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# Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information: http://www.tandfonline.com/loi/gpss20

Synthesis and Characterization of Zinc(II) Complexes with 3,4-Dimethoxybenzaldehyde Thiosemicarbazone: The Crystal Structure of [Zn(34-MBTSC)<sub>2</sub>Cl<sub>2</sub>]

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To cite this article: Aliakbar Dehno Khalaji , Gholamhossein Grivani , Morteza Rezaei , Karla Fejfarova & Michal Dusek (2013) Synthesis and Characterization of Zinc(II) Complexes with 3,4-Dimethoxybenzaldehyde Thiosemicarbazone: The Crystal Structure of [Zn(34-MBTSC)<sub>2</sub>Cl<sub>2</sub>], Phosphorus, Sulfur, and Silicon and the Related Elements, 188:8, 1119-1126, DOI: <u>10.1080/10426507.2012.738736</u>

To link to this article: <u>http://dx.doi.org/10.1080/10426507.2012.738736</u>

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Phosphorus, Sulfur, and Silicon, 188:1119–1126, 2013 Copyright © Taylor & Francis Group, LLC ISSN: 1042-6507 print / 1563-5325 online DOI: 10.1080/10426507.2012.738736

### SYNTHESIS AND CHARACTERIZATION OF ZINC(II) COMPLEXES WITH 3,4-DIMETHOXYBENZALDEHYDE THIOSEMICARBAZONE: THE CRYSTAL STRUCTURE OF [ZN(34-MBTSC)<sub>2</sub>CL<sub>2</sub>]

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#### **GRAPHICAL ABSTRACT**



**Abstract** The synthesis and characterization of three zinc(II) complexes  $[Zn(34-MBTSC)_2Cl_2]$ (1),  $[Zn(34-MBTSC)_2Br_2]$  (2), and  $[Zn(34-MBTSC)_2I_2]$  (3) of 3,4-dimethoxybenzaldehyde thiosemicarbazone (34-MBTSC) are reported. Elemental analysis (CHN) and spectral (FT-IR and <sup>1</sup>H-NMR) measurements have been used to characterize the complexes. In addition, the structure of the complex 1 has been determined by X-ray diffraction methods. In these complexes, the zinc(II) ion is bonded to two thioketonic sulfur donors in  $\eta^1$ -S bonding mode and to two halide ions in tetrahedral geometry.

Supplementary materials are available for this article. Go to the publisher's online edition of Phosphorus, Sulfer, and Silicon and the Related Elements for the following free supplemental files: Additional figures.

Keywords Zinc(II) complexes; thiosemicarbazone; X-ray diffraction; tetrahedral

#### INTRODUCTION

Thiosemicarbazone N,S donor ligands and their transition metal complexes have been studied because of their fungicidal, bactericidal, antiviral and antitumoral activities,<sup>1–6</sup>

We acknowledge Golestan University (GU) and Damghan University (DU) for partial support of this work, the institutional research plan No. AVOZ10100521 of the Institute of Physics and the project Pramium Academiae of the Academy of Sciences of the Czech Republic.

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Received 10 July 2012; accepted 8 October 2012.



Scheme 1 Chemical structure of zinc(II) complexes  $[Zn(34-MBTSC)_2 \times_2]$  (X = Cl (1), X = Br (2) and X = I (3)).

and interesting chemical and structural properties.<sup>7–12</sup> The biological activity of these compounds is strongly dependent on the nature of the heteroaromatic ring and the position of substituent to the ring, as well as the form of the thiosemicarbazone moiety.<sup>13</sup> They have been used for metal analyses;<sup>14</sup> and for device applications related to optical computing, storage, and information processing.<sup>15</sup> Thiosemicarbazones have a propensity to react with a wide range of transition metal ions.<sup>7–12,16–21</sup> They can bind to a metal ion via a variety of coordination modes in their neutral and anionic form.<sup>7–12,16–21</sup> They usually act as chelating N,S ligands with transition metal ions such as Zn(II), Hg(II), Ni(II), Co(II), Cu(II), and Cd(II), bonding through the sulfur and hydrazine nitrogen atoms<sup>16–19</sup> and as  $\eta^1$ -S bonding mode with Cu(I) and Ag(I) ions.<sup>7,8,20,21</sup> Only a few reports exist on  $\eta^1$ –S bonding mode of thiosemicarbazone ligands with Zn(II) ion.<sup>22,23</sup>

We describe here three zinc(II) complexes obtained from 3,4-dimethoxybenzaldehyde thiosemicarbazone (34-MBTSC) as a ligand (Scheme 1).

#### **RESULTS AND DISCUSSION**

The ligand 34-MBTSC was prepared by condensation of thiosemicarbazide with 3,4-dimethoxybenzaldehyde following the literature method.<sup>24</sup> The reaction of ZnX<sub>2</sub> with 34-MBTSC in ethanol-acetonitrile solvent mixture at r.t. results in monomeric zinc(II) complexes as shown in Scheme 1. The solubility of thiosemicarbazone ligand 34-MBTSC and its zinc(II) complexes was determined by adding the solvent to a known amount of compound until complete dissolution. It was found that all compounds are moderately soluble in common organic solvents, insoluble in water, and completely soluble in DMF and DMSO. In order to confirm the chemical composition of the synthesized complexes, CHN, XRD, FT-IR, and <sup>1</sup>H-NMR analyses were carried out with results are presented in the experimental section.

#### FT-IR Spectra

The main infrared vibration bands are reported in the experimental section. The presence of the  $\nu$ (N–H) band at 3181 cm<sup>-1</sup> shows that neutral thio-ligand is coordinated to zinc(II) metal center through thione sulfur.<sup>19,23,25</sup> In addition, the spectrum of the ligand shows a strong band at 1643 cm<sup>-1</sup> due to the –C=N– stretching vibration.<sup>19,23</sup>

In complexes, the  $\nu$ (C=N) vibration band shifted to lower wavenumbers, whereas  $\nu$ (N–H) vibration bands shifted to higher wavenumbers. The two bands corresponding to  $\nu$ (C=S) in complexes are significantly modified when compared to the free ligand. In

addition, the spectrum of the zinc(II) complexes **1–3** shows the vibration band  $\nu$ (Zn–S) at 412 cm<sup>-1</sup> (**1**), 411 cm<sup>-1</sup> (**2**), and 409 cm<sup>-1</sup> (**3**), which corresponds to the coordination bonds.<sup>19,23,29</sup>

#### <sup>1</sup>H-NMR Spectra

The <sup>1</sup>H-NMR spectral data of the ligand 34-MBTSC and its zinc(II) complexes were recorded in DMSO-d<sup>6</sup> with tetramethylsilane as an internal standard and are reported in the experimental section. The <sup>1</sup>H-NMR spectra of zinc(II) complexes suggest that they have similar structure (Figure 1), they also agree with the thione tautomer as indicated on the basis of FT-IR data. However, they are slightly modified, with some multiplicity of the signals and some small changes in the chemical shifts with respect to the free ligand.<sup>23</sup> Spectra for 1, 2, and 3 can be found in the Supplemental Materials (Figure S5)

#### **Crystal and Molecular Structure of 1**

An ORTEP view of  $[Zn(34-MBTSC)_2Cl_2]$  (1) with the atoms numbering scheme is given in Figure 2. Table 1 lists the selected bond lengths and angles. In the complex  $[Zn(34-MBTSC)_2Cl_2]$  (1), the thiosemicarbazone ligand 34-MBTSC is neutral and Smonodentate. Zinc(II) is coordinated by two chlorine ions and the thione sulfur atoms of two independent ligands. The two ligands are in Z position relative to C8–N2 and C8<sup>i</sup>–N2<sup>i</sup> bonds, respectively. The tetrahedral geometry around the zinc(II) ion in 1 is distorted because of the different bond distances and angles. The greatest angular distortion from an ideal tetrahedral geometry (109.5 °) occurs in the angles C11–Zn1–Cl1<sup>i</sup> [120.59(2) °] and



**Figure 1** <sup>1</sup>H-NMR spectrum of zinc(II) complex [Zn(34-MBTSC)<sub>2</sub> $\times_2$ ] (1).



**Figure 2** An ORTEP view of **1**, showing 50% probability displacement ellipsoids and the atom-numbering. Dashed lines indicate intramolecular N–H···S and N–H···Cl hydrogen bonds. Symmetry codes: (i) –x, y, –z+1/2.

Cl1–Zn1–S1 [98.404(17) °]. The Zn–S and Zn–Cl distances (Table 1) are similar to those found in other zinc(II) complexes with thiol sulfur atoms.<sup>22,23</sup> Additional representations of the structure are shown in the Supplemental Materials (Figures S 2 and S 3).

The length of the N2–C8 bond is 1.329(2) Å, the C7–N1 bond is 1.280(3) Å, and the N1–N2 bond is 1.384(2) Å. These distances are typical of a C–N single bond, C=N double bond, and N–N single bond, respectively. In addition, the bond length of the C8–S1 bond is 1.7209(19) Å, which is in between a single and a double bond as it has been already observed in the cases of the zinc(II) complexes with similar ligands.<sup>22,23</sup> The molecules of the two ligands are planar and the angle between the planes of the two phenyl rings is 87.969(50) °.

Complex 1 shows an intramolecular N–H $\cdots$ S and N–H $\cdots$ Cl hydrogen bonding via hydrogen atoms of NH<sub>2</sub> with chlorine and sulfur atoms (Figure 2, Table 2), while in the

Table 1 Selected bond distances (Å) and angles (°) of 1

Zn1–Cl1	2.2594(5)	Zn1–S1	2.3461(5)
N1-N2	1.384(2)	N2-C8	1.329(2)
N1-C7	1.280(3)	N3-C8	1.317(3)
S1–C8	1.7209(19)		
Cl1–Zn1–Cl1 <sup>i</sup>	120.59(2)	Cl1 <sup>i</sup> –Zn1–S1	114.592(17)
Cl1–Zn1–S1	98.404(17)	Cl1 <sup>i</sup> –Zn1–S1 <sup>i</sup>	98.404(17)
Cl1–Zn1–S1 <sup>i</sup>	114.592(17)	S1-Zn1-S1 <sup>i</sup>	110.87(2)
N2-N1-C7	114.92(16)	S1-C8-N2	116.14(14)
N1-N2-C8	119.11(16)	S1-C8-N3	124.27(14)
N1-C7-C1	121.03(18)	N2-C8-N3	119.59(17)

Symmetry codes: (i) -x, y, -z+1/2.

	Distance, Å			
D–H···A	D-H	Н…А	D····A	D–H…A, deg
N3–H3b…S1	0.817	3.168	3.717	126.947
N3-H3b···Cl1	0.817	2.964	3.687	148.834
C6-H6S1	0.960	2.943	3.840	156.038
N2–H2n···O2	0.866	2.085	2.941	169.694
С7–Н7…О2	0.960	2.724	3.496	137.873

 Table 2 Geometric parameters of hydrogen bond for 1

crystal packing of 1, the monomer units are linked to each other via several classical and non-classical intermolecular hydrogen bonds (N–H…O, C–H…O and C–H…S) (Table 2).

#### **EXPERIMENTAL**

All organic solvents used are commercially available and used as received without further purifications. The 3,4-dimethoxybenzaldehyde, thiosemicarbazide, ZnI<sub>2</sub>, ZnBr<sub>2</sub>, and ZnCl<sub>2</sub> were purchased from Merck Chemical Company. Fourier Transform Infrared spectra were recorded as a KBr disk on a FT-IR Perkin–Elmer spectrophotometer. Elemental analyses were carried out using a Heraeus CHN-O-Rapid analyzer. <sup>1</sup>H NMR spectra were measured on a BRUKER DRX-500 AVANCE spectrometer at 500 MHz for the Schiffbase ligands and its complexes. All chemical shifts are reported in  $\delta$  units downfield from TMS. X-ray powder diffraction pattern of the complex was recorded on a Bruker AXS

Empirical formula	$C_{20}H_{26}6_2Cl_2O_4S_2Zn$
Formula weight	614.9
Temperature	150
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 19.0691(3)  Å
	a = 9.9920(2)  Å
	a = 14.2504(2)  Å
	$\beta = 91.7482(13)^{\underline{0}}$
Volume, Å <sup>3</sup>	2717.83(8)
Calculated density, Z	4, 1.5022 Mg/m <sup>3</sup>
Absorption coefficient	$4.85 \text{ mm}^{-1}$
F(000)	1264
Theta range for data collection	4.65–63.67 <sup>o</sup>
Limiting indices	$-21 \le h \le 21$
	$-11 \le k \le 11$
	$-16 \le l \le 16$
Reflections collected/unique	$33279 / 2144 [R_{int} = 1.84]$
Refinement method	
Data/restraints/parameters	2236/3/168
Goodness-of-fit on $F^2$	1.84
Final <i>R</i> -indices $[I > 3\sigma(I)]$	$R_1 = 0.0293, wR_2 = 0.1032$
<i>R</i> -indices (all data)	$R_1 = 0.0304, wR_2 = 0.1046$
Largest diff. peak and hole	0.71 and $-0.33 \text{ e}\text{\AA}^{-3}$

 Table 3 Crystallographic data and structure refinement for 1

diffractometer D8 ADVANCE with Cu-K $\alpha$  radiation with nickel beta filter in the range  $2\theta = 10-80^{\circ}$ .

#### **Preparation of 34-MBTSC**

The ligand 34-MBTSC (Figure S 1, Supplemental Materials) was obtained from 3,4-dimethoxybenzaldehyde and thiosemicarbazide (1:1 molar ratio) in ethanol (10 mL) following the literature method.<sup>24</sup> Yield: 83%. Colorless crystals. Anal. Calc. for  $C_{10}H_{13}N_3O_2S$ : C, 50.20; H, 5.48; N, 17.56%. Found: C, 50.11; H, 5.35; N, 17.56%. IR (KBr pellet, cm<sup>-1</sup>): 3352, 3262, and 3181 (N–H), 1643, 1618, 1602, 1576 (C=N, C=C), 1161, 753 (C=S), 952 (N–N). <sup>1</sup>H-NMR (DMSO- $d_6$ ,  $\delta$ (ppm)): 3.76 (s, 3H<sub>7</sub>), 3.78 (s, 3H<sub>8</sub>), 6.93 (d, J = 8.55 Hz, H<sub>6</sub>), 7.11 (dd, H<sub>5</sub>), 7.50 (d, J = 1.35 Hz, H<sub>9</sub>), 7.94 (s, H<sub>1</sub>), 8.0 (s, H<sub>2</sub>), 8.14 (s, H<sub>4</sub>), 11.30 (s, H<sub>3</sub>).

#### Preparation of [Zn(34-MBTSC)<sub>2</sub>Cl<sub>2</sub>] (1)

To a stirring ethanolic (5 mL) solution of the 34-MBTSC ligand (0.408 g, 0.2 mmol) was added ZnCl<sub>2</sub> (0.028 g, 0.2 mmol) in acetonitrile (10 mL) and the mixture was stirred for 10 min in air at r.t. and was then left at r.t. for several days without disturbance yielding suitable crystals of **1** that subsequently were filtered off and washed with Et<sub>2</sub>O. Yield: 72%. Colorless crystals. Anal. Calc. for  $C_{20}H_{26}N_6Cl_2O_4S_2Zn$ : C, 39.07; H, 4.26; N, 13.67%. Found: C, 38.98; H, 4.11; N, 13.64%. IR (KBr pellet, cm<sup>-1</sup>): 3438, 3317, and 3217 (N–H), 1611, 1597, 1579, 1561 (C=N, C=C), 1165, 751 (C=S), 956 (N–N), 412 (Zn–S). <sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>,  $\delta$ (ppm)): 3.76 (s, 3**H**<sub>7</sub>), 3.79 (s, 3**H**<sub>8</sub>), 6.93 (d, J = 8.42 Hz, **H**<sub>6</sub>), 7.11 (dd, **H**<sub>5</sub>), 7.50 (d, J = 1.29 Hz, **H**<sub>9</sub>), 7.96 (s, **H**<sub>1</sub>), 8.02 (s, **H**<sub>2</sub>), 8.15 (s, **H**<sub>4</sub>), 11.32 (s, **H**<sub>3</sub>).

#### Preparation of [Zn(34-MBTSC)<sub>2</sub>Br<sub>2</sub>] (2)

This complex was prepared in a similar way as **1** using ZnBr<sub>2</sub>. Yield: 71%. Colorless crystals. Anal. Calc. for C<sub>20</sub>H<sub>26</sub>N<sub>6</sub>Br<sub>2</sub>O<sub>4</sub>S<sub>2</sub>Zn: C, 34.13; H, 3.72; N, 11.94%. Found: C, 33.96; H, 3.59; N, 12.03%. IR (KBr pellet, cm<sup>-1</sup>): 3439, 3316, and 3218 (N–H), 1610, 1595, 1580, 1557 (C=N, C=C), 1164, 751 (C=S), 955 (N–N), 411 (Zn–S). <sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>,  $\delta$ (ppm)): 3.77 (s, 3H<sub>7</sub>), 3.80 (s, 3H<sub>8</sub>), 6.94 (d, J = 8.48 Hz, H<sub>6</sub>), 7.12 (dd, H<sub>5</sub>), 7.55 (d, J = 1.38 Hz, H<sub>9</sub>), 7.95 (s, H<sub>1</sub>), 8.01 (s, H<sub>2</sub>), 8.15 (s, H<sub>4</sub>), 11.31 (s, H<sub>3</sub>).

#### Preparation of [Zn(34-MBTSC)<sub>2</sub>I<sub>2</sub>] (3)

This complex was prepared in a similar way as **1** using ZnI<sub>2</sub>. Yield: 68%. Colorless crystals. Anal. Calc. for  $C_{20}H_{26}N_6I_2O_4S_2Zn$ : C, 30.11; H, 3.28; N, 10.53%. Found: C, 29.99; H, 3.20; N, 10.66%. IR (KBr pellet, cm<sup>-1</sup>): 3431, 3311, and 3224 (N–H), 1605, 1592, 1577, 1547 (C=N, C=C), 1164, 750 (C=S), 955 (N–N), 409 (Zn–S). <sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>,  $\delta$ (ppm)): 3.77 (s, 3H<sub>7</sub>), 3.80 (s, 3H<sub>8</sub>), 6.94 (d, J = 8.51 Hz, H<sub>6</sub>), 7.12 (dd, H<sub>5</sub>), 7.50 (d, J = 1.31 Hz, H<sub>9</sub>), 7.95 (s, H<sub>1</sub>), 8.00 (s, H<sub>2</sub>), 8.14 (s, H<sub>4</sub>), 11.30 (s, H<sub>3</sub>).

#### **X-Ray Structure Determination**

Single crystal of the dimensions  $0.31 \text{ mm} \times 0.22 \text{ mm} \times 0.17 \text{ mm}$  of **1** was chosen for X-ray diffraction study. Crystallographic measurements were done at 150 K with four circle

CCD diffractometer Gemini (Oxford Diffraction) with mirrors-collimated Cu  $K\alpha$  radiation ( $\lambda = 1.54184$  Å), using the area detector atlas. The crystal structures were solved by direct methods with program SIR2002<sup>26</sup> and refined with the Jana2006 program package<sup>27</sup> by full-matrix least-squares technique on  $F^2$ . The molecular structure plots were prepared by ORTEP III.<sup>28</sup> Hydrogen atoms were mostly discernible in difference Fourier maps and could be refined to reasonable geometry. According to common practice they were nevertheless kept in ideal positions during the refinement except the ones bounded to nitrogen. The isotropic atomic displacement parameters of all hydrogen atoms were evaluated as  $1.2U_{eq}$  of the parent atom. Crystallographic data and details of the data collection and structure solution and refinements are listed in Table 3.

#### SUPPLEMENTARY DATA

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Center, CCDC No. 821654 (1). Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336 033, E-mail: deposit@ccdc.cam.ac.uk or website: http://www.ccdc.cam.ac.uk.

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