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Programmable self-assembly of water-soluble organo-heterometallic cages $[M_{12}M'_4L_{12}]$ using 3-(3,5-dimethyl-1H-pyrazol-4-yl)pentane-2,4-dione (H_2L)

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Supporting Information

Materials: All chemicals and solvents were of reagent grade and were purified according to conventional methods.

Instrumentation: ^{1}H NMR experiments of all compounds were recorded at 400 MHz on a Bruker Avance III HD 400 spectrometer using tetramethylsilane. ESI-MS and CSI-MS measurements were performed with an JEOL Accu-TOF-4G LC-plus mass spectrometer.

X-ray Structural Determinations:

X-Ray diffraction data of the crystals of complex **2**·12PF_6^- were carried out at 291K using synchrotron radiation (λ = 0.80010 Å) via the 3W1A in the IHEP with the approval of the Beijing Synchrotron Radiation Facility (BSRF). The diffraction data reduction and integration were performed by the HKL2000 software. Positions of the Pd and Fe atoms and most of the non-hydrogen atoms were found using the direct methods program in the Bruker SHELXTL software. All hydrogen atoms were placed in calculated positions in the final structure refinement. Data for **C1**·2PF_6^- and **1**·12PF_6^- were collected at 123 K on a Bruker Smart Apex CCD area detector equipped with a graphite monochromated MoKα radiation (λ = 0.71073 Å). The absorption correction for complex was performed using SADABS. The structure was solved by direct methods and refined by employing full-matrix least-squares on F^2 by using the SHELXTL software program and expanded using Fourier techniques.[1-2] All non-H atoms of the complexes were refined with anisotropic thermal parameters. The hydrogen atoms were included in idealized positions. In complexes **1**·12PF_6^- and **2**·12PF_6^-, the unit cell includes a large region of disordered solvent acetonitrile molecules, which could not be modelled as discrete atomic sites. We employed PLATON/SQUEEZE to calculate the diffraction contribution of the solvent acetonitrile molecules, thereby, to produce a set of solvent-free CH_3CN.

CCDC1524843(**C1**), 1524844(**1**) and 1524845(**2**) contains the supplementary crystallographic data for this paper. These data can be achieved via the Cambridge Crystallographic Data Centre deposit @ccdc.cam.Ac.uk,

http://www.ccdc.cam.ac.uk/deposit.

The final residuals along with unit cell, space group, data collection, and refinement parameters are presented in Table S1-S5.

Experimental Section:

Synthesis of ligand H_2L

Ligand 3-(3,5-dimethyl-1*H*-pyrazol-4-yl)petane-2,4-dione (**H_2L**) was synthesized according to a reported procedure with spectroscopic data consistent with the literature[3,4].

Self-assembly of dimetallic corners

Synthesis of $[(bpy)_2Pd_2(HL)_2](NO_3)_2$ (C1·$2NO_3^-$). H_2L (10.1mg, 0.052mmol) was added to a suspension of $bpyPd(NO_3)_2$ (20mg, 0.052mmol) in H_2O (3ml), and the mixture was stirred for 24 h at room temperature. Then, removing the mixture into 60℃ oil-bath for 8 h and the solution became clear. ^1H NMR confirmed the quantitative formation of **C1**·$2NO_3^-$. ^1H NMR (400 MHz, D_2O, $Si(CH_3)_4$ as external standard, 298 K, ppm): 8.55 (d, J = 8.0 Hz, 4H, py-H), 8.45 (s, 4H, py-H), 8.24 (d, J = 4.0 Hz, 4H, py-H), 7.82 (s, 4H, py-H), 2.37 (s, 12H, pz-H), 2.13 (s, 6H, diketone-H), 1.65 (s, 6H, diketone-H).

The mixture was filtered and a ten-fold excess of KPF_6 was added and vigorous stirring. The yellow precipitation were collected by centrifugation, washed with minimum amount of cold water and dried in vacuum to give **C1**·$2PF_6^-$ as yellow solid (23.12 mg, 0.025 mmol, 97.5% yield). ESI-MS (CH_3CN): *m/z* Calcd for [**C1**·PF_6^-]$^+$ 1056.59 found 1056.94; Calcd for [**C1**]$^{2+}$ 455.82 found 455.99. Elemental analyses calcd (%) for $C_{40}H_{42}F_{12}N_8O_4P_2Pd_2$: C, 39.95; H, 3.50; N, 9.32; Found: C, 39.88; H, 3.59; N, 9.28.

Synthesis of $[(dmbpy)_2Pd_2(HL)_2](NO_3)_2$ (C2·$2NO_3^-$). H_2L (9.4 mg, 0.05mmol) was added to a suspension of $dmbpyPd(NO_3)_2$ (20.7 mg, 0.05 mmol) in H_2O (3ml), and the mixture was stirred for 24 h at room temperature. Then, removing the mixture into 60 ^oC oil-bath for 8 h and the addition of 0.5 ml of acetone to the solution resulted in the suspention liquid became clear immediately. ^1H NMR confirmed the quantitative

formation of **C2**·2NO_3^-. ^{1}H NMR (400 MHz, D_2O, $Si(CH_3)_4$ as external standard, 300 K, ppm): 8.55 (d, *J* = 8.0 Hz, 4H, py-H), 8.45 (s, 4H, py-H), 8.24 (d, *J* = 4.0 Hz, 4H, py-H), 7.82 (s, 4H, py-H), 2.37 (s, 12H, pz-H), 2.13 (s, 6H, diketone-H), 1.65 (s, 6H, diketone-H).

The mixture was filtered and a ten-fold excess of KPF_6 was added and vigorous stirring. The yellow precipitation were collected by centrifugation, washed with minimum amount of cold water and dried in vacuum to give **C2**·2PF_6^- as yellow solid (23.73 mg, 0.0245 mmol, 98.1% yield). ESI-MS (CH_3CN): *m/z* Calcd for [**C2**·PF_6^-]$^+$ 1112.72 found 1112.99; Calcd for [**C2**]$^{2+}$ 483.88 found 484.02. Elemental analyses calcd (%) for $C_{44}H_{50}F_{12}N_8O_4P_2Pd_2$: C, 42.02; H, 4.01; N, 8.91; Found: C, 42.08; H, 4.16; N, 8.93.

Self-assembly of hetero-metallic cages

{[(bpy)Pd]$_{12}$Fe$_4$L$_{12}$}(PF$_6$)$_{12}$ (1·12PF_6^-**).** $NaHCO_3$ (8.4 mg, 0.104 mmol) was added to a suspension of **C1**·2NO_3^- (23.6 mg, 0.026 mmol) in H_2O (3 ml), and the mixture was stirred for 3 h at room temperature. Then, $FeCl_3$ (2.8 mg, 0.017 mmol) was added to the solution and the mixture was stirred for 8 h at 60 °C. The mixture was filtered and a ten-fold excess of KPF_6 was added and vigorous stirring. The red precipitation were collected by centrifugation, washed with minimum amount of cold water and dried in vacuum to give **1**·12PF_6^- as red solid (26.7 mg, 3.62 μmol, 85.2% yield). CSI-MS (CH_3CN): *m/z* Calcd for [**1**·9PF_6^-]$^{3+}$ 2328.58 found 2328.99; Calcd for [**1**·8PF_6^-]$^{4+}$ 1710.20 found 1709.76; Calcd for [**1**·7PF_6^-]$^{5+}$ 1339.17 found 1339.00; Calcd for [**1**·6PF_6^-]$^{6+}$ 1091.81 found 1091.68. Elemental analyses calcd (%) for $C_{240}H_{240}F_{72}Fe_4N_{48}O_{24}P_{12}Pd_{12}$: C, 38.81; H, 3.23; N, 9.06; Found: C, 38.58; H, 3.20; N, 8.46.

{[(dmbpy)Pd]$_{12}$Fe$_4$L$_{12}$}(PF$_6$)$_{12}$ (2·12PF_6^-**).** $NaHCO_3$ (8.4 mg, 0.10 mmol) was added to a suspension of **C2**·2NO_3^- (24.2 mg, 0.025 mmol) in H_2O (3 ml) and acetone (0.5 ml), and the mixture was stirred for 3 h at room temperature. Then, $FeCl_3$ (2.8 mg, 0.017 mmol) was added to the solution and the mixture was stirred for 8 h at 60 °C. The mixture was filtered and a ten-fold excess of KPF_6 was added and vigorous stirring. The red precipitation were collected by centrifugation, washed with minimum

amount of cold water and dried in vacuum to give **2**·12PF_6^- as red solid (27.6 mg, 3.56 μmol, 83.8% yield). CSI-MS (CH_3CN) : *m/z* Calcd for [**2**·9PF_6^-]$^{3+}$ 2440.79 found 2440.54; Calcd for [**2**·8PF_6^-]$^{4+}$ 1794.35 found 1794.12; Calcd for [**2**·7PF_6^-]$^{5+}$ 1406.49 found 1406.12; Calcd for [**2**·6PF_6^-]$^{6+}$ 1147.92 found 1147.94. Elemental analyses calcd (%) for $C_{264}H_{288}F_{72}Fe_4N_{48}O_{24}P_{12}Pd_{12}$: C, 40.84; H, 3.71; N, 8.66; Found: C, 40.75; H, 3.73; N, 8.21.

NMR and MS spectra

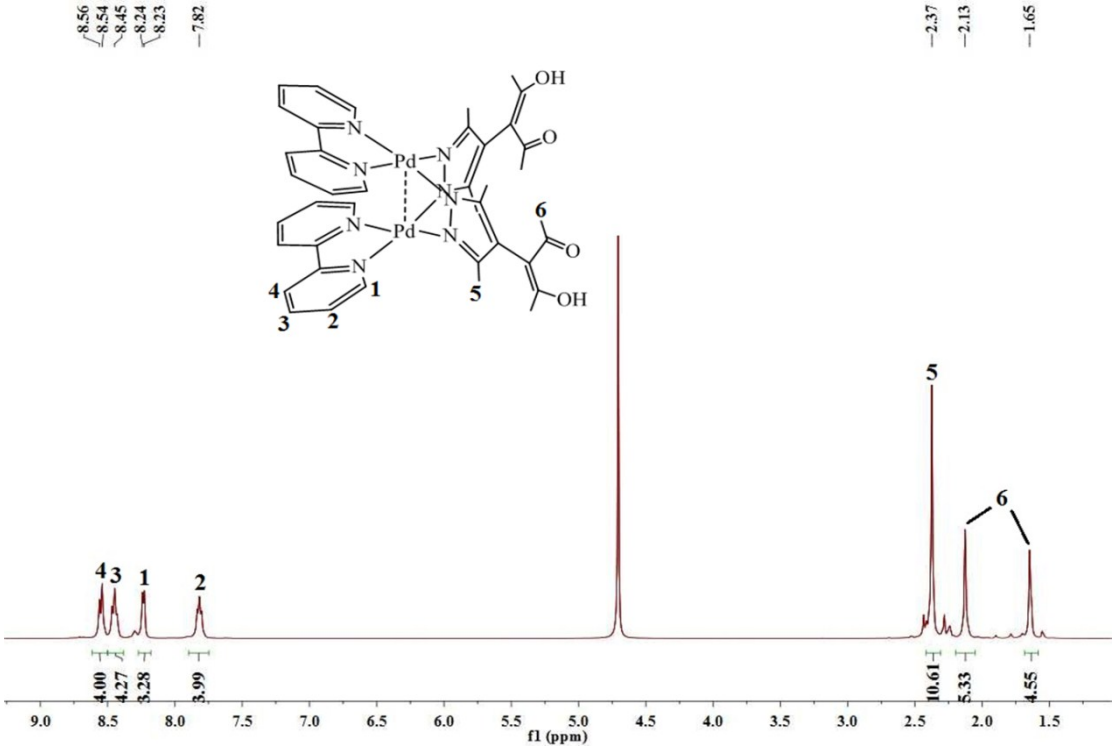

Fig. S1 ^{1}H NMR (400 MHz, 298 K) spectrum of **C1**·2NO_3^- in D_2O.

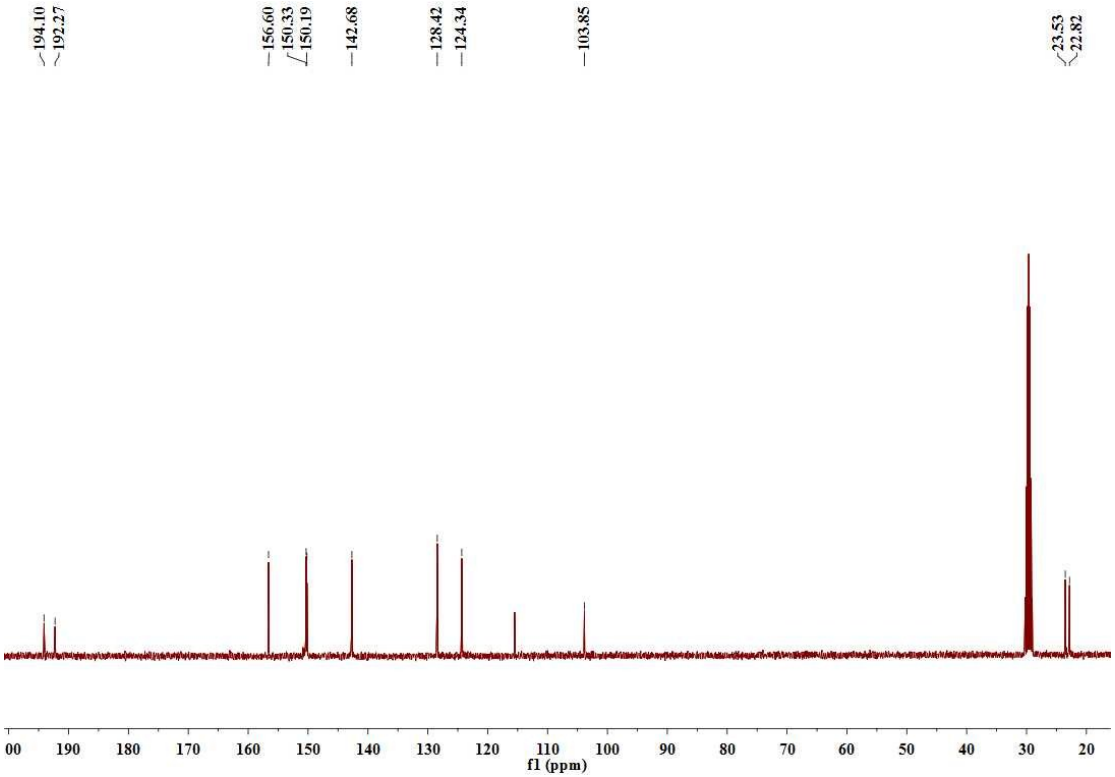

Fig. S2 ^{13}C NMR (100 MHz, 298 K) spectrum of **C1**·2NO_3^- in D_2O.

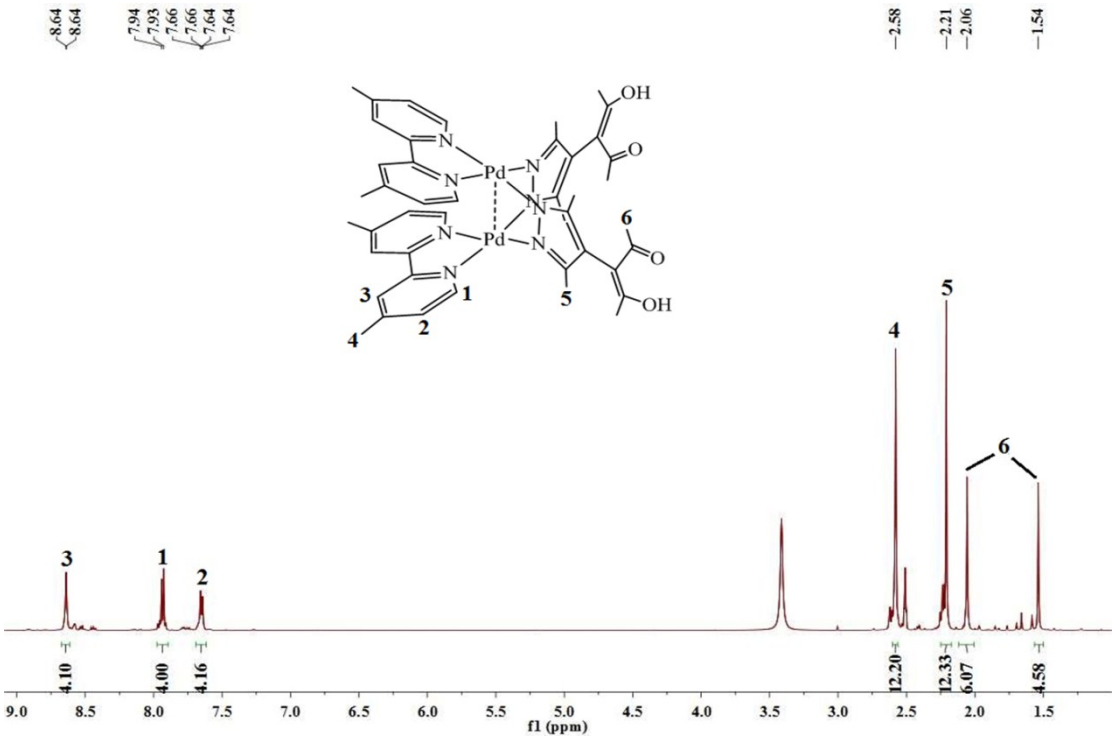

Fig. S3 ^{1}H NMR (400 MHz, 298 K) spectrum of **C2**·2NO_3^- in DMSO.

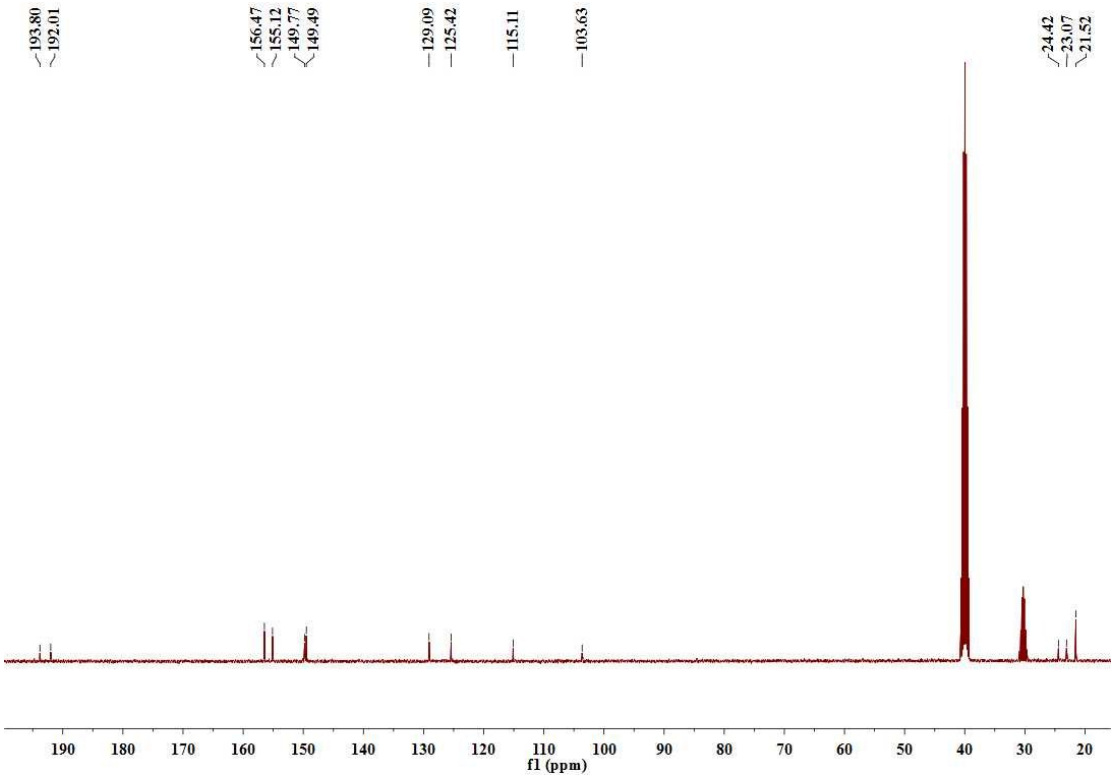

Fig. S4 ^{13}C NMR (100 MHz, 298K) spectrum of **C2**·2NO_3^- in DMSO

Analysis of the NMR spectra

All of ^{1}H NMR spectra for the ligand and the self-assembled complexes showed sets of high resolution signals. From the Figure S1 and S3 we could find that the dimetallic clips **C1**·2PF_6^- and **C2**·2PF_6^- were formed as the pure products and no any other products founded at all in D_2O and DMSO at room temperature. Compared with the pyrazole acetylacetonate ligand, the protons at acetylacetonate methyl of the dimetallic clips had obvious split into two groups. This is due to the ratation of the bond connecting acetylacetonate and pyrazole ligand before assembly, however, the bond would be subject to steric hindrance after assembly, so the chemical environment is different. This also proves the formation of the intermediates.

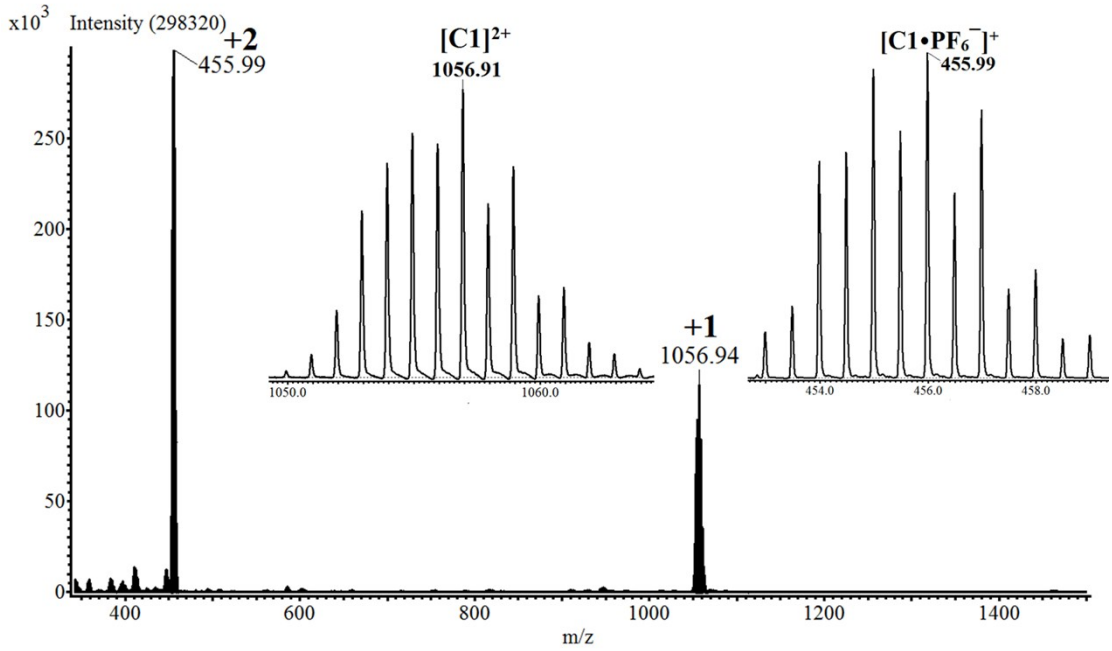

Fig. S5 ESI-MS spectrum of **C1**·2PF_6^- in acetonitrile. The inset shows the isotopic distribution of the species **[C1]**$^{2+}$ and **[C1**·PF_6^-**]**$^+$.

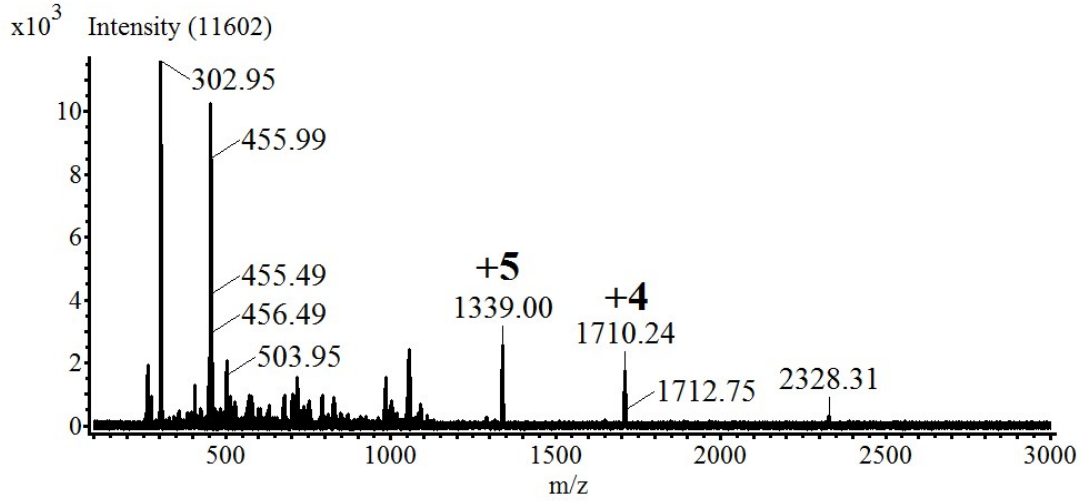

Fig. S6 ESI-MS spectrum of **1**·12PF_6^- in acetonitrile.

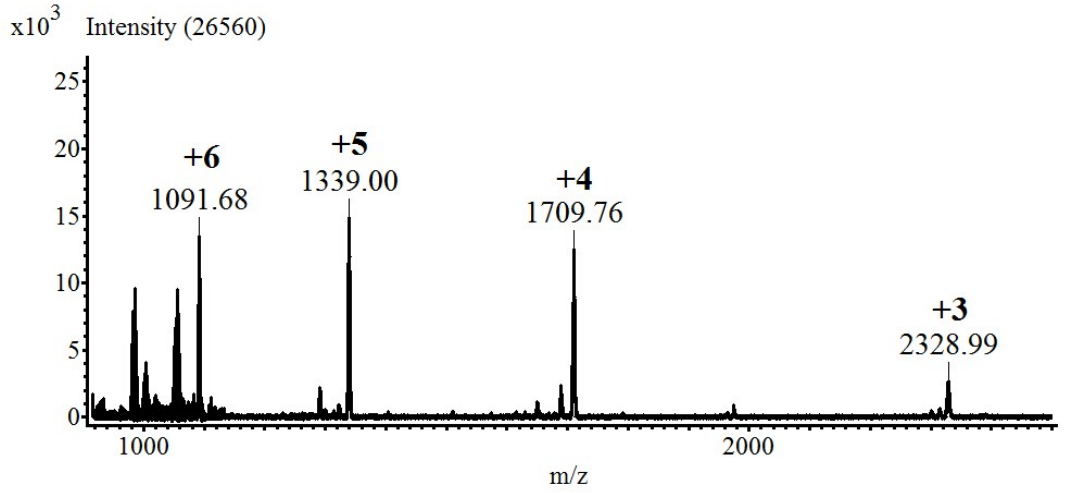

Fig. S7 CSI-MS spectrum of **1**·12PF_6^- in acetonitrile.

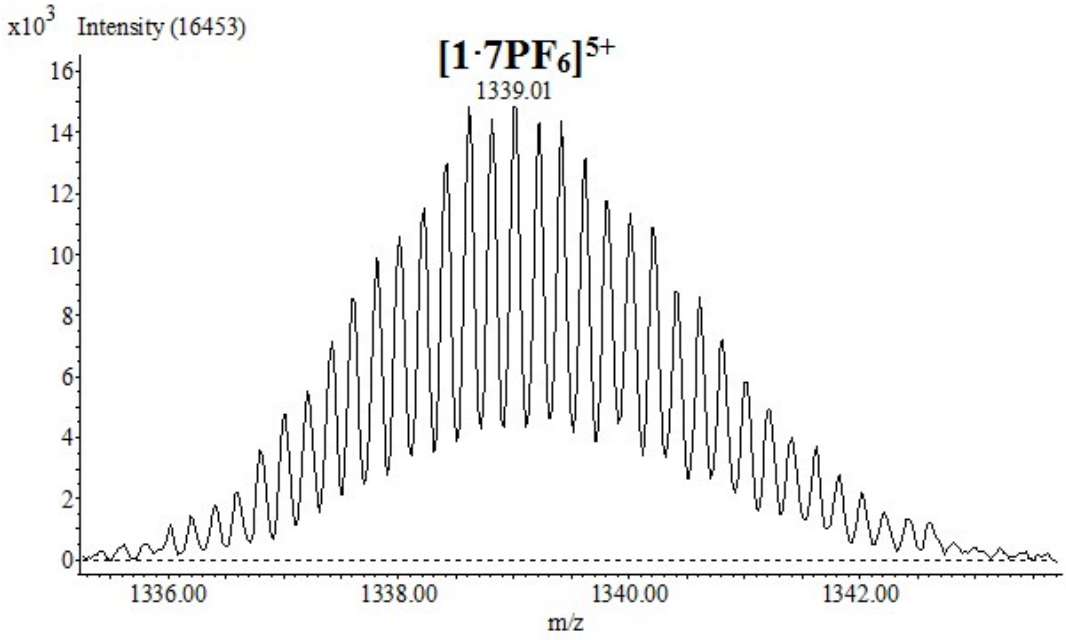

Fig. S8 The isotopic distribution of the species **[1**·7PF_6^-**]**$^{5+}$.

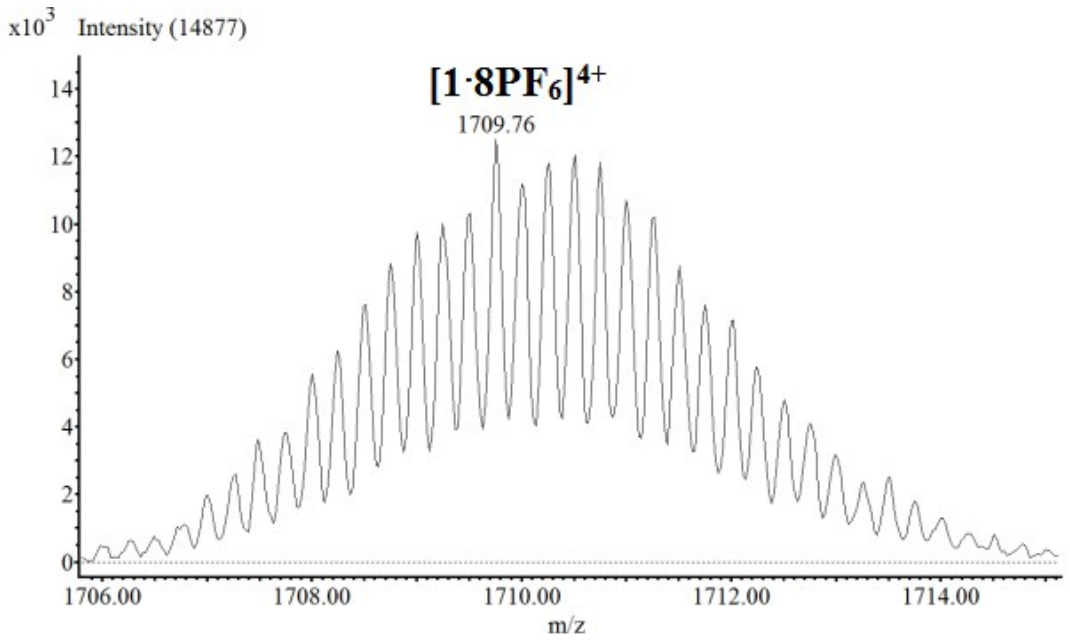

Fig. S9 The isotopic distribution of the species **[1**·8PF_6^-**]**$^{4+}$.

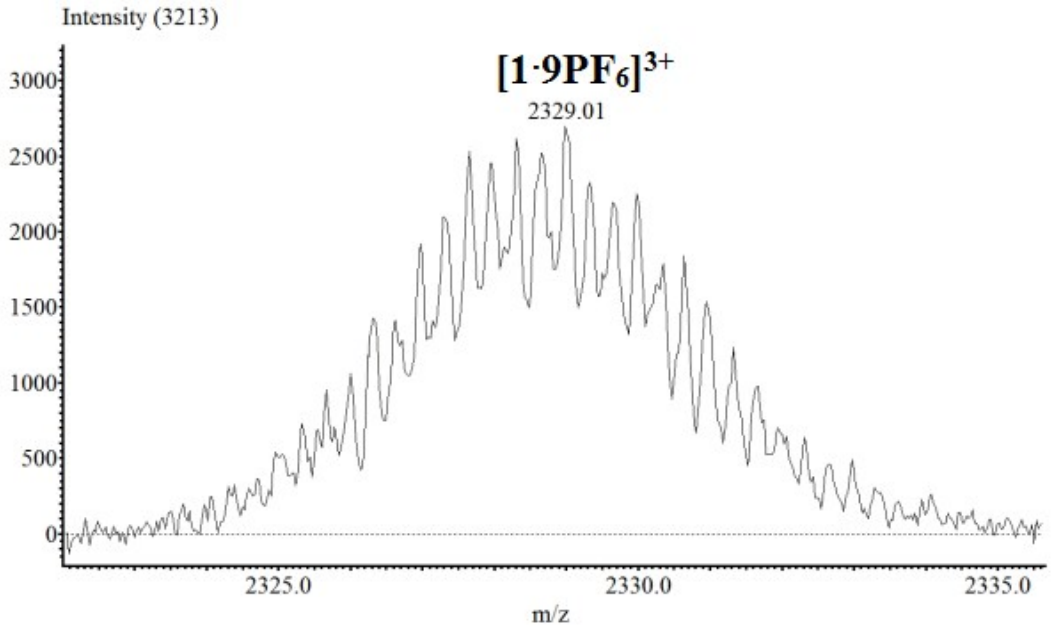

Fig. S10 The isotopic distribution of the species **[1**·9PF_6^-**]**$^{3+}$.

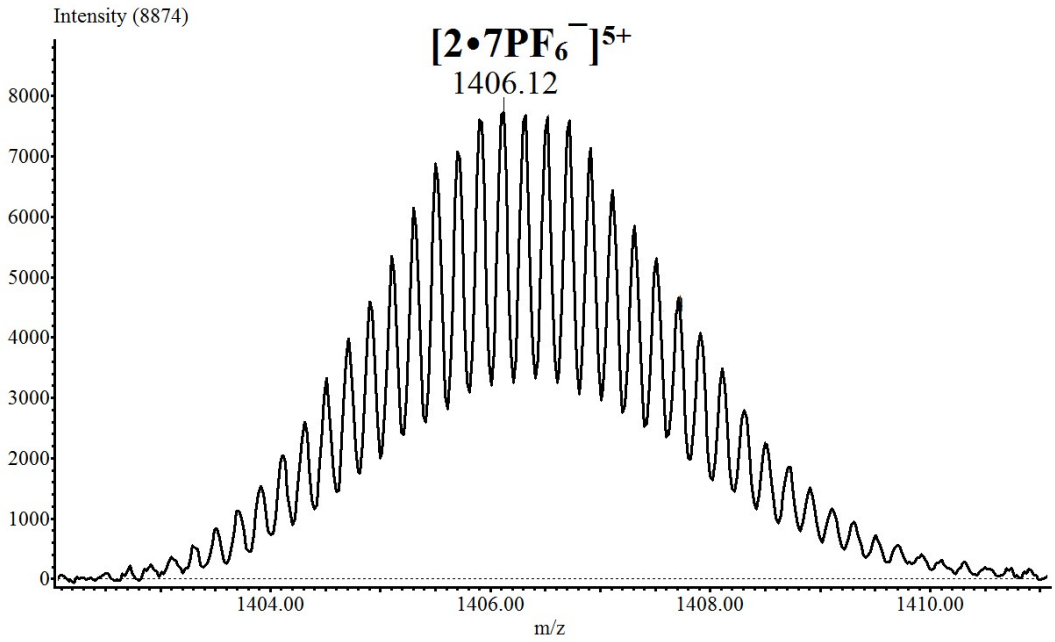

Fig. S11 The isotopic distribution of the species **[2**·7PF_6^-**]**$^{5+}$.

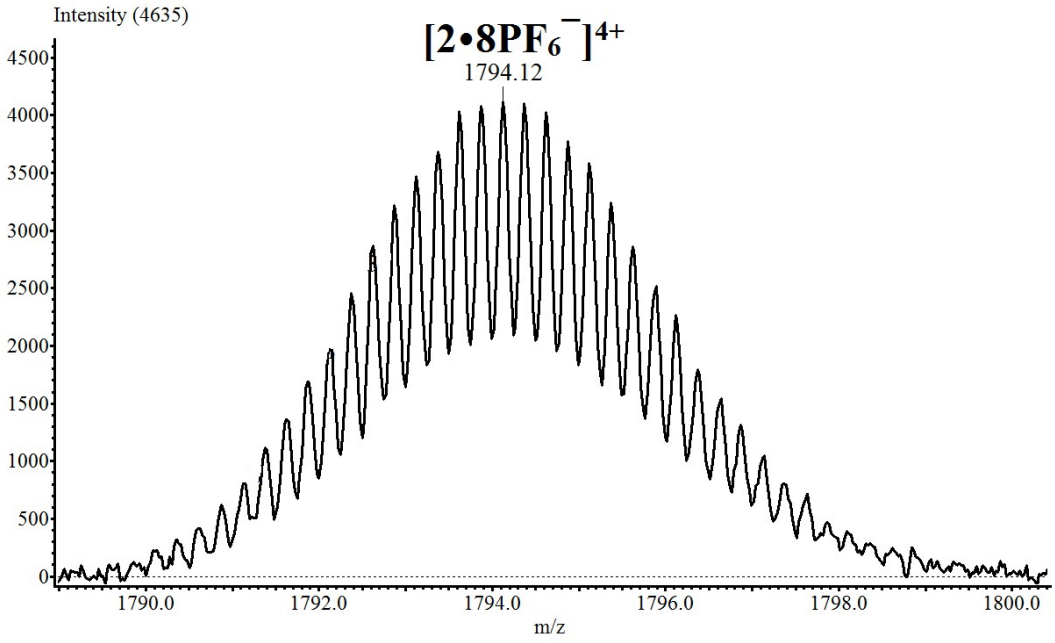

Fig. S12 The isotopic distribution of the species **[2**·8PF_6^-**]**$^{4+}$.

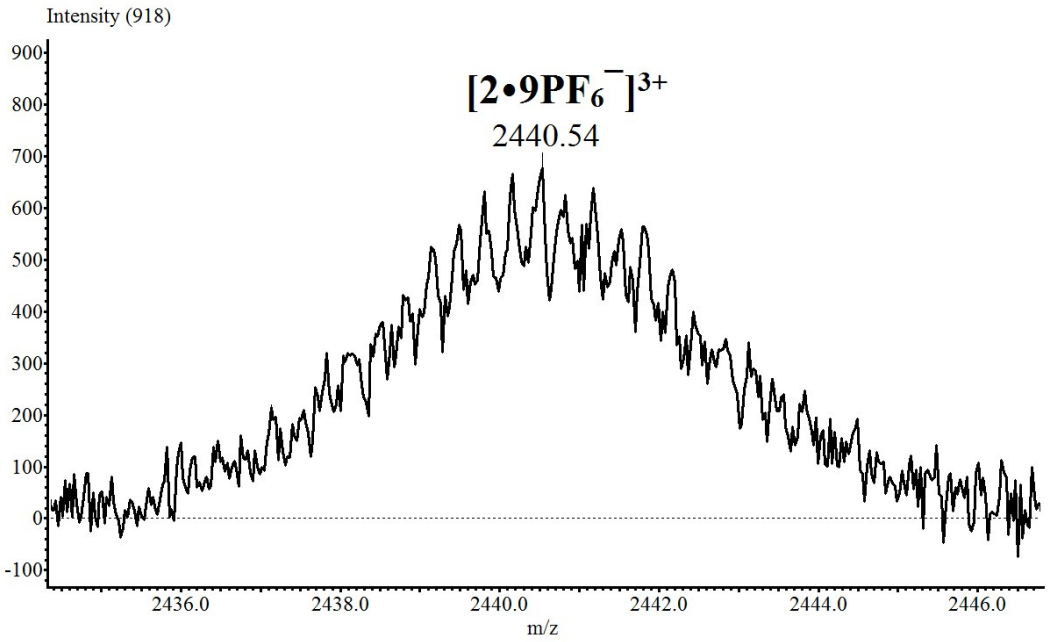

Fig. S13 The isotopic distribution of the species **[2**·9PF_6^-**]**$^{3+}$.

X-ray diffraction measurement

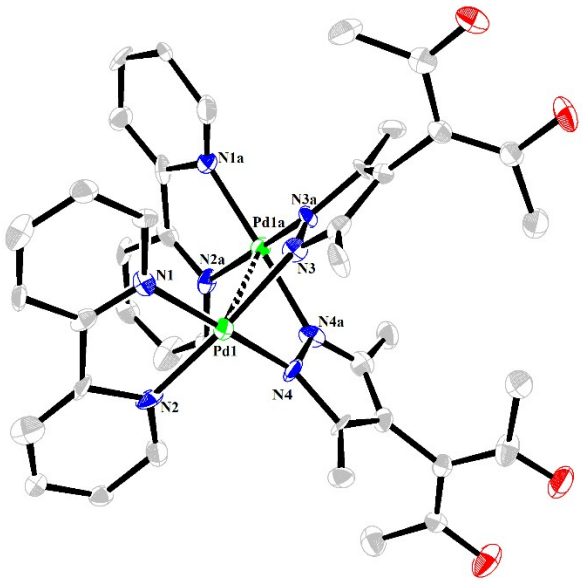

Fig. S14 ORTEP diagram of the molecular structure of **C1**•2PF_6^-. Thermal ellipsoids are shown at the 30% probability level. Counter ions and solvent molecules are omitted for clarity.

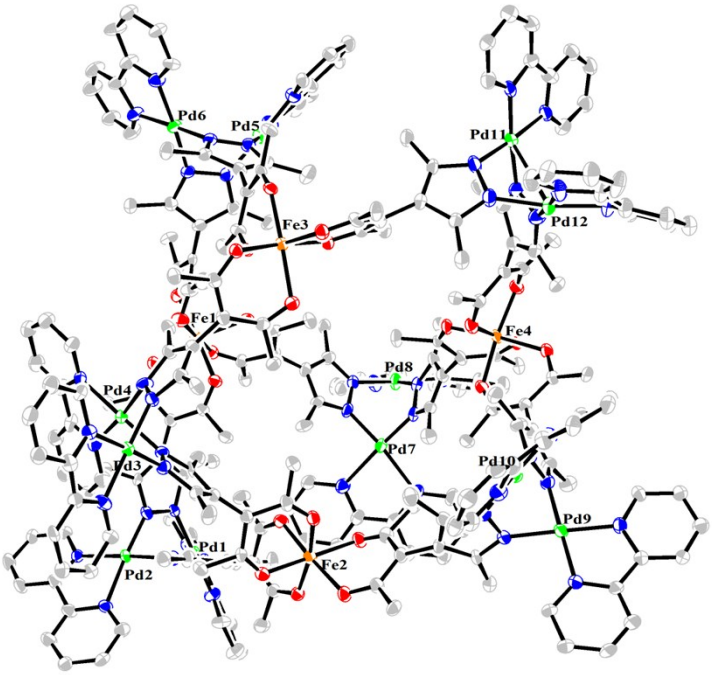

Fig. S15 ORTEP diagram of the molecular structure of **1**•12PF_6^-. Thermal ellipsoids are shown at the 30% probability level. Counter ions and solvent molecules are omitted for clarity.

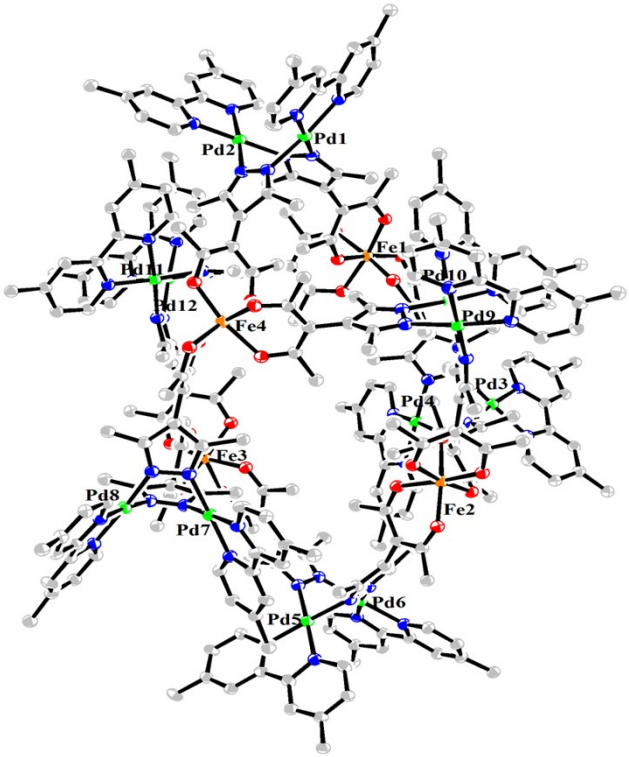

Fig. S16 ORTEP diagram of the molecular structure of **2**•12PF_6^-. Thermal ellipsoids are shown at the 30% probability level. Counter ions and solvent molecules are omitted for clarity.

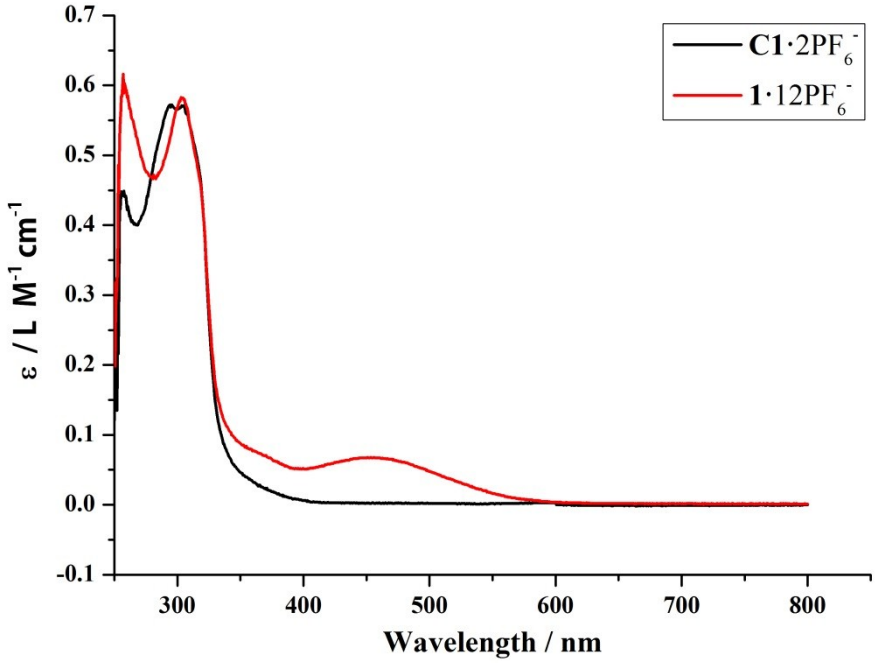

Fig. S17 UV-vis absorption spectra of compound **C1**·$2PF_6^-$ and **1**·$12PF_6^-$ (1×10^{-5} M) in CH_3CN at 25 °C (black, **C1**·$2PF_6^-$; red, compound **1**·$12PF_6^-$).

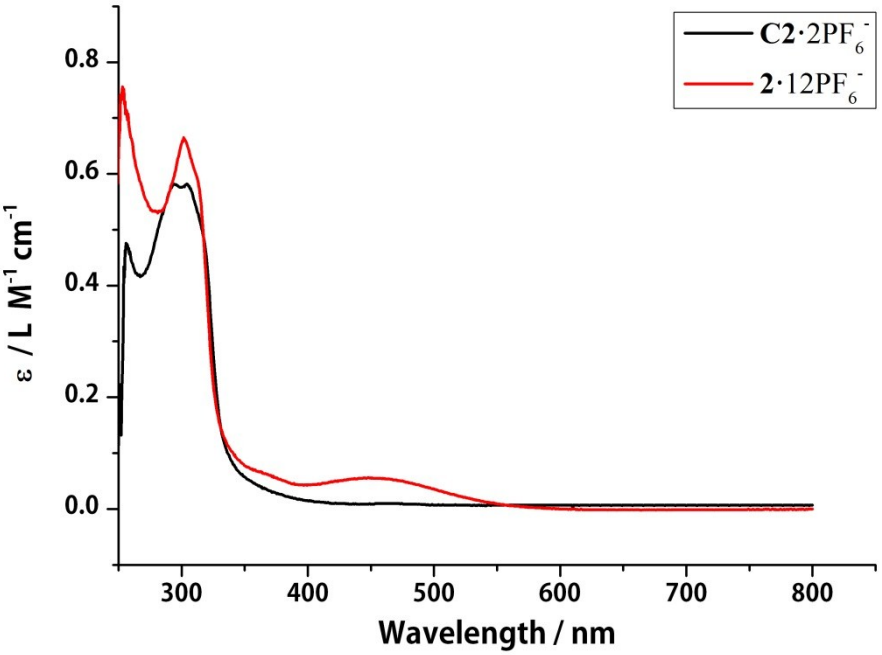

Fig. S18 UV-vis spectrum of compound **C2**·$2PF_6^-$ and **2**·$12PF_6^-$ (1×10^{-5} M) in CH_3CN at 25 °C (black, compound **C2**·$2PF_6^-$; red, compound **2**·$12PF_6^-$)

Table S1. Crystallographic data for complexes C**1**·2PF_6^-, **1**·12PF_6^- and **2**·12PF_6^-.

	C**1**·2PF_6^-	**1**·12PF_6^-	**2**·12PF_6^-
Formula	$C_{40}H_{42}F_{12}N_8O_4P_2Pd_2$	$C_{240}H_{240}F_{72}Fe_4N_{48}O_{24}P_{12}Pd_{12}$· 8($CH_3CN$)	$C_{264}H_{288}F_{72}Fe_4N_{48}O_{24}P_{12}Pd_{12}$ ·14(CH_3CN)
FW	1201.55	7749.02	8831.98
crystal system	orthorhombic	Triclinic	Triclinics
space group	Pnma	P-1	P-1
a [Å]	18.797(4)	25.0517(12)	25.6021(15)
b [Å]	18.398(4)	32.2634(14)	26.9162(12)
c [Å]	13.758(3)	35.368(2)	36.6641(13)
α [°]	90	108.9680(10)	68.991(2)
β [°]	90	101.156(2)	73.112(2)
γ [°]	90	108.3630(10)	64.351(3)
V [$Å^3$]	4757.9(18)	24223(2)	20985.3(18)
Z	4	2	2
ρcalcd, [g/cm^{-3}]	1.677	1.017	1.267
μ [mm^{-1}]	0.919	0.657	1.039
F(000)	2400	7384	7768
2θmax [°]	50.00	54.00	62.52
no. unique data	4209	105551	93235
Parameters	337	3757	3991
GOF [F^2][a]	1.061	0.794	1.010
R[$F^2>2\sigma(F^2)$]	0.0327	0.0408	0.0511
wR[F^2][b]	0.0892	0.1028	0.1235

Platon-squeeze details and solvents assignment for cage **1**:

The crystallography revealed that cage **1** crystallized in lower symmetry space group p-1 and two formula units existed in one cell. Based on the data refined using the platon-squeeze program, each unit has a formula of $[C_{264}H_{288}Fe_4N_{48}O_{24}Pd_{12}](PF_6)_{12}$, as well one void of 11674 $Å^3$(377 electrons) located in per unit cell. This residual electron density was assigned to ~17 molecules of the acetonitrile solvent [377=22×17.15]. Due to there are two formula units in one cell, it's possible that the formula unit includes ~8 (17/2=8.5) acetonitrile molecules. On the basis of these results, these solvent molecules were include in the sum formula, formula weight and calculated density, respectively. The tentative formula for cage **1** is $[C_{240}H_{240}Fe_4N_{48}O_{24}Pd_{12}](PF_6)_{12}$ $(CH_3CN)_8$.

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_platon_squeeze_void_average_x
_platon_squeeze_void_average_y
_platon_squeeze_void_average_z

_platon_squeeze_void_volume
_platon_squeeze_void_count_electrons
_platon_squeeze_void_content
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_chemical_formula_sum
'C256 H264 F72 Fe4 N56 O24 P12 Pd12 [solvent]'

Platon-squeeze details and solvents assignment for cage **2**:

The refined crystal structure indicated that cage **2** crystallized in lower symmetry space group p-1 with two formula in per cell. When we used the squeeze to refine this crystal data to solve all solvent molecules last time, the squeezed data reveals a formula $[C_{264}H_{288}Fe_4N_{48}O_{24}Pd_{12}](PF_6)_{12}$, as well a void of 7373 $Å^3$(604 electrons) located in per unit cell. This residual electron density was assigned to ~28 molecules of the acetonitrile solvent [604 = 22×27.45]. Due to there are two formula units in one cell, it's possible that the formula unit includes 14 (28 / 2=14) acetonitrile molecules.

After we refine this data of cage 2 again, there are 12 CH_3CN revealed in very well ordered within the cage structure in per cell by difference fourier diagram. The squeeze data reveals a formula $[C_{264}H_{288}Fe_4N_{48}O_{24}Pd_{12}](PF_6)_{12}(CH_3CN)_6$, and a void of 6148 $Å^3$(474 electrons) located in per cell. This residual electron density was assigned to 22 molecules of the acetonitrile solvent [474 = 22×21.54] in per cell. Due to there are two formula units in one cell, that the formula unit includes about 7－13 acetonitrile molecules taking account of 30% error (22 / 2 ≈ 10). According to the number of solvent molecules from the squeezed data last time, we have reason to guess there are about 8 molecules of acetonitrile solvent that are more precise than the number of 10 we calculated according to the residual electron density. On the basis of these results, these solvent molecules were include in the sum formula, formula weight and calculated density, respectively. So, the tentative formula for cage **2** is $[C_{264}H_{288}Fe_4N_{48}O_{24}Pd_{12}](PF_6)_{12}(CH_3CN)_{14}$.

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_platon_squeeze_void_nr

_platon_squeeze_void_average_x

_platon_squeeze_void_average_y

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2 0.767 0.416 0.193 13 -1 ' '

3 0.233 0.584 0.807 12 -1 ' '

_chemical_formula_moiety

' C264 H288 Fe4 N48 O24 Pd12, 12(F6 P),14(MeCN)'

_chemical_formula_sum

'C292 H330 F72 Fe4 N62 O24 P12 Pd12'

_chemical_formula_weight 8331.98

Table S2. Selected bond distances (Å) for complex **C1**·2PF_6^-, **1**·12PF_6^- and **2**·12PF_6^-.

C**1**·2PF_6^-		**1**·12PF_6^-		**2**·12PF_6^-	
Pd1-Pd1a	3.1357(8)	Pd1-Pd2	3.2323(4)	Pd1-Pd2	3.2388(4)
		Pd3-Pd4	3.2851(4)	Pd3-Pd4	3.2475(4)
		Pd5-Pd6	3.2197(4)	Pd5-Pd6	3.2513(4)
		Pd7-Pd8	3.2500(4)	Pd7-Pd8	3.2665(4)
		Pd9-Pd10	3.2027(4)	Pd9-Pd10	3.1346(4)
		Pd11-Pd12	3.1826(4)	Pd11-Pd12	3.2597(4)
		Fe1-Fe2	11.3165	Fe1-Fe2	11.7086
		Fe1-Fe3	11.3790	Fe1-Fe3	11.2921
		Fe1-Fe4	11.4655	Fe1-Fe4	11.2536

	Fe2-Fe3	11.9849	Fe2-Fe3	11.3407
	Fe2-Fe4	11.0181	Fe2-Fe4	11.4311
	Fe3-Fe4	10.7871	Fe3-Fe4	11.7767

Table S3. Selected bond distances (Å) for complex **C1**·2PF_6^-.

Bond Dist. (Å)		Bond Dist. (Å)	
Pd1-N1	2.025(2)	Pd1-N2	2.035(2)
Pd1-N3	2.012(2)	Pd1-N4	2.011(2)

Table S4. Selected bond distances (Å) for complex **1**·12PF_6^-.

	Bond Dist. (Å)		Bond Dist. (Å)
Pd1-N1	1.954(3)	Pd1-N2	2.093(3)
Pd1-N25	1.895(2)	Pd1-N27	1.934(3)
Pd2-N3	2.164(2)	Pd2-N4	1.992(2)
Pd2-N26	1.926(2)	Pd2-N28	1.922(2)
Pd3-N5	2.064(2)	Pd3-N6	1.932(3)
Pd3-N29	1.939(3)	Pd3-N31	1.951(2)
Pd4-N7	2.022(2)	Pd4-N8	1.966(3)
Pd4-N30	1.921(2)	Pd4-N32	1.940(3)
Pd5-N9	1.996(3)	Pd5-N10	1.974(3)
Pd5-N33	1.943(3)	Pd5-N35	1.915(3)
Pd6-N11	2.036(3)	Pd6-N12	1.971(3)
Pd6-N34	2.043(3)	Pd6-N36	1.915(3)

Pd7-N13	2.304(3)	Pd7-N14	2.345(3)
Pd7-N37	1.878(3)	Pd7-N39	1.928(3)
Pd8-N15	2.435(3)	Pd8-N16	2.312(3)
Pd8-N38	1.903(3)	Pd8-N40	1.949(3)
Pd9-N17	1.962(3)	Pd9-N18	2.023(3)
Pd9-N41	1.963(3)	Pd9-N43	1.962(3)
Pd10-N19	2.017(3)	Pd10-N20	2.055(3)
Pd10-N42	1.963(3)	Pd10-N44	1.988(3)
Pd11-N21	1.946(3)	Pd11-N22	2.070(3)
Pd11-N45	1.993(3)	Pd11-N47	1.884(3)
Pd12-N23	1.964(3)	Pd12-N24	1.990(3)
Pd12-N46	1.996(3)	Pd12-N48	1.983(3)
Fe1-O1	2.001(2)	Fe1-O2	1.968(2)
Fe1-O3	1.946(2)	Fe1-O4	1.994(2)
Fe1-O5	1.995(2)	Fe1-O6	2.006(2)
Fe2-O7	2.000(2)	Fe2-O8	2.012(2)
Fe2-O9	1.987(2)	Fe2-O10	1.997(2)
Fe2-O11	2.059(2)	Fe2-O12	2.001(2)
Fe3-O13	1.999(2)	Fe3-O14	2.146(2)
Fe3-O15	2.0176(19)	Fe3-O16	2.015(2)
Fe3-O17	1.991(2)	Fe3-O18	1.995(2)

Fe4-O19	2.007(2)	Fe4-O20	2.032(2)
Fe4-O21	2.013(2)	Fe4-O22	2.015(2)
Fe4-O23	2.013(2)	Fe4-O24	2.009(2)

Table S5. Selected bond distances (Å) for complex **2**·12PF_6^-.

	Bond Dist. (Å)		Bond Dist. (Å)
Pd1-N1	2.052(2)	Pd1-N2	2.000(2)
Pd1-N25	2.075(3)	Pd1-N27	2.033(2)
Pd2-N3	2.033(2)	Pd2-N4	1.980(2)
Pd2-N26	2.045(2)	Pd2-N28	2.021(2)
Pd3-N5	2.049(2)	Pd3-N6	2.030(3)
Pd3-N29	2.011(2)	Pd3-N31	2.028(2)
Pd4-N7	2.027(2)	Pd4-N8	1.997(2)
Pd4-N30	2.026(2)	Pd4-N32	2.085(2)
Pd5-N9	1.995(2)	Pd5-N10	2.049(2)
Pd5-N33	2.075(2)	Pd5-N35	1.996(2)
Pd6-N11	2.055(2)	Pd6-N12	2.023(2)
Pd6-N34	2.020(2)	Pd6-N36	2.039(2)
Pd7-N13	2.019(2)	Pd7-N14	2.021(2)
Pd7-N46	2.065(2)	Pd7-N48	2.064(2)
Pd8-N15	2.039(2)	Pd8-N16	2.013(2)
Pd8-N45	2.014(2)	Pd8-N47	1.996(2)

Pd9-N17	2.101(3)	Pd9-N18	2.031(2)
Pd9-N37	2.068(3)	Pd9-N39	1.997(2)
Pd10-N19	2.000(3)	Pd10-N20	2.009(2)
Pd10-N38	2.104(3)	Pd10-N40	2.050(2)
Pd11-N21	2.041(2)	Pd11-N22	2.036(3)
Pd11-N41	1.999(2)	Pd11-N43	2.077(3)
Pd12-N23	2.022(3)	Pd12-N24	1.945(2)
Pd12-N42	2.064(2)	Pd12-N44	2.078(2)
Fe1-O1	2.003(2)	Fe1-O2	1.978(2)
Fe1-O3	1.777(2)	Fe1-O4	2.000(2)
Fe1-O5	1.992(2)	Fe1-O6	2.001(2)
Fe2-O7	2.032(2)	Fe2-O8	2.048(2)
Fe2-O9	2.009(2)	Fe2-O10	2.022(2)
Fe2-O11	2.017(2)	Fe2-O12	1.990(2)
Fe3-O13	2.031(2)	Fe3-O14	2.052(2)
Fe3-O15	2.029(2)	Fe3-O16	2.003(2)
Fe3-O17	2.073(2)	Fe3-O18	2.018(2)
Fe4-O19	1.970(2)	Fe4-O20	2.052(2)
Fe4-O21	1.958(2)	Fe4-O22	2.037(2)
Fe4-O23	2.009(2)	Fe4-O24	2.064(2)

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