9)_3Sn]_3EDTAH$	3345m	1600sb	1380s	220	530w	455w
			1670m	1200s	470	555m	
14	$[(C_6H_5)_3Sn]_3EDTAH$	3340m	1630sb	1430m	200	370w	230w
			1660s	1260s	400	330w	260w
15	$[(C_6H_{11})_3Sn]_3EDTAH$	3340m	1625sb	1380s	245	500w	400w
			1670sh	1270s	400		

Table 3

^1H NMR data of diorgano and triorganotin(IV) complexes of EDTA

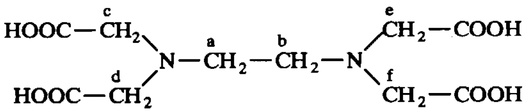


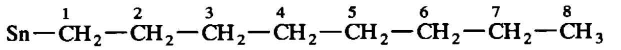
	Compound	$\overset{1}{CH_2}$ and $\overset{2}{CH_2}$	+ $\overset{3}{CH_2}$, $\overset{4}{CH_2}$, $\overset{5}{CH_2}$, $\overset{6}{CH_2}$	Sn–alkyl/phenyl/cyh -CH_2-	 -CH_3
	$EDTANa_4$ [a]	5.02 (s, 4H)	5.59–5.54 (m, 8H)	–	–
	$EDTAHNa_3$ [a]	3.85–3.76 (m, 4H)	6.05–5.89 (m, 8H)	–	–
	$EDTAH_2Na_2$ [a]	3.91–3.69 (m, 4H)	5.24–4.12 (m, 8H)	–	–
1	$(C_2H_5)_2Sn$ $EDTAH_2$ [b]	2.78–2.20 (bm, 14H, CH_2 = 12H, NH = 2H)		1.95–0.63 (m, 10H)	
2	$(n\text{-}C_3H_7)_2Sn$ $EDTAH_2$ [b]	3.90–3.10 (m, 14H, CH_2 = 12H, NH = 2H)		1.80–1.10 (m, 8H)	1.00–0.60 (t, 6H)
3	$(n\text{-}C_4H_9)_2Sn$ $EDTAH_2$ [b]	2.65–2.39 (m, 14H, CH_2 = 12H, NH = 2H)		1.68–1.19 (m, 12H)	1.05–0.55 (m, 6H)
4	$(n\text{-}C_8H_{17})_2Sn$ $EDTAH_2$ [b]	3.90–3.40 (m, 14H, CH_2 = 12H, NH = 2H)		1.80–1.10 (m, 28H)	1.00–0.60 (t, 6H)
5	$[(CH_3)_2Sn]_2EDTA$ [b]	1.99–1.45 (bm, 12H)		–	0.90 (s, 12H)
6	$[(C_2H_5)_2Sn]_2EDTA$ [b]	2.80–2.53 (bm, 12H)		2.03–1.30 (m, 8H)	1.20–0.98 (t, 12H)
7	$[(n\text{-}C_3H_7)_2Sn]_2EDTA$ [b]	4.15–3.65 (bm, 12H)		2.60–2.00 (m, 16H)	1.91–1.60 (t, 12H)
8	$[(n\text{-}C_4H_9)_2Sn]_2EDTA$ [b]	3.19–2.80 (m, 12H)		2.00–1.20 (m, 24H)	1.10–0.55 (m, 12H)
9	$[(n\text{-}C_8H_{17})_2Sn]_2EDTA$ [b]	2.49–2.12 (m, 12H)		1.51–1.10 (bm, 68H)	
10	$[(n\text{-}C_4H_9)_2Sn]_3$- $[EDTAH]_2$ [b]	2.62–2.36 (m, 26H, CH_2 = 24H, NH = 2H)		1.73–1.10 (m, 36H)	1.09–0.67 (d, 18H)
11	$[(n\text{-}C_8H_{17})_2Sn]_3$- $[EDTAH]_2$ [b]	2.53–2.30 (m, 26H, CH_2 = 24H, NH = 2H)		1.60–1.28 (m, 84H)	1.00–0.89 (m, 18H)
12	$[(n\text{-}C_4H_9)_3Sn]_3EDTAH$ [b]	2.90–2.09 (m, 13H, CH_2 = 12H, NH = 1H)		2.00–0.80 (bm, 81H)	–
14	$[(C_6H_5)_3Sn]_3EDTAH$ [b]	3.62–3.55 (m, 12H)		7.76–7.16 (m, 46H, Ph = 45H, NH = 1H)	–
15	$[(C_6H_{11})_3Sn]_3EDTAH$ [b]	3.54–2.90 (m, 13H, CH_2 = 12H, NH = 1H)		2.20–0.68 (bm, 99H)	–

[a] D_2O. [b] $CDCl_3 + DMSO\text{-}d_6$ (4:1).

Table 4

^{13}C NMR spectra data of diorgano and triorganotin(IV) complexes of EDTA





$$\overset{}{Sn}-\overset{1}{CH_2}-\overset{2}{CH_2}-\overset{3}{CH_2}-\overset{4}{CH_2}-\overset{5}{CH_2}-\overset{6}{CH_2}-\overset{7}{CH_2}-\overset{8}{CH_3}$$

	Compound	a, b	c, d, e, f	COO	Sn–R							
					1	2	3	4	5	6	7	8
	$EDTANa_4$ [a]	54.42	61.32	182.22	–	–	–	–	–	–	–	–
	$EDTAHNa_3$ [a]	53.75	60.12	178.99	–	–	–	–	–	–	–	–
	$EDTAH_2Na_2$ [a]	52.40	58.90	171.50	–	–	–	–	–	–	–	–
2	$(n\text{-}C_3H_7)_2Sn\ EDTAH_2$ [b]	48.88	59.36	169.63 168.40	17.64	16.45	12.51	–	–	–	–	–
3	$(n\text{-}C_4H_9)_2SnEDTAH_2$ [b]	48.61	57.71	168.39 166.58	26.76	26.43	25.08	13.44	–	–	–	–
11	$[(n\text{-}C_8H_{17})_2Sn]_3[EDTAH]_2$	58.39	61.73	167.89 170.81	33.85	31.83	29.33	25.67	27.74	22.63	21.96	14.03
14	$[(C_6H_5)_3Sn]_3EDTAH$ [b]	50.43	58.42	171.85 172.69	137.19 128.41	–	–	–	–	–	–	–
15	$[(C_6H_{11})_3Sn]_3EDTAH$ [b]	49.02	58.09	166.98 194.98	36.88	31.16	30.01	30.69	28.73	26.68	–	–

[a] D_2O. [b] $CDCl_3 + DMSO\text{-}d_6$ (4:1).

complexes **10–15**, a strong absorption in the region 1685–1660 cm^{-1} is assigned to the free hydrogen bonded carboxylates ...H–O–C=O...H–O, and another strong band in the range 1645–1600 cm^{-1} is due to the bidentately bonded carboxylate [5]. The magnitude of separation $\Delta\nu$, $[\Delta\nu = \nu(COO)_{asym} - \nu(COO)_{sym}]$, of the COO group is very useful for drawing structural inferences. In complexes **1–9**, the $\Delta\nu$ values in the range 445–430 are clearly from the unidentately bonded carboxylates [6], and the second set of $\Delta\nu$ values 285–250 for the same compounds are from the intermolecularly bonded carboxylate C=O (as C=O → Sn) as well as from the free-hydrogen-bonded carboxylate groups, which also absorb in the same region. For complexes **10–15**, $\Delta\nu$ values in the range 245–200 are from the bidentately bonded carboxylated [7] and are comparable to the sodium salts whereas $\Delta\nu$ values in the range 470–375 are from the hydrogen-bonded free carboxylate groups. Sn–C stretching frequencies appear at 600–500 cm^{-1} for alkyl derivatives and 330–250 cm^{-1} for the phenyl compounds [5]. Bands in the region 500–400 cm^{-1} are assigned to Sn–O stretching, as reported earlier [8].

^1H NMR data

The spectra of the di-, tri- and tetra-sodium salts of ethylenediaminetetraacetic acid (EDTA) were recorded in D_2O and those of the soluble complexes in a mixture of $CDCl_3$ and DMSO-d_6 (4:1) (Table 3). In the spectra of $EDTAH_2Na_2$ and $EDTAHNa_3$, the signals due to NH protons appear at 7.23 and 7.28 ppm, respectively, whereas in complexes **1–4**, **10–13** and **15**, the NH protons move upfield and appear along with all the CH_2 protons of EDTA as a multiplet; in complex **14**, the protons move downfield and appear along with the phenyl protons as a complex multiplet. All the CH_2 protons of EDTA undergo a high field shift, to differing extent, after complex formation. All the alkyl/phenyl/cyclohexyl protons appear in the expected region. The total number of protons calculated from the integration curves for all the complexes agrees with the total number of protons calculated from the expected molecular formula, thus confirming complex formation.

^{13}C NMR data

The spectra of the di-, tri- and tetra-sodium salts of ethylenediaminetetraacetic acid were recorded in D_2O and those of the complexes in a mixture of $CDCl_3$ and DMSO-d_6 (4:1) (Table 4). The number of signals found correspond with the presence of magnetically non-equivalent carbon atoms. In di- [9], tri- and tetra-sodium salts of EDTA, only one type of carboxylate group is present, as shown by the presence of only one signal, whereas in the case of complexes **2**, **3**, **11**, **14** and **15** the appearance of two signals for the carboxyl carbons clearly shows two types of carboxyl groups. The high-field signal is assigned to the free carboxylic acid group and the lower field signal to the carboxylate bonded to tin(IV). In the triphenyl complex, carboxylates are deshielded to a greater extent compared to the remaining complexes. The identification of the alkyl/phenyl/cyclohexyl carbons in all the complexes confirms complex formation.

Mössbauer data

The ^{119m}Sn Mössbauer spectra (Table 5) are best interpreted on the basis of the quadrupole splitting (QS) values [10]. In diorganotin(IV) complexes, when the

Table 5

^{119m}Sn Mössbauer data (80 K, mm s^{-1})

	Compound	IS (SnO_2) ±0.08	QS ±0.06	Line widths		ρ = QS/IS
				1	2	
3	$(n\text{-}C_4H_9)_2$Sn EDTAH$_2$ (1:1)	1.30	3.14	1.22	1.49	2.41
8	$[(n\text{-}C_4H_9)_2Sn]_2$EDTA (2:1)	1.28	2.98	1.04	1.42	2.33
10	$[(n\text{-}C_4H_9)_2Sn]_3$[EDTAH]$_2$ (3:2)	1.31	3.11	1.27	1.45	2.37
15	$[(C_6H_{11})_3Sn]_3$EDTAH (3:1)	1.39	2.99	1.06	1.32	2.15

donor atoms are highly electronegetive, 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 [6]. The Mössbauer data (IS 1.24–1.55 and QS 3.10–3.70 mm s^{-1}) for diorganotin carboxylates supports a distorted *trans* octahedral geometry around tin(IV) [11]. In the present investigation, the QS values in complexes **3** and **10** fall in the range of five and six coordinated distorted structures. However, the infrared data supports a five coordinate structure for complex **3** and a distorted octahedral structure for complex **10**. The isomer shift values in complexes **8** and **15** lie in the range typical of triorganotin(IV) carboxylates [12] while the quadrupole splitting values lie in the range of triorganotin derivatives of amino acids with trans trigonal bipyramidal geometry around the tin atom [13]. Hence five coordinated trigonal bipyramidal geometry around the tin atom has been confirmed for complexes **8** and **15**. The ρ value (QS/IS ratio) also supports a coordination number of tin higher than four.

Experimental

Materials

Trimethyl and tributyltinchlorides were obtained from Alfa products. Tricyclohexyltinchloride was obtained from Aldrich. Dibutyl- and dioctyltindichlorides were prepared by the reported method [14]. Di-, tri- and tetra-sodium salts of ethylenediaminetetraacetic acid were obtained from Alfa products.

Preparation of silver salts of ethylenediaminetetraacetic acid (EDTAH$_4$)

The silver salts of Na_2EDTAH_2, Na_3EDTAH and Na_4EDTA were prepared by the addition of the required amount of silver nitrate to a solution of sodium salt of EDTAH$_4$ in distilled water. The silver salt of the EDTA precipitated and was separated by filtration, washed several times with hot water and dried under vacuum.

$$EDTAH_2Na_2 + 2AgNO_3 \rightarrow EDTAH_2Ag_2 + 2NaNO_3$$

$$EDTAHNa_3 + 3AgNO_3 \rightarrow EDTAHAg_3 + 3NaNO_3$$

$$EDTANa_4 + 4AgNO_3 \rightarrow EDTAAg_4 + 4NaNO_3$$

Preparation of complexes

Di-, tri- and tetra-silver salts of ethylenediaminetetraacetic acid (EDTA) (1.0 mmol for 1:1, 2:1, 3:1 and 2.0 mmol for 3:2 (metal/ligand)) were dissolved in

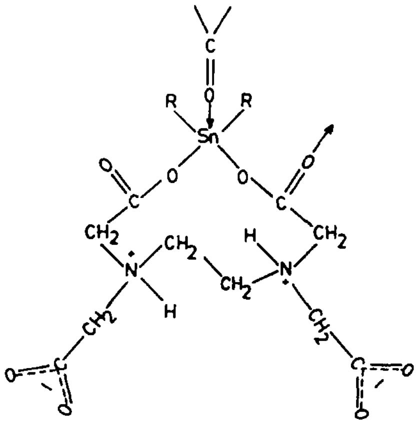


Fig. 1. R = Et, ^{n}Pr, ^{n}Bu, ^{n}Oct (1:1).

absolute ethanol (30 cm^3). To this solution, was added a solution of diorganotindichloride (1.0 mmol for 1:1, 2.0 mmol for 2:1 and 3.0 mmol for 3:2) and triorganotinchloride (3.0 mmol for 3:1) dissolved in absolute ethanol (10 cm^3). The reaction mixture was refluxed on a water bath. The pink-coloured silver chloride started separating out within 10–15 min. The reaction mixture was further refluxed for 4 h until all the silver chloride separated out. This was removed by filtration and washed with absolute ethanol. From the filtrate, the ethanol was removed by distillation leaving a solid or a liquid complex which was dried under vacuum. All the complexes were recrystallized from absolute ethanol.

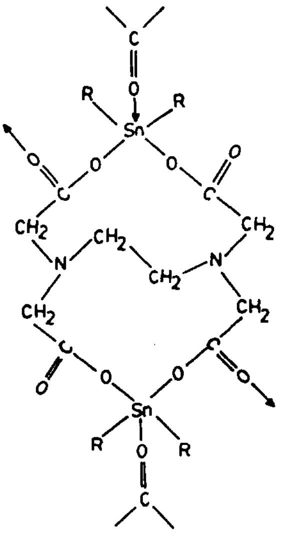


Fig. 2. R = Me, ^{n}Pr, ^{n}Bu, ^{n}Oct (1:2).

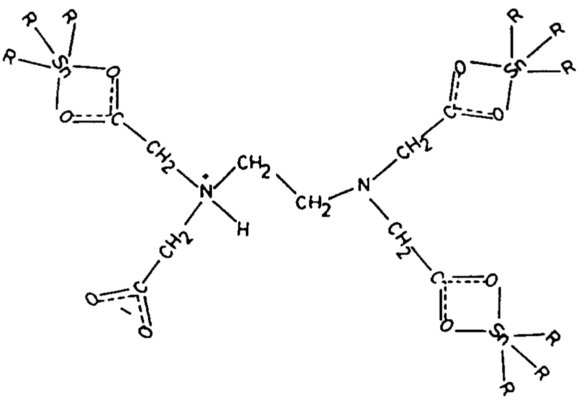


Fig. 3. R = Me, ^{n}Bu, Ph, Cyh (1:3).

Physical measurements

Elemental analysis for carbon, hydrogen and nitrogen were carried out by Microanalytical Service R.S.I.C., Panjab University, Chandigarh. Tin was estimated as SnO_2. Infrared spectra were recorded on Pye–Unicam SP3-300 spectrometer as KBr discs. The ^{1}H and ^{13}C NMR spectra were recorded on Brucker AC 200 spectrometer using tetramethylsilane as internal standard. Mössbauer spectra were recorded on a Harwell 6000 series spectrometer with samples at about 80 K and source Pd/Sn at room temperature; isomer shifts are relative to SnO_2 at room temperature.

Conclusions

Interesting structural information has been obtained from IR, NMR and Mössbauer spectral studies. Diorganotin(IV) 1:1 and 1:2 and triorganotin(IV) 1:3 complexes possess a five coordinate distorted trigonal bipyramidal geometry around the Sn atom (Figs. 1–3). The trinuclear 3:2 complexes have six coordinate distorted octahedral geometry around the Sn atom with one bridging R_2Sn moiety (Fig. 4).

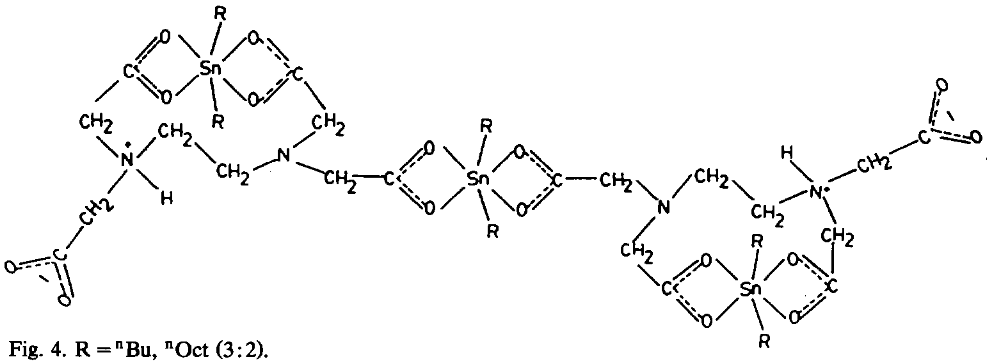


Fig. 4. R = ^{n}Bu, ^{n}Oct (3:2).

Acknowledgment

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References

1 A.I. Vogel, A Text Book of Quantitative Analysis, English Language Book Society and Longman, London, 1978.

2 A.E. Martell and M. Calvin, Chemistry of the Metal Chelate Compounds, Prentice Hall, New York, 1952.

3 T.N. Srivastava, Ind. J. Chem. 13 (1975) 724.

4 M.E. Lombardo and P.B. Hudson, US Patent 534,077 (Cl, 260 429.7 CO7); Chem. Abstr. 74 (1971) 23011w.

5 G.K. Sandhu, S.P. Verma, L.S. Moore and R.V. Parish, J. Organomet. Chem., 315 (1986) 309.

6 G.K. Sandhu, R. Gupta, S.S. Sandhu, R.V. Parish and K. Brown, J. Organomet. Chem., 279 (1985) 373.

7 G.K. Sandhu, R. Gupta, S.S. Sandhu and R.V. Parish, Polyhedron, 4 (1985) 81.

8 G.K. Sandhu, R. Hundal and E.R.T. Tiekink, J. Organomet. Chem., 412 (1991) 31.

9 L.F. Johnson and W.C. Jankowski, A Collection of Assigned Coded and Indexed Carbon-13 NMR Spectra, Wiley–Interscience, New York, 1972, p. 382.

10 R.V. Parish, in G.J. Long (Ed.), Mössbauer Spectroscopy Applied to Inorganic Chemistry, Vol. 1, Plenum, New York, 1984, p. 527.

11 R. Barbieri and R.H. Herber, J. Organomet. Chem., 42 (1972) 65.

12 G.K. Sandhu, G. Kaur, J. Holecek and A. Lycka, J. Organomet. Chem., 332 (1987) 75.

13 O.A. Bamgboye, T.T. Bamgboye and P.G. Harrison, J. Organomet. Chem., 306 (1986) 17.

14 J. Nosek, Coll. Czech. Chem. Commun., 29(3) (1964) 597; Chem. Abstr. 60 (1964) 8053.