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**A NEW Zn(II) COMPLEX OF UNUSUAL UNIDENTATE COORDINATION  
OF 4,4'-BIPYRIDINE, A NEW PRECURSOR FOR THE PREPARATION  
OF ZINC(II) OXIDE NANOPARTICLES**

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A 4,4'-bipyridine Zn(II) complex  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  is synthesized, characterized by elemental analysis, IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR spectroscopy, and studied by X-ray crystallography. The compound forms monomeric units as a result of unusual unidentate coordination of 4,4'-bipy ligands. The thermal stability of the compound is studied by thermal analyses. Furthermore, the complex is luminescent with emission maxima at 329 nm in the methanol solvent. Different sizes of zinc(II) oxide nanoparticles are prepared by calcination of the  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  compound at two different temperatures. These nanostructures are characterized by X-ray powder diffraction and scanning electron microscopy.

**Keywords:** zinc(II), 4,4'-bipyridine, coordination polymer, fluorescence, nanoparticles.

### INTRODUCTION

Zinc always occurs as a divalent cation and it is the second most abundant transition metal following iron [1–4]. In recent years, a wide variety of networks have been designed using the inorganic coordination polymers that provide very interesting information about supramolecular systems [5–7]. The Zn(II) ion is particularly appropriate for the construction of coordination polymers and networks [8–23]. The zinc(II) coordination polymers with 4,4'-bipyridine (4,4'-bipy) spacer ligands such as  $[\text{Zn}(4,4'\text{-bipy})(\text{detp})_2]_n$ ,  $\text{dpet} = \text{P,P-diphenyl-2-[3,5-dimethylpyrazol-1-yl]-ethylphosphinate}$  [10],  $[\text{Zn}(4,4'\text{-bipy})(\text{sal})_2]_n$  [10],  $[\text{Zn}(4,4'\text{-bipy})(\text{SC}\{\text{O}\}\text{CH}_3)_2]_n$ ,  $[\text{Zn}(4,4'\text{-bipy})(\text{SC}\{\text{O}\}\text{Ph})_2]_n$  [10],  $[\text{Zn}(4,4'\text{-bipy})(\text{SPh})_2]_n$ ,  $\{[\text{Zn}(4,4'\text{-bipy})(\text{SPh})_2] \cdot 0.25\text{CH}_2\text{Cl}_2 \cdot \text{H}_2\text{O}\}_n$  [11],  $[\text{Zn}(4,4'\text{-bipy})(\text{NCS})_2]_n$  [12],  $[\text{Zn}(4,4'\text{-bipy})\text{Cl}_2]_n$ ,  $[\text{Zn}(4,4'\text{-bipy})\text{Br}_2]_n$  [13],  $\{[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_2]\text{SiF}_6 \cdot 2\text{H}_2\text{O}\}_n$  [14],  $\{[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_2](\text{ClO}_4)_2 \cdot (2,4'\text{-bipy}) \cdot 2\text{H}_2\text{O}\}_n$  [15],  $[\text{Zn}(4,4'\text{-bipy})_2\text{SiF}_6]_n$  [16],  $\{[\text{Zn}(4,4'\text{-bipy})(\text{H}_2\text{O})_4](\text{NO}_3)_2 \cdot (4,4'\text{-bipy})\}_n$ ,  $\{[\text{Zn}(4,4'\text{-bipy})(\text{H}_2\text{O})_4](\text{NO}_3)_2 \cdot 2(4,4'\text{-bipy}) \cdot 3\text{H}_2\text{O}\}_n$ ,  $\{[\text{Zn}(4,4'\text{-bipy})(\text{H}_2\text{O})_4](\text{CF}_3\text{SO}_3)_2 \cdot 2(4,4'\text{-bipy})\}_n$  [17],  $[\text{Zn}(4,4'\text{-bipy})(\text{H}_2\text{O})_3(\text{ClO}_4)]\text{ClO}_4 \cdot 1.5(4,4'\text{-bipy}) \cdot \text{H}_2\text{O}\}_n$  [13],  $\{[\text{Zn}_2(4,4'\text{-bipy})_3(\text{NO}_3)_4] \cdot 2\text{H}_2\text{O}\}_n$  [18],  $[\text{Zn}(4,4'\text{-bipy})(\text{N}_3)_2]_n$  [19],  $\{[\text{Zn}(4,4'\text{-bipy}) \cdot (\text{H}_2\text{O})_2(\text{bpen})_2](\text{NO}_3)_2 \cdot 1.75\text{bpen} \cdot 0.25(4,4'\text{-bipy}) \cdot 4.45\text{H}_2\text{O}\}_n$  [21], and  $[\text{Zn}_2(4,4'\text{-bipy})(\text{OAc})_4]_n$  [22] have been studied in detail. In most of these complexes, the 4,4'-bipyridine ligands are coordinated to the zinc(II) ion as bridging, act as bridging ligands, and form one-, two- or three-dimensional coordi-

nation polymers. A zinc(II) complex with unidentate coordination of 4,4'-bipyridine has been reported in the literature [23]. We describe here on the preparation, thermal properties and crystal structure of  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  in the room temperature. Different sizes of zinc(II) oxide nanoparticles were prepared from calcinations of compound  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  at two different temperatures.

### EXPERIMENTAL

**Physical property measurements.** IR spectra were recorded using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and were uncorrected. The thermal behavior was measured with a PL-STA 1500 apparatus.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500 MHz and all chemical shifts are reported in  $\delta$  units downfield from  $\text{Me}_4\text{Si}$ .

**Preparation of  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$ .** To a magnetically stirred solution of the (4,4'-bipy) ligand (0.312 g, 2 mmol) in methanol (10 ml) was added dropwise a mixture of  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 3 \text{H}_2\text{O}$  (0.237 g, 1 mmol) and  $\text{NaClO}_4 \cdot \text{H}_2\text{O}$  (0.144 g, 1 mmol) in methanol (20 ml) at room temperature over 10 min. The reaction mixture was stirred for 1 h at room temperature. The white powder was filtered off, washed with acetone (5 ml), diethyl ether (5 ml) and dried in vacuum over silica gel at room temperature. The product was then dried in vacuum over  $\text{P}_4\text{O}_{10}$  at 50 °C. M. p. 115 °C. Yield: 0.315 g, 50 %. A single crystal suitable for X-ray analysis was obtained by slow evaporation of an acetonitrile solution of the product at room temperature. (Found: C, 56.45; H, 4.50; N, 13.20 %. calculated for  $\text{C}_{60}\text{H}_{56}\text{Cl}_2\text{N}_{12}\text{O}_{12}\text{Zn}$ : C, 56.53; H, 4.39; N, 13.19 %).

**IR** ( $\text{cm}^{-1}$ ) selected bonds:  $\nu(\text{O}-\text{H})_{\text{water}}$  3100–3250 (b),  $\nu(\text{C}-\text{H})_{\text{ar}}$  3025(m),  $\nu(\text{C}=\text{C})$ ,  $\nu(\text{C}=\text{N})$  1626(s), 1524(m), and  $\nu(\text{ClO}_4)$  1085 (vs).

**$^1\text{H}$  NMR** (DMSO;  $\delta$ ): 7.85 (d, 2H) and 8.75 (d, 2H) ppm.

**$^{13}\text{C}-\{^1\text{H}\}$  NMR** (DMSO;  $\delta$ ): 121.7, 144.9 and 151.1 ppm.

**Preparation of zinc(II) oxide nanoparticles.** The  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  compound was calcinated in 400 °C and 500 °C; the whole of organic components were combusted and zinc(II) oxide was produced. The XRD pattern shows the rest of the product is zinc(II) oxide.

**Determination of the structure of  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$ .** Crystallographic measurements were made at 298(2) K using a Siemens R3m/V diffractometer. The accurate unit cell parameters and the orientation matrix for data collection were obtained from the least squares refinement. The structure was solved by direct methods and refined by full matrix least squares techniques on  $F^2$ .

### RESULTS AND DISCUSSION

The reaction between 4,4'-bipy and a mixture of zinc(II) acetate with sodium perchlorate in methanol provided the crystalline material analyzed as  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$ . The IR spectrum displays absorption bands characteristic of 4,4'-bipy and water ligands and perchlorate anions. A very strong band at 1085  $\text{cm}^{-1}$  characterizes the  $\nu(\text{Cl}-\text{O})$  vibrations. The absorption bands with a variable intensity in the frequency range 1400–1590  $\text{cm}^{-1}$  correspond to the ring vibrations of the pyridyl moiety of 4,4'-bipy ligands. The broad absorption band for  $\nu(\text{HOH})$  at 3100–3250  $\text{cm}^{-1}$  indicates the presence of water molecules in this compound and is indicative of hydrogen bonding as confirmed by the crystal structure of this complex.

The  $^1\text{H}$  NMR spectrum of the DMSO solution of the complex displays two distinct doublets at *ca.* 7.85 and 8.75 ppm assigned to the aromatic ring protons of 4,4'-bipy ligands. The  $^{13}\text{C}$  NMR spectrum of the DMSO solution shows three distinct signals at 121.7 ppm, 144.9 ppm, and 151.1 ppm assigned to the three aromatic ring carbon atoms of 4,4'-bipy ligands.

In order to examine the thermal stability of the compound, thermogravimetric (TG) and differential thermal analyses (DTA) were carried out for  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  between

30 °C and 700 °C (Fig. 1). The compound is stable up to 115 °C, at which temperature it begins to melt. The TG curve indicates the release of four water and four 4,4'-bipy molecules up to 290 °C with two endothermic effects at 250 °C and 290 °C (observed 55.10 %, calcd. 55.65 %) to give the product of water- and guest-free  $[\text{Zn}(\text{4,4}'\text{-bipy})_2](\text{ClO}_4)_2$ , which is stable up to 310 °C. The weight loss of 24.10 % from 370 °C to 415 °C is equivalent to the loss of two coordinated 4,4'-bipy molecules (calcd. 24.50 %) with an exothermic effect at 410 °C. The decomposition of perchlorate anions takes place at 425.60 °C and the experimental mass loss of 13.75 % is consistent with the calculated value of 14.37 % for the elimination of 7 oxygen and 2 Cl atoms of two perchlorate anions with two endothermic effects at 510 °C and 590 °C. The solid residue formed at around 600 °C is suggested to be ZnO (observed 6.50, calcd. 6.36 %).

The UV/vis spectrum of the  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  complex displays intense absorption bands ranging from 220 nm to 2900 nm (Fig. 2, *a*), indicating that electronic transitions are mostly  $\pi$  to  $\pi^*$ , originating from the pyridyl rings of the 4,4'-bipy ligand. The fluorescence spectra of the 4,4'-bipy ligand and the  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  complex in MeOH are studied. The complex shows a broad emission band with the maximum intensity at 329 nm upon excitation at 295 nm (Fig. 2, *b*). These fluorescent emissions can be tentatively assigned to the intraligand fluorescent emission, since a similar emission at 347 nm upon excitation at 295 nm can also be observed for free 4,4'-bipy (Fig. 2, *c*). Generally, the intraligand fluorescent emission wavelength is determined by the energy gap between the  $\pi$  and  $\pi^*$  molecular orbitals of the free ligand, which is simply related to the degree of  $\pi$  conjugation in the system. Thus the unexpected slightly blue-shifted emission of the  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  complex probably results from 4,4'-bipy ligand coordination in the solution state that has an effect on their fluorescent property.

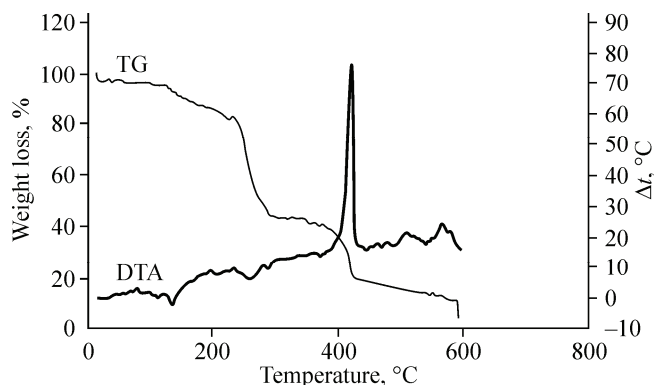


Fig. 1. Thermal behavior of  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$

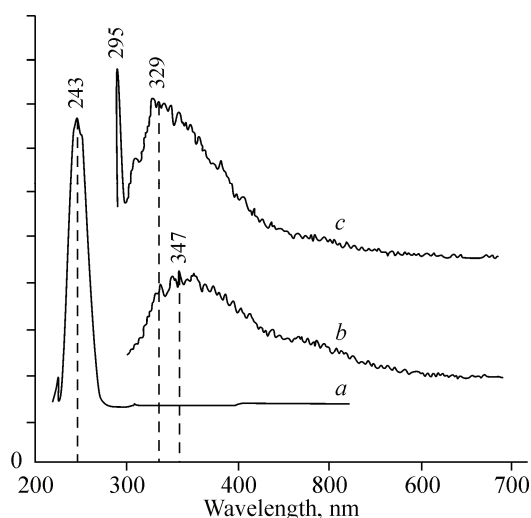


Fig. 2. Electronic absorption of  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4] \cdot (\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  (*a*), Absorption:  $c = 1.36 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$ , methanol,  $d = 1 \text{ cm}$ . Fluorescence spectra of the solution state of the 4,4'-bipy ligand (*b*) and the solution state of  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  (*c*),  $\lambda_{\text{exc}} = 295 \text{ nm}$

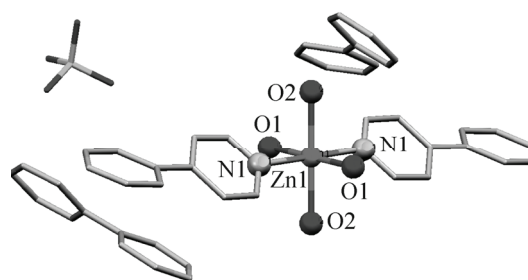


Fig. 3. Molecular structure of  $[\text{Zn}(\text{4,4}'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$

T a b l e 1

Crystal data and structure refinement for  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$ 

Empirical formula	$\text{C}_{60}\text{H}_{56}\text{Cl}_2\text{N}_{12}\text{O}_{12}\text{Zn}$
Molecular weight	1273.46
Temperature, K	298(2)
Wavelength, Å	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
$a, b, c, \text{Å}$	7.8805(7), 14.187(1), 14.830(1)
$\alpha, \beta, \gamma, \text{deg.}$	63.704(1), 88.039(1), 81.415(1)
$V, \text{Å}^3$	1468.8(2)
$Z$	1
$D_{\text{calcd}}, \text{g} \cdot \text{cm}^{-3}$	1.440
$\mu, \text{mm}^{-1}$	0.583
$F(000)$	660
Crystal size, mm	0.40×0.32×0.27
$\theta$ Range, deg.	1.62 to 25.50
Index ranges	$-9 \leq h \leq 9, -17 \leq k \leq 16, -17 \leq l \leq 17$
Reflections collected	11110
Independent reflections ( $R_{\text{int}}$ )	5412 (0.0186)
Reflections observed ( $>2\sigma$ )	5085
Absorption correction	Multi-scan
Max. and min. transmissions	0.858 and 0.800
Data / restraints / parameters	5085 / 0 / 394
Goodness-of-fit on $F^2$	1.161
$R [I \geq 2\sigma(I)]$	$R1 = 0.0548, wR2 = 0.1298$
$R$ (all data)	$R1 = 0.0577, wR2 = 0.1315$
Largest diff. peak and hole, $\text{e}/\text{Å}^{-3}$	0.375 and $-0.451$

The determination of the structure of  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  by X-ray crystallography (Table 1) showed the complex in the solid state (Fig. 3) to consist of mononuclear units similar to the reported one [23]. The zinc atoms are linked by four oxygen and two nitrogen atoms and can be considered six-coordinate with  $\text{O}_4\text{N}_2$  donor atom arrays. The coordination about Zn is distorted octahedral and two nitrogen atoms of 4,4'-bipy ligands with distances  $\text{Zn}-\text{N} = 2.117(2) \text{ Å}$ , and four oxygen atoms of water molecules with distances  $\text{Zn1}-\text{O2} = 2.130(2) \text{ Å}$ ,  $\text{Zn1}-\text{O1} = 2.149(2) \text{ Å}$  coordinate the Zn atoms. One nitrogen atom of 4,4'-bipy ligands is not coordinated to Zn atoms (Fig. 3) and thus this ligand acts as a less commonly unidentate ligand. The two nitrogen atoms are in *trans* position [ $\text{N1}-\text{Zn1}-\text{N1}^i = 180^\circ$ ]. By heating the  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2 \cdot 4(4,4'\text{-bipy})$  compound at  $400^\circ\text{C}$  (direct calcination) all the organic moieties would be decomposed and ZnO nanoparticles would be obtained finally. Zinc(II) oxide was iden-

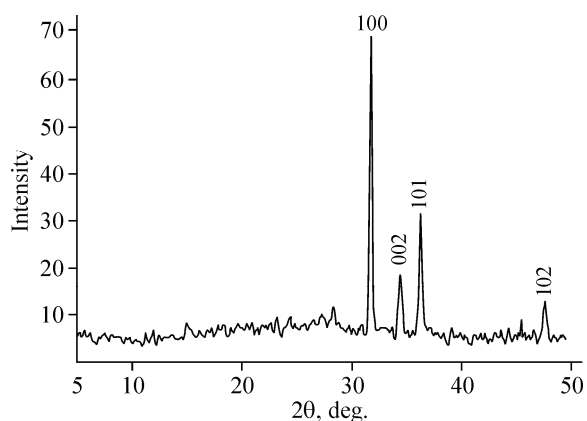


Fig. 4. XRD pattern of ZnO nanoparticles produced by calcination of compound 1

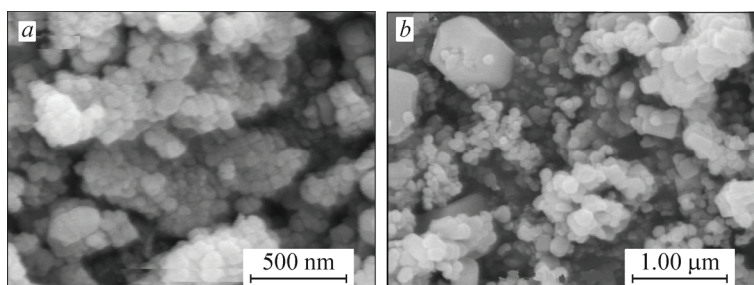


Fig. 5. SEM photograph of ZnO microparticles produced by calcination of precursor **1** at 400 °C (a) and (b)

tified by X-ray diffraction (Fig. 4) and the particle size was investigated with scanning electron microscopy (Fig. 5). Estimated by the Scherrer formula,  $D = 0.891\lambda/\beta\cos\theta$ , where  $D$  is the average grain size,  $\lambda$  is the X-ray wavelength (0.15405 nm), and  $\theta$  and  $\beta$  are the diffraction angle and the full-width at half maximum of an observed peak, respectively. The average particle size is 65 nm, which is in approximate agreement with that observed by scanning electron microscopy, as shown in Fig. 5, a. Also, zinc(II) oxide nanoparticles from the decomposition of  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4]\cdot(\text{ClO}_4)_2\cdot 4(4,4'\text{-bipy})$  were obtained at 500°C.

Fig. 5, b shows the SEM image of the residue obtained from the decomposition of the  $[\text{Zn}(4,4'\text{-bipy})_2(\text{H}_2\text{O})_4](\text{ClO}_4)_2\cdot 4(4,4'\text{-bipy})$  compound. This image shows that ZnO nanoparticles have a larger size than ZnO nanoparticles prepared at 400 °C.

**Supplementary material.** A complete list of bond lengths and angles, coordinates and displacement parameters have been deposited at the Cambridge Crystallography Data Centre. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number 288301.

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