Syntheses and molecular structures of half-sandwich iridium metallarectangles containing bridging 2,5-dihydroxy-1,4- benzoquinonato (dhbq) ligands

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ABSTRACT

Binuclear complex $(Cp^*IrCl)_2(dhbq)$ (dhbq = 2,5-dihydroxy-1,4-benzoquinonato) (**1**) was obtained by the reaction of $(Cp^*IrCl_2)_2$ with bridging ligand 2,5-dihydroxy-1,4-benzoquinone(H_2dhbq) in the presence of the base n-$BuNH_2$. After treatments of **1** with AgX (X = NO_3^-, $SO_3CF_3^-$) and then with N-linkers (pyrazine, 4,4′-bipyridine), the corresponding tetranuclear metallarectangular complexes $[(Cp^*Ir)_4(dhbq)_2(pyrazine)_2](NO_3)_4·CH_2Cl_2·5H_2O$ (**2**) and $[(Cp^*Ir)_4(dhbq)_2(4,4′-bipyridine)_2](SO_3CF_3)_4$ (**3**) were obtained in good yields. Both the products were characterized by IR, ^{1}H NMR and single crystal X-ray analyses and revealed that these tetranuclear complexes were constructed from half-sandwich metal corners with both dhbq and N-linkers to form rectangular cavities with the dimensions 8.0×6.9 Å (Ir–Ir separations) for **2**, 8.0×11.2 Å (Ir–Ir separations) for **3**, respectively. In additionally, in the solid structure of **2** the counteranions NO_3^- are located out of the cavities with the observed water molecules by hydrogen bonding of the type $N–O···H–O–H···O–N$. One-dimensional channels are observed in **3** along the b axis with intramolecular stacking, the similar arrangement is not found in the solid of **2**.

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1. Introduction

Self-assembly of supramolecular architectures with transition metal corners, such as triangles, squares, rectangles, polygons and even 3-dimensional "organometallic boxes" has attracted interest in last decade [1–4]. After the first case of molecular square $[\{Pd(en)(\mu\text{-}4,4′\text{-}bipy)\}_4](NO_3)_8$ reported by Fujita and co-workers in 1990 [5], a series of supramolecules with platinum or palladium were developed in host-guest chemistry – encapsulating compounds, recognizing or trapping guest molecules, microreactors for specific reactions [6–24]. Using metal centers as corners and organic linkers in the build up of molecular architectures led to materials with new properties.

Organometallic half-sandwich complexes have also been widely used as building blocks in the construction of supramolecular complexes [25–48]. Recently, the *fac*-Re(CO)₃ corner system was chosen to prepare molecular rectangles with 2,2′-bipyrimidine, dianionic benzimidazolate, sulfides, selenides or alkoxide bridging ligands [49,50]. Similarly, (arene)Ru and (cyclopentadienyl)M (M = Ir, Rh) have been intensively investigated by some groups and to generate metallacycles, rectangles, trigonal prisms, hexagonal prisms, and other supramolecular geometries [25–48]. We and the group of Süss-Fink have studied the formation of tetra- and hexa-nuclear complexes bearing dichloro or oxalate bridges con-

nected by two and three pyridyl-based linkers [39–48]. Also, the octanuclear "organometallic boxes" with oxalate bridges have been prepared [25,41]. Recently, several coordination complexes containing chloranilate (CA) as bis-bidentate ligands have been studied in order to expanding the "pillars" of the architectures. The synthesis and characterization of some ruthenium tetranuclear metallarectangles using 2,5-dichloro-1,4- benzoquinonato and 2,5-dihydroxy-1,4-benzoquinonato were reported, along with the anti-cancer activities and electrochemical behaviors [40,43,48]. The "complex-in-a-complex" systems bearing with ruthenium corners and two trigonal 2,4,6-tris(pyridin-4-yl)-1,3,5-triazine (tpt) panels and dhbq bridges also were studied and showed high cytotoxicity toward human ovarian cancer cells [45]. Within this context and in an effort to develop new supramolecular structures, a family of iridium di- or tetra-nuclear complexes was prepared and characterized.

Herein, we describe the formation of binuclear complex $(Cp^*IrCl)_2(dhbq)$ (dhbq = 2,5-dihydroxy-1,4-benzoquinonato) (**1**) by the reaction of $(Cp^*IrCl_2)_2$ with bridging ligand H_2dhbq in the presence of the base n-$BuNH_2$ (Scheme 1). After treatments of **1** with AgX (X = NO_3^-, $SO_3CF_3^-$) and N-linkers (pyrazine, 4,4′-bipyridine), the corresponding tetranuclear metallarectangular complexes $[(Cp^*Ir)_4(dhbq)_2(pyrazine)_2](NO_3)_4·CH_2Cl_2·5H_2O$ (**2**) and $[(Cp^*Ir)_4(dhbq)_2(4,4′-bipyridine)_2](SO_3CF_3)_4$ (**3**) were obtained in good yields (Scheme 2). Complexes **2** and **3** were characterized by IR, ^{1}H NMR and single crystal X-ray analyses and revealed that these tetranuclear complexes constructed from half-sandwich

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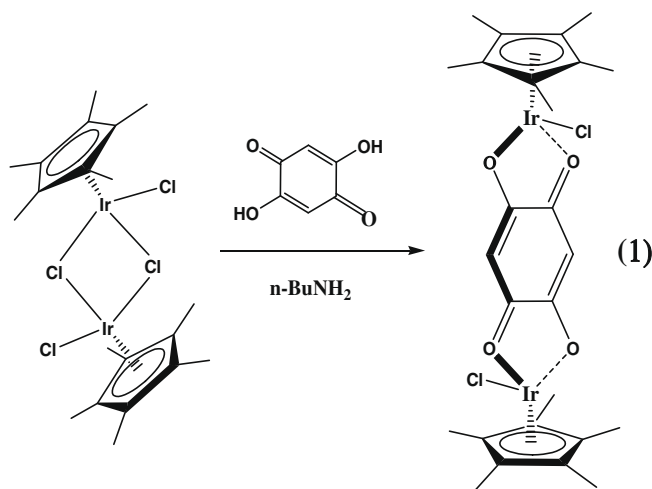
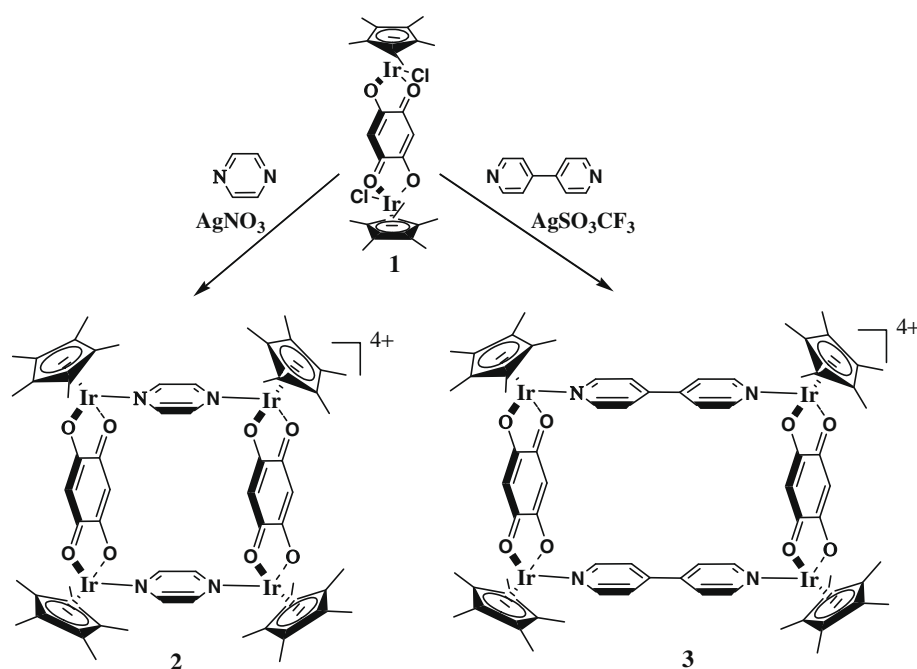**Scheme 1.** Synthesis of binuclear complex **1** in the presence of base.

metal corners with both dhbq and N-linkers formed rectangular cavities with the dimensions 8.0×6.9 Å (Ir–Ir separations) for **2**, 8.0×11.2 Å (Ir–Ir separations) for **3**.

2. Results and discussion

The tetranuclear rectangular complexes **2** and **3** were prepared following the two-step strategy, in which the binuclear complex **1** was used for the "first edge". The binuclear complex **1** as a precursor was synthesized by the mixture of $(Cp^*IrCl_2)_2$ and 2,5-dihydroxy-1,4- benzoquinone(H_2dhbq) in the presence of base in high yield (83%, Scheme 1). In the IR spectra of **1**, two strong bands were observed at 1376 and 1257 cm^{-1} for the dhbq ligands, a sharp band at 2965 cm^{-1} was also revealed the Cp* units. The ^{1}H NMR spectra exhibited a sharp singlet at $\delta = 1.73$ ppm due to the Cp* group, and the spectra showed $\delta = 5.93$ ppm singlets for dhbq ligands. Detailed structural information of **1** came from single crystal X-ray

diffraction analysis. Perspective drawing of binuclear complex **1** is shown in Fig. 1, and selected bond lengths and angles of **1** are given in Table 1. As shown in Fig. 1, the expected molecule contains two half-sandwich iridium metal corners, which are bridged by the dhbq ligand via its four oxygen atoms. Each metal center is thus coordinated by two chelate O_{dhbq} atoms and one Cl atom by a three-legged piano stool shape. The two chloride atoms are located on both sides of dhbq plane. The Ir–Ir separation is about 8.0 Å. The stacking of molecules **1** from b axis is shown in Fig. 1.

Tetranuclear complexes **2** and **3** bearing N–N liangds, which are formulated as $[Cp^*_4Ir_4(dhbq)_2(L)_2](X)_4$ (**2**, L = pyrazine, X = NO$_3^-$; **3**, L = 4,4′-bipyridine, X = OTf$^-$) were prepared by the reactions of **1** with bidentate ligands in the presence of AgX (Scheme 2). When pyrazine was added to the filtrate which was obtained from **1** and AgNO$_3$ in a 1:2 molar ratio in MeOH at room temperature, the complex **2** was obtained as dark red crystals in the yield of 73%. In the IR spectra of **2**, two strong bands were observed at 1384 and 1261 cm^{-1} for the dhbq ligands and 1542 cm^{-1} for the pyrazine groups, the Cp* signal at 2963 cm^{-1} was also observed. The ^{1}H NMR spectra exhibited a sharp singlet at $\delta = 1.74$ ppm due to the Cp* group, and the spectra showed $\delta = 5.93$ singlet for dhbq ligands, as well as $\delta = 8.76$ ppm for pyrazine protons. The molecular structure of **2** was determined by X-ray analysis. As expected, the tetranuclear complex is formed after the replacement of the neutral pyrazine for the Cl atom through the intermediate of the binuclear triflate salts. The perspective drawing is shown in Fig. 2, the cation of **2** include four half-sandwich iridium metal corners, which are bridged by the dhbq ligands through its four oxygen atoms and linked via the pyrazine ligand. The rectangular molecule shows a 8.0×6.9 Å cavity (Ir–Ir separations). Excluding the van der Waals radii (C 1.7 Å), the inner cavities are almost 4.6×3.5 Å. The rectangular angles are 83.8° (Ir1), 83.7° (Ir2). Interestingly, in the solid structure the counteranions of NO$_3^-$ are positioned out of the cavities with the observed water molecules by hydrogen bonding of the type N–O···H–O–H···O–N, which is revealed in the stacking view of Fig. 2. The selected bond lengths and angles of **2** are given in Table 2.

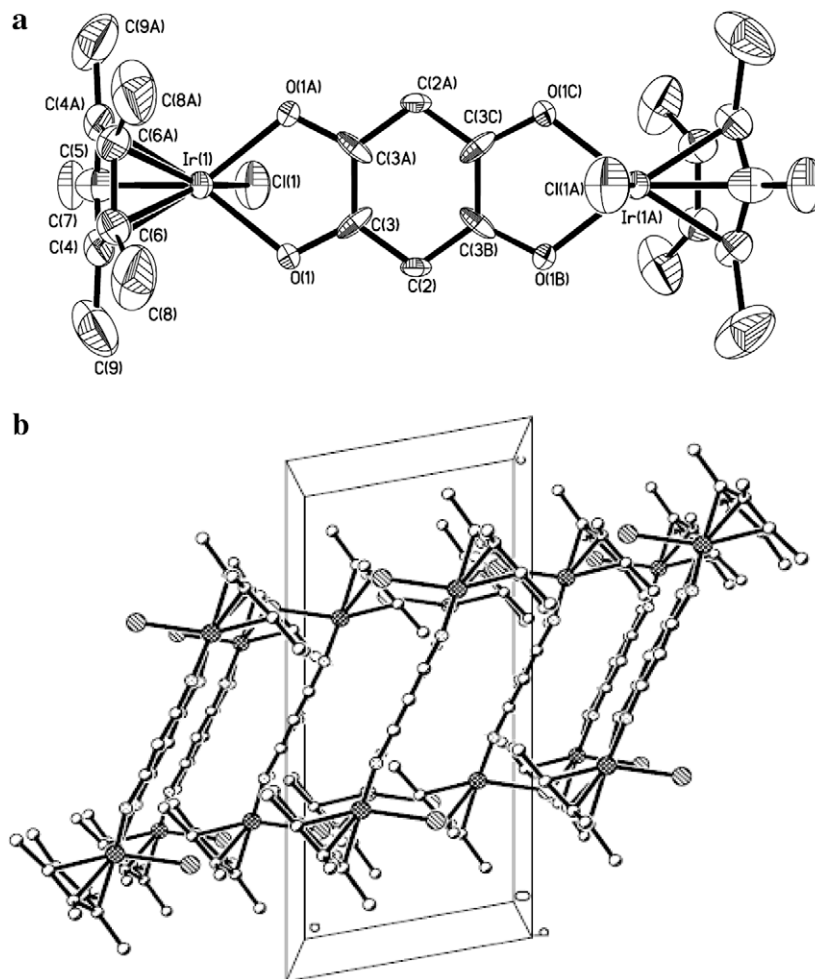**Scheme 2.** Synthesis of tetranuclear rectangular complexes **2** and **3**.

Fig. 1. (a) Molecular structure of binuclear complex 1; (b) stacking of the molecules of crystal 1. Hydrogen atoms have been omitted for clarity. Ellipsoids are shown at 30% probability level.

Table 1
Selected bond distances (Å) and angles (°) for **1**.

Bond distances (Å)			
Ir(1)–O(1A)	2.105(12)	Ir(1)–O(1)	2.105(12)
Ir(1)–Cl(1)	2.361(8)	O(1)–C(3)	1.35(2)
Bond angles (°)			
O(1A)–Ir(1)–O(1)	75.0(7)	O(1A)–Ir(1)–Cl(1)	83.2(4)
O(1)–Ir(1)–Cl(1)	83.2(4)		

Similarly, when 4,4′-bipyridine was added to the filtrate which was obtained from **1** and AgOTf in a 1:2 molar ratio in MeOH at room temperature, the complex **3** was obtained as dark red prismatic crystals in the yield of 68%. In the IR spectra of **3**, a sharp signal at 2966 cm^{-1} shown the Cp* units, both strong bands were observed at 1378 and 1261 cm^{-1} for the dhbq ligands and 1619 and 1527 cm^{-1} for the pyridine groups. The ^{1}H NMR spectra revealed a sharp singlet at δ = 1.68 ppm due to the Cp* group, and the spectra showed δ = 5.95 ppm singlet for dhbq ligands, as well as δ = 8.31 and 8.74 ppm for pyridine protons. The rectangular molecules of **3** are shown the expected larger cavity from the crystal study. As shown in Fig. 3, similar to complex **2**, this tetranuclear complex also contains four half-sandwich iridium metal corners formed a 8.0 × 11.2 Å cavity (Ir–Ir separations). The four iridium atoms are bridged by two dhbq ligands through chelate O atoms

and linked by the longer 4,4′-bipyridine ligands. The inner cavities excluding the van der Waals radii (C 1.7 Å) are almost 4.6 × 7.8 Å. The rectangular angles are 84.8° (Ir1), 84.3° (Ir2). Both the pyridyl planes in 4,4′-bipyridine just have a 3.1° twisted angle, much smaller than the reported similar CA-bridged rectangular structures about 22° [40,43]. From the packing view of **3**, the 1-dimensional channels are observed along the b axis with intramolecular stacking, which is shown in Fig. 3. A similar arrangement is not found in the crystal of **2**. The selected bond lengths and angles of **3** are given in Table 3.

3. Conclusion

In conclusion, we have described the formation of tetranuclear metallarectangular complexes [(Cp*Ir)$_4$(dhbq)$_2$(pyrazine)$_2$](NO$_3$)$_4$·CH$_2$Cl$_2$·5H$_2$O (**2**) and [(Cp*Ir)$_4$(dhbq)$_2$(4,4′-bipyridine)$_2$] (SO$_3$CF$_3$)$_4$ (**3**) from the precursor complex (Cp*IrCl)$_2$(dhbq) (**1**) in good yields. All the products were characterized by IR, ^{1}H NMR and single crystal X-ray analyses and revealed that these tetranuclear complexes were formed by the half-sandwich metal corners with both dhbq and N-linkers constructed rectangular cavities with the dimensions 8.0 × 6.9 Å (Ir–Ir separations) for **2**, 8.0 × 11.2 Å (Ir–Ir separations) for **3**. In additionally, in the solid structure of **2** the counteranions NO$_3^-$ are located out of the cavities with the observed water molecules by hydrogen bonding of the type N–
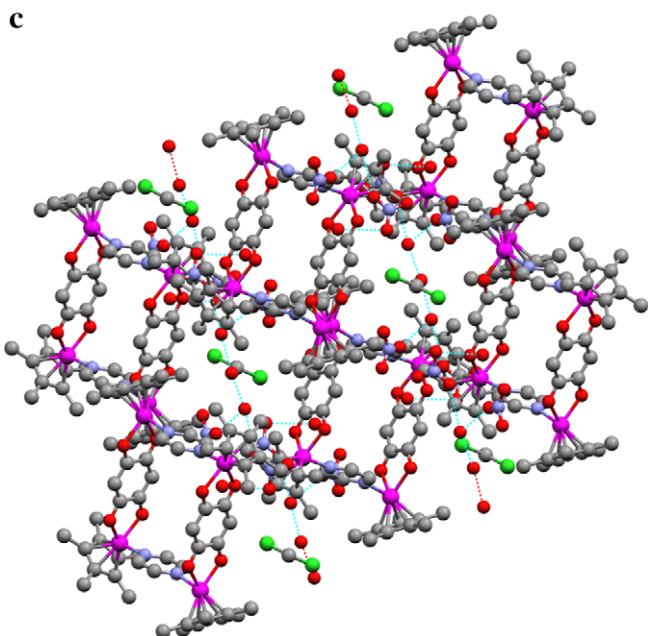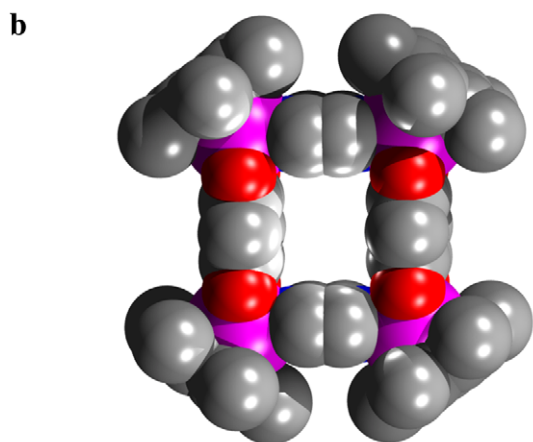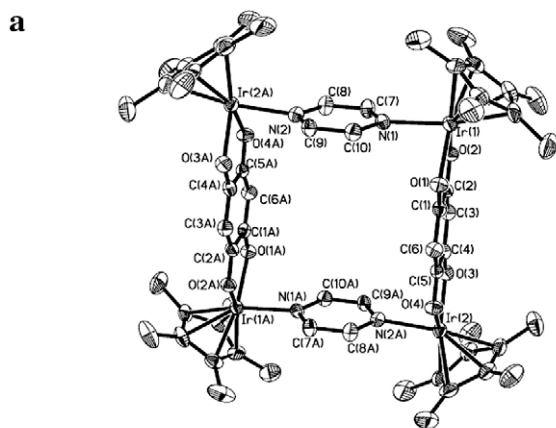
a

b

c

Table 2
Selected bond distances (Å) and angles (°) for **2**.

Bond distances (Å)			
Ir(1)–N(1)	2.092(5)	Ir(1)–O(2)	2.096(4)
Ir(1)–O(1)	2.111(4)	Ir(2)–N(2A)	2.099(5)
Ir(2)–O(4)	2.105(4)	Ir(2)–O(3)	2.108(4)
Bond angles (°)			
N(1)–Ir(1)–O(2)	84.86(17)	N(1)–Ir(1)–O(1)	84.89(17)
O(2)–Ir(1)–O(1)	76.22(15)	N(2A)–Ir(2)–O(4)	84.79(18)
N(2A)–Ir(2)–O(3)	84.30(18)	O(4)–Ir(2)–O(3)	76.14(16)

a

b

c

Fig. 2. (a) Molecular structure of rectangular cationic part of complex 2; (b) space-filling model of cationic part of complex 2; (c) stacking of the molecules of crystal 2 containing hydrogen bondings with the type N–O···H–O–H···O–N. Hydrogen atoms have been omitted for clarity. Hydrogen bondings were shown by the green dotted line. Ellipsoids are shown at 30% probability level. Ir: purple C: black O: red N: blue. (For interpretation of colour in figures, the reader is referred to the Web version of this article.)
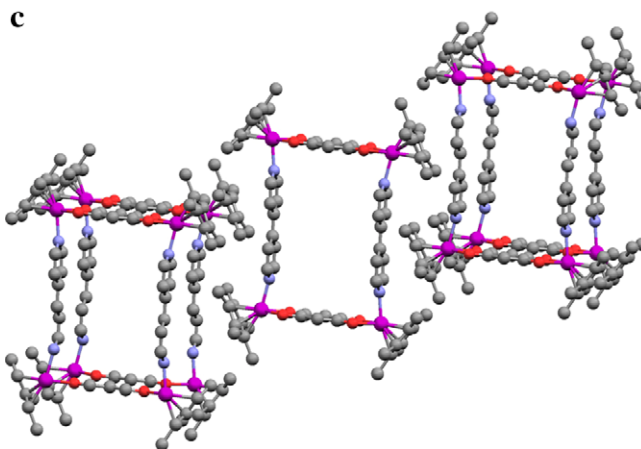
Fig. 3. (a) Molecular structure of rectangular cationic part of complex 3; (b) space-filling model of cationic part of complex 3; (c) stacking of the molecules of crystal 3. Hydrogen atoms have been omitted for clarity. Ellipsoids are shown at 30% probability level. Ir: purple C: black O: red N: blue. (For interpretation of colour in figures, the reader is referred to the Web version of this article.)

O···H–O–H···O–N. Meanwhile, 1-dimensional channels are observed in **3** along the b axis with intramolecular stacking. Further

investigations are sought to explore host-guest behavior of these metallarectangles. Based on the rational design principles for the

Table 3
Selected bond distances (Å) and angles (°) for **3**.

Bond distances (Å)			
Ir(1)–N(1)	2.098(8)	Ir(1)–O(1A)	2.108(4)
Ir(1)–O(1)	2.108(4)	Ir(2)–N(2)	2.064(8)
Ir(2)–O(2)	2.109(4)	Ir(2)–O(2A)	2.109(4)
Bond angles (°)			
N(1)–Ir(1)–O(1A)	84.7(2)	N(1)–Ir(1)–O(1)	84.7(2)
O(1A)–Ir(1)–O(1)	75.7(2)	N(2)–Ir(2)–O(2)	84.9(2)
N(2)–Ir(2)–O(2A)	84.9(2)	O(2)–Ir(2)–O(2A)	76.8(2)

construction of metallarectangular complexes, we are approaching the new molecules with designed properties.

4. Experimental

4.1. General considerations

All reactions were carried out under a nitrogen atmosphere using standard Schlenk techniques. All of the solvents were freshly distilled prior to use. CH_2Cl_2 was dried over CaH_2 and CH_3OH was distilled over Mg/I_2. $(Cp^*IrCl_2)_2$ was prepared according to reported procedures. Pyrazine, 4,4′-bipyridine and 2,5-dihydroxy-1,4-benzoquinone(H_2dhbq) were commercially availably and used as received. Infrared spectra were recorded on a Nicolet AVATAR-360IR spectrometer, whereas ^{1}H{400 MHz} NMR spectra were obtained on a Bruker DMX-500 spectrophotometer in $CDCl_3$ solvents. Elemental analyses were performed on Elementar vario EI Analyzer after vacuum the samples for days.

4.2. Syntheses and characterization

4.2.1. Synthesis and characterization of binuclear complex $(Cp^*IrCl)_2(dhbq)$ (**1**)

A mixture of 2,5-dihydroxy-1,4-benzoquinone(H_2dhbq) (0.25 mmol, 35 mg) and n-$BuNH_2$ (1.5 mmol, 0.15 ml) in MeOH was stirred at room temperature for 3 h. Then $(Cp^*IrCl_2)_2$ (0.25 mmol, 0.2 g) was added and the solution was kept to stir for 12 h. Then

the solvent was removed under reduced pressure, producing a dark red solid which was extracted from CH_2Cl_2. The residue solids were recrystallized from CH_2Cl_2/Et_2O to give crystals of **1** (180 mg, 83% yield). Elemental Anal. Calc. for C, 36.15; H, 3.73. Found: C, 36.04; H, 3.80%. ^{1}H NMR(400 MHz, $CDCl_3$): $\delta = 1.73$ (s, –CH_3, 30H), 5.93 (s, dhbq, 2H). IR (KBr disk): 2965, 1618, 1518, 1376, 1257, 1028, and 811 cm^{-1}.

4.2.2. Synthesis and characterization of tetranuclear complex $[(Cp^*Ir)_4(dhbq)_2(pyrazine)_2] (NO_3)_4 \cdot CH_2Cl_2 \cdot 5H_2O$ (**2**)

$AgNO_3$(0.23 mmol, 0.039 g) was added to a solution of **1** (0.1 mmol, 0.086 g) in MeOH and was kept to stir for 3 h. After filtration of AgCl, pyrazine (0.1 mmol, 0.008 g) was added, and the mixture was still stirred for 12 h. After removal of the solvents, the residue was extracted by CH_2Cl_2. Then slowly added Et_2O to the CH_2Cl_2 layer and kept it in room temperature for days until the dark red crystals were obtained (73 mg after vacuum for days, 73% yield). Elemental Anal. Calc. for C, 36.14; H, 3.64; N, 5.62. Found: C, 36.30; H, 3.72; N, 5.58%. ^{1}H NMR(400 MHz, $CDCl_3$): $\delta = 1.74$ (s, –CH_3, 60H), 5.93 (s, dhbq, 4H), 8.76 (s, pyrazine, 8H). IR (KBr disk): 2963, 1542, 1384, 1261, 1097, 1029, and 803 cm^{-1}.

4.2.3. Synthesis and characterization of tetranuclear complex $[(Cp^*Ir)_4(dhbq)_2(4,4′-bipyridine)_2] (SO_3CF_3)_4$ (**3**)

$AgSO_3CF_3$ (0.23 mmol, 0.059 g) was added to a solution of **1** (0.1 mmol, 0.086 g) in MeOH and was kept to stir for 3 h. After filtration of AgCl, 4,4′-bipyridine(0.1 mmol, 0.02 g) was added, and the mixture was still stirred for 24 h. After removal of the solvents, the residue was extracted by CH_2Cl_2. Then slowly added Et_2O to the CH_2Cl_2 layer and kept it in room temperature for days until the dark red crystals were obtained (85 mg after vacuum for days, 68% yield). Elemental Anal. Calc. for C, 36.59; H, 3.23; N, 2.25. Found: C, 36.54; H, 3.10; N, 2.34%. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 1.68$ (s, –CH_3, 60H), 5.95 (s, dhbq, 4H), 8.31(d, -pyridyl, 8H), 8.74 (d, -pyridyl, 8H). IR (KBr disk): 2966, 1618, 1527, 1378, 1261, 1102, 1030 and 803 cm^{-1}.

Table 4
Crystallographic data for compounds **1**–**3**.

	1	2	3
Empirical formula	$Ir_2C_{26}Cl_2O_4H_{32}$	$Ir_4C_{60}N_8O_{20}H_{72} \cdot CH_2Cl_2 \cdot 5H_2O$	$Ir_4C_{76}F_{12}N_4O_{20}S_4H_{80}$
T (K)	293(2)	293(2)	293(2)
Formula weight	863.82	2169.06	2494.48
Crystal size (mm^3)	$0.12 \times 0.10 \times 0.08$	$0.12 \times 0.10 \times 0.06$	$0.25 \times 0.15 \times 0.10$
Crystal system	monoclinic	monoclinic	monoclinic
Space group	C2/m	C2/c	C2/m
a (Å)	7.785(4)	21.600(6)	27.951(17)
b (Å)	12.550(7)	20.413(6)	10.556(7)
c (Å)	17.407(10)	22.715(7)	23.759(15)
α (°)	90	90	90
β (°)	100.945(9)	96.000(4)	115.906(8)
γ (°)	90	90	90
V ($Å^3$)	1669.8(16)	9961(5)	6306(7)
Z	2	4	2
ρ_{calc} (g/cm^3)	1.718	1.446	1.314
μ (Mo Kα) (mm^{-1})	8.145	5.441	4.339
Number of collected reflections	3490	23 872	13 161
Number of unique reflections	1549	10 732	5865
Number of parameters	86	462	218
Goodness-of-fit (GOF) on F^2	0.981	0.784	0.890
R_1, ωR_2 [I>2σ(I)][a]	0.0758, 0.1612	0.0350, 0.0723	0.0478, 0.1137
R_1, ωR_2 (all data)[a]	0.1180, 0.1718	0.0683, 0.0754	0.0686, 0.1203
Maximum and minimum residual density (e Å^{-3})	3.253 and −1.834	1.161 and −0.650	1.731 and −1.068

[a] $R_1 = \sum \|F_0| - |F_c\| / \sum |F_0|$ (based on reflections with $F_0^2 > 2\sigma F^2$); $wR_2 = [\sum[w(F_0^2 - F_c^2)^2]/ \sum[w(F_0^2)^2]]^{1/2}$; $w = 1/[\sigma^2(F_0^2) + (0.095P)^2]$; $P = [\max(F_0^2, 0) + 2F_c^2]/3$ (also with $F_0^2 > 2\sigma F_2$).

4.3. X-ray crystallography

Each crystal was mounted on glass fiber. Crystallographic measurements were made on a Bruker Smart Apex 100 CCD area detector using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 293 K. The structures were solved by directed methods (SHELXS-97) and refined on F^2 by full-matrix least squares (SHELX-97) using all unique data. There were disordered solvent molecules in complex **1**, **2** and **3** so the SQUEEZE algorithm was used to omit them. In complex **3**, all of the anions were also strongly disordered so that they were omitted by SQUEEZE algorithm before the structure was refined to convergence. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were placed in calculated positions with fixed isotropic thermal parameter. All hydrogen atoms of water molecules in **2** can not be found.

Crystal data, data collection parameters, and the results of the analyses of compounds **1**–**3** are listed in Table 4.

5. Supplementary material

CCDC 755468 (**1**), 755469 (**2**) and 755470 (**3**) contain the supplementary crystallographic data for **1**–**3**. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving. html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Acknowledgements

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