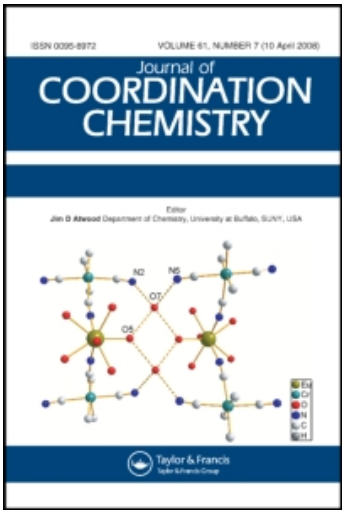

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

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Rufen Zhang[a]; Jiafeng Sun[a]; Chunlin[ab]
[a] Department of Chemistry, Liaocheng University, Liaocheng 252059, P.R. China [b] Taishan University, Taian 271021, P.R. China

To cite this Article Zhang, Rufen , Sun, Jiafeng and Chunlin(2006) 'Synthesis and characterization of diorganotin(IV) derivatives from rhodanine (HRd): crystal structures of $[(PhCH_2)_2Sn(Rd)(\mu\text{-}OH)]_2$ and $[n\text{-}Bu_2Sn(Rd)(\mu\text{-}OH)]_2$', Journal of Coordination Chemistry, 59: 17, 1945 — 1952
To link to this Article: DOI: 10.1080/00958970600734319
URL: http://dx.doi.org/10.1080/00958970600734319

Journal of Coordination Chemistry
Vol. 59, No. 17, 20 November 2006, 1945–1952

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Synthesis and characterization of diorganotin(IV) derivatives from rhodanine (HRd): crystal structures of $[(PhCH_2)_2Sn(Rd)(\mu\text{-}OH)]_2$ and $[n\text{-}Bu_2Sn(Rd)(\mu\text{-}OH)]_2$

RUFEN ZHANG†, JIAFENG SUN† and CHUNLIN MA*†‡

†Department of Chemistry, Liaocheng University, Liaocheng 252059, P.R. China
‡Taishan University, Taian 271021, P.R. China

(*Received in final form 20 January 2006*)

The diorganotin(IV) complexes, $[R_2Sn(Rd)(\mu\text{-}OH)]_2$ (R = Me (**1**), $PhCH_2$ (**2**), *n*-Bu (**3**), Ph (**4**); HRd = rhodanine), have been synthesized and characterized by IR and multinuclear (^1H, ^{13}C, ^{119}Sn) NMR spectroscopy. The structures of complexes **2** and **3** have been determined by single-crystal X-ray diffraction. Both crystal structures of **2** and **3** show the presence of asymmetrically bridging hydroxy groups leading to an Sn_2O_2 unit. Each atom in complex **1** is also coordinated by an N atom of ligand and two C atoms of the alkyl groups, so the Sn environment is based on a trigonal bipyramid. While in complex **2**, a weak intermolecular Sn–O interaction has also been found between the two adjacent molecules, so the geometry of the Sn atom can be best described as six-coordinate octahedral. The salient feature of the supramolecular structure of complex **3** is that of a 1D polymer, in which the discrete molecules are connected through weak intermolecular Sn···O interactions.

Keywords: Diorganotin(IV) complexes; Rhodanine; Crystal structures

1. Introduction

The environmental and biological chemistry of organotin(IV) complexes have been the subject of interest for some time due to their widespread use [1, 2]. In particular, a few diorganotin(IV) derivatives have been shown to exhibit *in vitro* antitumour properties against a wide panel of tumoral cell lines of human origin [3–5]. In order to explore the relationship between biological activity and structure, a large number of new organotin(IV) complexes have been synthesized and studied [6, 7]. Recently, more attention has been paid to the coordination chemistry of polydentate ligands that incorporate both thiol sulfur and heterocyclic nitrogen or oxygen donor sites because of their unusual geometries and moderate biological activities against various bacteria and fungi [8]. Molloy *et al*. [9] and ourselves [10, 11] crystallographically demonstrated for some organotin(IV) derivatives from heterocyclic thionates containing a nitrogen atom (or more) and an adjacent, exocyclic thioketo group, respectively, that the metal-ligand bonding occurs through sulfur.

*Corresponding author. Tel.: +86-635-8239121. Fax: +86-538-6715521. Email: macl@lctu.edu.cn

Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2006 Taylor & Francis
DOI: 10.1080/00958970600734319

Heterocyclic ligands such as rhodanine (HRd), which in spite of having only a five-membered ring possess several different endo- and exocyclic-donating atoms, is interesting because it can exist either neutral or as a mono-anion in solution, and also has a tautomeric equilibrium (see figure 1) according to the literature [12–14]. The HRd ligand has been extensively studied in the synthesis and coordination chemistry of complexes with transition or post-transition elements [15, 16] but few organotin(IV) complexes have been synthesized so far.

In order to widen the scope of coordination behavior of ligands containing {S, O, N} donors towards organotin(IV) derivatives in biological systems [17–19], we report studies on the synthesis of a series of diorganotin(IV) complexes $[R_2Sn(Rd)(\mu\text{-OH})]_2$ (R = Me (**1**), $PhCH_2$ (**2**), *n*-Bu (**3**), Ph (**4**)) and their characterization by IR and multinuclear (^1H, ^{13}C, ^{119}Sn) NMR spectroscopy. X-ray crystallography analyses of complexes **2** and **3** are also described.

2. Results and discussion

2.1. *Syntheses*

The synthetic procedure is shown in the following scheme 1. The Sn_2O_2 moiety in complexes **1**–**4**, which is similar to that found in bis[(μ-hydroxo)-(O,O′-diphenylthiophosphato)-diphenyl-tin(IV)] [20] and bis[acetyl-*t*-butyl-(μ-hydroxo)-tin(IV)] [21], can be considered as a hydrolyzed intermediate of the diorganotin moiety due to the presence of water in the solvent ethanol (95%).

2.2. *Spectroscopic studies*

2.2.1. IR studies of the complexes 1–4. A broad absorption at about 3000 cm^{-1} displayed by complexes **1**–**4** is attributed to ν(O–H) stretching, consistent with that

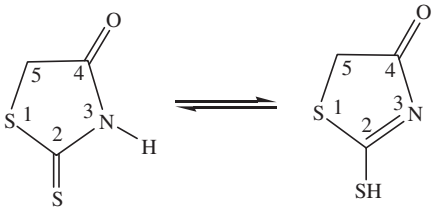
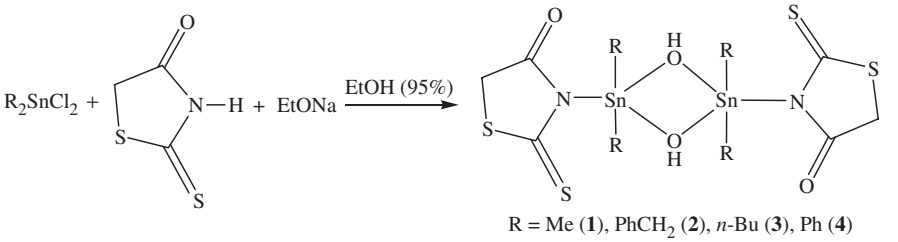


Figure 1. Tautomeric forms of HRd.



R = Me (**1**), $PhCH_2$ (**2**), *n*-Bu (**3**), Ph (**4**)

Scheme 1. Synthetic procedure of complexes **1**–**4**.

found in similar diorganotin complexes, $[^tBu_2Sn(O_2CCH_3)(\mu\text{-OH})]_2$ and $[^tBu_2Sn(\mu\text{-OH})]_2$ [21, 22]. The Sn–O and Sn–C absorptions at 450 and 550 cm^{-1}, respectively, are in the normal range of their stretching values [23].

2.3. *NMR data of the complexes 1–4*

The ^1H NMR spectra show the expected integration and peak multiplicities. In the spectrum of the free ligand a single resonance is observed at 10.7 ppm, which is absent in the spectra of the complexes **1**–**4**, indicating the replacement of the ligand proton by an alkyltin moiety on complex formation. The resonance for CH_2 in complexes **1**–**4** (4.15 ppm for **1**, 4.05 ppm for **2**, 4.12 ppm for **3**, 4.08 ppm for **4**) appears at the same position as in the ligand (4.10 ppm). The ^{13}C NMR spectral patterns are consistent with the formulation of the complexes **1**–**4**. The ^{119}Sn NMR spectra of complexes **1**–**4** exhibit only one sharp signal, and the δ values, −208.3 for **1**, −185.1 for **2**, −192.7 for **3**, −183.9 for **4**, are consistent with a five-coordinate structure in solution [24]. So it can reasonably be assumed that the structures in solution of complexes **1**–**4** are likely similar to those observed in the solid state.

2.4. *Molecular structures*

2.4.1. Structures of complexes 2 and 3. The labeling of the atoms of complexes **2** and **3** are shown in figures 2 and 3, respectively. All hydrogen atoms have been omitted for clarity. Tables 1 and 2 list selected bond lengths and angles for complexes **2** and **3**.

For complex **2**, each tin is pentacoordinate and the coordination environment is comprised of two benzyl substituents, one Rd moiety and two μ-OH groups. The coordination geometry around each tin is distorted trigonal bipyramidal with the axial positions occupied by one bridging hydroxy oxygen (O2A) and the heterocyclic nitrogen (N) (O2A–Sn–N = 154.19(14)°) while the equatorial positions are taken up by the two carbons (C4 and C11) of the benzyl substituents and the other bridging hydroxy

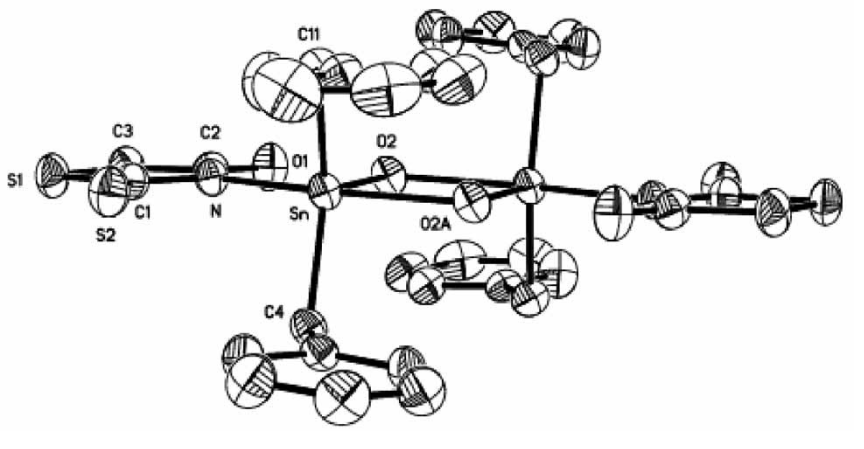


Figure 2. The molecular structure of $[(PhCH_2)_2Sn(Rd)(\mu\text{-OH})]_2$, all H atoms are omitted for clarity.

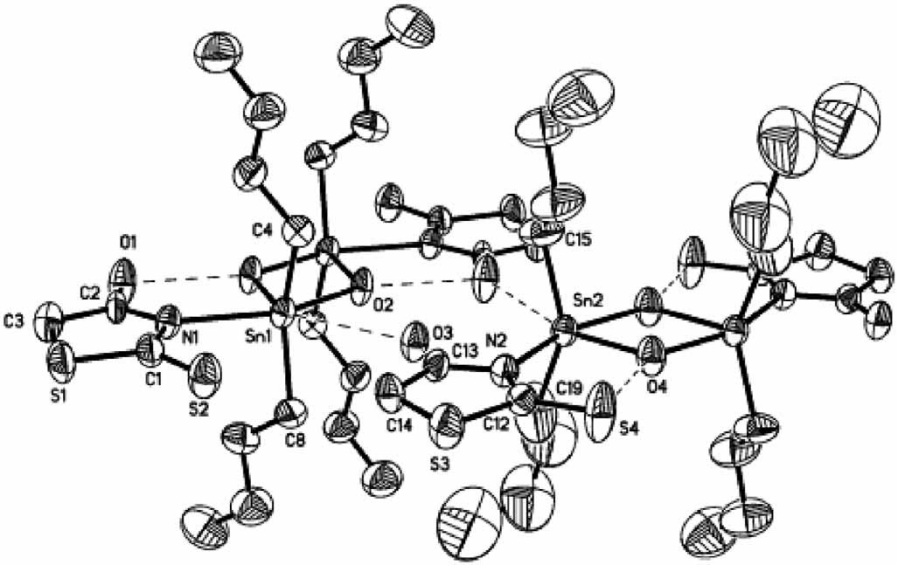


Figure 3. The molecular structure of $[n\text{-}Bu_2Sn(Rd)(\mu\text{-}OH)]_2$, all H atoms are omitted for clarity.

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

Bond lengths			
Sn–O2	2.026(3)	O1–C2	1.208(6)
Sn–C11	2.125(5)	C1–S1	1.736(5)
Sn–C4	2.145(5)	S1–C3	1.775(6)
Sn–O2A	2.282(4)	C1–S2	1.657(6)
Sn–N	2.277(4)		
Bond angles			
O2–Sn–C11	110.05(19)	C4–Sn–O2A	89.42(17)
O2–Sn–C4	111.77(19)	N–Sn–O2	82.22(15)
C4–Sn–C11	136.0(2)	N–Sn–C11	100.45(18)
O2–Sn–O2A	72.07(14)	N–Sn–C4	98.23(18)
O2A–Sn–C11	90.66(17)	N–Sn–O2A	154.19(14)

Symmetry transformations used to generate equivalent atoms, A: $2-x, 2-y, 2-z$.

oxygen (O2). The overall structure is a dimer with the two hydroxy groups acting as bridges between the two tin units, thus leading to the formation of a central Sn_2O_2 ring, which is similar to that found in $\{Me_2[(MeSO_2)_2N]Sn(\mu\text{-}OH)_2Sn[N(SO_2Me)_2]Me_2\}$ [23].

The hydroxyl bridges are not symmetrical with the Sn–O2 and Sn–O2A bond distances being 2.026(3) and 2.282(4) Å, respectively, which is similar to that reported in $[^tBu_2Sn(O_2CCH_3)(\mu\text{-}OH)]_2$ [21]. Each Sn in complex **2** is also coordinated by one N atom derived from the ligand and two C atoms from the benzyl groups. The corresponding Sn–N bond distance is 2.277(4) Å, which is significantly shorter than that found in $\{Me_2[(MeSO_2)_2N]Sn(\mu\text{-}OH)_2Sn[N(SO_2Me)_2]Me_2\}$, [2.475(2) Å] [23]. The Sn···O1 and Sn···S2 separation of 3.626 and 3.322 Å, respectively, are not indicative of bonding interactions. Furthermore, two intramolecular O–H···O hydrogen bonds,

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

Bond lengths			
Sn1–O2#1	2.040(5)	Sn2–O4#2	2.218(5)
Sn1–C4	2.119(6)	Sn2–C15	2.107(8)
Sn1–C8	2.118(7)	Sn2–C19	2.161(9)
Sn1–N1	2.374(5)	O1–C2	1.221(8)
Sn1–O2	2.190(5)	O3–C13	1.203(8)
Sn2–N2	2.364(5)	C1–S2	1.651(8)
Sn2–O4	2.046(5)	S4–C12	1.604(7)
Bond angles			
O2#1–Sn1–C8	107.1(3)	O4–Sn2–C15	108.7(3)
O2#1–Sn1–C4	106.5(3)	O4–Sn2–C19	106.1(4)
C8–Sn1–C4	146.3(3)	C15–Sn2–C19	145.1(5)
O2#1–Sn1–O2	69.2(2)	O4–Sn2–O4#2	69.6(2)
C8–Sn1–O2	95.8(3)	C15–Sn2–O4#2	94.3(3)
C4–Sn1–O2	94.4(2)	C19–Sn2–O4#2	96.8(3)
O2#1–Sn1–N1	83.05(19)	O4–Sn2–N2	87.14(19)
C8–Sn1–N1	91.6(2)	C15–Sn2–N2	92.4(3)
C4–Sn1–N1	94.0(2)	C19–Sn2–N2	90.2(3)
O2–Sn1–N1	152.29(19)	O4#2–Sn2–N2	156.71(19)

Symmetry transformations used to generate equivalent atoms, #1: $2-x$, $1-y$, $1-z$; #2: $2-x$, $-y$, $-z$.

O2–H2···O1 (O2–O1 = 2.612 Å, H2–O1 = 1.812 Å, O2–H2···O1 = 165.93°) and symmetric O2A–H2#···O1# (symmetry operation, A: $2-x$, $2-y$, $2-z$; #: $2-x$, $2-y$, $2-z$), are found between the uncoordinated ligand oxygens O1 or O1# (symmetry operation: $2-x$, $2-y$, $2-z$) and the bridging hydroxyl groups O2 or O2A (symmetry operation: $2-x$, $2-y$, $2-z$).

The asymmetric unit for complex **3** contains two dimers (figure 3), which are crystallographically inequivalent. The conformations of the two independent molecules A and B are almost the same, with only small differences in bond lengths and bond angles (table 2). The Sn in complex **3** also forms five primary bonds; two from the hydroxyl groups, two from the butyl groups and one from the N atom in the ligand. The distance of Sn–N (Sn1–N1 = 2.374(5) Å, Sn2–N2 = 2.364(5) Å) and Sn–O (Sn1–O2#1 = 2.040(5) Å, Sn1–O2 = 2.190(5) Å, Sn2–O4#2 = 2.218(5) Å, Sn2–O4 = 2.046(5) Å, symmetry operation, #1: $2-x$, $1-y$, $1-z$; #2: $2-x$,$-y$, $-z$) are similar to those found in complex **2**. Six weak intermolecular Sn–O interaction (Sn2–O1# = 3.147 Å, Sn1–O3# = 2.971 Å) has also been found between the two adjacent molecules, so the geometry of the Sn atom in complex **3** can be best described as six-coordinate distorted octahedron. The sum of the angles surrounded the two tin atoms (360° for Sn1, 360° for Sn2) are typical for the theoretical value, 360°, while the axial-Sn-axial angles (146.32 for Sn1, 145.13 for Sn2) are bent. The Sn···O and Sn···S separations in complex **3** are also not indicative of bonding interactions. Intramolecular O-H···S (O···S, 3.076 Å), O–H···O (O···O, 2.717 Å) hydrogen bonds are found between the uncoordinated ligand sulfur or oxygen atoms and the bridging hydroxyl groups. The salient feature of the supramolecular structure of complex **3** is that of a 1D polymer, in which the discrete molecules are connected through intermolecular weak interactions of Sn and O atoms (figure 4). The Sn···O distances 3.147 and 2.971 Å are less than the sum of van der Waals radii 3.68 Å [25].

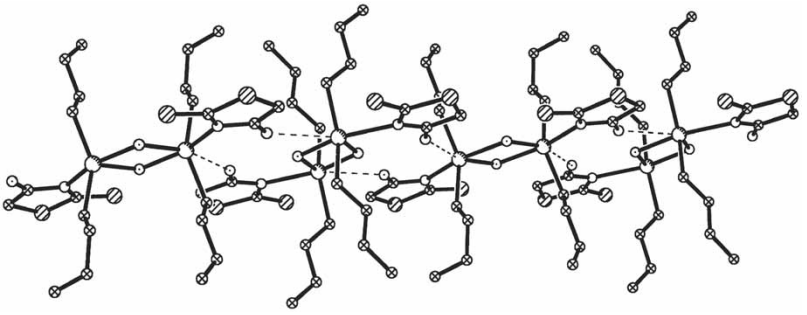


Figure 4. Perspective view showing the 1D chain of $[n\text{-}Bu_2Sn(Rd)(\mu\text{-}OH)]_2$, all H atoms are omitted for clarity.

3. Experimental

3.1. *Materials and measurements*

Dimethyltin dichloride, di-*n*-butyltin dichloride, diphenyltin dichloride and rhodanine were commercially available, and they were used without further purification. Dibenzyltin dichloride was prepared by a standard method reported in the literature [26]. The melting points were obtained with a Kofler micro melting point apparatus and are uncorrected. Infrared spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs and sodium chloride optics. ^1H, ^{13}C and ^{119}Sn NMR spectra were recorded on a Varian Mercury Plus 400 spectrometer operating at 400, 101 and 186.50 MHz, respectively; TMS was used as an internal standard and Me_4Sn was used as an external standard with chemical shifts given in ppm in $CDCl_3$ solvent. Elemental analyses were performed with a PE-2400II apparatus.

3.2. *Syntheses*

3.2.1. Syntheses of complexes 1–4. Rhodanine (0.266 g, 2 mmol) was added to a 30 mL ethanol (95%) solution of sodium ethoxide (0.136 g, 2 mmol) under a nitrogen atmosphere and stirred for 10 min. The appropriate diorganotin dichloride (1 mmol) was then added to the mixture. The reaction was continued for 12 h at 50°C, then cooled to room temperature and filtered. The filtrate solvent was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from a 2 : 1 ratio of ether to petroleum ether. Yellow crystals were formed.

1: Yield 82%, m.p. 134–136°C. Anal. Calcd for $C_{10}H_{18}N_2O_4S_4Sn_2$: C, 20.71; H, 3.13; N, 4.83. Found: C, 20.82; H, 3.25; N, 5.01%. IR (KBr, cm^{-1}): ν(OH) 3005, ν(Sn–C) 558, ν(Sn–O) 442. ^1H NMR ($CDCl_3$, ppm): δ 4.15 (s, 4H, ring-CH_2), 1.22 (s, 12H, $^2J(^{119}Sn\text{–}^1H) = 80$ Hz, Sn–CH_3). ^{13}C NMR ($CDCl_3$, ppm): δ 201.3 (C^2), 174.8 (C^4), 40.5 (C^5), 6.12 ($^1J^{13}C\text{–}^1H = 123$ Hz, Sn–CH_3). ^{119}Sn NMR ($CDCl_3$, ppm): δ −208.3.

2: Yield 85%, m.p. 140–144°C. Anal. Calcd for $C_{34}H_{34}N_2O_4S_4Sn_2$: C, 45.36; H, 3.81; N, 3.11. Found: C, 45.58; H, 3.56; N, 3.31%. IR (KBr, cm^{-1}): ν(OH) 3010, ν(Sn–C) 562, ν(Sn–O) 457. ^1H NMR ($CDCl_3$, ppm): δ 6.90–7.15 (m, 20H, $^2J(^{119}Sn\text{–}^1H) = 83$ Hz,

Sn–$CH_2C_6H_5$), 4.05 (s, 4H, ring-CH_2), 3.47 (s, 8H, Sn–$CH_2C_6H_5$). ^{13}C NMR ($CDCl_3$, ppm): δ 206.9 (C^2), 177.5 (C^4), 138.9, 128.4, 128.1, 125.1 (Sn–$CH_2C_6H_5$), 42.2 (C^5), 33.9 ($^1J^{13}C$–$^1H = 128$ Hz, Sn–$CH_2C_6H_5$). ^{119}Sn NMR ($CDCl_3$, ppm): δ −185.1.

3: Yield 80%, m.p. 110–112°C. Anal. Calcd for $C_{22}H_{42}N_2O_4S_4Sn_2$: C, 34.57; H, 5.54; N, 3.67. Found: C, 34.76; H, 5.32; N, 3.69%. IR (KBr, cm^{-1}): ν(OH) 3008, ν(Sn–C) 550, ν(Sn–O) 463. ^{1}H NMR ($CDCl_3$, ppm): δ 4.12 (s, 4H, ring-CH_2), 0.90–1.87 (m, 36H, $^2J(^{119}Sn–^1H) = 73$, Hz Sn–C_4H_9). ^{13}C NMR ($CDCl_3$, ppm): δ 204.4 (C^2), 175.1 (C^4), 42.7 (C^5), 28.4, 27.6, 26.1, 13.5 ($^1J^{13}C$–$^1H = 130$ Hz, Sn–C_4H_9). ^{119}Sn NMR ($CDCl_3$, ppm): δ −192.7.

4: Yield 87%, m.p. 126–128°C. Anal. Calcd for $C_{30}H_{26}N_2O_4S_4Sn_2$: C, 42.68; H, 3.10; N, 3.32. Found: C, 42.78; H, 3.32; N, 3.54%. IR (KBr, cm^{-1}): ν(OH) 2090, ν(Sn–C) 543, ν(Sn–O) 455. ^{1}H NMR ($CDCl_3$, ppm): δ 7.37–7.80 (m, 20H, $^2J(^{119}Sn–^1H) = 75$ Hz, Sn–C_6H_5), 4.08 (s, 4H, ring-CH_2). ^{13}C NMR ($CDCl_3$, ppm): δ 205.6 (C^2), 176.9 (C^4), 140.8, 136.6, 130.4, 129.2 ($^1J^{13}C$–$^1H = 156$ Hz, Sn–C_6H_5), 41.3 (C^5). ^{119}Sn NMR ($CDCl_3$, ppm): δ −183.9.

3.3. *X-ray crystallography*

Diffraction measurements for a $0.26 \times 0.23 \times 0.16\,mm^3$ sample of **2** and $0.32 \times 0.13 \times 0.10\,mm^3$ sample of **3** were carried out at 298 K on a Bruker Smart CCD 1000 diffractometer (graphite-monochromatized Mo-Kα radiation, $\lambda = 0.71073$ Å) with $\omega/2\theta$ scan technique. The structure was solved by direct methods and refined by a full-matrix least squares procedure based on F^2 using the SHELXL-97 system, and corrected for Lorentz and polarization effects but not for absorption. All non-H atoms were included in the model at their calculated positions. The crystal data and refinement details are given in table 3. CCDC deposition numbers: 238956 (**2**) and 233181 (**3**).

Table 3. Crystal data and refinement details for complexes **2** and **3**.

Complexes	**2**	**3**
Empirical formula	$C_{34}H_{34}N_2O_4S_4Sn_2$	$C_{22}H_{44}N_2O_4S_4Sn_2$
Formula weight	900.25	766.21
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	9.035(9)	12.241(10)
b (Å)	9.608(9)	12.772(10)
c (Å)	11.429(11)	12.912(11)
α (°)	103.779(11)	108.431(11)
β (°)	104.742(12)	98.074(13)
γ (°)	96.909(13)	115.510(11)
V (Å^3)	914.4(15)	1636(2)
Z	1	2
D_c (Mg m^{-3})	1.635	1.556
μ (Mo-Kα) (mm^{-1})	1.633	1.809
Reflections collected	4865	8386
Independent reflections	3205 [$R_{int} = 0.0250$]	5708 [$R_{int} = 0.0281$]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0385$, $wR_2 = 0.0895$	$R_1 = 0.0462$, $wR_2 = 0.1073$
R indices (all data)	$R_1 = 0.0527$, $wR_2 = 0.0949$	$R_1 = 0.0900$, $wR_2 = 0.1273$
$\Delta\rho$ (e Å^{-3})	0.885, −0.643	0.876, −0.660

Supplementary material

Crystallographic data (excluding structure factors) for the structure analysis of complexes **2** and **3** have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos 238956 and 233181. Copies may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; Email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

We thank the National Natural Foundation, P.R. China (20271025), and the National Natural Foundation of Shandong Province, P.R. China for financial support of this work.

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