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分子间卤键和氢键控制的网络结构的组装

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摘要 分别以四苯甲烷和四苯基卟啉为中心, 端位引入四氟碘代苯, 合成了两类四齿卤键供体分子, 并合成了 3,3',5,5'-四甲基-4,4'-联吡啶(TMBP)作为卤键受体分子. 以四苯甲烷为中心的卤键供体分子和 TMBP 基于分子间 I···N 卤键和 H···N 氢键在固相中自组装, 得到一类超分子网络结构. 晶体结构显示, 一个四面体卤键供体分子通过两组 I···N 卤键和两组 H···N 氢键结合四个 TMBP 分子, 与之对应, 一个 TMBP 分子通过一组 I···N 卤键和一组 H···N 氢键结合两个四面体分子, 形成单层网络结构, 网格为宽度 2.37 nm 的正方形结构, 层与层之间通过其它氢键和卤键进一步堆积. 卟啉类四齿卤键供体分子的晶体数据显示, 通过较为复杂的分子间 C—I···π 及 H···F 等弱相互作用, 供体分子自身进行平面组装, 层与层之间通过 π-π 堆积等作用进一步堆积.

关键词 卤键; 氢键; 晶体工程; 超分子化学; 自组装

Intermolecular Halogen and Hydrogen Bonding-Controlled Self-Assembly of Network Structures

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Abstract Two kinds of tetra-dentate halogen bonding donors were synthesized by introducing tetrafluoroiodide benzene into tetraphenylene methane and tetraphenyl porphyrin modules. 3,3',5,5'-Tetramethyl-4,4'-bipyridine (TMBP) was synthesized as halogen bonding acceptor. A type of supramolecular network structure was constructed by self-assembly of tetraphenylmethane-centered halogen donor and TMBP in solid phase based on intermolecular I···N halogen bonds and H···N hydrogen bonds. The crystal structure shows that one tetrahedral halogen bonding donor binds four TMBP molecules through two sets of I···N halogen bonds and two sets of H···N hydrogen bonds, correspondingly, one TMBP molecule binds two tetrahedral molecules through one set of I···N halogen bonds and one set of H···N hydrogen bonds to form a monolayer network structure distributed with square structures with grid width of 2.37 nm. And further stacking is controlled by other hydrogen and halogen bonds between layers. The crystal data of tetradentate halogen bonding donor molecules of porphyrins show that donor molecules assemble themselves in plane controlled by more complex weak intermolecular C—I···π and H···F interaction, and further stacking is controlled by π-π stacking between layers.

Keywords halogen bond; hydrogen bond; crystal engineering; supramolecular chemistry; self-assembly

1 Introduction

As a kind of robust non-covalent force similar to hydrogen bonding, halogen bonding has been widely used in the field of crystal engineering after more than two decades of

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development.[1] Two- and three-dimensional supramolecular assemblies based on polydentate halogen bonding donors were constructed.[2] The multi-mode halogen bonding interactions demonstrate the important role of halogen bonds as inducible forces in supramolecular self-assembly. And the application of halogen bonding-controlled assemblies has also been extensively explored, such as molecular recognition in solution phase,[3] ion transport across membranes,[4] *etc*. Tetraarylporphyrin and tetraphenylmethane fragments are widely used in the construction of 2D and 3D porous materials due to their specific spatial structures and easy derivatization.[5] There are many reports on the use of tetraarylporphyrin modules for constructing halogen bonding-controlled supramolecular assemblies,[6] while halogen bond-controlled assemblies based on tetraphenylmethane fragment are rarely reported.[7] We herein describe the synthesis of two kinds of molecules based on tetraphenylmethane center and tetraphenylporphyrin center, the ends of which are attached with iodotetrafluorobenzene units as halogen donor. These are two types of halogen donors with longer arm lengths, which facilitate the construction of assemblies with larger pore sizes. And 3,3',5,5'-tetramethyl-4,4'-bipyridine (TMBP) which is considered to be a better halogen acceptor was also synthesized.[8]

2 Resluts and discussion

Two tetradentate halogen bonding donors were synthesized, centered on tetraphenylene methane and tetraphenyl porphyrin with tetrafluoroiodobenzene terminated by amide bonds. Tetrafluoroiodobenzene has been proved to be an excellent halogen bonding donor fragment.[1c,9] The N—H of amide and its adjacent fluorine atoms form a six-membered N—H···F hydrogen bonding to control the orientation of the four tetrafluoroiodobenzene units, which was theoretically feasible and highly expected.[10] Compounds **1** and **2** (Figure 1) represent two types of halogen bonding donors with three-dimensional and two-dimensional spatial extension potential, which can be used as basic modules to study different spatial topologies controlled by halogen bonds. The corresponding halogen bond acceptor molecule **3** was also synthesized, and the mutual repulsion effect and electron-donating effect of the four methyl groups greatly enhanced the capacity of bipyridine as halogen bonding acceptor.[8] Co-crystals of compounds **1** and **3** and single-component crystals of compound **2** were obtained by crystal culture under the same conditions.

The synthesis of compounds **1**, **2**, and **3** is shown in Scheme 1. Compound **4** first reacted with 1.0 equiv. of *n*-butyllithium in dry tetrahydrofuran (THF) at −78 ℃, and then iodine elemental was added to obtain compound **5** in 67% yield. Compound **5**, after a further exchange reaction with *n*-butyllithium in dry THF at −78 ℃, reacted with solid carbon dioxide directly to give compound **6** in 68% yield. Compound **6** reacted with compound **7** under the condensation conditions of 2-(7-azabenzotriazol-1-yl)-*N*,*N*,*N*′,*N*′-tetramethyluronium hexafluorophosphate (HATU) and *N*,*N*-diisopropylethylamine (DIPEA) at room temperature to obtain compound **1** with a yield of 80%. Under the same conditions, compound **7** reacted with compound **8** to obtain compound **2** with a yield of 76%. Compound **3** was prepared by one-step radical coupling in the presence of sodium metal and trimethylchlorosilane from compound **9** at 0 ℃ to room temperature with a yield of 35%.

The co-crystals of compounds **1** and **3** and the single crystal of compound **2** were obtained by volatilizing the mixed solvent of methanol and dichloromethane (DCM) (V∶V=5∶1). Single crystal data display that there is no effective six-membered N—H···F hydrogen bond that can control the direction of tetrafluoroiodobenzene formed, and the four tetrafluoroiodines with amide bonds as the bonding

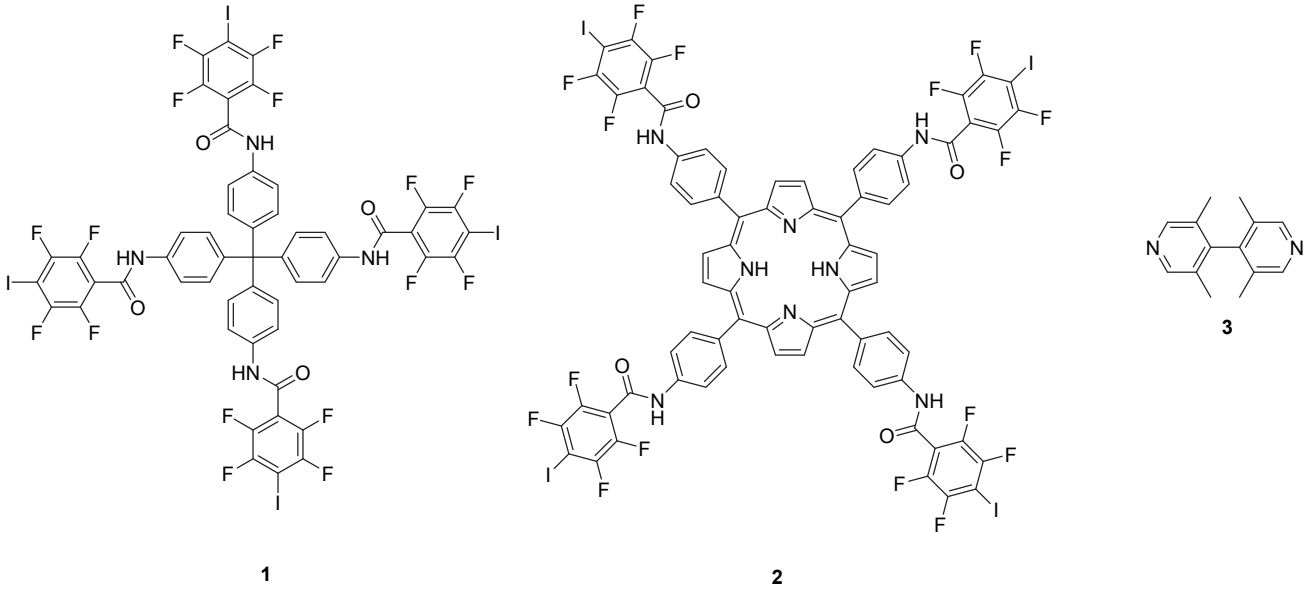
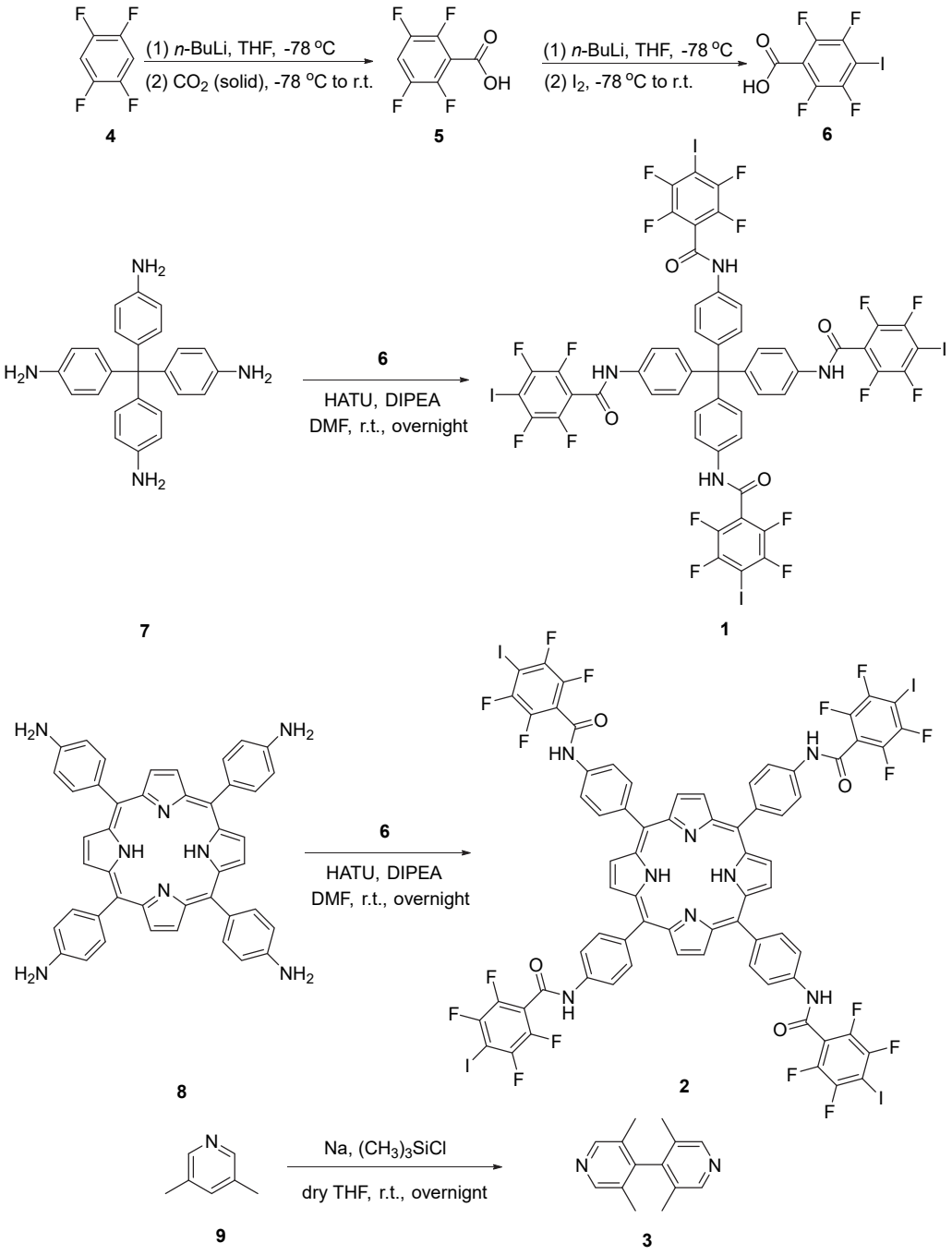


Figure 1 Structures of compounds **1**～**3**



Scheme 1 Synthetic routes for compounds **1**～**3**

site are distorted to different degrees. From the crystal data of compound **1**, it can be seen that the distances of the hydrogen atoms (amide units) and the nearest fluorine atoms are 0. 277, 0.277, 0.264, 0.264 nm, respectively. For compound **2**, the four data are 0.264, 0.256, 0.295, 0.264 nm (Figure 2, the sum of the van der Waals radium of H and F: 0.267 nm). The distances are close to or slightly larger than the sum of the van der Waals radium of hydrogen and fluorine atoms, it can be speculated that the extremely weak hydrogen bonds are not sufficient to control the orientation of tetrafluoroiodobenzene.

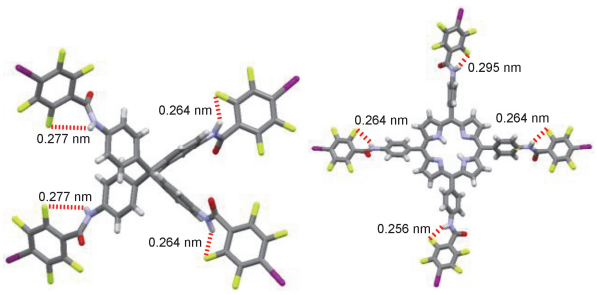


Figure 2 Crystal structures of compounds **1** and **2**

The co-crystal data of compounds **1** and **3** (Figure 3) shows that only two tetrafluoroiodines (A and B) are involved in strong halogen bonding formation, and the other two tetrafluoroiodines (C and D) are not involved in halogen bonding interaction. Unexpectedly, the adjacent amide nitrogen hydrogen atoms bind to two TMBP molecules through hydrogen bonds (Figure 3a). Then one tetrahedral halogen bonding donor binds four TMBP molecules through two sets of I···N halogen bonds (bond length: 0.274 nm, bond angle: 164.1°) and two sets of H···N hydrogen bonds (bond length: 0.202 nm, bond angle: 177.2°), correspondingly, one TMBP molecule binds two tetrahedral molecules through one set of I···N halogen bonds and one set of H···N hydrogen bonds (Figures 3b, 3c). From the bond angle close to 180° and the short distance between atoms, it can be seen that the hydrogen bond effect of these two groups of halogen bonds is very strong. The TMBP is slightly tilted and participates in the interaction of both halogen and hydrogen bonds, making the assembly present a non-planar layered network structure with wavy sides (Figures 4a, 4b). The grid has a square structure when viewed from the front, its length and width are both 2.37 nm, and the multi-layer supramolecular grid structure is obtained by further stacking between layers. However, this is a kind of dislocation stacking, which makes the positions of the grids between layers unable to keep overlapping (Figure 4c).

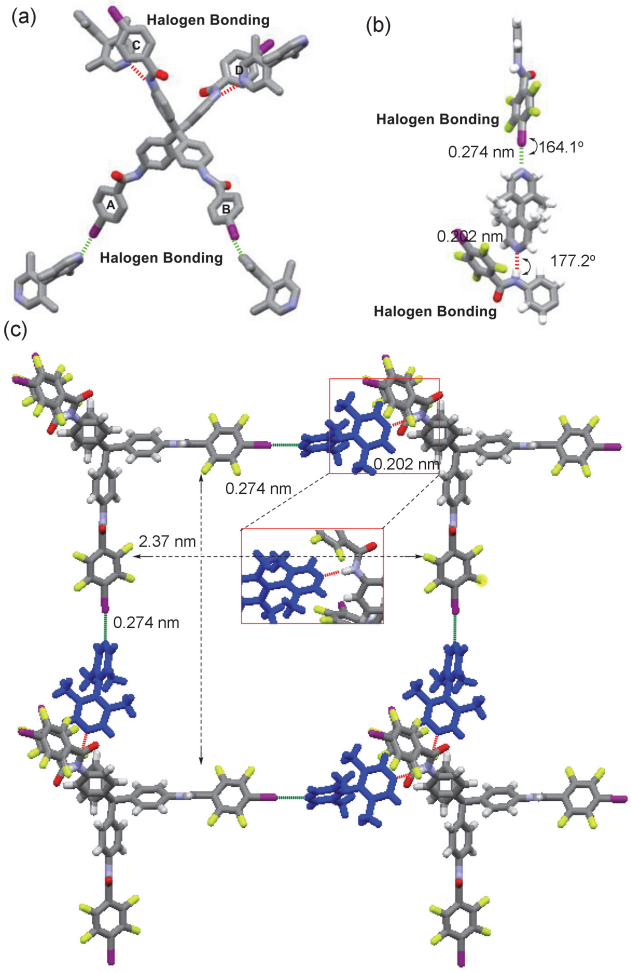


Figure 3 (a) Partial structure of one compound **1** molecule binding four TMBP molecules (H and F atoms were omitted for clarity), (b) partial structure of one TMBP molecule binding two compound **1** molecules (only the two terminal groups of compound **3** interacting with TMBP are retained, and the rest structures are omitted for clarity) and (c) a net unit of supramolecular assembly constructed by compounds **1** and **3**

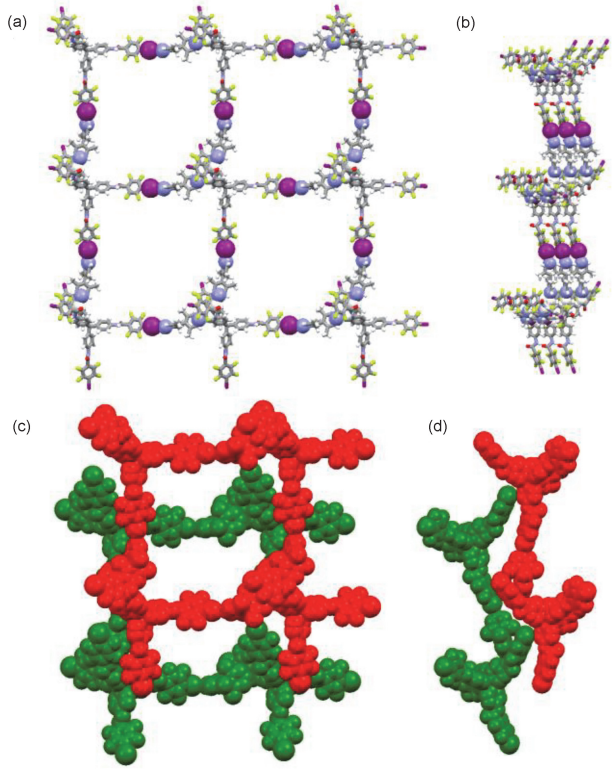


Figure 4 (a) Monolayer non-coplanar network structure formed in the solid phase of compounds **1** and **3**, (b) side view of the network, and (c, d) bilayer stacking patterns of compounds **1** and **3** and their side view

The purple balls represent iodine atoms, the light blue balls represent nitrogen atoms, and the white balls represent hydrogen atoms in (a) and (b)

The crystal data of compound **2** show that one porphyrin molecule interacts with other four porphyrin molecules on the same plane through C—I···π halogen bonds, H···F hydrogen bonds and other weak interactions, resulting in a planar network structure. And two types of rectangular grid structures of different sizes (A and B) are distributed in the network (Figure 5a). Then a planar supramolecular topology network structure with a heteroporous distribution is formed. Layer-to-layer interactions such as π-π stacking lead to multilayer structures with very small layer spacing (Figure 5b).

3 Conclusions

In this work, tetraphenylmethane- and porphyrin-based supramolecular network aggregates were constructed by

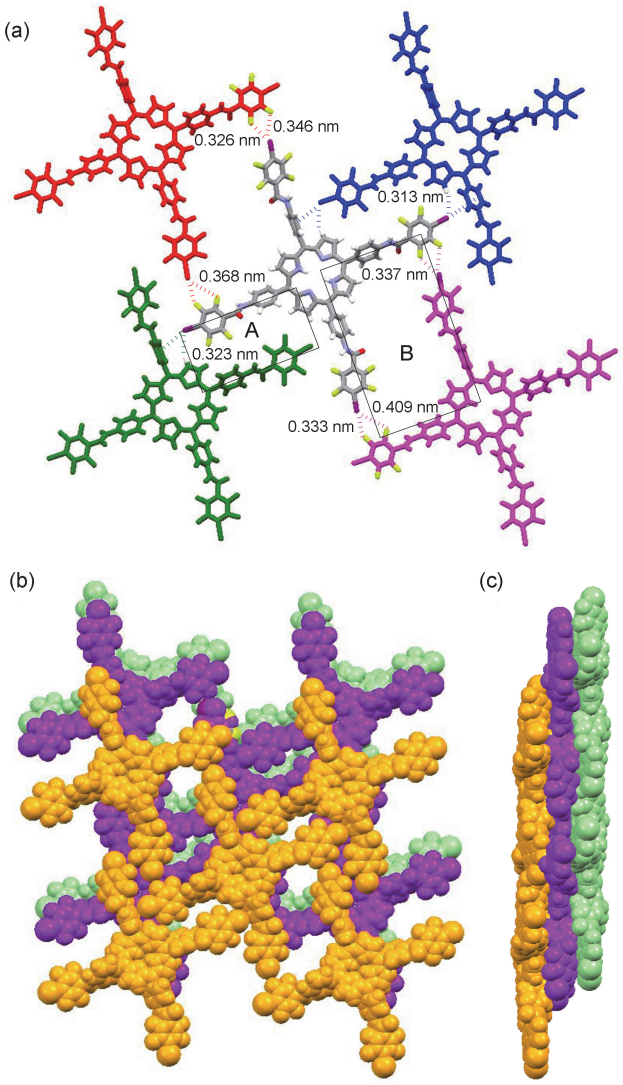


Figure 5 (a) Molecular arrangement in the same layer, (b) stacking patterns between layers and (c) side view of (b)

utilizing the induction of halogen and hydrogen bonds, and a clear halogen bond interaction mode in the crystal was observed. The formation of the three-dimensional network structure is affected by the distortion of the terminal tetrafluoroiodobenzene to different degrees. But supramolecular structures with larger-sized square grids were still got. On the basis of this work, we will focus on solving the twisting problem of terminal tetrafluoroiodobenzene, in order to obtain a topology extending into three-dimensional space. In addition, on the basis of the tetraphenylmethane and tetraphenylporphyrin centers, the length of the halogen bond donor fragment can be further regulated, and three-dimensional and two-dimensional network structures of different or larger sizes can be obtained, and the potential for subject identification can be further developed.

4 Experimental section

4.1 General information

All reagents were obtained from commercial suppliers and used without further purification unless otherwise noted. All reactions were carried out under the atmosphere of N_2 except the reactions of condensation reaction of amines and carboxylic acids under HATU and DIPEA condition.

^{1}H NMR and ^{13}C NMR spectra were recorded with a 400 MHz or 100 MHz spectrometer in the indicated solvents at 25 ℃. Chemical shifts are expressed using residual proton resonances of the deuterated solvents as the internal standards. The ^{19}F NMR spectra were recorded on 300 MHz spectrometer in the indicated solvents. Chemical shifts are expressed using $CFCl_3$ as external standard ($CFCl_3$: δ 0). Crystals were measured using a Bruker D8 Venture-Metaljet diffractometer equipped with an PHOTON II area detector and HELIOS multilayer optics monochrommated Cu-Kα and Ga-Kα radiation (λ=0. 154184 and 0.134139 nm). Crystal structures were solved by direct method and refined by full-matrix least-squares methods based on F^2 using SHELXL-2014 software. The co-crystals of compounds **1** and **3**, and the single crystal of compound **2** were obtained by volatilizing the mixed solvent of dichloromethane and methanol (V∶V=5∶1). The co-crystals of compounds **1** and **3**, and the single crystal of compound **2** have been deposited in Cambridge Crystallographic Data Centre (CCDC deposit Nos. 2166009, 2166016).

4.2 Synthesis of compound **5**

Based on a literature procedure,[9a] compound **4** (10.0 g, 66.6 mmol) was dissolved in dry THF (100 mL) and the mixture was cooled to −78 ℃. The solution was treated dropwise with *n*-butyllithium in hexanes (1.6 mol/L, 46 mL, 73.3 mmol) while maintaining the internal temperature below −60 ℃. The mixture was stirred at −70 ℃ for 20 min and then poured into a mixture of carbon dioxide ice (excess) and THF (20 mL). The reaction mixture was allowed to warm to 0 ℃ with the bath removed, and then the mixture was quenched with water (200 mL) followed by addition of *n*-hexane (100 mL). The layers were separated and the organic layer was washed with additional fresh water. Then the aqueous was adjusted to pH≈3 with con. HCl and extracted three times with dichloromethane, and the combined organic layers were washed with saturated NaCl, dried over Na_2SO_4 and concentrated to get 8.6 g of 2,3,5,6-tetrafluorobenzoic acid (**5**). White solid, 67% yield. m.p. 146.1～147.8 ℃ (Lit.[11] 148.0～148.5 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (s, 1H), 7.33～7.24 (m, 1H); ^{19}F NMR (300 MHz, $CDCl_3$) δ: −136.89～−137.02 (m, 1F), −137.69～−137.81 (m, 1F).

4.3 Synthesis of compound **6**

Based on a literature procedure,[8] compound **5** (4.0 g, 20.6 mmol) was dissolved in dry THF (90 mL) and the mixture was cooled to −78 ℃. The solution was treated dropwise with *n*-butyllithium in hexanes (1.6 mol/L, 30 mL, 51.5 mmol) while maintaining the internal temperature below −60 ℃. The mixture was stirred at −70 ℃ for 20 min and then I_2 was added by one portion. The reaction mixture was allowed to warm to room temperature and

stirred overnight. Then the mixture was quenched with water (100 mL) followed by addition of *n*-hexane (50 mL). The layers were separated and the organic layer was washed with additional fresh water. Then the aqueous was adjusted to pH≈3 with con. HCl and extracted three times with dichloromethane. The combined organic layers were washed with saturated NaCl, dried over Na_2SO_4 and concentrated to get crude product. The crude product was recrystallized in a mixed solvent of dichloromethane and *n*-hexane (*V*∶*V*=3∶1) to get 4.5 g of pure 2,3,5,6-tetrafluoro-4-iodobenzoic acid (**6**).[12] White solid, 68% yield. m.p. 156.1～157.6 ℃; ^{19}F NMR (300 MHz, $CDCl_3$) *δ*: −118.30～−118.41 (m, 1F), −136.37～−136.50 (m, 1F).

4.4 Synthesis of compound **1**

To a solution of **7** (100 mg, 0.53 mmol) and **6** (380 mg, 2.37 mmol) in *N*,*N*-dimethylformamide (DMF) (10.0 mL) was added HATU (500 mg, 1.31 mmol) and DIPEA (340 mg, 2.63 mmol). The solution was stirred at room temperature for 24 h, and then ice-water (30.0 mL) was added to the mixture. Insoluble solid was precipitated out of solution and filtered to get crude product. The crude product was recrystallized in a mixed solvent of dichloromethane and *n*-hexane (*V*∶*V*=3∶1) to get 335 mg of pure *N*,*N*′,*N*″,*N*‴-(methanetetrayltetrakis(benzene-4,1-diyl))tetrakis(2,3,5,6-tetrafluoro-4-iodobenzamide) (**1**). Light brown power, 80% yield. m.p. ＞300 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 11.01 (s, 4H), 7.62 (d, *J*=8.4 Hz, 8H), 7.20 (d, *J*=8.8 Hz, 8H); ^{19}F NMR (300 MHz, $CDCl_3$) *δ*: −120.57～−120.67 (m, 8F), −140.64～140.75 (m, 8F); HRMS (ESI) calcd for $C_{53}H_{20}F_{16}I_4N_4O_4Na$ $[M+Na]^+$ 1610.7305, found 1610.7195.

4.5 Synthesis of compound **2**

To a solution of **8** (100 mg, 0.15 mmol) and **6** (200 mg, 0.62 mmol) in DMF (20.0 mL) was added HATU (281 mg, 0.74 mmol) and DIPEA (96 mg, 0.74 mmol). The solution was stirred at room temperature for 24 h, and then ice-water (60.0 mL) was added to the mixture. Insoluble solid was precipitated out of solution and filtered to get crude product. The crude product was recrystallized in a mixed solvent of dichloromethane and *n*-hexane (*V*∶*V*=3∶1) to get 210 mg of pure *N*,*N*′,*N*″,*N*‴-(porphyrin-5,10,15,20-tetrayltetrakis(benzene-4,1-diyl))tetrakis(2,3,5,6-tetrafluoro-4-iodobenzamide) (**2**). Blue-violet solid, 76% yield. m.p. ＞300 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 11.46 (s, 4H), 8.94 (s, 8H), 8.27 (d, *J*=8.0 Hz, 8H), 8.15 (d, *J*=8.4 Hz, 8H); ^{19}F NMR (300 MHz, $CDCl_3$) *δ*: −120.37～−120.47 (m, 8F), −140.33～140.44 (m, 8F); HRMS (ESI) calcd for $C_{72}H_{30}F_{16}I_4N_8O_4$ $[M+H]^+$ 1882.8386, found 1882.8384.

4.6 Synthesis of compound **3**

Based on a literature procedure,[13] to a solution of 11.8 mL (103.5 mmol) of 3,5-lutidine and 13.1 mL (103.5 mmol) of Me_3SiCl in 200 mL of dry THF under an N_2 atmosphere was added sodium (2.5 g, 108.7 mmol) in 5 mL of THF. The rate of addition was controlled so that the reaction flask was never warm to the touch. After complete addition, the mixture was stirred at room temperature for 48 h. The reaction was quenched with EtOH (50 mL) which was added by dropwise slowly, and then water (50 mL) was added. The mixture was stirred at room temperature for 20 min. The organic phase was separated and the aqueous was extracted with dichloromethane (DCM) (50 mL×3). The combined organic phase was dried over Na_2SO_4 and concentrated to get crude product. The crude product was purify by column chromatography (eluent: petroleum ether/ethyl acetate, *V*∶*V*=2∶3) to get 2.0 g of pure 3,3',5,5'-tetramethyl-4,4'-bipyridine (**3**).[13] White solid, 18% yield. m.p. 124.3～125.9 ℃ (Lit.[12] 126 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.42 (s, 4H), 1.91 (s, 12H).

Supporting Information General methods, synthesis and characterization of new compounds, additional crystal structures, and ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of new compounds. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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