# Syntheses and Characterizations of Diorganotin(IV) Complexes with L-Cysteine: Crystal Structure of [(CH<sub>3</sub>)<sub>2</sub>Sn(L-C<sub>3</sub>H<sub>5</sub>NO<sub>2</sub>S)·H<sub>2</sub>O]

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ABSTRACT: Five diorganotin(IV) derivatives of Lcysteine have been synthesized and characterized by <sup>1</sup>H, <sup>13</sup>C, <sup>119</sup>Sn NMR, and IR spectroscopies along with elemental analyses. The diorganotin(IV) complexes were readily obtained from the reactions of diorganotin(IV) dichlorides and L-cysteine. The crystal structure of  $[(CH_3)_2Sn(L-C_3H_5NO_2S)\cdot H_2O]$  contains a onedimensional infinite "S" conformation polymeric chain, with the L-cysteine acting as a bridged tridentate ligand. The tin(IV) atom, bonding to two methyl carbons, amino nitrogen atom, thiol sulfur atom, and carboxylate oxygen atom, has a five-coordinated trigonal bipyramid environment. © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:636-641, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10218

# INTRODUCTION

The increasing interest in the chemistry of organotin compounds has led to extended studies on their interactions with different biomolecules, e.g., carbohydrates [1–3], nucleic acid derivatives [4–6], amino acids [7-10], and peptides [11-15]. Several reports revealed the versatile coordination chemical behavior of organotin cations toward molecules containing different types of donor sets, e.g., {O} [1–7], {S, O, N} [9], or {O, N} [7,9,10,15], including both solidstate and solution studies. The exponential increase of the industrial, agricultural, and biological applications of the organotin(IV) compounds during the last 50 years has led to their accumulation in the environment and finally in biological systems [16]. These compounds are generally very toxic, even at low concentration. On the other hand, many dialkyltin derivatives have been found to possess anticancer effects on different tumor cells and their structures in the solid-state are well characterized [17–19].

To widen the scope of investigations on the coordination behavior of ligands in biological systems toward organotins, here we report the synthesis of five diorganotin(IV) derivatives of L-cysteine, and they have been characterized by elemental analyses, <sup>1</sup>H, <sup>13</sup>C, <sup>119</sup>Sn NMR, and IR spectroscopy. Moreover, the crystal structure of [(CH<sub>3</sub>)<sub>2</sub>Sn(L-C<sub>3</sub>H<sub>5</sub>NO<sub>2</sub>S)·H<sub>2</sub>O](1) has been determined by X-ray diffraction, showing that the structural distortion for tin is a displacement from the tetragonal toward a trigonal bipyramidal. The reaction equation is shown in Scheme 1.

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where R= Me (1); Et (2); n-Bu(3); Ph (4); PhCH<sub>2</sub>(5)

### SCHEME 1

# EXPERIMENTAL

# Materials and Instrumentation

All the diorganotin(IV) dichlorides except dibenzyltin dichloride and L-cysteine were commercially available, and they were used without further purification. Dibenzyltin chloride was prepared by a standard method reported in the literature [20]. 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. <sup>1</sup>H, <sup>13</sup>C, and <sup>119</sup>Sn NMR spectra were recorded on a Bruker AMX-300 spectrometer operating at 300, 75.3, and 111.9 MHz, respectively. The spectra were acquired at room temperature (298 K) unless otherwise specified; <sup>13</sup>C spectra are broadband proton decoupled. The chemical shifts were reported in ppm with respect to the references and were stated relative to external tetramethylsilane (TMS) for <sup>1</sup>H and <sup>13</sup>C NMR, and to neat tetramethyltin for <sup>119</sup>Sn NMR. Elemental analyses were performed with a PE-2400II apparatus.

# Synthesis of the Diorganotin Complexes

 $[Me_2Sn(L-C_3H_5NO_2S)\cdot H_2O]$  (1). The reaction was carried out under nitrogen atmosphere with use of standard Schlenk technique. L-Cysteine (0.121 g, 1.0 mmol) and sodium ethoxide (0.136 g, 2.0 mmol) were added to a solution of benzene (30 ml).  $Me_2SnCl_2$  was then added (0.219 g, 1.0 mmol) to the mixture, which was stirred for 12 h at 40°C and then filtrated. The filtered solution was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ethanol (95%). Colorless crystals that contain one molecule of water as the solvent of crystallization was formed. Yield, 72%; m.p. 192–194°C. Anal. Calcd for C<sub>5</sub>H<sub>13</sub>NO<sub>3</sub>SSn (FW: 285.91): C, 21.00; H, 4.58; N, 4.90. Found: C, 20.96; H, 4.56; N, 4.88%. IR (KBr): 3020–3432 (b, NH<sub>2</sub>, H<sub>2</sub>O); 1611  $\nu_{as}$  (COO<sup>-</sup>); 1386  $\nu_{s}$  $(COO^{-})$ ; 569  $\nu_{as}$  (m, Sn–C); 530  $\nu_{s}$  (m, Sn–C); 642 (m, C–S); 536 (s, Sn–O); 405 (w, Sn  $\leftarrow$  N); 310 (s, Sn–S). <sup>1</sup>H NMR (CDCl<sub>3</sub>-D<sub>2</sub>O,  $\delta$  ppm): 0.68 [s, 6H, SnCH<sub>3</sub>,

<sup>2</sup>*J* (<sup>1</sup>H-<sup>119</sup>Sn = 76 Hz)], 3.36 (m, 2H, CH<sub>2</sub>), 4.16 (t, 1H, CH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, ppm):  $\delta$  0.99 [<sup>1</sup>*J*(<sup>13</sup>C-<sup>119</sup>Sn) = 594 Hz], 179.36 (C1), 57.68 (C2), 29.72 (C3). <sup>119</sup>Sn NMR (CDCl<sub>3</sub>):  $\delta$  –146.6 ppm.

 $[Et_2Sn(L-C_3H_5NO_2S)\cdot H_2O]$  (2). Complex 2 was prepared similarly to complex 1, by adding Et<sub>2</sub>SnCl<sub>2</sub> (0.247 g, 1.0 mmol) to L-cysteine (0.121 g, 1.0 mmol) and sodium ethoxide (0.136 g, 2.0 mmol) mixture, stirring for 16 h at 40°C. The solid was then recrystallized from ethanol (95%), which also contained one molecule of water as the solvent of crystallization. Yield, 76%; m.p. 208-210°C. Anal. Calcd for C<sub>7</sub>H<sub>17</sub>NO<sub>3</sub>SSn (FW: 295.93): C, 26.78; H, 5.41; N, 4.46. Found: C, 26.73; H, 5.39; N, 4.43%. IR (KBr): 3024–3309 (b, NH<sub>2</sub>, H<sub>2</sub>O); 1610  $\nu_{as}$  (COO<sup>-</sup>); 1388  $\nu_{s}$ (COO<sup>-</sup>); 592  $\nu_{as}$  (m, Sn–C); 545  $\nu_{s}$  (m, Sn–C); 646 (m, C–S); 530 (s, Sn–O); 408 (w, Sn  $\leftarrow$  N); 309 (s, Sn–S). <sup>1</sup>H NMR (CDCl<sub>3</sub>-D<sub>2</sub>O,  $\delta$  ppm): 1.32 (t, 6H, -CH<sub>3</sub>), 1.59(q, 4H, -CH<sub>2</sub>), 3.34 (m, 2H, CH<sub>2</sub>), 4.00 (t, 1H, CH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, ppm): δ 20.2 (<sup>α</sup>C), 9.8 (<sup>β</sup>C), 179.59 (C1), 58.12 (C2), 29.76 (C3). <sup>119</sup>Sn NMR (CDCl<sub>3</sub>):  $\delta$  -150.2 ppm.

 $[(n-Bu)_2Sn(L-C_3H_5NO_2S)]$  (3). Complex 3 was prepared similarly to complex 1, by adding  $Bu_2SnCl_2$ (0.303 g, 1.0 mmol) to L-cysteine (0.121 g, 1.0 mmol) and sodium ethoxide (0.136 g, 2.0 mmol) mixture, stirring for 10 h at 40°C. The solid was then recrystallized from benzene-hexane (v/v, 1:3). Yield, 70%; m.p. 210-212°C. Anal. Calcd for C<sub>11</sub>H<sub>23</sub> NO<sub>2</sub>SSn (FW: 352): C, 37.53; H, 6.53; N, 3.98. Found: C, 37.49; H, 6.51; N, 3.95%. IR (KBr): 3294 (bd, NH<sub>2</sub>); 1617 ν<sub>as</sub>  $(COO^{-})$ ; 1385  $\nu_{s}$   $(COO^{-})$ ; 587  $\nu_{as}$  (m, Sn–C); 517  $\nu_{s}$  (m, Sn−C); 640 (m, C−S); 550 (s, Sn−O); 416 (w, Sn ← N); 313 (s, Sn-S). <sup>1</sup>H NMR (CDCl<sub>3</sub>-D<sub>2</sub>O, δ ppm): 0.90 (t, 6H, -CH<sub>3</sub>), 1.32-1.79 (m, 12H, -CH<sub>2</sub>), 3.32 (m, 2H, CH<sub>2</sub>), 3.97 (t, 1H, CH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, ppm): δ 26.32 (°C), 26.6 (<sup>β</sup>C), 26.43 (<sup>γ</sup>C), 13.4 (<sup>δ</sup>C), 179.62 (C1), 58.10 (C2), 29.82 (C3). <sup>119</sup>Sn NMR (CDCl<sub>3</sub>):  $\delta$ -165.1 ppm.

 $[Ph_2Sn(L-C_3H_5NO_2S)]$  (4). Complex 4 was prepared similarly to complex 1, by adding Ph<sub>2</sub>SnCl<sub>2</sub> (0.343 g, 1.0 mmol) to L-cysteine (0.121 g, 1.0 mmol) and sodium ethoxide (0.136 g, 2.0 mmol) mixture, stirring for 16 h at 40°C. The solid was then recrystallized from benzene–hexane (v/v, 1:2). Yield, 68%; m.p. (dec.) 230°C. Anal. Calcd for C<sub>15</sub>H<sub>15</sub>NO<sub>2</sub>SSn (FW: 392): C, 45.96; H, 3.83; N, 3.57. Found: C, 45.91; H, 3.85; N, 3.52%. IR (KBr): 3300 (bd, NH<sub>2</sub>); 1615  $\nu_{as}$ (COO<sup>-</sup>); 1367  $\nu_{s}$  (COO<sup>-</sup>); 579  $\nu_{as}$  (m, Sn–C); 531  $\nu_{s}$  (m, Sn–C); 640 (m, C–S); 535 (s, Sn–O); 402 (w, Sn  $\leftarrow$  N); 308 (s, Sn–S). <sup>1</sup>H NMR (CDCl<sub>3</sub>-D<sub>2</sub>O,  $\delta$  ppm): 7.09– 7.78 (m, 10H), 3.38 (m, 2H, CH<sub>2</sub>), 4.11 (t, 1H, CH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, ppm):  $\delta$  138.0 (°C), 136.1 (<sup>β</sup>C), 129.6 (°C), 129.3 (<sup>8</sup>C), 179.18 (C1), 57.91 (C2), 30.13 (C3). <sup>119</sup>Sn NMR (CDCl<sub>3</sub>):  $\delta$  –197.8 ppm.

 $[(PhCH_2)_2Sn(L-C_3H_5NO_2S)]$  (5). Complex 5 was prepared similarly to complex 1, by adding (PhCH<sub>2</sub>)<sub>2</sub>SnCl<sub>2</sub> (0.371 g, 1.0 mmol) to L-cysteine (0.121 g, 1.0 mmol) and sodium ethoxide (0.136 g, 1.0 mmol)2.0 mmol) mixture, stirring for 12 h at 40°C. The solid was then recrystallized from benzene-hexane (v/v, 1:1). Yield, 70%; m.p. (dec.) 250°C. Anal. Calcd for C<sub>17</sub>H<sub>19</sub>NO<sub>3</sub>SSn (FW: 427.03): C, 47.82; H, 4.50; N, 3.28. Found: C, 47.80; H, 4.47; N, 3.24%. IR (KBr): 3293 (bd, NH<sub>2</sub>); 1619  $\nu_{as}$  (COO<sup>-</sup>); 1321  $\nu_{s}$  (COO<sup>-</sup>); 575  $\nu_{as}$  (m, Sn–C); 530  $\nu_{s}$  (m, Sn–C); 648 (m, C–S); 532 (s, Sn–O); 417 (w, Sn  $\leftarrow$  N); 307 (s, Sn–S). <sup>1</sup>H NMR (CDCl<sub>3</sub>-D<sub>2</sub>O, δ ppm): 7.29–7.55 (m, 10H), 2.95 (s, -CH<sub>2</sub>), 3.35 (m, 2H, CH<sub>2</sub>), 3.98 (d, 1H, CH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, ppm): δ 30.15 (\*CH<sub>2</sub>), 127–129.95 (-C<sub>6</sub>H<sub>5</sub>), 179.90 (C1), 57.86 (C2), 29.83 (C3). <sup>119</sup>Sn NMR (CDCl<sub>3</sub>):  $\delta$  –193.6 ppm.

# X-Ray Crystallography

A colorless crystal of complex **1** suitable for X-ray analysis  $(0.23 \times 0.15 \times 0.12 \text{ mm})$  was obtained on a Bruker SMART CCD 1000 diffractometer. Corrections were applied for Lorentz and polarization effects but not for absorption, satisfying the  $I \ge 2\sigma(1)$ and the structure was solved by direct methods and refined by a full-matrix least-squares procedure based on  $F^2$  using the SHELXL-97 program system. All non-H atoms were included in the model at their calculated positions. The positions of hydrogen atoms were calculated, and their contributions in structural factor calculations were included. The crystal belongs to orthorhombic space group  $P2_12_12_1$ , with a = 9.858(4), b = 10.635(4), c =11.329(4) Å, V = 1187.7(8) Å<sup>3</sup>, Z = 4,  $D_c = 1.599$  g  $\text{cm}^{-3}$ ,  $\mu = 2.298 \text{ mm}^{-1}$ , F(000) = 560.

# **RESULTS AND DISCUSSION**

Both complexes **1** and **2** contain one molecule of water as the solvent crystallization when they are recrystallized from the ethanol (95%). The complexes **3**, **4**, and **5** have little solubility in the ethanol (95%),

and so they are recrystallized from the benzenehexane, and results show that they do not contain water molecules.

The infrared spectra of the water-containing ligand and complexes **1** and **2** show one broad  $\nu$ (OH) band in the region 3020–3432 cm<sup>-1</sup>. Studies of all the diorganotin(IV) derivatives of the L-cysteine show that the vibration associated with the NH<sub>3</sub><sup>+</sup> group (2140 cm<sup>-1</sup> in the ligand) are missing and appear as  $\nu$ (NH<sub>2</sub>) in the range 3256–3309 cm<sup>-1</sup> (bd). The appearance of a new band of weak intensity in the region 402–417 cm<sup>-1</sup> are assigned to  $\nu$ (Sn  $\leftarrow$  N). This further confirms the coordination of the amino nitrogen to the organotin(IV) group [21].

The explicit feature in the infrared spectra of all the complexes is the absence of the band in the region 2550–2430 cm<sup>-1</sup>, which appears in the free-ligand as the  $\nu$ (S–H) vibration, indicating metal–ligand bond formation through this site. On the other hand, in the far-IR spectra, strong absorptions at 307–313 cm<sup>-1</sup> for all the complexes, which is absent in the spectrum of the ligand, are assigned to the Sn–S stretching mode of vibration. The values are consistent with that detected for a number of organotin(IV)–sulfur derivatives [22].

It is to be noted that the difference  $\Delta \nu [\nu_{as}(COO^{-}) - \nu_{s}(COO^{-})]$  is of importance since these frequencies can be used for determining the type of bonding between metal and carboxyl [23]. The difference  $\Delta \nu$  for complexes **1–5** is larger (224–260 cm<sup>-1</sup>) than the difference ( $\Delta \nu = 163$  $cm^{-1}$ ) for the appropriate sodium salt of L-cysteine, and so the carboxylate group coordinate, in a monodenate fashion, the organometallic ions [24]. The appearance of a new medium intensity in the far-IR spectra of all the complexes in the region 530–550 cm<sup>-1</sup>, which may be assigned to  $\nu$ (Sn–O), further supports the bonding of COO group to tin atom [21]. Medium-intensity bands in the region 569-592 and 517-545 cm<sup>-1</sup> can be assigned to  $\nu_{\rm as}({\rm Sn-C})$  and  $\nu_{\rm s}({\rm Sn-C})$ , respectively. This clearly indicates the existence of a bent C-Sn-C moiety in organotin complexes [25].

<sup>1</sup>H NMR data showed that the signal of the –SH proton in the spectrum of the ligand is absent in all complexes, indicating the removal of the SH proton and the formation of Sn–S bonds. This information agrees with what the IR data have revealed. In solution, the polymeric structure would be destroyed, and molecular species are expected to occur [26]. The  ${}^{2}J({}^{119}\text{Sn-}{}^{1}\text{H})$  of complex **1** has a value 76 Hz, typical of five-coordinated tin species [27]. The  ${}^{13}\text{C}$  NMR spectra show a significant download shift of all carbon resonance. The shift is a consequence of an electron density transfer from the ligand to the

acceptor [28]. Only one sharp signal is present in the <sup>119</sup>Sn NMR spectra of all the complexes **1–5**, and the values found are in the range –146.6 to –197.8 ppm, which are consistent with monomeric structure in solution and characteristic of the five-coordinated diorganotin derivatives [27].

# The X-Ray Structure of Complex 1

The crystal data and the most relevant experimental parameters used in the X-ray measurements and in the crystal structure analyses are reported in Table 1. The crystal structure and unit cells for complex 1 in crystal are shown in Figs. 1 and 2 respectively. Table 2 lists the selected bond lengths and angles. From the structure of the complex [dimethy] (L-cysteine)tin(IV)-water (1/1)], we can see that it belongs to a one-dimensional polymeric species, which is arranged regularly with the absorbing water molecules. The dimethyltin(IV) moiety is bonded by the deprotonated amino N atom and deprotonated thiol S atom and a carboxylic O atom from the neighboring L-cysteine molecule. The chelating sulfur and amino nitrogen of the ligand form a fivemembered chelate ring (bite angle N(1)–Sn(1)–S(1) $80.45(8)^{\circ}$ ) with the metal ion. The geometry about Sn is a little distorted trigonal bipyramidal, with two methyl C atoms and S atom occupying the equatorial plane: S(1)-Sn(1)-C(4) 118.0(3)°, S(1)-Sn(1)-C(5)  $113.1(2)^{\circ}$ , C(4)–Sn(1)–C(5) 128.9(4)°, sum of the angles is even 360°. The N<sub>amino</sub> and O<sub>carboxylate</sub> atoms are in the apical positions: O(1)-Sn(1)-S(1) $84.23(9)^{\circ}$ , O(1)-Sn(1)-C(5) 93.1(2)^{\circ}, O(1)-Sn(1)-C(4) 94.8(3)°, N(1)-Sn(1)-C(4) 93.1(3)°, N(1)-

 TABLE 1
 Experimental Data for the X-Ray Diffraction Studies of Complex 1

Empirical formula Formula weight Crystal system Space group a (Å) b (Å) c (Å) V (Å <sup>3</sup> )	$C_5H_{13}NO_3SSn$ 285.91 Orthorhombic $P2_12_12_1$ 9.858 (4) 10.635 (4) 11.329 (4) 1187 7 (8)
7	Λ
$\mu \text{ (mm}^{-1})$ $D_{c} \text{ (g cm}^{-3})$ F(000) Min, min/ $\theta^{\circ}$ Limiting indices	4 2.298 1.599 560 3.83° to 26.36° $-12 \le h \le 5, -13 \le k \le 13,$ $-14 \le l \le 14$
Total reflection Independent reflections Max. and min. transmission Data/restraints/parameters $R_1$ (all data) $wR_2$ (all data) $R_1$ (Final) $wR_2$ (Final) Goodness-of-fit on $F^2$ Residual electron density (e Å <sup>-3</sup> )	$\begin{array}{l} & \begin{array}{c} 6779\\ 2372 \ (R_{\text{int}}=0.0521)\\ 0.7700 \ \text{and} \ 0.6200\\ 2372/3/93\\ 0.0763\\ 0.0617\\ 0.0350\\ 0.0558\\ 0.617\\ 0.553 \ \text{and} \ -0.317 \end{array}$

Sn(1)–C(5) 92.2(2)°, N(1)–Sn(1)–S(1) 80.45(8)°, which are all close to 90°. Thus, the L-cysteine acts as a tridentate ligand that bonds to the tin atom through S, O, and N atoms. The selected distances and angles are in Table 2, e.g., Sn–N<sub>amino</sub> 2.371(3) and Å Sn(1)–O<sub>carboxylate</sub> 2.205(3) Å are little longer than those found in complex [SnMe<sub>2</sub>(L-tryptophyl-L-alaninato)·MeOH] Sn–N<sub>amino</sub> 2.272(5) Å



FIGURE 1 Crystal molecule of complex 1. Probability ellipsoids drawn at 30%.



FIGURE 2 Unit cell of complex 1.

and Sn–O<sub>carboxylate</sub> 2.107(6) Å, respectively [29]; Sn(1)–S(1) 2.409(2) Å, Sn(1)–C(4) 2.112(7) and Å, Sn(1)–C(5) 2.099(7) Å are all comparable to those observed in complex [SnMe<sub>2</sub>Cl(SNH<sub>2</sub>COOEt)] [30]. Furthermore, each monomeric unit forms a weak hydrogen bond with the neighboring molecule through the N(1) atom of the coordinated amino group and the uncoordinated O(2) atom of the carboxylate group. In this way, N(1) is 3.001 Å

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TABLE 2 Selected Bond Lend	oths (A) and Angles (°) of Complex	1

Bond length			
Sn(1)–C(4)	2.112 (7)	Sn(1)–O(1)	2.205 (3)
Sn(1)–C(5)	2.099 (7)	Sn(1)–N(1)	2.371 (3)
Sn(1)-S(1)	2.409 (2)	S(1)-C(3)	1.839 (7)
N(1)-C(2) <sup>#1</sup>	1.464 (5)	O(1)–C(1)	1.2920
C(2)-N(1) <sup>#2</sup>	1.464 (5)	O(2)-C(1)	1.2134
C(2)-C(3) <sup>#2</sup>	1.464 (7)	C(1)–C(2)	1.4853
C(3)-C(2) <sup>#1</sup>	1.464 (7)	H1B-O(2) <sup>#3</sup>	2.121
Bond angle			
O(1)—Šn(1)—N(1)	164.67 (14)	C(4)–Sn(1)–C(5)	128.9 (4)
O(1)–Sn(1)–S(1)	84.23 (9)	N(1)—Sn(1)—C(4)	93.1 (3)
O(1)-Sn(1)-C(4)	94.8 (3)	N(1)-Sn(1)-C(5)	92.2 (2)
O(1)-Sn(1)-C(5)	93.1 (2)	N(1)–Sn(1)–S(1)	80.45 (8)
S(1)–Sn(1)–C(4)	118.0 (3)	C(3)–S(1)–Sn(1)	96.9 (2)
S(1)–Sn(1)–C(5)	113.1 (2)	C(2) <sup>#2</sup> –N(1)–Sn(1)	111.9 (2)
C(1)-O(1)-Sn(1)	118.55 (7)	N(1) <sup>#2</sup> -C(2)-C(3) <sup>#2</sup>	110.2 (3)
C(2) <sup>#1</sup> –C(3)–S(1)	110.5 (4)	N(1)–H1B…O(2)	165.38

Symmetry transformations used to generate equivalent atoms: #1 - x, y + 1/2, -z + 3/2; #2 - x, y - 1/2, -z + 3/2; #3 - x + 1/2, -y, z - 1/2.

from O(2) and hydrogen bond distance is 2.121 Å from O(2) (symmetry operation: -x + 1/2, -y, 1 – z), which is similar to those observed in the complex [SnMe<sub>2</sub>(L-tryptophyl-L-alaninato)·MeOH] (N(1)···O(2<sup>I</sup>) 3.016(7) Å, H(1)···O(2<sup>I</sup>) 2.009(5) Å [29].

# SUPPLEMENTARY MATERIAL

Crystallographic data (excluding structure factors) for the structure analysis of complex **1** have been deposited with the Cambridge Crystallographic Data Center, CCDC no. 207121. Copies of this information 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).

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