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Hydrothermal syntheses, characterizations, and luminescence of three new complexes based on 4,6-dibenzoylisophthalic acid

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Hydrothermal syntheses, characterizations, and luminescence of three new complexes based on 4,6-dibenzoylisophthalic acid

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Three new complexes with embellished dicarboxylate, $[\text{Cu}_2^{\text{II}}\text{L}_2(2,2'\text{-bpy})_2(\text{H}_2\text{O})_2]$ (**1**), $[\text{Co}_2^{\text{II}}\text{L}_2(2,2'\text{-bpy})_2(\text{H}_2\text{O})_2]$ (**2**), and $[\text{Ni}^{\text{II}}\text{L}(4,4'\text{-bpy})(\text{H}_2\text{O})]$ (**3**) ($\text{H}_2\text{L} = 4,6\text{-dibenzoylisophthalic acid}$, $2,2'\text{-bpy} = 2,2'\text{-bipyridine}$, $4,4'\text{-bpy} = 4,4'\text{-bipyridine}$), were synthesized under hydrothermal conditions and characterized by elemental analysis, IR spectra and X-ray diffraction. Complexes **1** and **2** are binuclear structures with 16-membered rings. Complex **3** shows a 2-D sheet structure. Intermolecular $\text{O-H}\cdots\text{O}$ interactions extend **1** and **2** into 1-D chains. Luminescence properties have been investigated for **1** and **2**.

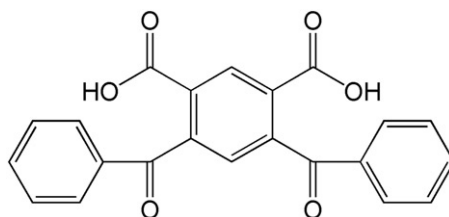
Keywords: Complexes; 4,6-Dibenzoylisophthalic acid; Luminescence

1. Introduction

Metal–organic frameworks (MOFs) have various architectures and potential applications in chemical separation, gas storage, ion exchange, luminescence, and heterogeneous catalysis [1–5]. Syntheses and properties of coordination polymers have attracted intense interest from chemists. Selection of ligands is very important in the construction of coordination polymers. As a family of multidentate O-donor ligands, organic aromatic carboxylates have been employed in the preparation of such metal–organic complexes [6–9]. In this article, we choose dicarboxylate with 2,2'-bipyridine and 4,4'-bipyridine as auxiliary ligands to give new complexes. 4,6-Dibenzoylisophthalic acid (H_2L , scheme 1) is selected for the six oxygens potentially available for metal coordination and the extended π -system from isophthalic acid which may be useful for luminescence of coordination polymers.

In previous work, we synthesized several coordination polymers with the functionalized isomorphous ligand [10–12]. Herein, we report the syntheses and structural characterizations of three new complexes, $[\text{Cu}_2^{\text{II}}\text{L}_2(2,2'\text{-bpy})_2(\text{H}_2\text{O})_2]$ (**1**), $[\text{Co}_2^{\text{II}}\text{L}_2(2,2'\text{-bpy})_2(\text{H}_2\text{O})_2]$ (**2**), and $[\text{Ni}^{\text{II}}\text{L}(4,4'\text{-bpy})(\text{H}_2\text{O})]$ (**3**). The fluorescence properties of **1** and **2** are studied.

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Scheme 1. The structure of H₂L.

2. Experimental

2.1. Materials and physical measurements

Reagents were purchased commercially and used without purification. H₂L was synthesized according to the literature [13]. FT-IR spectra were recorded using KBr pellets from 4000 to 400 cm⁻¹ on a Mattson Alpha-Centauri spectrometer. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 2400 Elemental Analyzer. The fluorescence spectra for **1** and **2** were obtained using a Cary Eclipse Luminescence spectrophotometer (VARIAN, USA).

2.2. Syntheses

2.2.1. Synthesis of [Cu^{II}L₂(2,2'-bpy)₂(H₂O)₂] (1**).** To a mixture containing H₂L (18.7 mg, 0.05 mmol), Cu(NO₃)₂ · 3H₂O (12.08 mg, 0.05 mmol) and 20 mL H₂O were added aqueous NaOH (0.05 mol L⁻¹) to adjust the pH to 4.1, and then 2,2'-bpy (7.8 mg, 0.05 mmol). The resulting mixture was stirred for 30 min, transferred to a Teflon-lined stainless steel vessel (25 mL), sealed, heated to 150°C, maintained at that temperature for 4 days, and cooled at a rate of 5°C h⁻¹ to room temperature. Blue cube-shaped crystals were obtained, washed by H₂O and air-dried (yield: 52% based on Cu(NO₃)₂ · H₂O). Anal. Calcd for C₆₄H₄₄N₄O₁₄Cu₂ (%): C, 62.94; H, 3.60; N, 4.58. Found: C, 63.05; H, 3.45; N, 4.60. IR (KBr, cm⁻¹): 3725s, 3056s, 1670s, 1610s, 1552m, 1446m, 1430w, 1340s, 865m, 796w, 765s.

2.2.2. Synthesis of [Co^{II}L₂(2,2'-bpy)₂(H₂O)₂] (2**).** To a mixture containing H₂L (18.7 mg, 0.05 mmol), Co(NO₃)₂ · 6H₂O (14.5 mg, 0.05 mmol) and 20 mL H₂O aqueous NaOH (0.05 mol L⁻¹) was added to adjust the pH to 6.0, and then 2,2'-bpy (7.8 mg, 0.05 mmol). The resulting mixture was stirred for 30 min, transferred to a Teflon-lined stainless steel vessel (25 mL), sealed, heated to 160°C, maintained at that temperature for 3 days, and cooled at a rate of 5°C h⁻¹ to room temperature. Red cube-shaped crystals were obtained, washed by H₂O, and air-dried (yield: 68% based on Co(NO₃)₂ · 6H₂O). Anal. Calcd for C₆₄H₄₄N₄O₁₄Co₂ (%): C, 63.42; H, 3.63; N, 4.62. Found: C, 63.56; H, 3.70; N, 4.57. IR (KBr, cm⁻¹): 3540s, 3060m, 1670s, 1598s, 1541m, 1368m, 1254m, 1042w, 767w, 737m, 702s.

Table 1. Crystal data and structure refinement for 1–3.

Complexes	1	2	3
Empirical formula	C ₆₄ H ₄₄ N ₄ O ₁₄ Cu ₂	C ₆₄ H ₄₄ N ₄ O ₁₄ Co ₂	C ₃₂ H ₂₂ N ₂ O ₇ Ni
Formula weight	1220.11	1210.89	605.23
Temperature (K)	293(2)	293(2)	293(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$	<i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions (Å, °)			
<i>a</i>	10.7978(11)	10.7384(10)	14.6052(13)
<i>b</i>	11.1925(12)	10.8177(2)	11.2097(7)
<i>c</i>	12.2253(13)	13.1070(5)	18.6370(18)
α	76.8120(10)	73.439(19)	90.00
β	68.0930(10)	66.630(118)	109.431(11)
γ	79.8660(10)	76.140(19)	90.00
Volume (Å ³), <i>Z</i>	1327.9(2), 1	1326.1(2), 1	2877.5(4), 4
Calculated density (g cm ⁻³)	1.526	1.516	1.397
Absorption coefficient (mm ⁻¹)	0.878	0.703	0.725
<i>F</i> (000)	626	622	1248
Goodness-of-fit on <i>F</i> ²	1.041	1.178	0.801
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ ^a = 0.0435, <i>wR</i> ₂ ^b = 0.0907	<i>R</i> ₁ ^a = 0.0405, <i>wR</i> ₂ ^b = 0.1292	<i>R</i> ₁ ^a = 0.0454, <i>wR</i> ₂ ^b = 0.0513
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0670, <i>wR</i> ₂ = 0.0975	<i>R</i> ₁ = 0.0430, <i>wR</i> ₂ = 0.1312	<i>R</i> ₁ = 0.1205, <i>wR</i> ₂ = 0.0568
Δ/σ (max/min)	0.288/−0.398	0.532/−0.364	0.517/−0.330

$$^a R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|. \quad ^b wR_2 = \Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]^{1/2}.$$

2.2.3. Synthesis of [Ni^{II}L(4,4'-bpy)(H₂O)] (3). To a mixture containing H₂L (18.7 mg, 0.05 mmol), Ni(NO₃)₂ · 6H₂O (14.5 mg, 0.05 mmol) and 20 mL H₂O aqueous NaOH (0.05 mol L⁻¹) was added to adjust the pH to 7.0, and 4,4'-bpy (9.6 mg, 0.05 mmol). The resulting mixture was stirred for 30 min, transferred to a Teflon-lined stainless steel vessel (25 mL), sealed, heated to 160°C, maintained for 3 days, and cooled at a rate of 5°C h⁻¹ to room temperature. Blue cube-shaped crystals were obtained, washed by H₂O, and air-dried (yield: 35% based on Ni(NO₃)₂ · 6H₂O). Anal. Calcd for C₃₂H₂₂N₂O₇Ni (%): C, 63.44; H, 3.63; N, 4.62. Found: C, 63.55; H, 3.68; N, 4.58. IR (KBr, cm⁻¹): 3423s, 1605s, 1564m, 1540w, 1412s, 1178m, 927w, 808s, 769m, 741m.

2.3. X-ray crystallography

Single crystals of 1–3 were glued to fiberglass for data collection on a SMART (Bruker, 2002) diffractometer equipped with a Mo-Kα radiation. Empirical absorption corrections were applied to the data using ABSCOR [14]. The structure was solved by direct methods followed by difference Fourier and refined by the full-matrix least-squares on *F*² using the SHELXXS-97 package [15]. All non-hydrogen atoms were refined anisotropically; hydrogens of organic ligand and coordinated water are located geometrically. The crystal data and structure refinement parameters for 1–3 are summarized in table 1; selected bond lengths and angles of 1–3 are listed in table 2.

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

Complex 1			
Cu1–O4	1.951(2)	Cu1–O1	1.9542(2)
Cu1–N2	2.002(2)	Cu1–N1	2.023(3)
Cu1#1–O1W	2.262(3)	Cu1–O4	1.951(2)
O4–Cu1–O1	92.37(8)	O4–Cu1–N2#1	167.34(10)
O1–Cu1–N2	96.25(9)	O4–Cu1–N1#1	89.98(10)
O1–Cu1–N1	171.83(10)	N2–Cu1–N1	80.25(11)
O4–Cu1–O1W#1	95.21(10)	O1–Cu1–O1W	96.44(11)
N2–Cu1–O1W	93.03(11)	N1–Cu1–O1W	91.13(11)
Complex 2			
Co1–O1	2.052(3)	Co1–O1W	2.071(4)
Co1–N1	2.105(4)	Co1–O4#2	2.115(3)
Co1–N2	2.122(4)	Co1–O3#2	2.373(3)
Co1–O3	2.373(3)	Co1–O4	2.115(3)
O1–Co1–O1W	99.93(16)	O1–Co1–N1	160.76(15)
O1W–Co1–N1	94.66(16)	O1–Co1–O4#2	93.33(13)
O1W–Co1–O4#2	93.40(16)	N1–Co1–O4#2	98.27(15)
O1–Co1–N2	90.82(15)	O1W–Co1–N2	90.46(16)
N1–Co1–N2	76.52(17)	O4–Co1–N2	173.78(15)
O1–Co1–O3#2	86.52(12)	O1W–Co1–O3#2	151.40(16)
N1–Co1–O3#2	86.62(14)	O4–Co1–O3#2	58.25(13)
N2–Co1–O3#2	117.49(14)		
Complex 3			
Ni1–N1	2.083(3)	Ni1–N2	2.049(2)
Ni1–O1	2.0307(19)	Ni1–O1W	2.081(2)
Ni1–O4	2.095(2)	Ni1–O3	2.1505(19)
Ni1–O3	2.1505(19)	Ni1–O4	2.095(2)
O1–Ni1–N2	94.05(12)	O1–Ni1–O1W	94.75(9)
N2–Ni1–O1W	91.92(13)	O1–Ni1–N1	87.21(12)
N2–Ni1–N1	178.74(12)	O1W–Ni1–N1	87.91(14)
O1–Ni1–O4	98.03(8)	N2–Ni1–O4	90.71(11)
O1W–Ni1–O4	166.73(9)	N1–Ni1–O4	89.18(12)
O1–Ni1–O3	159.54(8)	N2–Ni1–O3	93.26(12)
O1W–Ni1–O3	104.07(9)	N1–Ni1–O3	85.57(12)
O4–Ni1–O3	62.78(8)		

Symmetry mode: #1: $-x+1, -y+1, -z+2$, #2: $-x+1, -y+1, -z+1$.

3. Results and discussion

3.1. Structure description of **1**

Crystallographic analysis shows that **1** crystallizes in the triclinic space group $P\bar{1}$ with a binuclear structure. As shown in figure 1(a), Cu(II) is five-coordinate with two monodentate carboxylate oxygens (O1 and O4A) of two L^{2-} ligands, two nitrogens (N1 and N2) of 2,2'-bpy, and one coordination water molecule (O1W). The coordination geometry can be described as a slightly distorted square-pyramid with its equatorial plane defined by N1, N2, O1, and O4A and the axial position occupied by O1W. The Cu–O bond lengths (1.951(2)~2.262(3) Å) and the Cu–N bond lengths (2.002(2)~2.023(3) Å) are all within the normal range [16, 17]. Among them, the axial Cu1–O1W distance (2.262(3) Å) is slightly longer commensurate with Jahn–Teller sensitive Cu(II), comparable to that observed in other five-coordinate Cu(II) complexes [18–20]. The L^{2-} is monodentate (scheme S1a) in **1**. The two Cu(II) ions are linked by two L^{2-} anions through four monodentate carboxylates, giving Cu...Cu distance

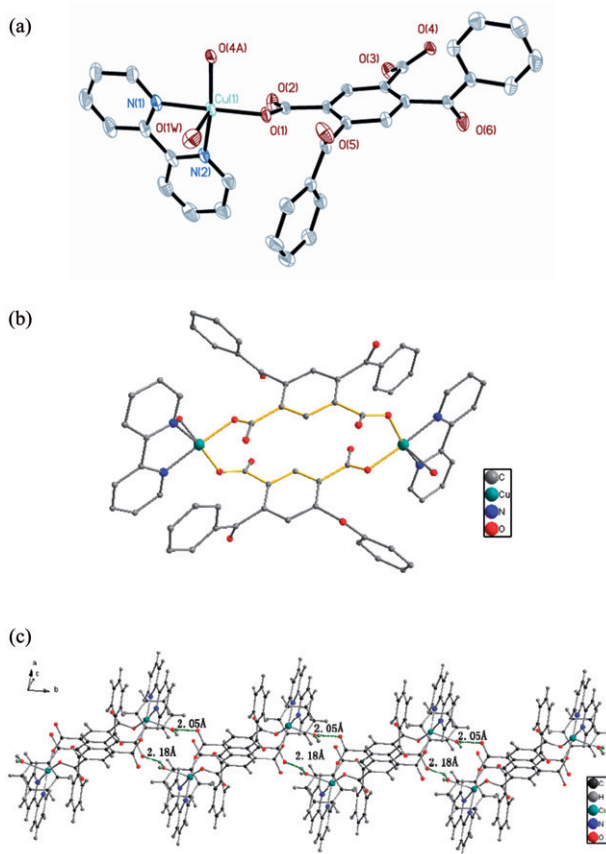


Figure 1. (a) Coordination environment of Cu(II) in **1**; symmetry codes: A: $1-x, 1-y, 2-z$, (b) the 16-membered ring in **1**, and (c) the 1-D chain *via* intermolecular O–H...O interactions in **1**.

of 9.196 Å and forming a 16-membered ring (figure 1b). A 1-D chain (figure 1c) is formed *via* intermolecular O–H...O interactions (table S1).

3.2. Structure description of **2**

As shown in figure 2(a), Co(II) is six-coordinate with a distorted octahedral geometry. Coordination involves two oxygens (O3A and O4A) from the chelating carboxylate of L^{2-} , two nitrogens (N1 and N2) of 2,2'-bpy, O1 from monodentate carboxylate of L^{2-} , and O1W of the aqua ligand. The Co–O/N bond distances are 2.052(3)–2.373(3) Å, in agreement with those of Co(II) analogs [21, 22]. The Co1–O3 distance is 2.373(3) Å and is the longest among the Co–O bond distances of **2** [23]. L^{2-} adopts chelate and monodentate coordination modes (scheme S1b) in **2**.

Complex **2** is similar to **1**, with a 16-membered ring (figure 2b) and two Co(II) ions linked by two L^{2-} through two monodentate and two chelate carboxylates, giving Co...Co distance of 9.274 Å. A 1-D chain (figure 2c) is formed *via* intermolecular O–H...O interactions (table S1) in **2**.

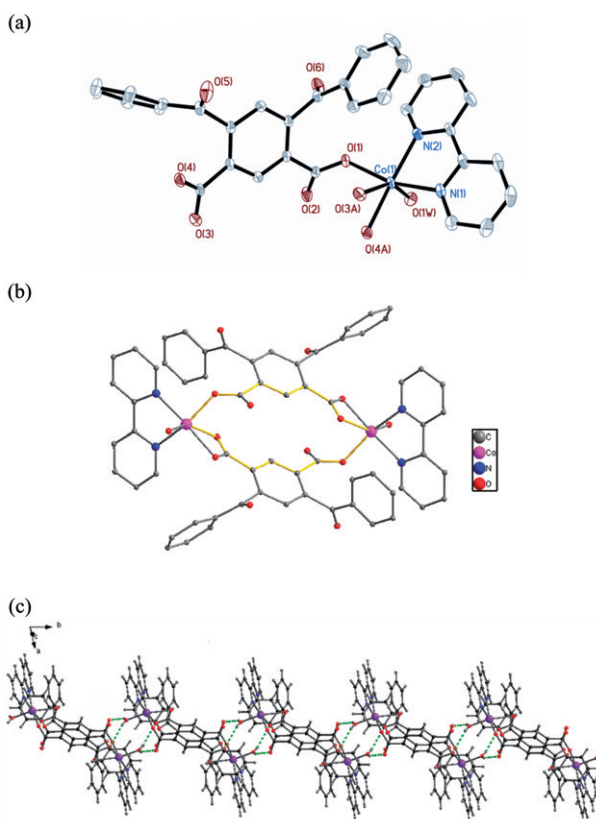


Figure 2. (a) Coordination environment of Co(II) in **2**; symmetry codes: A: $1-x$, $1-y$, $1-z$, (b) the 16-membered ring in **2**, and (c) the 1-D chain *via* intermolecular O-H...O interactions in **2**.

3.3. Structure description of **3**

Complex **3** crystallizes in monoclinic space group $P21/c$ with a 2-D layer structure. The asymmetric unit of **3** consists of one Ni(II), one L^{2-} , and one 4,4'-bpy. Each Ni(II) is six-coordinate with distorted octahedral geometry. The equatorial plane is defined by O1, O4A, O1W, and O3A and axial positions are occupied by N1 and N2 (N2–Ni1–N1 $178.74(12)^\circ$) (figure 3a). The Ni–O and Ni–N distances fall in the range $2.0307(19) \sim 2.1505(19) \text{ \AA}$ and $2.049(2) \sim 2.083(3) \text{ \AA}$, in agreement with those of Ni(II) analogs with carboxylic acid and 4,4'-bpy ligands [24, 25]. The L^{2-} in **3** exhibits chelate and monodentate coordination (scheme S1b) with Ni(II) to form wave-like 1-D covalent chains, which are linked into a 2-D sheet by rigid 4,4'-bpy (figure 3b).

3.4. Luminescence

The solid-state luminescence of **1** and **2** exhibit distinct emission bands at 380 and 385 nm (figure 4) upon excitation at $\lambda = 439$ and 425 nm, respectively. The solid-state photoluminescence of the free H_2L ligand ($\lambda_{em} = 480 \text{ nm}$, $\lambda_{ex} = 380 \text{ nm}$) was also analyzed (figure S1). It is clear that the emission bands of **1** and **2** are blue shifted

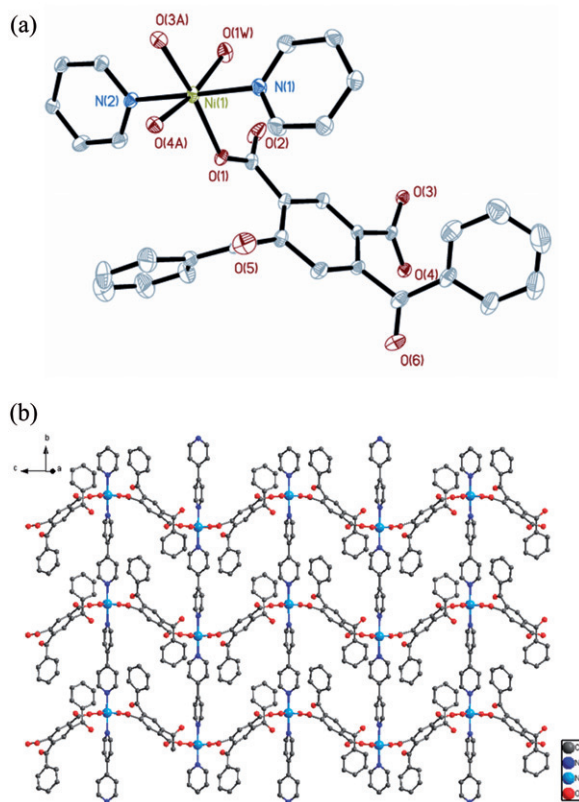


Figure 3. (a) Coordination environment of Ni(II) in **3**; symmetry codes: A: $x, 1/2 - y, -1/2 + z$ and (b) the 2-D sheet in **3**.

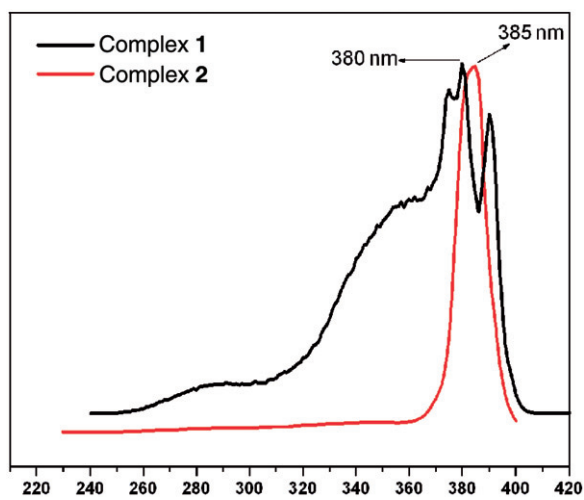


Figure 4. Solid-state photoluminescent spectra of **1** and **2**.

compared with free H₂L, suggesting intraligand π - π^* and charge-transfer transitions between the ligand and metal [26–28].

4. Conclusion

Three new complexes, [Cu^{II}L₂(2,2'-bpy)₂(H₂O)₂] (**1**), [Co^{II}L₂(2,2'-bpy)₂(H₂O)₂] (**2**), and [Ni^{II}L(4,4'-bpy)(H₂O)] (**3**), were synthesized and characterized; luminescence of **1** and **2** were examined. Complexes **1** and **2** would be candidates for photoactive materials. It is anticipated that more MOFs employing H₂L or other analogous ligands will be realized in functional materials.

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 705011, 713681, and 749248 for **1**, **2**, and **3**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44-1223-336033; Email: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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