

## 两个含 1-苄基咪唑的 Zn(II)和 Cu(II)新配合物:合成和结构研究

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**摘要:** 1-苄基咪唑(L)与对苯二甲酸和  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  反应生成一维聚合链  $[\text{ZnL}_2(\text{TP})]_n$  (**1**),在这个配合物中通过链间的  $\text{C}-\text{H} \cdots \pi$  相互作用形成了二维超分子层。1-苄基咪唑(L)和  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  反应生成四核配合物  $[\text{Cu}_4(\mu_4\text{-O})\text{Cl}_6\text{L}_4]$  (**2**),在这个配合物中通过  $\text{C}-\text{H} \cdots \text{Cl}$  氢键形成了一维超分子链。测定了 **1** 和 **2** 的荧光发射光谱。

**关键词:** 1-苄基咪唑; 锌(II); 铜(II);  $\text{C}-\text{H} \cdots \pi$  相互作用; 氢键

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### Two New Zn(II) and Cu(II) Complexes Based on 1-Benzylimidazole: Synthesis and Structural Studies

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**Abstract:** Reaction of 1-benzylimidazole (L) and terephthalic acid ( $\text{H}_2\text{TP}$ ) with  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  led to 1D polymeric chains  $[\text{ZnL}_2(\text{TP})]_n$  (**1**) (TP = terephthalate). In the crystal packing of **1**, 2D supramolecular layers are formed via interchain  $\text{C}-\text{H} \cdots \pi$  contacts. Reaction of 1-benzylimidazole (L) with  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  afforded a tetranuclear complex  $[\text{Cu}_4(\mu_4\text{-O})\text{Cl}_6\text{L}_4]$  (**2**). In the crystal packing of **2**, 1D supramolecular chains are formed by  $\text{C}-\text{H} \cdots \text{Cl}$  hydrogen bonds. CCDC: 663257, **1**; 663256, **2**.

**Key words:** 1-Benzylimidazole; Zinc(II); Copper(II);  $\text{C}-\text{H} \cdots \pi$  Contacts; Hydrogen bonds

Metallo-organic coordination networks with various ligands are extensively studied due to their intriguing structural diversity and potential applications as functional materials<sup>[1-2]</sup>. Most of coordination polymers were prepared by simple assembly of metal ions and organic ligands. However, the concept of the secondary build as the metal ion replacement is applied by eminent success in the design of highly porous and rigid metal-organic frameworks<sup>[3]</sup>. In the construction of coordination architectures, besides the regular covalent bonds<sup>[4]</sup>, multiple weak non-covalent forces, such as hydrogen bonds<sup>[5-6]</sup> and  $\text{C}-\text{H} \cdots \pi$  contacts<sup>[7-8]</sup>, also play

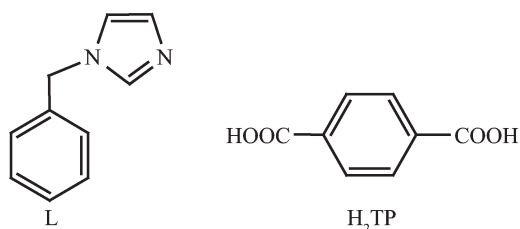
important roles, and they can further link discrete subunits or low-dimensional entities into high-dimensional supramolecular networks<sup>[9-10]</sup>. Ligands containing imidazolyl ring have received much attention for their wide-ranging antiviral activity<sup>[11]</sup>, and this kind of ligand can coordinate with a variety of transition metals to form one-, two- and three-dimensional coordination polymers through the use of nitrogen atom of imidazolyl<sup>[12-15]</sup>. Herein, we report the preparation, crystal structures, weak interactions and fluorescence emission spectra of two new complexes,  $[\text{Zn}(\text{L})_2(\text{TP})]_n$  (**1**) and  $[\text{Cu}_4(\mu_4\text{-O})\text{Cl}_6\text{L}_4]$  (**2**) (L=1-benzylimidazole, TP= tereph-

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thallate ion).

## 1 Experimental

### 1.1 Physical measurements

Melting points were determined with a Boetius Block apparatus. <sup>1</sup>H NMR spectra was recorded on a Varian Mercury Vx 300 spectrometer at 300 MHz with CDCl<sub>3</sub> as solvent. Chemical shifts,  $\delta$ , were reported in ppm relative to the internal standard TMS for <sup>1</sup>H NMR. Elemental analyses were measured using a Perkin-Elmer 2400C Elemental Analyzer. IR spectra (KBr) were taken on a Bruker Equinox 55 spectrometer. The luminescent spectra were conducted on a Cary eclipse fluorescence spectrophotometer.

### 1.2 Synthesis of 1-benzylimidazole (L)

A THF solution (40 mL) of imidazole (1.420 g, 20.8 mmol) was added to a suspension of oil-free sodium hydride (0.500 g, 20.8 mmol) in THF (40 mL) and stirred for 1 h at 60 °C. Then benzyl bromide (3.240 g, 19.0 mmol) was added dropwise to the above solution. The mixture was stirred for 22 h at 60 °C, and a brown solution was obtained. The solvent was removed with a rotary evaporator and H<sub>2</sub>O (50 mL) was added to the residue. Then the solution was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 30 mL), and the extracting solution was dried with anhydrous MgSO<sub>4</sub>. After removing CH<sub>2</sub>Cl<sub>2</sub>, a pale yellow solid was obtained. Yield: 2.690 g (90%), m.p. 76~78 °C. <sup>1</sup>H NMR  $\delta$  7.55 (s, 1H, 2-imH), 7.31-7.37 (m, 3H, PhH), 7.15 (d,  $J$  = 5.4, 2H, PhH), 7.09 (s, 1H, 4 or 5-imH), 6.90 (s, 1H, 4 or 5-imH), 5.12 (s, 2H, CH<sub>2</sub>) (imi = imidazole).

### 1.3 Synthesis of [Zn(L)<sub>2</sub>(TP)]<sub>n</sub> (1)

A *N,N*-dimethylformamide (DMF) solution (10 mL) containing terephthalic acid (H<sub>2</sub>TP) (80 mg, 0.5 mmol) was added to an aqueous solution (10 mL) of Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (148 mg, 0.5 mmol). After ca. 5 min of vigorous mixing, a DMF solution (8 mL) of 1-benzylimidazole (L) (158 mg, 1.0 mmol) was added and the pH value of the

solution was adjusted to ca. 7 by triethylamine. The filtrate of the above mixture was allowed to evaporate slowly under ambient condition, and colorless single crystals suitable for X-ray analysis were obtained within three weeks. Yield: 170 mg (31%), m.p. 240~242 °C. Anal. Calc. for C<sub>28</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub>Zn (%): C, 61.61; H, 4.43; N, 10.26. Found(%): C, 61.83; H, 4.27; N, 10.45. IR (KBr, cm<sup>-1</sup>): 3 123(s), 3 058(m), 3 029(m), 2 942(w), 2 888(w), 1 702(m), 1 598(m), 1 522(s), 1 456(s), 1 402(s), 1 231(m), 1 110(vs), 1 093(s), 1 022(m), 955(m), 716(vs), 707(vs), 654(m), 570(m).

### 1.4 Synthesis of [Cu<sub>4</sub>( $\mu_4$ -O)Cl<sub>6</sub>L<sub>4</sub>] (2)

A methanol solution (10 mL) containing 1-benzylimidazole (L) (158 mg, 1.0 mmol) was added to an aqueous solution (10 mL) of CuCl<sub>2</sub>·2H<sub>2</sub>O (170 mg, 1.0 mmol). After ca. 30 min of vigorous mixing, the filtrate was allowed stand at room temperature. The yellow block-shaped crystals were deposited slowly with the evaporation of the solvent within one week. Yield: 90 mg (36%), m.p. 254~256 °C. Anal. Calcd for C<sub>40</sub>H<sub>40</sub>Cl<sub>6</sub>Cu<sub>4</sub>N<sub>8</sub>O (%): C, 43.06; H, 3.61; N, 10.04. Found(%): C, 43.54; H, 3.96; N, 10.33. IR (KBr, cm<sup>-1</sup>): 3 137(m), 3 062(m), 3 025(m), 2 942(w), 2 809(w), 2 664(w), 2 540(w), 1 685(s), 1 612(s), 1 384(vs), 1 354(vs), 1 298(s), 1 242(m), 1 110(m), 1 086(m), 947(m), 831(m), 751(m), 729(m), 644(m).

### 1.5 X-ray Data Collection and Structure determinations

Single-crystal X-ray diffraction measurements of **1** (0.28 × 0.24 × 0.20 mm<sup>3</sup>) and **2** (0.28 × 0.22 × 0.20 mm<sup>3</sup>) were collected on a Bruker SMART 1000 CCD diffractometer operating at 50 kV and 30 mA using Mo *K* $\alpha$  radiation (0.071 073 nm). Data collection and reduction were performed using the SMART and SAINT software<sup>[16]</sup> with frames of 0.6° oscillation in the range 1.8° <  $\theta$  < 25°. An empirical absorption correction was applied using the SADABS program<sup>[17]</sup>. The structures were solved by direct methods and all non-hydrogen atoms were subjected to anisotropic refinement by full-matrix least squares on  $F^2$  using the SHELXTL package<sup>[18]</sup>. All hydrogen atoms were generated geometrically (C-H bond lengths fixed at 0.093~0.097 nm), assigned appropriated isotropic thermal parameters and included in structure factor calculations. Selected bond

lengths (nm) and angles ( $^{\circ}$ ) and crystal data and 1 and Table 2.

structure refinement for **1** and **2**, are presented in Table CCDC: 663257, **1**; 663256, **2**.

**Table 1** Selected bond lengths (nm) and angles ( $^{\circ}$ ) for **1** and **2**

<b>1</b>					
Zn1-O1	0.196(1)	Zn1-N1	0.201(1)		
O1-Zn1-O3	94.7(1)	O1-Zn1-N1	125.8(8)	N1-Zn1-N2	103.5(1)
O1-Zn1-N1	104.6(8)				
<b>2</b>					
Cu1-O1	0.191(3)	Cu4-O1	0.190(2)	Cu1-Cl3	0.237(1)
Cu2-O1	0.190(2)	Cu1-N1	0.195(3)	Cu1-Cl4	0.243(1)
Cu3-O1	0.190(2)	Cu1-Cl2	0.247(1)		
O1-Cu1-N1	178.9(1)	Cl3-Cu1-Cl4	129.4(6)	Cl4-Cu1-Cl2	111.4(5)
O1-Cu1-Cl3	86.5(9)	O1-Cu1-Cl2	82.8(8)	Cu1-O1-Cu2	110.3(1)
N1-Cu1-Cl3	94.4(1)	Cu1-Cl2-Cu2	80.2(4)	Cu1-O1-Cu3	107.7(1)
O1-Cu1-Cl4	84.1(9)	N1-Cu1-Cl2	96.4(1)	Cu1-O1-Cu4	109.1(1)
N1-Cu1-Cl4	95.3(1)	Cl3-Cu1-Cl2	116.4(7)	Cu2-O1-Cu3	110.2(1)

**Table 2** Crystal data and structure refinements for **1** and **2**

	<b>1</b>	<b>2</b>
Complex		
Chemical formula	$C_{28}H_{24}N_4O_4Zn$	$C_{40}H_{40}Cl_6Cu_4N_8O$
Formula weight	545.88	1115.66
Crysal system	Monoclinic	Monoclinic
Space group	$C2/c$	$P2_1/c$
$a$ / nm	2.457 2(1)	1.2028 (6)
$b$ / nm	0.640 3(3)	0.999 0(5)
$c$ / nm	1.944 2(1)	3.823 0(2)
$\beta$ / ( $^{\circ}$ )	124.632(5)	93.748(7)
$V$ / nm <sup>3</sup>	2.517(2)	4.585(4)
$Z$	4	4
$D_c$ / (Mg·m <sup>-3</sup> )	1.441	1.616
$\mu$ / mm <sup>-1</sup>	1.018	2.222
$F(000)$	1128	2248
Crystal size / mm	0.28×0.24×0.20	0.28 × 0.22 × 0.20
$\theta_{min}$ , $\theta_{max}$ / ( $^{\circ}$ )	2.01, 25.03	1.94, 25.03
$T$ / K	293(2)	293(2)
No. of data collected	6 407	24 054
No. of unique data	2 222	8 090
No. of refined params	169	520
Goodness-of-fit on $F^2$ <sup>a</sup>	1.055	1.028
Final $R$ indices <sup>b</sup> [ $I > 2\sigma(I)$ ] $R_1$ , $wR_2$	0.029 3, 0.075 6	0.037 2, 0.082 2
$R$ indices (all data) $R_1$ , $wR_2$	0.034 0, 0.077 5	0.056 3, 0.087 4

<sup>a</sup> GOF=[ $\sum \omega(F_o^2 - F_c^2)^2 / (n-p)$ ]<sup>1/2</sup>, where  $n$  is the number of reflection and  $p$  is the number of parameters refined.

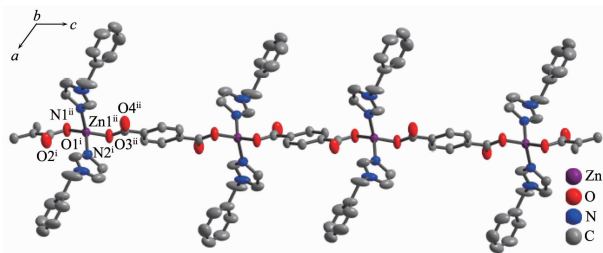
<sup>b</sup>  $R_1 = \sum (|F_o| - |F_c|) / \sum |F_o|$ ;  $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)]^{1/2}$ .

## 2 Results and discussion

### 2.1 Crystal structure of 1

1-benzylimidazole (L) was prepared from imidazole by alkylation with benzyl bromide, and obtained as a pale yellow solid. The ligand L is soluble in common organic solvents (such as  $\text{CH}_2\text{Cl}_2$ ,  $\text{CH}_3\text{OH}$ ,  $\text{CH}_3\text{CN}$  and DMF), therefore, crystallization of its complexes with inorganic metal salts occurs readily.

Complex **1** was prepared by the reaction of 1-benzylimidazole (L) and terephthalic acid with  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in the presence of  $\text{Et}_3\text{N}$  in DMF/ $\text{H}_2\text{O}$  solution. Complex **1** is stable and can retain their structural integrity at room temperature for a considerable length of time. Molecular structure of the complex **1** was demonstrated by X-ray analysis. In complex **1**, 1D polymeric chains are formed by  $\text{Zn}(\text{II})$  ions, bridging terephthalates and 1-benzylimidazole. The  $\text{Zn}(\text{II})$  is tetracoordinated with two oxygen donors from two different carboxyls and two nitrogen donors from two imidazyls to adopt the tetrahedral geometry (Fig.1a). The bond distances of  $\text{Zn1-O1}$  and  $\text{Zn1-N1}$  are 0.195(1) nm and 0.201(1) nm, respectively, and the bond angles around  $\text{Zn}(\text{II})$  center are in the range of  $94^\circ$ – $125^\circ$ . These values are comparable to analogous distances in other imidazole derivatives of  $\text{Zn}(\text{II})$  [19–20]. The X-ray crystal structural analysis of **1** shows that there are inversion centers in each  $\text{ZnL}_2\text{-TP-ZnL}_2$  unit. Dihedral angle of two imidazole rings bonded to same  $\text{Zn}(\text{II})$  center is  $58.2(1)$ . The two benzene rings from two adjacent terephthalates form the dihedral angle of  $38.5(2)^\circ$ . An interesting feature in the crystal packing of **1** is that 2-D supramolecular layers are formed via interchain  $\text{C-H}\cdots\pi$  contacts as shown in Fig.1b<sup>[7,8]</sup>, in which hydrogen

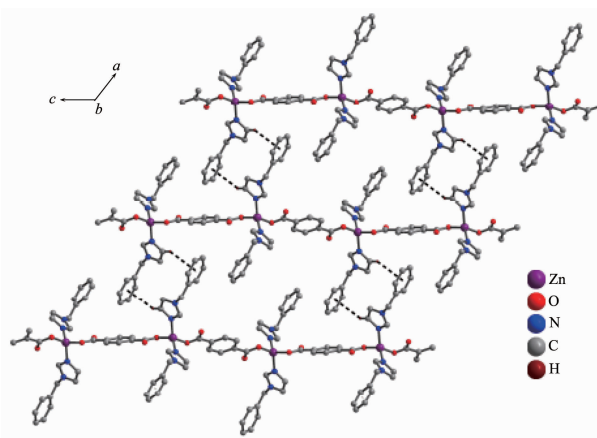


Symmetry operation: i:  $0.5+x, 0.5-y, -0.5+z$ ; ii:  $0.5-x, 0.5-y, -z$

All hydrogen atoms were omitted for clarity

Fig.1a 1D polymer chain of **1**

atoms are from imidazole rings ( $\text{H}\cdots\pi$  bond separation = 0.312(1) nm and  $\text{C-H}\cdots\pi$  angle =  $141.6(1)^\circ$ ).



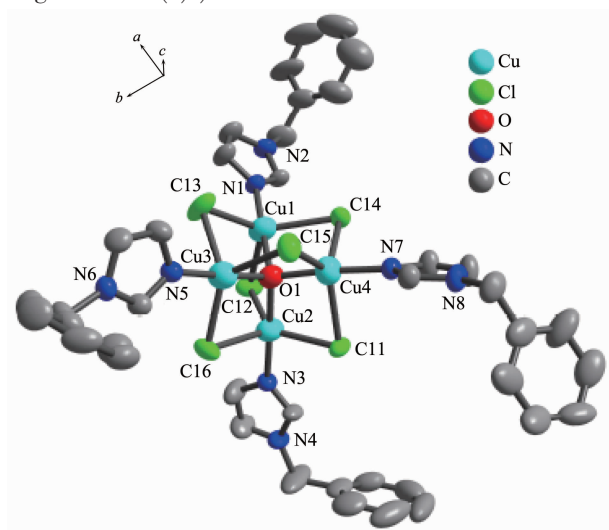
All hydrogen atoms except those participating in the  $\text{C-H}\cdots\pi$  contacts were omitted for clarity

Fig.1b 2D supramolecular layers formed via interchain  $\text{C-H}\cdots\pi$  contacts in **1**

### 2.2 Crystal structure of 2

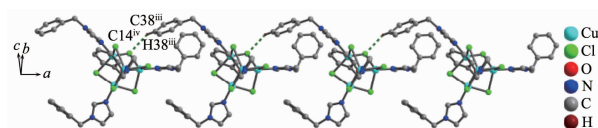
Complex **2** was prepared by reaction of 1-benzylimidazole (L) with  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in  $\text{CH}_3\text{OH}/\text{H}_2\text{O}$  solution. Complex **2** is stable and can retain their structural integrity at room temperature for a considerable length of time. Molecular structures of **2** was demonstrated by X-ray analysis. In **2**, a tetranuclear  $\text{Cu}_4(\mu_4\text{-O})$  core is formed via one oxygen atom and four copper(II) atoms, in which the oxygen atom lies in the center of core and four copper(II) lie in four tops of the tetrahedron (Fig.2a). Each pair of copper(II) center is bridged by a chlorine atom, and the six chlorine atoms form a larger octahedron geometry around the central  $\text{Cu}_4(\mu_4\text{-O})$  core. Additionally, the coordinated environment of each copper(II) atom is pentacoordinated with three bridging chlorine donors, one oxygen donor and one nitrogen donor to adopt a distorted trigonal bipyramid geometry, in which three bridging chlorine atoms lie in the equatorial plane, and oxygen atom and one nitrogen atom occupy axial sites of bipyramid. The  $\text{Cu-O}$  distances vary from 0.190(1) nm to 0.191(2) nm. The  $\text{Cu-Cl}$  distances are in the range of 0.235(2)–0.250(1) nm, and the  $\text{Cu-N}$  distances are in the range of 0.194(1)–0.196(1) nm. The  $\text{O-Cu-O}$  bond angles are in the range of  $108^\circ$ – $110^\circ$ , and the  $\text{O-Cu-N}$  angles range from  $174^\circ$  to  $179^\circ$ . The  $\text{Cu-Cl-Cu}$  angles

range from  $78.6^\circ$  to  $80.3^\circ$ , and the Cl-Cu-Cl angles range from  $102.5$  to  $134.1$ . The Cu $\cdots$ Cu mean distance is  $0.311(1)$  nm, and the O $\cdots$ Cl mean distance is  $0.295(1)$  nm. These values are comparable to analogous data reported<sup>[21-22]</sup>. Around the central Cu $_4(\mu_4\text{-O})$  core, the one imidazole ring with other three imidazole rings form the dihedral angles of  $69.6(3)^\circ$ ,  $55.4(3)^\circ$  and  $80.5(3)^\circ$ , respectively. Analysis of the crystal packing of **2** shows that 1D infinite chains are formed via the intermolecular C-H $\cdots$ Cl hydrogen bonds as shown in Fig.2b<sup>[5-6]</sup>, in which hydrogen atoms are from benzene rings (Cl $\cdots$ H separation =  $0.266(1)$  nm and C-H $\cdots$ Cl angle =  $147.7(1)^\circ$ ).



All hydrogen atoms were omitted for clarity

Fig.2a Perspective view of **2** and anisotropic displacement parameters depicting 30% probability



Symmetry operation: iii:  $x, 0.5-y, -0.5+z$ ; iv:  $-1+x, 0.5-x, -0.5+z$   
All hydrogen atoms except those participating in the hydrogen bonds were omitted for clarity

Fig.2b 1D supramolecular chain formed via C-H $\cdots$ Cl hydrogen bonds in **2**

### 2.3 Fluorescent emission spectra of L, **1** and **2**

As shown in Fig.3, the fluorescent emission spectra of L, **1** and **2** in dichloromethane at room temperature are obtained upon excitation at 230 nm. Ligand L shows a broad emission band centered at 345

nm, corresponding to intraligand transitions. For complexes **1** and **2**, the maximum emission bands are at 340 and 350 nm, respectively, and their fluorescence emission spectra are stronger than that of the corresponding ligand, which may be assigned to the result of the incorporation of metal-ligand coordination interactions. These results show that thus metal complexes might be candidates for potential photoactive materials<sup>[23-24]</sup>.

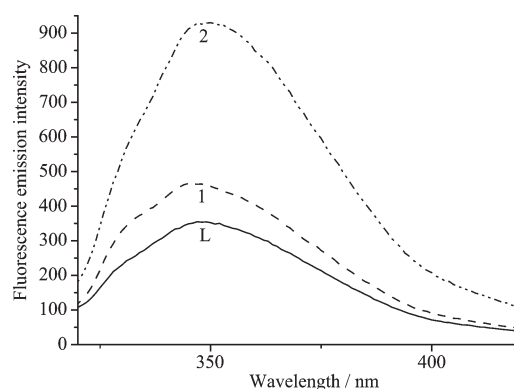


Fig.3 Emission spectra of L (—), **1** (---) and **2** (···) at 298 K in CH $_2$ Cl $_2$  ( $5.0 \mu\text{mol}\cdot\text{L}^{-1}$ ) solution

### 3 Conclusion

In summary, two new Zn(II) and Cu(II) complexes **1** and **2** have been prepared and characterized. In complex **1**, 1D polymeric chains are formed by Zn(II) ions, bridging terephthalates and 1-benzylimidazole. In the crystal packing of **1**, 2D supramolecular layers are formed via interchain C-H $\cdots$  $\pi$  contacts. In complex **2**, a Cu $_4(\mu_4\text{-O})$  core is formed by an oxygen atom lying in the center of core and four copper(II) lying in four tops of tetrahedron. In the crystal packing of **2**, 1-D infinite chains are formed through the C-H $\cdots$ Cl hydrogen bonds. The resultant structures of these complexes will provide interesting experimental data for supramolecular chemistry and crystal engineering. Further studies on new metal complexes from ligand L and analogous ligands are under way.

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