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A Zinc Porphyrin Bearing Two Lateral dpp-Containing Rings and Its [3]Pseudorotaxane (dpp: 2,9-diphenyl-1,10-phenanthroline)

Cécile Roche,[a] Angélique Sour,*[a] Frédéric Niess,[a] Valérie Heitz,[a] and Jean-Pierre Sauvage*[a]

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A zinc porphyrin attached to two dpp-containing rings was prepared from a 31-membered ring with an attached pendent aldehyde function and 5-mesityldipyrromethane by using the synthetic method introduced long ago by Lindsey and his group. The obtained porphyrin is of the ABAB type, with two mesityl groups on the 5- and 15-positions and two macrocycles on the 10- and 20-positions. From this compound, a [3]pseudorotaxane was obtained in good yield from the reaction of the porphyrin with two equivalents of a copper(I) salt followed by the addition of two equivalents of 2,9-dianisyl-1,10-phenanthroline.

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Introduction

The synthesis of porphyrin-containing catenanes and rotaxanes has experienced a rapid development since the first templated strategies reported more than 15 years ago.[1,2] These compounds are highly promising species in relation to models of the photosynthetic reaction centre and natural antenna, which are the key components of photosynthetic organisms. They are also potentially important in the field of molecular machines.[3,4] Besides the originally developed covalent approach, noncovalent strategies turned out to be very efficient, as they can lead to highly complex structures in a few steps from prefabricated porphyrinic compounds. Both the coordination chemistry approach and the organic one, based on π–π interactions between aromatic fragments and/or hydrogen bonds, have led to spectacular interlocking porphyrinic assemblies.[5–14] Interesting work on multicomponent but noninterlocking systems assembled through noncovalent interactions has also been reported by several research teams.[15–23]

By exploiting a copper(I)-based template approach, our group has described numerous porphyrin-containing catenanes and rotaxanes and their energy and electron-transfer properties.[3,4] Usually, the key components are porphyrin-coordinating ring conjugates.[24–26] The ring contains a dpp fragment that is ideally adapted to the formation of tetrahedral copper(I) complexes of the $Cu(dpp)_2^+$ family. Such complexes constitute the entanglements necessary for the formation of the desired interlocking structures. Along this line, the recently published work on a [3]rotaxane acting as an adaptable receptor is particularly illustrative.[27] The present report is concerned with the synthesis of a porphyrinic compound consisting of a central porphyrin bearing two laterally disposed dpp-containing macrocycles and its reaction with copper(I) atoms in the presence of additional chelates to afford a new [3]pseudorotaxane.

[a] Laboratoire de Chimie Organo-Minérale, Institut de Chimie, CNRS UMR 7177, Université de Strasbourg,
4, rue Blaise Pascal, 67070 Strasbourg Cedex, France
E-mail: a.sour@chimie.u-strasbg.fr
sauvage@chimie.u-strasbg.fr

Results and Discussion

Design of the System

The copper(I)-templated synthesis of multirotaxanes may necessitate the preparation of components containing several coordinating rings. An alternative strategy is that of the "daisy chain" approach,[28] based on ring-and-thread conjugates, which has also been successfully exploited in combination with copper(I)[29] to afford [3]- and [4]rotaxanes. Recently, we reported the high-yield formation of [4]rotaxanes consisting of two bismacrocycles and two rods able to thread the rings.[27] In the previous examples,[27,29] the connecting unit between the ring and the thread or between the two rings was located at the back of the 1,10-phenanthroline chelate (i.e., it involved the 5- and 6-positions) and it was extremely rigid. We wanted to make and study less-constrained systems consisting of coordinating rings attached to the central aromatic connector (porphyrins in the present case) by a single C–C linker located on the same side as the coordinating site of the 1,10-phenanthroline. In addition, it appeared particularly important to perform the reaction in a straightforward fashion with only a few steps within the overall synthetic process. The general

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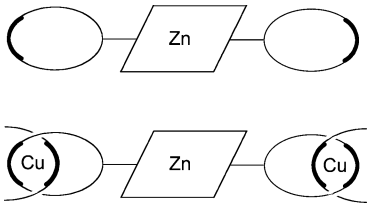
structure of the target compounds is represented in Scheme 1, as well as the corresponding threaded species ([3]pseudorotaxane).



Scheme 1. General structure of the compounds consisting of two peripheral rings attached to the central porphyrin on two *meso* carbon atoms *trans* to each other. The chelating unit is indicated as an arc of a circle and the porphyrin is represented by a lozenge. The copper(I)-driven threading of an open chain coordinating fragment should lead to a doubly threaded species consisting of three independent organic components ([3]pseudorotaxane).

The porphyrinic compound schematically represented in Scheme 1 could lead to a [4]rotaxane by threading two two-chelate molecular rods through the cyclic components of the molecule or, if the peripheral rings turn out to be flexible enough, to a [2]pseudorotaxane consisting of a single rod passing through the rings of the same porphyrinic compound.

Synthesis of the Porphyrin-Based Molecule Bearing Two Peripheral Coordinating Rings

The chemical structure of the target molecule is depicted in Figure 1 as is the sequence of chemical reactions leading to its preparation. The synthetic route chosen for the preparation of zinc porphyrin **1**, bearing two lateral rings, was to prepare 31-membered macrocycle **3** containing a benzalde-

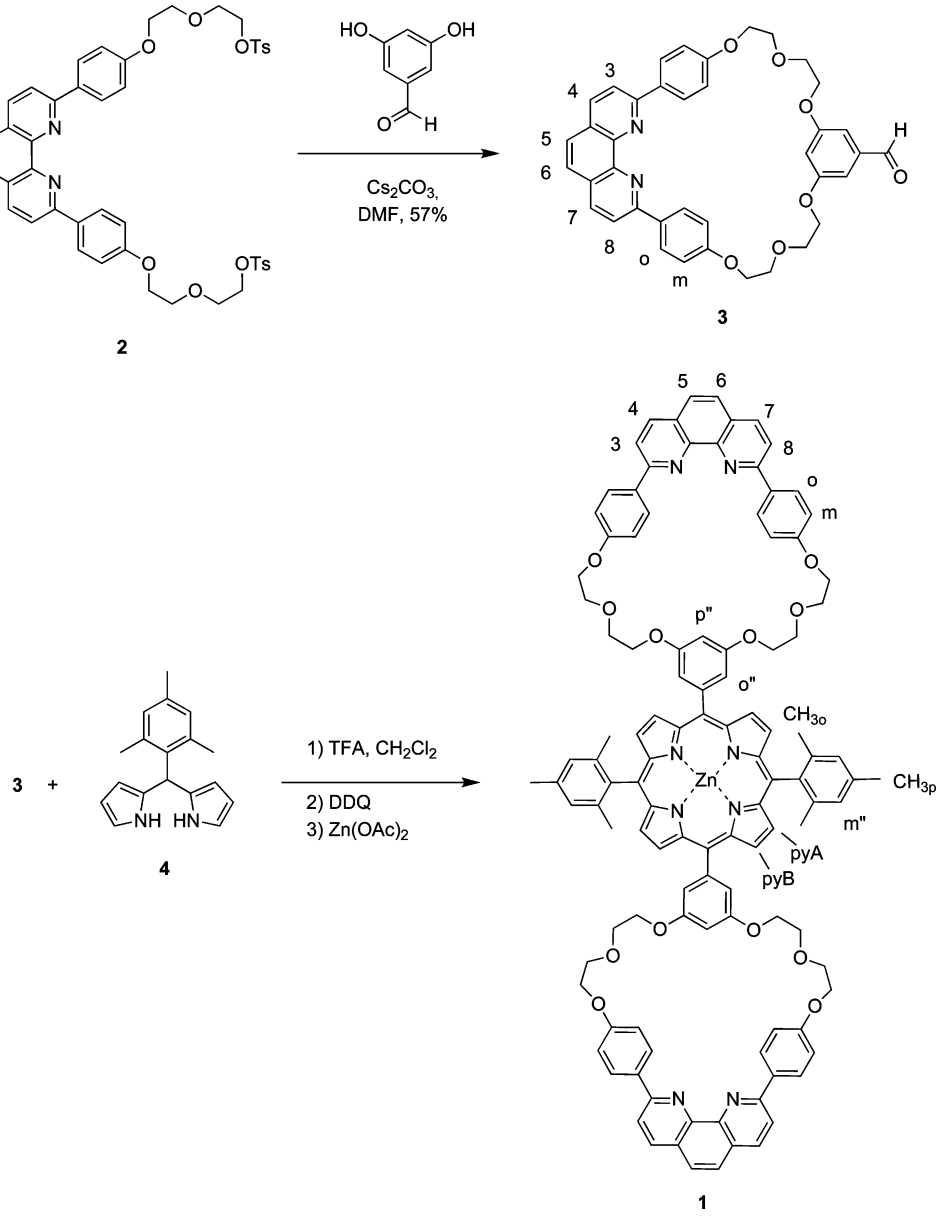


Figure 1. Preparation of compound **1**, consisting of a central Zn-complexed porphyrin nucleus attached to two coordinating 31-membered rings through single carbon–carbon bonds.

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hyde function and then to form the porphyrin by a condensation reaction with a dipyrryl methane derivative.

Macrocycle **3** was prepared by the cyclisation reaction of ditosylate **2** (obtained in two steps following a literature procedure)[24] with 3,5-dihydroxybenzaldehyde in the presence of Cs_2CO_3 at 90 °C. After purification, the compound was obtained as a white powder in a very satisfactory 57% yield. The porphyrin was then formed by condensation of macrocycle **3** with 5-mesityldipyrromethane (**4**). The latter compound was chosen as it increases the solubility of the corresponding porphyrin, it reduces scrambling of the various groups during the condensation reaction and it prevents π-stacking of the porphyrins. We chose the [2+2] McDonald-type condensation, a reaction introduced by Lindsey et al.,[30] to minimise the formation of the *cis*-A_2B_2-type porphyrin. We followed the condensation conditions described by Lindsey et al.[31] 5-Mesityldipyrromethane and the macrocyclic aldehyde [10 mM in CH_2Cl_2 containing 18 mM trifluoroacetic acid (TFA)] were stirred at room temperature. The reaction turned out to be very slow. After 20 h we obtained the desired porphyrin in poor yield (<2%). We performed other condensation reactions with different concentrations of TFA and observed that 5 equivalents of TFA were necessary to allow efficient condensation. After chromatographic purification, the porphyrinic site was metallated with zinc acetate. Further treatment with an EDTA solution to remove zinc(II) from the 1,10-phenanthroline chelate afforded compound **1** in 19% yield. It was fully characterised by ^{1}H NMR spectroscopy and mass spectrometry. The ^{1}H NMR spectrum of **1** in CD_2Cl_2 shows two doublets for the pyrrolic protons, which is in accordance with a 5,15-*meso*-substituted porphyrin.

Compound **1** was designed to be a good candidate for threading. 2,9-Dianisyl-1,10-phenanthroline is known to thread rapidly and to form stable four-coordinate Cu^I complexes. This chelate was thus selected as the threading element (Figure 2). Compound **1** and $Cu(CH_3CN)_4PF_6$ (2 equiv.) were first mixed in dichloromethane under an

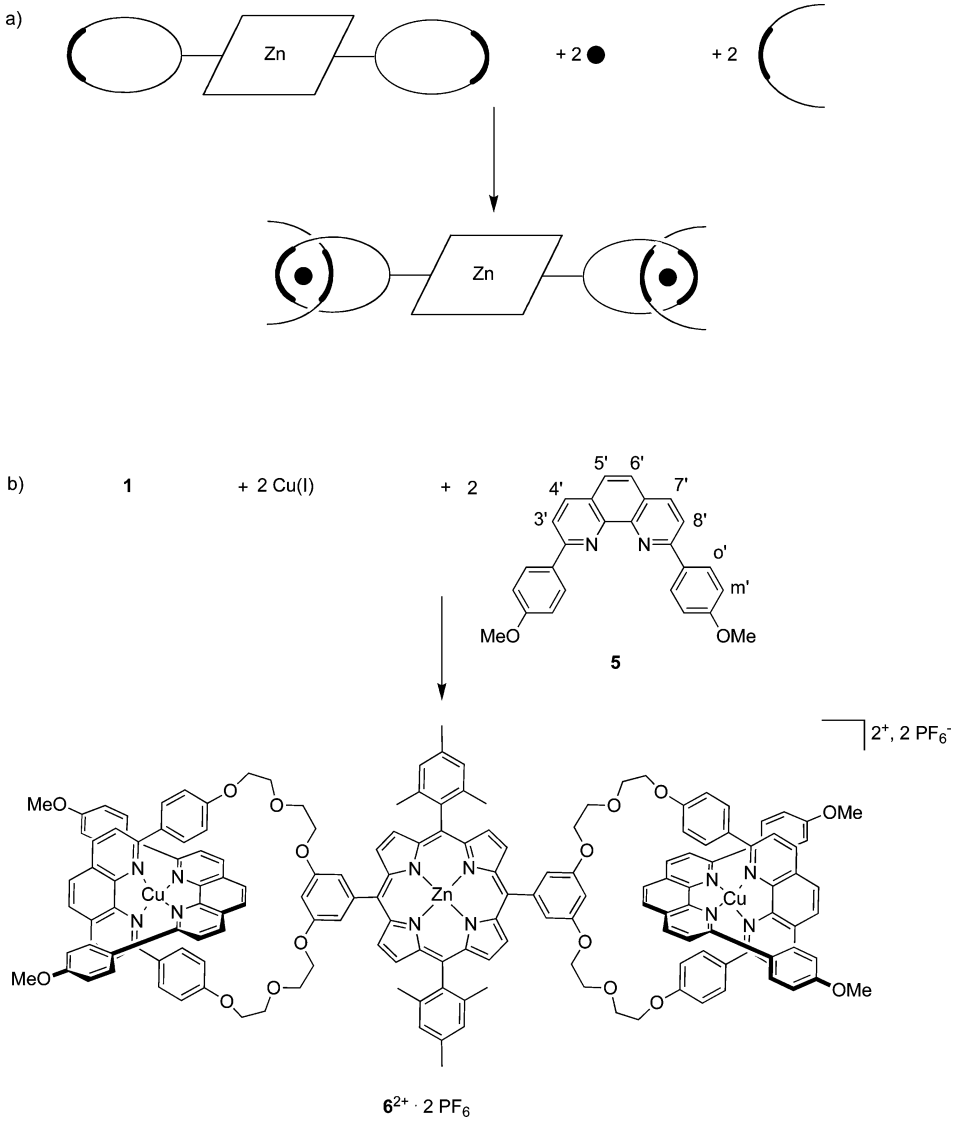

Figure 2. The threading reaction affording pseudorotaxane $\mathbf{6}^{2+}\cdot 2PF_6$. (a) Schematic representation of the double threading reaction and (b) the same reaction with the real molecules.

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atmosphere of argon and compound **5** (2 equiv.) was subsequently added, which led to desired pseudorotaxane $\mathbf{6}^{2+}\cdot 2PF_6$ in quantitative yield after workup. This compound was characterised by ^{1}H NMR, 2D COSY and ROESY spectroscopy and mass spectrometry.

The ^{1}H NMR spectrum of $\mathbf{6}^{2+}\cdot 2PF_6$ (Figure 3) shows that the threading reaction leading to this compound modifies the chemical shifts of most of the hydrogen atoms. In particular, the 3-, 8-, *o*- and *m*-protons undergo a strong upfield shift, which is characteristic of "threaded" complexes.[32] In addition, the CH_2O protons of the ring in compound **1** come out as only two distinct signals. The copper(I) threading enhances the dissymmetry of the CH_2O protons of the ring and four CH_2O signals are observed.

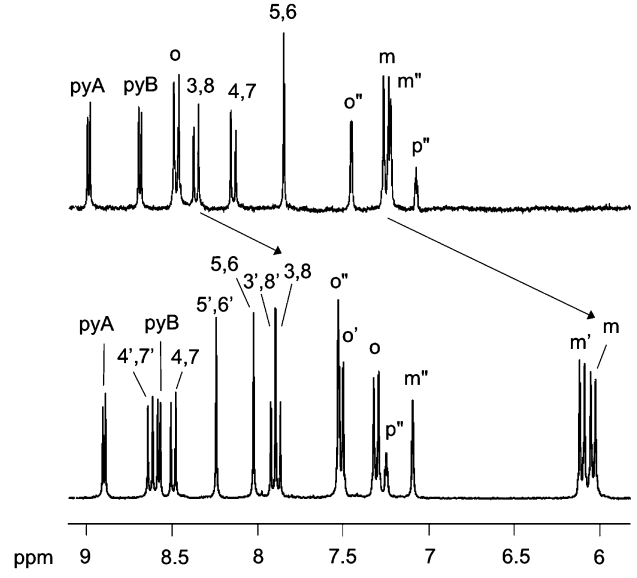

Figure 3. ^{1}H NMR spectra (aromatic region) of compounds **1** (upper) and $\mathbf{6}^{2+}$ (bottom) in CD_2Cl_2.

The absorption and emission spectra of compounds **1** and $\mathbf{6}^{2+}$ were measured in degassed dichloromethane at room temperature. The Soret band is typically observed at 421 nm, whereas the Q-bands are observed at 548 and 585 nm for both compounds. The ^{1}MLCT (metal-to-ligand charge transfer) band of tetrahedral copper(I) complexes containing two ligands of the dpp family, and thus similar to the present complexes, have weak extinction coefficients (a few thousands) relative to those of zinc porphyrins (ten to one hundred times more intense).[33] They are thus difficult to detect in the presence of a porphyrin as a result of spectral overlapping between the Soret and/or Q-bands of the porphyrin and the ^{1}MLCT band. The emission maxima are also observed at a typical wavelength (646 nm after excitation at 548 nm) for both compounds. The emission lifetimes of compounds **1** and $\mathbf{6}^{2+}$ were measured at 650 nm after excitation at 370 nm (which was the only excitation wavelength available for emission-lifetime measurements). The emission lifetime of zinc complex **1** was consistent with simple zinc-complexed porphyrin monomers (τ_1 = 1.85 ± 0.20 ns).[25] The emission lifetime of pseudorotaxane $\mathbf{6}^{2+}$ turned out to be much shorter (τ_2 = 0.15 ± 0.05 ns) than that of copper-free compound **1**. This observation is not especially surprising and is in line with previous work on related compounds containing zinc porphyrins and copper complexes of the $Cu(dpp)_2^+$ family.[25] The singlet excited state of the zinc porphyrin is rapidly quenched by the copper(I) complex owing to the presence of a low-lying triplet MLCT state.

Conclusions

A porphyrin attached to two coordinating macrocycles on the 5- and 15-positions of the tetrapyrrolic ring was synthesised by a condensation reaction. In a first step, a new dpp-containing ring was prepared in good yield by a classical macrocycle-forming reaction. The macrocycle is a 31-membered ring that contains a benzaldehyde group. In the presence of 5-mesityldipyrromethane and under Lindsey's conditions, this compound leads to the desired porphyrin in an acceptable yield of 19% after metallation by zinc acetate. In order to demonstrate that porphyrin **1** can be used in the construction of molecular machines based on relatively complex interlocking systems, a double prototypical threading reaction was carried out successfully with a simple chelate (2,9-dianisyl-1,10-phenanthroline) in the presence of copper(I).

Experimental Section

General: Nuclear magnetic resonance (NMR) spectra were acquired with a Bruker AM300 spectrometer. The spectra were referenced internally to residual proton solvent reference. Mass spectra (ES-MS) were recorded with a VG BOIQ triple quadrupole spectrometer by the Service de Spectrométrie de Masse (ISIS, Strasbourg). Dry solvents were distilled from suitable drying agents (CaH_2 for CH_2Cl_2 and CH_3CN). Commercial chemicals were at the best-available grade and used without further purification. Column chromatography was carried out by using silica gel (Merck Kieselgel, silica gel 60, 0.063–0.200 mm). Compound **2**,[24] $[Cu(MeCN)_4](PF_6)$,[34] 5-mesityldipyrromethane (**4**),[35] and 2,9-dianisyl-1,10-phenanthroline[36] were synthesised according to literature procedures. UV/Vis absorption spectra were recorded with a Kontron UVIKON 860 spectrophotometer. Emission spectra were recorded with a Fluorolog FL3–22 de HORIBA Jobin Yvon spectrophotometer. Lifetime measurements were detected by a TCSPC (time-correlated single-photon counting) technique with a NanoLED-370 electroluminescent diode as the excitation source.

Macrocycle 3: A suspension of Cs_2CO_3 (2.1 g, 5.9 mmol) in DMF (150 mL) was degassed under an atmosphere of argon and heated to 50 °C. In a separate flask, a mixture of ditosylate phenanthroline derivative **2** (1.00 g, 1.20 mmol) and 3,5-dihydroxybenzaldehyde (0.160 g, 1.20 mmol) in DMF (100 mL) was degassed, transferred to an addition funnel and added drop by drop to the suspension over a period of 4 h. The mixture was then heated to 90 °C and stirred under an atmosphere of argon for 16 h. The warm mixture was filtered, and the solid residue was washed with DMF (100 mL). After evaporation, the remaining solid was dissolved in dichloromethane, washed with water (3 × 100 mL) and dried with Na_2SO_4. After evaporation, the crude product was purified by silica gel

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chromatography (dichloromethane/methanol, 100:1) to yield macrocycle **3** as a pale-yellow solid (0.440 g, 57%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.84 (s, 1 H, aldehyde H), 8.41 (d, ^3J = 9.0 Hz, 4 H, H_o), 8.26 (d, ^3J = 8.4 Hz, 2 H, $H_{3,8}$), 8.07 (d, ^3J = 8.4 Hz, 2 H, $H_{4,7}$), 7.75 (s, 2 H, $H_{5,6}$), 7.14 (d, ^3J = 9.0 Hz, 4 H, H_m), 7.03 (d, ^4J = 2.4 Hz, 2 H, $H_{o''}$), 6.91 (t, ^4J = 2.3 Hz, 1 H, $H_{p''}$), 4.32–4.36 (m, 4 H, CH_2), 4.20–4.23 (m, 4 H, CH_2), 3.93–3.97 (m, 8 H, CH_2) ppm. ES-MS: *m*/*z* = 643.25 [M + H]$^+$. $C_{39}H_{34}N_2O_7{\cdot}0.5H_2O{\cdot}CH_2Cl_2$ (736.64): calcd. C 65.22, H 5.07, N 3.80; found C 65.38, H 5.05, N 3.56.

Zn^{II}–Porphyrin 1: To a solution of macrocyclic aldehyde **3** (350 mg, 0.546 mmol) in dry dichloromethane (55 mL) was added trifluoroacetic acid (218 μL, 2.93 mmol) and 5-mesityldipyrromethane (**4**; 147 mg, 0.546 mmol). The flask was protected from light, and the solution was stirred for 2 h under an atmosphere of argon. DDQ (2,3-dichloro-5,6-dicyanobenzoquinone; 210 mg, 0.869 mmol) was added, and the solution was stirred under an atmosphere of argon for an additional 1 h. A solution of Na_2CO_3 (1.4 g) in water (40 mL) was added, and the solution was stirred vigorously for 30 min. The two phases were separated, and the aqueous layer was washed with dichloromethane (2 × 15 mL). The organic solvents were evaporated, and the compound was purified by silica gel chromatography (dichloromethane/methanol, 100:0.5). After a second purification by flash chromatography (silica; dichloromethane/methanol, 100:0.2), the free-base porphyrin (97 mg, 0.053 mmol) was obtained and directly metallated. It was dissolved in dichloromethane (80 mL) and a solution of $Zn(OAc)_2{\cdot}2H_2O$ (95 mg, 0.430 mmol) in methanol (20 mL) was then added. The mixture was heated at reflux for 8 h. Then, an aqueous solution of [EDTA]-Na_4 (0.1 M, 45 mL) was added, and the biphasic mixture was vigorously stirred for 24 h at room temperature. The organic layer was separated and washed with water (3 × 30 mL). After evaporation, compound **1** was obtained as a violet solid (93 mg, 19%). ^{1}H NMR (300 MHz, CD_2Cl_2): δ = 8.92 (d, ^3J = 4.7 Hz, 4 H, H_{pyA}), 8.67 (d, ^3J = 4.6 Hz, 4 H, H_{pyB}), 8.45 (d, ^3J = 8.8 Hz, 8 H, H_o), 8.28 (d, ^3J = 8.4 Hz, 4 H, $H_{3,8}$), 8.08 (d, ^3J = 8.4 Hz, 4 H, $H_{4,7}$), 7.77 (s, 4 H, $H_{5,6}$), 7.42 (d, ^4J = 2.4 Hz, 4 H, $H_{o''}$), 7.20 (d, ^3J = 8.8 Hz, 8 H, H_m), 7.19 (s, 4 H, $H_{m''}$), 7.04 (t, ^4J = 2.4 Hz, 2 H, $H_{p''}$), 4.31 (m, 16 H, CH_2O), 3.91 (m, 16 H, CH_2O), 2.58 (s, 6 H, CH_{3p}), 1.75 (s, 12 H, CH_{3o}) ppm. UV/Vis (CH_2Cl_2): λ (log ε) = 421 (5.76), 549 (4.37), 585 (3.63) nm. ES-MS: *m*/*z* = 1833.51 [M + H]$^+$.

Pseudorotaxane 6^{2+}: A degassed solution of $[Cu(MeCN)_4](PF_6)$ (5.15 mg, 13.8×10^{-6} mol) in CH_3CN (3 mL) was added by cannula to a degassed solution of **1** (12.2 mg, 6.65×10^{-6} mol) in CH_2Cl_2 (10 mL), and the mixture was stirred under an atmosphere of argon for 30 min. A degassed solution of 2,9-dianisyl-1,10-phenanthroline (**5**; 5.42 mg, 13.8×10^{-6} mol) in CH_2Cl_2 (5 mL) was then added by cannula. After stirring for 3 h at room temperature, the solvent was evaporated. The solid was dissolved in acetonitrile and filtered. The solvent was then evaporated to give pseudorotaxane $\mathbf{6}^{2+}{\cdot}2PF_6$ in quantitative yield (18.0 mg). ^{1}H NMR (300 MHz, CD_2Cl_2): δ = 8.90 (d, ^3J = 4.8 Hz, 4 H, H_{pyA}), 8.64 (d, ^3J = 8.4 Hz, 4 H, $H_{4',7'}$), 8.58 (d, ^3J = 4.6 Hz, 4 H, H_{pyB}), 8.50 (d, ^3J = 8.2 Hz, 4 H, $H_{4,7}$), 8.25 (s, 4 H, $H_{5',6'}$), 8.04 (s, 4 H, $H_{5,6}$), 7.92 (d, ^3J = 8.3 Hz, 4 H, $H_{3',8'}$), 7.89 (d, ^3J = 8.0 Hz, 4 H, $H_{3,8}$), 7.53 (s, 4 H, $H_{o''}$), 7.52 (d, ^3J = 8.6 Hz, 8 H, $H_{o'}$), 7.32 (d, J = 8.4 Hz, 8 H, H_o), 7.29 (m, 2 H, $H_{p''}$), 7.09 (s, 4 H, $H_{m''}$), 6.12 (d, ^3J = 8.6 Hz, 8 H, $H_{m'}$), 6.05 (d, ^3J = 8.6 Hz, 8 H, H_m), 4.43 (m, 8 H, CH_2O), 3.97 (m, 8 H, CH_2O), 3.72 (m, 8 H, CH_2O), 3.67 (m, 8 H, CH_2O), 3.52 (s, 12 H, OMe), 2.35 (s, 6 H, CH_{3p}), 1.68 (s, 12 H, CH_{3o}) ppm. UV/Vis (CH_2Cl_2): λ (log ε) = 421 (5.84), 548 (4.51), 585 (3.79) nm. ES-MS: *m*/*z* = found 1371.37 [M / 2]$^+$.

Acknowledgments

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