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Structural chemistry of organotin carboxylates

XII *. Synthesis and characterization of diorganotin(IV) carboxylates containing 2-thiophene- or 2-furan-carboxylic acid. Crystal structure of [$Et_2Sn(O_2CC_4H_3S)_2$]

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Abstract

Reactions of diorganotin oxides with 2-thiophene- or 2-furan-carboxylic acids in 1:2 stoichiometry yielded complexes of the type [$R_2Sn(O_2CC_4H_3E)_2$] (I) (R = Me, Et, ^{n}Pr, ^{n}Bu; E = O or S). These complexes, on treatment with R_2SnCl_2 afforded the chloro complexes, [$R_2Sn(Cl)(O_2CC_4H_3E)$] (II) (R = Me, ^{n}Bu, E = S; R = ^{n}Pr, ^{n}Bu, E = O). On hydrolysis, complexes of the type I were converted into the corresponding dicarboxylatotetraorganodistannoxanes. All complexes were characterized by elemental analyses, IR and multinuclear magnetic resonance (^1H, ^{13}C and ^{119}Sn) data. The crystal structure of [$Et_2Sn(O_2CC_4H_3S)_2$] shows the Sn atom to exist in a skew-trapezoidal planar geometry defined by two asymmetrically coordinating carboxylate ligands (Sn–O 2.143(5), 2.556(5); 2.129(5), 2.473(5) Å) and two ethyl groups (Sn–C 2.110(9), 2.124(9) Å; C–Sn–C 151.7(4)°). In the crystal lattice, there is a weak Sn···O interaction of 2.891(6) Å between centrosymmetrically related molecules.

Introduction

Organotin(IV) carboxylates are widely used as biocides and in industry as homogeneous catalysts [1–4]. In order to allow exploration of relationships between their biocidal activity and structure, a number of investigations on such molecules have been reported in recent years. Studies on organotin compounds having carboxylate ligands with additional donor atoms, such a nitrogen, available for coordination to Sn, has revealed new structural types which may lead to compounds with different activity. For instance, the [$Me_2Sn(2\text{-pic})Cl$] [5] and [$Me_2Sn(2\text{-pic})_2$]

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* For Part XI see ref. 1.

[6] (pic = 2-picolinate anion) compounds have six- and seven- coordinate Sn atoms respectively, owing to the multidentate nature of the bridging picolinate ligands which utilize both the carboxylate O and the pyridyl N atoms in coordination to Sn. By contrast with the latter compound [6], other $[R_2Sn(O_2CR')_2]$ compounds contain six-coordinate Sn centres [1,7–12]. As an extension of our studies of organotin carboxylates with additional donor atoms residing on the carboxylate ligand [13], we now report a series of diorganotin(IV) compounds derived from 2-thiophene- or 2-furan-carboxylic acids, which were investigated to ascertain whether the S or O hetero atom coordinates the Sn atom.

Results and discussion

Reactions of diorganotin(IV) oxides with 2-thiophene- or 2-furan-carboxylic acid in 1 : 2 stoichiometry gave complexes of the type $[R_2Sn(O_2CC_4H_3E)_2]$ (I) (R = Me, Et, ^{n}Pr and ^{n}Bu; E = S or O). Treatment of these complexes with the corresponding R_2SnCl_2 gave the monochloro complexes, $[R_2Sn(Cl)(O_2CC_4H_3E)]$ (II) (R = Me, ^{n}Bu, E = S; R = ^{n}Pr, ^{n}Bu, E = O), in quantitative yields; see Table 1 for physical data for these compounds. All these complexes are sensitive to moisture and convert to the corresponding dicarboxylatotetraorganodistannoxanes, $\{[R_2Sn(O_2CC_4H_3E)]_2O\}_2$, which have been reported recently [13], in the presence of water.

Table 1

Physical and analytical data for diorganotin carboxylates

Compound	m.p. (°C)	Analyses (Found (calcd.) (%))			
		C	H	Cl	Sn
Ia $[Me_2Sn(O_2CC_4H_3S)_2]$	165–170	35.41 (35.76)	2.77 (3.00)	–	28.60 (29.45)
Ib $[Et_2Sn(O_2CC_4H_3S)_2]$	180–182	–	–	–	27.00 (27.53)
Ic $[Pr_2Sn(O_2CC_4H_3S)_2]$	146	–	–	–	24.92 (25.55)
Id $[Bu_2Sn(O_2CC_4H_3S)_2]$	106–108	43.90 (44.37)	4.91 (4.96)	–	24.10 (24.36)
Ie $[Me_2SnCl(O_2CC_4H_3S)]$	95	25.87 (27.00)	2.80 (2.91)	10.80 (11.39)	37.90 (38.12)
If $[Bu_2SnCl(O_2CC_4H_3S)]$	60– 65	38.88 (39.48)	5.04 (3.35)	8.80 (8.96)	–
IIa $[Me_2Sn(O_2CC_4H_3O)_2]$	185	39.27 (38.86)	3.20 (3.26)	–	30.07 (32.00)
IIb $[Et_2Sn(O_2CC_4H_3O)_2]$	84	42.07 (42.15)	3.84 (4.04)	–	29.74 (29.75)
IIc $[Pr_2Sn(O_2CC_4H_3O)_2]$	94	–	–	–	27.50 (27.79)
IId $[Bu_2Sn(O_2CC_4H_3O)_2]$	100	47.96 (47.51)	5.19 (5.32)	– –	25.90 (26.08)
IIe $[Pr_2SnCl(O_2CC_4H_3O)]$	85	–	–	9.90 (10.09)	32.90 (33.78)
IIf $[Bu_2SnCl(O_2CC_4H_3O)]$	85	41.00 (41.15)	5.21 (5.58)	9.90 (9.34)	30.90 (31.28)

Table 2

^{119}Sn{^{1}H} and ^{13}C{^{1}H} NMR data (ppm) for diorganotin carboxylates

Compound		δ^{119}Sn	Sn–R [a]	^{13}C NMR data				
				O_2CR'	C-2	C-3	C-4	C-5
	C_4H_3SCOOH	–	–	167.8	132.6	133.8	127.8	134.8
Ia	$[Me_2Sn(O_2CC_4H_3S)_2]$	−114	5.2 (630)	170.7	133.7	133.4	128.0	134.6
Ib	$[Et_2Sn(O_2CC_4H_3S)_2]$	−148	18.2 (607), 8.9	171.2	133.8	133.1	127.9	134.5
Ic	$[Pr_2Sn(O_2CC_4H_3S)_2]$	−141	[b]	[b]				
Id	$[Bu_2Sn(O_2CC_4H_3S)_2]$	−141	25.7 (579), 26.0, 26.4, 13.3	170.8	133.6	133.0	127.7	134.4
Ie	$[Me_2SnCl(O_2CC_4H_3S)]$ [b]		6.0	169.5	132.6	133.6	127.8	134.8
If	$[Bu_2SnCl(O_2CC_4H_3S)]$ [b]		26.3, 26.0, 25.8, 13.2	170.8	133.1	133.1	127.7	134.4
	C_4H_3OCOOH	–	–	163.8	143.9	120.2	112.3	147.5
IIa	$[Me_2Sn(O_2CC_4H_3O)_2]$	−105	5.1	165.8	144.7	118.9	111.8	146.4
IIb	$[Et_2Sn(O_2CC_4H_3O)_2]$	−136	[b]	[b]				
IIc	$[Pr_2Sn(O_2CC_4H_3O)_2]$	−131	28.2 (566), 17.9, 17.4	166.4	144.4	118.9	111.7	146.4
IId	$[Bu_2Sn(O_2CC_4H_3O)_2]$	−129	26.0 (566), 26.2, 25.7, 13.1	166.7	144.5	118.8	111.7	146.3
IIe	$[Pr_2SnCl(O_2CC_4H_3O)]$ [b]		28.9 (516), 18.1, 17.4	166.6	143.9	119.5	111.9	146.8
IIf	$[Bu_2SnCl(O_2CC_4H_3O)]$	−129 (broad)	26.4 (503), 25.9, 13.1	166.4	143.9	119.5	111.9	146.8

[a] Values in parentheses are $^{1}J(^{117/119}Sn-^{13}C)$. [b] Not recorded.

The assignment of IR bands of these complexes has been made by comparison with the IR spectra of related organotin compounds, the free acids and their sodium salts, R_2SnCl_2 and R_2SnO [14,15]. The magnitude of $\Delta\nu$ ($\Delta\nu = \nu_{asym}COO - \nu_{sym}COO$) of 210–250 cm^{-1} for I and II is comparable to those for the corresponding sodium salts, which indicates the presence of bidentate carboxylate groups [16]. The absorption bands in the regions 465–530 and 560–600 cm^{-1} were tentatively assigned to ν(Sn–O) and ν(Sn–C), respectively [17].

The ^{1}H NMR spectra showed the expected integration and peak multiplicities. The dimethyltin compounds exhibited a singlet for the methyl groups at δ 1.16 ppm with $^{2}J(^{119}Sn-C-^{1}H)$ of 82 Hz for Ia, 82.5 Hz for Ie and 76 Hz for IIa. The magnitude of ^{2}J(Sn–H) for I is comparable to that of $[Me_2Sn(O_2CCH_3)_2]$, 82.5 Hz [11], indicating a similar structure in solution.

The ^{13}C NMR spectra (see Table 2 for ^{13}C and ^{119}Sn data) for these complexes showed only one set of Sn–R and ligand carbon resonances. The C–Sn–C angle in $[Me_2Sn(O_2CC_4H_3S)_2]$ has been calculated to be 132° using Holecek's equation [18]. This value is comparable to that for $[Me_2Sn(O_2CCH_3)_2]$ (135.9°) [11] and $[Me_2Sn(O_2C_6H_4NH_2\text{-}4)_2]$ (135.3°) [19]. The ^{119}Sn chemical shifts for the bis complexes occur in the region −129 to −148 ppm and are consistent with those reported for the diorganotin diacetates and dibenzoates [20–22]. The ^{13}C and ^{119}Sn NMR data for the $[R_2Sn(O_2CC_4H_3E)_2]$ complexes are consistent with six coordinate Sn with chelated carboxylate ligands.

The carbonyl carbon signal is deshielded by 1.9 to 3.2 ppm on complexation, whereas the C-2 and C-5 carbon resonances showed little change in their position on

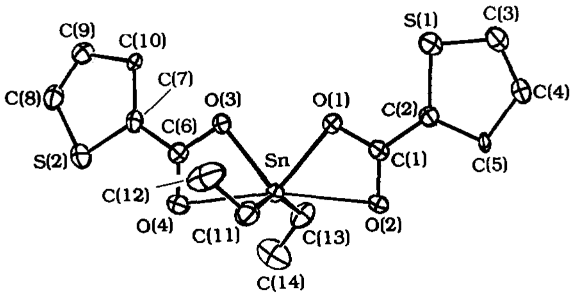

Fig. 1. Molecular structure and crystallographic numbering scheme employed for [$Et_2Sn(O_2CC_4H_3S)_2$].

complexation. This further suggests that the hetero atoms do not coordinate the Sn atom in I and II unlike that observed for the 2-pyridyl complexes [5,6,13]. This observation is supported by a crystal structure analysis of the [$Et_2Sn(O_2CC_4H_3S)_2$] compound.

The molecular structure of [$Et_2Sn(O_2CC_4H_3S)_2$] is shown in Fig. 1 and selected interatomic parameters are listed in Table 3. From Fig. 1 it can be seen that the Sn atom exists in a skew-trapezoidal planar geometry in which the basal plane is defined by the four O atoms derived from two chelating carboxylate ligands. The two remaining positions are occupied by the organic substituents which lie over the weaker Sn–O bonds. The carboxylate ligands chelate the Sn centre with asymmetric Sn–O bond distances and this asymmetry is reflected in the associated C–O bond distances; as expected the longer C–O bond distances are associated with the shorter Sn–O bonds (Table 3). The degree of asymmetry in the Sn–O bond distances is not equal however, with the difference between Sn–O bond distance for the two carboxylate ligands being 0.413 and 0.344 Å, respectively. There is no evidence of intra- or inter-molecular coordination to Sn by the S atoms of the

Table 3

Selected interatomic parameters (Å; deg) for [$Et_2Sn(O_2CC_4H_3S)_2$]

Sn–O(1)	2.143(5)	Sn–O(2)	2.556(5)
Sn–O(3)	2.129(5)	Sn–O(4)	2.473(5)
Sn–C(11)	2.110(9)	Sn–C(13)	2.124(9)
C(1)–O(1)	1.291(8)	C(1)–O(2)	1.237(8)
C(6)–O(3)	1.305(8)	C(6)–O(4)	1.243(9)
O(1)–Sn–O(2)	54.4(2)	O(1)–Sn–O(3)	82.0(2)
O(1)–Sn–O(4)	138.2(2)	O(1)–Sn–C(11)	100.6(3)
O(1)–Sn–C(13)	99.9(3)	O(2)–Sn–O(3)	136.3(2)
O(2)–Sn–O(4)	167.1(2)	O(2)–Sn–C(11)	90.1(3)
O(2)–Sn–C(13)	86.3(3)	O(3)–Sn–O(4)	56.2(2)
O(3)–Sn–C(11)	102.2(3)	O(3)–Sn–C(13)	99.7(3)
O(4)–Sn–C(11)	89.5(3)	O(4)–Sn–C(13)	88.0(3)
C(11)–Sn–C(13)	151.7(4)	Sn–O(1)–C(1)	102.3(4)
Sn–O(2)–C(1)	84.3(4)	Sn–O(3)–C(6)	99.7(4)
Sn–O(4)–C(6)	85.5(4)	O(1)–C(1)–O(2)	119.1(6)
O(1)–C(1)–C(2)	118.8(6)	O(2)–C(1)–C(2)	122.1(6)
O(3)–C(6)–O(4)	118.3(6)	O(3)–C(6)–C(7)	117.5(6)
O(4)–C(6)–C(7)	124.2(7)		

thiophene residues. This finding is consistent with previous observations which suggest that only pyridine type N atoms, present in the carboxylic acid R′ group, coordinate to Sn but not O and S atoms unless these are present as anions as in $[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CCH}_2\mathrm{CH}_2\mathrm{S})]_6$ for example [22].

While the structure of $[\mathrm{Et}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})_2]$ conforms to the structural motif found in similar formulations, there is a distinguishing feature, namely the presence of a close intermolecular Sn ··· O(2) contact. Two very recent contributions in this field have reported similar intermolecular contacts between centrosymmetrically related molecules of 3.451(5) Å in $[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_6\mathrm{H}_4\mathrm{Br}\text{-}4)_2]$ [12] and 2.96(1) Å in $[\mathrm{Me}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_6\mathrm{H}_5)_2]$ [1]. In the present compound the Sn ··· O(2)′ contact (where O(2)′ is related by the symmetry operation $-x, -y, -z$) is only 2.891(6) Å. It is worth noting that the sum of the covalent radii of Sn and O is 2.13 Å and while the Sn ··· O interaction is longer than this distance, it is less than the sum of the van der Waals radii for these atoms of 3.70 Å [23].

If the O(2)′ contact of 2.891(6) Å were to be considered a significant bonding interaction, then the Sn atom geometry would be one based on a distorted pentagonal bipyramid with the two axial positions being occupied by the organic substituents which define a C–Sn–C angle of 151.7(4)°. The sum of the angles about the basal plane is 360° with angles subtended at the Sn atom in the rather large range 54.4(2) to 102.9(2)°. In a recent report, describing the structure of $[\mathrm{Me}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_6\mathrm{H}_5)_2]$ [1], it was concluded on the basis of electronic arguments that a close intermolecular contact of 2.96(1) Å, i.e. similar to that found in $[\mathrm{Et}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})_2]$, was not to be considered a significant bonding interaction. Although comparison between the structure of $[\mathrm{Et}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})_2]$ and other structures of the general formula $[\mathrm{R}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CR}')_2]$ is difficult, owing to the presence of different R and R′ groups, there is no significant perturbation in the Sn atom geometry in the structure of $[\mathrm{Et}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})_2]$ compared to other related $[\mathrm{R}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CR}')_2]$ compounds, even those with no additional Sn ··· O interactions, to warrant its description as one based on a pentagonal bipyramid.

No crystals suitable for X-ray analysis were obtained for the $[\mathrm{R}_2\mathrm{Sn}(\mathrm{Cl})(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{E})]$ species and thus unambiguous structure assignment could not be made. In the literature there is one example of a comparable compound, namely $[\mathrm{Me}_2\mathrm{SnCl}\text{-}\mathrm{O}_2\mathrm{CCH}_3)]$ that was shown to be polymeric in the solid state [24]. This structure features trigonal bipyramidal Sn centres with the axial positions being occupied by bridging O atoms derived from the bidentate carboxylate ligands; the basal planes are defined by the two organic substituents and the chloride atom. This structure resembles closely those found for the ubiquitous *trans*-$\mathrm{O}_2\mathrm{SnR}_3$ structural type [25].

Experimental

Dibutyltinoxide, 2-thiophenecarboxylic acid and 2-furancarboxylic acid were obtained from Fluka. $\mathrm{R}_2\mathrm{SnCl}_2$ and $\mathrm{R}_2\mathrm{SnO}$ were prepared in the laboratory by standard methods. Dried analytical grade solvents were used in all experiments. The IR spectra were recorded as Nujol/Fluorolube mulls between CsI optics on a Perkin–Elmer 577 spectrophotometer. The ^{1}H, ^{13}C and ^{119}Sn NMR spectra were recorded on a Varian FT-80A NMR spectrometer operating at 79.54, 20.00 and 29.63 MHz, respectively. Chemical shifts are reported in ppm from internal chloro-form peak (7.26 ppm for ^{1}H and 77.0 ppm for ^{13}C) and external 33% Me_4Sn in C_6D_6 for ^{119}Sn. Microanalyses were performed by the Bio-organic Division of B.A.R.C.

Preparation of [$Et_2Sn(O_2CC_4H_3S)_2$]

To a benzene suspension of Et_2SnO (0.65 g, 3.34 mmol) was added a benzene solution of 2-thiophenecarboxylic acid (0.89 g, 6.99 mmol). The mixture was heated under reflux for 7 h. The water formed during the reaction was removed azeotropically with a Dean and Stark assembly. The clear solution thus obtained was reduced to 10 cm^3 under vacuum and hexane (20 cm^3) was added. On cooling this solution gave prismatic crystals in 65% yield. All the bis complexes were prepared by this method. Pertinent data for these compounds are given in Tables 1 and 2.

Preparation of [$^nPr_2Sn(Cl)(O_2CC_4H_3O)_2$]

To a benzene solution of [$^nPr_2Sn(O_2CC_4H_3O)_2$] (1.71 g, 4.0 mmol) was added a benzene solution of $^nPr_2SnCl_2$ (1.10 g, 4.0 mmol). The mixture was heated under reflux for 3 h. Excess solvent was evaporated under reduced pressure to give a white solid, which was recrystallized from hexane in 89% (2.51 g) yield.

Crystallography

Intensity data for [$Et_2Sn(O_2CC_4H_3S)_2$] was measured at room temperature on an Enraf–Nonius CAD4F diffractometer fitted with graphite monochromatized Mo-K_α radiation, $\lambda = 0.7107$ Å. The $\omega : 2\theta$ scan technique was employed to measure data up to a maximum Bragg angle of 22.5°. The data set was corrected for Lorentz and polarization effects however, no absorption correction was applied owing to the spherical nature of the crystal. Relevant crystal data are given in Table 4.

Table 4

Crystal data and refinement details for [$Et_2Sn(O_2CC_4H_3S)_2$]

Compound	[$Et_2Sn(O_2CC_4H_3S)_2$]
Formula	$C_{14}H_{16}O_4S_2Sn$
Molecular weight	431.1
Crystal system	monoclinic
Space group	$P2_1/n$
a, Å	13.671(3)
b, Å	7.963(1)
c, Å	15.986(2)
β, deg	91.87(1)
V, $Å^3$	1739.3
Z	4
D_c, g cm^{-3}	1.646
$F(000)$	856
μ, cm^{-1}	15.79
No. of data collected	3000
No. of unique data	2268
No. of unique reflections used with $I \geq 2.5\sigma(I)$	1813
R	0.041
g	0.0033
R_w	0.047
Residual ρ_{max}, e $Å^{-3}$	0.80

Table 5

Fractional atomic coordinates ($\times 10^5$ for Sn and $\times 10^4$ for remaining atoms) for $[Et_2Sn(O_2CC_4H_3S)_2]$

Atom	x	y	z
Sn	16465(3)	5848(6)	1623(3)
S(1)	1842(2)	−3210(3)	−2409(1)
S(2)	4475(2)	4268(3)	1579(2)
O(1)	1907(3)	−986(6)	−896(3)
O(2)	335(3)	−969(6)	−712(3)
O(3)	3185(3)	961(6)	110(3)
O(4)	2596(4)	2409(7)	1143(3)
C(1)	1034(5)	−1455(8)	−1121(4)
C(2)	904(5)	−2526(7)	−1848(4)
C(3)	1137(7)	−4263(11)	−3054(5)
C(4)	192(7)	−4213(11)	−2881(6)
C(5)	−70(4)	−3197(7)	−2152(3)
C(6)	3308(5)	2057(9)	711(5)
C(7)	4277(5)	2798(8)	830(4)
C(8)	5632(7)	4445(11)	1344(7)
C(9)	5904(6)	3468(10)	723(6)
C(10)	5127(4)	2379(7)	340(3)
C(11)	1548(6)	−1236(13)	1115(6)
C(12)	2425(9)	−1983(21)	1440(9)
C(13)	1147(7)	2795(11)	−462(8)
C(14)	923(37)	4409(30)	−91(18)
C(14′)	1655(31)	3461(45)	−907(28)

The structure was solved from the interpretation of the Patterson synthesis [26] and refined by a full-matrix least-squares procedure based on F [26]. All non-H atoms were refined with anisotropic thermal parameters and hydrogen atoms were included in each model at their calculated positions except for the disordered ethyl group. The methyl group, $C(14)H_3$, was found to be disordered over two positions such that the site occupancy factor for each was fixed at 50%. After the inclusion of a weighting scheme of the form, $w = [\sigma^2(F) + g|F|^2]^{-1}$, the refinement was continued until convergence; final refinement details are listed in Table 4. The analysis of variance showed no special features indicating that an appropriate weighting scheme had been applied. An examination of the thermal parameters associated with the pairs of atoms S(1), C(5) and S(2), C(10) indicated the possibility of positional disorder between each pair of atoms. Unlike that found recently for $\{[Me_2Sn(O_2CC_4H_3S)]_2O\}_2$ [27], it was not possible to model this disorder, indicating that it is only minor. Fractional atomic coordinates are listed in Table 5 and the numbering scheme employed is shown in Fig. 1 which was drawn with ORTEP [28] at 15% probability ellipsoids. Scattering factors were as incorporated in the SHELX76 program [26] and the refinement was performed on a SUN4/280 computer. Other crystallographic details (available from ERTT) comprise thermal parameters, H-atom parameters, all bond distances and angles, and tables of observed and calculated structure factors.

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