

A d¹⁰ Metal Coordination Polymer Containing a Thiodiphthalic Ligand: Crystal Structure and Luminescent Property

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Abstract. A new two-dimensional metal-organic coordination polymer $[\text{Cd}_2(2,3,2',3'\text{-sdpa})(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$ (**1**) were hydro(solvo)thermally synthesized by the reaction of 2,3,2',3'-sulfonyldiphthalic acid (2,3,2',3'-H₄sdpa) with Cd(NO₃)₂·4H₂O and characterized by elemental analysis, thermogravimetry, IR and luminescence spectroscopy, and X-ray diffraction. In complex **1**, two kinds of cadmium atoms are linked to-

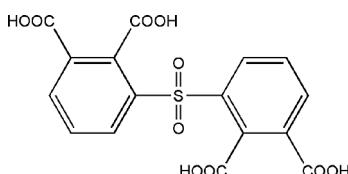
gether by anionic sdpa⁴⁻ ligands to form a 2D metal-organic network. The adjacent 2D layers further interact with each other through hydrogen bonds and $\pi\cdots\pi$ interactions to form a 3D supramolecular structure. The luminescence spectra and thermal properties of **1** were also investigated.

Introduction

Since controlling the molecular organization in the solid state can lead to materials with novel configurations and promising properties, the crystal engineering of metal-organic frameworks (MOFs) has grown into an active research area.^[1–7] The design and synthesis of metal-organic coordination polymers has become an exciting field in the past decade, and the reasonable design and selection of certain ligands are very crucial in the construction of desired coordination polymers.^[8,9] The self-assembly of multidentate organic ligands and metal ions has produced many MOFs, whose structures are affected by many factors such as the structural preference of metal ions, the shapes and sizes of the organic building blocks and solvent systems.^[10–13] Among previous reports, organic ligands with carboxylate groups are especially interesting because they can adopt a variety of coordination modes and result in diverse multidimensional structures.^[14–21] The carboxylate group can give rise to a wide variety of polynuclear complexes by bridging metal ions, so that organic aromatic polycarboxylate ligands were extensively used in the preparation of such metal-organic complexes with multidimensional networks and interesting properties.^[14,22–25] The flexible ligands have different conformations when they react with metal salts, and thus form metal complexes with great structural diversity.^[26] One of the keys to structural flexibility is to exploit the noncovalent interactions, such as hydrogen-bonding and $\pi\cdots\pi$ interactions, along with the directional covalent bond.^[27]

According to the situation above, in this paper we selected the relatively flexible sulfur-containing symmetrical ligand 2,3,2',3'-sulfonyldiphthalic acid (2,3,2',3'-H₄sdpa, Scheme 1)

to carry on a self-assembly and a novel 2D Cd^{II} coordination polymer was obtained. One of the features of this ligand is that it contains four carboxylate moieties; therefore the competition among these functional groups can result in novel multidimensional frameworks. Another noteworthy point for this ligand is that the relative flexibility of the sulfone functions is assumed to generate different torsion angles for the two phenyl planes and the carboxylate groups and further features various coordination conformations.



Scheme 1. Schematic representation of ligand 2,3,2',3'-sulfonyldiphthalic acid (2,3,2',3'-H₄sdpa).

In this study, we investigated one metal coordination polymer $[\text{Cd}_2(2,3,2',3'\text{-sdpa})(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$ (**1**) assembled from H₄sdpa ligand reacted with Cd(NO₃)₂·4H₂O. In this complex, two kinds of cadmium atoms are linked together by anionic sdpa⁴⁻ ligands to form a 2D metal-organic network. The adjacent layers further interact with each other through hydrogen bonding and $\pi\cdots\pi$ interactions to form a 3D supramolecular structure. The photoluminescent and thermal properties of complex **1** were studied as well.

Results and Discussion

Description of the Crystal Structure

Single-crystal XRD investigation revealed that $[\text{Cd}_2(2,3,2',3'\text{-sdpa})(\text{H}_2\text{O})_3]\cdot(\text{H}_2\text{O})_2$ (**1**) crystallizes in the triclinic system with the space group $P\bar{1}$. The metal-ligand connectivity pat-

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tern of complex **1** is depicted in Figure 1. The asymmetric unit of **1** contains two Cd^{II} ions, one 2,3,2',3'-sdpa⁴⁻ anion, three coordinated water molecules, and two solvated water molecules. The Cd1 ion is located in a distorted octahedral arrangement with O3, O4#1, O7#3, and O8#3 (from the carboxylate groups) on the equatorial plane and O2W and O3W (from the coordinated water) in the axial position (symmetry codes: #1: 2 - x , 2 - y , 2 - z ; #3: 2 - x , 2 - y , 1 - z). The Cd2 ion is also located in a distorted octahedral arrangement with O2, O5#2, O6#2, and O8#3 (from the carboxylate groups) on the equatorial plane and O1 (from the carboxylate groups) and O1W (from the coordinated water) in the axial position (symmetry codes: #2: 1 - x , 2 - y , 1 - z ; #3: 2 - x , 2 - y , 1 - z). As to 2,3,2',3'-sdpa⁴⁻, the dihedral angle between two phenyl rings is 74.1° and the band angle C₁-S₁-C₇ is 106.0°. The dihedral angles between the 2-, 3-, 2'-, and 3'-carboxylate groups and their corresponding phenyl rings are 68.3°, 27.3°, 68.4°, and 40.4°, respectively. The 3-COO⁻ group is dicoordinated, the 2-COO⁻, 2'-COO⁻, and 3'-COO⁻ groups are chelated and the 3'-COO⁻ group further bridges Cd1 and Cd2.

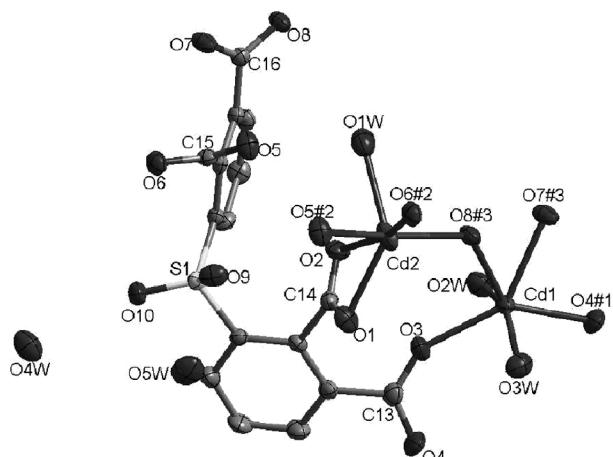


Figure 1. Connectivity pattern in the asymmetric unit of complex **1**. All hydrogen atoms are omitted for clarity. Symmetry codes: #1: $-x + 2, -y + 2, -z + 2$; #2: $-x + 2, -y + 2, -z + 1$; #3: $-x + 1, -y + 2, -z + 1$.

Each unit of complex **1** (shown in Figure 2) is comprised of four Cd^{II} ions, two 2,3,2',3'-sdpa⁴⁻ anions and six coordinated water molecules. As it is known, hydrogen bonding interactions are usually important in the synthesis of supramolecular architectures.^[28] The O5 atoms of 2'-COO⁻ groups form weak O(1W)-H(1WC)…O5 interactions with the water molecules, which play an important role in stabilizing the unit. The O…O distance is 2.688 Å and the O-H…O angle is 166.8°. The O4 atoms of the 3-COO⁻ groups from two neighboring sdpa⁴⁻ ligands connect the Cd1 atoms to form an infinite chain running along the *c* axis, as shown in Figure 3. Furthermore, there are weak O(3W)-H(3WB)…O(2W) interactions between the two coordinated watermolecules, which can further strengthen the skeleton of the chains.

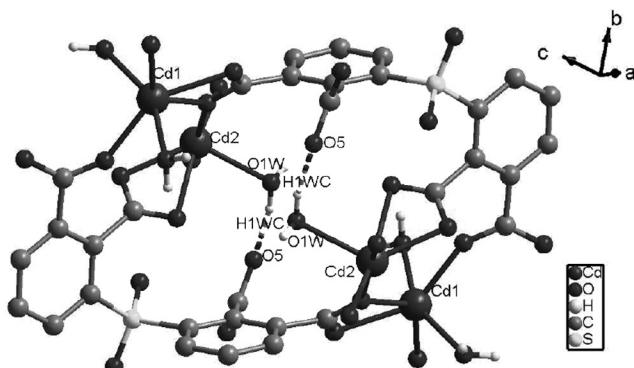


Figure 2. Unit of the cadmium coordination pattern of complex **1**.

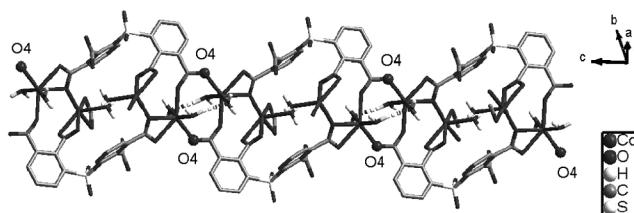


Figure 3. View of the one-dimensional cadmium coordination chain structure of complex **1**.

As shown in Figure 4, each Cd–sdpa chain connects to two other Cd–sdpa chains through the O5 and O6 atoms of 2'-COO⁻ groups to form a two-dimensional architecture in the *ac* plane, which layer is further stabilized by hydrogen bonding. The packing structure of complex **1** shows a three-dimensional supramolecular network via π…π stacking interactions (shown in Figure 5), which exist between the benzene ring and the benzene rings of neighboring ligands from different planes. The centroid…centroid distance between two rings is 3.551 Å. Further investigation of the structure shows that the structure is also stabilized by hydrogen bonds (geometrical parameters shown in Table 3).

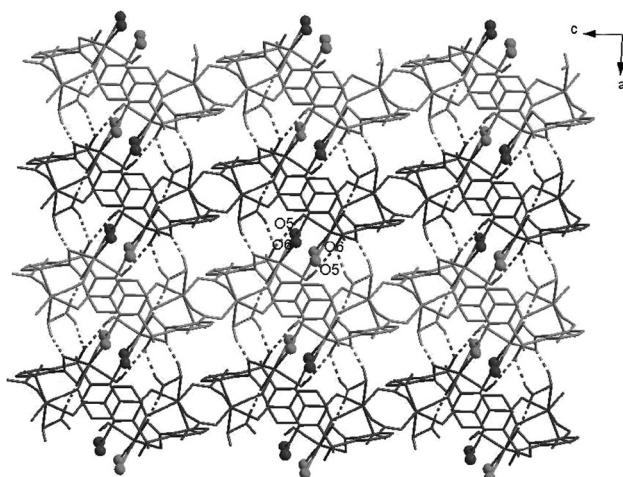


Figure 4. View of 2D layer structure of **1**.

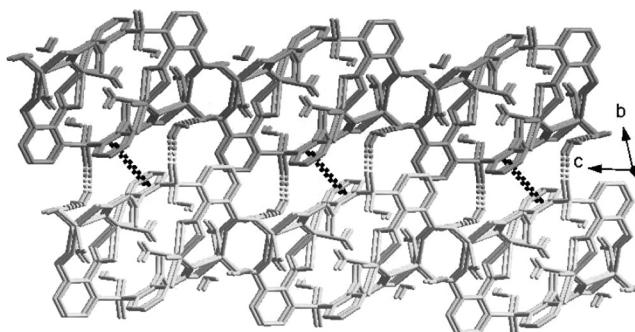


Figure 5. 3D supramolecular structure of complex **1**. Dashed lines indicate hydrogen bonds and $\pi\cdots\pi$ interactions.

Thermal Analysis of the Coordination Polymer **1**

Thermogravimetric analysis (TG) was carried out for complex **1**, and the result is shown in Figure 6. For **1**, the weight loss of 13.15 % (calcd. 12.77 %) below 360 °C corresponds to the loss of two solvent and three coordinated water molecules per formula. The second weight loss of 54.95 % from 360 to 440 °C corresponds to the release of one sdpa⁴⁻ (calcd. 55.31 %).

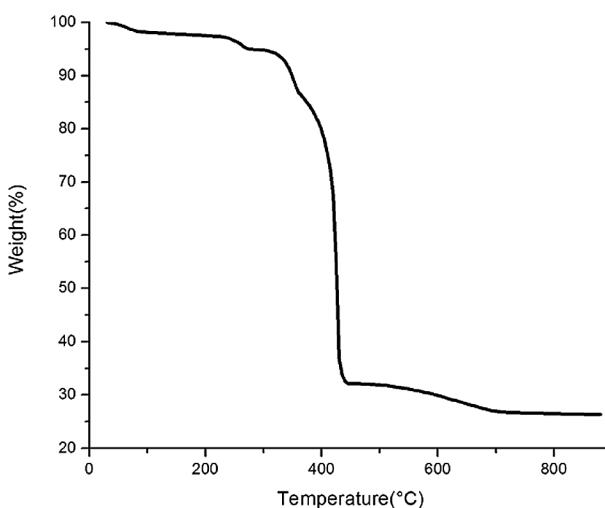


Figure 6. Thermogravimetric (TG) curve for complex **1**.

Photoluminescence Properties

Taking into account the excellent luminescent properties of d¹⁰ metal-organic polymers, the luminescent property of complex **1** was investigated in the solid-state at room temperature, as shown in Figure 7. Complex **1** exhibits an intense luminescent emission maximum at ca. 406 nm upon excitation at 355 nm, whereas the emission maximum of the ligand is at 396 nm in solid state.^[29] In comparison with the free ligand, the emission band of complex **1** is slightly red-shifted of about 10 nm, which is considered to mainly arise from the coordination of the Cd²⁺ ion and the ligand. The result suggests that the emission observed in complex **1** is neither metal-to-ligand

charge transfer (MLCT) nor ligand-to-metal charge transfer (LMCT) in nature. This phenomenon can be tentatively assigned to intraligand transfer $\pi^*\cdots\pi$ transitions (ligand-to-ligand charge transfer). Furthermore, the relative intensity of fluorescent emission for **1** is much stronger than that of the free ligand. The chelation of ligands to the metal atoms can effectively increase the conformational rigidity of the ligand and reduce the loss of energy by radiationless decay.^[30–32]

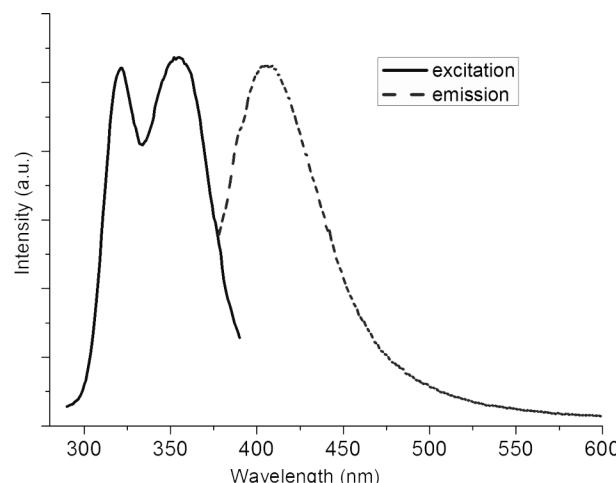


Figure 7. Excitation and emission spectra of **1** in solid state at room temperature.

Conclusions

In this paper, a new versatile multicarboxylate ligand was introduced to construct a novel coordination polymer under hydrothermal conditions. The results indicate that this ligand has the potential of forming 2D or multidimensional structures when it reacts with metal ions and plays an important role in mediating their optical properties. Complex **1** displays an intense emission peak and may be an excellent potential candidate for luminescent materials. It is believed that the results provide a promising access to the rational design and synthesis of metal-organic frameworks with specific structure and properties.

Experimental Section

Materials and General Methods: All reagents and solvents for syntheses were commercially available at analytical grade and used without further purification or with purification by standard methods prior to use. Elemental analyses for C and H were carried out with a Perkin-Elmer 240 elemental analyzer. The Fourier transform infrared (FT-IR) spectra were obtained in the range of 4000–400 cm⁻¹ as KBr pellets with a Bruker VECTOR 22 spectrometer. Thermogravimetric measurements were carried out from room temperature to 700 °C on crystalline samples in a nitrogen stream with a SDT 2960 thermal analyzer at a heating rate of 20 °C·min⁻¹. Luminescence spectra for the solid samples were recorded with a Hitachi 850 fluorescence spectrophotometer.

Synthesis of H₄sdpa and [Cd₂(2,3,2',3'-sdpa)(H₂O)₃]·2H₂O (1): Ligand 2,3,2',3'-sulfonyldiphthalic acid (2,3,2',3'-sdpa = H₄sdpa) was prepared according to the literature.^[29] Complex **1** was synthesized hydrothermally in a 23 mL Teflon-lined autoclave by heating a mixture of Cd(NO₃)₂·4H₂O (0.1 mmol, 30.8 mg), 2,3,2',3'-H₄sdpa (0.05 mmol, 19.7 mg), and one drop of Et₃N in water (10 mL) at 120 °C for 3 days under autogenous pressure. Afterwards, the reaction system was cooled to room temperature with a cooling rate of 5 °C·h⁻¹. Colorless block single crystals were collected in 74 % yield based on Cd(NO₃)₂·4H₂O.

Table 1. Crystal data and structure refinement parameters of coordination polymer **1**.

Empirical formula	C ₁₆ H ₁₆ Cd ₂ O ₁₅ S
Molecular mass	705.15
Temperature /K	296(2)
Crystal system	triclinic
Space group	P <bar{1}< td=""></bar{1}<>
Crystal size /mm	0.20 × 0.17 × 0.16
a /Å	7.5643(1)
b /Å	10.894(2)
c /Å	13.438(3)
α /°	73.83(3)
β /°	85.32(3)
γ /°	78.26(3)
V /Å ³	1040.9(4)
Z	2
D _{calcd.} /g·cm ⁻³	2.250
μ /mm ⁻¹	2.224
F(000)	688
Reflections collected	10369
Independent reflns	3634
R _{merg}	0.0213
Goodness-of-fit on F ²	1.109
Index ranges h, k, l	-8/8, -12/12, -15/15
Data / restraints / parameters	3634 / 0 / 307
R ₁ ^{a)} / wR ₂ ^{b)} [$I > 2\sigma(I)$]	0.0244 / 0.0506
R ₁ ^{a)} / wR ₂ ^{b)} (all data)	0.0283 / 0.0579
Largest diff. peak and hole /e Å ⁻³	0.831 / -1.002

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

Anal. found (calcd.) for C₁₆H₁₆Cd₂O₁₅S (705.15): C 27.16 (27.25); H 2.33 (2.29); S 4.52 % (4.54 %). IR (KBr pellets): $\tilde{\nu}$ = 3542 (m), 1617 (m), 1553 (s), 1462 (m), 1386 (s), 1312 (m), 1165 (m), 1150 (m), 1128 (w), 1097 (w), 911 (w), 865 (w), 774 (m), 705 (m), 613 (m), 573 (m), 451 (w) cm⁻¹.

Crystal Structure Determinations: Crystallographic data collections for complex **1** were carried out with a Bruker Smart Apex II CCD with graphite-monochromated Mo-K_α radiation ($\lambda = 0.71073$ Å) at room temperature using the ω -scan technique. Lorentz polarization and absorption corrections were applied. The structures were solved by direct methods with SHELXS-97 and refined with the full-matrix least-squares technique using the SHELXL-97 program. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms of the coordination water molecules, hydroxide groups, and ligands were included in the structure factor calculation at idealized positions by using a riding model and refined isotropically. The hydrogen atoms of the solvent water molecules were located from the difference Fourier maps, restrained at fixed positions and refined isotropically. Analytical expressions of neutral atom scattering factors were employed, and anomalous dispersion corrections were incorporated. Crystal data containing space group, lattice parameters and other relevant information for the title compound are summarized in Table 1. Relevant bond lengths and bond angles are displayed in Table 2. Geometrical parameters of hydrogen bonds are listed in Table 3.

CCDC-808450 contains the supplementary crystallographic data for the structure in this paper. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336-033; or E-Mail: deposit@ccdc.cam.ac.uk).

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Table 2. Selected bond lengths /Å and bond angles /° of **1**.

Cd(1)–O(4)#1	2.212(3)	Cd(1)–O3	2.234(3)
Cd(1)–O(3W)	2.268(3)	Cd(1)–O(2W)	2.300(3)
Cd(1)–O(7)#2	2.320(3)	Cd(1)–O(8)#2	2.422(3)
Cd(2)–O(8)#2	2.245(2)	Cd(2)–O(1)	2.260(3)
Cd(2)–O(1W)	2.315(3)	Cd(2)–O(6)#3	2.321(3)
Cd(2)–O(5)#3	2.370(3)	Cd(2)–O(2)	2.527(3)
O(4)#1–Cd(1)–O(3)	135.20(11)	O(4)#1–Cd(1)–O(3W)	90.91(12)
O(3)–Cd(1)–O(3W)	86.20(10)	O(4)#1–Cd(1)–O(2W)	88.24(12)
O(3)–Cd(1)–O(2W)	75.76(10)	O(3W)–Cd(1)–O(2W)	153.17(10)
O(4)#1–Cd(1)–O(7)#2	80.51(10)	O(3)–Cd(1)–O(7)#2	141.63(10)
O(3W)–Cd(1)–O(7)#2	110.67(11)	O(2W)–Cd(1)–O(7)#2	95.63(11)
O(4)#1–Cd(1)–O(8)#2	129.94(10)	O(3)–Cd(1)–O(8)#2	94.54(10)
O(3W)–Cd(1)–O(8)#2	86.52(10)	O(2W)–Cd(1)–O(8)#2	114.19(10)
O(7)#2–Cd(1)–O(8)#2	54.42(9)	O(8)#2–Cd(2)–O(1)	101.64(9)
O(8)#2–Cd(2)–O(1W)	93.52(10)	O(1)–Cd(2)–O(1W)	127.58(10)
O(8)#2–Cd(2)–O(6)#3	106.14(9)	O(1)–Cd(2)–O(6)#3	126.33(9)
O(1W)–Cd(2)–O(6)#3	95.63(10)	O(8)#2–Cd(2)–O(5)#3	161.91(9)
O(1)–Cd(2)–O(5)#3	90.12(10)	O(1W)–Cd(2)–O(5)#3	90.01(10)
O(6)#3–Cd(2)–O(5)#3	55.82(9)	O(8)#2–Cd(2)–O(2)	86.36(9)
O(1)–Cd(2)–O(2)	54.33(9)	O(1W)–Cd(2)–O(2)	77.37(10)
O(6)#3–Cd(2)–O(2)	166.17(9)	O(5)#3–Cd(2)–O(2)	111.72(9)

Symmetry codes: #1: -x + 2, -y + 2, -z + 2; #2: -x + 2, -y + 2, -z + 1; #3: -x + 1, -y + 2, -z + 1.

Table 3. Hydrogen bonds /Å, ° for complex **1**.

D-H···A	d(D-H)	d(H···A)	d(D···A)	∠(DHA)
O(5W)-H(5WB)···O(9)	0.85	2.02	2.821(4)	156.0
O(5W)-H(5WA)···O(2) ^{#4}	0.85	1.94	2.788(4)	171.7
O(4W)-H(4WB)···O(10)	0.85	2.27	2.978(4)	140.4
O(4W)-H(4WA)···O(4) ^{#5}	0.85	2.22	3.031(4)	158.4
O(3W)-H(3WB)···O(2W) ^{#1}	0.85	1.97	2.786(4)	159.4
O(3W)-H(3WA)···O(4W) ^{#6}	0.85	1.90	2.720(4)	162.6
O(2W)-H(2WB)···O(5W) ^{#7}	0.85	1.81	2.660(4)	172.3
O(2W)-H(2WA)···O(1) ^{#7}	0.85	1.84	2.665(4)	163.0
O(1W)-H(1WC)···O(5)	0.85	1.85	2.689(4)	166.8
O(1W)-H(1WA)···O(9) ^{#3}	0.85	2.40	3.229(4)	165.3

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