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Syntheses, characterization and crystal structures of new organogermanium complexes with 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole

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Abstract

A series of organogermanium(IV) complexes with 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole of the type, $R_nGe[S(C_8H_5N_2S_2)]_{4-n}$ ($n = 3$, $R = CH_3$, **1**; C_2H_5, **2**; C_4H_9, **3**; C_6H_5, **4**; $n = 2$, $R = CH_3$, **5**; C_2H_5, **6**; C_4H_9, **7**; C_6H_5, **8**) have been synthesized. All the complexes **1**–**8** have been characterized by elemental, IR and ^{1}H NMR analyses. Among them complexes **4** and **8** have also been characterized by X-ray crystallography diffraction analyses, which revealed that the structure of complex **4** is a tetrahedron while the germanium environment of complex **8** is a distorted octahedron. Furthermore, an intermolecular non-bonded sulfur–sulfur interaction was recognized in complex **8**. The IR and ^{1}H NMR spectral data indicate such an inclination that those structures of trialkylgermanium derivatives are apt to be four-coordinated and those of dialkylgermanium derivatives are likely to be six-coordinated.

Keywords: 2,5-Dimercapto-4-phenyl-1,3,4-thiodiazole; Organogermanium(IV); Non-bonded interaction; Crystal structures; X-ray crystallography diffraction

1. Introduction

The complexes of Group 14 elements (silicon, tin and germanium(IV)) can be obtained by using their tetrahalides with appropriate ligands [1]. Recently this field has attracted much attention in order to investigate the influences of the functional groups on the coordination modes and properties of these complexes [2,3]. However, until now most of the studies have been focused on the organotin and organosilicon compounds for either the diversity of their commercial application or the richness of their structural chemistry [4–8]. In our previous work, we have synthesized and characterized a variety of penta- or hexa-coordinate organotin complexes where in most cases the donor is nitrogen or sulfur [9,10]. From a theoretical point of view, we think it necessary to do an initial probe in the coordination chemistry of germanium complexes so that we report some details of the syntheses of a series of organogermanium complexes of the type $R_nGe[S(C_8H_5N_2S_2)]_{4-n}$ ($n = 3$, $R = CH_3$, **1**; C_2H_5, **2**; C_4H_9, **3**; C_6H_5, **4**; $n = 2$, $R = CH_3$, **5**; C_2H_5, **6**; C_4H_9, **7**; C_6H_5, **8**) and characterize them by elemental, IR, ^{1}H NMR analyses. X-ray crystallography analyses of the complexes **4** and **8** have also been given in the present paper.

2. Experimental

2.1. Materials and measurements

All reagents and solvents were purchased commercially and used without further purification unless otherwise noted. The melting points were obtained with Kofler micro melting point apparatus and were uncorrected. Infrared-spectra were recorded on a Nico-

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let-460 spectrophotometer using KBr discs and sodium chloride optics. ^{1}H NMR spectra were recorded on a JEOL-FX-90Q spectrometer using TMS as internal standard and $CDCl_3$ as solvent. The chemical shifts were reported in ppm. Elemental analyses were performed with a PE-2400II apparatus.

2.2. Syntheses of the complexes **1–8**

2.2.1. $(CH_3)_3Ge[S(C_8H_5N_2S_2)]$ (**1**)

The reaction was carried out under nitrogen atmosphere. The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.264 g, 1 mmol) and trimethylgermanium chloride (0.153 g, 1 mmol) were added to the solution of absolute benzene (20 ml) in a Schlenk flask, stirred for 12 h at 40 °C and then filtrated. The filtered solution was gradually reduced by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ether. White crystal complex **1** was formed. Yield: 91%. M.p. 138–140 °C. *Anal.* Found: C, 37.89; H, 4.01; N, 8.29; S, 28.21; Calc. for $C_{11}H_{14}GeN_2S_3$: C, 37.87; H, 4.12; N, 8.17; S, 28.00%. ^{1}H NMR ($CDCl_3$): 7.20–7.33 (m, 5H, C_6H_5–N), 1.71 (s, 9H, Ge–CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3028; ν(C–H), 2985, 2870; ν(Ge–C), 585; ν(Ge–S), 446; ν(C=N), 1595; ν(C–S), 981; ν(C=S), 1256.

2.2.2. $(C_2H_5)_3Ge[S(C_8H_5N_2S_2)]$ (**2**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.264 g, 1 mmol) and triethylgermanium chloride (0.195 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. After removing the solvent, the solid was redissolved in *n*-hexane. Colorless crystal complex **2** was formed. Yield: 87%. M.p. 124–126 °C. *Anal.* Found: C, 43.89; H, 5.66; N, 5.35; S, 25.17; Calc. for $C_{14}H_{20}GeN_2S_3$: C, 43.85; H, 5.78; N, 5.24; S, 24.94%. ^{1}H NMR ($CDCl_3$): 7.22–7.35 (m, 5H, C_6H_5–N), 1.76 (q, 6H, Ge–CH_2), 0.88 (t, 9H, CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3024; ν(C–H), 2979, 2865; ν(Ge–C), 579; ν(Ge–S), 457; ν(C=N), 1607; ν(C–S), 977; ν(C=S), 1256.

2.2.3. $(n\text{-}C_4H_9)_3Ge[S(C_8H_5N_2S_2)]$ (**3**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.264 g, 1 mmol) and tributylgermanium chloride (0.279 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. Yellow crystal complex **3** was obtained from benzene. Yield: 89%. M.p. 58–60 °C. *Anal.* Found: C, 51.25; H, 6.73; N, 6.12; S, 20.61; Calc. for $C_{20}H_{32}GeN_2S_3$: C, 51.21; H, 6.88; N, 5.97; S, 20.47%. ^{1}H NMR ($CDCl_3$): 7.15–7.32 (m, 5H, Ph–H), 1.79 (t, 6H, Ge–CH_2), 1.13–1.50 (m, 12H, CH_2), 0.86 (t, 9H, CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3020; ν(C–H), 2978, 2868; ν(Ge–C), 588; ν(Ge–S), 461; ν(C=N), 1603; ν(C–S), 973; ν(C=S), 1257.

2.2.4. $Ph_3Ge[S(C_8H_5N_2S_2)]$ (**4**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.264 g, 1 mmol) and triphenylgermanium chloride (0.339 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. Yellow crystal complex **4** was obtained from *n*-hexane. Yield: 89%. M.p. 166–168 °C. *Anal.* Found: C, 59.04; H, 3.73; N, 5.37; S, 18.30; Calc. for $C_{26}H_{20}GeN_2S_3$: C, 59.03; H, 3.81; N, 5.29; S, 18.15%. ^{1}H NMR ($CDCl_3$): 7.34–7.67 (m, 15H, Ph–H), 7.19–7.34 (m, 5H, C_6H_5–N). IR (KBr, cm^{-1}): ν(Ph–H), 3026; ν(Ge–C), 1098; ν(Ge–S), 458 cm^{-1}; ν(C=N), 1610; ν(C–S), 973; ν(C=S), 1257.

2.2.5. $(CH_3)_2Ge[S(C_8H_5N_2S_2)]_2$ (**5**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.528 g, 2 mmol) and dimethylgermanium dichloride (0.173 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. White crystal complex **5** was obtained from ether. Yield: 90%. M.p. 128–130 °C. *Anal.* Found: C, 39.22; H, 2.71; N, 10.30; S, 34.82; Calc. for $C_{18}H_{16}GeN_4S_6$: C, 39.10; H, 2.92; N, 10.13; S, 34.73%. ^{1}H NMR ($CDCl_3$): 7.20–7.32 (m, 5H, NC_6H_5), 1.78 (s, 6H, Ge–CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3048; ν(C–H), 2964, 2872; ν(Ge···N), 632; ν(Ge–C), 589; ν(Ge–S), 453; ν(C=N), 1626; ν(C–S), 990; ν(C=S), 1256.

2.2.6. $(C_2H_5)_2Ge[S(C_8H_5N_2S_2)]_2$ (**6**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.528 g, 2 mmol) and diethylgermanium dichloride (0.202 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. Colorless crystal complex **6** was obtained from ether-dichloromethane. Yield: 92%. M.p. 112–114 °C. *Anal.* Found: C, 41.44; H, 3.29; N, 9.88; S, 33.17; Calc. for $C_{20}H_{20}GeN_4S_6$: C, 41.35; H, 3.47; N, 9.64; S, 33.05%. ^{1}H NMR ($CDCl_3$): 7.22–7.35 (m, 5H, C_6H_5–N), 1.75 (q, 4H, Ge–CH_2), 0.94 (t, 6H, CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3055; ν(C–H), 2957, 2856; ν(Ge···N), 636; ν(Ge–C), 586; ν(Ge–S), 461; ν(C=N), 1628; ν(C–S), 982; ν(C=S), 1257.

2.2.7. $(n\text{-}C_4H_9)_2Ge[S(C_8H_5N_2S_2)]_2$ (7)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.528 g, 2 mmol) and dibutylgermanium dichloride (0.258 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. Yellow crystal complex **7** was obtained from *n*-hexane. Yield: 88%. M.p. 74–76 °C. *Anal.* Found: C, 45.37; H, 2.99; N, 8.91; S, 30.25; Calc. for $C_{24}H_{28}GeN_4S_6$: C, 45.25; H, 3.16; N, 8.79; S, 30.14%. ^{1}H NMR ($CDCl_3$): 7.22–7.34 (m, 5H, Ph–H), 1.73 (t, 4H, Ge–CH_2), 1.12–1.50 (m, 8H, CH_2), 0.91 (t, 6H, CH_3). IR (KBr, cm^{-1}): ν(Ph–H), 3047; ν(C–H), 2978, 2868; ν(Ge···N), 628; ν(Ge–C), 578; ν(Ge–S), 459; ν(C=N), 1624; ν(C–S), 974; ν(C=S), 1257.

2.2.8. $Ph_2Ge[S(C_8H_5N_2S_2)]_2$ (**8**)

The potassium salt of 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole (0.528 g, 2 mmol) and diphenylgermanium dichloride (0.298 g, 1 mmol) in benzene (20 ml) were stirred for 12 h at 40 °C and then filtrated. The filtered solution was gradually removed until solid product was obtained. The solid was then recrystallized from ether. Yellow crystal complex **8** was formed. Yield: 92%. M.p. 118–120 °C. *Anal*. Found: C, 53.13; H, 2.80; N, 8.36; S, 28.44; Calc. for $C_{28}H_{20}GeN_4S_6$: C, 53.03; H, 2.98; N, 8.27; S, 28.36%. ^1H NMR ($CDCl_3$): 7.35–7.64 (m, 10H, Ph–H), 7.21–7.35 (m, 5H, C_6H_5–N). IR (KBr, cm^{-1}): ν(Ph–H), 3061; ν(Ge–C), 1096; ν(Ge···N), 633; ν(Ge–S), 453; ν(C=N), 1626; ν(C–S), 986; ν(C=S), 1257.

2.3. X-ray crystallographic studies of complexes **4** and **8**

All X-ray crystallographic data were collected on a Bruker SMART CCD 1000 diffractometer. Criterion of observability was used for the solution and refinement. 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 at 298(2) K using graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) and corrected for Lorentz and polarization effects but not for absorption. 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.

3. Results and discussion

3.1. Syntheses of the complexes **1**–**8**

The synthesis procedure is given in Scheme 1.

3.2. IR spectroscopic studies of the complexes **1**–**8**

The assignment of IR bands of these complexes has been made by comparison with the IR spectra of their related precursors. A new absorption band, appearing at about 450 cm^{-1} for all eight complexes **1**–**8**, can be attributed to the vibrations of Ge–S bond according to the literature [11]. The strong peaks at about 1255 cm^{-1} have been attributed to the vibration of $\nu_{C=S}$ in all complexes **1**–**8**. Together with the absorption region ranging from 973 to 990 cm^{-1} attributed to the vibration of $\nu_{C-S,}$ all above messages indicate the coordination of the ligand to germanium by sulfur atom in all complexes **1**–**8**. So it can be concluded that complexes **1**–**4** are four-coordinated with a sulfur atom and three alkyl (or phenyl) groups substituted to the central germanium atom. The geometry at the germanium atom can be described as Scheme 2a. The conclusion well coincides with the following X-ray crystallographic analysis of complex **4**.

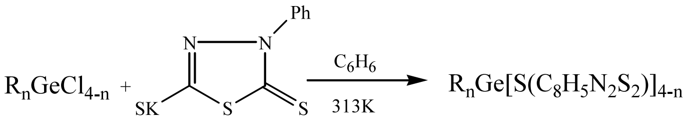
It is worthy to note that besides the absorption region belonging to Ge–S, all spectra of complexes **5**–**8** have another weak bands at about 630 cm^{-1} which can be attributed to Ge···N vibration [12]. Furthermore, the absorption band belonging to C=N in complexes **5**–**8** all shift about 20 cm^{-1} towards high-frequency compared with those of complexes **1**–**4**, which also prove that there exist the weak coordination from nitrogen atom of the ligand to germanium atom in complexes **5**–**8**. As a result, the probable geometry for complexes **5**–**8** can be described as distorted octahedron, see Scheme 2b. The following crystal structure of complex **8** well supports such a conclusion.

3.3. NMR data of the complexes **1**–**8**

The ^1H NMR spectra show that the chemical shifts of the phenyl group (Ge–C_6H_6) in complexes **4** and **8**, 7.34–7.67 ppm, and those of methylene connected directly with germanium in complexes **1**–**3** and complexes **5**–**7**, 1.72–1.79 ppm, upfield shift as compared with those of their corresponding precursors. All these data are similar to those cases appear in literature [13], indicating there may exist novel coordination of the ligand to germanium atom for all the eight complexes **1**–**8**. In addition, the resonance of the phenyl group connected with nitrogen atom (N–Ph) appears at 7.15–7.35 ppm for all complexes **1**–**8**.

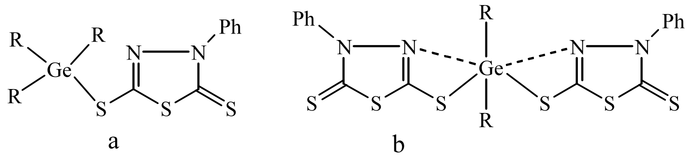
3.4. Crystal structures of complexes **4** and **8**

The crystal structures and unit cells or crystal packing of complexes **4** and **8** in crystal are shown in Figs. 1–4, respectively. All H atoms have been omitted for the purpose of clarity. Table 1 list the crystal data and structure refinement parameters for complexes **4** and **8**.



(n=3, R=CH_3, **1**; C_2H_5, **2**; C_4H_9, **3**; C_6H_5, **4**; n=2, R=CH_3, **5**; C_2H_5, **6**; C_4H_9, **7**; C_6H_5, **8**)

Scheme 1.



(n=3, R=CH_3, **1**; C_2H_5, **2**; C_4H_9, **3**; C_6H_5, **4**; n=2, R=CH_3, **5**; C_2H_5, **6**; C_4H_9, **7**; C_6H_5, **8**)

Scheme 2.

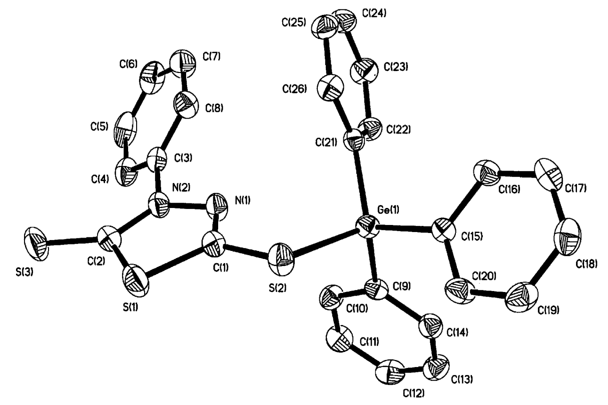


Fig. 1. Molecular structure of complex **4**.

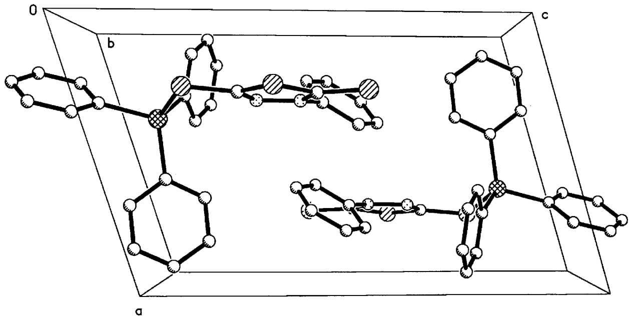


Fig. 2. Unit cell of complex **4**.

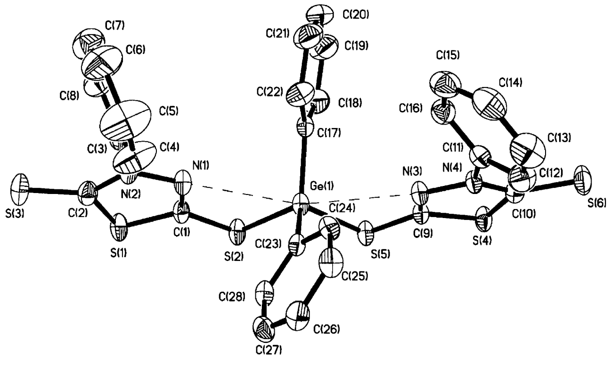


Fig. 3. Molecular structure of complex **8**.

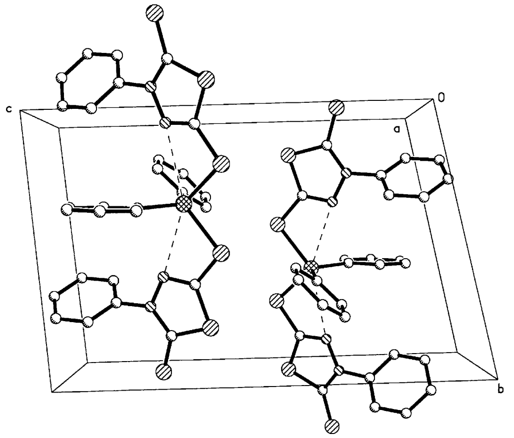


Fig. 4. Unit cell of complex **8**.

Tables 2 and 3 lists the selected bond lengths and angles of complexes **4** and **8**.

For complex **4**, the central germanium atom forms four primary bonds: three to the phenyl groups and one to the sulfur atom. Thus, complex **4** displays an almost perfect tetrahedral coordination sphere with four angles ranging from 108.91(8) to 111.79(10)°. The Ge–C bond lengths (1.927(2) to 1.937(3) Å) are in the normal range (1.93 Å for Ge–Cp) [2]. The Ge–S bond lengths (2.2798(9) Å) is a little longer than that reported in dichloro-(ferrocene-1,1′-dithiolato)-germane (2.19 Å) [2].

For complex **8**, the six-coordinate metal center displays a slightly distorted octahedral geometry with two carbons and two sulfur atoms covalently linked to the germanium atom. The valence extension is performed via the nitrogen atoms. The two chelating nitrogen atoms occupy *trans* positions (N(1)–Ge(1)–N(3), 148.65(10)°), while the cases for the sulfur bonding vary and they occupy *cis* positions (S(2)–Ge(1)–S(4), 93.15(5)°). Besides, the S and N equatorial ligating atoms belong to the same moiety (S(5)–Ge(1)–N(3): 59.74(8)°, S(2)–Ge(1)–N(1): 59.53(8)°), so their positions are fixed and the S–Ge–N angles can only admit very little deformation. The sum of angles between the germanium atom and the equatorial ligating atoms (i.e. two N and two S in each case) is 360.0°, typical for the ideal octahedral value of 360°. The Ge–C bond lengths (1.922(4) and 1.924(4) Å) are quite close to the ones in the literature [2]. It is worthy to note that in complex **8** both Ge–N bond lengths are markedly elongated (2.984(3) and 3.005(4) Å) compared to those reported in $GeCl_4$(bipy) and GeCl(bipy)(hcit) with values ranging from 2.000(3) to 2.068(4) Å [14], so it is obvious that the

Table 1
Crystal data and structure refinement parameters for complexes **4** and **8**

	Complex **4**	Complex **8**
Empirical formula	$C_{26}H_{20}GeN_2S_3$	$C_{28}H_{20}GeN_4S_6$
Formula weight	529.21	677.43
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions		
a (Å)	9.194(2)	9.956(6)
b (Å)	9.595(2)	10.700(6)
c (Å)	14.648(4)	15.026(8)
α (°)	85.277(4)	97.225(9)
β (°)	72.303(3)	91.511(9)
γ (°)	79.430(4)	111.855(7)
V (Å^3)	1209.2(5)	1469.2(14)
Z	2	2
D_{calc} (g cm^{-3})	1.453	1.531
Crystal size (mm)	0.40 × 0.40 × 0.30	0.30 × 0.20 × 0.09
F(0 0 0)	540	688
Total reflections	7091	8731
Independent reflections	4855	5909
Data/restraints/parameters	4855/0/369	5909/0/432
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0360$, $wR_2 = 0.0667$	$R_1 = 0.0397$, $wR_2 = 0.0784$
R indices (all data)	$R_1 = 0.0550$, $wR_2 = 0.0724$	$R_1 = 0.0842$, $wR_2 = 0.1075$
Goodness-of-fit	0.909	0.825
θ Range for data collection (°)	1.46–26.42	1.37–26.37
Absorption coefficient (mm^{-1})	1.543	1.495
Largest difference peak and hole (e Å^{-3})	0.445 and −0.358	0.344 and −0.425

Ge–N coordination is very weak. Concerning the Ge–S bond lengths (2.2588(15) Å for Ge(1)–S(2) and 2.2786(14) Å for Ge(1)–S(5)), we may note that they are similar to those in complex **4**.

Besides, intermolecular non-bonded S· · ·S interactions were recognized in the crystallographic analysis of complex **8,** which help the construction of the dimer of complex **8**. The non-bond S· · ·S distance (3.61 Å for S(5) · · ·S(2)# and 3.72 Å for S(2)· · ·S(2)#) is longer than those reported in literature [15,16] but close to the sum (3.70 Å) of the van der Waals radii (S and S) [17]. There have been several papers that have discussed the non-bonded S· · ·S interaction [18,19] and what we recognized in complex **8** can be regarded as a supplement to that kind of contact.

Table 3
Selected bond lengths (Å) and angles (°) for complex **8**

Bond lengths			
Ge(1)–C(23)	1.922(4)	Ge(1)–C(17)	1.924(4)
Ge(1)–S(2)	2.2588(15)	Ge(1)–S(5)	2.2786(14)
Ge(1)–N(1)	2.984(3)	Ge(1)–N(3)	3.005(4)
S(5)· · ·S(2)#	3.61	S(2)· · ·S(2)#	3.72
Bond angles			
C(23)–Ge(1)–C(17)	119.34(19)	C(23)–Ge(1)–S(2)	110.32(13)
C(17)–Ge(1)–S(2)	111.18(13)	C(23)–Ge(1)–S(5)	113.13(12)
C(17)–Ge(1)–S(5)	106.61(14)	S(2)–Ge(1)–S(5)	93.15(5)
C(23)–Ge(1)–N(1)	81.89(13)	C(17)–Ge(1)–N(1)	83.12(14)
S(2)–Ge(1)–N(1)	59.53(8)	S(5)–Ge(1)–N(1)	152.53(8)
C(23)–Ge(1)–N(3)	82.04(14)	C(17)–Ge(1)–N(3)	81.59(13)
S(2)–Ge(1)–N(3)	151.82(8)	S(5)–Ge(1)–N(3)	58.74(8)
N(1)–Ge(1)–N(3)	148.65(10)		

4. Conclusions

A series of germanium complexes based on the 2,5-dimercapto-4-phenyl-1,3,4-thiodiazole ligand containing two functional heterocyclic pendants have been synthesized and characterized. Detail studies on the structures and spectra of these complexes indicate that there exist such an inclination that the structures of those trialkylgermanium complexes **1**–**4** are apt to be of tetrahedral geometry and those dialkylgermanium complexes **5**–**8** are likely to construct octahedral geometry.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 206691 and 206692 for complexes **4** and **8**, respectively. Copies of this information may be obtained free of charge from the Director,

Table 2
Selected bond lengths (Å) and angles (°) for complex **4**

Bond lengths			
Ge(1)–C(21)	1.927(2)	Ge(1)–C(15)	1.934(2)
Ge(1)–C(9)	1.937(3)	Ge(1)–S(2)	2.2798(9)
Bond angles			
C(21)–Ge(1)–C(15)	111.50(10)	C(21)–Ge(1)–C(9)	113.59(11)
C(15)–Ge(1)–C(9)	111.79(10)	C(21)–Ge(1)–S(2)	110.95(8)
C(15)–Ge(1)–S(2)	99.15(8)	C(9)–Ge(1)–S(2)	108.91(8)

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