Article



A New Imide-pyrazine Zinc(II) Binuclear Complex; In Situ Synthesis and Crystal Structure Determination

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Cite This; *Inorg. Chem. Res.* **2022**, *6*, 155-160. DOI: 10.22036/j10.22036.2023.398659.1143 **Abstract:** A new zinc(II) binuclear complex, $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1) (where DPzCA is N-(2-pyrazylcarbonyl)-2-pyrazinecarboxamidate anion and 2-MPzCI is methyl pyrazine-2-carboximidate), was synthesized from the reaction of ZnCl₂.7H₂O and 2-cyanopyrazine in methanolic solution at 65 °C. This complex was thoroughly characterized by elemental analysis (CHN-O), IR, UV-Vis, ¹H NMR spectroscopy and its structure have been determined by the singlecrystal X-ray diffraction. The spectroscopic investigations and X-ray structural analysis indicated that by methanolysis and in the presence of ZnCl₂.7H₂O salt the 2-cyanopyrazine ligand converts to methyl pyrazine-2-carboximidate. Furthermore, the bis(2-pyrazinecarbonyl)amidate anionic ligand have been prepared from 2-cyanopyrazine in the presence of ZnCl₂.7H₂O salt in methanolic solution.



Keywords: Zinc(II) binuclear complex, Crystal structure, 2-Cyanopyrazine, Methyl pyrazine-2-carboximidate, N-(2-Pyrazylcarbonyl)-2-pyrazinecarboxamidate

1. INTRODUCTION

Zinc(II) cation is the second most abundant transition metal next to the iron(II) cation in the body. This cation has the most stable oxidation state of Zn^{2+} , with the spherical symmetry of electron shells, d¹⁰, is a hard acid in the hard-soft acids-bases system. Strongly bonded coordination compounds are formed by zinc(II) cation with hard donor atoms such as that of nitrogen and oxygen atoms.¹ Until now, several zinc(II) complexes with nitrogen and oxygen donor ligands were reported and were characterized by spectroscopy and X-ray diffraction method. These zinc(II) complexes have mononuclear, binuclear, trinuclear, tetranuclear, coordination polymer, and metal-organic frame works (MOFs) structures.²⁻¹⁰

Zinc(II) binuclear complexes containing monodentate *N*-donor and chelating *N*,*O*- and *N*,*O*,*N'*-donor ligands are important due to their application as antibacterial,¹¹⁻¹⁵ anticancer and cytotoxic agentes,^{16,17} in catalytic systems,^{18,19} and due to special optical properties.^{20,21} Furthermore, zinc(II) binuclear complexes may serve as model compounds for the active sites of many metalloenzymes and they play an essential role in many biological systems.^{22,23} Use of the binuclear complexes containing of a zinc(II)-zinc(II) core is also the most commonly used strategy for selective recognition of the pyrophosphate anion.²⁴⁻²⁶

The methyl pyrazine-2-carboximidate (2-MPzCI) as a multidentate ligand can be coordinated to metal center through nitrogen atom of pyrazine ring as a mono-dentate ligand, through one nitrogen atom of pyrazine ring and one nitrogen atom of amide group as chelating bidentate ligand and through one nitrogen atom of amide group and two nitrogen atoms from pyrazine ring as a tridendete ligand (Scheme 1). These three coordination modes for this ligand can lead to the preparation of many complexes with different structures, but so far only four monomeric and polymeric complexes with this ligand have been reported from the reaction of 2-cyanopyrazine with metal salts in methanolic solvent.^{27,28} On the other hand, the N-(2-pyrazylcarbonyl)-2-pyrazinecarboxamide (HDPzCA) compound is a polydentate ligand and can be coordinated to metal ions in anionic form from five nitrogen and two oxygen atoms. This multidentate property of this ligand may lead to metal-organic frameworks (MOFs) with special structures.^{29,30} The HDPzCA ligand was prepared from the reaction of 2-pyrazine carboxylic acid and 2pyrazine carboxamide in thionyl chloride at 120°C previously.30

Recently, we reported synthesis and crystal structure of two mononouclear $[Mn(2-MPyzCI)_2Cl_2].H_2O$ and $[Mn(2-MPyzCI)_2(H_2O)_2](NO_3)_2$ complexes.²⁸ Both complexes were prepared from the reaction of $MnX_2.4H_2O$ (X=Cl⁻ and NO_3^-) salts and 2-cyanopyrazine ligand in methanol

solvent at 45 °C. In these complexes, the 2-MPzCI ligand is formed by methanolysis of 2-cyanopyrazine in the presence of Mn(II) ions.²⁸ In continuation of these research works, I wish to report herein the synthesis and crystal structure of a new binuclear complex of $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1). Structural studies on this complex were performed by elemental analysis, IR, UV-Vis and ¹H NMR spectroscopic technique and X-ray crystallography. In this complex, the chelating 2-MPyzCI ligand and bridging DPzCA ligand were formed *via* the methanolysis and hydrolysis-imidation of 2cyanopyrazine, respectively.



Scheme 1. Possible coordination modes of methyl pyrazine-2carboximidate with metal ion

2. EXPERIMENTAL

Materials and physical methods

All reagents and solvents were purchased from Merck and used without further purification. Melting point was obtained on a Kofler Heizbank Rechart type 7841 melting point apparatus. Elemental analysis (C, H, N and O) was performed using a Heraeus CHN–O Rapid analyzer. IR spectrum (4000-250 cm⁻¹) was obtained as CsI pellets using a Shimadzu-470 spectrometer. ¹H-NMR spectrum was recorded in CD₃OD at room temperature on a Bruker AC-300 spectrometer for protons at 300.13 MHz, and electronic spectrum was recorded on a Shimadzu 2100 spectrometer using a 1 cm path length cell in methanol at room temperature.

Synthesis of [Zn₂(µ-DPzCA)(2-MPzCI)Cl₃] (1)

A solution of 2-cyanopyrazine (0.36 g, 0.30 mL, 3.36 mmol) in methanol (20 mL) was added to a solution of ZnCl₂.7H₂O (0.59 g, 2.24 mmol) in methanol (20 mL). In a round-bottom balloon equipped with a condenser, the resulting colorless solution was stirred for 20 min at 65 °C. The solution was then left to evaporate slowly at room temperature. After one week, colorless block crystals of **1** were isolated (yield 0.43 g, 63.7%, m.p. = 114 °C). IR (CsI, cm⁻¹): 3315m, 3162w, 3086w, 2980w, 1689s,1645s, 1603m, 1463m, 1385m, 1374s, 1210m, 1149m, 1052m, 1019s, 961m, 653m, 554m, 413m, 365m, 271m, 262m. UV-Vis: λ_{max} (CH₃OH, nm), 222, 269. ¹H-NMR (CD₃OD, ppm): δ (ppm) 4.75 (s, 3H, -OCH₃), 8.74 (m, 3H), 8.88 (d, 1H), 9.06 (d, 2H), 9.38 (s,1H) and 9.98 (s, 2H). Anal. Calcd. for C₁₆H₁₃Cl₃N₈O₃Zn₂ (%): C, 31.90; H, 2.16; N, 7.97; O, 7.97. Found: C, 31.68; H, 2.15; N, 7.90; O, 8.01.

X-Ray structure analysis

The X-ray diffraction measurements was made on a Bruker APEX II CCD area detector diffractometer at 298 K for 1 (Mo-

K α radiation, graphite monochromator, $\lambda = 0.71073$ Å). The structure of this complex was solved by SHELX-97 and absorption correction was done using the SADABS program.³¹ Some software including Bruker APEX II (data collection and cell refinement), Bruker SHELXTL (data reduction), and WinGX (publication material) were properly used.³²⁻³⁵ The ORTEP and crystal packing diagram for title complex were drawn with the Mercury 2.4 program.³⁶

3. RESULTS AND DISCUSSION

Synthesis of [Zn₂(µ-DPzCA)(2-MPzCI)Cl₃] (1)

Complex 1 was prepared from the reaction of three equivalents of 2-cyanopyrazine ligand with two equivalents of ZnCl₂.7H₂O salt in CH₃OH at 65 °C. Suitable crystals of this complex were obtained for X-ray diffraction measurement by slow evaporation of methanolic solution at room temperature. The synthetic route of this complex is shown in Scheme 2. The proposed mechanism for the synthesis of complex 1 from reaction of ZnCl₂.7H₂O salt with 2-cyanopyrazine ligand in methanol is shown in Scheme 3. As shown in this Scheme, the methyl pyrazine-2-carboximidate (2-MPzCI) and N-(2-pyrazylcarbonyl)-2-pyrazinecarboxamide (HDPzCA) ligands were formed *via* the methanolysis and hydrolysis-imidation of 2-cyanopyrazine, respectively in the presence of ZnCl₂.7H₂O salt in methanolic solution.²⁸



Scheme 2. The preparation method of complex 1



Scheme 3. The proposed mechanism for the formation of 2-MPzCI and HDPzCA from 2-cyanopyrazine ligand in the presence of $ZnCl_2.7H_2O$ salt and in methanol solvent

Spectroscopic characterization of $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1)

The infrared absorption bands for title complex are listed in the experimental section. The infrared spectrum for this complex shows several medium and weak absorption bands in the region of 3315-2980 cm⁻¹, which are assigned to the N-H stretching vibrations of amide group and C-H vibrations of pyrazine rings and methyl groups.^{27,37-39} The infrared spectrum of the free 2-cyanopyrazine ligand shows a medium absorption band at 2241 cm⁻¹ for cyanid group (C=N).⁴⁰ This absorption band disappears in the infrared spectrum of the title complex, showing that the nitrile group is converted to carboximidate group. The strong band at 1689 cm⁻¹ in the infrared spectrum of title complex is assigned to v(C=O) of carboxamidate. Furthermore, the strong band at 1645 cm⁻¹ is assigned to v(C=NH) of carboxamide.^{28,41} Several medium and strong bands observed in the range 1463-1385 cm⁻¹ are assigned to C=N and C=C stretching vibrations.28,37,41 Also, the absorption band at 1374 cm⁻¹ is assigned to v(=C-O-)stretching vibration which mixed with $\delta(NH)$ of the imino ether group. The absorption band observed at 1210 cm⁻¹ is assigned to δ (O-CH₃). In addition, the absorption bands at 1149 and 961 cm⁻¹ are assigned to vas(C-O-C) and v_s(C–O–C), respectively.^{28,37,41} Far infrared spectrum of title complex was recorded between 500 and 250 cm⁻¹. The medium absorption bands observed at 413 and 365 cm⁻¹ are assigned to the Zn-N stretching vibrations.^{28,37,42} The Zn-Cl stretching vibrations are assigned to bands at 271 and 262 cm^{-1.43}

The UV-Vis spectra of methanol solutions of 1 have two broad bands at around 222 and 269 nm which can be assigned to the $\pi \rightarrow \pi^*$ transitions.^{3,44}

The ¹H-NMR data for title complex was prepared in CD₃OD solution at room temperature and the results are listed in the experimental section. The ¹H NMR spectrum of **1** exhibited a singlet band at 4.75 ppm for the methoxy group, a multiplet band at around 8.74 ppm for three protons, a doublet band at 8.88 ppm for one proton, a doublet band at 9.06 ppm for two protons, a singlet band at 9.38 ppm for two protons and a doublet band at 9.98 ppm for two protons. Furthermore, due to the proton exchange with water protons in the CD₃OD solvent, in ¹H-NMR spectrum of **1**, =NH protons can not be seen. Also, the intensities of the signals in the ¹H-NMR spectrum is in agreement with a 1:1 (2-MPzCI:DPzCA) stoichiometry proposed in Scheme 2.

Description of the molecular structure of $[Zn_2(\mu - DPzCA)(2-MPzCI)Cl_3]$ (1)

Crystallographic data for title complex are given in Table 1 and selected bond lengths and angles are presented in Table 2. The coordination polyhedral and molecular structure of binuclear $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1) complex with the atomic labeling scheme are shown in Figs. 1 and 2, respectively. As shown in these figures, the structure of the complex 1 consists of two Zn(II) atoms bridged by one N-(2-pyrazylcarbonyl)-2-pyrazinecarboxamidate ligand. The complex 1 is binuclear and each zinc(II) cation is pentacoordinated, but the sources of the coordination cations of

the two zinc(II) cations are different with respect to each other. The Zn1 is surrounded by two nitrogen atoms (N1 and N3 from a methyl pyrazine-2-carboximidate ligand), two oxygen atoms (O2 and O3 from a bridged N-(2pyrazylcarbonyl)-2-pyrazinecarboxamidate ligand) and one chloride ion (Cl1). Furthermore, the Zn2 is surrounded by three nitrogen atoms (N4, N6 and N7 from а bridged N-(2-pyrazylcarbonyl)-2pyrazinecarboxamidate ligand) and two chloride ions (Cl2 and Cl3). The coordination geometry around each zinc(II) cation is five-coordinated in a distorted square-pyramidal configuration ($\tau = 0.718$ for Zn1 and 0.602 for Zn2; $\tau =$ $(\beta - \alpha)/60$, where $\beta = N1-Zn1-O3$ 166.4° and N4-Zn2-N7 148.2° for Zn1 and Zn2, respectively, and $\alpha = O2-Zn1-$ Cl1 123.3° and Cl2-Zn2-Cl3 112.06°, for Zn1 and Zn2, respectively). The τ parameter being 0 and 1 for perfect square-pyramidal and trigonal-bipyramidal geometries, respectively (Scheme 4).45-47.

Table 1. Crystallographic and structural refinement data for 1					
Formula	$C_{16}H_{13}Cl_{3}N_{8}O_{3}Zn_{2} \\$				
Formula weight	602.47				
Temperature /K	298(2)				
Wavelength λ /Å	0.71073				
Crystal system	Triclinic				
Space Group	Pī				
Crystal size /mm ³	0.45×0.40×0.35				
<i>a</i> /Å	8.0708(13)				
b /Å	12.4270(16)				
c /Å	13.2272(18)				
α /°	77.548(11)				
β /°	78.805(12)				
γ /°	73.914(11)				
Volume /Å ³	1231.9(3)				
Ζ	2				
Density (calc.) /g cm ⁻¹	1.624				
θ ranges for data collection	2.55-27.00				
F(000)	600				
Absorption coefficient mm ⁻¹	2.305				
Index ranges	$\text{-}9 \leq h \leq 10$				
	$\text{-}15 \leq k \leq 15$				
	$0 \leq l \leq 16$				
Data collected	5364				
Unique data (R _{int})	5364, (0.0582)				
Parameters, restrains	289, 0				
Final R_1 , wR_2 (Obs. data)	0.0452, 0.1582				
Final R_1 , wR_2 (All data)	0.0582, 0.1752				
Goodness of fit on F^2 (S)	1.006				
Largest diff peak and hole /e.Å $^{\rm -3}$	0.847, -0.855				
CCDC No.	2264655				

Table 2. Selected bond distances/Å and bond angles/° for 1					
Zn1-N1	2.303(11)	N3-Zn1-Cl1	108.1(3)		
Zn1-N3	2.102(10)	O2-Zn1-N1	89.5(4)		
Zn1-Cl1	2.292(4)	O3-Zn1-N1	166.4(4)		
Zn1-O2	2.030(9)	N3-Zn1-N1	75.4(4)		
Zn1-O3	2.049(10)	Cl1-Zn1-N1	89.7(3)		
Zn2-Cl2	2.258(4)	N4-Zn2-N6	76.0(4)		
Zn2-Cl3	2.281(4)	N4-Zn2-N7	148.2(4)		
Zn2-N4	2.142(11)	N6-Zn2-N7	75.5(4)		
Zn2-N6	2.143(8)	N4-Zn2-Cl2	102.7(3)		
Zn2-N7	2.150(11)	N6-Zn2-Cl2	110.1(3)		
O2-Zn1-O3	87.4(4)	N7-Zn2-Cl2	100.2(3)		
O2-Zn1-N3	126.3(4)	N4-Zn2-Cl3	94.6(3)		
O3-Zn1-N3	95.8(4)	N6-Zn2-Cl3	137.8(3)		
O2-Zn1-Cl1	123.3(3)	N7-Zn2-Cl3	97.0(3)		
O3-Zn1-Cl1	103.1(3)	Cl2-Zn2-Cl3	112.06(16)		



Figure 1. The coordination polyhedral of $[{\rm Zn}_2(\mu\text{-}DPzCA)(2\text{-}MPzCI)Cl_3]$ (1).



Figure 2. The molecular structure of $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1), with the atom-numbering scheme and 30% probability displacement ellipsoids.



 $\alpha = \beta = 180^{\circ}$

 $\alpha = 120^{\circ} \text{ and } \beta = 180^{\circ}$

Scheme 4. The perfect square-pyramidal and trigonal-bipyramidal geometries with the basal angles α and β^{46}

Furthermore, in this complex, the Zn1-Cl (2.292(4) Å), Zn1-N (2.303(11) and 2.102(10) Å) and Zn1-O (2.030(9) and 2.049(10) Å) are similar to [Zn(2,2'-bipy)(curc)Cl] (2,2'-bipy is 2,2'-bipyridine and curc is curcumin).⁴⁸ Also, the Zn2-N (2.142(11), 2.143(8) and 2.150(11) Å) and Zn2-Cl (2.258(4) and 2.281(4) Å) are similar to

 $[Zn(^{tertbut}terpy)Cl_2]$ ($^{tertbut}terpy$ is 4,4',4"-tri-tert-butyl-2,2':6',2"-terpyridine).⁴⁹

The mean planes of rings A (N1/C1/C2/N2/C3/C4), B (N4/C7/C8/N5/C9/C10) and C (N7/C14/C15/N8/C16/C13) make the following dihedral angles with each other: A/B = 59.02, A/C = 43.82 and B/C = 21.29°.

Crystal packing diagram for **1** is shown in figures 3 and 4. As it is shown in these figures, the head-to-tail $N...\pi$ interactions between the N atoms and pyrazine rings, N5…Cg7ⁱⁱ [distance = 3.520 Å, symmetry code: -x,2-y, 2-z, where Cg7 is centroid of the ring (N7/C14/C15/N8/16/C13)] and intermolecular N–H…Cl, C–H…N and C–H…Cl hydrogen bonds (Table 3) are effective in the stabilization of the crystal structure and the formation of the 3D supramolecular complex.



Figure 3. Crystal packing diagram for $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1). Intermolecular N–H···Cl, C–H···N and C–H···Cl hydrogen bonds are shown as dashed lines. Symmetry codes: (i) 1-x,2-y,1-z and (ii) -x,2-y,2-z.



Figure 4. Crystal packing diagram for $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1). Intermolecular N–H···Cl, C–H···N and C–H···Cl hydrogen bonds and N- π contacts are shown as dashed lines. Symmetry codes: (i) 1-x,2y,1-z and (ii) -x,2-y,2-z.

Table 5. Hydrogen bond geometry for T in crystal packing						
D-H…A	D-H/ Å	H…A/Å	D…A/Å	D-H···A/°	Symmetry code	
N3-H3B···Cl1	0.86	2.590	3.342(1)	147	-x,2-y,2-z	
C1-H1····Cl2	0.93	2.730	3.365(2)	126	1-x,1-y,2-z	
C9-H9…Cl2	0.93	2.853	3.441(1)	122	1-x,1-y,2-z	
C15-H15…Cl2	0.93	2.916	3.825(2)	166	1-x,1-y,2-z	
C2-H2…N8	0.93	2.703	3.560(2)	154	-	

 Table 3. Hydrogen bond geometry for 1 in crystal packing

4. CONCLUSIONS

In this research work, a new zinc(II) binuclear complex, $[Zn_2(\mu-DPzCA)(2-MPzCI)Cl_3]$ (1), has been synthesized. This complex was fully characterized by spectroscopic methods and its structure was determined by the single crystal diffraction analysis. This compound is binuclear complex and the coordination geometry around each central metal is five-coordinated in a distorted squarepyramidal configuration. It is notable that in this compound, nonclassical N-H···Cl, C-H···N and C-H···Cl hydrogen bonds and N- π interactions mostly stabilize the crystal lattice. Furthermore, in this binuclear complex, and in the presence of ZnCl₂.7H₂O salt, the 2cyanopyrazine converts to N-(2-pyrazylcarbonyl)-2pyrazinecarboxamide by hydrolysis-imidation. Also, pyrazine-2-carboximidate methanolyzed in methanol solvent and the present of ZnCl₂.7H₂O.

CONFLICTS OF INTEREST

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