Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information for

Nano-sized Heterometallic Macrocycles based on 4-pyridinylboron-capped Iron(II) Clathrochelates: Syntheses, Structures and Properties

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Contents

Materials and instrumentations

General procedures: All manipulations were performed under an atmosphere of nitrogen using standard Schlenk techniques. However, once the reactions were completed, subsequent workups were done without precaution, as the compounds are air-stable for several weeks. Solvents were purified by standard methods prior to use. The starting material $[Cp^*IrCl(\mu\text{-}Cl)]_2$ was prepared as described earlier,[1] the 1,4-benzenebis(N,N′-dicyclohexylamidine) ($H_2\mathbf{L}_1$) was obtained by a known procedure[2] and the metallaligands $FeNx_3(B4\text{-}Py)_2$ (L^{Fe1})/ $FeDm_3(B4\text{-}Py)_2$ (L^{Fe2}) were prepared according to the literature method.[3] Other chemicals were obtained commercially and used without further purification.

Elemental analyses were performed on an Elementar III Vario EI analyzer. ^{1}H NMR (400 MHz) spectra were obtained on a Bruker DMX-400 spectrometer in $CDCl_3$ solution. IR spectra of the solid samples (KBr tablets) in the range 400-4000 cm^{-1} are measured on a Nicolet Avatar-360 spectrophotometer. UV-vis spectra of solutions in CH_2Cl_2 were recorded in the ranges 190–1100 nm with a Agilent 8453 spectrophotometer.

Cyclic voltammetry (CV) experiments were carried out in CH_2Cl_2 solutions with 0.1 M $((n\text{-}C_4H_9)_4N)(PF_6)$ as supporting electrolyte using a model Parstat 2273 (Princeton Applied Research, USA) potentiostat with a conventional and one-compartment three-electrode cell (10 mL of solution). A glassy carbon (GC) disk electrode with an active surface area of 0.125 cm^2 was used as a working electrode in solution. The electrode was thoroughly polished and rinsed before measurements. A platinum counter electrode and a saturated calomel electrode (SCE) reference electrode were applied. The measurements were performed at scan rates of 200 mV s^{-1}. All solutions were thoroughly deaerated by passing argon through the solution before the CV experiments and above the solution during the measurements.

Synthesis of ligand H_2L_2:

To a solution of 4,4'-Dibromobiphenyl (3.1 g, 10.0 mmol) in Et_2O (50 mL) was added 18.8 mL of *n*BuLi (1.6 M in C_6H_{14}, 30.0 mmol, three equivalents) at room temperature. The reaction mixture was stirred strongly at 40 °C for 12 h, affording a slight yellow cream slurry of the dilithio-salt 4,4'-$Li_2(C_6H_4)_2$. The solvent was removed by filtration and the remaining solid was washed with Et_2O (3 × 20 mL). The dilithio-salt was subsequently reslurried in THF (100 mL) and 1,3-dicyclohexylcarbodiimide (4.1 g, 20.1 mmol) was added at 0 °C affording a beige slurry. The mixture was stirred for 12 h followed by the slow addition of distilled water to afford a clear orange solution that was stirred at ambient temperature for 2 h. The solution was washed with water and the product was extracted with ether (3 × 20 mL). Then the volatiles was removed under reduced pressure to afford crude products that was recrystallised from Et_2O at -30 °C as colourless crystals (2.7 g, 48%). *Data for* $H_2\mathbf{L_2}$: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 1.19-1.83 (m, 40H, Cy), 3.09 (m, 4H, NCH), 7.31 (d, 4H, C_6H_4), 7.64 (d, 4H, C_6H_4); IR (KBr disk): ν = 3434, 2922, 2850, 1635, 1479, 1315, 1151, 1102, 978, 890, 826, 738, 613 cm^{-1}; elemental analysis calcd (%) for $C_{38}H_{54}N_4$: C 80.51; H 9.60; N 9.88; found C 80.28; H 9.81; N 9.60.

Synthesis of dinuclear complexes 1, 2:

1.4 mL (2.2 mmol) of *n*BuLi (1.6 M solution in hexane) was added dropwise to a solution of $H_2\mathbf{L_1}$ (492 mg, 1.0 mmol)/$H_2\mathbf{L_2}$ (567 mg, 1.0 mmol) in THF (30 mL) at -78 °C. The reaction mixture was stirred for 30 min, then slowly warmed up to room temperature and further stirred for 2 h. The result slurry was added via cannula to the rapidly stirred slurry of $[Cp^*IrCl(\mu\text{-}Cl)]_2$ (875 mg, 1.1 mmol) in THF (20 mL) at 0 °C. Then, the mixture was stirred at 70 °C for 12 h to give a dark red solution. The volatiles were removed under reduced pressure and the product was separated from lithium chloride by extraction with CH_2Cl_2. Then, the concentrated solution was recrystallized by adding hexane to give yellow crystals (**1**, 789 mg, 65%; **2**, 735mg, 57%). *Data for complex **1***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.85-1.94 (m, 40H, Cy), 1.78 (s, 30H, Cp*), 2.61 (m, 4H, NCH), 7.17 (d, br, 4H, C_6H_4); IR (KBr disk): ν = 2926, 2851, 1489, 1362, 1343, 1228, 1102, 1075, 1028, 989, 852, 611 cm^{-1}. *Data for complex **2***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.98-1.98 (m, 40H, Cy), 1.81 (s, 30H, Cp*), 2.67 (m, 4H, NCH), 7.24 (d, 2H, C_6H_4), 7.37 (d, 2H, C_6H_4), 7.64 (d, 4H, C_6H_4); IR (KBr disk): ν = 2924, 2851, 1488, 1362, 1344, 1260, 1226, 1099, 1072, 1028, 988, 887, 832, 639, 576, 519 cm^{-1}.

Synthesis of tetra-nuclear complexes 3a, 3b:

A solution of **1** (122 mg, 0.1 mmol) and $\mathbf{L^{Fe1}}$ (66 mg, 0.1 mmol)/$\mathbf{L^{Fe2}}$ (58 mg, 0.1 mmol) in CH_2Cl_2 (20 mL) was added to a solution of AgOTf (52 mg, 0.2 mmol) in 10 mL CH_2Cl_2 via cannula at -78 °C. After vigorous stirring for 2 h in the darkness, the colour of the mixture changed and the reaction mixture was further stirred for 6 h at room temperature. Subsequently, the solution was filtered to remove undissolved compounds. The filtrate was concentrated and the residue was washed with Et_2O (Yield: **3a**, 181 mg, 86%; **3b**, 164 mg, 81%). The suitable crystals **3a** were obtained through the diffusion of ether into the solution of the complexes in dichloromethane after 3 days. *Data for complex* **3a**: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.86-1.59 (m, 80H, Cy), 1.72 (s, 60H, Cp*), 1.74 (s, 24H, β-CH_2), 2.76 (m, 8H, NCH), 2.93 (s, 24H, α-CH_2), 6.76 (s, 4H, C_6H_4), 7.34 (s, 4H, C_6H_4), 7.76 (d, 8H, py), 8.58 (d, 8H, py); IR (KBr disk): ν = 2932, 2854, 1637, 1489, 1263, 1203, 1154, 1061, 1031, 986, 969, 947, 827, 810, 692, 638, 573, 517, 495 cm^{-1}; elemental analysis calcd (%) for $C_{164}H_{220}B_4F_{12}Fe_2Ir_4N_{24}O_{24}S_4$: C 46.99; H 5.29; N 8.02; found C 47.12; H 5.43; N 7.95. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 230 (20.8), 276 (12.7), 450 (6.2). *Data for complex **3b***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.88-1.59 (m, 80H, Cy), 1.73 (s, 60H, Cp*), 2.46 (s, 36H, CH_3), 2.78 (m, 8H, NCH), 6.85 (s, 4H, C_6H_4), 7.34 (s, 4H, C_6H_4), 7.76 (d, 8H, py), 8.59 (d, 8H, py); IR (KBr disk): ν = 2931, 2852, 1638, 1490, 1249, 1203, 1157, 1031, 1017, 935, 807, 690, 574, 517, 500 cm^{-1}; elemental analysis calcd (%) for $C_{152}H_{208}B_4F_{12}Fe_2Ir_4N_{24}O_{24}S_4$: C 45.24; H 5.20; N 8.33; found C 45.39; H 5.24; N 8.21. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 228 (18.1), 273 (10.4), 442 (4.1).

Synthesis of tetra-nuclear complexes 4a, 4b:

A solution of **2** (129 mg, 0.1 mmol) and **L**$^{\mathbf{Fe1}}$ (66 mg, 0.1 mmol)/**L**$^{\mathbf{Fe2}}$ (58 mg, 0.1 mmol) in CH_2Cl_2 (20 mL) was added to a solution of AgOTf (52 mg, 0.2 mmol) in 10 mL CH_2Cl_2 via cannula at -78 °C. After vigorous stirring for 2 h in the darkness, the colour of the mixture changed and the reaction mixture was further stirred for 6 h at room temperature. Subsequently, the solution was filtered to remove undissolved compounds. The filtrate was concentrated and the residue was washed with Et_2O (Yield: **4a**, 191 mg, 88%; **4b**, 178 mg, 85%). The suitable crystals **4a** were obtained through the diffusion of ether into the solution of the complexes in dichloromethane after 4 days in high yield. *Data for complex **4a***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.92-1.74 (m, 80H, Cy), 1.76 (s, 60H, Cp*), 1.86 (s, 24H, β-CH_2), 2.99 (s, 24H, α-CH_2), 2.84 (m, 8H, NCH), 6.80 (d, 4H, C_6H_4), 7.31 (d, 4H, C_6H_4), 7.68 (d, 4H, C_6H_4), 7.74 (d, 4H, C_6H_4), 7.79 (d, 8H, py), 8.65 (d, 8H, py); IR (KBr disk): ν = 2933, 2856, 1636, 1485, 1260, 1203, 1155, 1061, 1031, 986, 969, 947, 830, 810, 692, 638, 573, 517 cm^{-1}; elemental analysis calcd (%) for $C_{176}H_{228}B_4F_{12}Fe_2Ir_4N_{24}O_{24}S_4$: C 48.66; H 5.29; N 7.74; found C 48.91; H 5.39; N 7.54. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 228 (20.4), 274 (24.3), 449 (6.7). *Data for complex **4b***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 0.89-1.73 (m, 80H, Cy), 1.77 (s, 60H, Cp*), 2.50 (s, 36H, CH_3), 2.84 (m, 8H, NCH), 6.78 (d, 4H, C_6H_4), 7.31 (d, 4H, C_6H_4), 7.67 (d, 4H, C_6H_4), 7.74 (d, 4H, C_6H_4), 7.81 (d, 8H, py), 8.66 (d, 8H, py); IR (KBr disk): ν = 2931, 2855, 1637, 1484, 1258, 1203, 1156, 1031, 1015, 935, 832, 805, 689, 638, 573, 517, 500 cm^{-1}; elemental analysis calcd (%) for $C_{164}H_{214}B_4F_{12}Fe_2Ir_4N_{24}O_{24}S_4$: C 47.06;

H 5.15; N 8.03; found C 47.19; H 5.28; N 7.90. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 227 (22.9), 273 (28.9), 443 (7.1).

Synthesis of dinuclear complexes 5a, 5b:

A mixture of [Cp*IrCl(μ-Cl)]$_2$ (80 mg, 0.1 mmol) and $\mathbf{L}^{\mathbf{Fe1}}$ (66 mg, 0.1 mmol)/$\mathbf{L}^{\mathbf{Fe2}}$ (58 mg, 0.1 mmol) in CH_2Cl_2 (20 mL) was stirred at room temperature for 3 h. The filtrate was concentrated and the residue was washed with Et_2O (Yield: **5a**, 136 mg, 94%; **5b**, 130 mg, 95%). The suitable crystals **5a**/**5b** were obtained through the diffusion of ether into the solution of the complexes in dichloromethane after 4 days in high yield. *Data for complex **5a***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 1.56 (s, 30H, Cp*), 1.83 (s, 12H, β-CH_2), 2.91 (s, 12H, α-CH_2), 7.57 (d, 4H, py), 8.85 (d, 4H, py); IR (KBr disk): ν = 2937, 2864, 1638, 1491, 1230, 1201, 1059, 969, 946, 829, 811, 691, 584, 495 cm^{-1}; elemental analysis calcd (%) for $C_{48}H_{62}B_2Cl_4FeIr_2N_8O_6$: C 39.74; H 4.31; N 7.72; found C 39.64; H 4.25; N 7.53. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 229 (16), 283 (9.5), 449 (6.5). *Data for complex **5b***: ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 1.57 (s, 30H, Cp*), 2.42 (s, 18H, CH_3), 7.58 (d, 4H, py), 8.88 (d, 4H, py); IR (KBr disk): ν = 2966, 2917, 1615, 1492, 1236, 1202, 1091, 1013, 934, 838, 806, 689, 595, 500 cm^{-1}; elemental analysis calcd (%) for $C_{42}H_{56}B_2Cl_4FeIr_2N_8O_6$: C 36.75; H 4.11; N 8.16; found C 36.63; H 4.14; N 8.06. UV–Vis (CH_2Cl_2): λ_{max}/nm ($\varepsilon \cdot 10^{-3}$ mol^{-1} l cm^{-1}): 230 (24.8), 278 (15.7), 444 (9.8).

Single-Crystal Structure Determination

Single-crystal XRD data of the compounds was collected on a Bruker APEX DUO diffractometer. Complexes **3a**, **4a**, **5a** and **5b** were collected with Mo-*Kα* radiation (λ = 0.71073 Å) at 173 K. These structures were solved by direct methods, using Fourier techniques, and refined on F^2 by a full-matrix least-squares method. All calculations were carried out with the SHELXTL program.[4]

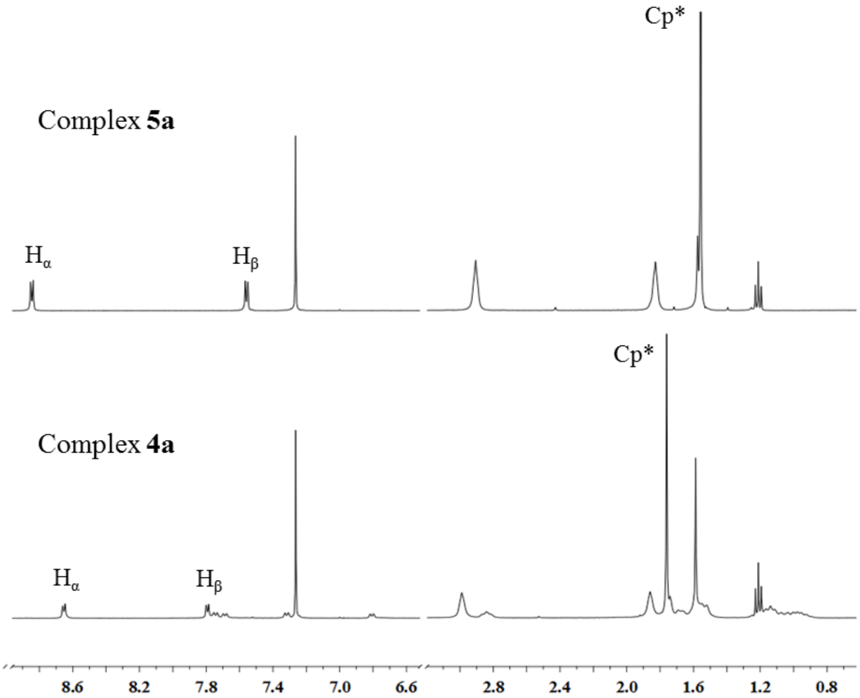

Fig. S1 The ^{1}H NMR spectra (CDCl$_3$) of complexes **4a** (bottom) and **5a** (top).

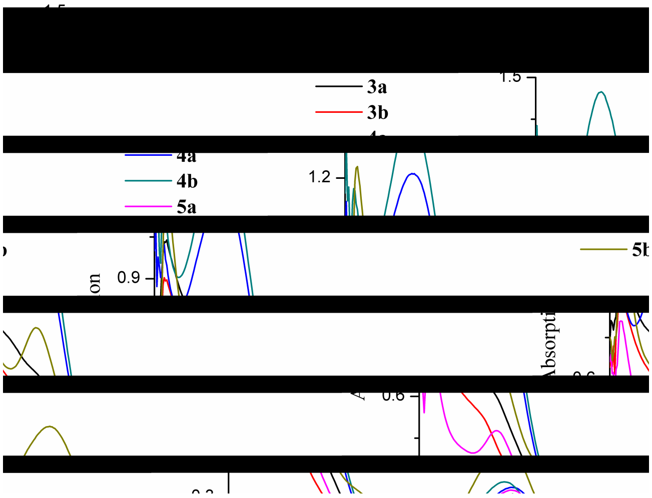
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Fig. S2 UV-vis spectra of complexes **3a**, **3b**, **4a**, **4b**, **5a** and **5b**.

[redacted]

Fig. S3 Cyclic voltammogram for 0.2 mM dichloromethane solutions of complex **4a** on a glassy carbon (GC) electrode at a scan rate of 200 mV s^{-1} vs. SCE (saturated calomel electrode).

Table S1 Selected bond lengths [Å] and angles [°] for compound **3a** (A -x+1,-y,-z+1).

Ir(1)-N(1)	2.136(6)	Ir(1)-N(2)	2.136(5)
Ir(1)-N(11)	2.117(5)	Ir(2)-N(3)	2.105(5)
Ir(2)-N(4)	2.150(5)	Ir(2)-N(12A)	2.112(5)
Fe(1)-N(5)	1.909(5)	Fe(1)-N(6)	1.877(5)
Fe(1)-N(7)	1.914(5)	Fe(1)-N(8)	1.895(5)
Fe(1)-N(9)	1.926(5)	Fe(1)-N(10)	1.901(5)
N(2)-Ir(1)-N(1)	60.9(2)	N(11)-Ir(1)-N(1)	85.6(2)
N(11)-Ir(1)-N(2)	84.4(2)	N(3)-Ir(2)-N(4)	61.2(2)
N(3)-Ir(2)-N(12A)	86.5(2)	N(12A)-Ir(2)-N(4)	83.0(2)
N(8)-Fe(1)-N(5)	78.4(2)	N(6)-Fe(1)-N(9)	77.8(2)
N(10)-Fe(1)-N(7)	77.9(2)		

Table S2 Selected bond lengths [Å] and angles [°] for compound **4a**.

Ir(1)–N(1)	2.119(13)	Ir(1)–N(2)	2.125(13)
Ir(1)–N(9)	2.132(11)	Ir(2)–N(3)	2.084(15)
Ir(2)–N(4)	2.105(14)	Ir(2)–N(17)	2.137(12)
Ir(3)–N(5)	2.086(14)	Ir(3)–N(6)	2.127(14)
Ir(3)–N(10)	2.128(12)	Ir(4)–N(7)	2.137(12)
Ir(4)–N(8)	2.095(11)	Ir(4)–N(18)	2.198(12)
Fe(1)–N(11)	1.872(13)	Fe(1)–N(12)	1.892(12)

Fe(1)–N(13)	1.908(13)	Fe(1)–N(14)	1.890(12)
Fe(1)–N(15)	1.884(13)	Fe(1)–N(16)	1.886(11)
Fe(2)–N(19)	1.936(13)	Fe(2)–N(20)	1.894(13)
Fe(2)–N(21)	1.920(11)	Fe(2)–N(22)	1.913(11)
Fe(2)–N(23)	1.882(13)	Fe(2)–N(24)	1.900(11)
N(1)–Ir(1)–N(2)	62.0(5)	N(1)–Ir(1)–N(9)	88.3(4)
N(2)–Ir(1)–N(9)	87.7(5)	N(3)–Ir(2)–N(4)	59.6(6)
N(3)–Ir(2)–N(17)	87.7(5)	N(4)–Ir(2)–N(17)	87.3(5)
N(5)–Ir(3)–N(6)	64.5(5)	N(5)–Ir(3)–N(10)	86.1(5)
N(6)–Ir(3)–N(10)	88.4(6)	N(8)–Ir(4)–N(7)	59.8(5)
N(8)–Ir(4)–N(18)	86.2(4)	N(7)–Ir(4)–N(18)	89.0(5)
N(11)–Fe(1)–N(14)	79.4(5)	N(15)–Fe(1)–N(12)	78.6(5)
N(16)–Fe(1)–N(13)	77.7(5)	N(22)–Fe(2)–N(19)	77.1(5)
N(23)–Fe(2)–N(20)	78.7(5)	N(24)–Fe(2)–N(21)	79.8(5)

Table S3 The oxidation (E_{ox}) and reduction (E_{red}) potentials (mV) for the complexes.

Complex	Oxidation, E_{ox}			Reduction, E_{red}		
	Ea	*Ec*	*ΔE*	*Ec*	*Ea*	*ΔE*
3a	1360	1130	230	-1180	irreversible	
3b	1350	1150	200	-1190	irreversible	
4a	1290	1120	170	-1110	irreversible	
4b	1350	1140	210	-1200	irreversible	
5a	1440	1130	310	-1180	irreversible	
5b	1470	1150	320	-1190	irreversible	

Table S4 Selected bond lengths [Å] and angles [°] for compound **5a** and **5b**.

	5a	**5b**		**5a**	**5b**
Ir(1)–N(1)	2.107(10)	2.120(4)	Ir(1)–Cl(1)	2.398(3)	2.3956(13)
Ir(1)–Cl(2)	2.396(3)	2.4010(13)	Ir(2)–N(2)	2.132(9)	2.099(4)
Ir(2)–Cl(3)	2.399(3)	2.400(0)	Ir(2)–Cl(4)	2.403(3)	2.3970(12)
Fe(1)–N(3)	1.889(10)	1.904(4)	Fe(1)–N(4)	1.914(10)	1.906(4)
Fe(1)–N(5)	1.916(11)	1.899(4)	Fe(1)–N(6)	1.899(9)	1.916(4)
Fe(1)–N(7)	1.914(10)	1.912(4)	Fe(1)–N(8)	1.943(11)	1.903(4)
N(1)–Ir(1)–Cl(1)	85.8(3)	85.93(12)	N(1)–Ir(1)–Cl(2)	86.2(3)	86.57(12)
Cl(1)–Ir(1)–Cl(2)	89.69(12)	89.13(5)	N(2)–Ir(2)–Cl(3)	85.9(3)	86.461(5)
N(2)–Ir(2)–Cl(4)	86.4(3)	85.63(11)	Cl(3)–Ir(1)–Cl(4)	88.34(12)	90.031(4)
N(3)–Fe(1)–N(4)	77.9(4)	77.64(16)	N(5)–Fe(1)–N(6)	79.0(4)	78.02(16)

N(8)–Fe(1)–N(7)	79.0(4)	78.09(16)

Table S5 Crystallographic data and structure refinement parameters for complex **3a**, **4a**, **5a**, **5b**.

Complex	**3a**	**4a**	**5a**	**5b**
empirical formula	$C_{204}H_{324}B_4Cl_{16}F_{12}Fe_2Ir_4N_{24}O_{36}S_4$	$C_{194}H_{270}B_4Cl_{12}F_{12}Fe_2Ir_4N_{24}O_{27}S_4$	$C_{52}H_{70}B_2Cl_{12}FeIr_2N_8O_6$	$C_{46}H_{64}B_2Cl_{12}FeIr_2N_8O_6$
T[K]	173(2)	173(2)	173(2)	173(2)
Mr	5536.03	5075.72	1790.43	1712.32
crystal size [mm^3]	0.11 × 0.06 × 0.05	0.12 × 0.11 × 0.05	0.20 × 0.14 × 0.07	0.16 × 0.16 × 0.04
crystal system	monoclinic	monoclinic	triclinic	monoclinic
space group	$P2_1/c$	$C2$	P-1	$P2_1/c$
a [Å]	20.2774(19)	58.688(4)	10.557(2)	14.9176(11)
b [Å]	22.715(2)	21.7043(16)	16.941(4)	19.1802(14)
c [Å]	26.453(3)	17.5541(13)	20.082(5)	23.1519(17)
α [°]	90	90	106.900(3)	90
β [°]	108.321(2)	94.1160(10)	90.895(4)	104.4350(10)
γ [°]	90	90	93.715(4)	90
V [Å^3]	11566.2(19)	22303(3)	3427.0(13)	6415.2(8)
Z	2	4	2	4
ρ_{calc} (g cm^{-3})	1.590	1.512	1.735	1.773
μ (Mo$_{K\alpha}$) [mm^{-1}]	2.718	2.763	4.601	4.911
F(000)	5656	10296	1760	3352
θ range [°]	1.058-26.167	0.70-25.01	1.06-25.01	1.410-26.249
limiting indices	$-23 \le h \le 25$	$-69 \le h \le 69$	$-12 \le h \le 12$	$-18 \le h \le 18$
	$-28 \le k \le 25$	$-25 \le k \le 21$	$-15 \le k \le 20$	$-16 \le k \le 23$
	$-32 \le l \le 18$	$-20 \le l \le 20$	$-23 \le l \le 23$	$-28 \le l \le 28$
collected reflns	56194	67613	20021	42071
unique reflns	22990	36117	11925	12888
absorption correction	semiempirical	semiempirical	semiempirical	semiempirical
max./min. transmission	0.745/0.577	0.8742/0.7328	0.7389/0.4597	0.745/0.522
data/restraints/parameters	22990/62/1190	36117/381/2298	11925/109/743	12888/139/753
goodness of fit	1.002	1.092	1.065	1.049
R_1/wR_2 [$I > 2\sigma(I)$][a]	0.0525/0.1539	0.0742/ 0.2010	0.0629/0.1746	0.0315/0.0865
R_1/wR_2 (all data)[a]	0.0738/0.1696	0.0986/ 0.2198	0.0926/0.2108	0.0396/0.0914

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

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