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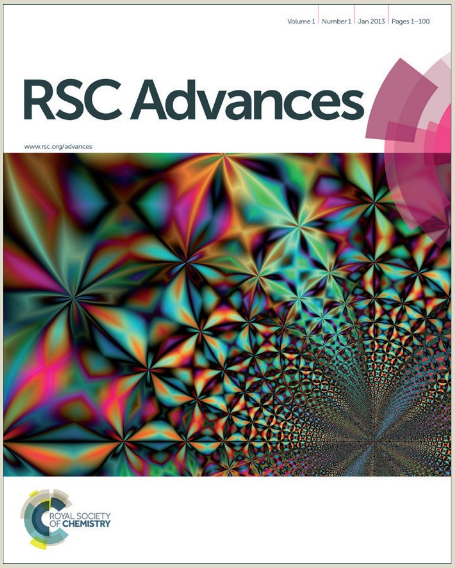

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A Novel Trinuclear Cd(II) Cluster-based Metal–Organic Framework: Synthesis, Structure and Luminescence Property

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A novel three-dimensional metal-organic framework (MOF), $[H_3O][Cd_3Cl(PZC)_3(H_2O)_3]$ (**1**), has been synthesized based on trinuclear Cd(II) cluster and H_2PZC ligand (H_2PZC = 1H-pyrazole-4-carboxylic acid). Compound 1 crystallizes in trigonal crystal system with the space group of *R*3*m*. Trinuclear Cd(II)-cluster as secondary building unit (SBU) are further connected by H_2PZC ligand to form a two-fold interpenetrated 3D framework. Compound **1** exhibits excellent water-stability and good luminescence properties for various organic solvents and different metal ions. The systematically investigating for luminescence indicates that compound **1** possesses unique performance for the detection of acetone, Fe^{2+} and Na^+ based on a fluorescence quenching and enhancing mechanism, thus it can be regarded as a potential luminescence probes for the detection of acetone, Fe^{2+} and Na^+.

Introduction

During the last decade, metal-organic frameworks (MOFs), as a novel functional material, have gained comprehensive attentions, not only because of their diverse topologies, but also potential applications in catalysis, gas storage, chemical separations, nonlinear optics, drug delivery, luminescence, and so on.[1-14] The interesting architectures of MOFs mainly rely on the assembly of various metals and linkers. Linkers usually employ organic ligands which possess N- or O-donors to design and synthesis.[15-28] In order to obtain stable and multifarious structures, introducing metal clusters SBU and interpenetration have turned out to be the effective methods. The method of designing metal clusters is intensely influenced by many factors, such as the coordination number of metal ions, various ligand, solvent, temperature and the pH of system.[29-44] Interpenetration usually reduces pore channel and adsorption capacity, but it also can give rise to other properties, for example, interpenetrating construction is in favor of improving its water stability.[45-49]

In this paper, we report a novel 3D Cd(II)-organic framework $[H_3O][Cd_3Cl(PZC)_3(H_2O)_3]$ (**1**) which assembled from trinuclear Cd(II)-cluster SBU and 1H-pyrazole-4-carboxylic acid (H_2PZC) ligand. The centers of trinuclear cadmium are occupied by the chlorine anion, the self-assembly of the trinuclear SBUs and ligands form a 2-fold interpenetrated 3D framework. Due to the interpenetration, compound **1** displays good water stability. By virtue of introducing d^{10} cadmium metal ion, the compound possesses excellent luminescent properties. It is worth pointing out that the sensitive luminescent phenomena were studied when compound **1** immersed in different organic solvents and metal ions solution. Powder X-ray diffraction (PXRD), thermogravimetric analyses (TGA) and differential thermal analysis (DTA), structures and luminescent properties were investigated in detail.

Experimental

Materials and methods

All chemicals were obtained from commercial sources and used without further purification. PXRD were collected on a Rigaku D/max-2550 diffractometer with Cu Kα radiation (λ = 1.5418 Å). Elemental analyses (C, H, and N) were achieved by vario MICRO (Elementar Germany). TGA and differential thermal analysis (DTA) were performed on NETZSCH STA449C thermogravimetric analyzer used in air with a heating rate of 10 °C min^{-1}. Fluorescence spectra were collected on a Fluoromax-4 spectrophotometer for the solid powder samples and **1**-solvent samples at ambient temperature. The slit width and filter of instrument is 4 nm and 450 nm, respectively.

Synthesis of compound 1

A mixture of $CdCl_2 \cdot 2.5H_2O$ (45.6 mg, 0.2 mmol), H_2PZC (11.2 mg, 0.1 mmol), DMF (2 mL), H_2O (1.5 mL) and EtOH (1.5 mL) were sealed in 25 mL Teflon-lined autoclave and heated at 110 ^oC for 3 days. After the mixture cooled to room temperature, needlelike colorless crystals were obtained with a yield of 61% (based on $CdCl_2 \cdot 2.5H_2O$). Elemental analysis (%) calc. for $C_{12}H_{15}Cd_3ClN_6O_{10}$, C 18.57, H 1.93, N 10.82; found: C 18.10, H 2.02, N 10.62. The experimental PXRD pattern of compound **1** agrees well with the simulated one based on the single-crystal X-ray data, indicating that compound **1** is a pure phase (Supporting Information Fig. S1).

X-ray Crystallography

Crystallographic data for compound **1** was measured by a Bruker Apex II CCD diffractometer using graphite-monochromated Mo-*K*α (λ = 0.71073 Å) radiation at room temperature. The structure was solved by direct methods and refined by full-matrix least squares on F^2 using version 5.1.[50] All the metal atoms were located first, and then the oxygen, nitrogen and carbon atoms of the compound were subsequently found in difference Fourier maps. The hydrogen atoms of the ligand were placed geometrically. All non-hydrogen atoms were refined anisotropically. The final formula was derived from crystallographic data combined with elemental and thermogravimetric analysis data. The detailed crystallographic data and selected bond lengths and angles for compound **1** are listed in Tables 1 and Table S2† respectively. Crystallographic data for **1** (1422767) has been deposited with Cambridge Crystallographic Data Centre. Topology information for **1** was calculated by TOPOS 4.0.[51]

Results and discussion

Structure description of compound 1

Single-crystal X-ray diffraction analysis reveals that compound **1** crystallizes in the trigonal crystal system with space group of *R*3*m*. As shown in Fig. 1, compound **1** possesses $[Cd_3(\mu_3\text{-}Cl)(CO_2)_3N_6]$ SBU with the bridging Cl^- residing at the centre of the three Cd atoms, while the three cadmium atoms are chelated by six crystallographic equivalent PZC^{2-} ligands and terminal coordinated by three water molecules. Every cadmium center coordinates two nitrogen atoms (N1, N2), two oxygen atoms (O1, O2) from three H_2L ligands, one oxygen atom (O3) from terminal coordinated water molecule, and one central chlorine ion (Cl1) from $CdCl_2 \cdot 2.5H_2O$ metal salt. The Cd-N and Cd-O bond lengths range in the normal distance of 1.927-1.940 and 1.938-1.947 Å, respectively. Each trinuclear unit is further connected to other neighboring units through six PZC^{2-} ligands to form a 3D two-fold interpenetrating framework.

For the purpose of illustrating the structure of compound **1** more clearly, its topology is further analyzed. The PZC^{2-} ligands are simplified as linear rods to connect two Cd SBUs, and the trinuclear SBU clusters can be regarded as 6-connected nodes (Fig. 2). Consequently, the structure of **1** can be described as a 6-connected network, which belongs to **pcu** topology with a Schläfli symbol of $(4^{12}.6^3)$.

Table 1 Crystal data and structure refinement for **1**

compound	**1**
formula	$C_{12}H_{15}Cd_3ClN_6O_{10}$
fw	775.95
temp (K)	293(2)
crystal system	Trigonal
space group	*R*3*m*
a (Å)	14.6793(6)
b (Å)	14.6793(6)
c (Å)	9.5183(5)
α (°)	90.00
β (°)	90.00
γ (°)	120.00
V (Å^3)	1776.24(14)
Z	3
Dc (Mg/m^3)	2.176
Absorption coefficient (mm^{-1})	2.839
F(000)	1110
Reflections collected/Unique (Rint)	3387 / 777 [R(int) = 0.0634]
Goodness on fit	1.202
Final R indices [I>2σ(I)]	$R_1 = 0.0279$, $wR_2 = 0.0767$
R indices (all data)	$R_1 = 0.0279$, $wR_2 = 0.0767$

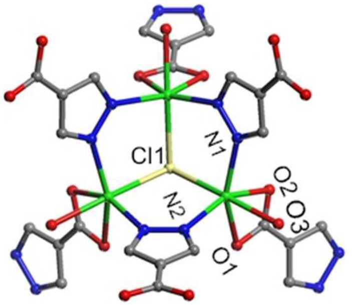

Fig. 1 Secondary building unit of compound **1**.

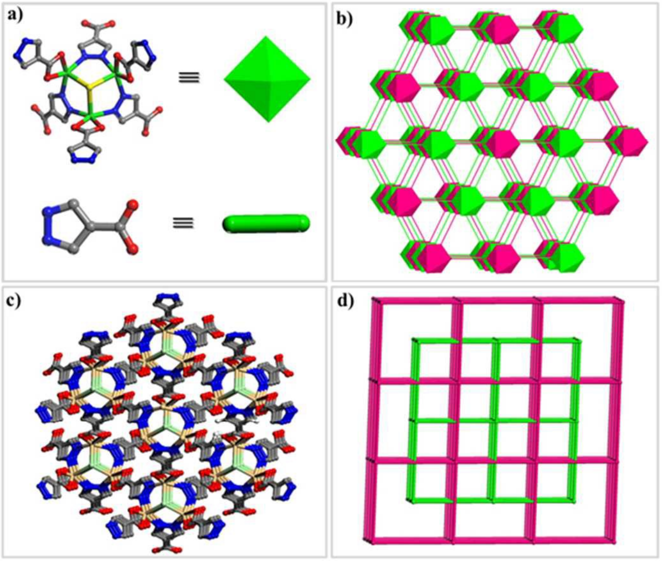

Fig. 2 Description of the structure of compound **1**: a) topology simplification of ligand and trinuclear cluster SBU; b) polyhedral view of the two-fold interpenetrated **pcu** net; c) ball and stick model of the 3D framework along the c-axis; d) schematic representation of the topology of interpenetrated net.

Thermogravimetric analysis

TGA and DTA measurement indicate that compound **1** exhibits three-step weight loss (Fig. 3). First slight weight loss of 9.2% below 300 ^{o}C may be ascribed to the removal of three terminal water and H_3O^+molecules (calcd 9.4%). The weight loss of 42.3% between 300 and 700 ^{o}C can be associated to the removal of organic ligands and structure collapse (calcd 41%). The last remaining at 700°C of 48.5% is attributed to the formation of CdO (calcd 49.6%).

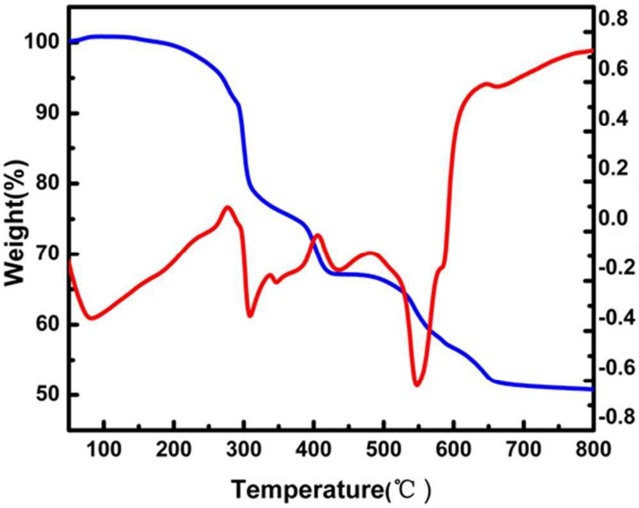

Fig. 3 TGA and DTA curves of compound **1**.

Compound **1** exhibits excellent water-stability, which due to the structure is a two-fold interpenetrating 3D framework. The crystals of compound **1** are soaked in water and kept at room temperature for 2h, 4h, 8h and 16 hours. It is worth noting that compound **1** still retain its crystallinity when it is immersed in water for 12 h, but a part framework of compound **1** collapsed after immersing it in water for 16 h, which can be proved by PXRD (Fig. 4).

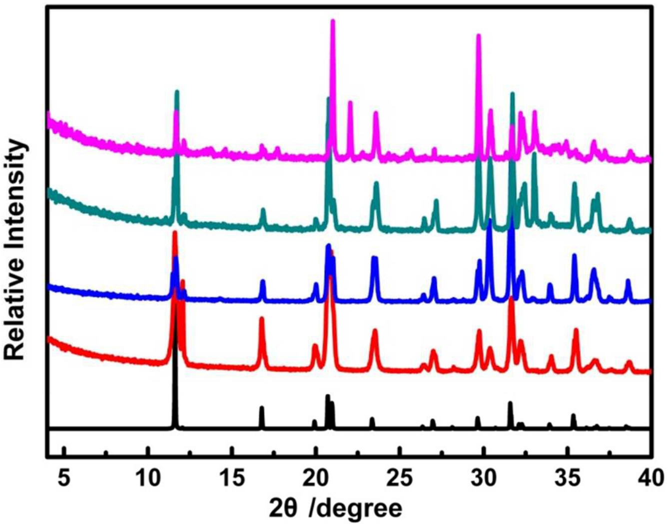

Fig. 4 The PXRD patterns of compound **1** for water stability; Simulated (black), in water for 2 h (red), 4 h (blue), 8 h (green), 16 h (pink).

Luminescent Properties

MOFs consisted of d^{10} metal ions own remarkable luminescent property and have potential to become promising luminescent materials.[52-53] So the luminescence properties of compound **1** and H_2PZC ligand were measured in the solid state at room temperature. As shown in Fig. 5, the free ligand is observed with emission maxima at 465 nm (λ_{ex} = 395 nm). Compound **1** exhibits the same emission characteristic with the ligands, and the emission peak is 468 nm (λ_{ex} = 395 nm). The enhancement of emission band in compound **1** should be ascribed to the interaction between H_2PZC ligand and the Cd center, which increased the conjugate level and reduced the non-radioactive loss. It is noticeable that the luminescent peak of compound **1** has slightly red-shift compared with the H_2PZC ligand, which could due to the reduction of the energy difference between HOMO and LUMO.[54]

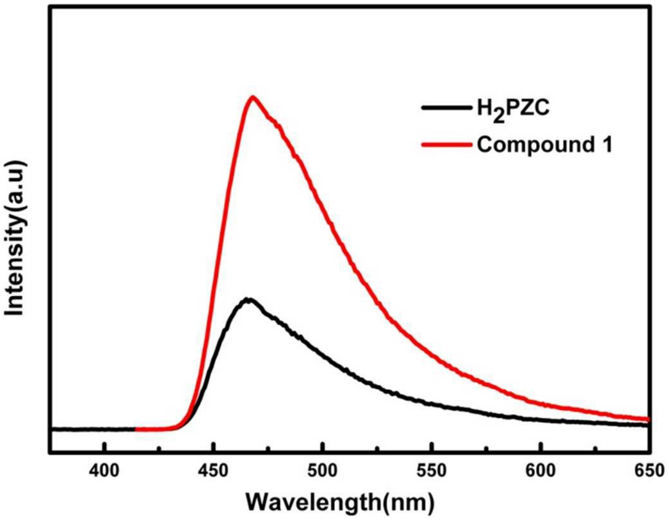

Fig. 5 Solid-state emission spectra of H_2PZC and the compound **1**

Further luminescence sensing measurements were investigated to explore the influence of a variety of guest molecules and metal ions towards compound **1**. The uniform powder of compound **1** was prepared by grinding 150 mg sample for 20 min. A series of solvent emulsions were prepared by introducing 3 mg ground powder sample of compound **1** into 3.0 mL acetone (CH_3COCH_3), dichloromethane (CH_2Cl_2), trichloromethane ($CHCl_3$), 1-propanol (1-PA), 2-propanol (2-PA), tetrahydrofuran (THF), acetonitrile (MeCN), methanol (MeOH), ethanol (EtOH), ethyl acetate (EtOAC), dimethyl sulfoxide (DMSO) and 1,4-Dioxane (DOA), respectively. By using ultrasound, the solvents and samples form stable emulsions before florescence study. As shown in Fig. 6a, all measure factors are same to that of compound **1**. The most noteworthy feature is that the luminescent wen is observably changing in different organic solvents. The acetone emulsions exhibits the most enhanced behavior,[55] which could be attribute to the interaction between the acetone solvent and compound **1** to increase the conjugate level. Nevertheless, the luminescent intensity in other solvent are all decrease, the order is Me_2CO > MeOH > CH_3Cl > EtOH > 1-PA> EtOAC> DMSO > MeCN >

2-PA > THF > DOA > CH_2Cl_2. Although the mechanism of response to organic solvents is still unclear, the interaction between organic solvents and crystals are essential to the excellent performance.

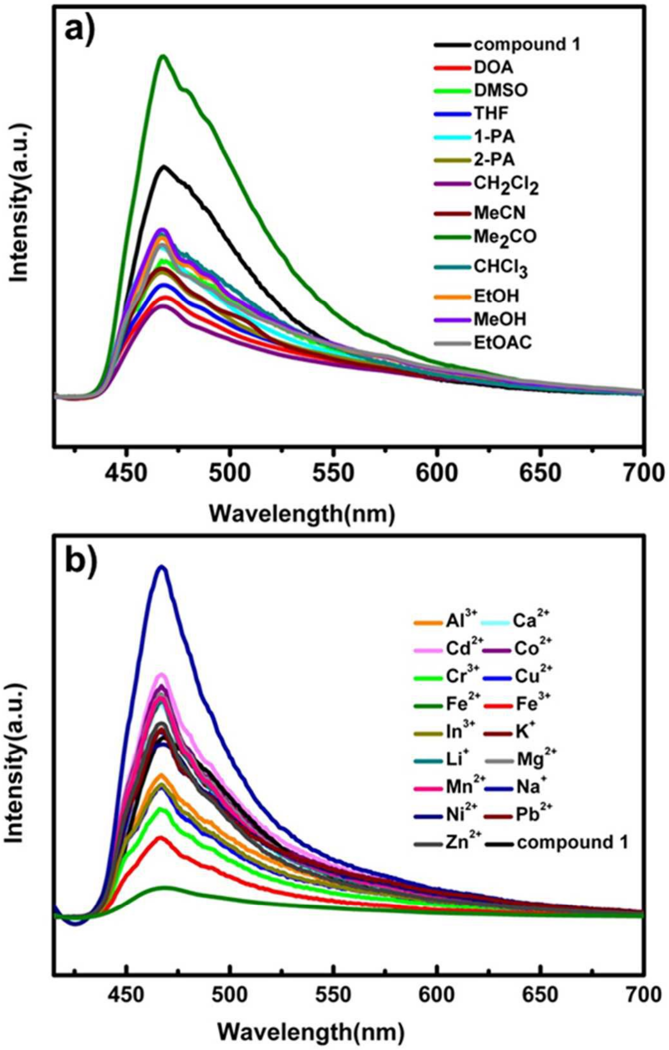

Fig. 6 (a) Emission spectra of compound **1** in different volatile organic solvents; (b) Emission spectra of compound **1** in different metal ions solutions.

The luminescence sensor of compound **1** for metal ions was also developed. The metal ions emulsions were prepared by adding 3 mg ground powder of compound **1** into 0.1 mL metal ion solutions (0.1 mol/L metal ions solution of DMF). Then the emulsions were dealt with ultrasound to form stable emulsions before florescence study. The luminescence measurements indicate that different metal ions display diverse effects on the luminescence intensity. As shown in Fig. 6b and Fig. S3, the interaction with monovalent ions enhances the luminescence intensity, particular for Na^+. Similarly, when contact with divalent ions, the luminescence intensity also enhanced, but Fe^{2+} nearly quenched. While for trivalent ions the luminescent intensity has negligible effect. On the basis of compound **1** is stable in different solutions, for comparison, the fluorescence of the ligand in different solvents and metal ions have also been measured (Fig. S4). Although the mechanism of response to metal ion is still unclear now, it may be associated with the d-d orbit transition of these metal ions, and the emission energy transformed into d-d spin transition.[56] Compound **1** is a potential probe material to detect Fe^{2+} and Na^+.

Conclusions

In summary, by using H_2PZC as ligand and $CdCl_2 \cdot 2.5H_2O$ as metal source, we have successfully synthesized a novel three-dimensional MOF based on trinuclear Cd(II)-cluster SBU. Compound **1** possesses commendable water and thermal stability. Owing to the existence of d^{10} metal ions, compound **1** exhibits excellent luminescent property in different organic solvents and metal ions. It is worth noting that the luminescent intensity of acetone, Na^+ and Fe^{2+} emulsions are observably changed, the acetone and Na^+ emulsion exhibits significant enhanced behavior, while the Fe^{2+} emulsion shows nearly quenching behavior. These behaviors indicate that compound **1** may be used to detect acetone, Na^+ and Fe^{2+} as a potential luminescent probe material. The synthesis of compound **1** may enrich the family of luminescent MOFs.

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Notes and references

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†Electronic Supplementary Information (ESI) available: Selected bond lengths and angles, additional figures PXRD, CCDC 1422767. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/b000000x

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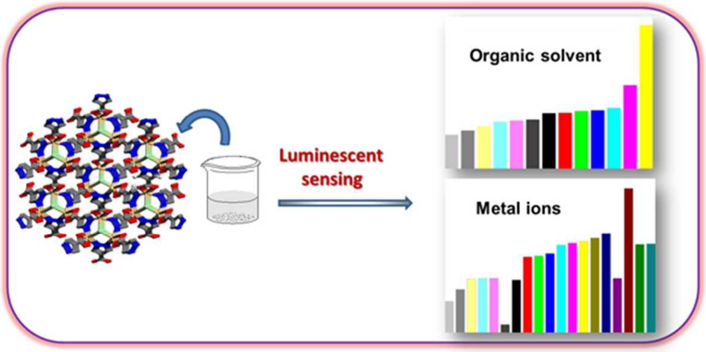
TOC:

A novel trinuclear Cd(II) cluster-based Metal–Organic Framework: synthesis, structure and luminescence property

Jia Wang, Shuo Yao, Guanghua Li, Qisheng Huo, Lirong Zhang* and Yunling Liu*



A novel luminescent Cd-MOF based on trinuclear Cd cluster and H_2PZC ligand has been constructed, the obtained Cd-MOF material exhibits high water stability and good luminescence properties for various organic solvents and different metal ions.