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A trefoil-shaped macrocycle with [12]-imidazolium cations

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ABSTRACT

A trefoil-like two-dimensional (2D) C_{3v} symmetric organic [12]-imidazolium cation H_{12}–**2**$(PF_6)_{12}$ featuring three [4]-imidazolium macrocycles was synthesized in three steps. The reaction of a dodecakis H_{12}–**1**$(PF_6)_{12}$ imidazolium salt with Ag_2O resulted in the formation of a hexanuclear Ag^I dodecacarbene assembly $[Ag_6($**1**$)](PF_6)_6$. Upon UV irradiation, the photodimerization of the cinnamic ester pendants of $[Ag_6($**1**$)](PF_6)_6$ led to the generation of a trefoil-like complex $[Ag_6($**2**$)](PF_6)_6$ containing three closed metallacycles. Removal of metal ions allowed for the synthesis of the target molecule. All complexes were fully characterized by NMR spectroscopy (^1H, $^{13}C\{^1H\}$ and 2D NMR) and high-resolution electrospray ionization mass spectrometry (HR-ESI-MS).

The discrete macrocyclic compounds, which contain both artificial metal-macrocycles (triangles, rhomboids, squares, hexagons, *etc.*) [1–12] and non-metal-macrocycles (crown ethers, cyclodextrins, cyclophanes, pillararenes, *etc.*) [13–18], have made significant progress in the application of host-guest chemistry, selective absorption/recognition, molecular electronic devices and supramolecular catalysis fields over the past decades [19–24]. Among these macrocyclic compounds, the polyimidazolium cyclophanes have drawn considerable attention as unique receptors due to the ionic hydrogen bond $(C\text{-}H)^+\cdots X^-$ between the imidazolium units and guests recently [23,25-31]. The polyimidazolium cyclophanes can also be utilized as precursors for the formation of functionalized assemblies bearing *N*-heterocyclic carbenes (NHCs) [32–47].

So far, attempts to make [*n*]-polyimidazolium-based ($n > 4$) cyclophanes by one-pot macrocyclization or stepwise macrocyclization processes have proved unsuccessful [48–50], probably due to the multiple incorporations of the dihaloalkanes and bis-imidazole precursors with required connectivity becomes more difficult as reactive sites increases. For example, Beer and co-workers isolated an organic polyimidazolium cation featuring 8 internal imidazolium units only as few crystals incidentally [48]. To overcome this limitation, our group developed a metal-carbene template approach and a series of three-dimensional [*n*]-imidazolium ($n = 6$, 12, 16 or 18) cages were controllably constructed [51–55].

In addition, most of the known polyimidazolium cationic compounds only have one macrocyclic unit of polyimidazolium. It should be noted that the increase in the number of macrocycles can bring potential properties [56–60]. For example, Stoddart and co-workers reported a trefoil-like rotaxane, which was used for a molecule elevator [57]. Therefore, the design and synthesis of the polyimidazolium-based compounds featuring more than one macrocyclic unit are imminent and challenging.

Based on these results, we attempted to construct a trefoil-like 2D C_{3v} symmetric organic [12]-imidazolium cation featuring three [4]-imidazolium macrocycles. Unfortunately, direct synthesis of compound **IV** by the one-pot method using the olefin-modified dodecakisimidazolium salt **I** as the precursor failed after various trials. Here, we reported an alternative method by using the precursor **I** to react with Ag_2O to generate the hexanuclear Ag^I assembly **II**. Then photochemically induced [2+2] cycloaddition reaction was performed with complex **II**, thereby leading to a trefoil-like complex containing three closed metallacycles. Finally, all Ag^I ions in complex **III** could be eventually removed to provide the target compound **IV** (Scheme 1).

The preparation of 1,2,3,4,5,6-hexakis(4-(1*H*-imidazol-1-yl)phenyl)benzene (**L**) and *N*-cinnamic-ester-appended dodecakisimidazolium precursor H_{12}–**1**$(PF_6)_{12}$ is shown in Scheme 2. Firstly, the compound **L** was obtained from imidazole and 1,2,3,4,5,6-hexakis(4-iodophenyl)benzene [61] by the solid-state reaction using an Ullmann coupling protocol (Fig. S1 in Supporting information) [62,63]. Subsequently, compound **L** and *N*-*p*-bromomethylbenzene-*N*'-cinnamic acid methyl ester imidazolium

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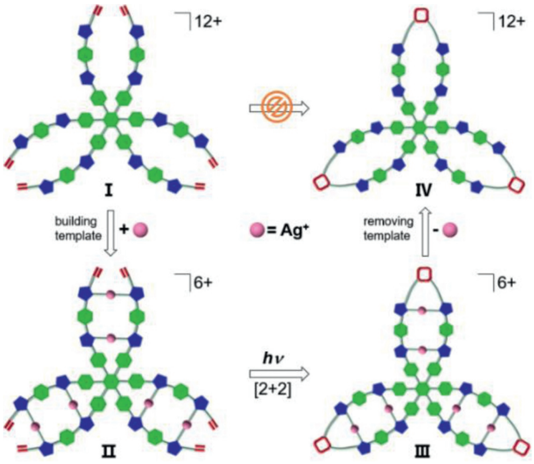


Scheme 1. Cartoon presentation of the synthetic strategy for the construction of a trefoil-like dodecakisimidazolium cation.

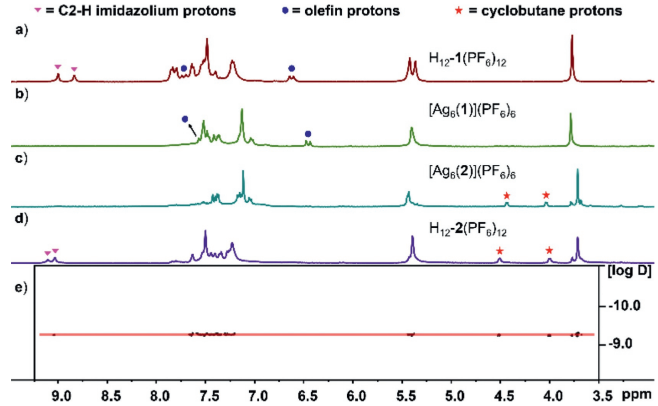
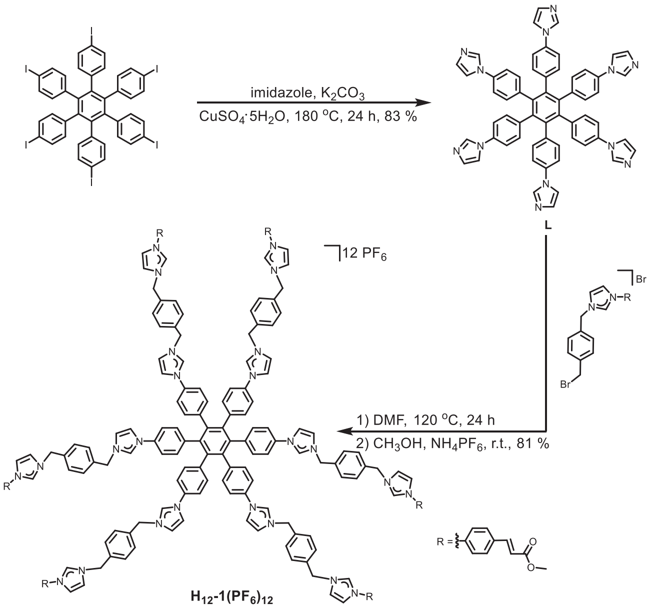


Fig. 1. Partial ^{1}H NMR spectra (400 MHz, 298 K) in CD_3CN of (a) dodecakisimidazolium salt H_{12}–**1**$(PF_6)_{12}$; (b) complex $[Ag_6(\mathbf{1})](PF_6)_6$ before irradiation; (c) complex $[Ag_6(\mathbf{2})](PF_6)_6$ obtained after irradiation; (d) dodecakisimidazolium salt H_{12}–**2**$(PF_6)_{12}$; (e) ^{1}H DOSY spectrum of H_{12}–**2**$(PF_6)_{12}$.



Scheme 2. Synthesis of compounds **L** and H_{12}–**1**$(PF_6)_{12}$.

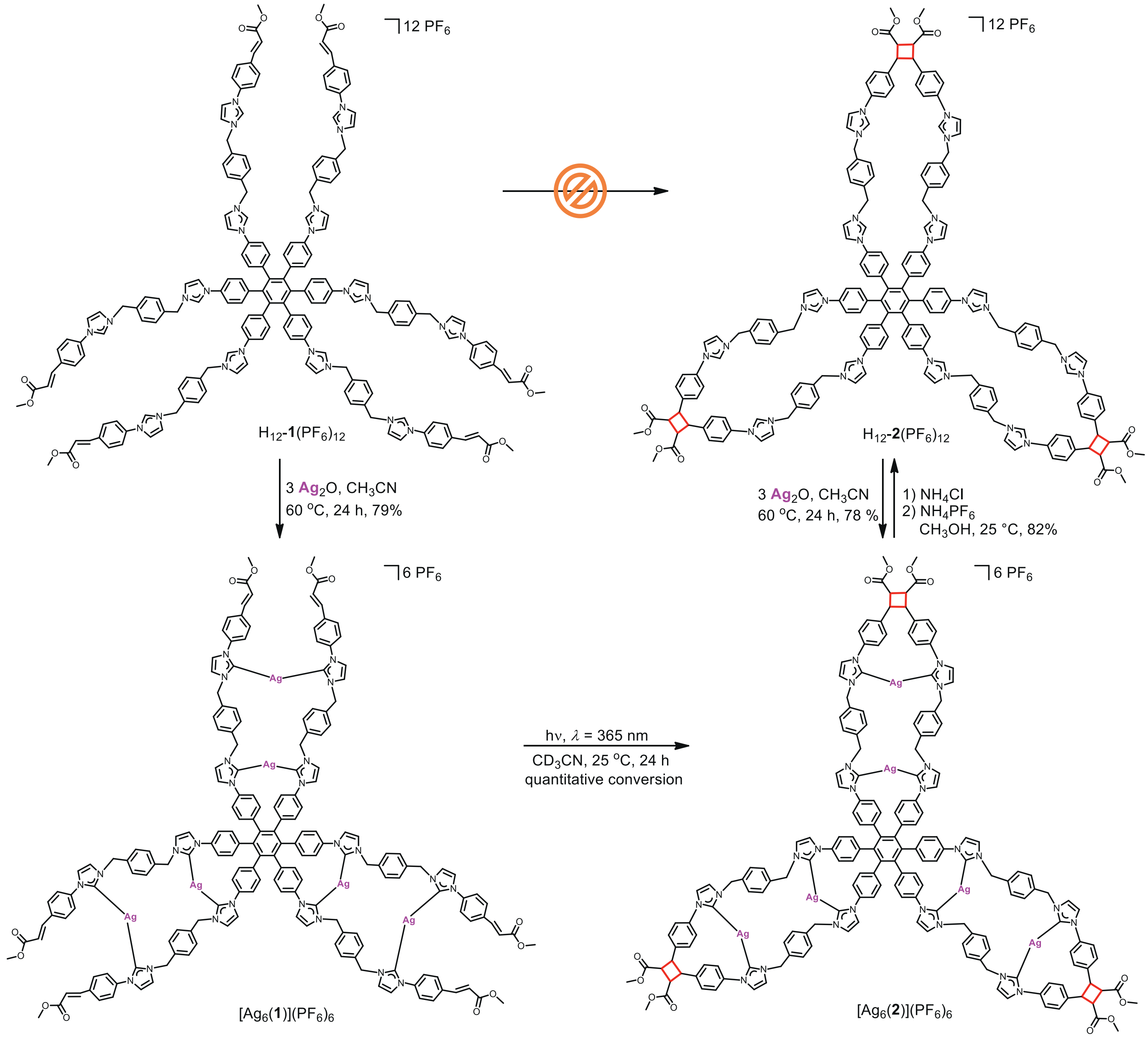
bromide was mixed to yield the corresponding dodecakisimidazolium precursor H_{12}–**1**$(Br)_{12}$, followed by the anion exchanged with NH_4PF_6 in CH_3OH to form hexafluorophosphate H_{12}–**1**$(PF_6)_{12}$ in 81% yield as a white solid. The hexafluorophosphate salt of H_{12}–**1**$(PF_6)_{12}$ is excellently soluble in DMSO, DMF, and CH_3CN, whereas almost insoluble in ether and dichloromethane.

The formation of the dodecakisimidazolium precursor H_{12}–**1**$(PF_6)_{12}$ was verified by nuclear magnetic resonance (NMR) spectroscopy (^{1}H, ^{13}C{^{1}H}, and 2D NMR) and high-resolution electrospray ionization mass spectrometry (HR-ESI-MS) (Figs. S2-S7 in Supporting information). The ^{1}H NMR spectrum of H_{12}–**1**$(PF_6)_{12}$ in CD_3CN reveals two characteristic resonances for the C2-H protons on imidazolium at δ 9.00 and 8.84 ppm, respectively (Fig. 1a). HR-ESI-MS spectrum provided further support for the formation of dodecakisimidazolium salt. The peaks of the highest intensity for H_{12}–**1**$(PF_6)_{12}$ were discovered at *m/z* 632.4934 (calcd. for $[H_{12}$–**1**$+6PF_6]^{6+}$ 632.5114), *m/z* 1021.2212 (calcd. for $[H_{12}$–**1**$+8PF_6]^{4+}$ 1021.2495).

The reaction of H_{12}–**1**$(PF_6)_{12}$ with an excess of Ag_2O generated the 2D C_{3v} symmetric hexanuclear Ag^{I} dodecacarbene assembly $[Ag_6(\mathbf{1})](PF_6)_6$ in 79% yield (Scheme 3), the compound $[Ag_6(\mathbf{1})](PF_6)_6$ contains two triangular metal silver ions form an inner Ag_3 triangle inserted inside a larger outer Ag_3 arrangement. The reaction was achieved in CH_3CN under the exclusion of light, and the carbene complex was separated as a white solid after workup. The formation of the hexanuclear silver complex was unambiguously confirmed by NMR and HR-ESI-MS studies (Figs. S8-S14 in Supporting information). In the ^{1}H NMR spectra of complex $[Ag_6(\mathbf{1})](PF_6)_6$, the characteristic resonance of the C2-H protons of H_{12}–**1**$(PF_6)_{12}$ at δ 9.00 and 8.84 ppm were found to have disappeared (Fig. 1b). The ^{13}C{^{1}H} NMR spectra in CD_3CN showed the two expected C_{NHC} signals at δ 182.1 and 180.7 ppm, accompanied by two slightly different imidazolium rings. These values are within the normal range compared with previously reported for such derivatives [64–88]. Diffusion-ordered NMR spectroscopy (DOSY) confirmed the formation of a signal configuration. All proton signals of complex $[Ag_6(\mathbf{1})](PF_6)_6$ displayed a closely related diffusion coefficient for $D = 6.46 \times 10^{-10}$ m^2/s ($\log D = -9.19$). The HR-ESI-MS spectrum further demonstrated the formation of hexanuclear silver complex. For example, the HR-ESI-MS spectrum for $[Ag_6(\mathbf{1})](PF_6)_6$ appeared two peaks at *m/z* 593.4065 (calcd for $[Ag_6(\mathbf{1})]^{6+}$ 593.4365), *m/z* 741.0843 (calcd for $[Ag_6(\mathbf{1})+PF_6]^{5+}$ 741.1168), these theoretical isotopic distributions are consistent with the experimental peaks (Fig. S14 in Supporting information). Unfortunately, all attempts to obtain single crystals of the hexanuclear silver complex $[Ag_6(\mathbf{1})](PF_6)_6$ used for X-ray analysis failed. However, based on the related assemblies [56–60], the 2D C_{3v} symmetric structure $[Ag_6(\mathbf{1})](PF_6)_6$ could be speculated in Scheme 3.

The photochemical reaction of the *N*-cinnamic-ester-appended hexanuclear Ag^{I} dodecacarbene assembly $[Ag_6(\mathbf{1})](PF_6)_6$ was subsequently investigated (Scheme 3). The complex $[Ag_6(\mathbf{1})](PF_6)_6$ was treated with a Philips high-pressure mercury lamp ($\lambda = 365$ nm) in CD_3CN solvent at ambient temperature converted exclusively into the organometallic complex $[Ag_6(\mathbf{2})](PF_6)_6$. The photochemical [2+2] cyclization reaction was finished within 24 h and the conversion was easily demonstrated by ^{1}H, ^{13}C{^{1}H} NMR spectroscopy.

The formation of the hexanuclear complex $[Ag_6(\mathbf{2})](PF_6)_6$ featuring three cyclobutanes bridged was monitored by NMR spectroscopy (Figs. S15-S20 in Supporting information). Before irradiation, the ^{1}H NMR spectrum from the olefin protons of $[Ag_6(\mathbf{1})](PF_6)_6$ features characteristic two doublet resonances of the cinnamic ester groups at δ 7.56 ($^{3}J = 16.1$ Hz, partly overlapped by other resonances) and 6.46 ($^{3}J = 16.1$ Hz) ppm (in CD_3CN, Fig. 1b). The ^{1}H NMR spectrum revealed that olefin protons resonances of H_{12}–**1**$(PF_6)_{12}$ (Fig. 1a) slightly shifted upfield after producing



Scheme 3. Preparation of the hexanuclear Ag^I dodecacarbene assembly $[Ag_6(\mathbf{1})](PF_6)_6$, of the organometallic complex $[Ag_6(\mathbf{2})](PF_6)_6$ and of the dodecakisimidazolium cation H_{12}–$\mathbf{2}(PF_6)_{12}$.

to $[Ag_6(\mathbf{1})](PF_6)_6$. The $^{13}C\{^1H\}$ NMR spectrum of $[Ag_6(\mathbf{1})](PF_6)_6$ features signals for the C=C carbon atoms were observed at δ 143.4 and 120.2 ppm (Fig. S9 in Supporting information). After UV-irradiation ($\lambda = 365$ nm) for 24 h, the intensity of the olefin doublet resonances completely vanished and two new multiplets for the cyclobutane protons of $[Ag_6(\mathbf{2})](PF_6)_6$ generated at δ 4.43 and 4.03 (both d, $^3J = 5.51$ Hz) ppm (in CD_3CN, Fig. 1c), while two characteristic peaks for cyclobutane carbons were also found at δ 52.7 and 46.1 ppm (Fig. S16 in Supporting information). Additionally, the formation of $[Ag_6(\mathbf{2})](PF_6)_6$ featuring three cyclobutanes have demonstrated by the HR-ESI-MS spectrum, showed peaks of highest intensity at *m/z* 593.4272 (calcd for $[Ag_6(\mathbf{2})]^{6+}$ 593.4365), *m/z* 741.1054 (calcd. for $[Ag_6(\mathbf{2})+PF_6]^{5+}$ 741.1168) (Fig. S21 in Supporting information). Subsequently, UV–vis spectra also clearly revealed the disappearance of the characteristic absorption peaks at around 280 nm, attributed to the C=C double bonds (Fig. S29 in Supporting information). The photochemical [2+2] cycloaddition reaction of precursor H_{12}–$\mathbf{1}(PF_6)_{12}$ was performed under the same experimental conditions. However, no target photoproduct was found except the *trans-cis* photoisomerization as indicated by ^{1}H NMR spectroscopy (Fig. S22 in Supporting information).

In our previous work, it has been confirmed that the newly generated terminal cyclobutanes can connect two poly-NHC ligands after removing the silver ions [51–55]. Inspired by these results, the next generation of trefoil-like dodecakisimidazolium cation H_{12}–$\mathbf{2}(PF_6)_{12}$ was prepared. The addition of excess NH_4Cl in CH_3OH successfully bring the liberation of the six Ag^+ ions from hexanuclear complex $[Ag_6(\mathbf{2})](PF_6)_6$, followed by anion exchange with NH_4PF_6 provided hexafluorophosphate H_{12}–$\mathbf{2}(PF_6)_{12}$ in 82% yield as a white solid (Scheme 3). The trefoil-like dodecakisimidazolium cation H_{12}–$\mathbf{2}(PF_6)_{12}$ was fully characterized by NMR spectroscopy and HR-ESI mass spectrometry (Figs. S23-S28 in Supporting information). In the ^{1}H NMR spectrum of H_{12}–$\mathbf{2}(PF_6)_{12}$, two resonances

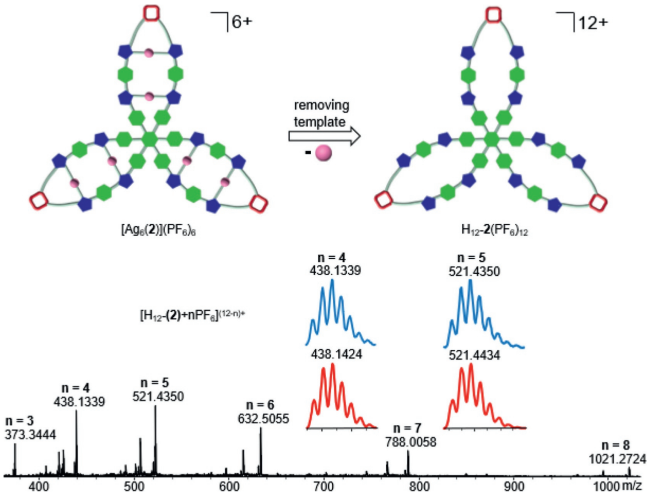


Fig. 2. HR-ESI-MS spectrum (position ion mode) of H_{12}–$\mathbf{2}(PF_6)_{12}$, inset experimental (blue) and calculated (red) isotope distributions for peaks corresponding to $[H_{12}$–$\mathbf{2}+4PF_6]^{8+}$ and $[H_{12}$–$\mathbf{2}+5PF_6]^{7+}$.

were observed at δ 9.10 and 9.03 ppm (Fig. 1d) because of imidazolium groups of dodecakisimidazolium salt H_{12}–$\mathbf{2}(PF_6)_{12}$ have two different chemical environments. This is consistent with the dodecakisimidazolium precursor starting material. The intensity ratio of the cyclobutane protons and the imidazolium protons was checked to be 1:1:1:1, indicating the fully de-metalation of complex $[Ag_6(\mathbf{2})](PF_6)_6$. All proton signals of H_{12}–$\mathbf{2}(PF_6)_{12}$ showed a relatively narrow interval with a diffusion coefficient of $D = 5.62 \times 10^{-10}$ m^2/s ($\log D = -9.25$), verifying that it is a single assembly (Fig. 1e). Moreover, the HR-ESI-MS spectrum of compound H_{12}–$\mathbf{2}(PF_6)_{12}$ (positive-ion mode) revealed peaks of highest intensity at $m/z = 438.1339$ (calcd for $[H_{12}$–$\mathbf{2}+4PF_6]^{8+}$ 438.1424), $m/z = 521.4350$ (calcd for $[H_{12}$–$\mathbf{2}+5PF_6]^{7+}$ 521.4434) (Fig. 2). Furthermore, the addition of 3 equiv. Ag_2O to H_{12}–$\mathbf{2}(PF_6)_{12}$ resulted in regeneration of the hexanuclear cycloaddition product $[Ag_6(\mathbf{2})](PF_6)_6$. This reaction was achieved after 24 h at 60 °C under the exclusion of light (Scheme 3). The HR-ESI-MS spectrum data were similar to those of $[Ag_6(\mathbf{2})](PF_6)_6$ obtained from the photochemical cycloaddition of $[Ag_6(\mathbf{1})](PF_6)_6$ (Fig. S21 in Supporting information).

In summary, based on the rational design of a hexagonal poly-NHC precursor, we described the synthesis of two different discrete 2D C_{3v} symmetric hexanuclear Ag^I dodecacarbene metallacycles and corresponding trefoil-like 2D C_{3v} symmetric organic [12]-imidazolium cation featuring three [4]-imidazolium macrocycles. To the best of our knowledge, this is the first example of 2D polyimidazolium derivative containing three [4]-imidazolium macrocycles. In addition, this approach potentially prepares newly functional two-dimensional polyimidazolium materials under study in our group.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.01.064.

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