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Synthesis and Characterization of Binuclear Zn(II) Complex of N_2O_2S Symmetrical Potentially Pentadentate Schiff Base Ligand

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The reaction of 1-chloro-2-nitrobenzene with 2-aminothiophenol yield the 2-((nitrophenyl)thio)aniline and then 2,2'-thiodianiline of this compound was prepared. Then, the symmetrical potentially pentadentate N_2O_2S Schiff base ligand 2,2'-(((thiobis(1,2-phenylene))bis(azanilidene))bis(methenilidene))diphenol (H_2L) was obtained by added salicylaldehyde. These compounds have been characterized by FT-IR, ^{1}H NMR and Mass Spectrometry. Binuclear Zn(II) complex of H_2L was prepared and structurally characterized by X-ray crystallography. The coordination geometry around Zn(II) is intermediate between trigonal bipyramidal and square pyramidal, $\tau = 0.54$.

Keywords: Schiff base complex, Pentadentate ligand, Zn(II) complex, Binuclear complex, N_2O_2S donor

INTRODUCTION

The chemistry of metal complexes with Schiff base ligands containing ONS donors has been studied in order to gain an understanding of the processes such as the redox properties of various metalloenzymes in living systems, catalytic reactions, formation of compounds with unusual properties, metal-metal interactions, mimic the functions of natural vitamins and proteins and so on [1-9]. The complexation sites in living systems are usually N, O, S or C donors coming from histidine, tyrosine, aspartic acid, glutamic acid and cysteine [6,10-12]. Schiff base ligands containing NOS donors are more frequently used in these model compound studies. Due to the presence of hard and soft donors in their structure, these ligands have structure similarities with natural biological systems and they readily coordinate with a wide range of transition metal ions [13,14].

Zinc is an essential metal ion in biological processes and has either a structural or catalytic role in several metalloproteins. Some of the particular properties of the zinc(II) ion include its flexible coordination geometry that can easily be four-, five-, or six-coordinate, fast ligand exchange, Lewis acidity, intermediate polarizability (hard-soft), strong binding to suitable sites [6,10].

Binuclear zinc complexes may serve as model compounds for the active sites of many metalloenzymes and play an essential role in many biological systems [15,16]. The zinc(II) ion is known to have a high affinity towards nitrogen and sulfur donors [17]. The preparation of complexes which contain two metal ions separated by distances of 3.5-6 Å is of considerable interest, at these distance no direct interaction between the metal ions is expected. Yet a substrate could interact simultaneously with both ions [18]. Alkaline phosphatase is dimeric and each monomer has two zinc(II) and one magnesium(II) in its active site. The Zn-Zn distance in this metalloenzyme is 4 Å. Metal-substituted derivatives indicate that one of the zinc(II) is five-coordinate, but another zinc(II) and magnesium are six-coordinate. Five-coordinated zinc(II) is essential for activity, but full catalytic efficiency is reached only when all metal ions are present [19,20].

Recently, several zinc(II) complexes of ligands with NOS coordination have been reported where some of them

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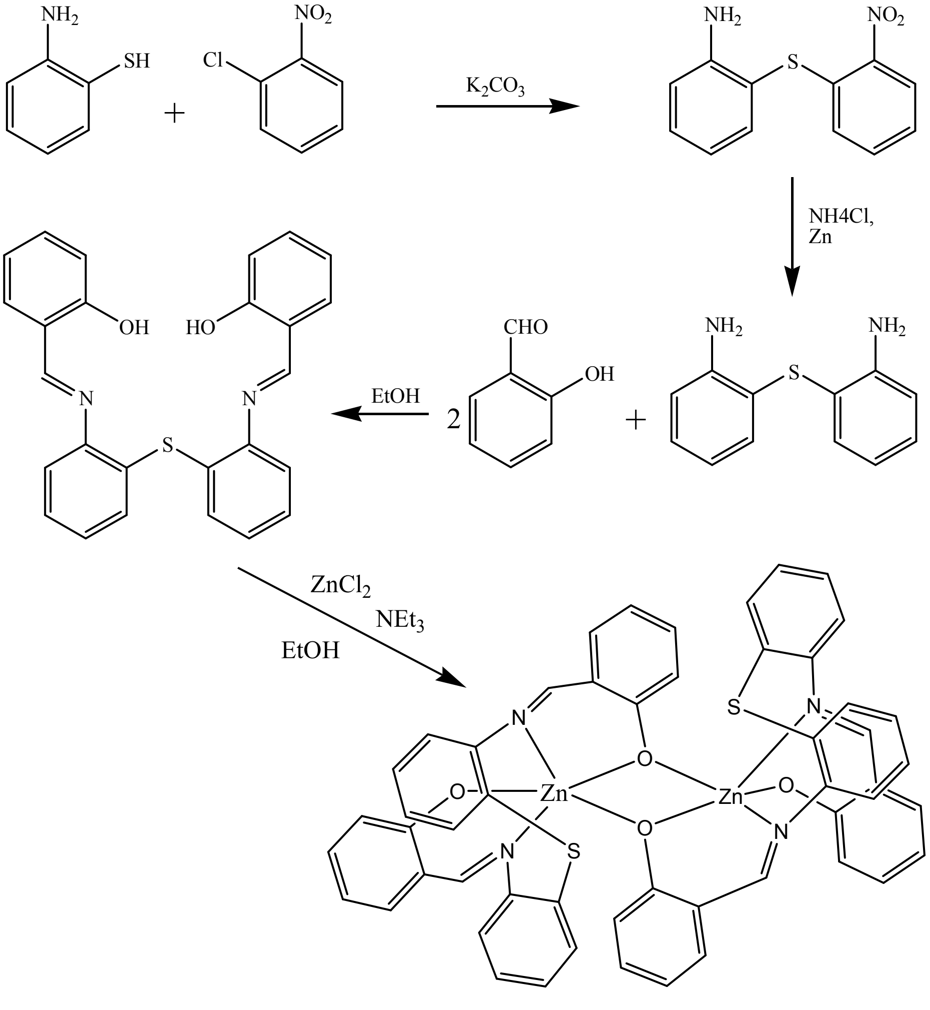

Scheme 1. Synthesis of the ligand (H_2L) and binuclear zinc(II) complex

are thioether Schiff base ligands [4,21,22]. Our group previously reported the synthesis and characterization of zinc(II) complexes derived from symmetric and unsymmetric thioether Schiff base ligands [23,24]. Herein, we present the synthesis and characterization of symmetrical potentially pentadentate N_2O_2S Schiff base ligand obtained by the condensation of 2,2'-thiodianiline and 2-hydroxybenzaldehyde (Scheme 1) [25]. Binuclear zinc(II) complex of this N_2O_2S donor thioether Schiff base ligand has been synthesized. The X-ray structural analysis of zinc(II) complex displayed the presence of two five-coordinate zinc atoms separated by 3.187 Å and the phenolate oxygen atom act as bridging ligand (Fig. 1).

EXPERIMENTAL

Chemical and Starting Materials

2-Aminothiophenol, 1-chloro-2-nitrobenzene, 2-hydroxybenzaldehyde, K_2CO_3, $ZnCl_2$, were commercial products and used without further purification. Other reagents and solvents were of analytical grade and purchased commercially.

Instrumentation

Elemental analyses were performed in a CHNS-O-2400 II Perkin-Elmer. Infrared spectra were recorded in ATR, using a Bruker FT-IR Equinax-55 spectrophotometer (4000-

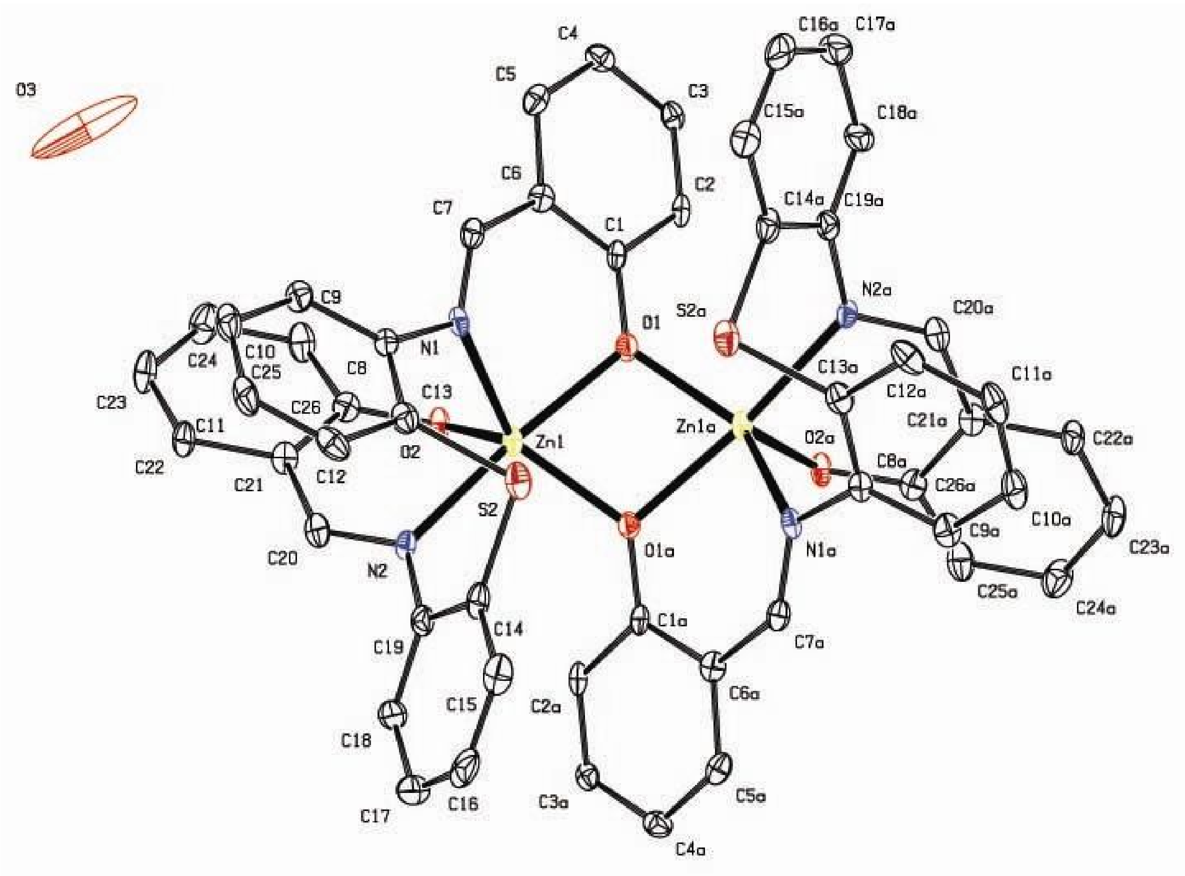

Fig. 1. Crystal structure of $[ZnL]_2.H_2O$ showing 50% displacement ellipsoids.

400 cm^{-1}). MS spectra were obtained using a QP-1100EX Shimadzu GC-MS (EI at 70 eV). Conductivity measurements were carried out in 10^{-3} mol dm^{-3} DMF solutions at 25 °C using a Metrohm 660 conductometer. The electronic spectra were taken in GBC UV-Vis Cintra 101 spectrophotometer with 1 cm quartz, in the range 200-1100 nm at 25 °C. ^{1}H NMR spectra were taken in $CDCl_3$ on a Bruker NMR 400 MHz spectrometer using $Si(CH_3)_4$ as an internal standard.

X-ray Crystal Structure Determination

Single crystal of Zn(II) complex suitable for X-ray analyses was selected from its obtained by open evaporation of ethanolic solution of the reaction mixture at 298 K. The details of the X-ray crystal data and of the structure solution and refinement are given in Table 1. The X-ray diffraction measurements were made on a STOE IPDS-G diffractometer with graphite monochromated Mo Kα radiation (λ = 0.71073 Å). Cell constants and an orientation matrix for data collection were obtained by least-squares refinement of diffraction data from 3898 unique reflections. The structure was solved by direct methods and refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms. The hydrogen atoms were located in their calculated positions and refined using a riding model.

Synthesis of the 2-((Nitrophenyl)thio)aniline

2-Aminothiophenol (5.0 g, 40 mmol), 1-chloro-2-nitrobenzene (6.3 g, 40 mmol) and potassium carbonate (5.5 g, 40 mmol) were mixed and then fused together at 130-140 °C with constant stirring for 4 h. The mixture was then cooled, and absolute ethanol (70 ml) was added. The mixture was stirred for 15 min and then filtered off. The

Table 1. Crystal Data and Structure Refinement for $[ZnL]_2\cdot H_2O$

	$[ZnL]_2\cdot H_2O$
Empirical formula	$C_{52}H_{38}N_4O_5S_2Zn_2$
Formula weight	993.72
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C 2/c
Unit cell dimensions	a = 16.646(3) Å
	b = 15.646(3) Å β = 97.96(3)°
	c = 17.109(3) Å
Volume	4413.0(14) $Å^3$
Z	4
Density (calculated)	1.496 mg m^{-3}
Absorption coefficient	1.237 mm^{-1}
F(000)	2040
Crystal size	0.40 × 0.15 × 0.05 mm^3
θ range for data collection	2.40-25.00°
Limiting indices	$-17 \leq h \leq 19$, $-18 \leq k \leq 18$, $-20 \leq l \leq 20$
Reflections collected/unique	11573/3898 [$R_{(int)}$ = 0.1889]
Completeness to θ =	25.00, 99.9%
Absorption correction	Numerical
Max. and min transmission	0.9407 and 0.6375
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3898/0/294
Goodness-of-fit on F^2	1.042
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0967, wR_2 = 0.1576
R indices (all data)	R_1 = 0.1832, wR_2 = 0.1828
Largest diff. peak and hole	0.849 and -0.407 e $Å^{-3}$

volume of the filtrate was reduced to about 10 ml. The precipitate formed in the concentrated solution was decanted off and washed twice with Et_2O (2 × 10 ml) (Scheme 1). yielded 7.94 g (81%). IR (ATR, cm^{-1}) 3378, 3301 ν(NH_2); 1530, 1301 ν(NO_2); 743 ν(C-S); ^1H NMR ($CDCl_3$, ppm): δH 4.34 (2H, br), 6.62 (1H, t), 6.63 (1H, t), 6.72 (1H, d), 6.73 (1H, d), 7.19 (4H, m); EI-MS (m/z): (248, 198, 154, 124, 93).

Synthesis of the 2,2'-Thiodianiline

The 2,2'-thiodianiline was prepared by reaction of 2-((nitrophenyl)thio)aniline, ammonium chloride and zinc dust similarly to the procedure of Keypour *et al.* [26]. A mixture of 2-((nitrophenyl)thio)aniline (2.5 g, 10 mmol), ammonium chloride (4 g) and water (2 ml) in ethanol 100 ml was heated to boiling and 3 g zinc dust was added. After refluxing for 24 h it was filtered and the volume was reduced to 20 ml. Then excess water was added to the mixture and the pH was adjusted to 12 with potassium hydroxide. The solution was extracted with chloroform (2 × 10 ml). The chloroform extracts were combined and dried over anhydrous sodium sulfate. The chloroform was removed and the product was obtained as an oil (Scheme 1). yielded 1.44 g (67%). IR (ATR, cm^{-1}) 3374, 3292 ν(NH_2); 745 ν(C-S); ^1H NMR ($CDCl_3$, ppm): δH 4.22 (4H, br), 6.70-6.76 (4H, m), 7.13 (2H, t), 7.23 (2H, d); EI-MS (m/z): (216, 198, 182, 124, 93).

Synthesis of the 2,2'-(((Thiobis(1,2-phenylene))bis(azanilidene))bis(methenilidene))diphenol (H_2L)

2,2'-(((Thiobis(1,2-phenylene))bis(azanilidene))bis(methenilidene))diphenol was prepared according to the procedure of Guo *et al.* [25]. A solution of 2,2'-thiodianiline (1.08 g, 5 mmol) in ethanol (30 ml) was added dropwise to a refluxing solution of 2-hydroxybenzaldehyde (1.22 g, 10 mmol) in ethanol (20 ml). After refluxing for 8 h the solution was vacuum evaporated to yield the crude product and rubbed with diethyl ether and n-hexane and the precipitated green powder was collected (Scheme 1). Yield: 1.76 g (83%). IR (ATR, cm^{-1}) 1613 ν(C=N); 751 ν(C-S); 2500-3300 ν(O-H); ^1H NMR ($CDCl_3$, ppm): δH 6.90 (2H, t), 6.99 (2H, d), 7.19-7.26 (6H, m), 7.31-7.35 (6H, m), 8.54 (2H, s), 12.94 (2H, br); EI-MS (m/z): (424, 320, 228, 196, 136, 91). UV-Vis in DMF (λ, nm) 280 (logε = 4.38), 370 (logε = 4.07); Λ_m (DMF) 24.4 Ω^{-1} cm^2 mol^{-1}.

Synthesis of the $[ZnL]_2.H_2O$

A solution of zinc(II) chloride (1 mmol) in ethanol (30 ml) was added dropwise to a refluxing solution of $N_2S_2O_2$ donor thioether Schiff base ligand (H_2L) (0.424 g, 1 mmol) in ethanol (20 ml). After refluxing for 2 h, a solution of triethylamin was added and the reaction mixture was refluxed for 1 h. Binuclear zinc(II) complex and crystal of $[ZnL]_2.H_2O$ suitable for X-ray work was obtained by slow evaporation of ethanolic solution of the complex (Scheme 1). Yield: 377 mg (76%). Anal. Calcd. for $C_{52}H_{38}N_4O_5S_2Zn_2$: C, 62.85; H, 3.85; N, 5.64%. Found: C, 63.46; H, 3.79; N, 5.67%; IR (ATR, cm^{-1}) 1605 ν(C=N); 755 ν(C-S); UV-Vis in DMF (λ, nm) 280 (logε = 4.68), 400 (logε = 4.31); Λ_m (DMF) 3.5 Ω^{-1} cm^2 mol^{-1}.

RESULTS AND DISCUSSION

The IR spectrum for 2-((nitrophenyl)thio)aniline shows bands at 3378 and 3301 cm^{-1} assignable to the aromatic primary amine vibrations and a band at 743 cm^{-1} due to the thioethers stretch. The vibration peaks of NO_2 are observed in 1530 and 1301 cm^{-1}. The IR spectrum for 2,2'-thiodianiline shows bands at 3374 and 3292 cm^{-1} ν(NH_2) and 745 cm^{-1} ν(C-S) and the absence of the vibration peaks of the nitro group of the starting material exhibit that the nitro group converted to the amine group. The IR spectrum for the ligand (H_2L) confirm the presence of imine (1613 cm^{-1}) and the absence of carbonyl and amine functional groups of the starting materials. In this spectrum, a broad band in the 2500-3200 cm^{-1} region is assigned to the ν(O-H) vibration in ligand. The vibration peak of C-S is observed in 751 cm^{-1}. The IR spectrum of binuclear zinc(II) complex of this N_2O_2S donor thioether Schiff base ligand shows band in the 1613 cm^{-1} attributed to ν(C=N) and the vibration peaks of C-S are observed in 751 cm^{-1}. The vibration peak of C-S was not shifted relative to the similar band of the ligand that shows the thioether sulfur atoms is not coordinated to the metal ion.

^1H NMR results, obtained for prepared compounds at ambient temperature in $CDCl_3$, are presented in Section 2. The ^1H NMR spectrum of 2-((nitrophenyl)thio)aniline shows a broad signal at δH 4.34 ppm correspond to the NH_2

Table 2. Selected Bond lengths (Å) and Bond Angels (deg) for $[ZnL]_2•H_2O$

Bond lengths	d (Å)	Bond angels	a (˚)
Zn(1)-N(1)	2.120(7)	O(1)-Zn(1)-O(2)	98.1(2)
Zn(1)-N(2)	2.150(7)	O(1)-Zn(1)-O(1a)	74.6(3)
Zn(1)-O(1)	2.093(6)	O(1)-Zn(1)-N(1)	83.7(3)
Zn(1)-O(1a)	1.993(6)	O(1)-Zn(1)-N(2)	173.5(3)
Zn(1)-O(2)	1.967(6)	O(1a)-Zn(1)-N(1)	141.0(2)
Zn(1)-Zn(1a)	3.187	O(1a)-Zn(1)-O(2)	115.1(2)
S(1)-C(14)	1.771(11)	O(1a)-Zn(1)-N(2)	100.9(3)
S(1)-C(13)	1.794(10)	O(2)-Zn(1)-N(1)	99.5(3)
N(1)-C(7)	1.283(11)	O(2)-Zn(1)-N(2)	88.1(2)
N(1)-C(8)	1.427(11)	N(1)-Zn(1)-N(2)	97.4(3)
N(2)-C(19)	1.397(12)	Zn(1)-O(1)-Zn(1a)	102.5(3)
N(2)-C(20)	1.301(10)	C(7)-N(1)-Zn(1)	123.3(6)
O(1)-C(1)	1.317(10)	C(7)-N(1)-C(8)	118.8(8)
O(2)-C(26)	1.309(11)	C(8)-N(1)-Zn(1)	116.3(6)

protons and shows doublet and triplet signals in the δH 6.62-7.19 ppm regions correspond to the aromatic ring protons. The ^{1}H NMR spectrum of the 2,2'-thiodianiline indicate that this compound has synthesized. The broad signal at δH 4.22 ppm in the ^{1}H NMR spectrum of this diamine is assigned to the NH_2 protons and the aromatic ring protons exhibit doublet and triplet signals at δH 670-7.23 ppm regions. The ^{1}H NMR spectrum of the ligand (H_2L) exhibit a singlet resonance at δH 8.54 ppm, which has been assigned to the azomethine proton (CH=N) and, shows the aromatic ring protons in the region δH 6.90-7.35 ppm. In the ^{1}H NMR spectrum, the broad signal with δH 12.94 ppm is assigned to the OH groups.

The electronic spectrum of the ligand (H_2L) shows two absorption bands at 280 nm (logε = 4.39) and 370 nm (logε = 4.07), that the band at around 370 nm is the $n\rightarrow\pi^*$ transition involving the promotion of one of the lone-pair electrons of the nitrogen atom to the antibonding π orbital associated with the azomethene groups, while the 280 nm band involves a $\pi\rightarrow\pi^*$ transition which includes the π orbital functions of the azomethene groups and benzene rings [27]. The electronic spectrum of the binuclear zinc(II) complex exhibited a band at 280 nm (logε = 4.68, $\pi\rightarrow\pi^*$) and a high intensity band at 400 nm (logε = 4.31) assigned as a metal → ligand charge transfer band.

The EI mass spectra of the 2-((nitrophenyl)thio)aniline, 2,2'-thiodianiline and Schiff bases ligand (H_2L), provide strong evidence for the formation of these compounds. The peaks in the spectra of the compounds are observed at m/z 248, 216, 424 corresponding to molecular weights $[M]^+$, respectively. The four fragment ions observed at m/z 198, 154, 124 and 93 in the spectrum of the 2-((nitrophenyl)thio)aniline, m/z 198, 182, 124 and 93 in the spectrum of the 2,2'-thiodianiline and m/z 320, 228, 196 and 136 in the spectrum of the Schiff bases ligand (H_2L), further supported to the formation of these compounds.

The molar conductance values for both ligand (H_2L) and binuclear zinc(II) complex measured in DMF solution (1 ×

10^{-3} mol dm^{-3}) fall in the range 3.5-24.4 ohm^{-1} cm^{2} mol^{-1}. These observed values of the molar conductance are well within the expected range for non-electrolytes [28]. Also, the elemental analyses of the complex indicated the absence of counter ions in the formula.

Description of the Crystal Structure

Yellow crystals of $[ZnL]_2.H_2O$ for X-ray crystallographic study were obtained by slow evaporation of an aqueous solution at room temperature. The structure of $[ZnL]_2.H_2O$ determined by single crystal X-ray diffraction, is shown in Fig. 1. Selected bond lengths and bond angles are given in Tables 2. The X-ray crystal structure of the complex revealed the presence of two Zn(II) atoms. One molecule of complex has present two five-coordinated zinc atoms separated by 3.187 Å and the zinc atoms in each molecular unit have only a weak interaction with together. The presence of two metal ions at a suitable distance from each other is a prerequisite for the formation of an activation space necessary for the coordination of other donors and/or organic substrates [29]. The structure of $[ZnL]_2.H_2O$ confirms a distorted trigonal bipyramidal geometry around the each Zn(II) center. The Schiff base behave as tetradentate dinegative ligand through the both imine N and the both phenolic O atoms (N_2O_2 donors) in binuclear zinc(II) complex and one of the phenolate oxygen atoms act as bridging ligand in each Zn(II) center and the Zn(II) ion is bound through the N_2O_3 atoms (Fig. 1). The bond angels O(1)-Zn(1)-N(2) and O(1a)-Zn(1)-N(1) are 173.5° and 141.0° (β and α) respectively, thus the geometric parameter $\tau = (\beta-\alpha)/60$ for this complex is 0.54, and confirms a distorted trigonal bipyramidal geometry [30]. The angle between the plane formed by N(1) O(1a) O(2) and the plane formed by N(1a) O(1) O(2a) is 40.13°. Zinc atom distance to the plane formed by N(1) O(1a) O(2) is 0.234 Å. In each molecular unit of the complex, two zinc atoms and two bridging phenolate oxygen atoms formed a distorted square as a butterfly geometry. The angle between the plane formed by Zn(1) O(1) O(1a) and the plane formed by Zn(1a) O(1) O(1a) is 22.89° and the Zn(1a) is 0.631 Å out of the Zn(1) O(1) O(1a) plane. The complex molecule contains a center of symmetry thereby marking the two metal ions chemically and crystallographically equivalent in the solid state.

CONCLUSIONS

We report the successful syntheses of a pentadentate N_2O_2S donor thioether Schiff base ligand (H_2L). Then, binuclear Zn(II) complex of this ligand was prepared and structurally characterized by spectroscopic methods and X-ray crystallography. The solid state structure of $[ZnL]_2.H_2O$ shows that the ligand is tetradentate and with bridging phenolate oxygen atom has a distorted trigonal bipyramidal geometry around the Zn(II) center.

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Appendix A. Supplementary Data

CCDC 1405410 contains the supplementary crystallographic data for $[ZnL]_2.H_2O$. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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