Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

Studies Towards the Synthesis of Pd(II)-containing [2] and [3]Catenanes in Aqueous Media

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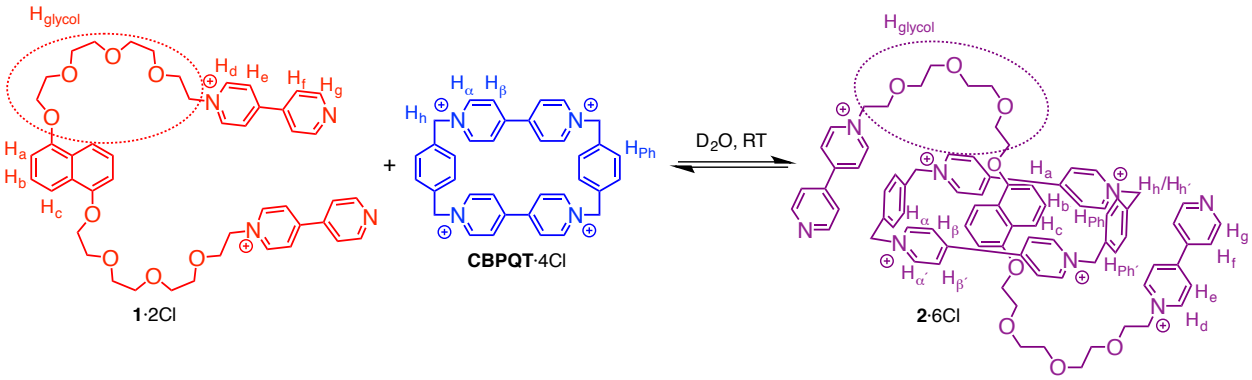
Section A. Materials / General Methods / Instrumentation

All reagents were purchased from commercial suppliers and used without further purification. Axle **1**·2Cl[1] and **CBPQT**·4Cl,[2] were prepared according to literature procedures. All other reagents used were commercial grade chemicals from freshly opened containers. Milli-Q water was purified with a Millipore Gradient A10 apparatus. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 300, Bruker Advance III and Agilent DD2 spectrometers equipped with a dual cryoprobe for ^{1}H and ^{13}C with working frequencies of 300, 500 and 600 MHz, respectively (^{1}H NMR), and 75, 125 and 150 MHz, respectively (^{13}C NMR) using the deuterated solvent as lock and the residual protiated solvent as internal standard. Chemical shifts are reported in ppm relative to the signals corresponding to the residual non-deuterated solvents (D_2O: δ_H = 4.79 ppm). ^{13}C NMR spectra were recorded with the simultaneous decoupling of ^{1}H nuclei. Mass spectrometry experiments were carried out in a LC-Q-q-TOF Applied Biosystems QSTAR Elite spectrometer for low- and high-resolution ESI.

Section B. Synthetic Procedures and Characterization

*Preparation of pseudorotaxane **2**·6Cl*



Scheme S1. Self-assembly of pseudorotaxane **2**·6Cl.

A solution of **1**·2Cl (12.1 mg, 0.014 mmol) and **CBPQT**·4Cl (9.3 mg, 0.014 mmol) in D_2O (2.8 mL), containing NaCl (115 mg, 1.96 mmol), was stirred at room temperature. After a few minutes the mixture reaches the equilibrium, becoming purple color and yielding the pseudorotaxane **2**·6NO_3 as major species along with a small amount of **CBPQT**·4Cl and **1**·2Cl. The following signals correspond to new species, pseudorotaxane **2**·6Cl. *^{1}H NMR (500 MHz, D_2O, 298 K) δ*: 9.05 (8H, 2d), 8.90 (4H, d, *J* = 6.5 Hz), 8.87 (4H, d_{broad}), 8.42 (4H, d, *J* = 6.7 Hz), 7.97 (4H, d, *J* = 4.8 Hz), 7.92 (8H, 2s), 7.58 (4H, d, *J* = 5.1 Hz), 7.41 (4H, d, *J* = 5.2 Hz), 6.40 (2H, d, *J* = 7.9 Hz), 6.06 (2H, t, *J* = 8.0 Hz), 5.71 (8H, s), 4.94-4.92 (4H, m), 4.36-4.35 (4H, m), 4.26-4.25 (4H, m), 4.16-4.14 (4H, m), 4.11-4.10 (4H, m), 4.00-3.99 (4H, m), 3.85-3.82 (8H, m), 2.42-2.40 (2H, d, *J* = 8.2 Hz). *^{13}C NMR (125 MHz, D_2O, 298 K) δ:* 153.0 (C), 151.07 (C), 149.5 (CH), 145.4 (CH), 145.1 (C), 145.0 (CH), 143.6 (CH), 142.5 (C), 136.3 (C), 131.3 (CH), 131.0 (CH), 128.2 (CH), 126.0 (CH),

125.4 (CH), 124.7 (CH), 124.4 (C), 122.8 (CH), 108.3 (CH), 104.2 (CH), 70.5 (CH_2), 69.7 ($2CH_2$), 69.6 (CH_2), 68.5 ($2CH_2$), 68.0 (CH_2), 64.8 (CH_2), 60.7 (CH_2).

*^{1}H NMR comparative of **CBPQT**·4Cl, pseudorotaxane **2**·6Cl and axle **1**·2Cl*

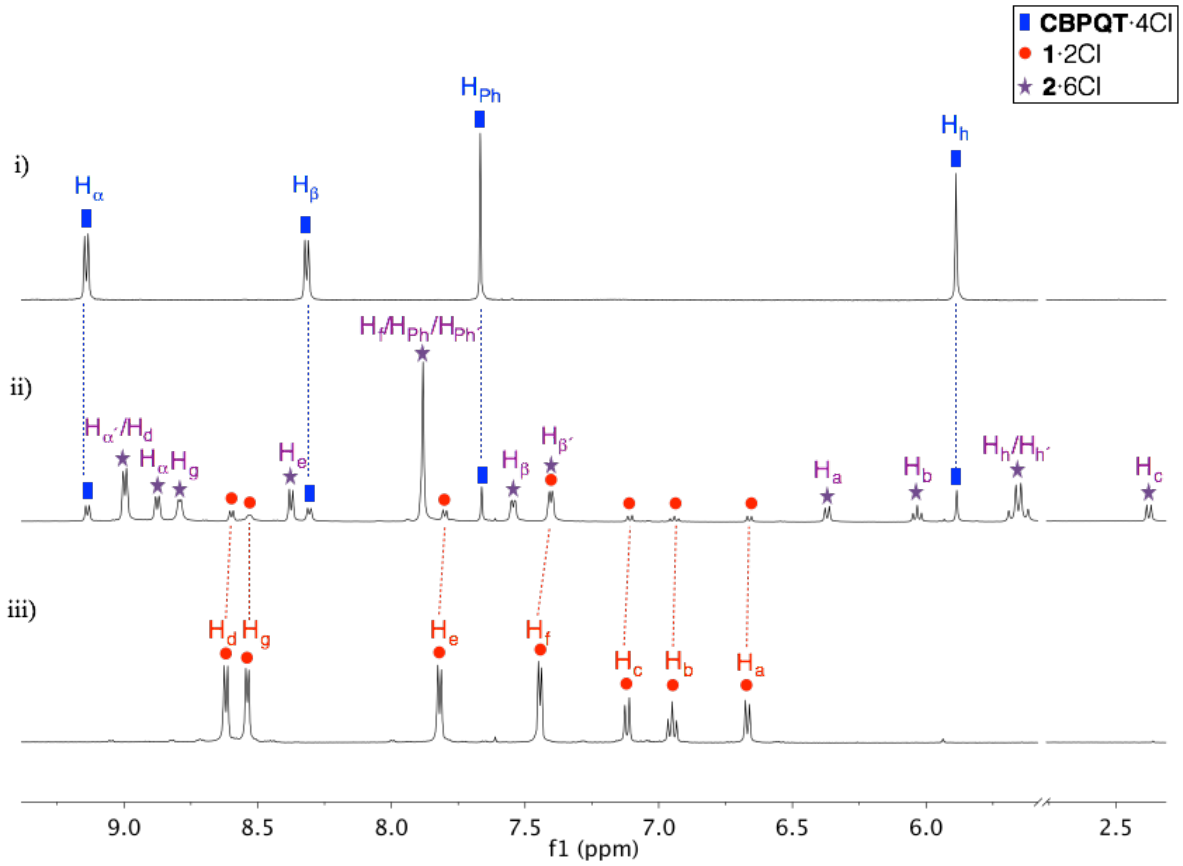


Fig. S1. Partial ^{1}H NMR spectra (500 MHz, D_2O, 298 K) of: i) **CBPQT**·4Cl, ii) an equimolar mixture of **1**·2Cl and **CBPQT**·4Cl (5 mM), iii) **1**·2Cl.

*Variable temperature ^{1}H NMR experiments of pseudorotaxane **2**·6Cl*

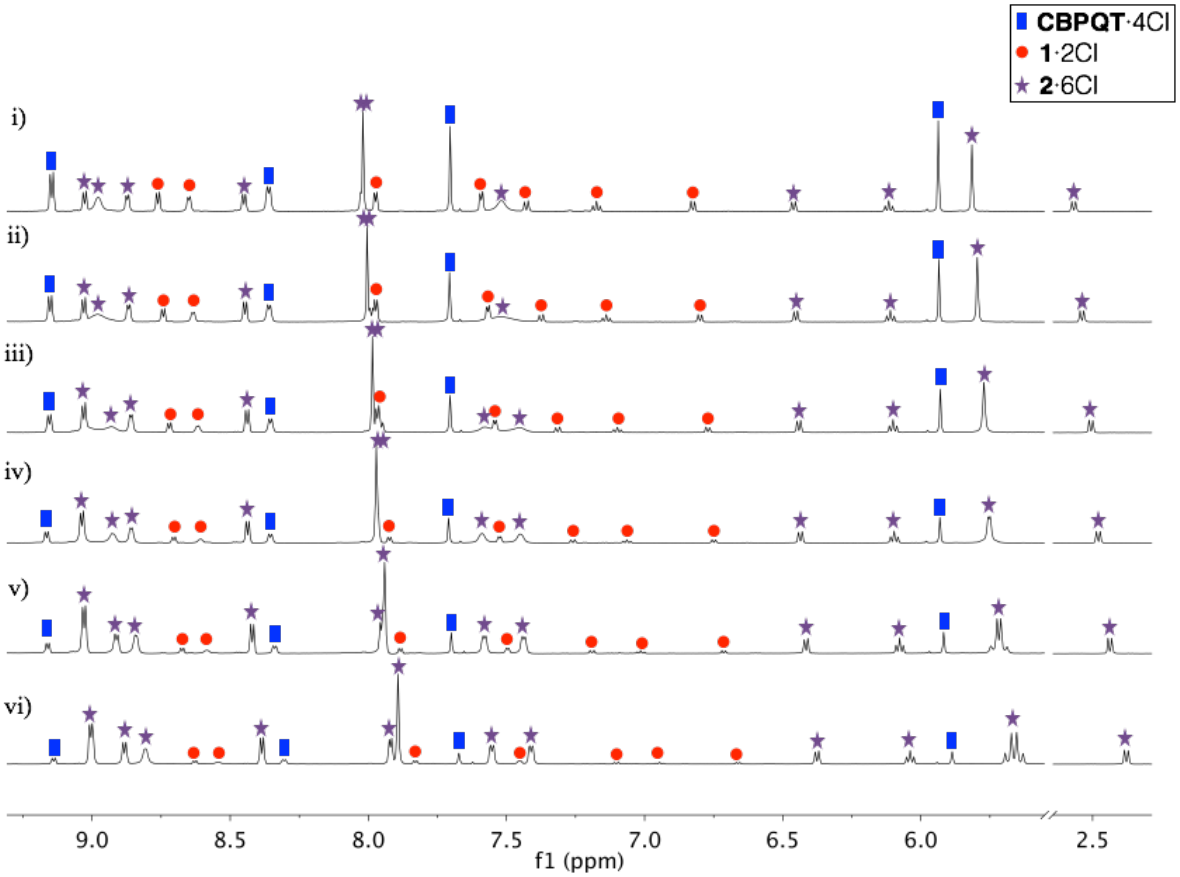


Fig. S2. Variable temperature partial ^{1}H NMR spectra (600 MHz, D_2O) of an equimolar mixture of **1**·2Cl and **CBPQT**·4Cl (5 mM) at: i) 348 K, ii) 338 K, iii) 328 K, iv) 318 K, v) 308 K and vi) 298 K.

*^{1}H NMR comparative of pseudorotaxane **2**·6Cl with and without addition of salt (NaCl)*

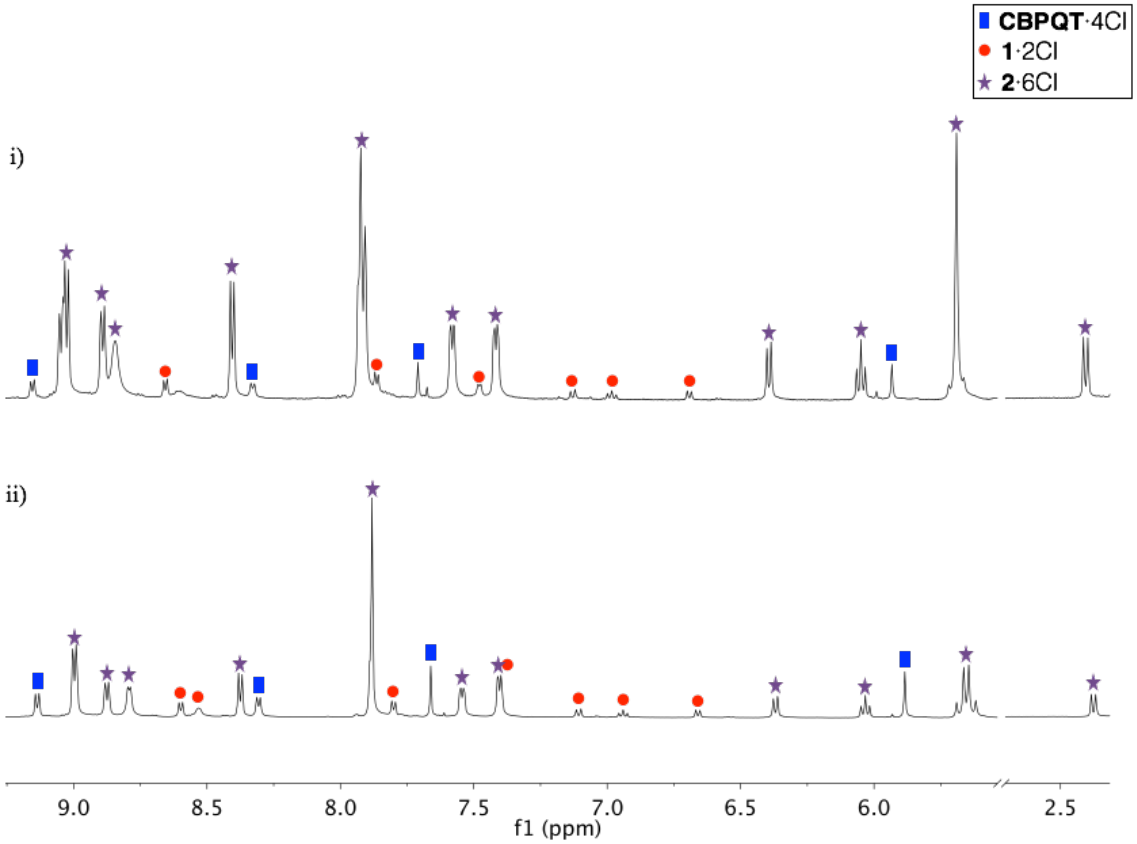


Fig. S3. Partial ^{1}H NMR spectra (500 MHz, D_2O, 298 K) of an equimolar mixture of **1**·2Cl and **CBPQT**·4Cl (5 mM): i) containing salt (NaCl (0.7 M)) and ii) without salt.

*NMR characterization of pseudorotaxane **2**·6Cl in the presence of salt (NaCl)*

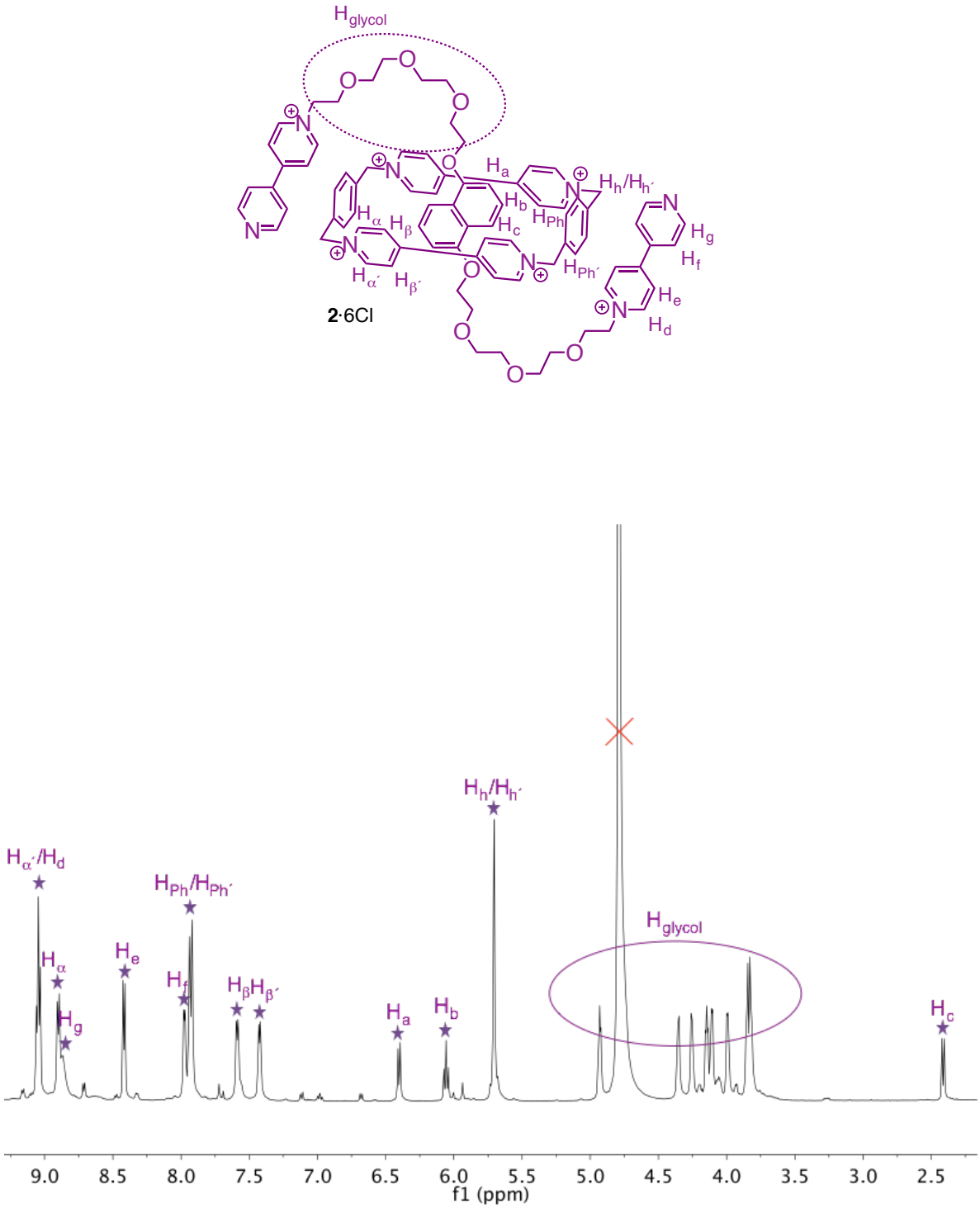


Fig. S4. Annotated ^{1}H NMR spectrum (500 MHz, D_2O, 298 K) of pseudorotaxane **2**·6Cl in the presence of NaCl. Red cross denotes residual solvent peak.

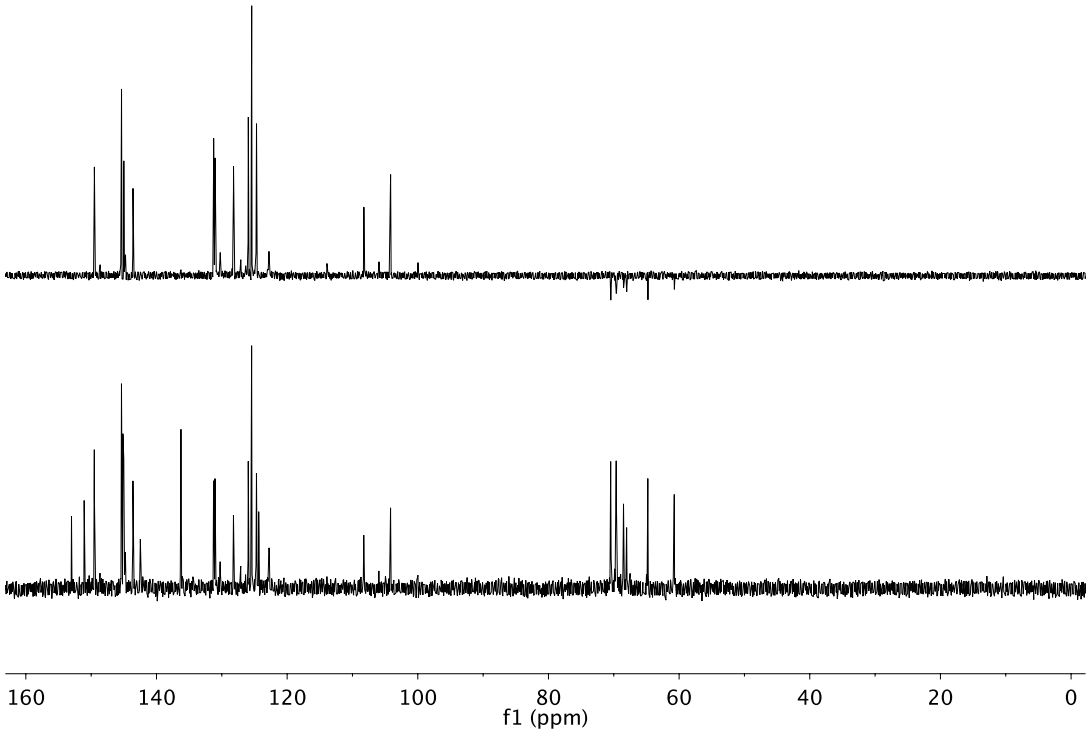


Fig. S5. ^{13}C NMR and DEPT-135 spectra (125 MHz, D_2O, 298 K) of pseudorotaxane **2**·6Cl in the presence of NaCl.

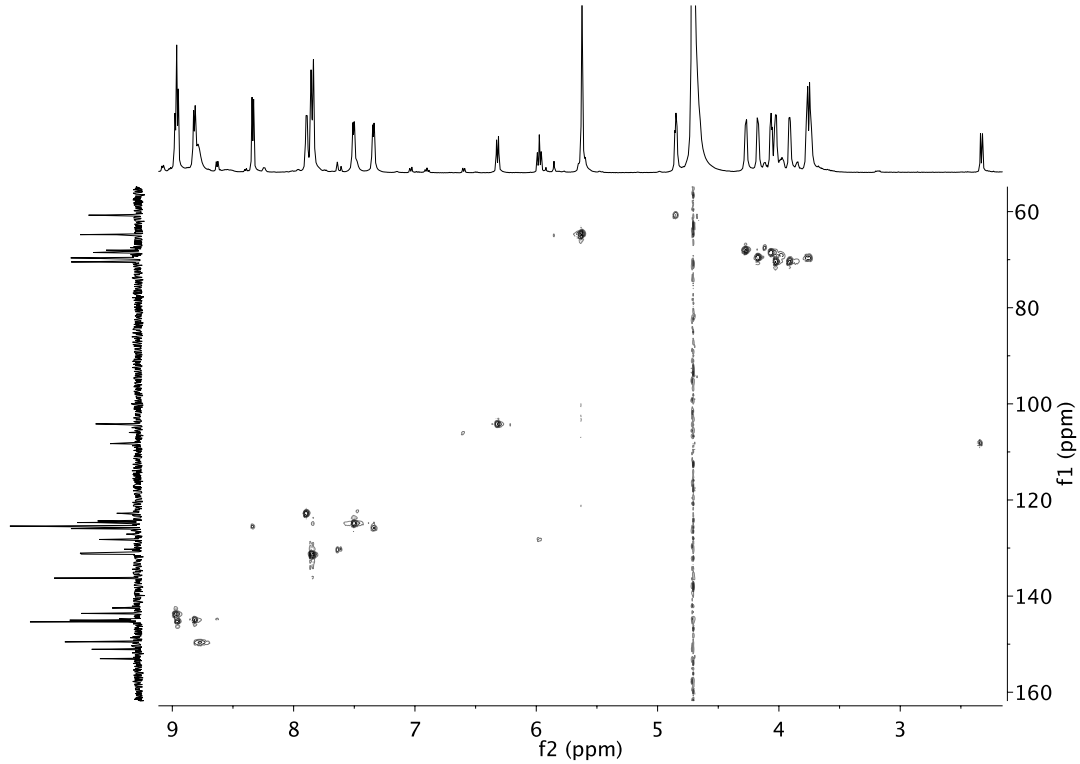


Fig. S6. HSQC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of pseudorotaxane **2**·6Cl in the presence of NaCl.

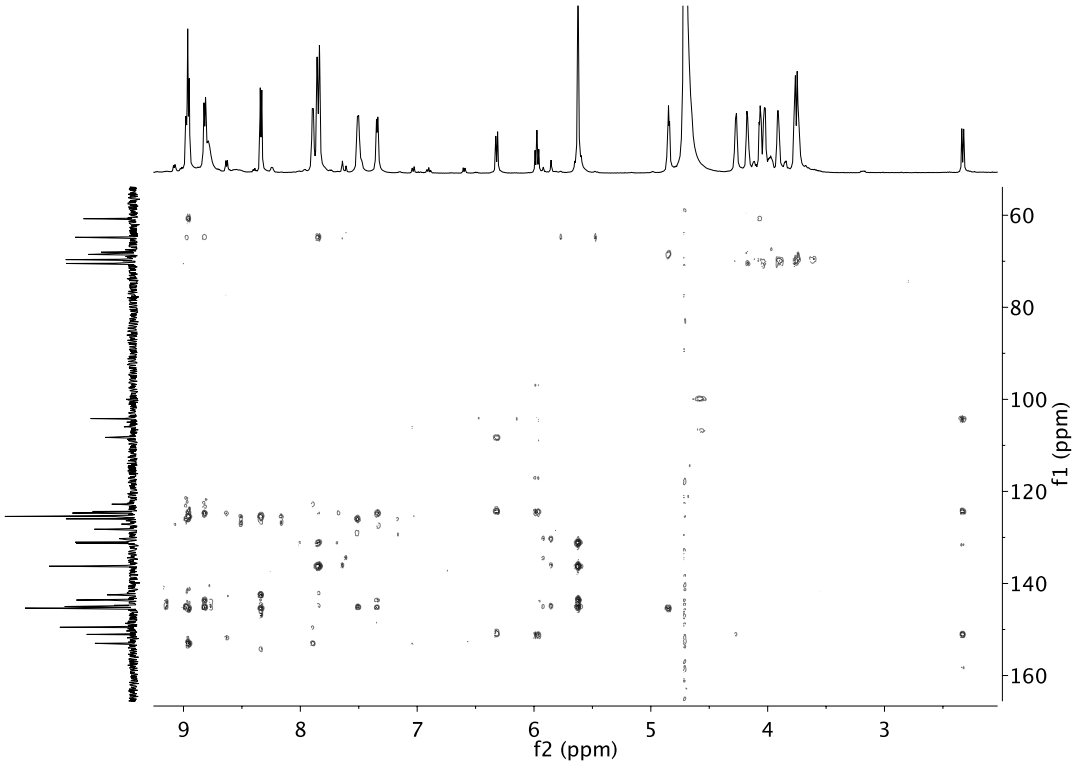


Fig. S7. HMBC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of pseudorotaxane **2**·6Cl in the presence of NaCl.

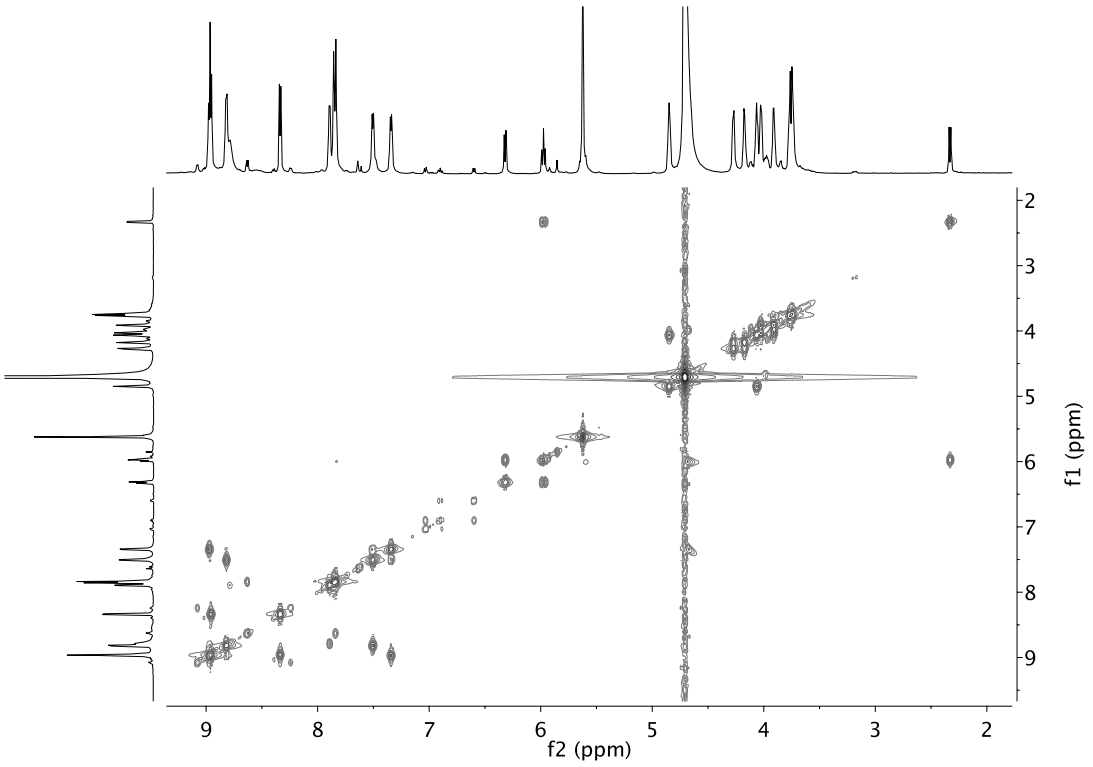
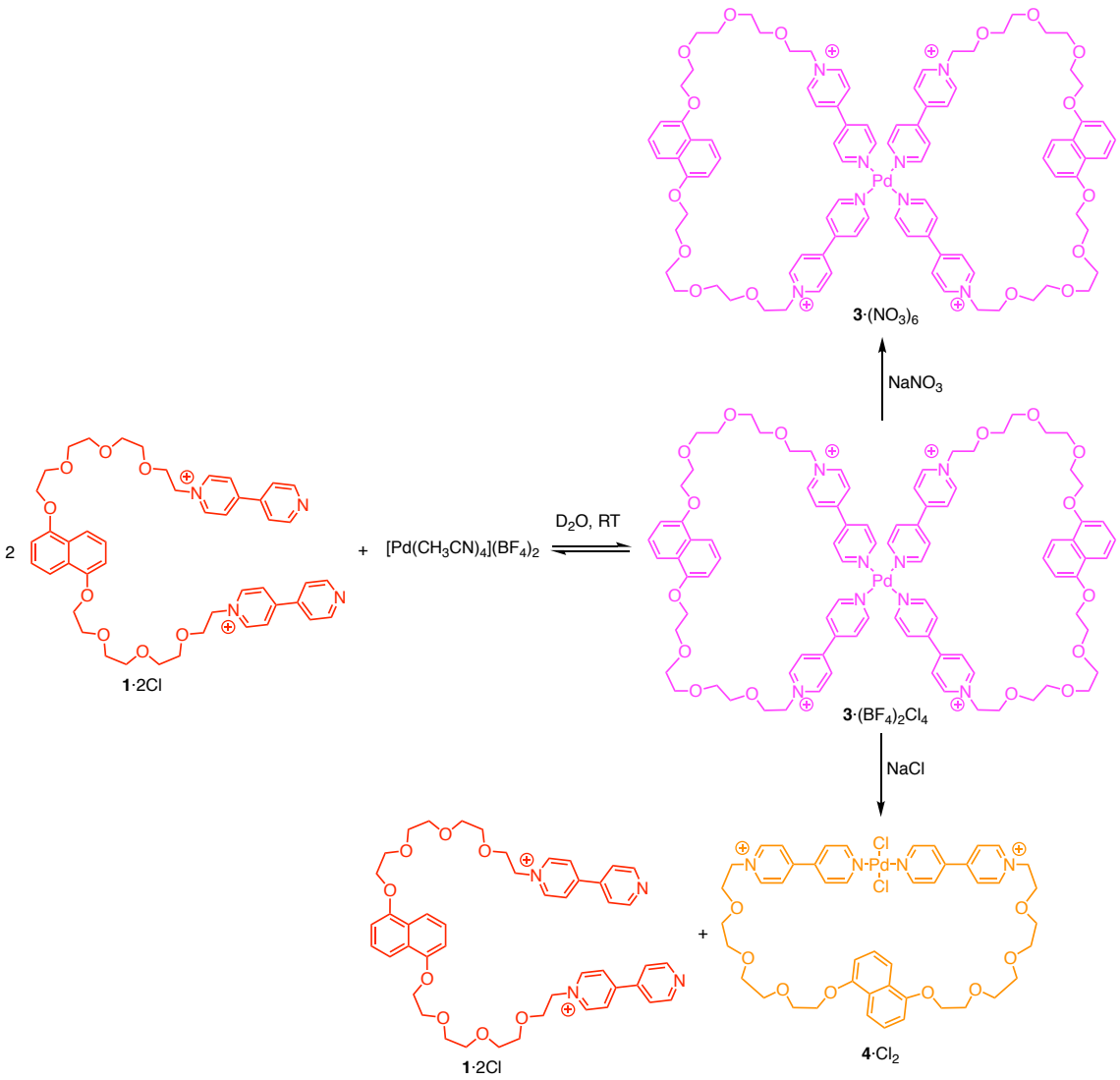


Fig. S8. COSY (500 MHz, D_2O, 298 K) of pseudorotaxane **2**·6Cl in the presence of NaCl.

Salt effect in the self-assembly of the metallacyclophanes $\mathbf{3}^{6+}$ *and* $\mathbf{4}^{2+}$



Scheme S2. Effect of salt addition in the self-assembly of the metallacyclophanes $\mathbf{3}^{6+}$ and $\mathbf{4}^{2+}$.

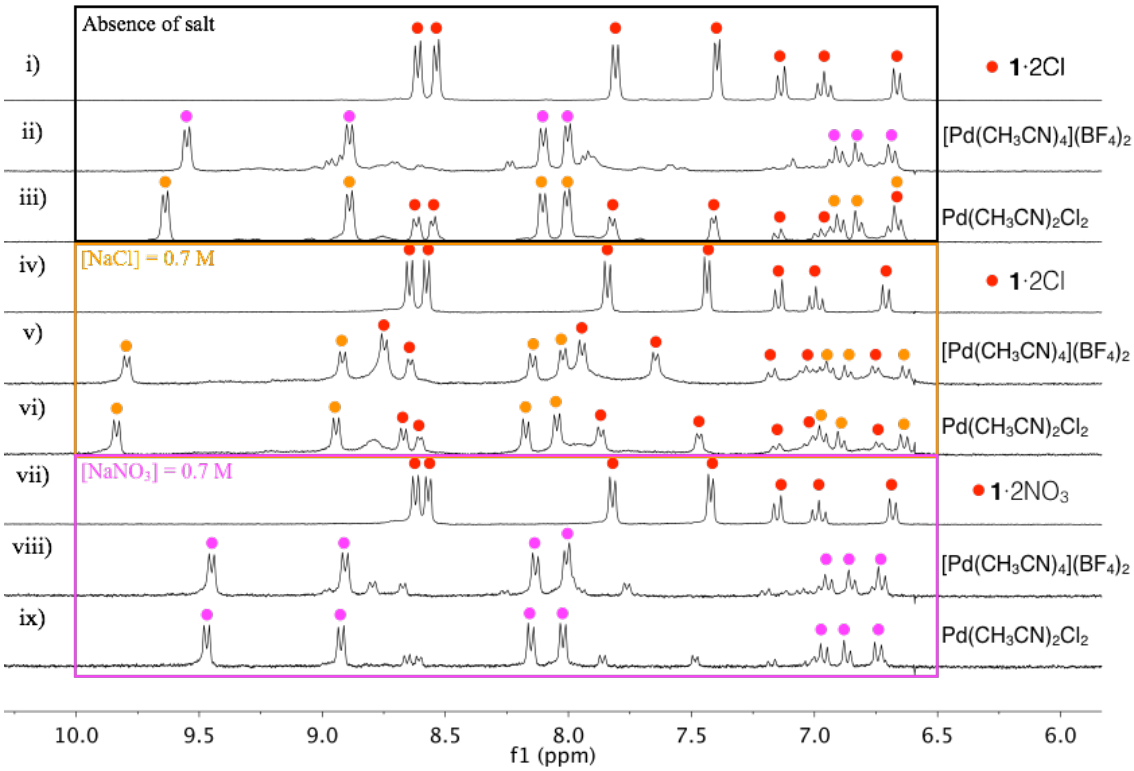
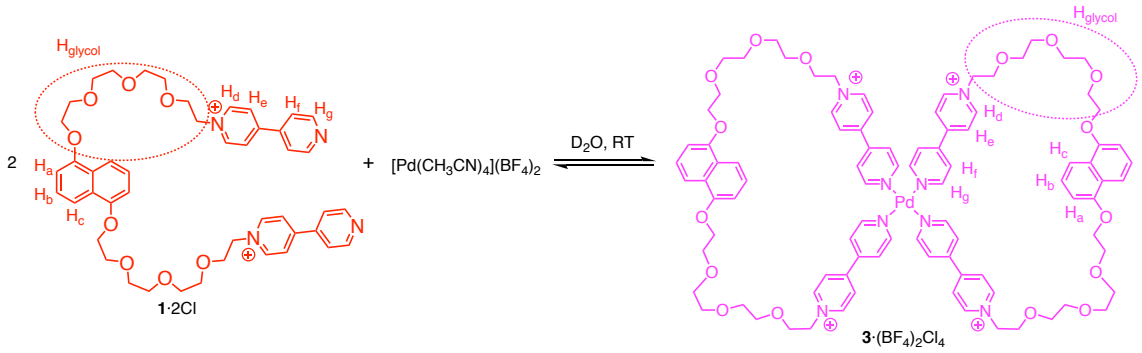


Fig. S9. Partial ^{1}H NMR spectra (300 MHz, D_2O, 298 K) of a solution containing **1**·2Cl (1 equiv., 5 mM) and the corresponding palladium complex ($[Pd(CH_3CN)_4](BF_4)_2$ or $Pd(CH_3CN)_2Cl_2$) (0.5 equiv., 2.5 mM) in the absence and in the presence of salt (NaCl or $NaNO_3$). Color code: ligand $\mathbf{1}^{2+}$, metallacyclophane $\mathbf{3}^{6+}$, metallacyclophane $\mathbf{4}^{2+}$.

Preparation of metallacyclophane ***3***$\cdot(BF_4)_2Cl_4$



Scheme S3. Self-assembly of metallacyclophane **3**$\cdot(BF_4)_2Cl_4$.

A solution containing **1**·2Cl (12.1 mg, 0.014 mmol) and $[Pd(CH_3CN)_4](BF_4)_2$ (3.1 mg, 7.2×10^{-3} mmol) in D_2O (2.8 mL) was stirred at room temperature resulting in the self-assembly of the metallacyclophane **3**$\cdot(BF_4)_2Cl_4$ as major product. *^{1}H NMR (500 MHz, D_2O, 298 K) δ*: 9.53 (8H, d, *J* = 6.7 Hz), 8.83 (8H, d, *J* = 6.8 Hz), 8.05 (8H, d, *J* = 6.9 Hz), 7.94 (8H, d, *J* = 6.8 Hz), 6.86 (4H, t, *J* = 8.0 Hz), 6.77 (4H, d, *J* = 7.8 Hz), 6.60 (4H, d, *J* = 8.3 Hz), 4.79 (8H, hidden), 4.26 (8H, m), 3.97-3.95 (16H, m), 3.71-3.61 (24H, m), 3.56-3.54 (8H, m). *^{13}C NMR (125 MHz, D_2O, 298 K) δ:* 153.0 (C), 152.2 (CH), 149.7 (C), 145.4 (CH), 145.0 (C), 125.8 (CH), 125.4 (C), 125.3 (CH), 125.2 (CH), 113.0 (CH), 106.0 (CH), 70.4 (CH_2), 70.1 (CH_2), 69.6 (CH_2), 69.5 (CH_2), 69.2 (CH_2), 68.4 (CH_2), 67.5 (CH_2), 61.1 (CH_2). HRMS-ESI *(m/z):* calculated for [**3**–2Cl–2BF_4]$^{4+}$ 439.1568, found 439.1680.

NMR characterization of metallacyclophane ***3***$\cdot(BF_4)_2Cl_4$

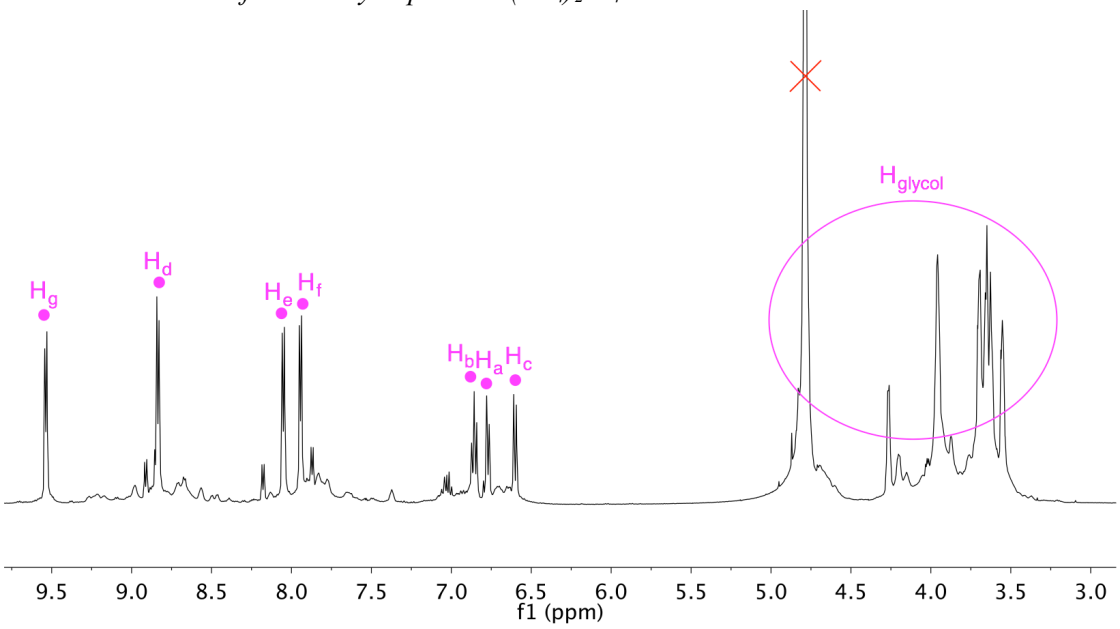


Fig. S10. Annotated ^{1}H NMR spectrum (500 MHz, D_2O, 298 K) of metallacyclophane **3**$\cdot(BF_4)_2Cl_4$. Red cross denotes residual solvent peak.

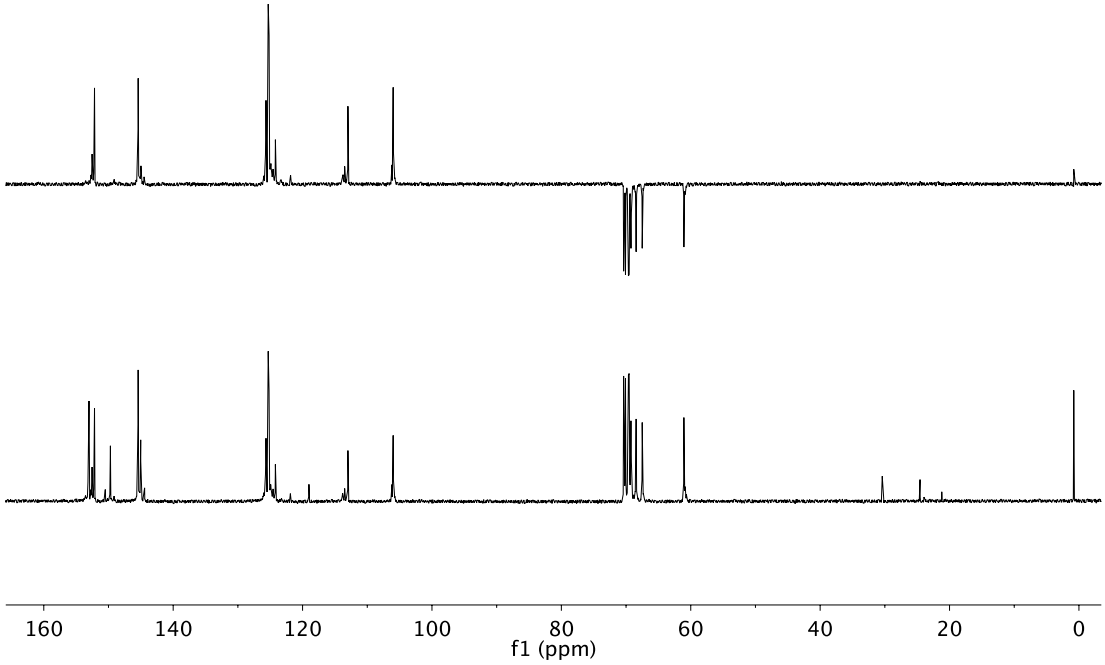


Fig. S11. ^{13}C NMR and DEPT-135 spectra (125 MHz, D_2O, 298 K) of metallacyclophane **3**·$(BF_4)_2Cl_4$.

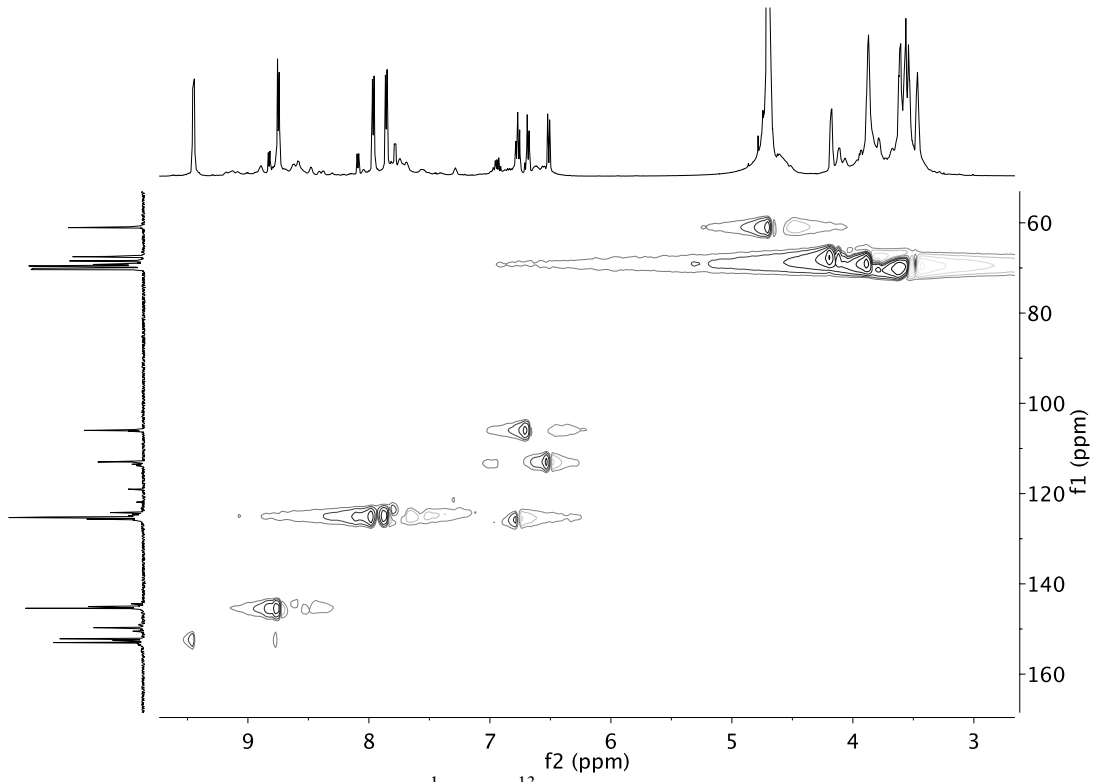


Fig. S12. HSQC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of metallacyclophane **3**·$(BF_4)_2Cl_4$.

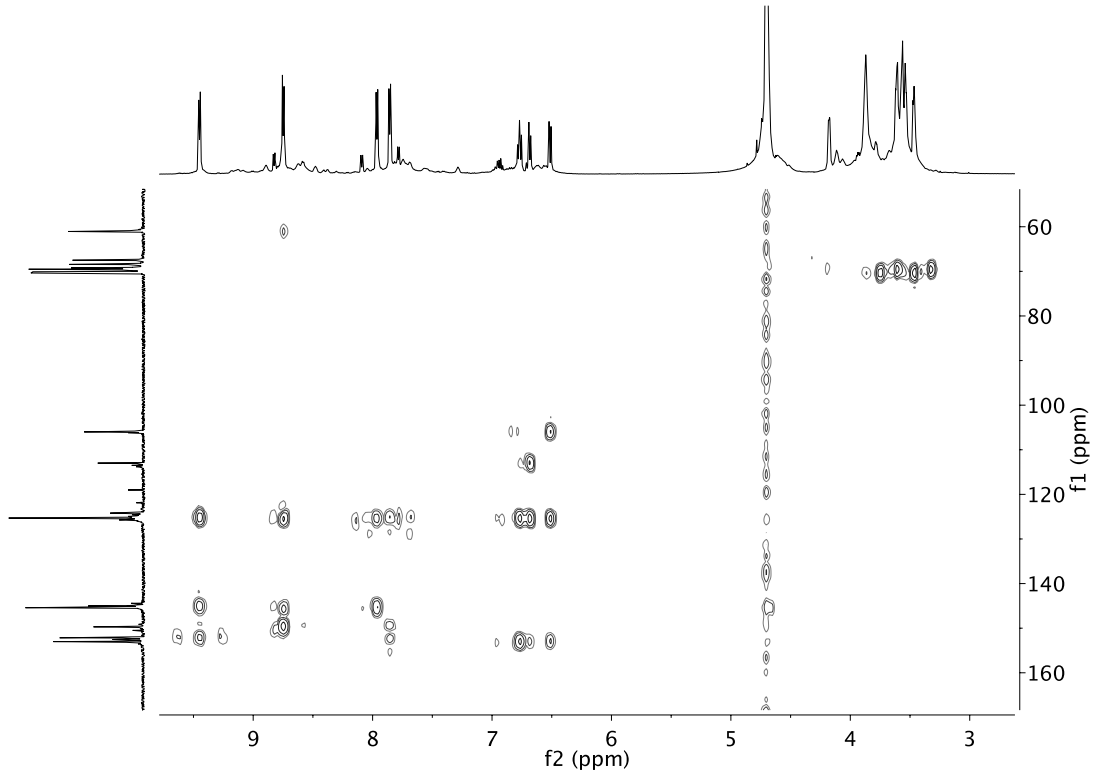


Fig. S13. HMBC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of metallacyclophane **3**·$(BF_4)_2Cl_4$.

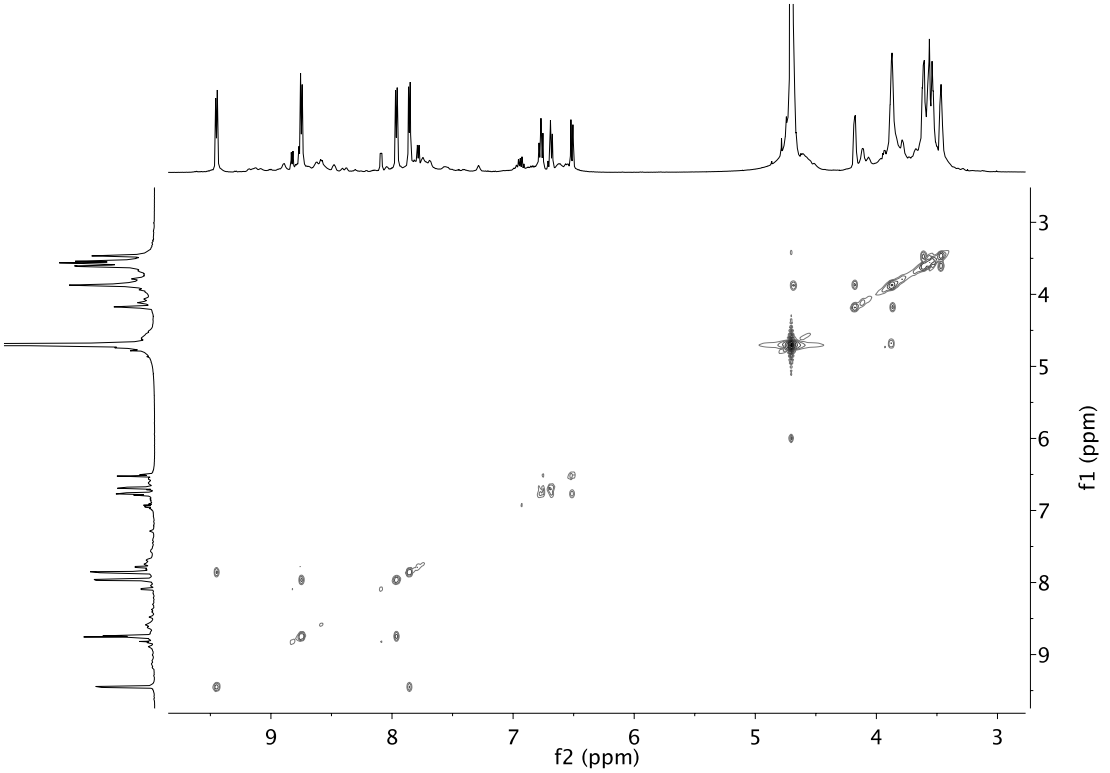


Fig. S14. COSY (500 MHz, D_2O, 298 K) of metallacyclophane **3**·$(BF_4)_2Cl_4$.

Mass Spectrometry characterization of metallacyclophane ***3***$\cdot(BF_4)_2Cl_4$

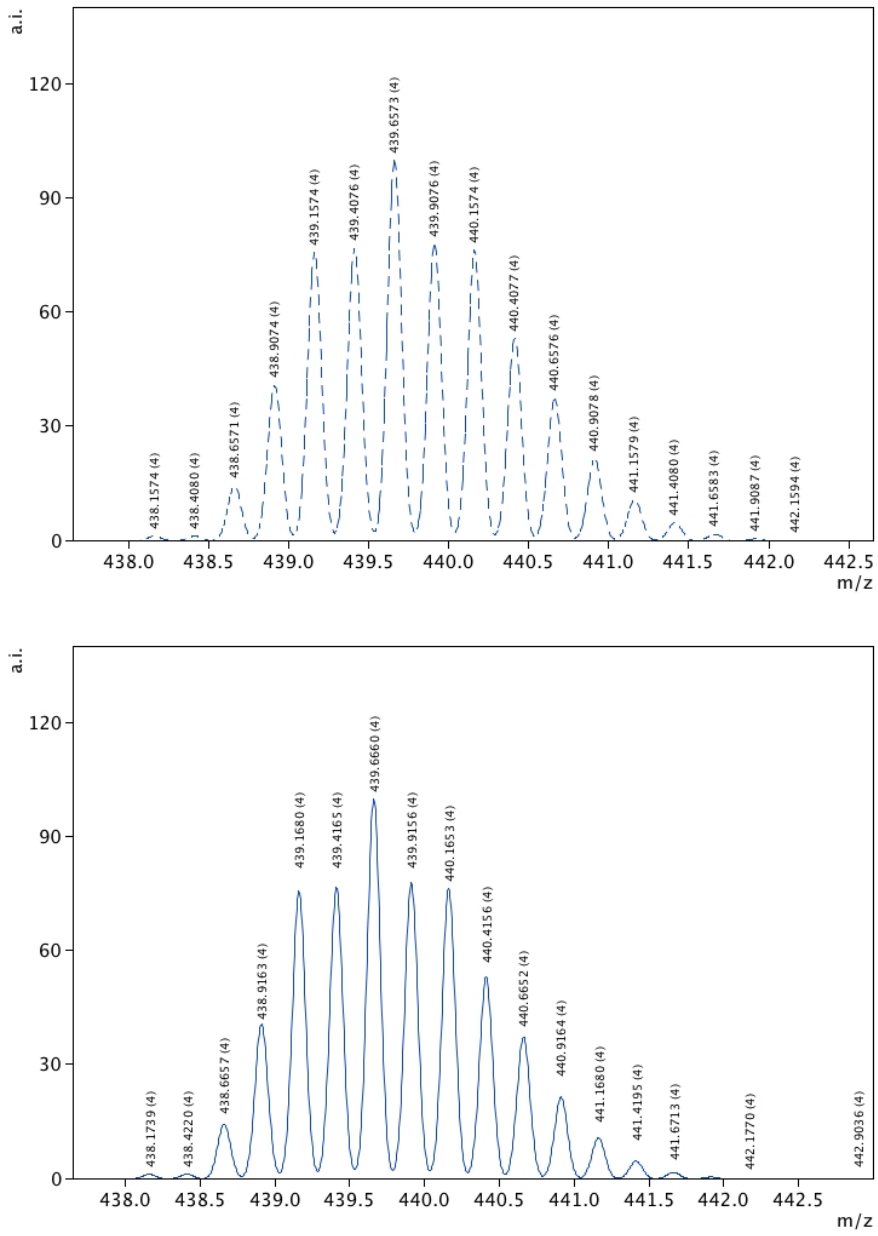
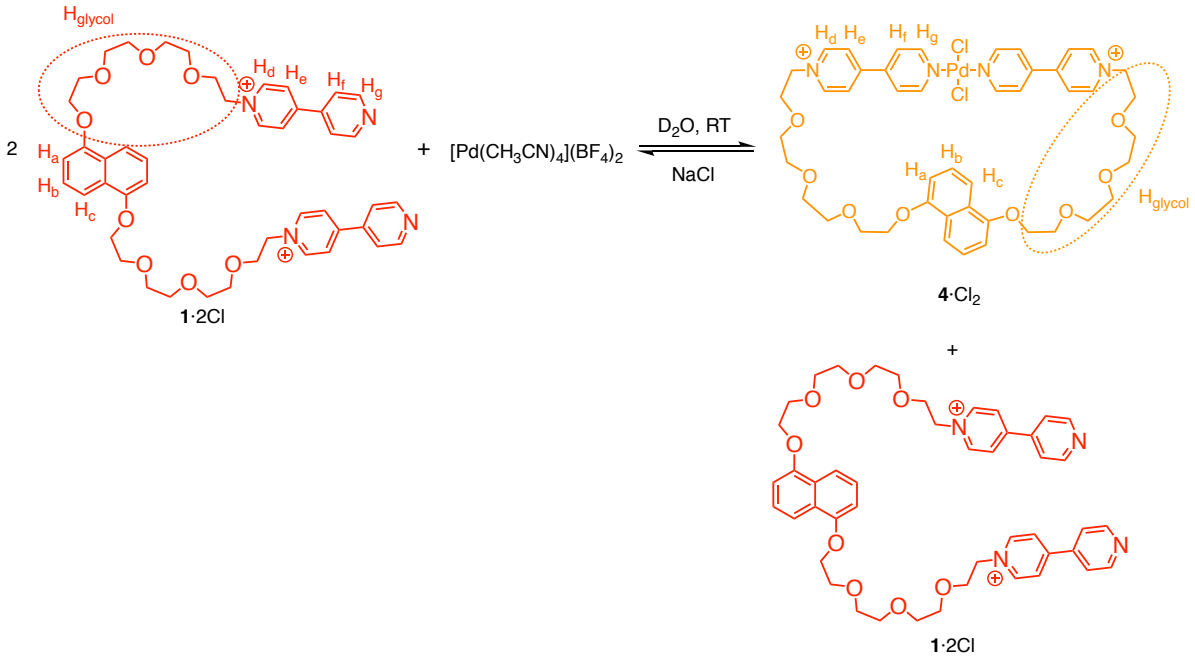


Fig. S15. HRMS-ESI theoretical isotopic distribution (above) and experimental isotopic distribution (below) for the fragment [**3**-2Cl-2BF_4]$^{4+}$.

Preparation of metallacyclophane ***4***·Cl_2



Scheme S4. Self-assembly of metallacyclophane **4**·Cl_2 using NaCl.

NaCl (29.3 mg, 0.5 mmol) was added over a solution containing **1**·2Cl (6.2 mg, 7.2×10^{-3} mmol) and $[Pd(CH_3CN)_4](BF_4)_2$ (1.6 mg, 3.6×10^{-3} mmol) in D_2O (1.44 mL). The mixture was stirred at room temperature resulting in the self-assembly of the metallacyclophane **4**·Cl_2 along with free axle **1**·2Cl. The next signals belong to metallacyclophane **4**·Cl_2. *^{1}H NMR (500 MHz, D_2O, 298 K) δ*: 9.75 (4H, d, *J* = 5.9 Hz), 8.87 (4H, d, *J* = 6.5 Hz), 8.09 (4H, d, *J* = 6.6 Hz), 7.96 (4H, d, *J* = 6.2 Hz), 6.89 (2H, t, *J* = 8.1 Hz), 6.81 (2H, d, *J* = 7.8 Hz), 6.54 (2H, d, *J* = 8.4 Hz), 4.79 (4H, hidden), 4.30 (4H, m), 4.00-3.97 (8H, m), 3.82-3.58 (16H, m). *^{13}C NMR (125 MHz, D_2O, 298 K) δ:* 153.0 (C), 152.5 (CH), 149.8 (C), 145.4 (CH), 144.8 (C), 125.4 (CH), 125.3 (C), 125.1 (CH), 124.7 (CH), 113.0 (CH), 106.1 (CH), 70.5 (CH_2), 70.2 (CH_2), 69.7 (CH_2), 69.6 (CH_2), 69.4 (CH_2), 68.5 (CH_2), 67.6 (CH_2), 61.1 (CH_2). HRMS-ESI *(m/z):* calculated for [**4**–2Cl]$^{2+}$ 483.1171, found 483.1165; calculated for [**4**–4Cl]$^{2+}$ 448.1482, found 448.1503.

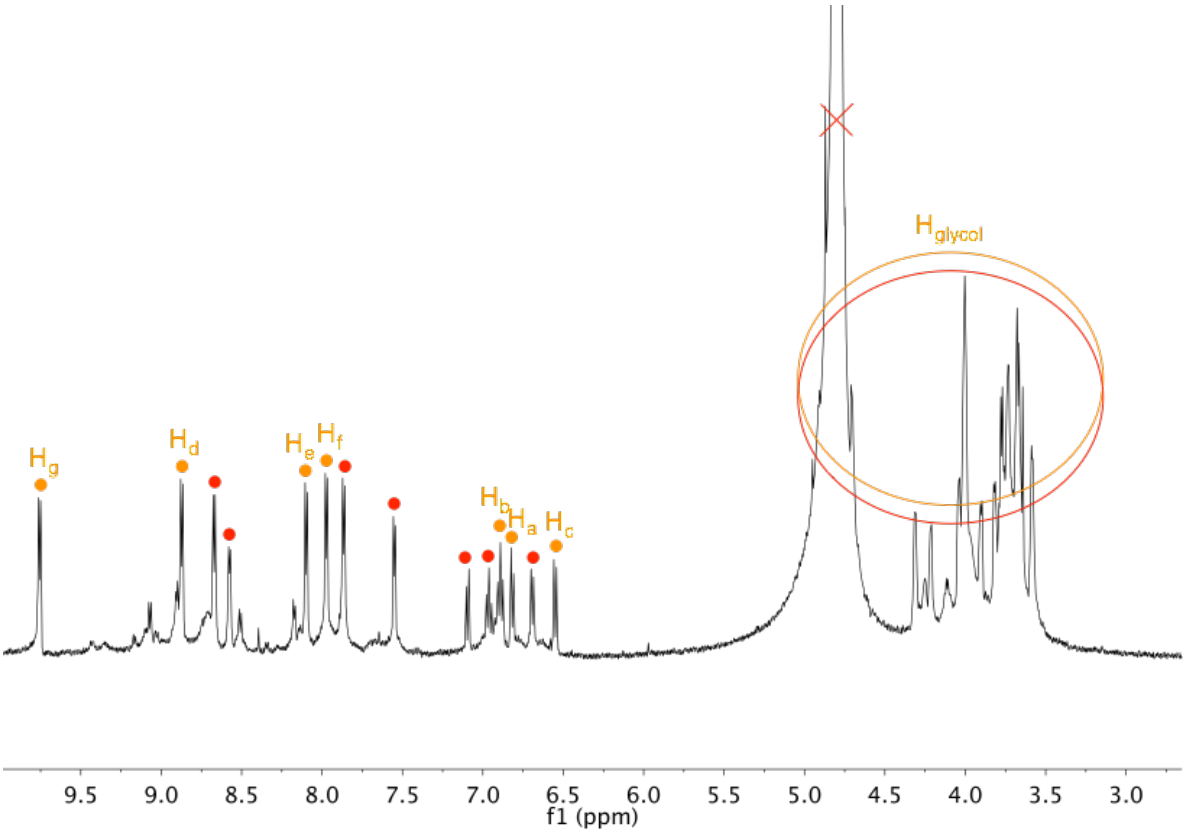


Fig. S16. Annotated ^{1}H NMR spectrum (500 MHz, D_2O, 298 K) of metallacyclophane **4**·Cl_2. Red cross denotes residual solvent peak.

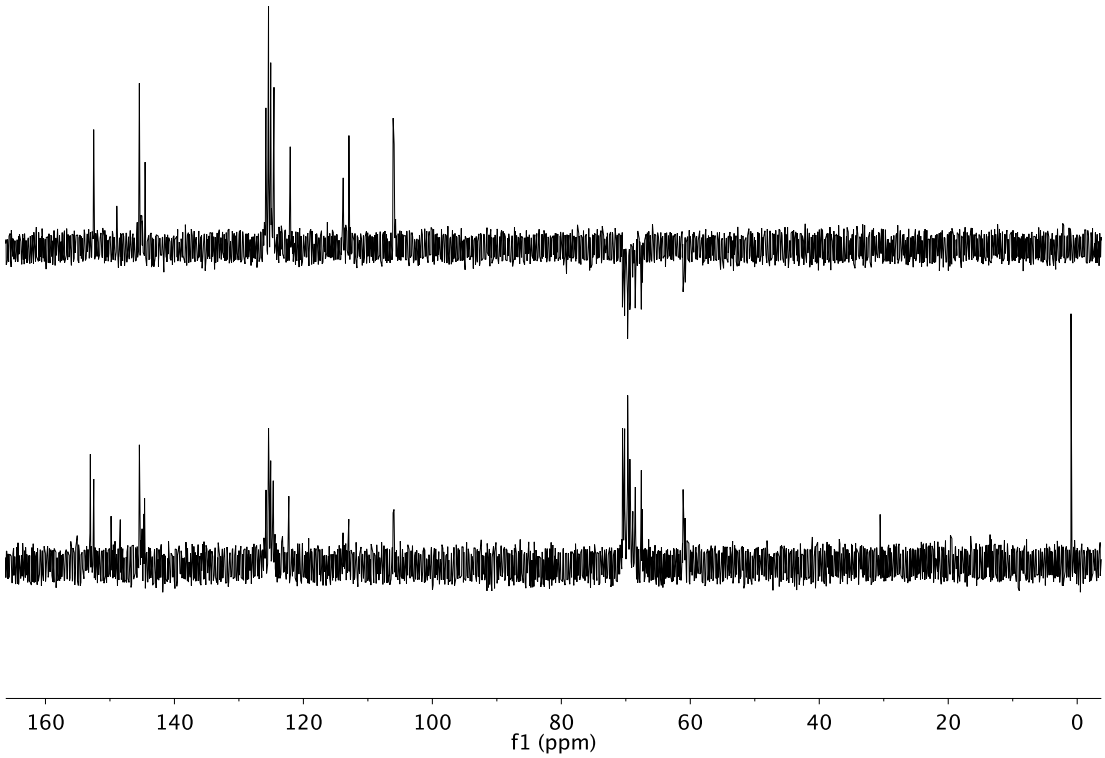


Fig. S17. ^{13}C NMR and DEPT-135 spectra (125 MHz, D_2O, 298 K) of metallacyclophane **4**·Cl_2.

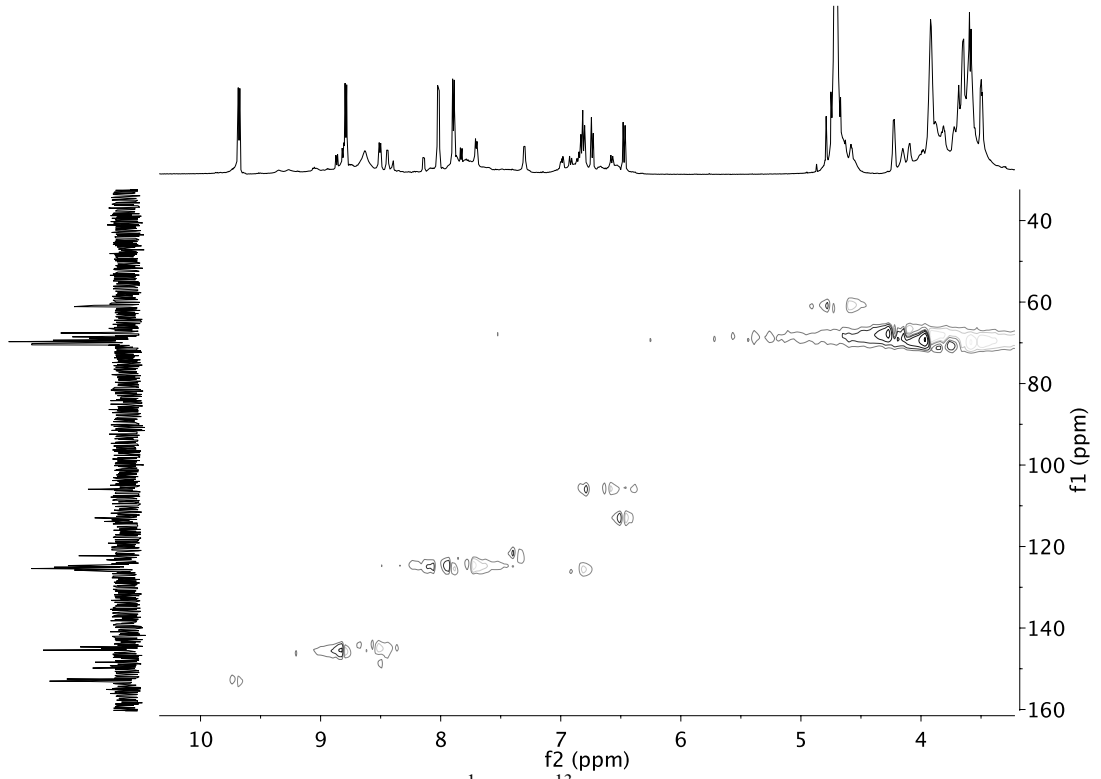


Fig. S18. HSQC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of metallacyclophane **4**·Cl_2.

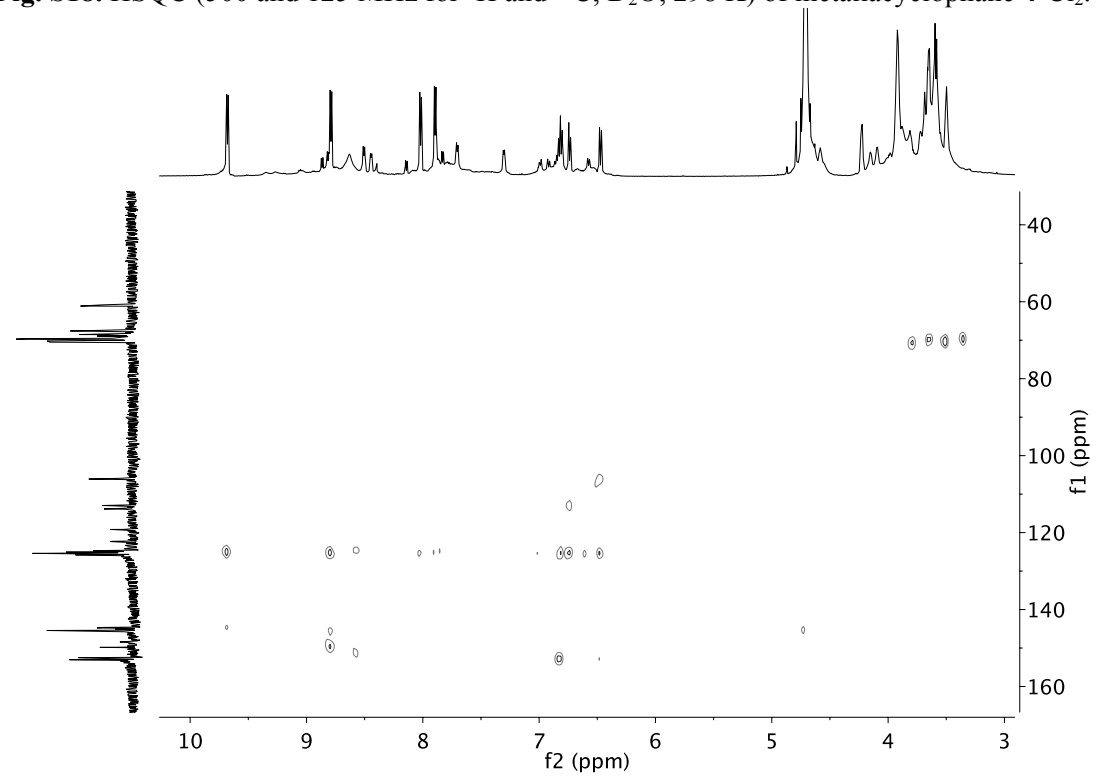


Fig. S19. HMBC (500 and 125 MHz for ^{1}H and ^{13}C, D_2O, 298 K) of metallacyclophane **4**·Cl_2.

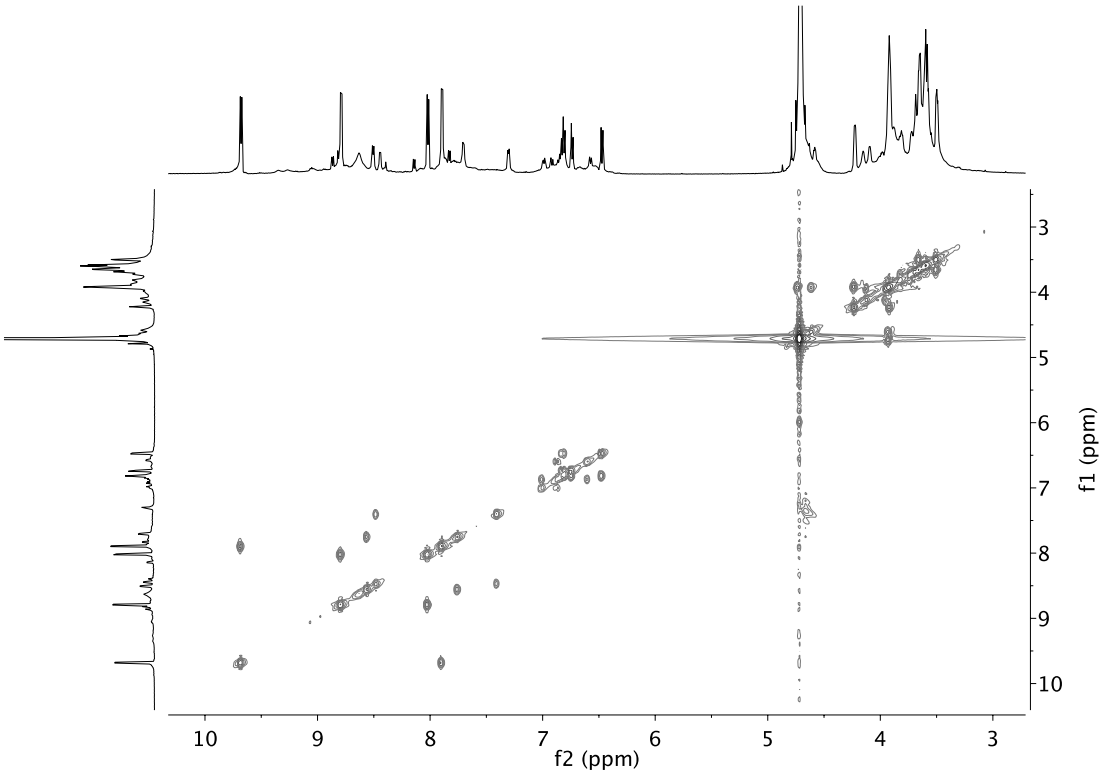


Fig. S20. COSY (500 MHz, D_2O, 298 K) of metallacyclophane **4**·Cl_2.

*Mass spectrometry characterization of metallacyclophane **4**·Cl_2*

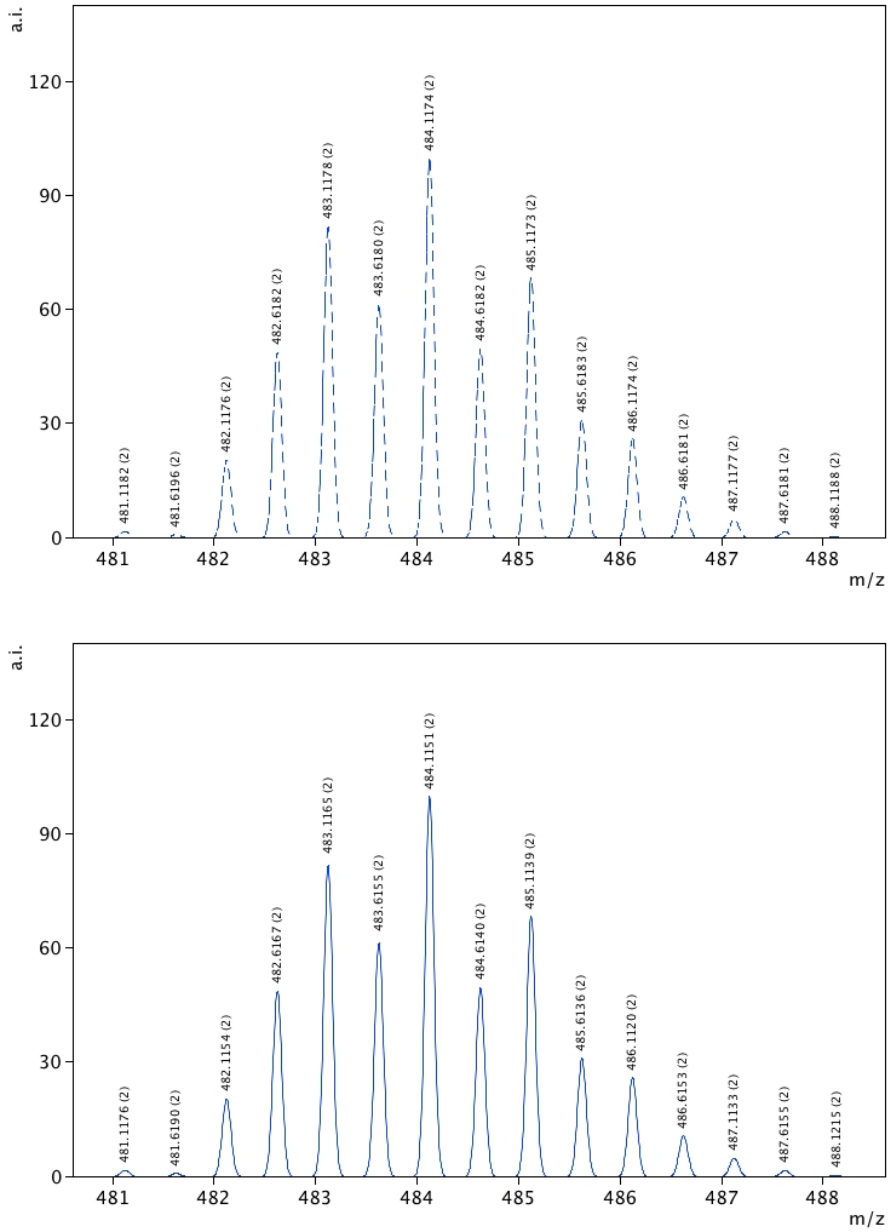


Fig. S21. HRMS-ESI theoretical isotopic distribution (above) and experimental isotopic distribution (below) for the fragment [**4**-2Cl]$^{2+}$.

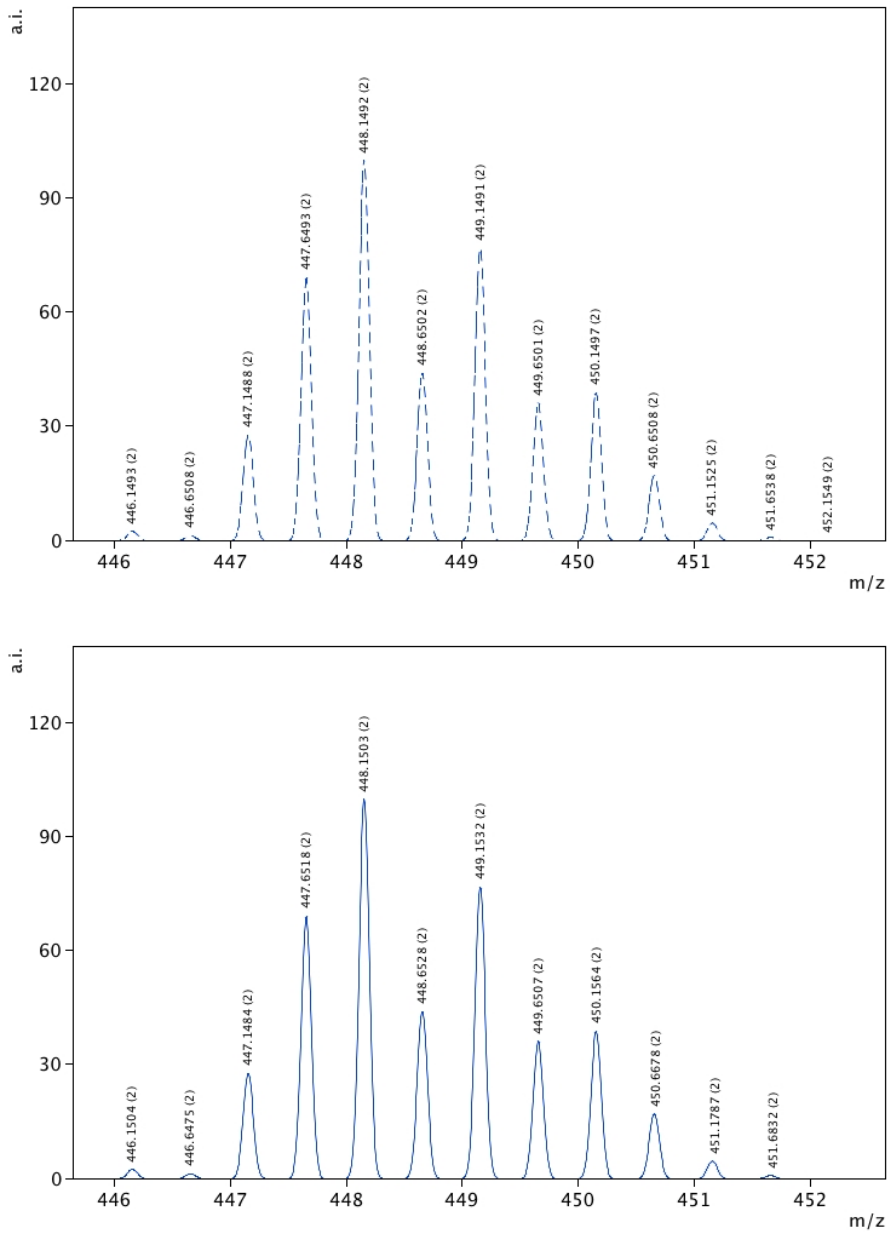


Fig. S22. HRMS-ESI theoretical isotopic distribution (above) and experimental isotopic distribution (below) for the fragment $[\mathbf{4}\text{-}4Cl+2e^-]^{2+}$.

Catenane self-assembly test using $NaNO_3$

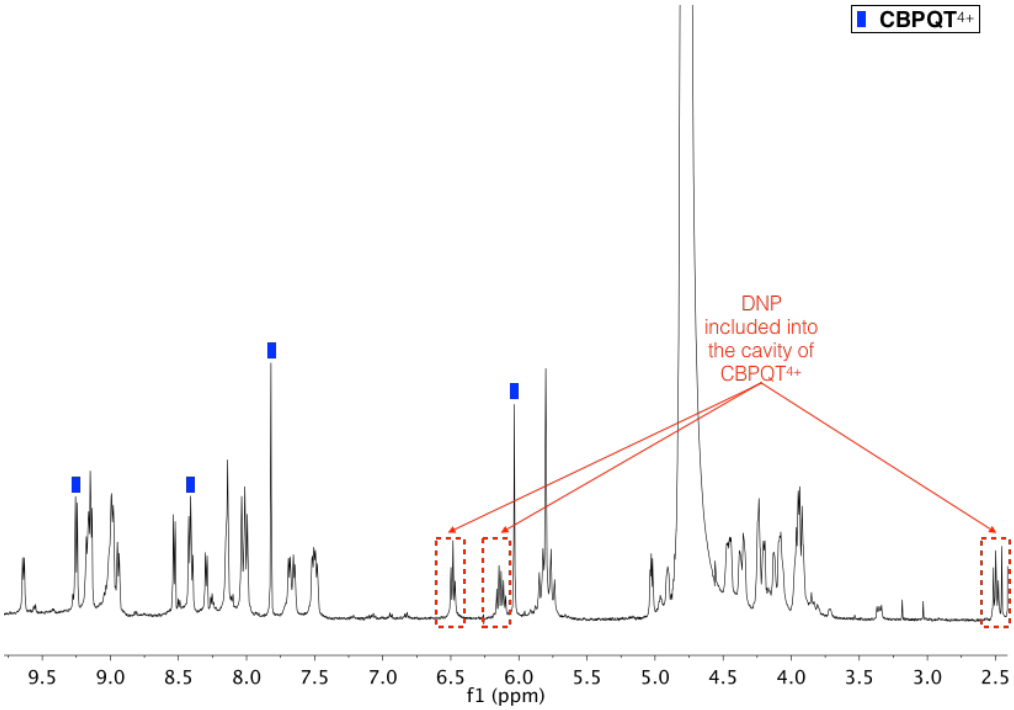


Fig. S23. ^{1}H NMR spectra (500 MHz, D_2O, 298 K) of an equimolar mixture of **1**·2Cl, **CBPQT**·4Cl and $[Pd(CH_3CN)_4](BF_4)_2$ (5 mM) in the presence of $NaNO_3$ (0.7 M).

Catenane self-assembly test using NaCl

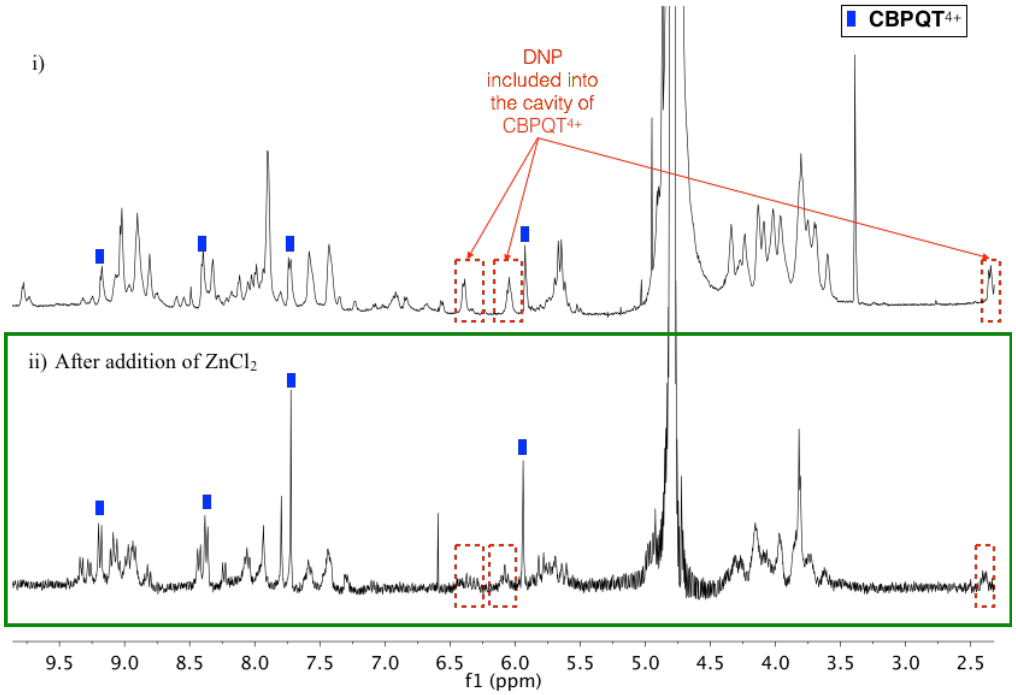
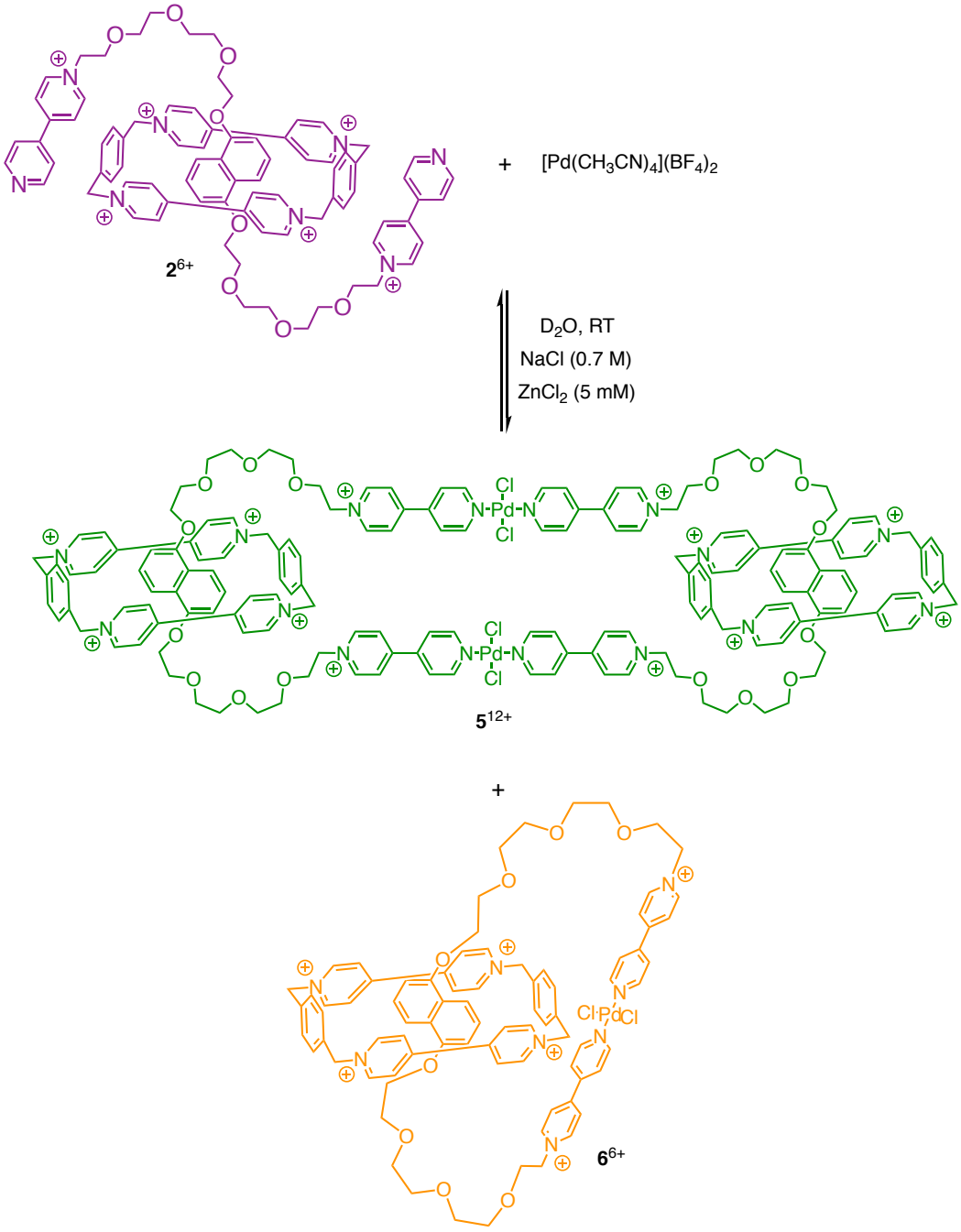


Fig. S24. ^{1}H NMR spectra (500 MHz, D_2O, 298 K) of: i) an equimolar mixture of **1**·2Cl, **CBPQT**·4Cl and $[Pd(CH_3CN)_4](BF_4)_2$ (5 mM) in the presence of NaCl (0.7 M); ii) after addition of $ZnCl_2$ (10 equiv.).

Preparation of [3]catenane $\mathbf{5}^{12+}$ *and [2]catenane* $\mathbf{6}^{6+}$



Scheme S5. Self-assembly of the [3]catenane $\mathbf{5}^{12+}$ and the [2]catenane $\mathbf{6}^{6+}$.

Over a solution containing **1**·2Cl (3.9 mg, 4.7×10^{-3} mmol), **CBPQT**·4Cl (3 mg, 4.7×10^{-3} mmol) and NaCl (38.3 mg, 0.65 mmol) in D_2O (0.9 mL), was added $[Pd(CH_3CN)_4](BF_4)_2$ (2.1 mg, 4.7×10^{-3} mmol) and $ZnCl_2$ (6.4 mg, 4.7×10^{-2} mmol). The mixture was stirred at room temperature producing the self-assembly of two species, [3]catenane $\mathbf{5}^{12+}$ and [2]catenane $\mathbf{6}^{6+}$.

The following signals correspond to [3]catenane $\mathbf{5}^{12+}$. *^{1}H NMR (500 MHz, D_2O, 298 K) δ*: 9.22 (8H, d, *J* = 6.5 Hz), 9.01 (8H, d, *J* = 6.6 Hz), 8.94 (8H, d, *J* = 7.1 Hz), 8.80 (8H, d, *J* = 6.6 Hz), 8.32 (8H, d, *J* = 6.5 Hz), 8.01 (8H, d, *J* = 6.9 Hz), 7.83 (8H, s), 7.80 (8H, s), 7.42 (8H, d, *J* = 5.3 Hz), 7.29 (8H, d, *J* = 6.2 Hz), 6.30 (4H, d, *J* = 8.1 Hz), 5.94 (4H, t, *J* = 8 Hz), 5.64 (8H, d, *J* = 13.8 Hz), 5.58 (8H, d, *J* = 13.6 Hz), 4.89 (8H, t_{broad}), 4.27-3.77 (56H, multiplets), 2.21 (4H, d, *J* = 8.1 Hz).

The following signals correspond to [2]catenane $\mathbf{6}^{6+}$. *^{1}H NMR (500 MHz, D_2O, 298 K) δ*: 9.27 (4H, d, 6.3 Hz), 9.05 (4H, d, *J* = 6.5 Hz), 8.94 (4H, d), 8.87 (4H, d, *J* = 6.5 Hz), 8.37 (4H, d, *J* = 6.5 Hz), 8.18 (4H, d, *J* = 6.4 Hz), 8.01 (4H, d_{broad}), 7.67 (4H, d_{broad}), 7.52 (4H, d, *J* = 5.9 Hz), 7.37 (4H, d, *J* = 6.6 Hz), 6.30 (2H, d, *J* = 8.1 Hz), 6.04 (2H, t, *J* = 8 Hz), 5.78 (4H, d, *J* = 13.4 Hz), 5.70 (4H, d, *J* = 13.4 Hz), 4.93 (4H, t_{broad}), 4.25 (4H, m), 4.10 (8H, m), 3.92 (4H, m), 3.81 (4H, m), 3.77 (8H, m), 2.36 (2H, d, *J* = 8.2 Hz).

*^{1}H NMR comparative of **CBPQT**$^{4+}$, pseudorotaxane **2**$^{6+}$ and catenanes **5**$^{12+}$ and **6**$^{6+}$*

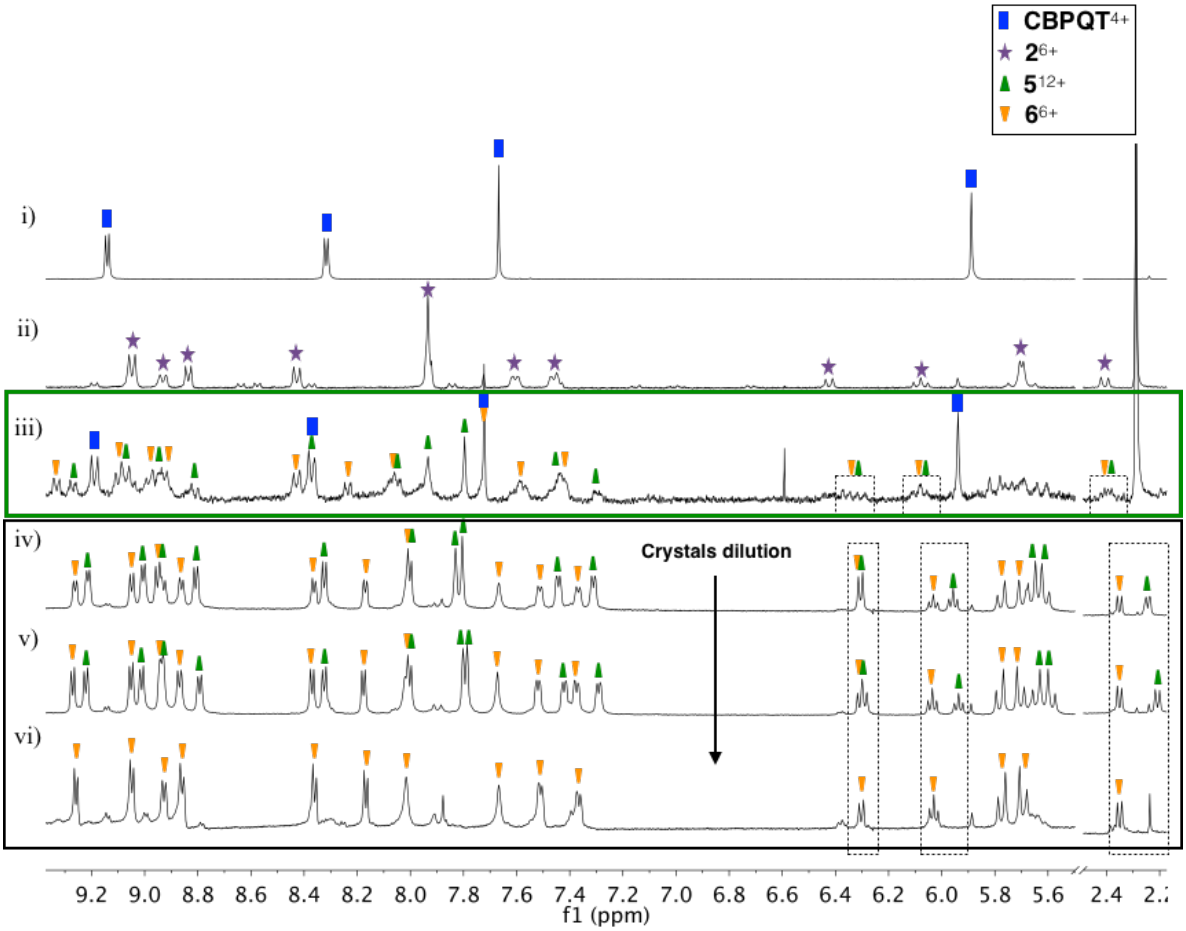


Fig. S25. Partial ^{1}H NMR spectra (500 MHz, D_2O, 298 K) of: i) **CBPQT**·4Cl; ii) an equimolar mixture of **1**·2Cl and **CBPQT**·4Cl (5 mM) in the presence of NaCl (0.7 M); iii) the previous mixture after addition of $[Pd(CH_3CN)_4](BF_4)_2$ (1 equiv.) and $ZnCl_2$ (10 equiv.), same spectrum as S24 ii). iv), v) and vi) are the partial ^{1}H NMR spectra (500 MHz, D_2O, 298 K) resulting from the dilution experiments of the crystals belonging to the [3]catenane **5**·$(ZnCl_4)_2Cl_8$.

Section C. Crystal Structures

*Crystal structure of pseudorotaxane [**2**$(ZnCl_3)_2$][$(ZnCl_4)_2$]*

Addition of $ZnCl_2$ (10 equiv.) to an equimolar solution of **CBPQT**$^{4+}$ and **1**$^{2+}$ (5 mM) in 0.7 M aq NaCl produced precipitation of a purple solid. The liquid fraction was separated from the precipitate, collected and stored for several days at room temperature producing purple plate-like crystals corresponding to [**2**$(ZnCl_3)_2$][$(ZnCl_4)_2$].

Suitable crystals for [**2**$(ZnCl_3)_2$][$(ZnCl_4)_2$] were selected and mounted in inert oil and transferred to the cold gas stream of a Kappa Apex 2 diffractometer. The crystals were kept at 100.00 K during data collection. Using Olex2[3], the structures were solved with the ShelXT[4] structure solution program using Direct Methods and refined with the ShelXL[5] refinement package using Least Squares minimization.

Table S1. Summary of crystallographic X-ray experimental data and refinement for [**2**$(ZnCl_3)_2$][$(ZnCl_4)_2$].

	[**2**$(ZnCl_3)_2$][$(ZnCl_4)_2$]
Empirical formula	$C_{82}H_{92}Cl_{14}N_8O_{14}Zn_4$
M_r	2171.41
Temperature [K]	100.0
Crystal size [mm^3]	0.387 × 0.176 × 0.046
Crystal system	Triclinic
Space group	P-1
a [Å]	14.5280(10)
b [Å]	16.6911(12)
c [Å]	20.7097(14)
α [º]	105.917(3)
β [º]	91.108(3)
γ [º]	105.758(3)
V [$Å^3$]	4623.9(6)
Z	2
ρ_{calcd} [Mg m^{-3}]	1.560
μ [mm^{-1}]	5.432
F(000)	2220
θ range [º]	4.46 to 133.388°
hkl ranges	-14 ≤ h ≤ 17, -19 ≤ k ≤ 19, -24 ≤ l ≤ 24
Reflections collected	55819
Independent reflections	16113
R_{int}	0.0372
Final *R* indices [$I>2\sigma(I)$]	R_1 = 0.0685, wR_2 = 0.1951
R indices (all data)	R_1 = 0.0737, wR_2 = 0.2022
Goodness-of-fit on F^2	1.051

Crystal structure of [3]catenane $\mathbf{5}\cdot(ZnCl_4)_2Cl_8$

Slow evaporation of an equimolar mixture of axle $\mathbf{1}^{2+}$, $\mathbf{CBPQT}^{4+}$ and $[Pd(CH_3CN)_4](BF_4)_2$ (5 mM) in D_2O (0.7 M NaCl) containing $ZnCl_2$ (10 equiv.) produced purple crystals corresponding to $\mathbf{5}\cdot(ZnCl_4)_2Cl_8$.

Suitable crystals for $\mathbf{5}\cdot(ZnCl_4)_2Cl_8$ were selected and mounted in inert oil and transferred to the cold gas stream of a Kappa Apex 2 diffractometer. The crystals were kept at 100.02 K during data collection. Using Olex2[3], the structures were solved with the ShelXT[4] structure solution program using Direct Methods and refined with the ShelXL[5] refinement package using Least Squares minimization.

Solvent Treatment Details: Total solvent accessible volume / cell = 836.3 $Å^3$ [17.0%] Total electron count / cell = 302.5 The solvent masking procedure as implemented in Olex2 was used to remove the electronic contribution of solvent molecules from the refinement. As the exact solvent content was not known, only the atoms used in the refinement model are reported in the formula here.

Table S2. Summary of crystallographic X-ray experimental data and refinement for $\mathbf{5}\cdot 8Cl\cdot 2ZnCl_4$.

	$\mathbf{5}\cdot(ZnCl_4)_2Cl_8$
Empirical formula	$C_{164}H_{192}Cl_{20}N_{16}O_{26}Pd_2Zn_2$
M_r	3855.86
Temperature [K]	100.02
Crystal size [mm^3]	0.311 × 0.033 × 0.01
Crystal system	Triclinic
Space group	P-1
a [Å]	12.2761(4)
b [Å]	19.5771(7)
c [Å]	20.4123(8)
α [º]	91.369(2)
β [º]	90.147(2)
γ [º]	97.5953(19)
V [$Å^3$]	4861.2(3)
Z	1
ρ_{calcd} [Mg m^{-3}]	1.317
μ [mm^{-1}]	0.765
F(000)	1988
θ range [º]	1.996 to 53.038°
hkl ranges	$-15 \le h \le 15$, $-24 \le k \le 24$, $-25 \le l \le 25$
Reflections collected	58662
Independent reflections	20008
R_{int}	0.0622
Final *R* indices [$I>2\sigma(I)$]	$R_1 = 0.0710$, $wR_2 = 0.1879$
R indices (all data)	$R_1 = 0.1108$, $wR_2 = 0.2055$
Goodness-of-fit on F^2	1.031

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