

Available online at www.sciencedirect.com



Polyhedron 23 (2004) 1981-1986



Syntheses, characterizations and crystal structures of new triorgano-tin or -germanium complexes with 1H-5-mercapto-1,2,3-triazolato

Chunlin Ma^{a,b,*}, Junhong Zhang^a, Rufen Zhang^a

^a Department of Chemistry, Liaocheng University, 34 Wenhua Road, Liaocheng 252059, China
^b Department of Chemistry, Taishan University, Taian 271021, China

Received 15 March 2004; accepted 4 May 2004

Abstract

A series of triorganotin and triorganogermanium(IV) complexes with 1H-5-mercapto-1,2,3-triazolato, of the type $R_3MSC_2H_2N_3$ (M = Sn, R = CH₃, (1); C₂H₅, (2); *n*-C₄H₉, (3); C₆H₅, (4); M = Ge, R = CH₃, (5); C₂H₅, (6); *n*-C₄H₉, (7); C₆H₅, (8)), have been synthesized. All the complexes 1–8 have been characterized by elemental, IR and ¹H NMR analyses. Among them complexes 4 and 8 have also been characterized by X-ray crystallography diffraction analyses, which revealed that the Sn and Ge atom environments are distorted tetrahedral coordination polyhedrons, coordinated to three phenyl groups and to one sulfur atom of the ligand. The packing of complexes 4 and 8 is stabilized into a one-dimensional infinite chain by intermolecular N–H···N hydrogen bonds. © 2004 Elsevier Ltd. All rights reserved.

Keywords: 1H-5-mercapto-1,2,3-triazolato; Organotin; Organogermanium(IV); Crystal structure; X-ray crystallography

1. Introduction

Metal thiolato complexes have been extensively studied because of their ability to adopt various nuclearities and their relevance in biology, since they form the inorganic part of the biologically active centers of some metalloproteins and enzymes [1-3]. Recently, attention has been paid to the coordination chemistry of heterocyclic thiol/thione donors, which can give potential access to new compounds with unusual structures and reactivities [4]. For example, triazyl- and tetrazole-thiol organometallic derivatives have been reported. A related class of such compounds is thiosemicarbazones in which the structure unit HS–C–N–N or S=C–N–N can bond to metal ions through S or N or both atoms [5–8].

As a result of our continuing interest in the coordination of main group metals with polydentate S, N ligands [9–11], here we report the studies on the syntheses of a series of organotin and organogermanium complexes $R_3MSC_2H_2N_3$ (M = Sn, R = CH₃, (1); C_2H_5 , (2); n-C₄H₉, (3); C₆H₅, 4; M = Ge, R = CH₃, (5); C₂H₅, (6); C₄H₉, (7); C₆H₅, (8)) and their characterization by elemental, IR and ¹H 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 the sodium salt of the ligand (1H-5mercapto-1,2,3-triazole) were purchased commercially and used without further purification unless otherwise noted. The melting points were obtained with a Kofler micro-melting point apparatus and were uncorrected. Infrared-spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs and sodium chloride optics. ¹H NMR spectra were recorded on a JEOL-FX-90Q spectrometer using TMS as internal standard and

^{*}Corresponding author. Tel.: +86-635-8238121; fax: +86-635-8238274/86-538-671-5521.

E-mail address: macl@lctu.edu.cn (C. Ma).

 $CDCl_3$ as solvent. The chemical shifts are reported in ppm. Elemental analyses were performed with a PE-2400II apparatus.

2.2. Syntheses of the complexes 1–8

2.2.1. $(CH_3)_3 Sn(SC_2H_2N_3)$ (1)

The reaction was carried out under nitrogen atmosphere. The sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol) and trimethyltin chloride (0.199 g, 1 mmol) were added to a solution of absolute benzene (20 ml) in a Schlenk flask, stirred for 12 h at 40 °C and then filtered. The filtered solution was gradually reduced by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ether. The white crystal complex 1 was formed. Yield, 89%. M.p.: 121-123 °C. Anal. Calc. for C₅H₁₁N₃SSn: C, 22.76; H, 4.20; N, 15.93. Found: C, 22.72; H, 4.18; N, 15.96%. IR (KBr, cm⁻¹): 3251 (br, N–H), 3052 (w, C– H), 1605 (m, C=C), 1596 (C-N), 1442 (m, N=N), 1156, 1078 (m, C-S), 560, 535 (m, Sn-C), 319 (m, Sn-S). ¹H NMR (CDCl₃): δ 10.9 (br, 1H, N-H), 1.53 (m, 9H, CH₃), 7.30 (s, 1H, =CH).

2.2.2. $(C_2H_5)_3Sn(SC_2H_2N_3)$ (2)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), triethyltin chloride (0.241 g, 1 mmol) and benzene (20 ml). The colorless crystal complex **2** was obtained from *n*-hexane. Yield: 82%. M.p.: 124–126 °C. *Anal.* Calc. for C₈H₁₇N₃SSn: C, 31.41; H, 5.60; N, 13.74. Found: C, 31.38; H, 5.46; N, 13.79%. IR (KBr, cm⁻¹): 3250 (br, N–H), 3050 (w, C–H), 1605 (m, C=C), 1595 (C–N), 1443 (m, N=N), 1152, 1076 (m, C–S), 561, 524 (m, Sn–C), 317 (m, Sn–S). ¹H NMR (CDCl₃): δ 10.5 (br, 1H, N–H), 1.76 (q, 6H, –CH₂), 0.88 (t, 9H, CH₃), 7.28 (s, 1H, =CH).

2.2.3. $(n-C_4H_9)_3Sn(SC_2H_2N_3)$ (3)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), tributyltin chloride (0.325 g, 1 mmol) and benzene (20 ml). The colourless crystal complex **3** was obtained from benzene. Yield: 80%. M.p.: 58–60 °C. *Anal.* Calc. for C₁₄H₂₉N₃SSn: C, 43.11; H, 7.50; N, 10.78. Found: C, 43.14; H, 7.53; N, 10.76%. IR (KBr, cm⁻¹): 3249 (br, N–H), 3050 (w, C–H), 1605 (m, C=C), 1595 (C–N), 1443 (m, N=N), 1152, 1076 (m, C–S), 565, 540 (m, Sn–C), 316 (m, Sn–S). ¹H NMR (CDCl₃): δ 10.6 (br, 1H, N–H), 1.79 (t, 6H, –CH₂), 1.13–1.50 (m, 12H, CH₂), 0.86 (t, 9H, CH₃), 7.28 (s, 1H, =CH).

2.2.4. $Ph_3Sn(SC_2H_2N_3)$ (4)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-

triazole (0.123 g, 1 mmol), triphenyltin chloride (0.385 g, 1 mmol) and benzene (30 ml). The yellow crystal complex **4** was obtained from hexane–dichloromethane. Yield, 89%. M.p.: 128–130 °C. *Anal.* Calc. for $C_{20}H_{17}N_3Sn$: C, 53.37; H, 3.81; N, 9.33; S, 7.12. Found: C, 53.24; H, 3.76; N, 9.28; S, 6.97%. IR (KBr, cm⁻¹): 3250 (br, N–H), 3050 (w, C–H), 1605 (m, C=C), 1595 (C–N), 1443 (m, N=N), 1152, 1076 (m, C–S), 561, 533 (m, Sn–C), 317 (m, Sn–S). ¹H NMR (CDCl₃): δ 11.0 (br, 1H, N–H), 7.31–7.96 (m, 15H, Ph–H), 7.28 (s, 1H, =CH).

2.2.5. $(CH_3)_3 Ge(SC_2H_2N_3)$ (5)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), trimethylgermanium chloride (0.153 g, 1 mmol) and benzene (20 ml). The yellow crystal complex **5** was obtained from hexane–dichloromethane. Yield, 78%. M.p.: 120–122 °C. *Anal.* Calc. for C₅H₁₁N₃SGe: C, 27.57; H, 5.02; N, 19.29. Found: C, 27.40; H, 5.06; N, 19.05%. IR (KBr, cm⁻¹): 3251 (br, N–H), 3120 (w, C–H), 1608 (m, C=C), 1595 (C–N), 1445 (m, N=N), 1160, 1073 (m, C–S), 587, 505 (m, Ge–C), 455 (m, Ge–S). ¹H NMR (CDCl₃): δ 10.8 (br, 1H, N–H), 1.71 (s, 9H, Ge–CH₃), 7.29 (s, 1H, =CH).

2.2.6. $(C_2H_5)_3Ge(SC_2H_2N_3)$ (6)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), triethylgermanium chloride (0.195 g, 1 mmol) and benzene (20 ml). The yellow crystal complex **6** was obtained from hexane–dichloromethane. Yield, 79%. M.p.: 119–121 °C. *Anal.* Calc. for C₈H₁₇N₃SGe: C, 36.97; H, 6.60; N, 16.17. Found: C, 36.94; H, 6.57; N, 16.19%. IR (KBr, cm⁻¹): 3252 (br, N–H), 3121 (w, C–H), 1608 (m, C=C), 1596 (C–N), 1443 (m, N=N), 1161, 1073 (m, C–S), 579, 516 (s, Ge–C), 457 (m, Ge–S). ¹H NMR (90 MHz, CDCl₃): δ 10.8 (br, 1H, N–H), 1.76 (q, 6H, Ge–CH₂), 0.88 (t, 9H, CH₃), 7.30 (s, 1H, =CH).

2.2.7. $(n-C_4H_9)_3Ge(SC_2H_2N_3)$ (7)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), triphenylgermanium chloride (0.279 g, 1 mmol) and benzene (20 ml). The yellow crystal complex 7 was obtained from hexane–dichloromethane. Yield, 71%. M.p.: 49–51 °C. *Anal.* Calc. for C₁₄H₂₉N₃SGe: C, 48.89; H, 8.50; N, 12.21. Found: C, 48.87; H, 8.53; N, 12.19%. IR (KBr, cm⁻¹): 3250 (br, N–H), 3120 (w, C–H), 1605 (m, C=C), 1592(C–N), 1442 (m, N=N), 1162, 1073 (m, C–S), 585, 531 (m, Ge–C), 461 (m, Ge–S). ¹H NMR (CDCl₃): δ 10.7 (br, 1H, N–H), 1.79 (t, 6H, Ge–CH₂), 1.13–1.50 (m, 12H, CH₂), 0.86 (t, 9H, CH₃), 7.30 (s, 1H, =CH).

2.2.8. $Ph_3Ge(SC_2H_2N_3)$ (8)

The synthesis procedure was the same as in Section 2.2.1, using the sodium salt of 1H-5-mercapto-1,2,3-triazole (0.123 g, 1 mmol), triphenylgermanium chloride (0.339 g, 1 mmol) and benzene (25 ml). The yellow crystal complex **8** was obtained from hexane–dichloromethane. Yield, 73%. M.p.: 130–132 °C. *Anal.* Calc. for C₂₀H₁₇N₃SGe: C, 59.46; H, 4.24; N, 10.40. Found: C, 59.40; H, 4.19; N, 10.36%. IR (KBr, cm⁻¹): 3250 (br, N–H), 3120 (w, C–H), 1608 (m, C=C), 1595 (C–N), 1445 (m, N=N), 1160, 1073 (m, C–S), 591, 535 (m, Ge–C), 453 (m, Ge–S). ¹H NMR (CDCl₃): δ 11.2 (br, 1H, N–H), 7.33–7.74 (m, 15H, Ph–H), 7.30 (s, 1H, =CH).

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. The φ/ω , scan technique was employed. Corrections were applied for Lorentz and polarization effects but not for absorption, satisfying $I \ge \sigma(I)$. 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 K α 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 was as in the following Scheme 1:

3.2. IR spectroscopic studies of the complexes 1-8

The assignment of the IR bands of all the complexes 1–8 have been made by a comparison with the IR

spectra of related organotin(IV) and organogermanium(IV) compounds. A new absorption band that appears at about 309–316 cm⁻¹ for complexes 1–4 can be attributed to the vibration of the Sn–S bond, which is consistent with that detected for a number of organotin(IV)–sulfur derivatives [12], and the absorption band that appears at 453–461 cm⁻¹ for complexes **5–8** can be attributed to the vibrations of the Ge–S bond according to the literature [13].

In complexes 1–4, two strong or medium absorptions are observed at 560–565 and 524–540 cm⁻¹. They are due to v_{asym} and v_{sym} Sn–C stretching vibrations, respectively. In complexes 5–8, v_{asym} and v_{sym} Ge–C stretching vibrations are at 579–591 and 505–535 cm⁻¹, respectively. This suggests that there are no-planar SnC₃ fragments for all the complexes 1–8.

The middle intensity bands observed at 1592–1596 cm⁻¹ in the spectra of all the complexes **1–8** have been assigned to v(C-N), according to the literature [14], together with the absorption region ranging from 1442 to 1445 cm⁻¹ attributed to the vibration of v(N=N), which are all consistent with the free ligand. In the 2800–2600 cm⁻¹ region the ligands exhibit a broad band typical of N–H stretching. In the organotin(IV) and organogermanium(IV) adducts this shifts to 3250 cm⁻¹. The position and character of the broadened N–H stretching band are consistent with the presence of a NH···N intermolecular hydrogen bond, as reported in the literature [15].

All the above data indicate the coordination of the ligand to tin or germanium only by the sulfur atom in all the complexes 1-8. So it can be concluded that complexes 1-8 are four-coordinate, with a sulfur atom and three alkyl (or phenyl) groups bonded to the central metal atom. The conclusion well coincides with the following X-ray crystallographic analysis of complexes 4 and 8.

3.3. ¹H NMR data of the complexes 1-8

The ¹HNMR spectra show that the chemical shifts of the proton on the phenyl group $(Sn-C_6H_6)$ 7.31–7.96 ppm for **4** and $(Ge-C_6H_6)$ 7.30–7.74 ppm for **8** and those of the methylene connected directly with tin in complexes **1–3** and with germanium in complexes **5–7**,



M=Sn, R=CH₃, 1; C₂H₅, 2; n-C₄H₉, 3; C₆H₅, 4; M=Ge, R=CH₃, 5; C₂H₅, 6; C₄H₉, 7; C₆H₅, 8

1.53–1.79 ppm, upfield shift when compared with those of their corresponding precursors. All these data are similar to those cases that appear in the literature [16], indicating there may exist coordination of the ligand to the metal atom for all the complexes. In addition, the broad resonance of the (N–H) proton appears at 10.5-11.2 ppm for all the complexes 1-8.

3.4. Crystal structures of complexes 4 and 8

The structure details of complexes 4 and 8 are shown in Figs. 1 and 2, respectively. The intermolecular hydrogen-bond packing of complexes 4 and 8 are similar, one of which is displayed in Fig. 3. Single crystal X-ray diffraction study details are shown in Table 1, and selected bond lengths and angles are listed in Table 2. The complexes 4 and 8 exhibit the tin(IV) and the germanium(IV) in a distorted tetrahedral geometry defined by three phenyl groups and one sulfur atom, which exists as a bridge between the tin or the germanium atom and the triazole ring.

For complex 4, Sn–C bond lengths (2.121(5) to 2.135(5) Å) are comparable with the complex: triphenyl(5-mercapto-1-phen-1,2,3,4-tetralato)tin (2.120– 2.127 Å) [17]. The Sn–S distance is 2.4354(15) Å, which is consistent with the sum of the covalent radii (2.42 Å) [18] and the complex [Cy₂NeophylSnS₂P(OC₆H₄[']Bu-4)₂] (2.477(3) Å) [19], and this distance is much shorter than the sum of the van der Waals' radii (4.0 Å) [18]. The S–Sn–C bond angles are more acute and the C–Sn– C bond angles are larger than the theoretical tetrahedral angle, the largest deviations occurring in S(1)–Sn(1)– C(15) [101.32(14)°] and C(15)–Sn(1)–C(9) [116.12(17)°].

For complex 8, the germanium atom environment is similar to that of the tin atom in complex 4. The Ge–C bond lengths (1.925(2)-1.929(3) Å) are in the normal range (1.93 Å for Ge-Cp) [20]. The Ge–S bond length (2.2406(11) Å) is a little longer than that reported in



Fig. 2. SHELX view of complex 8 (30% probability thermal ellipsoids).

dichloro-(ferrocene-1,1'-dithiolato)-germanium (2.19 Å) [20], and is consistent with that in complex 4-CH₃C₆H₅CS₂GePh₃ (2.2526(8) Å) [21]. The angles around the center Ge element range from 103.00° to 113.40°, which all depart from the theoretical tetrahedral angle.

Furthermore, the salient feature of the superamolecular structure of complexes **4** and **8** is that of a one-dimensional hydrogen-bonded polymer, as shown in Fig. 3, in which N(3) of the triazole bonds to N(1) of a neighboring triazole through a hydrogen bond $[H(1)\cdots N(1)^i 1.966 \text{ Å}, N(3)-H(1)\cdots N(1)^i 173.93^\circ \text{ for } 4);$ H(1)…N(1)ⁱ 1.880 Å, N(3)-H(1)…N(1)ⁱ 165.56° for **8**, (i) X + 1/2, -Y + 1/2, -Z], the parameters of which are consistent with that reported, H(1)…N(1) 1.92(3) Å, for the complex [(C₆H₅)₃SnSCNH₂=N-N=C(CH₃)(C₆H₄-2-OH)] [22].



Fig. 1. SHELX view of complex 4 (30% probability thermal ellipsoids).



Fig. 3. A drawing showing the intermolecular hydrogen-bonds in complex 8 (the intermolecular hydrogen-bonds in complex 4 are similar to complex 8).

Table 1		
Crystal data and	refinement details for complexes 4 and 8	

Data and details	4	8
Empirical formula	$C_{20}H_{17}N_3SSn$	$C_{20}H_{17}N_3SGe$
Formula weight	450.12	404.02
Temperature (K)	298(2)	298(2)
Wavelength (nm)	0.71073	0.71073
Crystal system	monoclinic	orthorhombic
Space group	$P2_{1}/c$	P2(1)2(1)2(1)
Unit cell dimensions		
a (Å)	9.539(2)	9.184(4)
b (Å)	21.892 (5)	9.657(4)
<i>c</i> (Å)	9.441(2)	21.283(8)
β (°)	93.846 (4)	90
$V(Å^3)$	1967.1(8)	1887.7(12)
Ζ	4	4
D_{calc} (Mg/m ³)	1.520	1.422
Absorption coefficient (mm ⁻¹)	1.412	1.740
Maximum and minimum transmission factors	0.8162 and 0.6768	0.7803 and 0.6059
F(000)	896	824
Crystal size (mm)	0.30 imes 0.20 imes 0.15	0.32 imes 0.18 imes 0.15
θ Data collection (°)	1.86–26.44	1.91–26.37
Index ranges	$-11 \leqslant h \leqslant 11, \ -15 \leqslant k \leqslant 27,$	$-10 \leqslant h \leqslant 11, \ -12 \leqslant k \leqslant 11,$
	$-11 \leq l \leq 11$	$-21 \leq l \leq 26$
Reflections collected	11458	10994
Independent reflections	4034	3842
R _{int}	0.0543	0.0382
Completeness to $\theta = 26.37$	99.7%	99.5%
Refinement method	full-matrix least-squares on F^2	full-matrix least-squares on F^2
Absorption correction	semi-empirical from equivalents	semi-empirical from equivalents
Number of parameters	294	294
Goodness-of-fit on F^2	0.903	0.857
Final <i>R</i> indices $[I > 2.0\sigma(I)]$	$R_1 = 0.0409, wR_2 = 0.0726$	$R_1 = 0.0293, wR_2 = 0.0467$
<i>R</i> indices (all data)	$R_1 = 0.0881, wR_2 = 0.0859$	$R_1 = 0.0455, wR_2 = 0.0495$
Largest difference peak and hole $(e A^{-3})$	0.584 and -0.724	0.289 and -0.223

4. Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this paper have been de-

posited with the Cambridge Crystallographic Data Center as supplementary publication Nos. CCDC-192514 and CCDC-207060. Copies of the data can be obtained free of charge on application to the Director,

Table 2 Selected bond lengths (Å) and angles (°) for complexes ${\bf 4}$ and ${\bf 8}$

4		8	
Bond lengths			
Sn(1)-S(1)	2.4354(15)	Ge(1)-S(1)	2.2406(11)
Sn(1)–C(8)	2.121(5)	Ge(1)–C(3)	1.926(2)
Sn(1)–C(9)	2.135(5)	Ge(1)–C(9)	1.925(3)
Sn(1)–C(15)	2.133(4)	Ge(1)–C(15)	1.929(3)
S(1)–C(2)	1.746(4)	S(1)–C(2)	1.743(3)
N(1)–N(2)	1.316(5)	N(1)–N(2)	1.314(3)
N(2)–N(3)	1.329(5)	N(2)–N(3)	1.321(3)
N(1)–C(2)	1.348(5)	N(1)-C(2)	1.347(3)
N(3)–C(1)	1.317(6)	N(3)–C(1)	1.316(4)
N(3)–H(1)	0.858	N(3)–H(1)	0.985
$N(1)^i \cdots H$	1.966	$N(1)^i \cdots H$	1.880
Bond angles			
C(8) - Sn(1) - S(1)	110.20(13)	C(3)-Ge(1)-S(1)	109.00(8)
C(9)-Sn(1)-S(1)	107.11(15)	C(9)-Ge(1)-S(1)	110.08(11)
C(15)-Sn(1)-S(1)	101.32(14)	C(15)-Ge(1)-S(1)	103.00(10)
C(8) - Sn(1) - C(15)	113.45(19)	C(3)-Ge(1)-C(9)	108.70(13)
C(8)–Sn(1)–C(9)	108.2(2)	C(15)-Ge(1)-C(9)	112.49(14)
C(9)-Sn(1)-C(15)	116.12(17)	C(3)-Ge(1)-C(15)	113.40(11)
$N(3)-H(1)\cdots N(1)^{I}$	173.93	$N(3)-H(1)\cdots N(1)^{i}$	165.56
C(1)-S(1)-Sn(1)	98.83(16)	C(1)-S(1)-Sn(1)	99.79(10)
N(2)–N(1)–C(2)	109.7(4)	N(2)-N(1)-C(1)	108.8(2)
N(1)–N(2)–N(3)	105.7(4)	N(1)–N(2)–N(3)	106.4(2)
C(1)–N(3)–N(2)	112.0(4)	C(2)–N(3)–N(2)	111.4(3)
N(1)-C(1)-S(1)	122.2(3)	N(1)-C(1)-S(1)	122.3(2)
C(1)-C(2)-S(1)	130.6(4)	C(2)-C(1)-S(1)	129.8(2)
N(3)-C(1)-C(2)	105.4(5)	N(3)-C(2)-C(1)	105.8(3)
N(1)-C(2)-C(1)	107.2(4)	N(1)-C(1)-C(2)	107.6(3)

CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.ck).

Acknowledgements

This work was supported by the Natural Science Foundation of Shandong Province, and the Key Teachers Foundation from the State Education Ministry of China (2050).

References

- E.I. Stiefel, K. Matsumoto, in: Transtion Metal Sulfur Chemistry, ASC Symposium Series, 653, American Chemistry Society, Washington, DC, 1995, p. 2.
- [2] E.S. Rasper, Coord. Chem. Rev. 165 (1997) 475.
- [3] B. Krebs, G. Henkel, Angew. Chem., Int. Ed. Engl. 30 (1991) 769.
- [4] C.J. Jones, Chem. Soc. Rev. 27 (1998) 289.
- [5] I. Awad, A.A. Abdel-Rahman, E. Bakite, J. Chem. Technol. Biotechnol. 51 (1991) 483.
- [6] R. Cea-Olivares, O. Jimenez-Sandoval, G. Espinosa-Perez, C. Silvestru, J. Organomet. Chem. 484 (1994) 33.
- [7] R. Cea-Olivares, O. Jimenez-Sandoval, G. Espinosa-Perez, C. Silvestru, Polyhedron 13 (1994) 2809.

- [8] S. Bhandari, M.F. Mahon, J.G. McGinley, K.C. Molly, C.E. Roper, J. Chem. Soc., Dalton Trans. (1998) 3425.
- [9] C.L. Ma, F. Li, D.Q. Wang, H.D. Yin, J. Organomet. Chem. 667 (2003) 5.
- [10] C.L. Ma, F. Li, Chin. J. Chem. 21 (2003) 146.
- [11] H.D. Yin, R.F. Zhang, L.Y. Zhang, C.L. Ma, ACH-Models Chem. 137 (2000) 103.
- [12] C.V.R Moura, A.P.G. de Sousa, R.M. Silva, A.R. Abras, M. Horner, A.J. Bortoluzzi, C.A.L. Filgueiras, J.L. Wardell, Polyhedron 18 (1999) 2961.
- [13] R.A. Chadha, J.E. Drake, A.B. Sarkar, Inorg. Chem. 25 (1986) 2201.
- [14] A. Růžička, A. Lyčka, R. Jambor, P. Novák, L. Čísařová, M. Holčapek, M. Erben, J. Holeček, Appl. Organomet. Chem. 17 (2003) 168.
- [15] A. Tarassoli, T. Sedaghat, Inorg. Chim. Commun. 2 (1999) 595.
- [16] Y. Ye, Q. Zeng, L.Z. Liu, Synth. Commun. 31 (2001) 2373.
- [17] J. Bravo, M.B. Cordero, J.S. Casas, A. Sanchez, S. Sordo, E.E. Castellano, J. Zukerman-Schpector, J. Organomet. Chem. 482 (1994) 147.
- [18] J.E. Huheey, Inorganic Chemistry, third ed., Harper Int, Cambridge, 1983.
- [19] F. Yuan, Y.Q. Huang, Q.L. Xie, Appl. Organomet. Chem. 16 (2002) 660.
- [20] S. Zurcher, V. Gramlich, A. Togni, Inorg. Chim. Acta 291 (1999) 355.
- [21] S. Kato, K. Tani, N. Kitaoka, K. Yanada, H. Mifune, J. Organomet. Chem. 611 (2000) 190.
- [22] S.W. Ng, V.G. Kumar Das, J. Organomet. Chem. 377 (1989) 211.