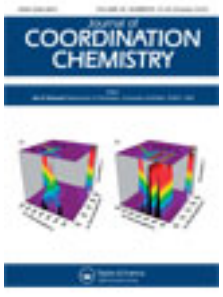

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Synthesis and characterization of complexes of organotin(IV) with 2-thiazoline-2-thione

Abbas Tarassoli [a] & Somayeh Azizi-Talooky [a]
[a] Department of Chemistry , College of Science, Shahid Chamran University , Ahvaz , Iran
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Synthesis and characterization of complexes of organotin(IV) with 2-thiazoline-2-thione

ABBAS TARASSOLI* and SOMAYEH AZIZI-TALOOKY

Department of Chemistry, College of Science, Shahid Chamran University, Ahvaz, Iran

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The reaction of 2-thiazoline-2-thione (TZDSH) with SnR_2Cl_2 (R=Ph **1**, Me **2**, Bu **3**) in dry ethanol in the presence of sodium ethoxide leads to $[SnR_2(C_3H_4NS_2)_2]$ (**1**, **2**, and **3**), respectively. Reaction between TZDSH and $SnPh_2Cl_2$ in dichloromethane and dry ethanol in an inert atmosphere produces $[SnPh_2Cl_2(C_3H_5NS_2)_2]$ (**4**). The yields of the products were over 80%. These new complexes have been characterized by IR, UV-Vis, multinuclear (^1H, ^{13}C, and ^{119}Sn) NMR spectroscopy, and mass spectrometry, as well as elemental analysis.

Keywords: Diphenyltin dichloride; 2-Thiazoline-2-thione; Neutral ligand; Anionic ligand; ^{119}Sn NMR spectroscopy

1. Introduction

Coordination chemistry of organotin compounds has developed rapidly because of their biological and commercial applications [1–3]. Interaction of heterocyclic thioamides, bearing functional groups, N(H)–C(=S)– ↔ –N=C(–SH)–, with metals has been reported [2–6]. These thioligands also have biochemical significance and have been used as medicines [1, 4]. The formation of organotin complexes of these types of ligands may enhance biological activity. Thioamides may coordinate to a metal ion with a variety of coordination modes including: (I) monodentate, (II) chelating, (III) bidentate, (IV) bridging, (V) tridentate, and (VI) polydentate (scheme 1). Thus, a variety of structural motifs can be obtained by use of thioamides [5–7].

One class of such thioamides is 2-thiazoline-2-thione (TZDSH), which may exist in two tautomeric forms **1a** (thione form) and **1b** (thiol form) [8] as shown in figure 1. Spectroscopic studies show that the thione form (**1a**) is preferred. This ligand can coordinate either as a neutral [9] or anionic ligand [10], by loss of a proton. To continue our study of ligands containing competing N and S reactive sites [11–15], interaction of the neutral TZDSH, and anionic TZDS forms of 2-thiazoline-2-thione with organotin(IV) halides have been investigated here. New complexes of organotin(IV), such as $[SnPh_2(C_3H_4NS_2)_2]$ (**1**), $[SnMe_2(C_3H_4NS_2)_2]$ (**2**), $[SnBu_2(C_3H_4NS_2)_2]$ (**3**), and $[SnPh_2Cl_2(C_3H_5NS_2)_2]$ (**4**), are produced. These complexes have been characterized by

*Corresponding author. Email: tarassoli@scu.ac.ir

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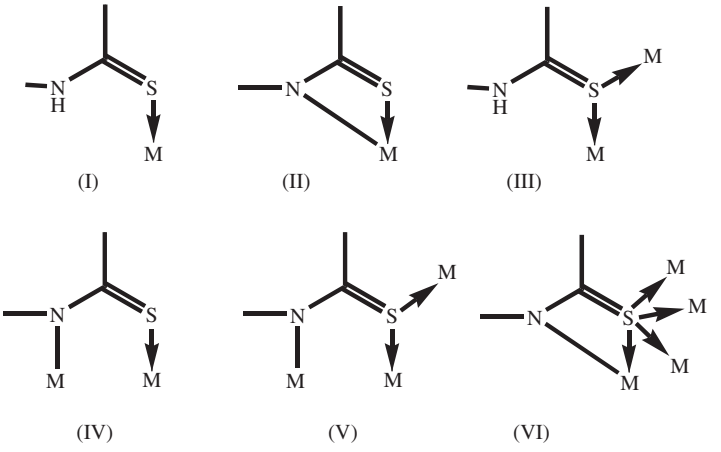


Scheme 1. Possible coordination modes of thioamide to metal ion.

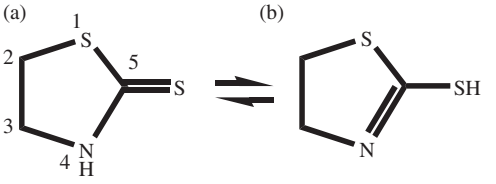


Figure 1. Tautomeric forms of thione and thiol.

elemental analysis, IR, UV-Vis, multinuclear (^{1}H, ^{13}C, and ^{119}Sn) NMR spectroscopy, as well as mass spectrometry.

2. Experimental

2.1. *Materials and instrumentation*

All experiments requiring inert atmosphere were carried out in N_2-flushed glove bags or standard Schlenk apparatus. All solvents, sodium ethoxide, 2-thiazoline-2-thione, dimethyltin dichloride, and dibutyltin dichloride were purchased from Merck, Germany, and diphenyltin dichloride was purchased from Acros Company. IR spectra were recorded using KBr pellets from 4000 to 400 cm^{-1} on a Bomen FT-IR spectrophotometer. NMR spectra were recorded on a Bruker Avance 400 MHz at ambient temperature. ^{1}H NMR (400.13 MHz) and ^{13}C NMR (100.61 MHz) were recorded using $CDCl_3$ and DMSO-d_6 with TMS as external reference. ^{119}Sn NMR spectra of complexes were recorded at 149.21 MHz in $CDCl_3$ and DMSO-d_6 with $SnMe_4$ as external reference. C, H, and N analyses were performed by the microanalytical service of the N.I.O.C Research Institute of Petroleum Industry. The chloride contents of **4** were measured by potentiometric titration (argentometry).

Mass spectra of complexes were recorded on a Shimadzu spectrometer Model GCMS-QP 1000 EX. UV-Vis spectra of complexes were recorded on a double beam GBC model Cintral 101 spectrophotometer in DMSO and $CHCl_3$.

2.2. *Synthesis of [$SnPh_2(C_3H_4NS_2)_2$] (1)*

2-Thiazoline-2-thione (0.300 g, 2.52 mmol) was dissolved in 20 mL of dry ethanol. Then a sodium ethoxide solution in ethanol (0.9 mL, 2.52 mmol) was added and stirred for 30 min at room temperature. Diphenyltin dichloride (0.433 g, 1.26 mmol) in 10 mL of dry ethanol was added dropwise to the above mixture, which was then stirred for 8 h at reflux under nitrogen. After refluxing, the reaction mixture was stirred for another 14 h at room temperature. After filtration, the solvent was removed on a rotary evaporator and an oily product was obtained. Light brown solid was formed by washing the product with diethyl ether and drying under vacuum.

Yield 82%, m.p. 138°C. Anal. Calcd for $C_{18}H_{18}N_2S_4Sn$ (%): C, 42.46; H, 3.53; N, 5.49. Found (%): C, 42.60; H, 3.52; N, 5.26. IR (KBr, cm^{-1}): ν(C–H), 2944, 2898; $\nu(C–H)_{Ar}$, 3076; ν_s(Sn–Ph), 695; ν_{as}(Sn–Ph), 730; ν(C=S), 672; ν(C=N), 1659. ^{1}H NMR (δ, ppm; $CDCl_3$): 3.59 (t, 4H, C^2H), 4.01 (t, 4H, C^3H), 7.74 (d, 4H, $H^{(2,6)}$Ph), $^3J(^{119}Sn\text{-}^1H) = 120.1$ Hz, 7.38–7.55 (m, 6H, $H^{(3,4,5)}$Ph); $^{13}C\{^1H\}$ NMR (δ, ppm; $CDCl_3$): 201.83 (C^5), 33.72 (C^2), 51.42 (C^3), 128–137 ($C^{(1-6)}$ Ph) (scheme 3); ^{119}Sn NMR (δ, ppm; $CDCl_3$): −46.43. Ms: *m*/*z* 510 (M^+), 309 197, 153, 152, 150, 119, 78, 77, 51, UV-Vis (nm): 334.

2.3. *Synthesis of [$SnMe_2(C_3H_4NS_2)_2$] (2)*

The synthesis procedure was the same as section 2.2. A solution of $SnMe_2Cl_2$ (0.368 g, 1.68 mmol) in dry ethanol (10 mL) was added dropwise to a solution of 2-thiazoline-2-thione (0.400 g, 3.36 mmol) in 20 mL dry ethanol which contained sodium ethoxide in ethanol (1.2 mL, 3.36 mmol) to afford an oily product.

Yield 81%, m.p. 198°C. Anal. Calcd for $C_8H_{14}N_2S_4Sn$ (%): C, 24.95; H, 3.63; N, 7.27. Found (%): C, 24.57; H, 3.63; N, 6.99. IR (KBr, cm^{-1}): ν(C–H), 2960, 3004; ν_s(Sn–C), 518; ν_{as}(Sn–C), 568; ν(C=S), 676; ν(C=N), 1654. ^{1}H NMR (δ, ppm; $CDCl_3$): 3.59 (t, 4H, C^2H), 4.01 (t, 4H, C^3H), 1.23 (s, 6H, CH_3), $^2J(^{119}Sn\text{-}^1H) = 82.74$ Hz; $^{13}C\{^1H\}$ NMR (δ, ppm; $CDCl_3$): 201.96 (C^5), 33.73 (C^2), 51.31 (C^3), 1.02 (C (Me)); ^{119}Sn NMR (δ, ppm; $CDCl_3$): −61.61. Ms: *m*/*z* 386 (M^+), 355, 268, 152, 150, 135, 119, 76, 15, UV-Vis (nm): 335.

2.4. *Synthesis of [$SnBu_2(C_3H_4NS_2)_2$] (3)*

The synthesis procedure was the same as Section 2.2. A solution of $SnBu_2Cl_2$ (0.509 g, 1.68 mmol) in dry ethanol (10 mL) was added dropwise to a solution of 2-thiazoline-2-thione (0.400 g, 3.36 mmol) in 20 mL dry ethanol which contained sodium ethoxide in ethanol (1.2 mL, 3.36 mmol) to give an oily product.

Yield 80%, m.p. 152°C. Anal. Calcd for $C_{14}H_{26}N_2S_4Sn$ (%): C, 35.84; H, 5.54; N, 5.96. Found (%): C, 36.11; H, 5.53; N, 5.78. IR (KBr, cm^{-1}): ν(C–H), 2918, 3002; $\nu(C–H)_{Bu}$, 2858, 2954; ν_s(Sn–C), 522; ν_{as}(Sn–C), 573; ν(C=S), 673; ν(C=N), 1629.

^{1}H NMR (δ, ppm; $CDCl_3$): 3.59 (t, 4H, C^2H), 4.01 (t, 4H, C^3H), 0.97-1.8 (18H, Bu), J(HH) = 7.12 Hz; ^{13}C $\{^{1}H\}$NMR (δ, ppm; $CDCl_3$): 201.90 (C^5), 33.97 (C^2), 51.71 (C^3), 13.6 and 26.3–28.1 (C^{1-4}(Bu)); ^{119}Sn NMR (δ, ppm; $CDCl_3$): +27.16. Ms: m/z 470 (M^+), 440, 413, 412, 207, 177, 152, 150, 119, 76, 57, 29, 27, 26, 15, 14, UV-Vis (nm): 338.

2.5. *Synthesis of $[SnPh_2Cl_2(C_3H_5NS_2)_2]$ (4)*

2-Thiazoline-2-thione (0.200 g, 1.68 mmol) was dissolved in 10 mL of dichloromethane. Then diphenyltin dichloride (0.288 g, 0.84 mmol) solution in dichloromethane (12 mL) and dry ethanol (4 mL) was added dropwise to the reaction mixture, and the contents stirred for 9 h at reflux under nitrogen. After refluxing, the mixture was stirred for another 16 h at room temperature. The mixture was filtered and the solvent was removed on a rotary evaporator, and an oily product was obtained. Light yellow solid was formed by washing the product twice with dichloromethane and diethyl ether, and then dried under vacuum.

Yield 84%, m.p. 120°C. Anal. Calcd for $C_{18}H_{20}N_2Cl_2S_4Sn$ (%): C, 37.14; H, 3.43; N, 4.81; Cl, 12.18. Found (%): C, 37.32; H, 3.41; N, 4.93; Cl, 12.41. IR (KBr, cm^{-1}): ν(N–H), 3211 (br); ν(C–H), 2876, 2930; ν(C–H)$_{Ar}$, 3037, 3140; ν_s(Sn–Ph), 695; ν_{as}(Sn–Ph), 737; ν(C=S), 679. ^{1}H NMR (δ, ppm; $SO(CD_3)_2$): 10.24 (s, 2H, NH), 3.51 (t, 4H, C^2H), 3.88 (t, 4H, C^3H), 7.89 (d, 4H, $H^{(2,6)}$Ph), $^{3}J(^{119}Sn\text{-}^{1}H) = 162.3$ Hz, 7.26–7.44 (m, 6H, $H^{(3,4,5)}$Ph); ^{13}C $\{^{1}H\}$NMR (δ, ppm; $SO(CD_3)_2$): 199.38 (C^5), 33.38 (C^2), 51.92 (C^3), 127–136 ($C^{(1-6)}$ Ph) (scheme 3); ^{119}Sn NMR (δ, ppm; $SO(CD_3)_2$): −163.97. Ms: m/z: 582 (M^+), 344, 309, 232, 230, 197, 155, 153, 152, 120, 119, 78, 77, 51, UV-Vis (nm): 339.

3. Results and discussion

Reactions of diorganotin dichloride with 2-thiazoline-2-thione were carried out in a 1 : 2 stoichiometric ratio for all the complexes. The synthesis procedures are shown in scheme 2 and proposed structures are illustrated in scheme 3.

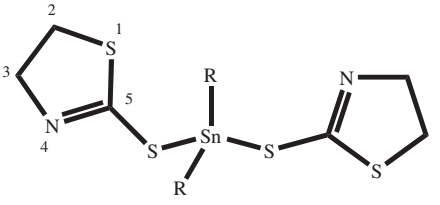
The new complexes (**1**–**4**) were fully characterized by UV-Vis, FT-IR, mass, and (^{1}H, ^{13}C and ^{119}Sn) NMR spectroscopy, as well as elemental analyses. All attempts to grow single crystals of these compounds suitable for X-ray crystallography were unsuccessful. Spectroscopic data are discussed below.

$$2C_3H_5NS_2 + SnR_2Cl_2 \xrightarrow[(EtOH,\ dry)\ 75^{o}C]{EtONa} [SnR_2(C_3H_4NS_2)_2] + 2NaCl + 2EtOH$$

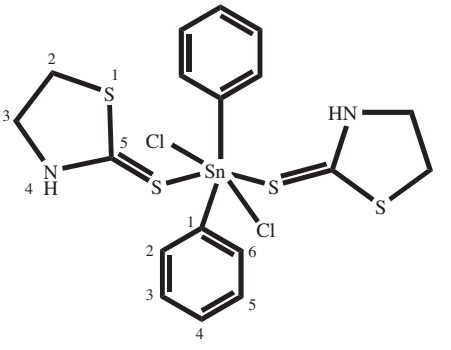
(R = Ph 1, Me 2, Bu 3)

$$2C_3H_5NS_2 + SnPh_2Cl_2 \xrightarrow[CH_2Cl_2]{40^{o}C} [SnPh_2Cl_2(C_3H_5NS_2)_2]$$

Scheme 2. The synthesis methods for complexes **1**–**4**.



Complexes **1**, **2** and **3**



Complex **4**

Scheme 3. The proposed structures for complexes **1**–**4**.

Table 1. Selected FT-IR bands (cm^{-1}) for the ligand (TZDSH) and its complexes.

Compound	N–H	Thioamide (I)	Thioamide (II)	Thioamide (III)	C=S	C=N	Sn–C
TZDSH	3139	1515	1297	1051	698	–	–
1	–	1521	1302	1042	672	1659	695, 730
2	–	1520	1303	1042	676	1654	518, 568
3	–	1520	1301	1046	673	1629	522, 573
4	3211	1523	1305	1044	679	–	695, 737

3.1. *IR spectra*

IR spectra for **1**–**3** show characteristic absorptions at 1659, 1654, and 1629 cm^{-1}, related to the C=N bond (Supplementary material). In these complexes the absorption of N–H is eliminated, indicating that the ligand is coordinated to tin as an anionic ligand. In **4** the broad absorption at 3211 cm^{-1} is seen for the N–H group, shifted to higher wavenumber compared to the free ligand, corresponding to the neutral ligand (Supplementary material). In addition, the ν(C=S) band of the free ligand at 698 cm^{-1} appears at 672, 676, and 673 cm^{-1} for **1**, **2**, and **3**, respectively, and at 679 cm^{-1} for **4**, shifted to lower wavenumbers in the complexes. These variations are consistent with a decrease in the C=S bond order upon coordination. This also indicates that the ligand is coordinated to metal through exocyclic sulfur and not *via* nitrogen. The most significant FT-IR bands of the uncoordinated TZDH and **1**–**4** are summarized in table 1.

3.2. *^{1}H NMR spectra*

^{1}H NMR spectra of **1**, **2**, and **3** in $CDCl_3$ show the C^2H and C^3H protons at δ 3.59 and 4.01 ppm, respectively, which are slightly shifted downfield compared to the free ligand. In these complexes the N–H signal is eliminated, indicating the ligand is coordinated to tin anionic. In **1**, the proton signals of $H^{(2,6)}$ from phenyl group are at δ 7.74 ppm with

$^3J(^{119}Sn–^1H)$ equal to 120.1 Hz [16, 17] and other signals related to protons of phenyl $H^{(3,4,5)}$ are at δ 7.38–7.55 ppm (scheme 3). In **2**, the proton signals of methyl appear at δ 1.23 ppm with $^2J(^{119}Sn–^1H) = 82.7$ Hz [18] and substitution of the coupling constant in the Lockhart–Manders equation [19] gives a value of 134.40° for Me–Sn–Me angle. Therefore, in solution $–SnMe_2$ moiety is not linear. In **3**, the proton signals of butyl appear at δ 0.97–1.80 ppm with $J(HH) = 7.1$ Hz. For this complex, $^2J(^{119}Sn–^1H)$ value cannot be extracted from the spectrum because of the complexity of the methylene multiplets. The ^1H NMR spectrum of **4** in DMSO-d_6 shows the N–H signal of the ligand at δ 10.09 ppm, slightly shifted to lower energy region at δ 10.24 ppm in this complex, indicating ligand is deprotonated and coordinated as a neutral ligand. The C^2H and C^3H protons appear at δ 3.51 and 3.88 ppm, respectively, and the proton signals of $H^{(2,6)}$ from phenyl are at δ 7.89 ppm with $^3J(^{119}Sn–^1H) = 162.3$ Hz [16, 17]; other signals related to protons of phenyl $H^{(3,4,5)}$ appear at δ 7.26–7.44 ppm (scheme 3).

3.3. *^{13}C NMR spectra*

The ^{13}C NMR spectra of **1**, **2**, and **3** in $CDCl_3$ show C^2 at δ 33.72, 33.73, and 33.97 ppm, respectively (scheme 3), with a small downfield shift in comparison to the ligand signal at 33.40 ppm, while C^3 shows a small upfield shift at δ 51.42, 51.31, and 51.7 ppm, respectively, instead of 51.91 ppm for TZDSH [20]. After coordination of thione sulfur of 2-thiazoline-2-thione to tin, C^5 in ^{13}C NMR spectra of ligand (δ 199.39 ppm) moves downfield at δ 201.83, 201.96, and 201.90 ppm, respectively. The characteristic aromatic signals are at δ 128–137 ppm in **1**, and methyl carbon atoms are seen at δ 1.02 ppm and butyl carbon atoms of **3** show signals at 13.6 and 26.3–28.1 ppm. ^{13}C NMR spectra of **4** in DMSO-d_6 exhibited the C^5 signal at 199.38 ppm, C^2 at δ 33.38 ppm and the C^3 at δ 51.92 ppm (scheme 3) almost without change relative to the free ligand, indicating coordination occurs *via* thione form (figure 1). Characteristic phenyl signals appear at δ 127–136 ppm in this complex.

3.4. *^{119}Sn NMR spectra*

^{119}Sn NMR spectra of **1**, **2**, and **3** in $CDCl_3$ show signals at δ −46.43, −61.61, and +27.16 ppm, respectively. These resonances appear at lower magnetic field than those of the organotin halides [$SnPh_2Cl_2$, $\delta - 27$ ppm, $SnMe_2Cl_2$, $\delta + 137$ ppm, and $SnBu_2Cl_2$, $\delta + 123$ ppm], consistent with a four-coordinate tin [21–25]. The ^{119}Sn NMR spectra of **4** in DMSO-d_6 exhibited a signal at $\delta - 163.97$ ppm. As reported [24, 26, 27] ^{119}Sn chemical shift is strongly dependent on the coordination number of tin and an increase in coordination number produces a large up-field shift, thus we conclude that Sn is six-coordinate in **4** [28].

3.5. *UV-Vis spectra*

The $n \rightarrow \pi^*$ absorption of C=N for 2-thiazoline-2-thione appears at λ_{max} 332 nm, slightly shifted to λ_{max}/nm (log ε): 334 (3.38), 335 (3.05), 338 (3.21), and 339 (3.84) for **1**–**4**, respectively.

3.6. *Mass spectra*

Mass spectral data for **1**–**4** are consistent with the structures proposed on the basis of other spectroscopic data. Mass spectrum of **1** shows expected peaks at m/z 510 (M^+), 309, 197, 152, 151, 119, 78, 77, and 51. The peak at 119 is attributable to the 2-thiazoline-2-thione (TZDS). The peak at 77 is related to the S=C=S group resulting from the decomposition of the ligand. The peaks at 77 and 51 corresponding to Ph and (Ph–C_2H_2) ions resulted from the phenyl ring and Ph-fragmentation. The peaks at m/z 150, 151, 152, and 197 can be attributed to the ^{118}Sn–S, ^{119}Sn–S, ^{120}Sn–S, and ^{120}Sn–Ph, respectively. These Sn-containing ions are due to the principal isotopes ^{118}Sn, ^{119}Sn, and ^{120}Sn with naturally relative abundance of 24.01, 8.58, and 32.97%, respectively [27].

Mass spectrum of **2** exhibits peaks at m/z 386 (M^+), 355, 268, 152, 151, 150, 119, 76, and 15. The peak at 119 is due to the 2-thiazoline-2-thione (TZDS). The peak at 76 is related to S=C=S resulting from decomposition of the ligand. The peak at 15 is due to the methyl group. The peaks at 152, 151, 150, and 268 are attributable to the Sn-containing fragments ^{120}Sn-S, ^{119}Sn-S, ^{120}Sn$(CH_3)_2$, and ^{120}Sn$(CH_3)_2$ (TZDH), respectively.

Mass spectrum of **3** shows peaks at m/z 470 (M^+), 440, 412, 352, 209, 177, 152, 150, 119, 76, 57, 29, 27, 26, 15, and 14. The peak at 119 is attributable to the 2-thiazoline-2-thione (TZDS). The peak at 76 is related to the S=C=S moiety resulting from decomposition of the ligand. The peak at 57 is due to the butyl group. Peaks at 152, 150, 209, and 280 are related to the Sn-containing ions ^{120}Sn–S, ^{119}Sn–S, Bu-^{120}Sn–S, and ^{120}Sn$(CH_2)_3$ (TZDS), respectively. Finally the peaks at 14, 15, and 29 correspond to –CH_2, methyl, and ethyl groups, respectively.

Mass spectrum of **4** exhibits expected ions peaks at m/z: 582 (M^+), 344, 309, 232, 230, 197, 155, 152, 120, 119, 78, 77, and 51. The peaks at 120 and 119 correspond to 2-thiazoline-2-thione (TZDSH) and (TZDS), respectively. The peak at 77 is related to the S=C=S skeleton resulting from decomposition of the ligand. The peak at 78 indicates the presence of the phenyl group, along with a series of less intense peaks at m/z 153, 155, 197, and 230, which are attributable to the Sn-containing ions ^{120}Sn–S, ^{119}Sn–Cl, ^{120}Sn–Ph, and ^{120}Sn–S–Ph moieties, respectively. Finally the mass spectra of **1**–**4** showed relatively low intensity molecular ion (M^+) for **1**–**4**, as is generally the case for tin complexes [29].

4. Conclusion

Although attention has already been paid to coordination chemistry of heterocyclic thiol/thione donors and a number of organotin complexes have been reported, examples of these types are rare and limited [1–3, 22]. 2-Thiazoline-2-thione is an excellent candidate for reaction with organotin halides and can adopt variable coordination numbers. This ligand can also give potential access to new products with unusual structures. In this research, the synthesis and characterization of four new organotin(IV) complexes based on 2-thiazoline-2-thione have been described. The spectroscopic studies show that this N, S-ligand with considerable flexibility is coordinated monodentate binding through the exocyclic sulfur in these complexes. In **1**, **2**, and **3** the ligand is coordinated anionic to afford a four-coordinate tin(IV) structure,

while in **4** the ligand is coordinated as a neutral ligand and the ^{119}Sn NMR data are consistent with six-coordinate tin(IV) in solution.

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