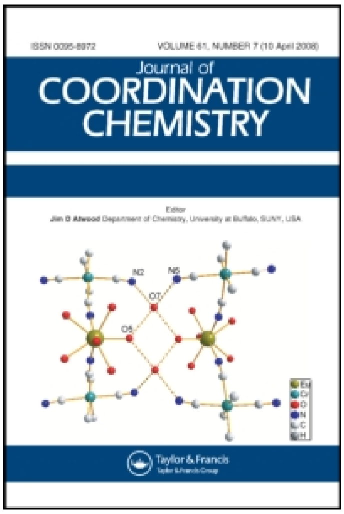

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Synthesis and crystal structure of two new Bi(III) complexes $\{(Me_2NCS_2)_3Bi\}_2$ and $\{[(CH_2)_5NCS_2]_2BiI\}_2$

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Six bismuth(III) complexes containing dithio-ligands formulated as $(R_2NCS_2)_3Bi$ [$R_2NCS_2M = Me_2NCS_2Na$, $C_4H_8NCS_2Na$, Bz_2NCS_2Na] and $[(R_2NCS_2)_2BiI]_2$ [$R_2NCS_2M = C_5H_{10}NCS_2Na$, $^{n}Bu_2NCS_2Na$, $OC_4H_8NCS_2Na$] have been obtained by reactions of bismuth(III) halides with dithiocarbamate ligands in 1 : 2 or 1 : 3 stoichiometry. All compounds were characterized by elemental and IR analyses. The crystal structures of complexes **1** and **4** have been determined by X-ray single crystal diffraction. The structure analyses reveal that Bi^{III} in complex **1** adopts a distorted pentagonal–pyramidal coordination, due to its stereochemically active lone pair of electrons. A long $Bi \cdots S$ contact of 3.218 (3) Å leads to dimeric associations of molecules in the crystal structure. The structure of complex **4** is six-coordination with a distorted octahedral configuration. Intramolecular $S \cdots S$ weak interactions contribute to the stability and lead to a one-dimensional chain structure.

Keywords: Bismuth(III) halides (I); Dithiocarbamate; Dimer; Crystal structure; X-ray diffraction

1. Introduction

Dialkyl-substituted dithiocarbamate anions have proved to be highly versatile chelating agents for the separation of metals as metal chelates using gas chromatography [1]. Their performance in liquid–liquid extraction and other analytical procedures [2] makes them attractive for the determination of metals using gas chromatography [3]. Some dialkyl-substituted dithiocarbamate salts have also shown interesting biological effects which include anti-alkylation [4, 5] or anti-HIV properties [6, 7]. They are also used as effective antidotes for cadmium detoxication [8, 9]. The ability of dtc to bind to metals has been known for many years, forming chelates with virtually all transition elements [10]. Water-soluble dialkyldithiocarbamate complexes have been tested in various medical applications [11]. However, main-group metal complexes with dithiocarbamate have been rarely studied, and few reports have appeared on the syntheses and structures of bismuth(III) complexes with dithiocarbamate. Bismuth complexes are used for the treatment of gastrointestinal disorders and may also be useful for the treatment of other diseases. Bi(III) exhibits highly variable coordination [12–19] and often an irregular coordination geometry. The coordination chemistry of Bi(III) with carboxylates and

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aminocarboxylates is dominated by intermolecular interactions which lead to polymeric structures. Bi(III) binds strongly to the thiolate sulfur of the tripeptide glutathione, however these adducts are also labile which allows rapid translocation of Bi(III) inside cells.

As a continuation of our interest in sulphur-containing ligands, we have synthesized six new bismuth(III) complexes $(R_2NCS_2)_3Bi$ [$R_2NCS_2M = Me_2NCS_2Na$, $C_4H_8NCS_2Na$, Bz_2NCS_2Na] and $[(R_2NCS_2)_2BiI]_2$ [$R_2NCS_2M = C_5H_{10}NCS_2Na$, $^{n}Bu_2NCS_2Na$, $O(CH_2CH_2)_2NCS_2Na$]. All the complexes have been characterized by elemental analyses and IR spectra. The crystal structure of complexes **1** and **4** has been determined by X-ray diffraction.

2. Experimental

2.1. *Preparation of the N,N′-dialkyldithiocarbamate sodium [Na(R₂dtc)]*

To a stirred solution of dialkyl-secondary amine (0.05 mol) in ethanol [(5 mL) or methanol] was added, at less than 4°C, carbon disulfide (3.1 mL, 0.052 mol) and sodium hydroxide (50% aqueous solution, 4 mL). After stirring for 4–5 h, evaporation of the volatile was performed without heating. Pure [Na(R_2dtc)] was obtained by recrystallization from components of ethanol (or methanol). Yield: 80–90%.

$$CS_2 + R_2NH + MOH \xrightarrow[\text{or } CH_3OH]{CH_3CH_2OH} R_2NCS_2M + H_2O$$

$$R_2NCS_2M = Me_2NCS_2Na,\quad Et_2NCS_2Na,\quad (CH_2)_4NCS_2Na,\quad (CH_2)_5NCS_2Na,$$
$$O(CH_2CH_2)_2NCS_2Na,\quad Bz_2NCS_2Na,\quad {}^{n}Bu_2NCS_2Na$$

2.2. *Synthesis*

2.2.1. Preparation of $(Me_2NCS_2)_3Bi$ (1). To a stirred solution of BiI_3 (0.1179 g, 0.2 mmol) in acetonitrile (20 mL) was added Me_2NCS_2Na (0.0858 g, 0.6 mmol). The reaction mixture was stirred for 4 h at 40°C. An orange-red-coloured solution was obtained and then filtered. The solvent is gradually removed by evaporation under vacuum until a solid is obtained. The solid is recrystallized from ethanol and the orange-red crystals of **1** are formed. Yield 80%. M.p. 258°C(dec.). Anal. Calcd (%) for $C_9H_{18}N_3S_6Bi$ (Mw = 569.60): C, 18.98; H, 3.19; N, 7.38. Found: C, 19.12; H, 3.33; N, 7.30.

2.2.2. Preparation $[(CH_2)_4NCS_2]_3Bi$ (2). Complex **2** is prepared in the same way as **1**. The orange solid was recrystallized from acetonitrile to give orange crystals, yield 85%, m.p. 240°C(dec.). Anal. Calcd (%) for $C_{15}H_{24}N_3S_6Bi$ (Mw = 647.74): C, 27.81; H, 3.73; N, 6.49. Found: C, 27.65; H, 3.90; N, 6.57.

2.2.3. Preparation of (Bz_2NCS_2)$_3$Bi (3). Complex **3** is prepared in the same way as **1**. The yellow solid product was recrystallized from ethanol to give yellow crystals, yield 69%. M.p. 285°C(dec.). Anal. Calcd (%) for $C_{45}H_{42}N_3S_6Bi$ (Mw = 1026.21): C, 52.67; H, 4.13; N, 4.09. Found: C, 52.50; H, 4.35; N, 4.23.

2.2.4. Preparation of {[(CH_2)$_5NCS_2$]$_2$BiI}$_2$ (4). Dithiopiperidylcarbamate (0.0733 g, 0.4 mmol) was dissolved in 10 mL of methanol. BiI_3 (0.118 g, 0.2 mmol) dissolved in 5 mL of methanol was added dropwise to the complex in solution with continuous stirring. The reaction mixture was stirred for 2 h and the volume of the solution was reduced to 10 mL. The resulting orange-yellow solution gave a pale yellow solid on evaporation. The solid was filtered, washed with CCl_4 and methanol and then dried over anhydrous calcium chloride. Yield 78%. M.p. 267°C(dec.). Anal. Calcd (%) for $C_{24}H_{40}N_4S_8Bi_2I_2$ (Mw = 1312.84): C, 21.96; H, 3.07; N, 4.27. Found: C, 21.78; H, 3.01; N, 4.43.

2.2.5. Preparation of [($^n Bu_2NCS_2$)$_2$BiI]$_2$ (5). Complex **5** is prepared in the same way as **4**. BiI_3(0.1179 g, 0.2 mmol) and $^n Bu_2NCS_2Na$ (0.0909 g, 0.4 mmol) were used. The yellow solid product was recrystallized from acetonitrile to give yellow crystals. Yield 78%. M.p. 267°C(dec.). Anal. Calcd (%) for $C_{36}H_{72}N_4S_8Bi_2I_2$ (Mw = 1489.27): C, 29.03; H, 4.87; N, 3.76. Found: C, 29.33; H, 4.56; N, 3.92.

2.2.6. Preparation of {[O(CH_2CH_2)$_2NCS_2$]$_2$BiI}$_2$ (6). Complex **6** is prepared in the same way as **4**. BiI_3(0.1179 g, 0.2 mmol) and Et_2NCS_2Na (0.0451 g, 0.2 mmol) were used. The yellow solid product was recrystallized from acetonitrile to give yellow crystals. Yield 78%. M.p. 259°C(dec.). Anal. Calcd (%) for $C_{20}H_{32}N_4O_4S_8Bi_2I_2$ (Mw = 1320.78): C, 18.19; H, 2.44; N, 4.24. Found: C, 18.36; H, 2.52; N, 4.20.

2.3. *Physical measurements*

The melting points were obtained with a Kolfer micro melting point apparatus and were uncorrected. The elemental analyses were performed on a PE-2400-II elemental analyzer. IR spectra were recorded on a Nicolet-460 spectrophotometer, as KBr discs.

2.4. *Crystal structure determination*

Crystals having approximate dimensions of $0.29 \times 0.22 \times 0.11\,mm^3$ **1** or $0.25 \times 0.18 \times 0.11\,mm^3$ **2** were mounted in a fiber. All measurements were made on a Bruker Smart 1000 CCD diffractometer with graphite-monochromated Mo-Kα (0.71073 Å) radiation at 298(2) K using the ϕ/ω scan technique. A total of 4899 or 5010 reflections for **1** or **4** were collected, of which 3231 or 3369 reflections were considered as observed [$I > 2\sigma(I)$] and used for the structure determination. Semi-empirical absorption corrections were applied.

The structures were solved by direct methods followed by Fourier syntheses. Structure refinement was carried out with full-matrix least-squares procedures using the SHELXL-97 programme package [20]. Hydrogen atoms were placed at calculated

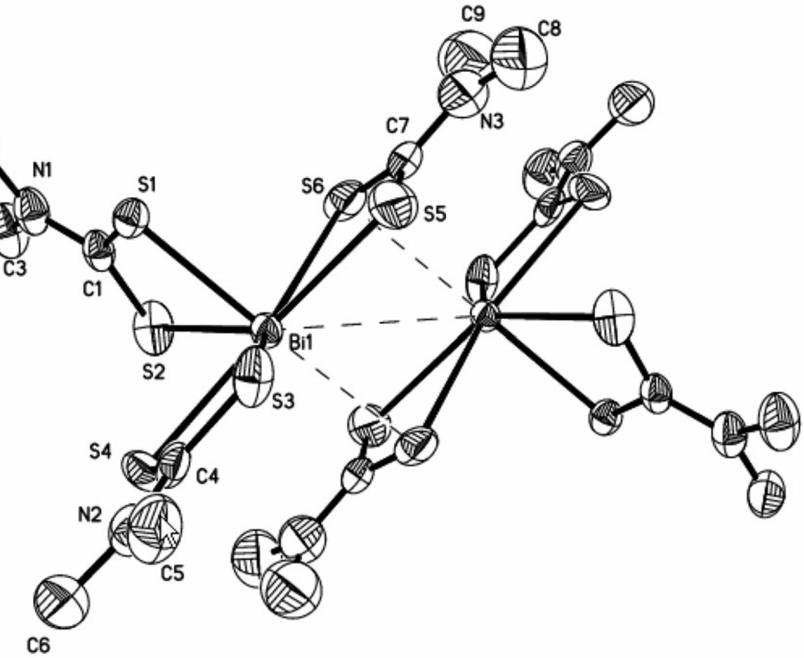

Figure 1. Molecular structure of **1** with 30% probability ellipsoids.

positions with C–H distances of 0.96 Å and riding on carrier atoms. Anisotropic refinement including all non-H atoms converged to $R = 0.0380$ **1** or $R = 0.0372$ **4** for all observed reflections.

2.5. *Crystal data*

For **1**, $C_9H_{18}N_3S_6Bi$, $M = 569.60$, triclinic, space group $P\bar{1}$ $a = 9.961(3)$, $b = 9.964(3)$, $c = 11.461(3)$ Å, $\alpha = 64.516(3)$, $\beta = 84.504(3)$, $\gamma = 64.699(3)$, $V = 922.9(4)$ Å^3, $Z = 2$, $F(000) = 544$, $D_x = 2.050\ \mathrm{g\,cm^{-3}}$, $S = 1.000$, $(\Delta\rho)_{\max} = 0.2606$, $(\Delta\rho)_{\min} = -0.1555\ \mathrm{e\,Å^{-3}}$.

For **4**, $C_{24}H_{40}N_4S_8Bi_2I_2$, $M = 1312.84$, triclinic, space group $P\bar{1}$, $a = 10.260(4)$, $b = 10.271(4)$, $c = 10.375(4)$ Å, $\alpha = 109.062(6)$, $\beta = 104.474(6)$, $\gamma = 98.784(6)^\circ$, $V = 967.2(7)$ Å^3, $Z = 2$, $F(000) = 612$, $D_x = 2.254\ \mathrm{g\,cm^{-3}}$, $S = 1.000$, $(\Delta\rho)_{\max} = 0.3739$, $(\Delta\rho)_{\min} = -0.1673\ \mathrm{e\,Å^{-3}}$.

3. Results and discussion

3.1. *Description of the crystal structures*

Complex **1** is depicted in figure 1 and the bond distances and angles are reported in table 1. The central bismuth is six-coordinate by six sulfur atoms belonging to three *N*,*N*-dimethyldithiocarbamate ligands. The resulting BiS6 coordination polyhedron approximates a pentagonal pyramid with atoms Bi1 and S2–S6 at the base of the

Table 1. Selected bond distances (Å) and angles (°) for complexes **1** and **4**.

1			
Bi(1)–S(1)	2.611(2)	Bi(1)–S(5)	2.829(3)
Bi(1)–S(3)	2.807(3)	Bi(1)–S(6)	2.927(3)
Bi(1)–S(4)	2.803(3)	N(1)–C(2)	1.459(13)
Bi(1)–S(2)	2.838(3)	N(1)–C(1)	1.328(12)
N(1)–C(3)	1.461(13)	N(2)–C(4)	1.340(13)
N(2)–C(5)	1.422(18)	N(2)–C(6)	1.440(18)
N(3)–C(7)	1.352(16)	N(3)–C(9)	1.43(2)
N(3)–C(8)	1.44(2)	S(1)–C(1)	1.763(10)
S(2)–C(1)	1.693(11)	S(3)–C(4)	1.730(13)
S(4)–C(4)	1.697(13)	S(5)–C(7)	1.692(16)
S(6)–C(7)	1.710(15)		
S(1)–Bi(1)–S(4)	87.20(9)	S(4)–Bi(1)–S(3)	63.83(11)
S(1)–Bi(1)–S(3)	89.49(9)	S(1)–Bi(1)–S(5)	92.48(10)
S(4)–Bi(1)–S(5)	141.60(13)	S(3)–Bi(1)–S(5)	77.77(12)
S(1)–Bi(1)–S(2)	65.99(9)	S(4)–Bi(1)–S(2)	78.38(11)
S(3)–Bi(1)–S(2)	136.02(9)	S(2)–Bi(1)–S(5)	135.72(12)
S(1)–Bi(1)–S(6)	86.79(8)	S(4)–Bi(1)–S(6)	156.29(12)
S(3)–Bi(1)–S(6)	138.97(11)	S(5)–Bi(1)–S(6)	61.63(13)
S(2)–Bi(1)–S(6)	78.22(10)	N(1)–C(1)–S(2)	121.5(8)
N(1)–C(1)–S(1)	118.2(8)	S(2)–C(1)–S(1)	120.3(6)
4			
Bi(1)–S(2)	2.646(2)	Bi(1)–S(3)	2.656(2)
Bi(1)–S(4)	2.686(2)	Bi(1)–S(1)	2.847(2)
Bi(1)–I(1)	3.2730(12)	N(1)–C(6)	1.473(10)
Bi(1)–I(1)#1	3.3093(12)	N(1)–C(2)	1.469(9)
I(1)#1–Bi(1)	3.3093(12)	N(1)–C(1)	1.297(9)
S(2)–C(1)	1.754(7)	S(1)–C(1)	1.700(8)
S(4)–C(7)	1.729(7)	S(3)–C(7)	1.715(7)
S(2)–Bi(1)–S(3)	95.39(8)	S(2)–Bi(1)–S(4)	88.36(7)
S(3)–Bi(1)–S(4)	67.22(6)	S(2)–Bi(1)–S(1)	64.89(6)
S(3)–Bi(1)–S(1)	85.25(7)	S(4)–Bi(1)–S(1)	139.87(7)
S(2)–Bi(1)–I(1)	86.68(6)	S(3)–Bi(1)–I(1)	148.36(5)
S(1)–Bi(1)–I(1)	123.39(6)	S(4)–Bi(1)–I(1)	81.32(5)
S(2)–Bi(1)–I(1)#1	174.77(5)	S(3)–Bi(1)–I(1)#1	89.81(6)
S(4)–Bi(1)–I(1)#1	93.23(6)	S(1)–Bi(1)–I(1)#1	116.18(5)
I(1)–Bi(1)–I(1)#1	88.64(4)		

Symmetry code: (#1 for **4**) −*x*, −*y*, −*z*.

pyramid and S1 in the apical position. the five equatorial Bi–S bonds are long [S2 to S6; mean = 2.841(3) Å] while the apical donor atom forms the strongest bond [Bi1–S1 = 2.611(2) Å]. The r.m.s. deviation from the mean plane for atoms S2–S6 is 0.2117 Å, and Bi1 deviates from this mean plane by 0.2934(3) Å, in the opposite sense to S1. The Bi^{III} lone pair of electrons may project in a direction roughly trans to the Bi1–S1 bond.

In $(Me_2NCS_2)_3Bi$, Bi^{III} adopts a distorted pentagonal–pyramid, due to its stereochemically active lone pair of electrons. A long Bi$\cdots$S contact of 3.218(3) Å leads to dimeric associations of molecules in the crystal structure.

The N–C bond distances in this complex (mean = 1.340(16) Å) are manifestations of the presence of a partial double bond. IR spectra of the above complex show ν(C–N) values at 1488 cm^{-1}, which lie between the single and double bond values, indicating contribution of the thioureide form to the dithiocarbamate [21].

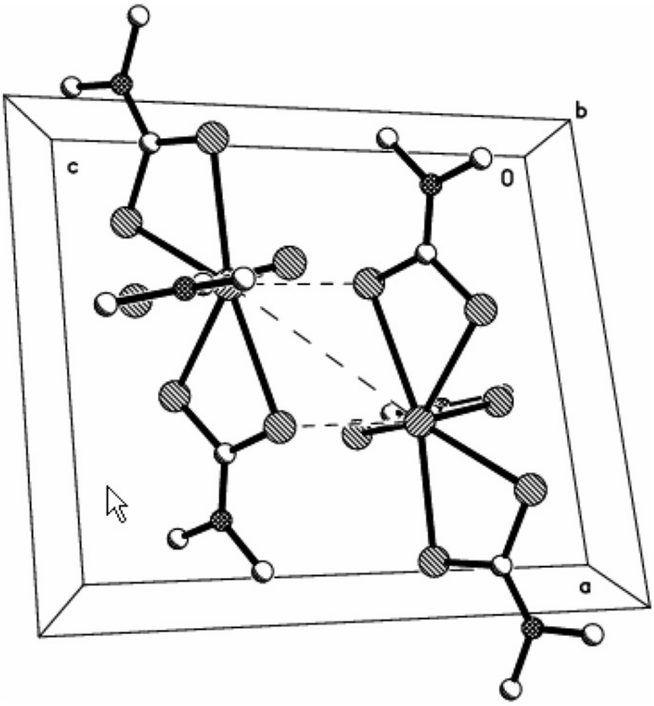

Figure 2. Unit cell of complex **1**.

The molecular structure of **4** is shown in figure 3 along with the numbering scheme. The complex is dimeric and $Bi(C_5H_{10}NCS_2)_2$ units are linked by iodide bridges. The two dithiocarbamate ligands are strongly coordinated through their negatively charged sulfur atoms.

The N(1)–C(1)–S(1) angles (123.2(6)°) are considerably larger than the N(1)–C(1)–S(2) angles (119.3(6)°). The N(1)–C(1) bond distance (1.297(9) Å) indicates the presence of a partial double bond and supports the contribution of the thioureide form to the dithiocarbamate ligand as indicated by an earlier study based on IR data [21, 22]. The −I inductive effect of the iodide does not influence the C–N bond distances in this compound.

Of the two Bi–I bonds, one is shorter (3.2730(12) Å) than the other (3.3093(12) Å). The Bi–I bond distances are normal, as observed in the diethyl analogue [23] and the other parameters of the dithiocarbamate ligands are normal.

As shown in figure 3, the structure of $[Bi(C_5H_{10}NCS_2)_2I]_2$ consists of a binuclear centrosymmetric neutral complex in which four sulphur atoms from two ligand molecules and two iodine coordinate to each bismuth atom, resulting in a distorted octahedron. Two iodine, related by a centre of symmetry, are bridging, so that the two octahedra share an edge. The central Bi atom is surrounded equatorially by I(1), S(1), S(3, S(4) and axially by S(2), I(1)#1($-x$, $-y$, $-z$). In this complex, because the piperidydithiocarbamate ligands bond with Bi in bidentate fashion, the angle of equatorial sulfur and iodine deviate from the standard octahedral angle. For example: the angles of three sulfur atoms and one iodine atom around Bi are S(1)–Bi(1)–I(1) 123.39(6)°, S(2)–Bi(1)–S(1) 64.89(6)°, S(2)–Bi(1)–S(4) 88.36(7)° and

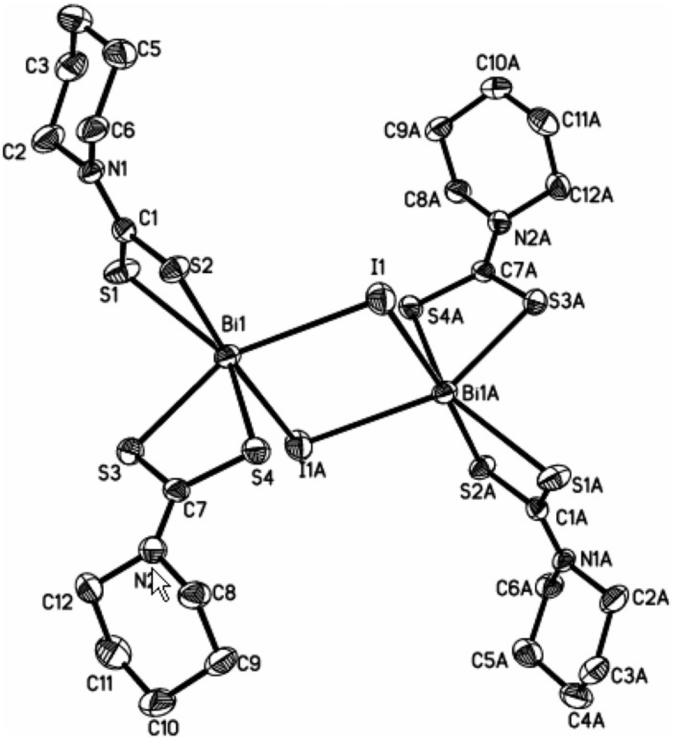

Figure 3. Molecular structure of **4** with 30% probability ellipsoids.

S(4)–Bi(1)–I(1) 81.32(5)°. All angles deviate from 90°, and the sum of these angles is 357.16°, which show that these atoms are not co-planar. Furthermore, the angle S(2)–Bi(1)–I(1)#1 being axial place is 174.77(5)°, deviating from 180°. The Bi atom of this complex is distorted octahedron bipyramidal coordination geometry.

In addition to the Bi–I coordination, the molecules are held together by weak interaction among S(1)–S(3)#2 $(1-x, -y, -z)$, S(3)–S(1)#2 $(1-x, -y, -z)$ and S(1)–S(1)#2 $(1-x, -y, -z)$, of neighbouring molecules. In fact this dithiocarbamate moiety may be considered as both a dithiocarbamate bridge and as a chelating ligand since the S(1)–S(3)#23.460(3), S(3)–S(1)#23.460(3) and S(1)–S(1)#2 3.338(5) are within the sum of the estimated van der Waal's radii. A representation of the scheme is shown in figure 5, where the S–S interaction has been highlighted. These interactions appear to reinforce the dithiocarbamate bridges in the formation of the polymeric structure.

3.2. *IR spectra*

Assignment of the IR bands of these complexes have been made by comparison with the IR spectra of related complexes with sodium dithiocarbamate. A new absorption band appears at $441 \sim 459\,\mathrm{cm}^{-1}$, characteristic vibration of Bi–S.

The most significant IR bands of the prepared compounds are shown in the table below. The band in the range $1481 \sim 1512\,\mathrm{cm}^{-1}$ is attributed to the ν(C–N) stretching [24]. The band in the range $981 \sim 995\,\mathrm{cm}^{-1}$, belonging to the ν(C–S)

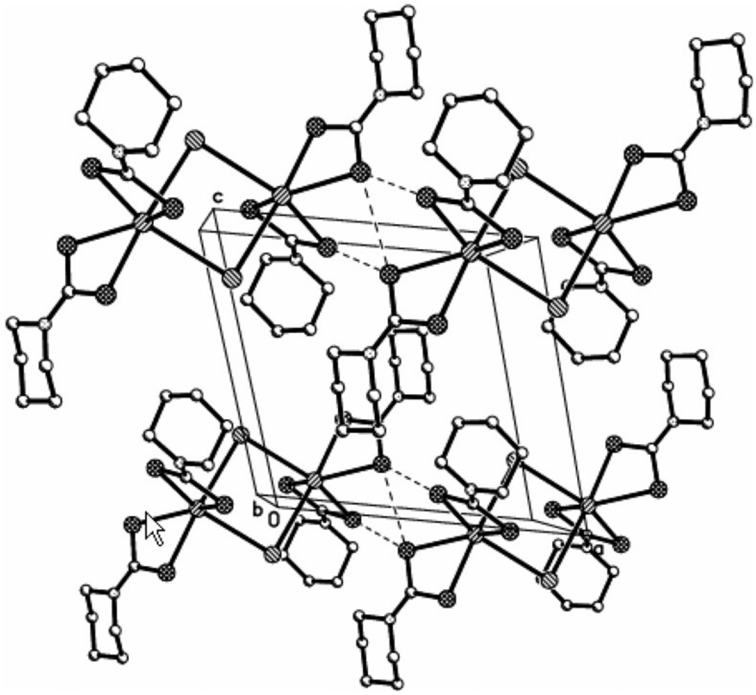

Figure 4. Unit cell of complex **4**.

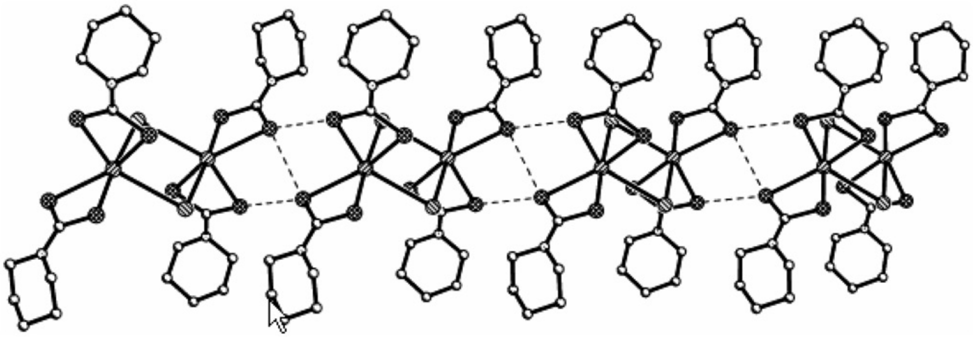

Figure 5. One-dimensional chain network of complex **4**.

stretching vibration [24, 25], is considered indicative of dithiocarbamate as a bidentate ligand [26].

Most significant IR band (cm^{-1}) of complexes **1**–**6**.

Compound	**1**	**2**	**3**	**4**	**5**	**6**
ν(Bi–S)	441	454	446	459	451	455
ν(C–N)	1514	1485	1510	1512	1481	1485
$\nu(CS_2)_{asym}$	1147	1155	1135	1147	1147	1128
$\nu(CS_2)_{sym}$	981	994	995	989	989	986

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 284463 for complex **1** and CCDC No. 283595 for complex **4**. 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; Email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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References

[1] M.L. Riekkola, O. Makitie, M. Sundberg. *Kem. Kemi.*, **6**, 523 (1979).
[2] G.D. Thorn, R.A. Ludwig. *The Dithio carbamate and Related Complex*, pp. 1–9, Elsevier, Amsterdam (1962).
[3] M.L. Riekkola, T. Pakkanen, L. Niinisto. *Acta Chim. Scand.*, **A37**, 807 (1983).
[4] A. Gringeri, P.C. Keng, R.F. Borch. *Cancer Res.*, **48**, 5708 (1988).
[5] M.H. Zeng, H. Liang, R.X. Hu. *Chinese J. Appl. Chem.*, **8**, 837 (2001).
[6] T.L. Sheng, X.T. Wu, P. Lin. *Polyhedron*, **18**, 1049 (1999).
[7] L.J. Farrugia, F.J. Lawlor, N.C. Norman. *J. Chem. Soc., Dalton Trans.*, 1163 (1995).
[8] Rdglinski, N.C. Norman (Eds). *J. Chemistry of Arsenic Antimony and Bismuth*, p. 403, Blackie Academic & Professional, London (1998).
[9] D.A. Atwood, A.H. Cowley, R.D. Hernandez, R.A. Jones, L.L. Rand, S.G. Bott, J.L. Atwood. *Inorg. Chem.*, **32**, 2972 (1993).
[10] L.Z. Xu, P.S. Zhao, S.S. Zhang. *Chin. J. Chem.*, **19**, 436 (2001).
[11] R.G. Pearson. *J. Am. Chem. Soc.*, **85**, 3533 (1963).
[12] J. Xie, T. Funakoshi, H. Shimada, S. Kojima. *Res. Commun. Mol. Pathol. Pharmacol.*, **86**, 245 (1994).
[13] R. Luckay, J.H. Reibenspies, R.D. Hancock. *J. Chem. Soc. Chem. Comm.*, 2365 (1995).
[14] J.J.R. Frausto da Silva, R.J.P. Williams. *The Biological Chemistry of the Elements: The Inorganic Chemistry of Life*, p. 538, Clarendon Press, Oxford (1991).
[15] R.D. Hancock, I. Cukrowski, J. Baloyi, J. Baloyi, J. Mashishi. *J. Chem. Soc. Dalton Trans.*, 2895 (1993).
[16] R.D. Hancock, I. Cukrowski, I. Antunes, E. Curkrowska, J. Mashishi, K. Brown. *Polyhedron*, **14**, 1699 (1995).
[17] W. Frank, G.J. Reiss, J. Schneiderm. *Angew. Chem. Int. Ed. Engl.*, **34**, 2416 (1995).
[18] F. Lazarini. *Cryst. Struct. Comm.*, **8**, 69 (1979).
[19] P. Kopi-Maier, T. Klapotke. *Inorg. Chim. Acta*, **152**, 49 (1988).
[20] G.M. Sheldrick. *SHELX-97, Program for Crystal Structure Analysis*, University of Gottingen, Germany (1997).
[21] D. Coucouvains. *Proj. Inorg. Chem.*, **11**, 233 (1970).
[22] D. Coucouvains. *Proj. Inorg. Chem.*, **26**, 301 (1979).
[23] G. Mckie, C.L. Raston, G.L. Rowbottom, A.H. White. *J. Chem. Soc., Dalton Trans.*, 1360 (1981).
[24] H.D. Yin, C.H. Wang, R.F. Zhang. *Chin. J. Inorg. Chem.*, **16**, 619 (2000).
[25] H.D. Yin, C.L. Ma. *Chin. Appl. Chem.*, **20**, 108 (2000).
[26] J.S. Casas, A. Sanchez, J. Bravo, G.F. Soledad, E.E. Castellano, M.M. Jones. *Inorg. Chim. Acta*, **158**, 119 (1989).