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Journal of Organometallic Chemistry 691 (2006) 1631–1636
Journal of Organometallic Chemistry
www.elsevier.com/locate/jorganchem

Synthesis and crystal structures of new complexes of di- and tribenzyltin 2-amino-1-cyclopentene-1-carbodithioates

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Received 24 November 2005; accepted 24 November 2005
Available online 27 January 2006

Abstract

Treatment of dibenzyltin dichloride with 1 equiv. of 2-amino-1-cyclopentene-1-carbodithioic acid (ACDA) gave $Bz_2SnCl(ACDA)$ (**1**) that contains five coordinate tin. Reaction of 2 equiv. of ammonium 2-amino-1-cyclopentene-1-carbodithioic acid (AACD) with Bz_2SnCl_2 and then recrystallization from THF produced $Bz_2Sn(ACDA)_2 \cdot THF$ **2** in which the coordination geometry around the Sn is highly distorted from octahedral. $Bz_3Sn(ACDA)$ (**3**) was obtained from reaction of Bz_3SnCl with 1 equiv. of AACD. The crystal structure of **3** indicates a Sn–S′ interaction [3.0823(5) Å] that distorts the tin coordination geometry from that of an ideal tetrahedron. In **1**–**3** the tin atom is also coordinated to one carbon atom of each benzyl group. The products were characterized by IR and NMR (^1H, ^{13}C, ^{119}Sn) spectroscopy and elemental analysis.
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Keywords: Tin complexes; Organotin(IV) compounds; Dithiocarboxylato ligands; Crystal structures; Coordination number

1. Introduction

Increasing industrial use of organotin(IV) compounds containing an Sn–S bond, especially as stabilizers of polyvinyl chlorides [1] and recognition of the importance of this bond for the biological properties of organotin compounds [2], have together spurred on the study of thiolates of tin [3]. Therefore, interest in ACDA complexes of organotin species arises in part because of their remarkable diversity in structures as well as because of their probable antitumoral activities and industrial uses [4–7].

The presence of competing reactive centers in ACDA adds to the interest in the study of such a sulfur–nitrogen containing ligand [8]. Chelating agents, which are akin to this type of ligands would have tendency to exhibit ambidentate nature. The ligands may act in monodentate fashion, fully or partially bidentate and even bridging as well as bidentate [9], so that the coordination number of metal may appear ambiguous [10]. Triorganotin derivatives of bidentate ligands are reported to have tetragonal [11] or trigonal bipyramidal (Tbp) [12] geometry depending upon the nature of organo-group as well as the electronegativity of the atoms attached with central tin atom. Replacement of one of the organo-groups by more electronegative atom, e.g., Cl leads to the Tbp geometry around tin [13,14]. For diorganotin bis(1,1-dithiolates) four crystal structures have been reported [15].

In this paper, we report our observation on di- and tribenzyltin(IV) complexes with ACDA in which ACDA behaves as a (S, $\overline{S}$) donor anisobidentate ligand. We describe the synthesis and crystal structure of $Bz_2SnCl(ACDA)$ (**1**) in which the Sn atom is in a distorted trigonal dipyramid environment. Examples of structurally studied pentadentate diorganotin complexes of the type R_2ClSnS_2C are limited. In some of these complexes the Sn atom is asymmetrically bonded to the two S atoms with an average difference in the Sn–S bond lengths of 0.3 Å [16], similar to what observed in **1**. The synthesis and crystal structure of $Bz_2Sn(ACDA)_2$ (**2**) are also reported. This

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doi:10.1016/j.jorganchem.2005.11.060

Table 1
Summary of crystal data for **1**–**3**

	$Bz_2SnCl(ACDA)$ **1**	$Bz_2Sn(ACDA)_2$ **2**	$Bz_3Sn(ACDA)$ **3**
Empirical formula	$C_{20}H_{22}ClNS_2Sn$	$C_{26}H_{30}N_2S_4Sn \cdot C_4H_8O$	$C_{27}H_{29}NS_2Sn$
Formula weight	494.65	689.55	550.32
Crystal size (mm^3)	$0.3 \times 0.3 \times 0.2$	$0.20 \times 0.10 \times 0.05$	$0.3 \times 0.3 \times 0.3$
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_{1/C}$ (no. 14)	$P2_{1/C}$ (no. 14)	$P\bar{1}$ (no. 2)
a (Å)	18.4613(4)	13.0498(4)	8.8579(3)
b (Å)	11.2587(3)	11.8810(4)	10.0472(4)
c (Å)	20.4525(4)	20.4455(5)	15.2369(6)
α (°)	90	90	96.556(2)
β (°)	95.502	98.928(2)	95.891(2)
γ (°)	90	90	109.521(2)
Z	8	4	2
V ($Å^3$)	4231.5(2)	3131.6(2)	1255.03(8)
μ (mm^{-1})	1.53	1.11	1.20
θ Range (°)	3.71–30.03	3.78–30.04	3.75–30.02
Index range	$-25 \leqslant h \leqslant 22$	$-18 \leqslant h \leqslant 16$	$-12 \leqslant 1 \leqslant 12$
	$-10 \leqslant k \leqslant 15$	$-13 \leqslant k \leqslant 16$	$-14 \leqslant k \leqslant 12$
	$-28 \leqslant l \leqslant 26$	$-23 \leqslant l \leqslant 28$	$-21 \leqslant l \leqslant 17$
Number of reflections collected	24829	22937	11118
Number of unique reflections [R_{int}]	12164 [0.048]	9116 [0.054]	7253 [0.039]
Number of reflections with $I > 2\sigma(I)$	8815	6472	6702
R_1, wR_2 ($I > 2\sigma(I)$)	0.039, 0.094	0.042, 0.079	0.027, 0.073
(all data)	0.065, 0.107	0.073, 0.089	0.031, 0.075
Number of data/restraints/parameters	12164/0/451	9116/0/359	7253/0/288
GOF on F^2	1.039	1.028	1.174

compound contains two bidentate dithioate ligands giving rise to a distorted octahedral geometry around the Sn atom. We also report the synthesis and crystal structure of $Bz_3Sn(ACDA)$ (**3**) in which due to the presence of the second S atom, the tin coordination geometry is highly distorted from tetrahedral (see Table 1).

2. Results and discussion

2.1. Synthesis

The complex $Bz_2SnCl(ACDA)$ (**1**) was obtained from reaction of Bz_2SnCl_2 with 1 equiv. of 2-amino-1-cyclopentene-1-carbodithioic acid (ACDA) in MeOH. When Bz_2SnCl_2 was treated with 2 equiv. of ACDA, again only mono-substitution occurred, while treatment of the former with 2 equiv. of the ammonium salt of ACDA, ammonium 2-amino-1-cyclopentene-1-carbodithioic acid (AACD), in CH_2Cl_2 and then recrystallization from THF led to $Bz_2Sn(ACDA)_2 \cdot THF$ **2**. Similarly, Bz_3SnCl reacted with one mole proportion of AACD to give $Bz_3Sn(ACDA)$ (**3**) (see Scheme 1).

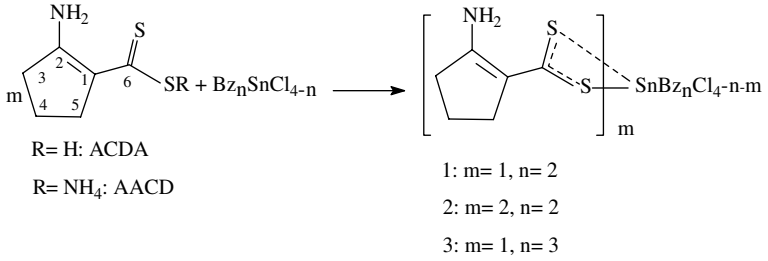
2.2. Vibrational spectra

The IR spectrum of ACDA exhibits a weak intensity band at ≈2550 cm^{-1} due to ν(S–H) which is missing in the spectrum of **1**, but a new band in the range 360–345 cm^{-1} assigned to ν(Sn–S) is seen in the spectra of all the products. An additional band in ≈280 cm^{-1} for **1** may be assigned to ν(Sn–Cl). The band due to ν_{asym} CSS at ≈900 cm^{-1} of the ligand spectra is used in distinguishing the type of sulfur bonding to tin center [19]. It is symmetrically split for **3** indicating the unidentate mode of chelation, but the presence of asymmetrically split bands for **1** and **2** confirm the unequal involvement of the two S atoms in the complexation.

2.3. NMR spectra

A comparison of ^1H NMR spectrum of **1** with that of ACDA shows the disappearance of the signal for the –SH proton upon formation of Sn–S bond. In the spectrum of ACDA, two broad signals assigned to the $–NH_2$ protons are observed at 6.1 and 11.2 ppm, indicating the non-equivalence of these protons because of the involvement of one of them in the (NH—S═) hydrogen bond. In the spectra of the complexes one of the two signals (6.1 ppm) appears almost in the same region while the second signal shifts upfield. This shift may be ascribed to weakening of the hydrogen bond on complexation. The positions of the signals assigned to $CH_2(3)$, $CH_2(4)$ and $CH_2(5)$ do not show significant shifts.

Unfortunately, because of the low solubility of **1** and **2** in common solvents, for these compounds ^{13}C and ^{119}Sn NMR spectra could not be obtained. In the ^{13}C NMR spectrum of **3**, chemical shifts are very similar to those of the ligands. The small shifts in the position of C(6) is considered due to the deshielding of this carbon upon deprotonation of the thiol group and coordination through the other sulfur atom.



Scheme 1.

The ^{119}Sn NMR spectrum of **3** shows a sharp signal at −36.151 ppm that is consistent with the presence of four-coordinate organotin(IV) dithioate species for which a range of 144 to −120 ppm was previously reported [7,20].

2.4. Description of the crystal structures

2.4.1. Crystal structure of $Bz_2SnCl(ACDA)$ (1)

For compound **1**, the selected bond lengths and bond angles are given in Table 2. The unit cell contains two independent molecules that show only minor differences. Atomic numbering scheme and a view of one of the two molecules are represented in Fig. 1. The geometry around the Sn atom is pentacoordinated distorted trigonal bipyramid with two benzylic carbon atoms and S(1) in the equatorial plane. As in all similar molecules, the chloride ion occupies an axial position [7,16,21,22], while the other apical position is occupied by S(2) atom. The sum of the ligand–Sn–ligand angles in the trigonal girdle of the compound is 357.17° instead of the ideal 360°. The Sn–S(2) bond is markedly elongated compared to the Sn–S(1) bond. This makes the dithioate coordination unsymmetrical. Because of being part of a chelate, the atom S(2) cannot occupy exactly the corresponding *trans* axial position, the angle Cl–Sn–S(2) being 158.92° (3) and also the angle S(1)–Sn–S(2) is not 90° but only 69.15(2)°. While the C–Sn–C in the equatorial plane for most of carbodithioate complexes with tin is about 125–128°, similar to $Me_2SnCl[SCH_2CH(NH_2)CO_2Et$ [21], in **1** this angle is only 119.99(15)°. In **1** there are an intramolecular hydrogen bond between one of the two proton atoms of NH_2 and S(2) and an intermolecular hydrogen bond between the other NH_2 proton and Cl of neighboring molecule [x, $-y+1.5$, $z+0.5$].

Table 2
Selected bond lengths (Å) and bond angles (°) for **1**

Sn–C(14)	2.148(3)	Sn–C(7)	2.156(3)
Sn–S(1)	2.4446(8)	Sn–S(2)	2.7465(8)
S(1)–C(1)	1.754(3)	S(2)–C(1)	1.721(3)
NH(1)–S(2)	2.40	NH(2)–Cl	2.59
Sn–Cl	2.4652(8)		
C(14)–Sn–C(7)	119.99(15)	C(14)–Sn–S(1)	117.01(11)
C(7)–Sn–S(1)	120.17(9)	C(14)–Sn–Cl	101.55(9)
C(7)–Sn–Cl	95.38(10)	S(1)–Sn–Cl	89.81(3)
C(14)–Sn–S(2)	89.69(9)	C(7)–Sn–S(2)	94.26(10)
S(1)–Sn–S(2)	69.15(2)	Cl–Sn–S(2)	158.92(3)
C(1)–S(1)–Sn	91.50(10)	C(1)–S(2)–Sn	82.50(10)
C(8)–C(7)–Sn	107.5(2)	C(15)–C(14)–Sn	118.6(2)
N–H(1)–S(2)	134	N–H(2)–Cl	151

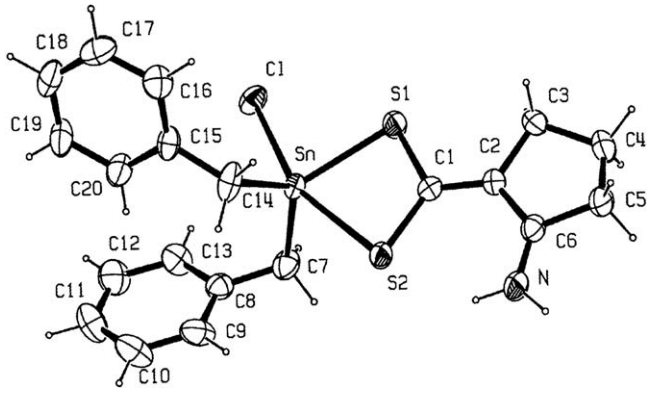


Fig. 1. Crystal structure of $Bz_2SnCl(ACDA)$ (**1**).

2.4.2. Crystal structure of $Bz_2Sn(ACDA)_2 \cdot THF$ (2)

Relevant bond lengths and bond angles are given in Table 3. A view of the molecule including numbering scheme is shown in Fig. 2. The crystals of **2** contain a molecule of THF from which it was recystallized. The dithioate ligands are coordinated asymmetrically with short Sn–S(1) [2.5082(8) Å] and Sn–S(3) [2.5006(7) Å] and long Sn–S(2) [3.1001(8) Å] and Sn–S(4) [2.9530(8) Å] distances. The long

Table 3
Selected bond lengths (Å) and bond angles (°) for **2**

Sn–S(1)	2.5082(8)	Sn–S(2)	3.1001(8)
Sn–S(3)	2.5006(7)	Sn–S(4)	2.9530(8)
Sn–C(13)	2.166(3)	Sn–C(20)	2.169(3)
S(1)–C(1)	1.759(3)	S(2)–C(1)	1.703(3)
S(3)–C(7)	1.757(3)	S(4)–C(7)	1.704(3)
N(1)H(1)–S(2)	2.29	N(2)H(2)–S(4)	2.34
N(1)H(2)–O(1)	1.99		
C(13)–Sn–C(20)	130.64(11)	S(3)–Sn–S(1)	82.89(2)
C(13)–Sn–S(3)	113.85(9)	C(20)–Sn–S(3)	105.43(8)
C(13)–Sn–S(1)	104.50(8)	C(20)–Sn–S(1)	109.03(8)
C(1)–S(1)–Sn	98.72(10)	C(7)–S(3)–Sn	95.74(10)
Sn–C(13)–C(14)	113.33(19)	Sn–C(20)–C(21)	116.26(18)
S(2)–Sn–S(4)	149.81(2)	N(1)–H(2)–O(1)	173
N(1)–H(1)–S(2)	139	N(2)–H(2)–S(4)	137

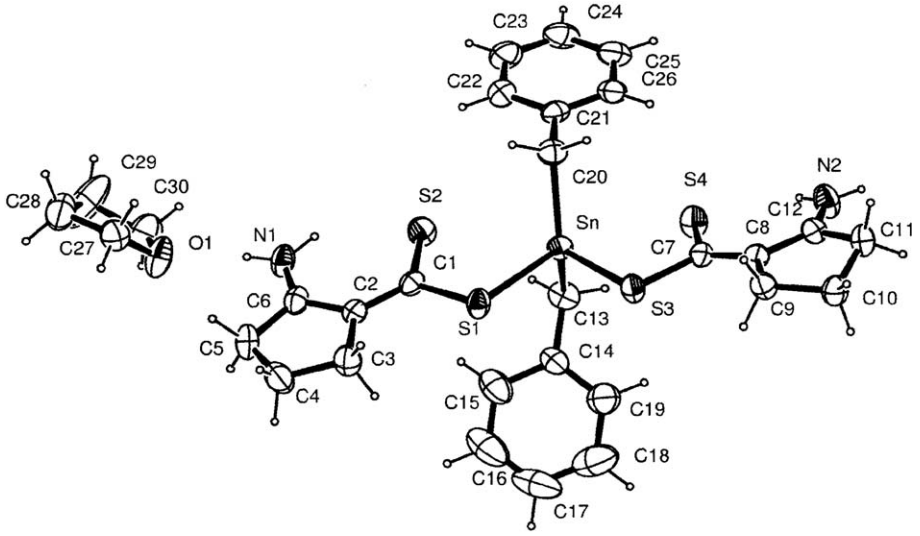


Fig. 2. Crystal structure of $Bz_2Sn(ACDA)_2 \cdot THF$ (**2**).

Sn–S distances are significantly less than the sum of the van der Waals radii (4.0 Å), and the coordination number of tin is assigned as six. The overall geometry at tin is, however, highly distorted from *trans* octahedral: the C–Sn–C angle is only 130.64 which is intermediate between *cis* and *trans*, the tin and four S atoms of the ligands are nearly coplanar but distorted from square-planar geometry, so that Sn–S(2) is about 0.05 Å longer than Sn–S(4) and also while *cis* S(1)-Sn–S(3) angle is only 82.89(2)°, the *cis* S(2)–Sn–S(4) is 149.81(2)°. In both anisobidentate ligands each shorter tin-sulfur bond is associated with a longer carbon–sulfur bond and vice versa; this is in consonance with the bonding asymmetry of the ligands. The bond angles subtended at the tin atom by the methylene carbons and S(1) and S(3) atoms range from 104.50(8)° to 113.85(9)° demonstrating that the tin–carbon bonds are bent toward the longer tin–sulfur bonds. This is obviously a consequence of repulsion between the bonding electron pairs around the central tin atom. Electronic and steric arguments have also been invoked to account for the distortion of similar structures from regular octahedral geometry. The geometry and bond lengths of the SnC_2S_4 core are comparable with those observed for most other octahedral complexes, however, different from $[^tBu_2Sn(IV)]^{2+}$ ethylene bis-dtc and $Ph_2Sn(S_2CNEt_2)_2$ complexes [15,20,22–24].

2.4.3. Crystal structure of $Bz_3Sn(ACDA)$ (**3**)

A perspective view of **3** is shown in Fig. 3 and selected bond lengths and bond angles are listed in Table 4. Similar to **1** and **2**, in **3** the Sn atom is asymmetrically bonded to the two S atoms, however, with a difference in the Sn–S bond lengths of 0.6203 Å. Sn–S(1) bond length is close to the sum of the covalent radii of tin and sulfur (2.42 Å), while the Sn–S(2) may be considered as a weak coordinative bond. Thus, the complex contains essentially four-coordinate tin, but with wide distortion from the normal

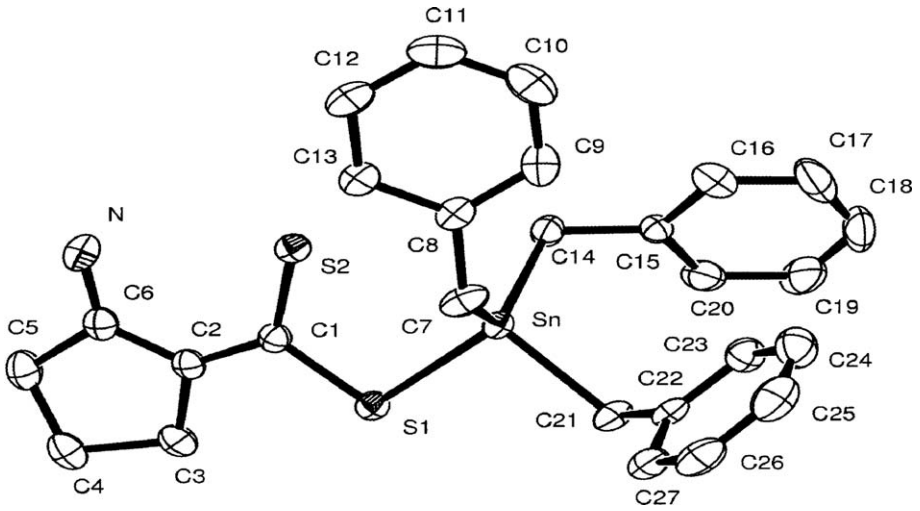


Fig. 3. Crystal structure of $Bz_3Sn(ACDA)$ **3**.

Table 4
Selected bond lengths (Å) and bond angles (°) for **3**

Sn–S(1)	2.4620(5)	Sn–S(2)	3.0823(5)
Sn–C(7)	2.165(2)	Sn–C(14)	2.169(2)
Sn–C(21)	2.197(2)	S(1)–C(1)	1.761(2)
S(2)–C(1)	1.698(2)		
C(7)–Sn–C(14)	119.63(9)	C(7)–Sn–C(21)	104.08(8)
C(14)–Sn–C(21)	108.78(8)	C(7)–Sn–S(1)	113.82(7)
C(14)–Sn–S(1)	111.07(6)	C(21)–Sn–S(1)	96.40(6)
C(1)–S(1)–Sn	98.57	C(8)–C(7)–Sn	115.80(13)
C(15)–C(14)–Sn	112.54(13)	C(22)–C(21)–Sn	110.49(13)
S(1)–C(1)–S(2)	118.39(11)		

tetrahedral environment. Wide variations in C–Sn–C [104.08(8)–119.63(9)°] and S–Sn–C angles [96.40(6)–113.82(7)°] are seen which show the significance of deviations from the tetrahedral value. In this molecule C(21) lies adjacent to the coordinated sulfur atom (S(1)). The Sn–C bond distances are close to those of triphenyltin dithiocarbamates [3,25].

3. Experimental

3.1. General procedure

Starting materials were purchased from commercial sources and used without further purification, except for benzyl chloride and triethylamine, which were distilled at 63 °C/8 mm Hg and 89 °C, respectively, before use. ACDA, AACD [17], di- and tribenzyltin chlorides [18] were prepared using the literature methods. IR spectra were obtained on FT BOMEM MB 102 or SHIMADZU IR-470 infrared spectrophotometers from KBr disks. ^{1}H, ^{13}C and ^{119}Sn NMR spectra were recorded with a Bruker DRX500 AVANCE spectrometer.

3.2. Synthesis of $Bz_2SnCl(ACDA)$ (*1*)

A solution of ACDA (0.400 g, 2.52 mmol) in methanol (10 cm^3) was added dropwise to a solution of Bz_2SnCl_2 (0936 g, 2.52 mmol) in the same solvent (10 cm^3). A crystalline pale yellow precipitate was formed gradually which was filtered off, washed with methanol and recrystallized from methanol to give yellow plates, m.p. 154–156 °C. Yield: 1.1 g (88%). Anal. Calc. for $C_{20}H_{22}ClNS_2Sn$: C, 48.56; H, 4.48; N, 2.83. Found: C, 48.28; H, 4.55; N, 2.82%. ^{1}HNMR: δ 1.86 ($^{3}J_{HH} = 7.37$ Hz, 2H, quintet, H(4)), 2.69 ($^{3}J_{HH} = 7.73$ Hz, 2H, t, H(3)), 2.76 ($^{3}J_{HH} = 7.21$ Hz, 2H, t, H(5)), 3.11 ($^{2}J_{119SnH} = 86.27$ Hz, 4H, s, CH_2–Ph), 6.25 and 8.71 (1H, s, –NH_2), 7.02–7.32 (10H, m, aromatic-H).

3.3. Synthesis of $Bz_2Sn(ACDA)$ (*2*)

AACD (0400 g, 2.27 mmol) was dissolved in warm methanol (10 cm^3) and the resultant solution was added to a solution of Bz_2SnCl_2 (0.420 g, 1.13 mmol) in methanol (10 cm^3). The mixture was stirred for 2 h, during which time an orange precipitate was gradually formed which then was filtered off, washed with methanol and recrystallized from THF at −20 °C to give yellow plates, m.p. 164–166 °C. Yield: 0.38 g (55%). Anal. Calc. for $C_{26}H_{30}N_2S_4Sn \cdot C_4H_8O$: C, 52.25; H, 5.55; N, 4.09. Found: C, 52.04; H, 5.48; N, 4.09%. ^{1}HNMR: δ 1.86 ($^{3}J_{HH} = 7.34$ Hz, 4H, quintet, H(4)), 2.65 ($^{3}J_{HH} = 7.70$ Hz, 4H, t, H(3)), 2.80 ($^{3}J_{HH} = 7.01$ Hz, 4H, t, H(5)), 3.33 ($^{2}J_{119SnH} = 85.87$ Hz, 4H, s, CH_2–Ph), 5.81 and 9.74 (1H, s, –NH_2), 6.97–7.25 (10H, m, aromatic-H).

3.4. Synthesis of $Bz_3Sn(ACDA)$ (*3*)

To a solution of Bz_3SnCl (0.970 g, 2.27 mmol) in CH_2Cl_2 (10 cm^3), a solution of AACD (0.400 g, 2.27 mmol) in methanol (30 cm^3) was added and the mixture was stirred for 18 h. The solution, which its volume was reduced to 20 cm^3, then was kept at −4 °C to give yellow hexagonal plates, m.p. 90 °C. Yield: 0.9 g (72%). Anal. Calc. for $C_{27}H_{29}NS_2Sn$: C, 58.92; H, 5.31; N, 2.55. Found: C, 58.37; H, 5.33; N, 2.63%. ^{1}HNMR: δ 1.78 ($^{3}J_{HH} = 7.34$ Hz, 2H, quintet, H(4)), 2.58 ($^{3}J_{HH} = 7.77$ Hz, 2H, t, H(3)), 2.60 ($^{2}J_{119SnH} = 66.85$ Hz, 6H, s, CH_2–Ph), 2.87 ($^{3}J_{HH} = 7.41$, H, t, H(5)), 5.88 and 10.34 (1H, s, –NH_2), 6.78 ($^{3}J_{HH} = 7.32$ Hz, 6H, δ, *o*-H), 7.00 ($^{3}J_{HH} = 7.12$ Hz, 3H, t, *p*-H), 7.14 ($^{3}J_{HH} = 7.57$ Hz, 6H, t, *m*-H). ^{13}CNMR: δ 19.4 (C(4)), 25.1 ($^{1}J_{119SnC} = 301.1$ Hz, $^{1}J_{117SnC} = 287.7$ Hz, *C*H_2–Ph), 35.7 (C(3)), 36.9 (C(5)), 120.8 (C(1)), 123.9 ($^{4}J_{SnC} = 21.01$ Hz, *m*-C), 127.9 ($^{5}J_{SnC} = 30.1$ Hz, *p*-C), 128.4 ($^{3}J_{SnC} = 17.1$ Hz, *m*-C), 140.8 ($^{2}J_{SnC} = 40.26$ Hz, *i*-C), 168.0 (C(2)), 204.6 (C(6)), ^{119}Sn NMR: δ −36.151 ppm.

3.5. X-ray crystallography

Data were collected on kappa CDD diffractometer at 173 K by use of Mo Kα radiation and the structures were solved by direct methods. Further details are given in Table 1. Full-matrix least-squares against F^2 (SHELXL-97) was used for refinement with non-H atoms isotropic and H atoms in riding mode anisotropic.

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 167034 (**1**), 167035 (**2**) and 167036 (**3**). Copies of the data may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgments

The authors thank Professor Colin Eaborn and Dr. J. David Smith for helpful discussions. Support of this work

by National Research Council of Iran (Project No. 1956) and School of Chemistry, Physics and Environmental Science, University of Sussex (for X-ray facilities) is gratefully acknowledged.

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