# Chloro-diorganotin(IV) complexes of 4-methyl-1-piperidine carbodithioic acid: Synthesis, X-ray crystal structures, spectral properties and antimicrobial studies

Saira Shahzadi [a], Saqib Ali [a,*], Moazzam H. Bhatti [b,*], Mohammmad Fettouhi [c], Muhammad Athar [d]

[a] Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan
[b] Department of Chemistry, Allama Iqbal Open University, H/8, Islamabad, Pakistan
[c] Department of Chemistry, King Fahad University of Petroleum and Minerals, 31261 Dhahran, Saudi-Arabia
[d] State Key Laboratory of Inorganic Synthesis and Preparative Chemistry, College of Chemistry, Jilin University, Changchun 130012, PR China

## Abstract

Chloro-diorganotin(IV) complexes of 4-methyl-1-piperidine carbodithioic acid (4-MePCDTA) have been synthesized by the reaction with diorganotin dichloride in 1:1 molar ratio in anhydrous toluene. These newly synthesized complexes have been characterized by elemental, IR, multinuclear NMR ($^1$H and $^{13}$C) and mass spectrometric studies. The crystal structures of complex **1** [Me$_2$SnCl(4-MePCDT)] and **3** [Ph$_2$SnCl(4-MePCDT)] have been determined by X-ray single crystal analysis, which show trigonal bipyramid geometry. These complexes were tested for their antimicrobial activity against six different plant and human pathogens. The screening results show that the complexes exhibit higher antibacterial and antifungal activity than the free ligand.
© 2005 Elsevier B.V. All rights reserved.

*Keywords:* Chloro-organotin complexes; Dithiocarbamates; NMR; Mass; Antimicrobial study; X-ray structures

## 1. Introduction

Complexing agents with dithio functional group have been widely used in industry as rodent repellents, vulcanization additives in rubber manufacturing, additives in lubricants and in agriculture as fungicides on almond trees, stone fruits and vegetables [1]. Interest in complexes of both main group and transition metals with sulfur donor ligands arises in past because of their varied structures and biological activity [2–4].

Sulfur containing molecules are currently under study as chemoprotectants in platinum-based chemotherapy. In particular, thiocarbonyl and thiol donors have shown promising properties for chemical use in modulating cisplatin nephrotoxicity [5,6].

In recent years much attention have been paid to the synthesis, characterization and biological activities of various organotin(IV) derivatives with sulfur ligands like thione or dithiocarbamate [7–9]. In addition to biological activities the organotin derivatives are also subjected to thermal and CVD studies [10,11]. In continuation to our interest in synthetic and structural aspects of organotin(IV) complexes of dithiocarbamate [12,13], we report here the synthesis, spectral characterization, antimicrobial activity and crystal structures of chloro-diorganotin(IV) derivatives of 4-methyl-1-piperidine carbodithioic acid (Fig. 1).

* Corresponding authors. Tel.: +92512875027; fax: +92512873869 (S. Ali), Tel./fax: +92519250081 (M.H. Bhatti).
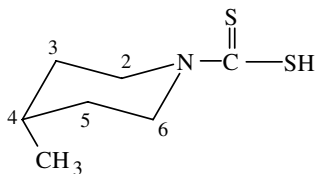*E-mail addresses:* drsa54@yahoo.com (S. Ali), moazzamhussain_b@yahoo.com (M.H. Bhatti).

Fig. 1. The structure and numbering scheme of 4-methyl-1-piperidine carbodithioic acid.

## 2. Experimental

### 2.1. Materials and instrumentation

Analytical grade dimethyltin dichloride, dibutyltin dichloride, diphenyltin dichloride and 4-methyl piperidine were procured from Aldrich Chemical Company. $CS_2$ was purchased from Riedel-de-Haën. All solvents were dried before used by the literature methods [14a]. 4-Methyl-1-piperidine carbodithioic acid is prepared by the literature method [14b,14c].

Carbon, hydrogen, nitrogen and sulfur analyses were performed with a Perkin–Elmer 2400 Series II instrument. IR spectra in the range 4000–250 cm$^{-1}$ were obtained on a Bio-Rad FTIR spectrophotometer with samples investigated as KBr discs. The $^1$H and $^{13}$C NMR spectra were recorded on a Bruker Advance 500 spectrometer operating at 500 MHz. Mass spectra were recorded on MAT-112S mass spectrometer.

### 2.2. Syntheses

#### 2.2.1. Synthesis of Me$_2$SnCl(4-MePCDT) (1)

The preparation of complex **1** was carried out at room temperature. Me$_2$SnCl$_2$ (1 mmol), 4-MePCDTA (1 mmol) and Et$_3$N (1 mmol) were added to 100 cm$^3$ of dry toluene and refluxed for 4 h. The precipitated salt was removed by filtration and the filtrate was evaporated under reduced pressure. The product was recrystallized from chloroform–hexane to give colorless crystals. Yield: 96%, m.p. 122–123 °C. Anal. Calc. for C$_9$H$_{18}$NS$_2$ClSn: C, 30.16; H, 5.02; N, 3.91; S, 17.87. Found: C, 30.20; H, 5.08; N, 3.89; S, 17.89%. $^1$H NMR (DMSO-$d_6$, ppm), $^nJ(^1$H, $^1$H), $^nJ[^{119}$Sn, $^1$H], 2.50 (m, 4H), 1.67 (m, 4H), 1.77 (m, 1H), 0.93 (d, 3H, CH$_3$, (7.5)), {0.48 (s, 6H), SnCH$_3$, [79.6]}. $^{13}$C NMR (DMSO-$d_6$, ppm), 193.9 (CSS), 51.74 (C-2), 33.3 (C-3), 30.3 (C-4), 22.7 (CH$_3$), 14.6 (SnCH$_3$). IR (KBr, cm$^{-1}$), 328 $v$(Sn–Cl), 428 $v$(Sn–S), 555 $v$(Sn–C), 966 $v$(C=S), 1075 $v$(C–S).

#### 2.2.2. Synthesis of Bu$_2$SnCl(4-MePCDT) (2)

The procedure is the same as that of complex **1**, and complex **2** was recrystallized from chloroform–diethyl ether to give crystalline product. Yield: 80%, m.p. 92–93 °C. Anal. Calc. for C$_{15}$H$_{30}$NS$_2$ClSn: C, 40.72; H, 6.78; N, 3.16; S, 14.47. Found: C, 40.76; H, 6.80; N, 3.20; S, 14.42%. $^1$H NMR (DMSO-$d_6$, ppm), $^nJ(^1$H, $^1$H), 2.50 (m, 4H), 1.69 (m, 4H), 1.78 (m, 1H), 0.92 (d, 3H, CH$_3$, (7.4)), {1.77–1.30 (m, 12H), 0.89 (t, 6H), SnCH$_2$CH$_2$CH$_2$CH$_3$}. $^{13}$C NMR (DMSO-$d_6$, ppm, $^nJ[^{119/117}$Sn, $^{13}$C]), 192.75 (CSS), 51.94 (C-2), 33.42 (C-3), 30.22 (C-4), 21.3 (CH$_3$), {22.0 [577/553], 28.84 [34], 27.57 [87], 13.62, SnCH$_2$CH$_2$CH$_2$CH$_3$}. IR (KBr, cm$^{-1}$), 320 $v$(Sn–Cl), 441 $v$(Sn–S), 532 $v$(Sn–C), 968 $v$(C=S), 1086 $v$(C–S).

#### 2.2.3. Synthesis of Ph$_2$SnCl(4-MePCDT) (3)

Crystalline complex **3** was made in the same way of complex **1**. It was recrystallized in chloroform–hexane. Yield: 90%, m.p. 192–193 °C. Anal. Calc. for C$_{19}$H$_{22}$NS$_2$ClSn: C, 47.30; H, 4.56; N, 2.90; S, 13.27. Found: C, 47.35; H, 4.60; N, 2.88; S, 13.25%. $^1$H NMR (DMSO-$d_6$, ppm), $^nJ(^1$H, $^1$H), 2.50 (m, 4H), 1.60 (m, 4H), 1.78 (m, 1H), 0.92 (d, 3H, CH$_3$, (7.5)), {7.96–7.53 (m, 10H, SnC$_6$H$_5$)}. $^{13}$C NMR (DMSO-$d_6$, ppm,), 190.21 (CSS), 53.17 (C-2), 33.22 (C-3), 30.24 (C-4), 21.29 (CH$_3$), {142.45, 135.15, 128.53, 129.21, SnC$_6$H$_5$}. IR (KBr, cm$^{-1}$), 325 $v$(Sn–Cl), 450 $v$(Sn–S), 552 $v$(Sn–C), 960 $v$(C=S), 1065 $v$(C–S).

### 2.3. X-ray crystallography

All X-ray crystallographic data (Table 1) were collected on a Bruker AXS Smart Apex diffractometer. Correction for semi-empirical from equivalents was applied, 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 data were collected with graphitemonochromated Mo K$\alpha$ radiation ($\lambda = 0.71073$ Å) at 295 K.

Table 1
Crystal data and structure refinement parameters for complex (**1**) and (**3**)

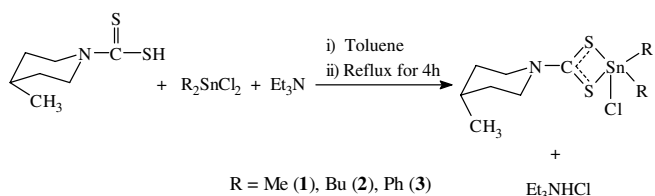|  | Complex (**1**) | Complex (**3**) |
|---|---|---|
| Empirical formula | C$_9$H$_{18}$NS$_2$ClSn | C$_{19}$H$_{22}$NS$_2$ClSn |
| Formula weight | 358 | 482 |
| Crystal system | Triclinic | Monoclinic |
| Space group | $P\bar{1}$ | $P21/c$ |
| *Unit cell dimension* |  |  |
| $a$ (Å) | 7.110 (8) | 13.581 (13) |
| $b$ (Å) | 10.621 (12) | 9.637 (9) |
| $c$ (Å) | 11.2185 (12) | 17.171 (17) |
| $\alpha$ (°) | 113.377 (2) | 90.00 |
| $\beta$ (°) | 97.562 (2) | 112.828 (10) |
| $\gamma$ (°) | 107.290 (2) | 90.00 |
| $V$ (Å$^3$) | 711.86 (14) | 2071.60 (3) |
| $Z$ | 2 | 4 |
| $D_{calc}$ (g cm$^{-3}$) | 1.673 | 1.547 |
| Crystal size (mm) | $0.31 \times 0.24 \times 0.08$ | $0.33 \times 0.19 \times 0.17$ |
| $F(000)$ | 356 | 968 |
| Total reflections | 3296 | 4977 |
| Independent reflections | 2941 | 4231 |
| All indices (all data) | $R_1 = 0.0319$, $wR_2 = 0.669$ | $R_1 = 0.0337$, $wR_2 = 0.0678$ |
| Final $R$ indices [$I > 2\sigma(I)$] | $R_1 = 0.0272$, $wR_2 = 0.0647$ | $R_1 = 0.0273$, $wR_2 = 0.0652$ |
| Goodness-of-fit | 1.056 | 1.053 |
| $\theta$ Range for data collection (°) | 2.06–28.32 | 1.63–28.26 |
| Data/restrains/parameters | 3296/0/130 | 4977/0/218 |

## 3. Results and discussion

### 3.1. Synthesis

The ligand was obtained from the reaction of 4-methylpiperidine with carbon disulphide in dry methanol as shown in the following equation:
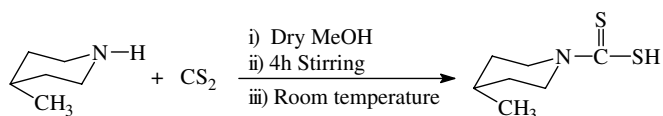
The organotin derivatives **1–3** were obtained from the reaction of $R_2SnCl_2$ with 4-MePCDTA in 1:1 molar ratio in the presence of triethyl amine. The general chemical reaction is given as:

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

### 3.2. IR spectra

The comparison is made between the spectra of complexes and precursor. The N–H bond stretching in $R_2NH$ (secondary amine) in the range of 3200 $cm^{-1}$ disappeared when it reacted with carbon disulfide, indicating the formation of the ligand. The disappearance of –SH bond stretch-ing of free ligand in the region 2754 $cm^{-1}$ and appearance of Sn–S stretching in the range of 450–428 $cm^{-1}$ and Sn–C in the range of 555–525 $cm^{-1}$ indicate the formation of complexes [15]. The characteristic bands observed in the spectra of **1–3** complexes in the region 328–320 $cm^{-1}$ are assigned to Sn–Cl stretching mode of vibration [15].

### 3.3. NMR spectroscopy

The $^1H$ NMR spectral data of 4-methyl-1-piperidine carbodithioic acid shows single resonance at 1.27 ppm, which is absent in the spectra of the complexes, indicating the replacement of the dithiocarboxylic acid proton by a diorganotin moiety. The $^nJ[^{119}Sn, ^1H]$ of dimethyltin deriv-ative has a value of 79.6 Hz falling in the range for five coordinated tin atoms [16a].

In $^{13}C$ NMR, the assignment of the $^{13}C$ signal for –CSS group is straightforward and is assigned in the range 193.9–190.21 ppm for chloro-diorganotin(IV) complexes indicat-ing the coordination of sulfur to the tin atom. R groups attached to tin moiety give signals in the expected range. The order of the magnitude of the coupling constant in **2**, $^1J[^{119}Sn, ^{13}C]$, is same as reported for the analogous five coordinated derivatives [16b,16c].

### 3.4. Mass spectrometry

The conventional EI mass spectral data for the ligand and compounds **1–3** are recorded and different fragmenta-tion patterns have been proposed and are listed in Schemes 1–4 along with $m/z$ and % intensity. In the mass spectrum of 4-methyl-1-piperidine carbodithioic acid, molecular ion
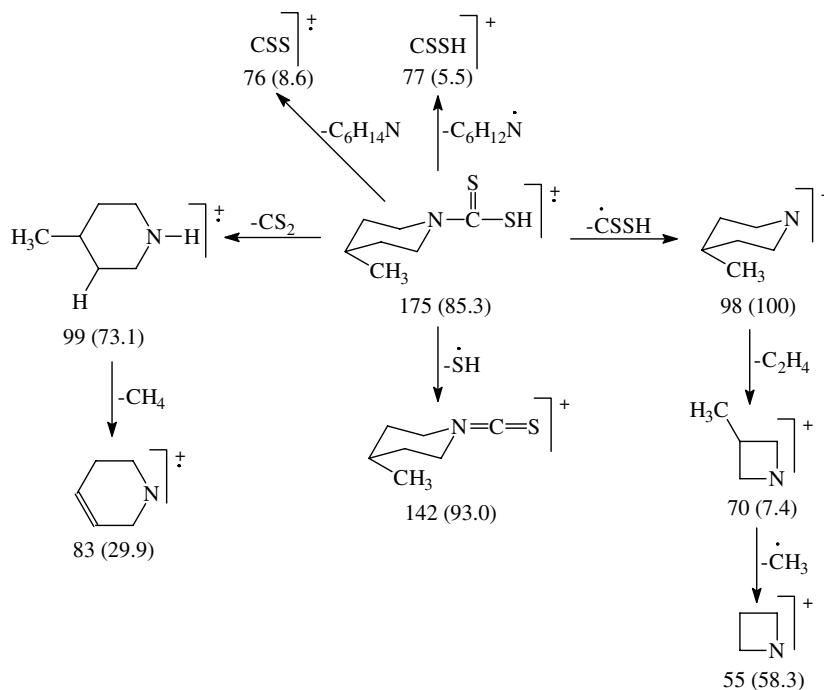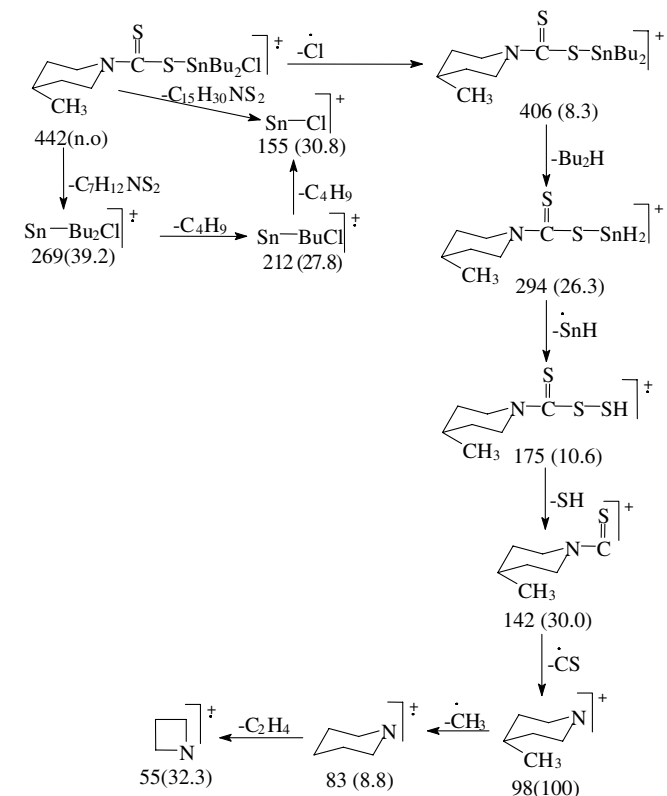


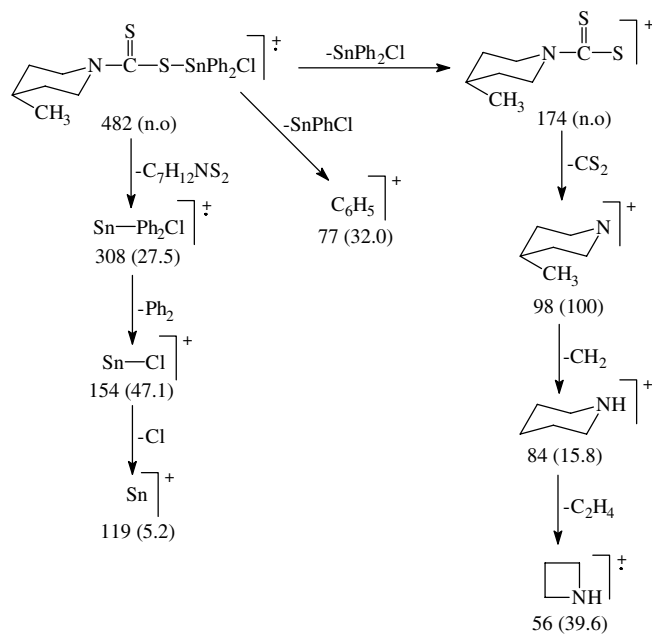Scheme 1. Fragmentation pattern for ligand (HL).

peak is observed at $m/z$ (%) 175 (85.3). The base peak is observed at $m/z$ (%) 98 (100) due to $[C_6H_{12}N]^+$ fragment. The other fragments at $m/z$ (%) 142 (93.0), 76 (8.6), 83 (29.9), 99 (73.1) are observed for $[C_7H_{12}NS]^+$, $[CSS]^+$,
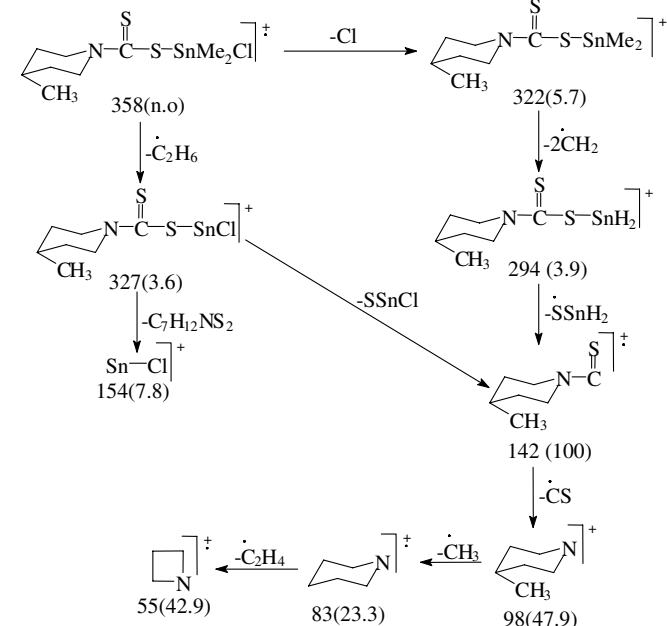
Scheme 2. Fragmentation pattern for compound (**1**).

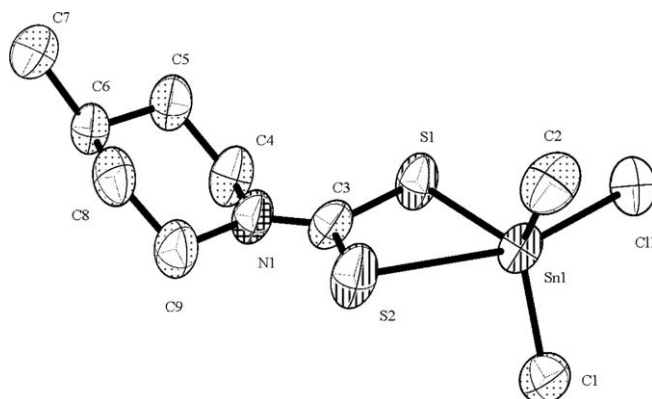Scheme 3. Fragmentation pattern for compound (**2**).

Scheme 4. Fragmentation pattern for compound (**3**).

$[C_5H_9N]^+$ and $[C_6H_{13}N]^+$, respectively. The fragmentation patter is given in Scheme 1.

In the mass spectral data for **1**–**3** most fragment ions occur in group of peaks as a result of tin isotopes. For simplicity the mass spectral fragmentation data reported here is related to the principal isotope $^{120}$Sn [17]. The molecular ion peak is not observed in all complexes. In general, the complex **1** has base peak (100%) at $m/z$ 142 due to fragment $[C_7H_{12}NS]^+$ while complexes **2** and **3** have base peak due to the fragment $[C_6H_{12}N]^+$ at $m/z$ (%) 98 (100). The other possible fragments with $m/z$ (%) are reported in Schemes 2–4.

### 3.5. Crystal structures

Figs. 2 and 3 show the molecular structures and atomic numbering scheme of complexes **1** and **3**, respectively. The intermolecular bond distances and angles are given in
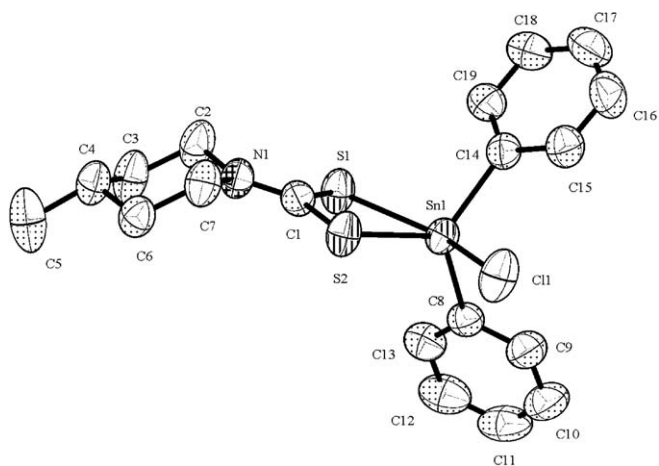
Fig. 2. ORTEP drawing of the X-ray structure of complex (**1**).

Fig. 3. ORTEP drawing of the X-ray structure of complex (**3**).

ideal bond angle of 360° [18a]. In both complexes the one of Sn–S bond length (in **1** Sn–S(1) 2.466(7) Å, in **2** Sn–S(2) 2.461(6) Å) is shorter than the other (in **1** Sn–S(2) 2.739(9) Å, in **2** Sn–S(1) 2.657(6) Å) suggesting the unsymmetrical coordination of the ligand. Further the shorter Sn–S bond lengths are very close to the sum of the covalent radii of tin and sulfur and the longer Sn–S distances are significantly less than the sum of van der Waals radii (4.0 Å) [18b]. The Sn–C distances (in **1** Sn–C(1) 2.109(3) Å; Sn–C(2) 2.112(3) Å, in **2** Sn–C(8) 2.129(2) Å; Sn–C(14) 2.137(2) Å) are similar to those found in earlier reports [12,13]. The Sn–Cl bond lengths (in **1** Sn–Cl(1)

Table 2. From both figures and tables it can be seen that the coordination geometry about tin atom is penta-coordinated distorted trigonal bipyramidal with two R groups attach to tin and S(1)/(S2) occupying equatorial positions while Cl(1) and S(2)/S(1) occupy the apical position. In this way, the ligand behaves as a bidentate species and chelates the tin atom by means of sulfur atoms similar to Ph$_2$SnCl(PCDT) and Me$_2$SnCl(PCDT) where PCDT = piperidine-1-carbodithioate [12,13]. Due to being apart of chelate the angle S(1)–Sn–S(2) is not 90° but only 68.25(2)°, so the S(2) cannot occupy exactly the corresponding *trans* apical position of Cl(1) and the angle between the apical groups is 154.19(3) for **1** [12,13]. For complex **2** the S(2)–Sn–S(1) is only 69.98(18)° and the angle Cl(1)–Sn–S(1) is 153.55(2)° for the same reason. These values are again comparable with the analogous Ph$_2$SnCl(PCDT) and Me$_2$SnCl(PCDT) where PCDT = piperidine-1-carbodithioate. The sum of the equatorial angles formed in complexes **1** and **3** is 358.37(10)° and 359.59(10)°, respectively, showing some distortion from

Table 2
Selected bond lengths (Å) and bond angles (°) for complex (**1**) and (**3**)

| Complex (**1**) | | Complex (**3**) | |
|---|---|---|---|
| *Bond lengths (Å)* | | | |
| Sn(1)–C(1) | 2.109(3) | Sn(1)–C(8) | 2.129(2) |
| Sn(1)–C(2) | 2.112(3) | Sn(1)–C(14) | 2.137(2) |
| Sn(1)–S(1) | 2.466(7) | Sn(1)–S(2) | 2.461(6) |
| Sn(1)–Cl(1) | 2.485(8) | Sn(1)–Cl(1) | 2.467(6) |
| Sn(1)–S(2) | 2.739(9) | Sn(1)–S(1) | 2.657(6) |
| S(1)–C(3) | 1.743(3) | S(1)–C(1) | 1.720(2) |
| S(2)–C(3) | 1.697(3) | S(2)–C(1) | 1.745(2) |
| N(1)–C(3) | 1.317(3) | N(1)–C(1) | 1.302(3) |
| N(1)–C(9) | 1.469(4) | N(1)–C(2) | 1.474(3) |
| N(1)–C(4) | 1.471(3) | N(1)–C(7) | 1.481(3) |
| *Bond angles(°)* | | | |
| C(1)–Sn(1)–C(2) | 126.88(14) | C(8)–Sn(1)–C(14) | 114.63(8) |
| C(1)–Sn(1)–S(1) | 112.96(9) | C(8)–Sn(1)–S(2) | 122.21(6) |
| C(2)–Sn(1)–S(1) | 118.53(10) | C(14)–Sn(1)–S(2) | 122.75(6) |
| C(1)–Sn(1)–S(2) | 94.76(9) | C(8)–Sn(1)–C(11) | 96.76(6) |
| C(2)–Sn(1)–S(2) | 92.50(11) | C(14)–Sn(1)–C(11) | 96.60(6) |
| S(1)–Sn(1)–S(2) | 68.25(2) | S(2)–Sn(1)–S(1) | 69.98(18) |
| Cl(1)–Sn(1)–S(2) | 154.19(3) | Cl(1)–Sn(1)–S(1) | 153.55(2) |
| C(3)–S(1)–Sn(1) | 91.23(9) | C(1)–S(1)–Sn(1) | 83.94(7) |
| N(1)–C(3)–S(2) | 123.6(2) | N(1)–C(1)–S(1) | 123.48(17) |
| N(1)–C(3)–S(1) | 119.6(2) | N(1)–C(1)–S(2) | 120.41(17) |
| N(1)–C(4)–C(5) | 110.2(2) | N(1)–C(2)–C(3) | 110.7(2) |

Table 3
Antimicrobial activity[a] of the free ligand and their chloro-diorganotin(IV) complexes

| Comp. | Fungi | | | | | |
|---|---|---|---|---|---|---|
| | *T. longifusus* | *C. albicans* | *A. flavus* | *M. canis* | *F. solani* | *C. glaberata* |
| HL | 10 | 0 | 0 | 10 | 28 | 38 |
| (**1**) | 30 | 0 | 0 | 0 | 40 | 70 |
| (**2**) | 30 | 60 | 0 | 60 | 40 | 20 |
| (**3**) | 30 | 60 | 10 | 60 | 0 | 90 |
| R | 70 | 110.8 | 20 | 98.4 | 73.2 | 110.8 |
| | Bacteria | | | | | |
| | *E. coli* | *B. subtilis* | *S. flexenari* | *S. aureus* | *P. aeruginosa* | *S. typhi* |
| HL | 12 | 15 | 15 | 20 | 20 | 20 |
| (**1**) | 18 | 20 | 20 | 22 | 22 | 20 |
| (**2**) | 26 | 25 | 25 | 24 | 20 | 20 |
| (**3**) | 18 | 18 | 20 | 20 | 20 | 18 |
| R | 35 | 38 | 32 | 38 | 29 | 28 |

[a] The test done using the tube diffusion method (antifungal) and agar well diffusion method, well diameter, 6 mm; R, Standard drug; Miconazole and Amphotericin.B (antifungal agent); Imipenum (antibacterial agent).

2.485(8) Å, in **2** Sn–Cl(1) 2.467(6) Å) lie in the range of the normal covalent radii, 2.37–2.60 Å [12,13,18].

### 3.6. Microbial assay

The free ligand 4-methyl-1-piperidine carbodithioic acid and their chloro-diorganotin(IV) complexes were tested against the bacterial strains *Escherichia coli*, *Bacillus subtilis*, *Shigella flexenari*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Salmonella typhi* by agar well diffusion method [19] and the antifungal activity against *Trichophyton longifusus*, *Candida albicans*, *Aspergillus flavus*, *Microsporum canis*, *Fusarium solani* and *Candida glaberata*. The results are listed in Table 3.

The antibacterial studies exhibited that the complexes **1**–**3** have high activity towards all tested bacteria than the free ligand. The antifungal studies by tube diffusion method exhibited that the complexes **1**–**3** have high activity towards all the tested fungi than the free ligand. It may be concluded that metal coordination increases the activity as compared to free ligand.

### 4. Conclusions

Chloro-diorganotin(IV) complexes of 4-methyl-1-piperidine carbodithioic acid have been synthesized and characterized. Detailed studies of reported complexes indicate that their structures are trigonal bipyramidal geometry. These complexes also checked for their antimicrobial activity against different bacteria and fungi by agar well diffusion and tube diffusion methods, respectively. The screening results show that reported compounds **1**–**3** exhibit high antimicrobial activity as compared to free ligand (4-methyl-1-piperidine carbodithioic acid).

### 5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 277901 and 277900 for complexes **1** and **3**, respectively. Copies of these information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CBZ 1EZ, UK (fax: +44 1223 336 033; email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

### References

[1] E.I. Stiefel, K. Matsumoto, in: Transition Metal Sulfur Chemistry ASC Symposium Series, vol. 653, American Chemical Society, Washington, DC, 1995, p. 2.
[2] P. Yang, M. Guo, Coord. Chem. Rev. 189 (1999) 185.
[3] R.K. Chadha, R. Kumar, D.G. Tuck, Can. J. Chem. 65 (1987) 1336.
[4] T.A. Al-Allaf, I.A. Mustafa, S.E. Al-Mukhtar, Transition Met. Chem. 18 (1993) 1.
[5] R.T. Dorr, in: H.M. Pinedo, J.H. Schornagel (Eds.), Platinum and Other Metal Coordination Compounds in Cancer Chemotherapy, vol. 2, Plenum Press, New York, 1996, p. 131.
[6] N. Ferrell, Catalysis by Metal ComplexesTransition Metal Complexes as Drugs and Chemotherapeutic Agents, vol. 11, Kluwer Academic Publishers, Dordrecht, 1989, p. 44.
[7] L. Pellerito, L. Nagy, Coord. Chem. Rev. 224 (2002) 111.
[8] A.K. Saxena, F. Huber, Coord. Chem. Rev. 95 (1989) 109.
[9] M.N. Xanthopoulou, S.K. Hadjikakou, N. Hadjiliadis, M. Schürmann, K. Jurkschat, A. Michaelides, S. Skoulika, T. Bakas, J. Binolis, S. Karkabounas, K. Charalabopoulos, J. Inorg. Biochem. 96 (2003) 425.
[10] (a) D.C. Menezes, G.M. de Lima, A.O. Porto, C.L. Donnici, J.D. Ardisson, A.C. Doriguetto, J. Ellena, Polyhedron 23 (2004) 2103;
(b) R. Sharma, N.K. Kaushik, J. Therm. Anal. Calor. 78 (2004) 953.
[11] A.T. Kana, T.G. Hibbert, M.F. Mahon, K.C. Molloy, I.P. Parkin, L.S. Price, Polyhedron 20 (2001) 2989.
[12] S. Ali, S.U. Ahmad, S. Rehman, S. Shahzadi, M. Parvez, M. Mazhar, Appl. Organomet. Chem. 19 (2005) 200.
[13] S. Ali, S.U. Ahmad, S. Shahzadi, S. Rehman, M. Parvez, M. Mazhar, Appl. Organomet. Chem. 19 (2005) 201.
[14] (a) W.F.F. Armarego, C.L.L. Chai, Purification of Laboratory Chemicals, fifth ed., Butterworth, Oxford, 2003;
(b) A. Benedetti, A.C. Fabretti, C. Preti, J. Chem. Crystallogr. 18 (1988) 685;
(c) A.C. Fabretti, C. Preti, J. Chem. Crystallogr. 19 (1989) 957.
[15] H.P.S. Chauhan, N.M. Shaik, J. Inorg. Biochem. 99 (2005) 538.
[16] (a) T.P. Lockhart, W.F. Manders, Inorg. Chem. 25 (1986) 892;
(b) B. Wrackmeyer, Annu. Rep. NMR Spectrosc. 16 (1985) 73;
(c) B. Wrackmeyer, Annu. Rep. NMR Spectrosc. 38 (1999) 203.
[17] M.H. Bhatti, S. Ali, M. Mazhar, M. Danish, M.A. Choudhary, Turk. J. Chem. 23 (1999) 329.
[18] (a) A. Tarassoli, T. Sedaghat, M.L. Helm, A. Norman, J. Coord. Chem. 56 (2003) 1179;
(b) C. Ma, J. Zhang, R. Zhang, Can. J. Chem. 81 (2003) 1070.
[19] A. Rehman, M.I. Choudhary, W.J. Thomsen, Bioassay Techniques for Drug Development, Harwood Academic Publishers, The Netherlands, 2001, p. 14.