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Synthesis and Spectral Studies of Diorganotin Heterocyclic Dithiocarbamate Complexes and Crystal Structures of $(2\text{-}F\text{-Bz})_2Sn(Cl)S_2CN(CH_2CH_2)_2NEt$ and $(3\text{-}Cl\text{-Bz})_2Sn[S_2CN(CH_2CH_2)_2NEt]_2 \cdot 0.5HN(CH_2CH_2)_2NH$

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ABSTRACT: *Some new diorganotin(IV) complexes of heterocyclic dithiocarbamate having general formula* $R_2Sn(Cl)S_2CNR'_2$ *and* $R_2Sn(S_2CNR'_2)_2$ *[R = 2-F-Bz, 3-Cl-Bz;* $NR'_2 = N(CH_2CH_2)_2NMe$, $N(CH_2CH_2)_2NEt$, *and* $N(CH_2CH_2)_2NBz$*] have been prepared, respectively. Elemental analyses, IR, and NMR spectral data characterized all compounds. The crystal structures of* $(2\text{-F-Bz})_2Sn(Cl)S_2CN(CH_2CH_2)_2NEt$ **2** *and* $(3\text{-Cl-Bz})_2Sn[S_2CN(CH_2CH_2)_2NEt]_2{\cdot}0.5\ HN(CH_2CH_2)_2NH$ **5** *were determined by single crystal X-ray diffractometer. In the crystal of complex* **2**, *the tin atom is rendered five-coordination in a trigonal bipyramidal configuration by coordinating with S atoms of dithiocarbamate groups. For complex* **5**, *the central Sn atom exists in a skew-trapezoidal planar geometry defined by two asymmetrically coordinated dithiocarbamate ligands and two 3-chlorobenzyl groups.* Heteroatom Chem 16:271–277, 2005; DOI 10.1002/hc.20096

INTRODUCTION

Metal *N,N*-dialkyldithiocarbamates have been synthesized by the reaction of metal halides on sodium *N,N*-dialkyldithiocarbamate in both aqueous and nonaqueous solutions [1–3]. Recently, interest in dithiocarbamate complexes of diorganotin(IV) species arises because of their varied structures and biological activities [4–6]. On the basis of crystallographic studies of dithiocarbamate complexes of diorganotin(IV), a variety of coordination environments around the central tin atom, ranging from tetrahedral to distorted octahedral, with ligands having anisobidentate or monodentate character, have been reported [7–14]. As an extension of our studies of organotin(IV) complexes with dithiocarbamate ligands, we have synthesized and characterized two types of diorganotin(IV) complexes of heterocyclic dithiocarbamate having general formula $R_2Sn(Cl)S_2CNR'_2$ and $R_2Sn(S_2CNR'_2)_2$ [R = 2-*F*-Bz, 3-*Cl*-Bz; $NR'_2 = N(CH_2CH_2)_2NMe$, $N(CH_2CH_2)_2NEt$, and $N(CH_2CH_2)_2NBz$] and determined the crystal structures of $(2\text{-}F\text{-Bz})_2Sn(Cl)S_2CN(CH_2CH_2)_2NEt$ **2** and $(3\text{-}Cl\text{-Bz})_2Sn[S_2CN(CH_2CH_2)_2NEt]_2 \cdot 0.5\ (CH_2CH_2NH)_2$ **5**. The results of this study are reported in the present paper.

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EXPERIMENTAL

Materials and Instrumentation

Anhydrous sodium dithiocarbamates were prepared according to the method described in the literature [15]. All solvents used in this work are of reagent grade and were dried by the standard methods prior to use. IR spectra were recorded on a Nicolet-460 spectrophotometer, using KBr as disks. NMR spectra were obtained with a Mercury Plus-400 NMR spectrometer, and the chemical shifts are given in ppm relative to Me_4Si in $CDCl_3$. Elemental analyses were performed on a PE-2400-II elemental analyzer.

Syntheses of Complexes, $R_2Sn(Cl)S_2CNR'_2$ **1–3**

Anhydrous sodium dithiocarbamate (1.2 mmol) was added to a 30 mL absolute benzene solution of bis(2-fluorobenzyl)tin dichloride (1.0 mmol). Then the mixture was heated under reflux for 5 h. The precipitated sodium chloride was removed by filtration, and the filtrate was concentrated to obtain white precipitate under reduced pressure. The products were recrystallized from dichloromethane-hexane to give crystals.

*$(2\text{-}F\text{-}Bz)_2Sn(Cl)S_2CN(CH_2CH_2)_2NMe$ (**1**).* Yield 62%. mp 104–105°C. ^{1}H-NMR ($CDCl_3$): δ 6.83–7.22 (8 H, m, Ar-H); 2.85 (3H, s, NCH_3); 3.67 (8H, t, NCH_2CH_2N); 2.78 (4H, t, $ArCH_2Sn$) ppm. ^{13}C-NMR ($CDCl_3$): δ 131.48, 130.86, 129.52, 127.84, 126.59, 125.74, 29.34 (2-*F*-$C_6H_4CH_2$); 198.63 (CS_2); 54.53, 51.64, 45.60 (NCH_2, NCH_3) ppm.^{119}Sn-NMR δ –216.27 ppm. IR (KBr, cm^{-1}): 1494 (s, C—N); 1112, 996 (s, CS_2); 452 (m, Sn—S). Anal. Calcd for $C_{20}H_{23}ClF_2N_2S_2Sn$: C, 43.94; H, 4.21; N, 4.92; S,11.64. Found: C, 43.86; H, 4.23; N, 5.11; S, 11.71%.

*$(2\text{-}F\text{-}Bz)_2Sn(Cl)S_2CN(CH_2CH_2)_2NEt$ (**2**).* Yield 65%. mp 88–90°C. ^{1}H-NMR ($CDCl_3$): δ 6.96–7.26(8 H, m, Ar-H); 3.86 (8H, t, NCH_2CH_2N); 3.22 (4H, t, $ArCH_2Sn$); 2.52 (2H, m, NCH_2); 1.15 (3H, t, CH_3) ppm. ^{13}C-NMR ($CDCl_3$): δ 131.45, 130.96, 130.71, 127.09, 127.00, 125.48, 29.28 (2-*F*-$C_6H_4CH_2$); 197.60 (CS_2); 51.85, 51.67, 51.48, 43.61 (NCH_2, NCH_2CH_3) ppm. ^{119}Sn-NMR δ –221.35 ppm. IR (KBr, cm^{-1}): 1490 (s, C—N); 1125, 1017 (s, CS_2); 448 (m, Sn—S). Anal. Calcd for $C_{21}H_{25}ClF_2N_2S_2Sn$: C, 44.84; H, 4.51; N, 4.92; S,11.04. Found: C, 44.90; H, 4.49; N, 4.99; S, 11.42%.

*$(2\text{-}F\text{-}Bz)_2Sn(Cl)S_2CN(CH_2CH_2)_2NBz$ (**3**).* Yield 54%. mp 84–86°C ^{1}H-NMR ($CDCl_3$): δ 6.95–7.38 (13 H, m, Ar-H, Ph-H); 3.85 (2H, s, NCH_2Ph); 3.60 (8H, t, NCH_2CH_2N); 2.51 (4H, t, $ArCH_2Sn$) ppm. ^{13}C-NMR ($CDCl_3$): δ 130.94, 129.17, 128.53, 127.06, 125.33, 123.91, 29.65 (2-*F*-$C_6H_4CH_2$); 198.81 (CS_2); 54.53, 51.64, 45.60, 118.52, 128.23, 129.04 (NCH_2, $NCH_2C_6H_5$) ppm. ^{119}Sn-NMR δ –227.46 ppm. IR (KBr, cm^{-1}): 1491 (s, C—N); 1125, 1022 (s, CS_2); 450 (m, Sn—S). Anal. Calcd for $C_{26}H_{27}ClF_2N_2S_2Sn$: C, 50.14; H, 4.31; N, 4.52; S,10.14. Found: C, 50.06; H, 4.36; N, 4.49; S, 10.28%.

Syntheses of Complexes, $R_2Sn(S_2CNR'_2)_2$, **4–6**

Sodium dithiocarbamate (2.5 mmol) was dissolved in 15 mL dichloromethane and added dropwise to a solution of di(3-*Cl*-benzyl)tin dichloride (1.0 mmol) in the same solvent. The mixture was kept stirring for 14 h at 30°C. It was then filtered to remove the white solid. The solvent from the filtrate was removed in vacuo, and a white precipitate was obtained. The product was dissolved in hot acetone, and a few drops of methanol were added to give colorless crystals.

*$(3\text{-}Cl\text{-}Bz)_2Sn[S_2CN(CH_2CH_2)_2NMe]_2$ (**4**).* Yield 71%. mp 134–135°C. ^{1}H-NMR ($CDCl_3$): δ 6.97–7.26 (8H, m, Ar-H); 2.91 (6H, s, NCH_3); 3.83 (16H, t, NCH_2CH_2N); 2.71 (4H, t, Hz, $ArCH_2Sn$) ppm. ^{13}C-NMR ($CDCl_3$): δ 133.28, 131.56, 130.87, 129.32, 128.95, 127.18, 29.07 (3-*Cl*-$C_6H_4CH_2$); 195.84 (CS_2); 54.26, 51.83, 45.52 (NCH_2, NCH_3) ppm. ^{119}Sn-NMR δ –408.61 ppm. IR (KBr, cm^{-1}): 1491 (s, C—N); 1124, 1038 (s, CS_2); 462 (m, Sn—S). Anal. Calcd for $C_{26}H_{34}Cl_2N_4S_4Sn$: C, 43.44; H, 4.78; N, 7.90; S,17.54. Found: C, 43.34; H, 4.76; N, 7.78; S, 17.80%.

*$(3\text{-}Cl\text{-}Bz)_2Sn[S_2CN(CH_2CH_2)_2NEt]_2{\cdot}0.5\ HN(CH_2CH_2)_2NH$ (**5**).* Yield 78%. mp 166–168°C. ^{1}H-NMR ($CDCl_3$): δ 7.04–7.34 (8H, m, Ar-H); 3.86, 4.01 (24H, t, NCH_2CH_2N); 3.12 (4H, t, $ArCH_2Sn$); 2.42 (4H, m, NCH_2); 1.09 (6H, t, CH_3); 1.03 (2H, m, NH) ppm. ^{13}C-NMR ($CDCl_3$): δ 134.31, 132.49, 130.43, 128.96, 126.58, 125.74, 28.96 (3-*Cl*-$C_6H_4CH_2$); 196.93 (CS_2); 52.49, 51.78, 51.25, 51.19, 45.54 (NCH_2, NCH_2CH_3) ppm. ^{119}Sn-NMR δ –412.52 ppm. IR (KBr, cm^{-1}): 1480 (s, C—N); 1015, 992 (s, CS_2); 452 (m, Sn—S). Anal. Calcd for $C_{30}H_{43}Cl_2N_5S_4Sn$: C, 45.44; H, 5.52; N, 8.91; S,16.11. Found: C, 45.52; H, 5.48; N, 8.85; S, 16.20%.

*$(3\text{-}Cl\text{-}Bz)_2Sn[S_2CN(CH_2CH_2)_2NBz]_2$ (**6**).* Yield 57%. mp 143–145°C. ^{1}H-NMR ($CDCl_3$): δ 6.75–7.38 (18 H, m, Ar-H, Ph-H); 3.87 (4H, s, NCH_2Ph); 3.75 (16H, t, NCH_2CH_2N); 2.91 (4H, t, $ArCH_2Sn$) ppm. ^{13}C-NMR ($CDCl_3$): δ 134.12, 133.48, 132.52, 132.04, 130.60, 129.74, 30.34 (3-*Cl*-$C_6H_4CH_2$); 197.60 (CS_2);

53.94, 52.56, 43.87, 116.89, 125.49, 130.16 (NCH_2, $NCH_2C_6H_5$) ppm. ^{119}Sn-NMR δ –404.73 ppm. IR (KBr, cm^{-1}): 1486 (s, C—N); 1138, 1007 (s, CS_2); 458 (m, Sn—S). Anal. Calcd for $C_{38}H_{42}Cl_2N_4S_4Sn$: C, 52.34; H, 4.78; N, 6.40; S,14.64. Found: C, 52.30; H, 4.85; N, 6.42; S, 14.70%.

Crystallographic Measurements

All X-ray crystallographic data were collected on a Bruker smart-1000 CCD diffractometer with graphite monochromated Mo Kα (0.71073 Å) radiation. The structures were solved by direct method and difference Fourier map using SHELXL-97 program, and refined by full-matrix least-squares on F^2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located at calculated positions and refined isotropically. The crystallographic data are summarized in Table 1.

RESULTS AND DISCUSSION

IR Spectroscopic Properties

Of particular interest in the IR spectra are the C—N, C—S, and Sn—S stretching frequencies. The former two can be used to differentiate between mono and bidentate modes of binding of dithiocarbamate ligand [16]. For bidentate binding only two individual single bands are observed for C—N and C—S absorptions which occur near 1500 cm^{-1} and 1000 cm^{-1}, respectively, whereas in the case of monodentate binding doublets are observed in the above regions. In the IR spectra of complexes **1–6**, the strong band in the region ranging from 1475 cm^{-1} to 1490 cm^{-1} may be assigned to ν(C—N), which is similar to that reported for analogous tin complexes [7–9]. The presence of only one intense absorption band at about 1000 cm^{-1} that can be attributed to ν(C—S) indicates the bidentate nature of the dithiocarbamate moiety in the complexes [17]. On the other hand, a new strong absorption band at about 450 cm^{-1} for all complexes, which is absent in the spectra of the free groups, is assigned to the Sn—S stretching mode of vibration. These information suggest that the sulfur atoms of dithiocarbamate group in six complexes are linked to tin atoms in an anisobidentate fashion. The analysis is in agreement with X-ray single crystal diffraction results.

The ^{1}H-NMR spectra of complexes **1–6** show that the chemical shifts of the protons on the benzyl group exhibit two signals about 6.75–7.38 ppm as multiplet and 2.51–3.22 ppm as a triplet which is caused by the tin (^{119}Sn) hydrogen coupling. For complexes **1** and **3**, the NCH_2CH_2N protons give rise to triplet signals at 3.60–3.86 ppm and the NCH_3 protons lead to a singlet signal at 2.32, 2.83 ppm.

TABLE 1 Crystallographic Data of Complexes **2** and **5**

Complex	**2**	**5**
Molecular formula	$C_{21}H_{25}ClF_2N_2S_2Sn$	$C_{30}H_{43}Cl_2N_5S_4Sn$
Formula weight	561.69	791.52
Temperature (K)	273(2)	293(2)
Crystal system	Monoclinic	Triclinic
Space group	*C*2/*c*	*Pi*
a (Å)	18.354(4)	9.433(7)
b (Å)	9.0683(17)	14.292(11)
c (Å)	30.695(6)	15.100(11)
α (°)	90	66.075(9)
β (°)	107.181(5)	82.933(10)
γ (°)	90	71.233(10)
V (Å^3)	4880.9(17)	1762(2)
Z	8	2
*D*cal (g/cm^3)	1.529	1.492
μ (mm^{-1})	1.352	1.142
F (000)	2256	812
Theta range (°)	$1.39 < \theta < 25.03$	$1.63 < \theta < 25.03$
Reflections collected	11895	9078
Independent reflections	4160 ($R_{int} = 0.0337$)	6125 ($R_{int} = 0.0241$)
Data, restraints, and parameters	4160, 12, 265	6125, 5, 372
Goodness-of-fit on F^2	1.066	1.010
Final *R* indices R_1, wR_2	0.0575, 0.1324	0.0446, 0.1273
R indices (all data) R_1, wR_2	0.0775, 0.1446	0.0667, 0.1460
Residual electron density (e·Å^{-3})	1.037, −0.618	0.801, −0.729

TABLE 2 Selected Bond Distances (Å) and angles (°) of Complex **2**

Sn(1)–C(8)	2.146(8)	Sn(1)–S(2)	2.659(2)
Sn(1)–C(15)	2.142(9)	S(1)–C(1)	1.736(6)
Sn(1)–S(1)	2.4567(17)	S(2)–C(1)	1.701(6)
Sn(1)–Cl(1)	2.4592(19)		
C(8)–Sn(1)–C(15)	116.7(4)	S(1)–Sn(1)–Cl(1)	87.25(6)
C(8)–Sn(1)–S(1)	133.4(4)	C(8)–Sn(1)–S(2)	94.3(3)
C(15)–Sn(1)–S(1)	108.9(3)	C(15)–Sn(1)–S(2)	98.0(3)
C(8)–Sn(1)–Cl(1)	93.9(2)	S(1)–Sn(1)–S(2)	69.71(6)
C(15)–Sn(1)–Cl(1)	99.7(3)	Cl(1)–Sn(1)–S(2)	154.53(7)

In the ^{13}C NMR spectra of compounds **1–6**, chemical shifts are similar to those of the corresponding dithiocarbamate ligand. The ^{13}C NMR data also show a slightly downfield shift in the position of C_{C-N} signals and an upfield shift in the position of C_{CSS} signal. This result is consistent with previous studies [5,6,18].

^{119}Sn NMR spectroscopic data are very useful for determining the coordination number of tin, its molecular geometry, and stereochemistry. In general tin-119 chemical shifts move to lower frequency with increasing coordination number. According to the information reported literature [21], the ^{119}Sn chemical shift values for compounds **1–3** indicate that a signal appear at −227.46 to −221.35 ppm, suggesting that the tin atoms exhibit five-coordinate in the solution. And the ^{119}Sn NMR data for compounds **4–6** are found at −412.52 to −404.73 ppm indicating six-coordination environment around the central tin atoms in three complexes.

Molecular Structure of Complex **2**

The selected bond distances and angles of complexes **2** and **5** are given in Tables 2 and 3, respectively, and their molecular structures are shown in Figs. 1 and 2. Projections of the unit cell are depicted in Figs. 3 and 4.

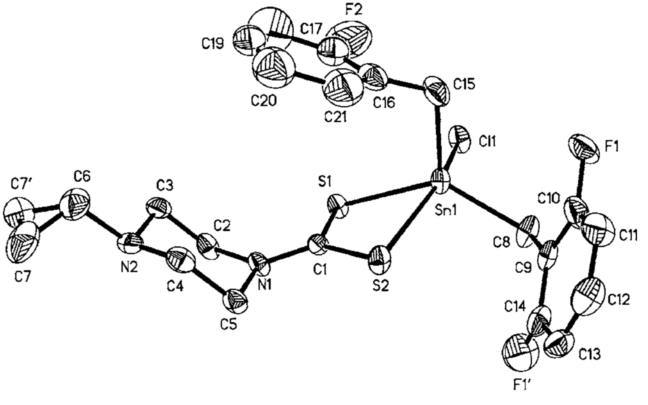

FIGURE 1 Molecular structure of complex **2**.

As can be seen from Fig. 1, the crystal structure of complex **2** is made up of discrete molecules, with no significant intermolecular interactions. The tin atom is rendered five-coordination in a trigonal bipyramidal configuration [Sn(1)–S(1) 2.4567(17) Å, Sn(1)–S(2) 2.659(2) Å, Sn(1)–Cl(1) 2.4592(19) Å, Sn(1)–C(8) 2.146(8) Å, Sn(1)–C(15) 2.142(9) Å]. The structure is similar to those observed for the complexes $Ph_2SnCl(S_2CNEt_2)$ [7], $^tBu_2SnCl(S_2CNEt_2)$ [7], and (4-*F*-Bz)$_2$Sn(Cl)S$_2$CN(CH$_2$CH$_2$)$_2$O [18].

The geometry of complex **2** is loosely based on a trigonal bipyramid, with atoms C(8), S(1), and C(15) occupying equatorial positions. The sum of the equatorial angles (359.1°) at the tin atom by the two coordinated carbon atoms and one sulfur atom [C(15)–Sn(1)–S(1) 108.9(3)°, C(8)–Sn(1)–S(1) 133.4(4)°, C(8)–Sn(1)–C(15) 116.7(4)°] deviates only by 0.9° from the 360°, so the atoms C(8), S(1), C(15),

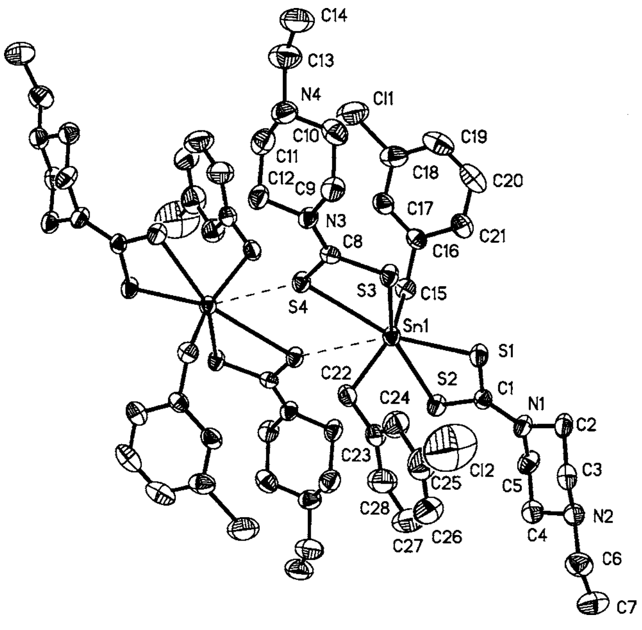

FIGURE 2 Molecular structure of complex **5**.

TABLE 3 Selected Bond Distances (Å) and Angles (°) of Complex **5**

Sn(1)–C(15)	2.170(5)	Sn(1)–S(1)	2.520(2)
Sn(1)–C(22)	2.171(5)	Sn(1)–S(4)	2.893(2)
Sn(1)–S(3)	2.555(2)	Sn(1)–S(2)	2.840(2)
S(1)–C(1)	1.727(5)	S(2)–C(1)	1.707(5)
S(3)–C(8)	1.726(5)	S(4)–C(8)	1.697(5)
Sn(1) ··· S(4)#	3.638(3)		
C(15)–Sn(1)–C(22)	147.7(2)	S(3)–Sn(1)–S(4)	65.18(5)
C(15)–Sn(1)–S(3)	102.74(14)	S(1)–Sn(1)–S(4)	150.64(5)
C(22)–Sn(1)–S(3)	99.18(16)	C(15)–Sn(1)–S(2)	84.69(15)
C(15)–Sn(1)–S(1)	100.71(16)	C(22)–Sn(1)–S(2)	86.87(16)
C(22)–Sn(1)–S(1)	104.46(15)	S(3)–Sn(1)–S(2)	152.24(5)
S(3)–Sn(1)–S(1)	85.48(5)	S(1)–Sn(1)–S(2)	66.80(5)
C(15)–Sn(1)–S(4)	85.42(15)	S(4)–Sn(1)–S(2)	142.55(4)
C(22)–Sn(1)–S(4)	82.54(15)		

and Sn(1) are approximately in the same plane. The Cl atom occupies approximately one apical position of the trigonal bipyramid. Conversely, due to the constraint of the chelate [the angle of [S(1)—Sn(1)—S(2)] is not 90° only 69.71(6)°], the S(2) atom cannot exactly occupy one of the two axial positions of the trigonal bipyramid, the angle Cl(1)—Sn(1)—S(2) being 154.53(7)°.

The S—C bond lengths [S(1)—C(1) 1.736(6) Å, S(2)—C(1) 1.701(6) Å] appear to be characteristic of the dithiocarbamate group, and the two distances are all intermediate between the values expected "single" and "double" bond [13]. Therefore, in the crystal of compound **2**, the tin atom exists in a five-coordination trigonal bipyramidal geometry by coordinating with S atoms of dithiocarbamate group.

Molecular Structure of Complex 5

From Fig. 2, it can be seen that the Sn atom exists in a skew-trapezoidal planar geometry in which the basal plane is defined by the four S atoms derived from two chelating dithiocarbamate ligands. The two remaining positions are occupied by two 3-chlorobenzyl groups, which lie over the weaker

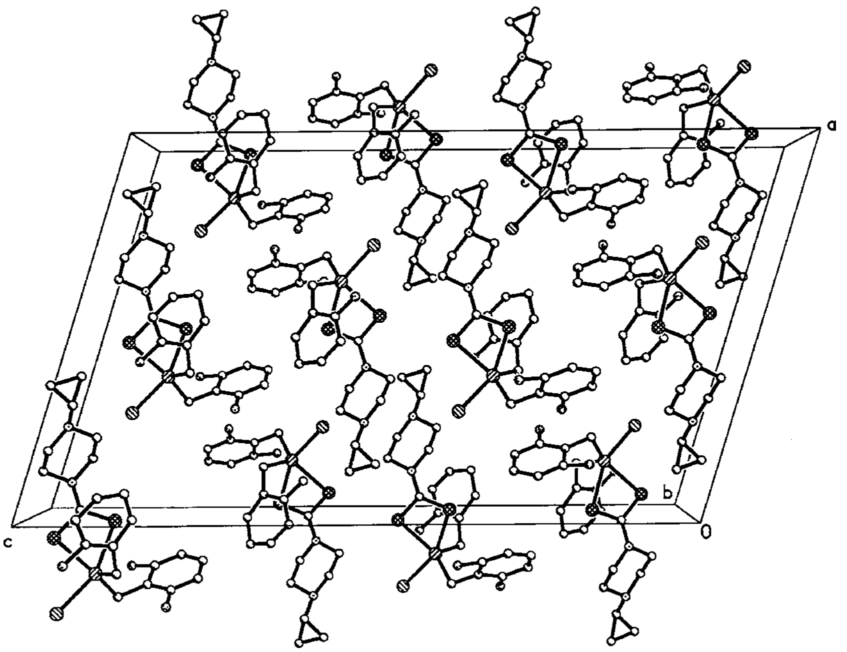

FIGURE 3 Projection of the unit cell of complex **2**.

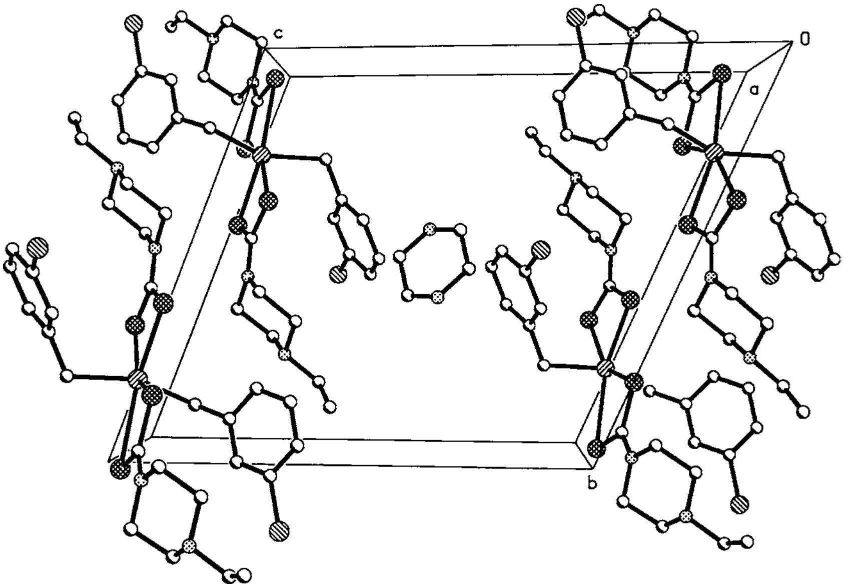

FIGURE 4 Projection of the unit cell of complex **5**.

Sn—S bonds and define a C(15)—Sn(1)—C(22) angle of 147.7(2)°. The degree of asymmetry in the mode of coordination of each dithiocarbamate ligand, while comparable, is not equivalent. The first ligand forms Sn(1)—S(1) and Sn(1)—S(2) bond distances of 2.520(2) and 2.840(2) Å, respectively, and the other forms Sn(1)—S(3) and Sn(1)—S(4) bond at 2.555(2) and 2.893(2) Å, respectively. These differences yield values of 0.32 and 0.338 Å, respectively for Δ(Sn—S), and are reflected in the associated C—S bond distances. As expected, the C—S bond distances [S(1)—C(1) 1.727(5) Å, S(3)—C(8) 1.726(5) Å] for the S atoms bound strongly to the Sn center are longer than the C—S bonds [S(2)—C(1) 1.707(5) Å, S(4)—C(8) 1.697(5) Å] for the S atoms which form the weaker Sn—S bonds to the Sn atom. The geometry is similar to that observed for $Me_2Sn(S_2CNEt_2)_2$ [19].

In addition, a distinguishing feature, namely the presence of close intermolecular nonbonded Sn(1) ··· S(4)# 3.638(3) Å contact (from a centrosymmetrically related molecule), is noted in the crystallographic analysis of complex **5** (see Fig. 4). The contact of Sn(1) and S(4)# is significantly longer than the sum of the covalent radii of tin and sulfur atoms (2.42 Å) and less than the sum of the van der Waals radii for these atoms of 4.0 Å [20].

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 255212 for complex **2** and CCDC No. 255211 for complex **5**. Copies of this information may be obtained from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1233-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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