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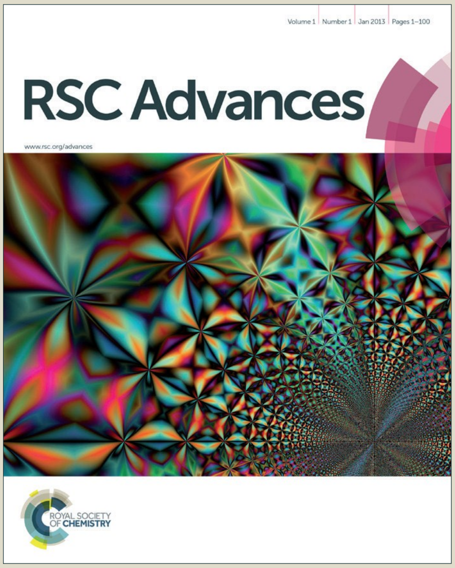

ROYAL SOCIETY OF CHEMISTRY

www.rsc.org/advances

Journal Name

RSC Publishing

ARTICLE

Cite this: DOI: 10.1039/x0xx00000x

Received 00th January 2014,
Accepted 00th January 2014

DOI: 10.1039/x0xx00000x

www.rsc.org/

A highly connected (5,5,18)-c trinodal MOF with a 3D diamondoid inorganic connectivity: Tunable luminescence and white-light emission

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A new heterometallic MOF, namely [$Cd_2K_4(2,3\text{-}PDC)_4$] (**1**) (2,3-$H_2$PDC = 2,3-Pyridine dicarboxylic acid), has been successfully synthesized from Cd^{2+} and K^+ salts with 2,3-H_2PDC under the solvothermal conditions. Single crystal X-ray diffraction studies reveal that **1** is a three-dimensional, highly connected trinodal (5,5,18)-c net. Compound **1** features a rare 3D diamondoid Cd(II)-K(I) inorganic connectivity constructed by a {Cd_2K_4} cluster. The K^+ ion in the structure functions not only as a charge balancer but also as a connector in the construction of MOF. Tunable blue-green to white luminescence of **1** was achieved by variation of the excitation wavelength and the inclusion amount of Eu^{3+} ions in the host framework of **1**.

Introduction

Metal-organic framework (MOF) has emerged as a new class of material which has attracted great attention due to their pleasing architectures and potential applications for materials science, catalysis and biochemistry.[1] It has been well demonstrated that the multi-nodal and highly connected nets offer great advantage to increase the topological diversity and stability of the framework.[2] To date, a variety of MOFs have been constructed which usually exhibit uninodal 3-, 4- and 6-connected topology, and those having higher connectivity numbers are uncommon.[3] This may be because the construction of such systems are severely hampered by the available number of coordination sites of the metal centre and the sterically demanding nature of the organic ligand.[4] One way to construct high-connected framework is to utilize clusters as building blocks, because they can enhance the coordination number and reduce the steric hindrance of organic ligands.[5] Benefited from this strategy, MOFs having binodal networks with high-connected nodes such as (3,12)-, (5,12)-, (4,24)- and (3,36)-nodes have been successfully obtained.[6] By comparison, trinodal frameworks with high-connectivity (>10) are still very rare.[7]

White-light-emitting diodes (WLEDs) are regarded as an indispensable solid-state light source for the next generation lighting industry and display systems because of their unique properties such as energy savings, environmental friendliness, small volume, and long persistence.[8] Up to now, the search for efficient materials for WLEDs is mainly focused on inorganic oxides, organic molecules, polymers and quantum dots. More recently, MOFs have been considered as a new class of light-emitting materials with the possibility of colour tuning of the light emission, including the production of white light.[9] The inorganic-organic hybrid character enables them to produce a diversity of optical phenomenon different from classical light-emitting materials. It has been documented that the white-light emission can be achieved by co-doping Eu^{3+} and/or Tb^{3+} ions in a single framework through variation of the stoichiometric ratio of different lanthanide ion.[10] Unfortunately, this approach still remains a great challenge because on one hand it is difficult to obtain mixed lanthanide MOFs due to the specific recognition requirements by different lanthanide ions and on the other hand it is hard to control the composition of different lanthanide ions in the same framework. Another alternative approach for white-light emission is to encapsulate Ln^{3+} species in microporous luminescent MOFs constructed from transition metals. Such host–guest systems will offer a new possibility to realize multiband emissions including white-light emission by judicious selection of suitable host framework and adjusting the encapsulation amounts of different Ln^{3+} complexes.[11]

Herein we report a highly connected (5,5,18)-c trinodal net, [$Cd_2K_4(2,3\text{-}PDC)_4$] (**1**) (2,3-$H_2$PDC = 2,3-pyridinedicarboxylic acid), featuring a 3D heterometallic inorganic connectivity with a diamondoid topology. The emission of **1** can be tuned from green to blue *via* variation of the excitation wavelength and a white-light emission can be readily realized by encapsulating luminescent Eu^{3+} ions into the framework of **1**.

Experimental

Materials and methods

All reagents were obtained from commercial sources and used without further purification. Thermogravimetric experiment was performed using a TGA/NETZSCH STA-449C instrument heated from 30–1000°C (heating rate of 10°C/min, nitrogen stream). IR spectrum was recorded on a Spectrum-One FT-IR spectrophotometer using KBr pellets. Powder X-ray diffraction (XRD) patterns were recorded on crushed single crystals in the 2θ range 5–50° using Cu-Kα radiation. Element analysis (C, H and N) were measured with an Elemental Vairo ELIII analyzer. Fluorescence spectra for the solid samples were performed on an Edinburgh Analytical instrument FLS920. The diffuse-reflectance spectrum has been measured on a Lambda 900 spectrophotometer using $BaSO_4$ powder as 100% reflectance at room temperature.

Synthesis of [Cd_2K_4(2,3-PDC)$_4$] (1).

A mixture of $Cd(NO_3)_2\cdot 4H_2O$ (0.2mmol), KNO_3 (0.5mmol) and 2,3-Pyridine dicarboxylic acid (0.4mmol) was placed in a 20 mL of Teflon-lined stainless steel vessel with 6 mL of DMF/ CH_3OH (V/V = 1:1). The mixture was heated to 150 ºC in 4 h and kept this temperature for two days, and then cooled slowly to 30 ºC in one day. Colourless crystals of **1** were collected in 79% yield based on $Cd(NO_3)_2\cdot 4H_2O$. Elemental analysis calcd. for **1** $C_{28}H_{12}Cd_2K_4N_4O_{16}$ (1041.62) (%): C, 32.26; H, 0.096; N, 5.38. Found (%): C, 31.71; H, 0.101; N, 5.31. IR data (cm^{-1}): 3408m, 3020w, 1598s, 1489s, 1384vs, 1107s, 948s, 866s, 831s, 779m, 719m, 663m, 601w, 545w, 443m (Fig. S5).

Crystal Structure Determination

Single-crystal X-ray diffraction data were collected at room temperature on a Rigaku Diffractometer with a Mercury CCD area detector (Mo Kα; λ = 0.71073 Å). Empirical absorption corrections were applied to the data using the Crystal Clear program.[12] The structure was solved by direct methods using SHELXTL and refined by full-matrix least-squares on F^2 using SHELX-97 program.[13] Metal atoms were located from the *E*-maps. Other non-hydrogen atoms were located in successive difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were positioned geometrically. Selected bond lengths and angles are given in Tables S1 and S2. Crystallographic data and other pertinent information are summarized in Table 1.

Results and Discussion

Synthesis and description of Crystal Structures

Colourless crystals of **1** were synthesized by the solvothermal reaction of $Cd(NO_3)\cdot 4H_2O$, KNO_3 with 2,3-H_2PDC in a 2 : 5 : 3 molar ratio, in a 6 mL mixed solvent of DMF and methyl alcohol at 150°C for 2 days. Single crystal X-ray diffraction studies reveal that **1** crystallizes in the tetragonal space group *I*4(1)/a. The asymmetric unit contains half Cd^{2+} ion, one K^+ ion and one 2,3-PDC^{2-} ligand. The Cd1 atom is hexa-coordinated and located in an octahedral environment by four O (O4, O4c, O2a and O2b) and two N atoms (N1, N1c) from four different 2,3-PDC^{2-} ligands (Fig. 1a). The K1 ion is located in a strongly deviated octahedral environment, completed by six O atoms (O1a, O1d, O2b, O3b, O3e and O4) from four different 2,3-PDC^{2-} ligands. The 2,3-PDC^{2-} ligand in **1** adopts a μ_7-$\eta^{1:1:2:1:2}$ coordination mode to connect five K and two Cd atoms through four carboxylate O and one N atom (Fig. S1). It is worth noting that such a coordination mode has not been observed in MOFs based on 2,3-PDC^{2-} ligand. Four KO_6 polyhedra are organized into a {K_4} cluster through corner-sharing (Fig. 1b). Each {K_4} cluster is in turn connected to two CdO_4N_2 polyhedra (Fig. S2) *via* shared O2···O4 edges to form a heterometallic {K_4Cd_2} cluster, which acts as a building unit in the construction of a 3D diamondoid inorganic connectivity (Fig. 1c). Thus, compound **1** can be viewed as an organic 2,3-PDC^{2-} net embodied by a 3D inorganic connectivity (Fig. 1d).

Table 1 Crystal Data and Structure Refinement of **1**

Compounds	**1**
CCDC	1400307
Formula	$C_{14}H_6N_5O_8CdK_2$
M*r*	520.81
Crystal system	tetragonal
Space group	*I*4(1)/a
a (Å)	14.3031(5)
b (Å)	14.3031(5)
c (Å)	16.4279(15)
α (deg)	90
V (Å^3)	3360.8(3)
Z	8
D_c (g cm^{-3})	2.059
M (mm^{-1})	1.84
F(000)	2032
GOF	1.149
$R_1{}^a$	0.0458
$wR_2{}^a$	0.1676

$^aR = \sum(||F_o| - |F_c||)/\sum|F_o|$, $wR = \{\sum w[(F_o^2 - F_c^2)^2]/\sum w[(F_o^2)^2]\}^{1/2}$

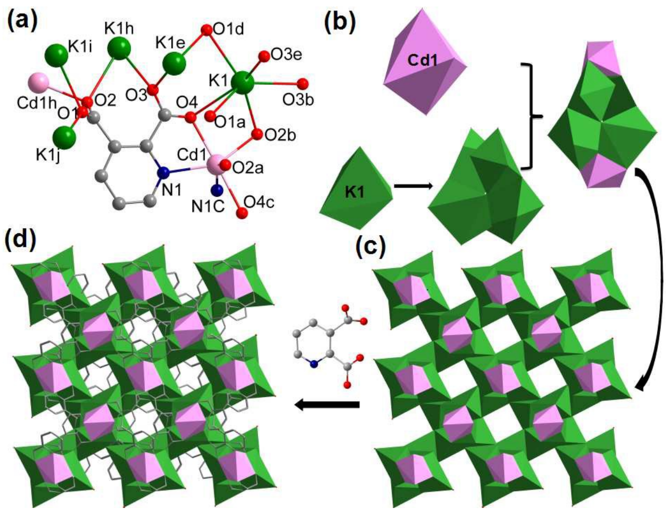


Fig. 1 (a) The coordination environment of the Cd(II) and K(I) ions in compound **1**. (b) Schematic representation of the organization of K1 and Cd1 polyhedra. (c) Construction of a 3D inorganic building block. (d) View of inorganic framework encapsulated by the organic component.

In order to gain a better visualization and understanding of the structure of **1**, topological analysis has been performed. Accordingly, the Cd1 atom and the {K_4} cluster can be viewed as two subunits: each Cd1 unit being linked by four 2,3-PDC^{2-} ligands and one {K_4} cluster can be regarded as a 5-connected node (Fig. 2b), while each {K_4} cluster being surrounded by other four {K_4} clusters, twelve 2,3-PDC^{2-} ligands and two Cd1 units can be considered as a 18-connected node (Fig. 2c). The 2,3-PDC ligand, linking three {K_4} clusters and two Cd1 units acts as a 5-connected node (Fig. 2a). Therefore, **1** can be regarded as a (5,5,18)-connected net (Fig. 2d), with a total point symbol of $\{3^4{\cdot}4^6\}_2\{3^4{\cdot}4^4{\cdot}5^2\}_4\{3^{24}{\cdot}4^{44}{\cdot}5^{44}{\cdot}6^{39}{\cdot}7^2\}$. Apparently, the K^+ ion not only compensates the charge of the framework, but also functions as a connector to extend the dimension of the inorganic connectivity. When the K^+ ions in **1** are omitted, the 3D inorganic building block is completely disrupted, resulting in a new anionic interpenetrating structure consisting of two identical 3D diamondoid Cd^{2+}–2,3-PDC^{2-} networks (Fig. S3). MOFs constructed from a 3D inorganic connectivity are rare. Among them, there is only one example with a 3D diamondoid inorganic connectivity.[14]

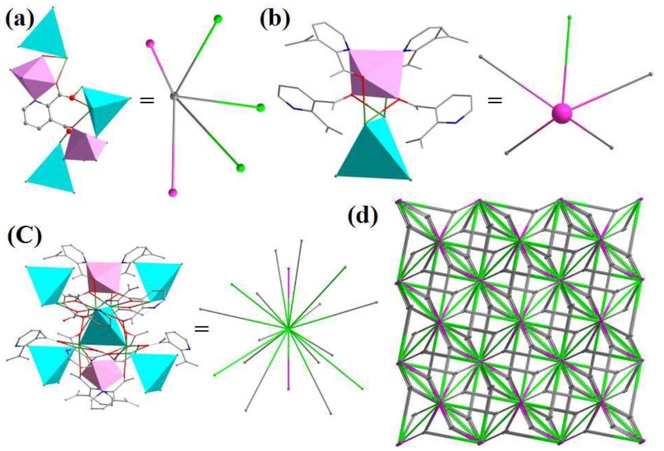


Fig. 2 Schematic representation for the nodes of (a) 2,3-PDC^{2-} anion. (b) Cd1 unit. (c) {K_4} cluster. (d) The (5,5,18-c) topological net in **1** (the atoms in gray, pink, and green represent 2,3-PDC^{2-}, Cd1 and {K_4} cluster, respectively).

IR spectra, Thermal characterization and Conductivity

Powder XRD data of **1** were recorded at ambient temperature. The peak positions of simulated and experimental patterns are in good agreement with each other, indicating the phase purity of the as-synthesized sample (Fig. S4). In the IR spectrum of **1**, the absence of strong absorption associated with the carboxyl group at around 1701 cm^{-1} indicates that the 2,3-BPC^{2-} ligand is completely deprotonated, as confirmed by the single-crystal X-ray analysis (Fig. S5). In order to evaluate the thermal stability of **1**, thermogravimetric analysis was performed. The TGA curve of **1** shows that it can stable up to 200 °C, and after that it begins to decompose upon further heating (Fig. S6).

To explore the conductivity of **1**, the measurement of diffuse reflectivity for the powder sample was carried out. The band gap (Eg) can be determined as the intersection point between the energy axis and the line extrapolated from the linear portion of the adsorption edge in a plot of the Kubelka-Munk function F against E.[15] As shown in Fig. 3, the corresponding well-defined optical absorption associated with Eg can be assessed at 3.3 eV, which indicates that compound **1** is a potential wide gap semiconductor material.[16]

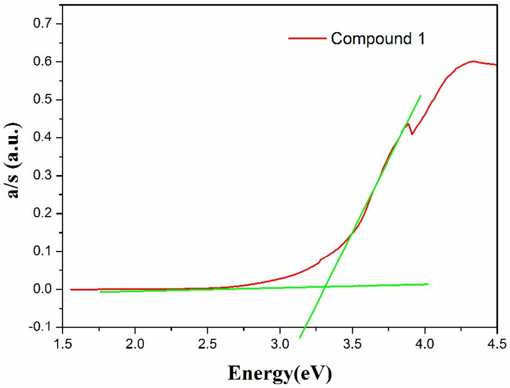


Fig.3 The diffuse reflectance UV-vis-NIR spectrum of K-M function vs. energy (ev) of compound **1**.

Tunable Photoluminescence and White-light emission

Compound **1** exhibits two maximum emissions at 420 and 498 nm under excitation at 350 and 360 nm (Fig. S7). Tunable blue-green luminescence was observed in **1** with different excitation wavelengths ranging from 310 to 380 nm (Fig. 4).[17] When the excitation wavelength varied from 310 to 340 nm, the emission intensity at 420 and 498 nm increases, reaching the maximum when excited at 340 nm. As the excitation wavelength moves from 340 to 380 nm, the emission intensity at 420 and 498 nm gradually decreases and finally the two emission peaks merge into a broad emission at 470 nm. Because the emission peak at 420 nm is similar to that of the 2,3-H_2PDC (Fig. S8), it is reasonable to assign this emission to intra-ligand π-π* transition of the ligand, while the emission peak at 498 nm should be originated from LMCT(O–Cd).[18]

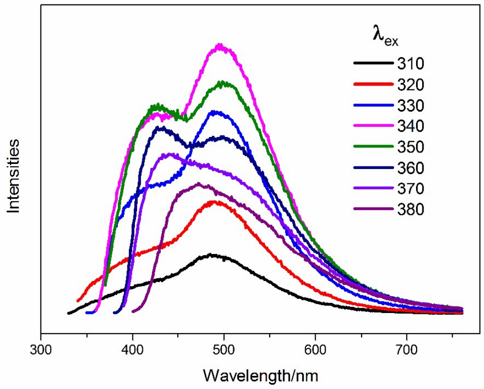
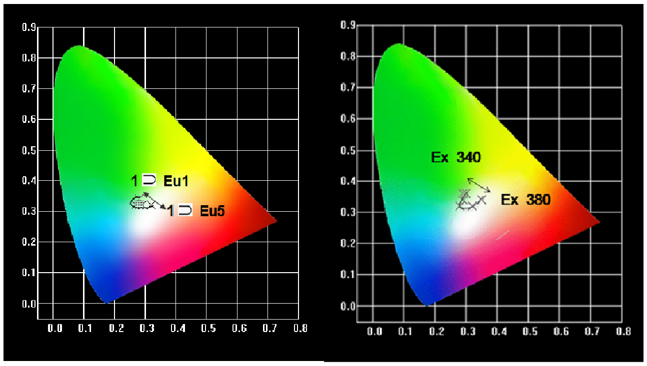


Fig.4 Solid-state emission spectra of **1** by variation of the excitation light under the same conditions.

The tunable luminescent property of **1** indicates that it emits broad blue-green light by varying the excitation wavelengths through 310–380 nm. This result gives us an opportunity to obtain whit-light emission by using **1** in combination with a red emitting component. In order to do this, dried sample of **1** (100 mg) was immersed in a CH_3OH solution (5 ml) of $Eu(NO_3)_3$

with different concentrations for 24 h (Table S3). Then the filtered solid was washed with CH_3OH several times to remove any residual Eu^{3+} ion on the sample surface, affording Eu^{3+}-included compounds **1 ⊃ Eu1** to **1 ⊃ Eu5**. The amount of Eu^{3+} encapsulated in the framework of **1** increases by increasing the concentration of Eu^{3+} in the CH_3OH solution. The maximum encapsulated amount of the Eu^{3+} ion is 1.94% as confirmed by ICP. The solid-state emission spectra of **1 ⊃ Eu1** to **1 ⊃ Eu5** excited at 375 nm exhibit the characteristic transitions $^5D_0$ to ^7F_J (J = 0–4) of Eu^{3+} at around 578, 590, 612, 650 and 699 nm (Fig. S10). The CIE coordinates of **1 ⊃ Eu1** to **1 ⊃ Eu5** are 0.28, 0.33), (0.27, 0.32), (0.28, 0.32), (0.29, 0.32) and (0.32, 0.32), respectively. As the content of Eu^{3+} ion is increased, the emission gradually moves from blue-green range to white area (Fig. 5a). Compounds with less Eu^{3+} ion exhibit a dominant blue-green emission, while the compound **1 ⊃ Eu5** with 1.94% Eu^{3+} displays white-light emission (Fig. S9). Thus, the emission color balance between the host and the Eu^{3+} ion can be easily tuned by varying the amount of encapsulated Eu^{3+}. Meanwhile, the emission of **1 ⊃ Eu5** at different excitation wavelengths was also investigated. When excited at 340, 350 and 360 nm, it exhibits green emission with CIE coordinates of (0.30, 0.36), (0.29, 0.36) and (0.29, 0.34), respectively (Fig. S10). As the excitation wavelength is shifted towards lower energy (λ_{ex} = 370, 375 and 380 nm), the main emission peaks of Eu^{3+} and the host are comparable in intensity, and the complex emission falls in the white region, as illustrated in Fig. 5b. The corresponding CIE coordinates are (0.35, 0.34), (0.32, 0.32) and (0.28, 0.32), respectively, which are very closed to the international pure white-light CIE chromaticity coordinates (0.333, 0.333).[19]



(a) (b)

Fig.5 (a) The CIE values of **1 ⊃ Eu1** to **1 ⊃ Eu5**; (b) The CIE chromaticity of **1 ⊃ Eu5** (λ_{ex} = 380, 375, 370, 360, 350 and 340 nm).

Conclusions

In summary, a highly connected (5,5,18)-c trinodal MOF built from a novel 3D Cd(II)-K(I) diamondoid inorganic connectivity has been synthesized and structurally characterized. It exhibits tunable blue-green luminescence through varying the excitation wavelength. Blue-green to white-light emitting materials can be obtained by encapsulating different amount of Eu^{3+} in the host framework. Moreover, white-light emission can be achieved *via* optimizing the concentration of Eu^{3+} in the framework and the excitation wavelength. These Eu^{3+}-encapsulated MOFs may have potential applications in the design of colour tunable and white-light emitting materials.

Acknowledgements

This work was supported by the National Basic Research Program of China (973 Program, 2012CB821702), the National Natural Science Foundation of China (21233009, 21173221 and 21571175) and the State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences.

Notes and references

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†Electronic Supplementary Information (ESI) available: Additional figures, IR, TGA, XRD patterns and the emission spectra of the ligand and the Eu^{3+}-encapsulated materials. Tables for bond lengths and angles and the ICP results. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/b000000x/.

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Graphical Abstract

A highly connected (5,5,14)-c trinodal MOF featuring a 3D diamondoid inorganic connectivity was synthesized. Tunable color and white light emission can be achieved by inclusion of Eu^{3+} into the framework.