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Group IIB Metal Directed Complexes with Flexible Benzimidazole-Based Connector[1]

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Abstract—Four group IIB metal complexes, namely $[ZnCl_2(Mbibix)]_n$ (**I**), $[Cd_2(Ac)_4(Mbibix)_2] \cdot (H_2O)$ (**II**), $[HgCl_2(DMF)(Mbibix)]_2$ (**III**) and $[HgI_2(Mbibix)]_2$ (**IV**), have been achieved via the reaction of corresponding metal salts and organic spacer Mbibix (Mbibix = 1-(3-((2-methyl-1*H*-benzimidazol-1-yl)methyl)benzyl)-2-methyl-1*H*benzimidazole). Crystal structure analyses reveal that complex **I** is one-dimensional (1D) zigzag chain, whereas **II**, **III** and **IV** are binuclear motifs (CIF files CCDC nos. 1006224 (**I**), 1006225 (**II**), 1006226 (**III**), 1006227 (**IV**)). These structures are further stacked into 3D supramolecular architectures via hydrogen bonding and $\pi \cdots \pi$ interactions. The result indicates that the metal centers imparted their influences on the structures and fluorescence behaviors.

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INTRODUCTION

The design and syntheses of well-defined supramolecular architectures has been of increasing interest, which makes chemists to construct novel materials with molecular-level precision [1–3]. Such material with expected topologies and properties is affected by several factors, in which metal coordination sites, as well as the nature of organic building blocks have been proved significant. Consequently, metal-driven assembly is currently considered as a powerful tool to design intriguing supramolecular architectures, because it bears regulated coordination preference and the profound effect on physical and chemical properties of structures [4, 5]. For example, the metal-directed M-TCNQF$_4$ nanowires, electroactive cage have been achieved [6–9], and the effect of metal centers on the conformation of organic spacer have been also studied in the reported $[Zn(Btdc)(2,20\text{-}Bipy)]_n$ and $\{[Cd_2(Btdc)_2(2,20\text{-}Bipy)] \cdot DMF\}_n$ complexes [10]. Generally, due to the coordination lability, Zn(II) and Cd(II) atoms can readily accommodate all kind of architectures and a selection of topological types of 1D, 2D and 3D motifs are given; by contrast, the formation of polymers with Hg(II) center is disproportionately sparse.

Much effort has been devoted to the synthesis of coordination architecture containing group IIB metal centers for their potential photoluminescence behaviors, in which the current prevalent topics is to rationally design organic spacer with efficient coordination groups and proper conjugated systems [11, 12]. The organic spacer takes on conformation freedom responsive to donor affinity of different metal centers, which results in a remarkable class of materials bearing diverse molecular structures and functions [13]. Rich possibilities of unusual coordination geometries with interesting spectral properties may be anticipated for the incorporation of two equivalent bulky and aromatic benzimidazole moieties. In this article, such units are extended via 1,3-dimethylene-benzene platform giving rise to a ditopic organic connector. The presence of $-CH_2-$ segment endows the ditopic connector's freely bending and rotating to satisfy metal coordination tendency, which often causes structural versatility [14]. Consequently, four new complexes with distinct structural features, $[ZnCl_2(Mbibix)]_n$ (**I**), $[Cd_2(Ac)_4(Mbibix)_2] \cdot (H_2O)$ (**II**), $[HgCl_2(DMF)(Mbibix)]_2$ (**III**) and $[HgI_2(Mbibix)]_2$ (**IV**) (Mbibix = 1-(3-((2-methyl-1*H*-benzimidazol-1-yl)methyl)benzyl)-2-methyl-1*H*-benzimidazole), were constructed, in which the metal centers impart their effect on the structures and luminescence behaviors.

EXPERIMENTAL

Materials and methods. All chemicals with reagent-grade quality were purchased from commercial sources and used without further purification. Mbibix was synthesized according to the previously methods [15]. IR data were recorded on a Bruker tensor 27 spectrophotometer with KBr pellets in the region of 400–4000 cm^{-1}. Elemental analyses (C, H, and N) were carried out on a Flash EA 1112 elemental

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analyzer. Thermogravimetric experiment was performed on a Mettler toledo TGA/SDTA instrument at a heating rate of 10°C min^{-1}.

Synthesis of I. A solution of $ZnCl_2$ (0.1 mmol, 0.0136 g) in 5 mL methanol was mixed with a equiv of Mbibix (0.1 mmol, 0.0365 g) in DMF (2 mL). The resulting mixture was left at room temperature for three weeks to yield colorless rod crystal **I**. The yield was 45% based on Zn.

For $C_{24}H_{22}N_4Cl_2Zn$

anal. calcd., %:	C, 57.34;	H, 4.41;	N, 11.14.
Found, %:	C, 57.14;	H, 4.46;	N, 11.28.

IR (KBr; ν, cm^{-1}): 3503 w, 2929 w, 1670 m, 1612 m, 1461 s, 1412 s, 1350 m, 1292 w, 1019 m, 752 s.

Synthesis of II, III and IV follows the similar procedure except that the corresponding metal salt was used: $Cd(Ac)_2 \cdot 3H_2O$ for **II** (0.1 mmol, 0.0231 g); $HgCl_2$ for **III** (0.1 mmol, 0.0271 g); HgI_2 for **IV** (0.1 mmol, 0.0454 g). The yield of colorless block crystals was: 43% (**II**) based on Cd; 52% (**III**) and 58% (**IV**) based on Hg.

For $C_{56}H_{56}Cd_2N_8O9$ (**II**)

anal. calcd., %:	C, 55.59;	H, 4.67;	N, 9.26.
Found, %:	C, 55.47;	H, 4.58;	N, 9.35.

IR (KBr; ν, cm^{-1}): 3446 m, 3023 w, 1563 s, 1506 m, 1480 m, 1415 s, 1335 m, 1162 w, 1015 w, 766 s, 678 s.

For $C_{54}H_{58}N_{10}O_2Cl_4Hg_2$ (**III**)

anal. calcd., %:	C, 45.61;	H, 4.11;	N, 9.85.
Found, %:	C, 45.52;	H, 4.13;	N, 9.77.

IR (KBr; ν, cm^{-1}): 3445 m, 2926 w, 1669 s, 1612 m, 1509 s, 1455 s, 1412 s, 1292 m, 1094 m, 1013 w, 757 s.

For $C_{48}H_{44}N_8I_4Hg_2$ (**IV**)

anal. calcd., %:	C, 35.12;	H, 2.70;	N, 6.83.
Found, %:	C, 35.23;	H, 2.78;	N, 6.77.

IR (KBr; ν, cm^{-1}): 3446 m, 1667 m, 1611 m, 1507 s, 1470 s, 1451 s, 1406 s, 1354 m, 1224 m, 1157 m, 1029 w, 983 w, 745 s.

X-ray structure determination. The single crystal suitable for X-ray determination was selected and mounted on a glass fiber. The data of **I** and **III** were collected at room temperature on a Rigaku Saturn 724 CCD with graphite monochromated MoK_α radiation (λ = 0.71073 Å). While **II** and **IV** were recorded on a SuperNova with graphite monochromated radiation CuK_α (λ = 1.54184 Å) for **II**, at 100 K; MoK_α radiation (λ = 0.71073 Å) for **IV**, at 160 K. The structures were solved by direct methods and expanded with Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement was based on observed reflections and variable parameters. All calculations were performed with the SHELXL-97 crystallographic software package [16–19]. Table 1 showed crystallographic crystal data and processing parameters for all complexes, and Table 2 listed their selected bond lengths and bond angles.

Supplementary material for structures **I–IV** has been deposited with the Cambridge Crystallographic Data Centre (nos. 1006224 (**I**), 1006225 (**II**), 1006226 (**III**), 1006227 (**IV**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The asymmetric unit of complex **I** consists of one Zn(II) center, one neutral Mbibix and two Cl^- anions. The Zn(II) atom lies in a distorted tetrahedral coordinated geometry which is completed by two chlorine anions and two nitrogen atoms from Mbibix. Each bidentate Mbibix bridges two Zn(II) centers to form a 1D zigzag pattern with Zn···Zn separation of 11.799 Å running in the *z* axis direction (Fig. 1). In Mbibix spacer, both methylene groups join the benzene ring and benzimidazole moieties, which make Mbibix spacer assume *trans* conformation to aggregate metal centers. The dihedral angles between the two methylbenzimidazole segments and the least-squares plane of phenyl group are 83.107° and 76.026°, respectively, with two methylbenzimidazole moieties twisted by 26.737°, which confirm that, based on the more conformational freedom offered by methylene, the methylbenzimidazole moiety adopts different direction to meet the metal coordination tendency.

It is anticipated that such π···π stacking interactions, together with other secondary interactions such as van der Waals' forces, hydrogen bonding, and electrostatic attractions, may function to facilitate the self-assembly of complexes. The halogen bonding interactions are present in **I** (Cl(2)···H(11)' 2.95, Cl(1)···H(12)' 2.96 Å), which extend the 1D chains into 2D supramolecular architecture in the *y*, *z* plane. Along the *x* axis, the intermolecular hydrogen bonds (Cl(1)···H(19)' 2.82 and Cl(2)···H(9)' 2.87 Å) further interlinked the 1D arrays into 3D supramolecular lattice [20].

The subtle change of experimental factor will affect the final structure, in which the replacement of zinc chloride in **I** by cadmium acetate results in a zero-dimensional binuclear species **II** and crystallized in the monoclinic crystal group P_2. As shown in Fig. 2, the asymmetric unit is consisted of one independent cadmium cation, one Mbibix, two carboxylate anions and half a water molecule. The coordination sphere of Cd(II) center is completed by two N-atoms from two

Table 1. Crystal data and structure refinement for complexes **I**–**IV**.

Parameter	Value			
	I	**II**	**III**	**IV**
Fw	502.73	1209.89	1422.08	820.85
Temperature, K	293(2)	100.01(10)	293(2)	160(10)
Crystal system	Monoclinic	Monoclinic	Triclinic	Triclinic
Space group	$P2_1/c$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$
a, Å	13.243(3)	11.8458(3)	9.4239(19)	9.3677(3)
b, Å	12.104(2)	16.9391(3)	12.763(3)	11.2635(5)
c, Å	15.767(3)	13.7822(4)	13.339(3)	11.6473(4)
α, deg	90	90	63.83(3)	86.979(3)
β, deg	113.99(3)	110.684(3)	86.63(3)	84.479(3)
γ, deg	90	90	72.04(3)	86.398(3)
V, Å^3	2309.2(8)	2587.24(11)	1364.1(5)	1219.48(8)
Z	4	2	1	2
ρ_{calcd}, g cm^{-3}	1.446	1.553	1.731	2.235
μ, mm^{-1}	1.314	7.127	5.867	8.861
$F(000)$	1032	1232	696	760
θ Range for data collection, deg	2.20–25.00	4.30–76.48	2.61–27.94	2.93–29.57
Reflection collected/unique (R_{int})	22708/4070 (0.1088)	11588/5325 (0.0423)	17508/6490 (0.0399)	10760/4982 (0.0325)
Reflections with $I > 2\sigma(I)$	3577	4331	5372	4374
Refined parameters	282	344	329	282
Goodness-of-fit on F^2	1.173	1.004	1.059	1.042
Final R_1, wR_2^*	0.0991, 0.1998	0.0344, 0.0782	0.0452, 0.0858	0.0305, 0.0606
Largest diff. peak and hole, eÅ^{-3}	0.653 and –0.317	0.442 and –0.773	0.605 and –1.191	2.225 and –1.147

* $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$; $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$.

different Mbibix and four carboxylate oxygen atoms from two chelated carboxylate moieties. Two adjacent Cd(II) centers are held together by paired Mbibix connectors to form a centrosymmetric binuclear motif with intramolecular Cd···Cd separation of 13.963 Å.

Molecule with functional carboxyl group for hydrogen bonding has been widely employed. Based on the C···O interactions (C···O 3.232, 3.206, 3.389 Å for C(5)–H(5)···O(1), C(7)–H(7)···O(2) and C(22)–H(22)···O(4)), such dimeric motifs are further extended into 3D supramolecular architecture. By comparison with **I** and **II**, the metal center with distinct coordination tendencies in the assembled process directs structural differences, which may dominate the final coordination architectures.

It is observed that the centrosymmetric binuclear complexes **III** and **IV** adopt very similar solid state organization. Also, the complex **III** is different from **I**, which indicates the radii and nature of metal centers can impart their influence on the group IIB metal complex structures. The fundamental building unit of complex **III** is defined by one Hg(II) center, one Mbibix molecule, two Cl$^-$ anions, and one DMF (Fig. 3a). Hg(II) center adopts distorted tetrahedral coordination geometry, which is completed by two N-atom derived from two Mbibix and two terminal Cl$^-$ anions. In binuclear **III**, the separation between adjacent Hg(II) centers is 13.9834 Å. Each Mbibix adopting *cis* form, the flexibility of methylene endows the dihedral angles between two terminal

Table 2. Selected bond lengths (Å) and angles (deg) for complexes **I**–**IV***

Bond	*d*, Å	Bond	*d*, Å
I			
Cl(1)–Zn(1)	2.256(2)	Cl(2)–Zn(1)	2.235(2)
N(1)–Zn(1)	2.048(6)	N(3)–Zn(1)	2.049(6)
II			
N(4)–Cd(1)	2.293(3)	Cd(1)–N(1)	2.303(2)
Cd(1)–O(1)	2.252(2)	Cd(1)–O(4)	2.323(2)
Cd(1)–O(2)	2.479(3)	Cd(1)–O(3)	2.317(3)
III			
Cl(1)–Hg(1)	2.4186(16)	Cl(2)–Hg(1)	2.4277(19)
Hg(1)–N(3)	2.269(4)	Hg(1)–N(1)$^{\#1}$	2.365(4)
IV			
Hg(1)–I(1)	2.6911(4)	Hg(1)–I(2)	2.6783(4)
Hg(1)–N(4)	2.366(3)	Hg(1)–N(1)	2.398(4)
Angle	ω, deg	Angle	ω, deg
I			
N(1)Zn(1)N(3)	106.4(2)	N(1)Zn(1)Cl(2)	109.75(17)
N(3)Zn(1)Cl(2)	114.59(17)	N(1)Zn(1)Cl(1)	110.19(17)
N(3)Zn(1)Cl(1)	104.43(18)	Cl(2)Zn(1)Cl(1)	111.29(9)
II			
N(4)$^{\#1}$Cd(1)N(1)	86.65(9)	N(4)$^{\#1}$Cd(1)O(4)	112.66(11)
N(4)$^{\#1}$Cd(1)O(2)	157.37(9)	N(4)$^{\#1}$Cd(1)O(3)	104.33(11)
III			
N(3)Hg(1)N(1)$^{\#1}$	85.78(14)	N(3)Hg(1)Cl(1)	115.52(11)
N(1)$^{\#1}$Hg(1)Cl(1)	107.36(11)	N(3)Hg(1)Cl(2)	116.68(11)
N(1)$^{\#1}$Hg(1)Cl(2)	109.78(11)	Cl(1)Hg(1)Cl(2)	116.63(6)
IV			
I(2)Hg(1)I(1)	136.930(13)	N(4)$^{\#1}$Hg(1)I(1)	111.34(9)
N(4)$^{\#1}$Hg(1)I(2)	103.20(10)	N(4)$^{\#1}$Hg(1)N(1)	90.92(13)
N(1)Hg(1)I(1)	97.95(9)	N(1)Hg(1)I(2)	106.6(10)

* Symmetry transformation used to generate equivalent atoms: $^{\#1}$ 2 – *x*, –*y*, 1 – *z* (for **II**); –*x* + 2, –*y* + 1, –*z* + 2 (for **III**); –*x*, 1 – *y*, –*z* (for **IV**).

methylbenzimidazole and benzene groups are 88.517° and 82.997°, respectively. Besides, the dihedral angle is 53.560° for two methylbenzimidazole segments in Mbibix spacer.

Complex **IV** is isostructural with complex **III**, and there is no solvent in the crystalline. The Mbibix spacer possesses *trans* conformation linking two adjacent Hg(II) centers. In the binuclear structure, the centroid distance between two benzene rings is 6.2552 Å, which is larger than that of complex **III**, and it may be the iodide ion with larger size affected the donor arrangement in Hg(II) coordination sphere (Fig. 3b).

For complex **III**, along the *y* axis, the centroid separations between the benzene ring and another imidazole ring of methylbenzimidazole groups is 3.734 Å with dihedral angle of 0.381°, which exhibits the existence of the π···π interactions. Besides, the hydrogen bond Cl(2)···H(17)' is equal to 2.86 Å, which is within the normal range of reported values [20]. The structure is further consolidated by extensive weak C–H···O (C(6)'···O(1) 3.380, C(17)'···O(1) 3.358, C(10)'···O(1) 3.433, C(16)'···O(1) 3.446 Å) and C–H···Cl (Cl(2)···H(29*C*)' 3.012 Å) interactions, such values are similar with those reported crystal structures [21–23]. These interactions extend the dimeric moieties into a 3D molecular architecture.

For complex **IV**, via *x* axis, the binuclear structures are stacked and linked together by the C–H···π interactions (H···π 2.903 Å) to sustain a supramolecular

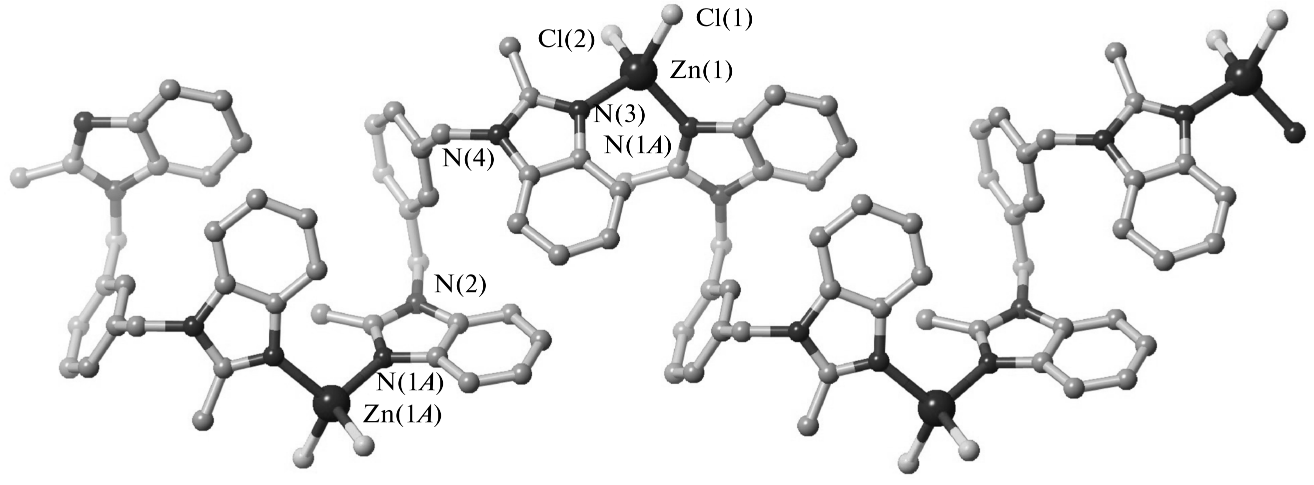

Fig. 1. The diagram showing the coordination environment for the Zn(II) center in 1D complex **I**. All the hydrogen atoms are omitted for clarity.

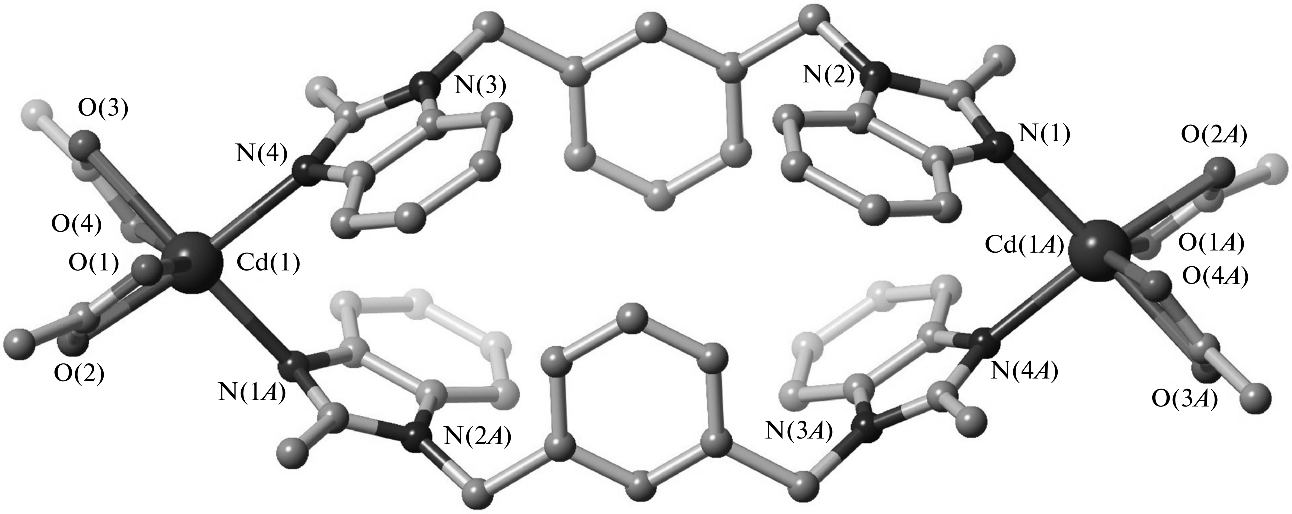

Fig. 2. Coordination arrangement of Cd(II) in complex **II**.

chain (Fig. 4). In the *x*, *z* plane, the π···π interactions between two parallel benzene ring of methylbenzimidazole groups with the centroid separation of 3.561 Å, and the distance of **I**(1)···H(9*A*) is 3.183 Å assist the solid state stabilization. In the *y* axis, the distance is 3.197 Å for I(2)···H(15), and similar C–H···I interactions have been discussed in other hydrogen bonded systems [24]. Upon these interactions, the 3D supramolecular lattice was generated. In **III** and **IV**, the different secondary interactions may function to facilitate the methyl-benzimidazole groups with contrary orientations.

Complexes **I**–**IV** are air-stable and can retain their crystalline integrity at ambient temperature. The TGA curve shows that the weight loss of 1.68% for **II** from 52 to 265°C (calcd. 1.49%) corresponds to the loss of one lattice water per formula, and the dehydrated complex is stable up to 265°C. Complexes including group IIB metal centers and aromatic organic tectons are considered as important candidates for solid photoluminescent material. Accordingly, solid state fluorescence properties of complexes **I**–**IV** were investigated at ambient temperature. As shown in Fig. 5, strong emissions were found at 370 (λ_{ex} = 298 nm) and 369 nm (λ_{ex} = 323 nm) for complexes **I** and **II**, respectively, which is in line with the reported Zn(II) or Cd(II) complexes [25–27]. By contrast, Mbibix shows strong fluorescence emission band at 309 nm, upon excited at 292 nm. Unfortunately, only weak emissions of complexes **III** (λ_{em} = 377 nm) and **IV** (λ_{em} = 397 nm) were observed, and the result imparts that the mercury ion as heavy metal atom does negatively

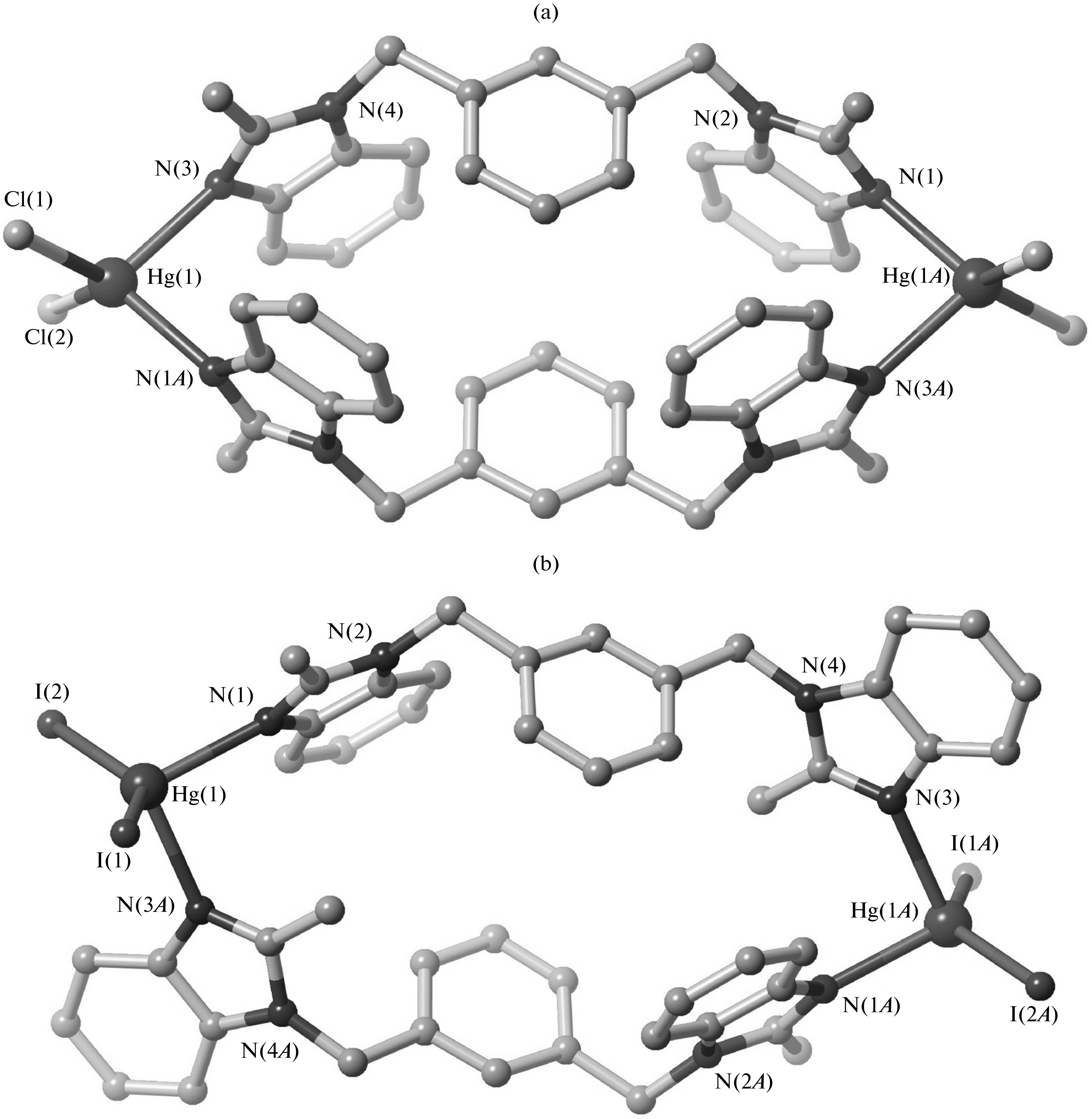

Fig. 3. Coordination arrangement of Hg(II) atom in complexes **III** (a) and **IV** (b).

impact the fluorescence of **III** and **IV** [28, 29]. In contrast to the free ligand, the emission maximums of complexes **I–IV** have changed, which may be attributed to the changes of HOMO–LUMO energy gap caused by the ligand coordinating to metal centers.

Thus, this work presents four group IIB metal complexes with different coordination motifs as well as the extended supramolecular architectures in virtue of various secondary interactions such as H-bonding, C–H···π and aromatic stacking. The metal centers impart their effects on the structures and fluorescence behaviors.

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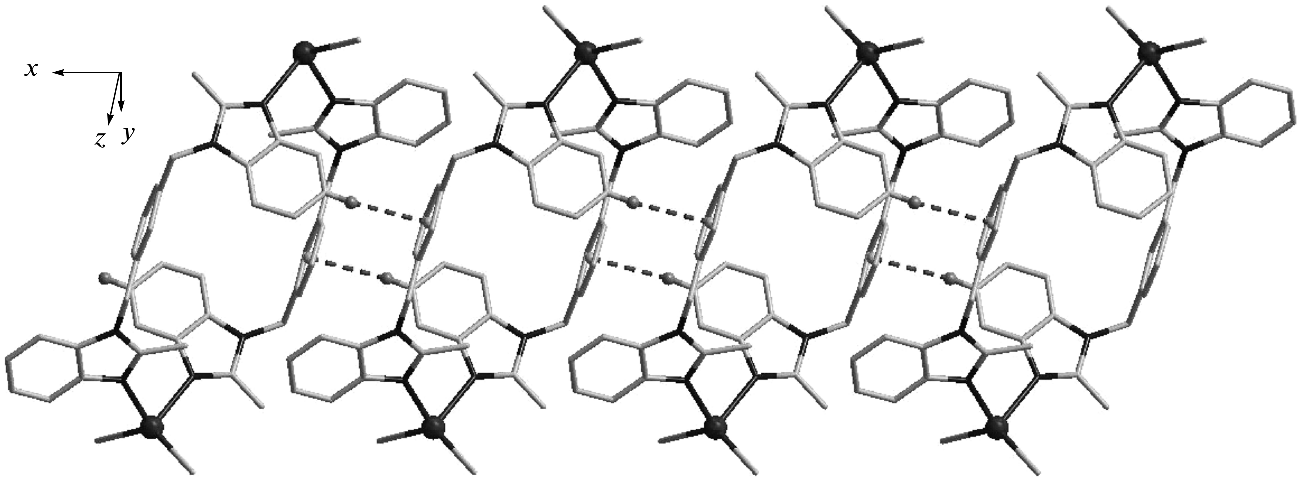

Fig. 4. The 1D supramolecular chain of complex **IV** through C–H···π interactions.

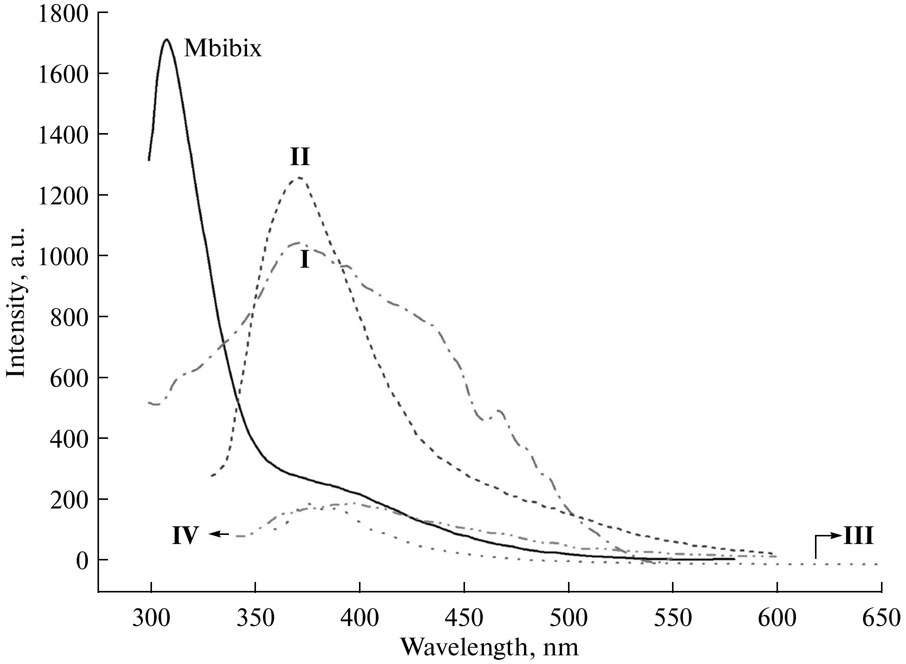

Fig. 5. The solid-state photoluminescence spectra of complexes **I–IV** (λ_{ex} = 298 (**I**), 323 (**II**), 345 (**III**), 332 (**IV**).

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