Electronic Supplementary Information for

Ring Rotation of Ferrocene in Interlocked Molecules in Single Crystals

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General Methods. Synthesis and characterization of [*N*-(4-Methylbenzyl)ammoniummethylferrocene]$^{+}(PF_6)^{-}$ was performed on the basis of the method described in the literature.[1] $NaBH_4$, ferrocenecarboxaldehyde, NH_4PF_6, HCl aq. (37 wt%), lithium aluminium hydride, NaOH pellet, hydroxylamine hydrochloride, and DB30C10, DB18C6, CH_2Cl_2, and, diethyl ether were purchased from Sigma-Aldrich. Anhydrous toluene, MeOH, EtOH, and THF were purchased from Sigma-Aldrich in Sure/Seal container and were stored under N_2 gas. 4-Methylphenzylamine and methylamine were purchased from Alfa Aesar. ^{1}H NMR spectra were record on Varian-Unity INOVA-500 spectrometer. The chemical shifts were referenced with respect to $CHCl_3$ (δ_H = 7.26) and CD_2HCN (δ_H = 1.94) for ^{1}H as internal standards. Elemental analysis was carried out with a CHN-O-Rapid elemental analyzer (Foss. Heraeus, Germany). High-resolution field-desorption mass spectra (HRFDMS) were observed by JEOL JMS-T200GC AccuTOF GCx. DSC was conducted using Perkin Elmer Diamond DSC at a scan rate of 5 °C min^{-1}.

A microscope (Olympus, BX51) equipped with a temperature-control stage (TMS 92 and HMS600, both from Linkam Co.) was used to monitor the crystal-to-crystal phase transition. A crystal was placed on the stage, and the stage was heated or cooled a rate of 5 °C min^{-1}. The Imagesource DFK 51AU02 charge-coupled device (CCD) camera with a video rate of 12 fps was used to capture the images and movies of crystals.

Synthesis of [ferrocenylmethyl(methy)ammonium]$^{+}(PF_6)^{-}$. A pipe was connected with two round bottom flasks and one of which is filled with dry toluene (10 ml) solution containing ferrocenecarboxaldehyde (150 mg, 0.7 mmol), and the other flask contains the methylamine solution. The methylamine solution was gradually heated from room temperature to 50 °C, and the methylamine gas can go into the reaction through the pipe as bubbles continuously. Heating for 3 to 4 hours until the bubbling

speed slow down, and keep stirring for 12 more hours. The solvent was removed under reduced pressure to provide methyliminomethylferrocene as a light brown solid without further purification.

The above product was dissolved in dry THF/dry MeOH (15 ml: 25 ml) at room temperature. $NaBH_4$ (26.5 mg, 0.7 mmol) was added to the solution, and stirred for 2 hr at room temperature, a further portion of $NaBH_4$ (26.5 mg, 0.7 mmol) was added to the reaction mixture. Stirring was continued for 12 hr before the product being quenched with 1N HCl aq. (2 ml). The solvent was partitioned between 1N KOH aq. (10 ml) and Dichloromethane (10 ml). The organic extract was dried over $MgSO_4$, filtered and concentrated under reduced pressure to give methylaminomethylferrocene (194.8 mg, 91% yield) as dark brown oil.

A suspension of methylaminomethylferrocene in 6N HCl aq. (6 ml) was stirred for 0.5 hr at room temperature. Before stirring, a sonicator is necessary to make powder well suspended in HCl solution to react efficiently. The residual powder was washed with H_2O (10 ml) then dried in vacuum oven. To a suspension of $[(Fc\text{-}Me)\text{-}H]^+Cl^-$ in acetone (100 ml) was added NH_4PF_6 (207 mg, 0.64 mmol), and the mixture was stirred for 1hr at room temperature. The precipitate was removed by filtration, and the evaporation of the filtrate gave the title compound $[ferrocenylmethyl(methy)ammonium]^+(PF_6)^-$ as a yellow solid. ^1H NMR spectrum (500 MHz, CD_3CN, r.t.): δ_H 2.55 (s, 3H, CH_3), 3.95 (s, 2H, CH_2), 4.21 (s, 5H, C_5H_5), 4.28 (m, 2H, C_5H_4), 4.34 (m, 2H, C_5H_4) (Fig. S1). HRFDMS calcd. for $C_{12}H_{16}NFe$ $[M+H]^+$ = 230.0627, found *m/z* = 230.0633 (error = 2.6 ppm). (Fig. S2) Anal. Calcd. for $C_{12}H_{16}F_6FeNP$: C, 38.43; H, 4.30; N, 3.73. Found: C, 39.03; H, 4.58; N, 3.85.

Preparation of crystals of complex 1. [*N*-(4-Methylbenzyl)ammoniumethyl ferrocene]$^{+}(PF_6)^{-}$ (15.8 mg, 0.034 mmol) and DB30C10 (18.4 mg, 0.034 mmol) were dissolved in 1.8 mL acetone:CH_2Cl_2 = 51:49 vol%. This solution was placed in diethylether vapor atmosphere at room temperature for 2 days to give tangerine-colored crystals of **1** (9.0 mg, 0.009 mmol, 26% yield). ^{1}H NMR spectrum (500 MHz, CD_3CN, r.t.): δ_H 2.33* (s, 3H, Me), 3.57 (m, 8H, OCH_2), 3.63 (m, 8H, OCH_2), 3.76 (m, 8H, OCH_2), 4.04 (s, 2H, NCH_2), 4.07-4.09* (m, 10H, OCH_2, NCH_2), 4.18 (s, 5H, C_5H_5), 4.25-4.36 (s, 4H, C_5H_4), 6.88-6.93 (m, 8H, $C_6H_{4(catechol)}$), 7.21-7.30 (d, 4H, C_6H_4) (Fig. S3). HRFDMS calcd. for $C_{47}H_{62}FeNO_{10}$ $[M+H]^+$ (*m*/*z*): 856.3718, found: 856.3722 (error 0.47 ppm) (Fig. S4).

Preparation of crystals of complexes 2 and 3. [Ferrocenylmethyl(methy) ammonium]$^{+}(PF_6)^{-}$ (7.3 mg, 0.019mmol) and DB18C6 (7.0 mg, 0.019 mmol) were dissolved in 0.6 mL CH_2Cl_2. This solution was placed in a diethylether vapor atmosphere at room temperature for 2 days to give a mixture of tangerine-colored blocks (**2**) and yellowish thin plates (**3**). Total solid yield: 8.8 mg, 0.012 mmol, 63% (calculated for **3**). ^{1}H NMR spectrum for a mixture of **2** and **3** (500 MHz, CD_3CN, r.t.): δ_H 2.56 (m, 3H, CH_3), 3.84 (s, 8H, OCH_2), 3.94 (s, 2H, NCH_2), 4.10 (m, 8H, OCH_2), 4.18 (s, 5H, C_5H_5), 4.27 (m, 2H, C_5H_4), 4.32 (m, 2H, C_5H_4) , 6.90 (m, 4H, C_6H_4) (Fig. S5). The molar ratio between **2** and **3** was estimated to be **2**:**3** = 15:85 from peaks at 5.44 ppm for CH_2Cl_2 and 2.56 ppm for CH_3 in the ^{1}H NMR spectrum. HRFDMS calcd. for a mixture of **2** and **3** for $C_{32}H_{40}NO_6Fe$ $[M+H]^+$ = 590.2200, found *m/z* = 590.2213 (error = 2.2 ppm) (Fig. S6). Anal. Calcd. for $C_{34}H_{42}NO_6F_6FePCl_2$ (**3**): C, 48.31; H, 5.16; N, 1.71. Found: C, 48.33; H, 5.10; N, 1.78.

Single-crystal X-ray crystallography. Single-crystal X-ray crystallography was performed for the pseudorotaxane crystals using Rigaku XtaLAB Synergy-DW system

and Bruker Single Crystal D8 Venture diffractometer. CCDC 2033783-2033808 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

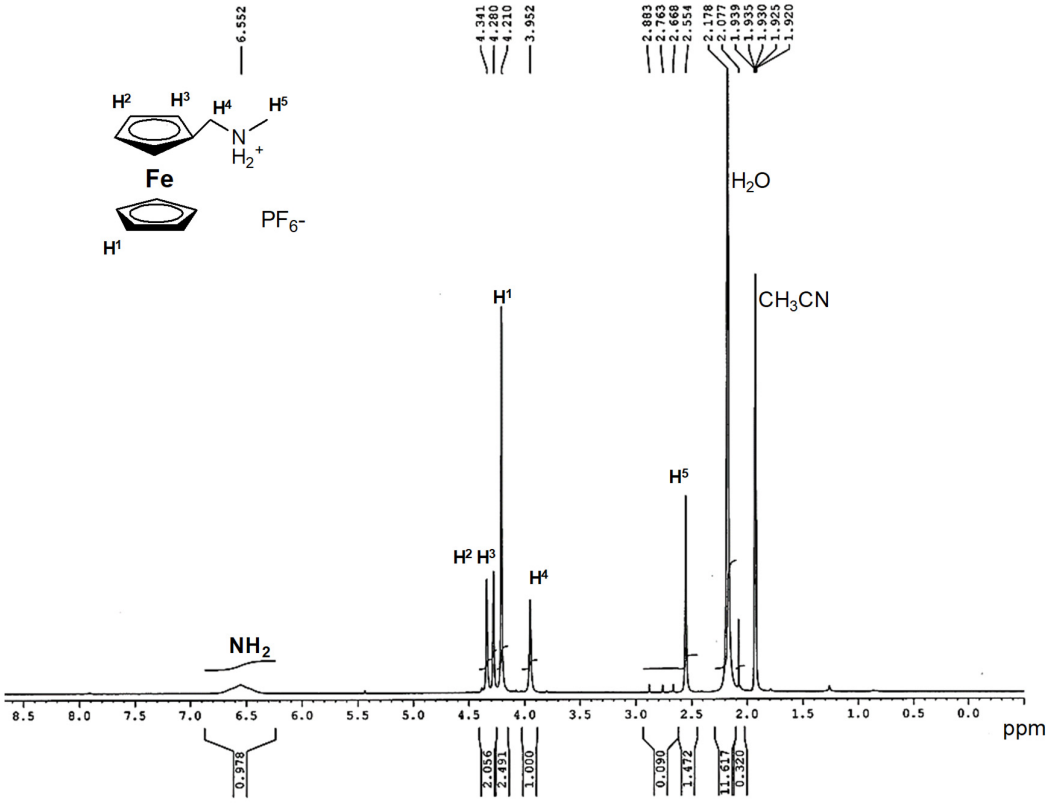

Figure S1 ^{1}H NMR spectrum of [ferrocenylmethyl(methy)ammonium]$^{+}$(PF$_6$)$^{-}$ in CD$_3$CN.

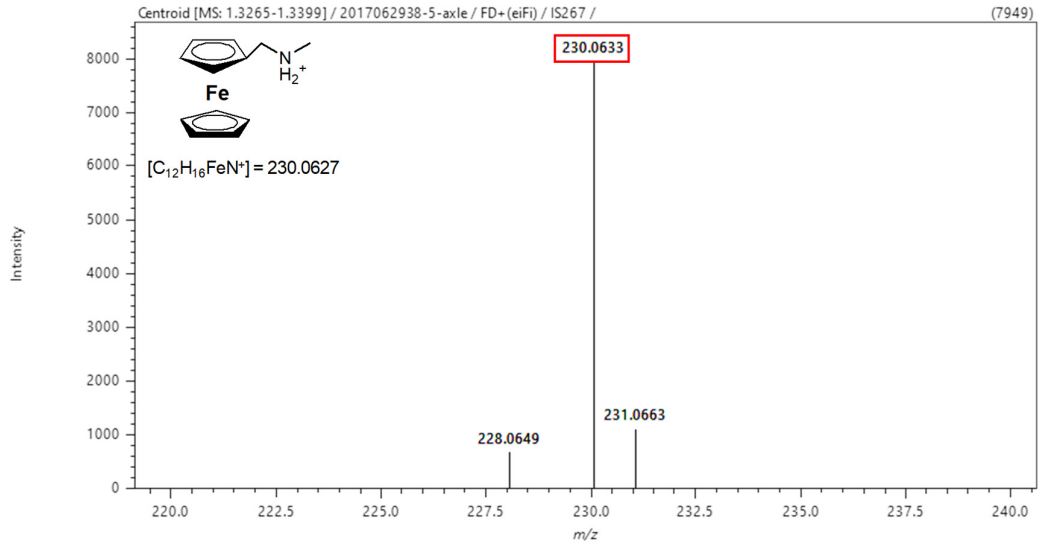

Figure S2 High-resolution field desorption mass spectrum (HRFDMS) of [Ferrocenylmethyl(methy)ammonium]$^{+}$(PF$_6$)$^{-}$. The main peak at *m*/*z* = 230.0633 corresponds to [C$_{12}$H$_{16}$FeN^{+}] = 230.0627 (error = 2.6 ppm).

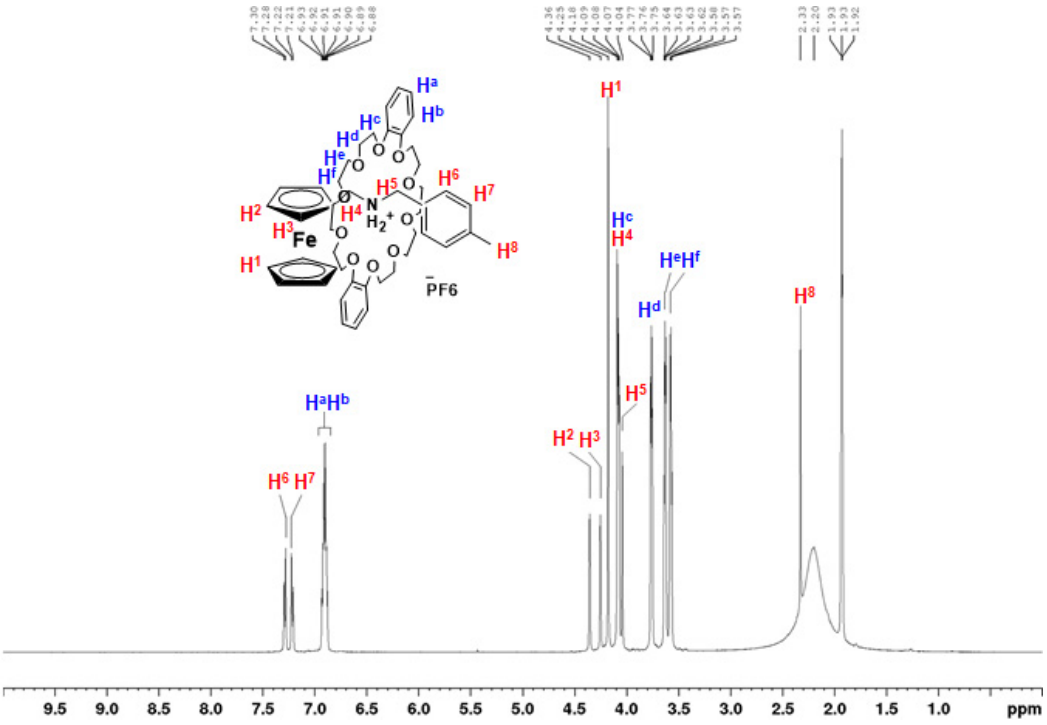

Figure S3 ^{1}H NMR spectrum of complex **1** in CD_3CN.

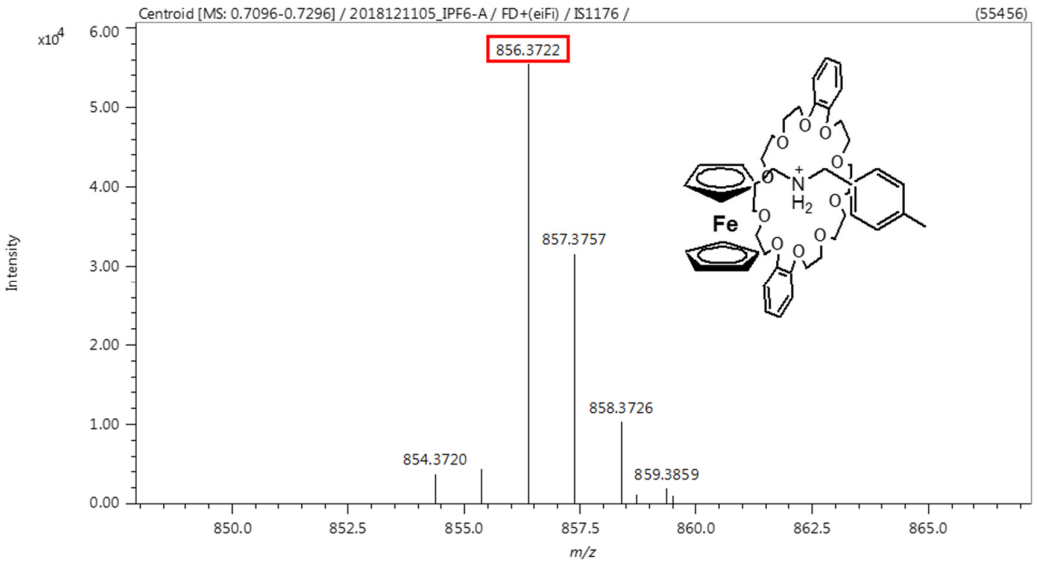

Figure S4 High-resolution field desorption mass spectrum (FDMS) of complex **1**. The main peak at *m*/*z* = 856.3722 corresponds to [$C_{47}H_{62}FeNO_{10}^{+}$] = 856.3718 (error = 0.47 ppm).

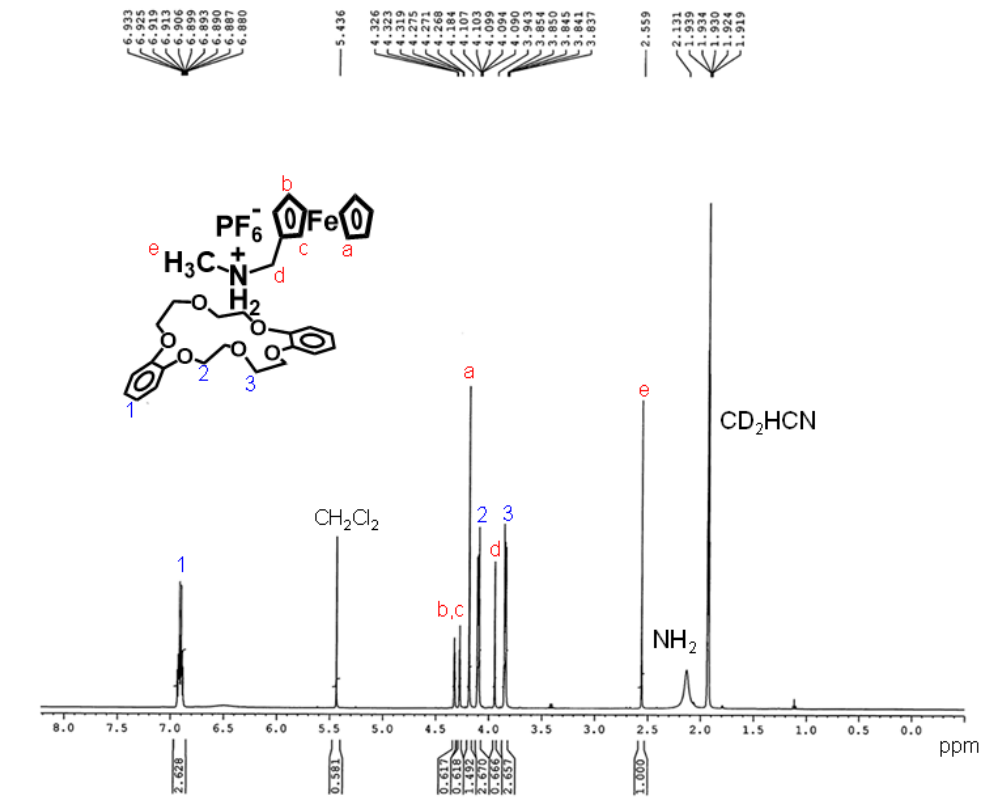

Figure S5 ^{1}H NMR spectrum of a mixture of complexes **2** and **3** in CD_3CN.

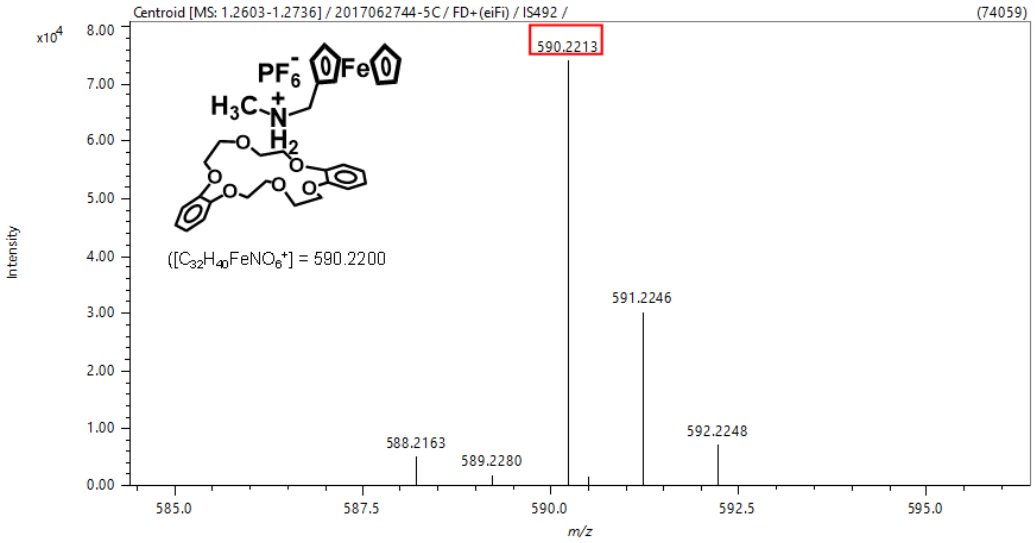

Figure S6 High-resolution field desorption mass spectrum (HRFDMS) of a mixture of complexes **2** and **3**. The main peak at *m*/*z* = 590.2213 corresponds to $[C_{32}H_{40}FeNO_6^+]$ = 590.2200 (error = 2.2 ppm).

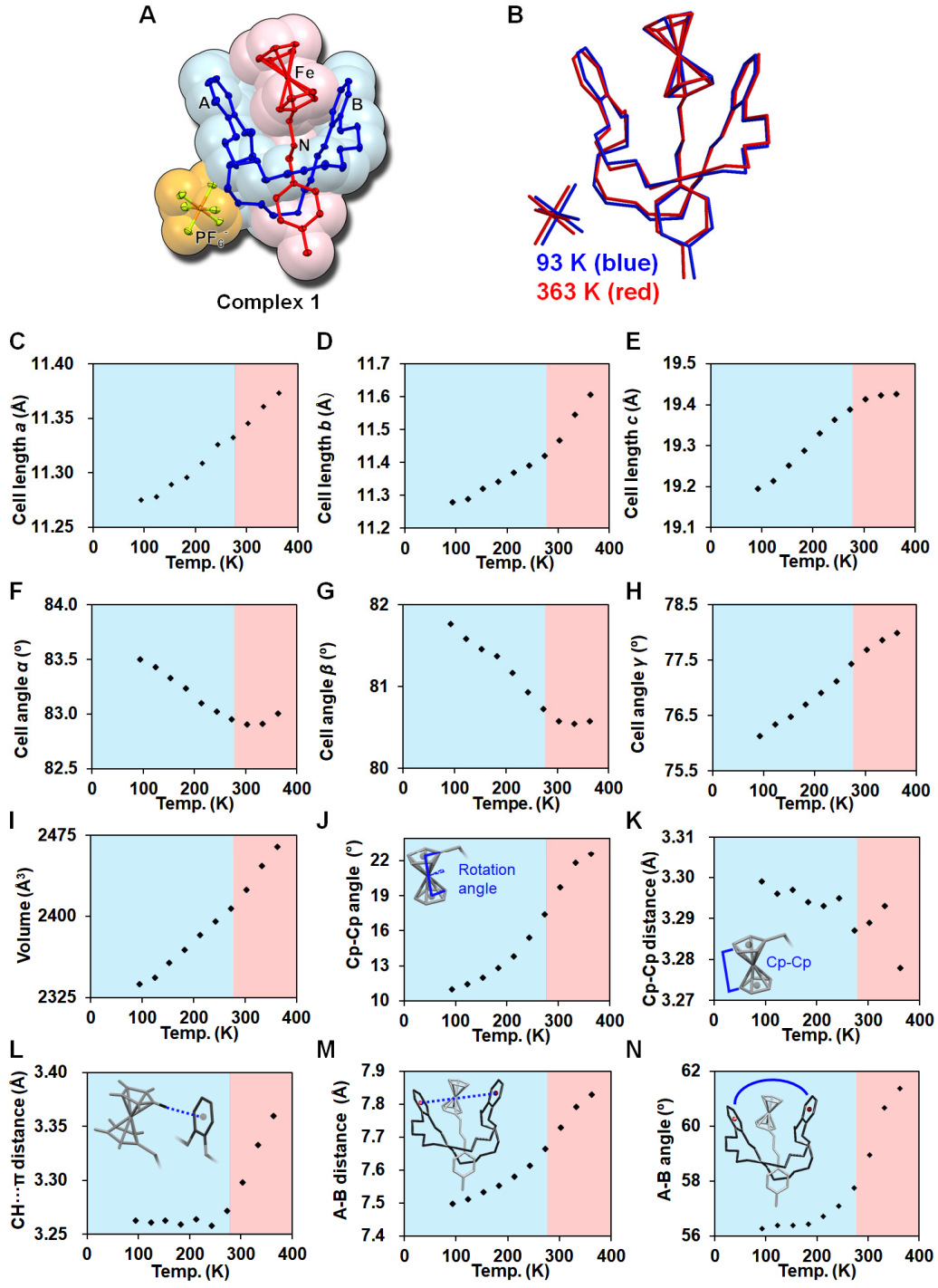

Figure S7 (A) Molecular structure of **1** at 93 K. (B) Overlay of the molecular structures of **1**. Temperature dependence of (C-I) unit cell parameters and volume, (J) internal rotation-angle of Cp rings, (K) distance between Cp rings, (L) C–H···π distance, (M) distance between catechols A and B, and (N) plane angle between catechols A and B.

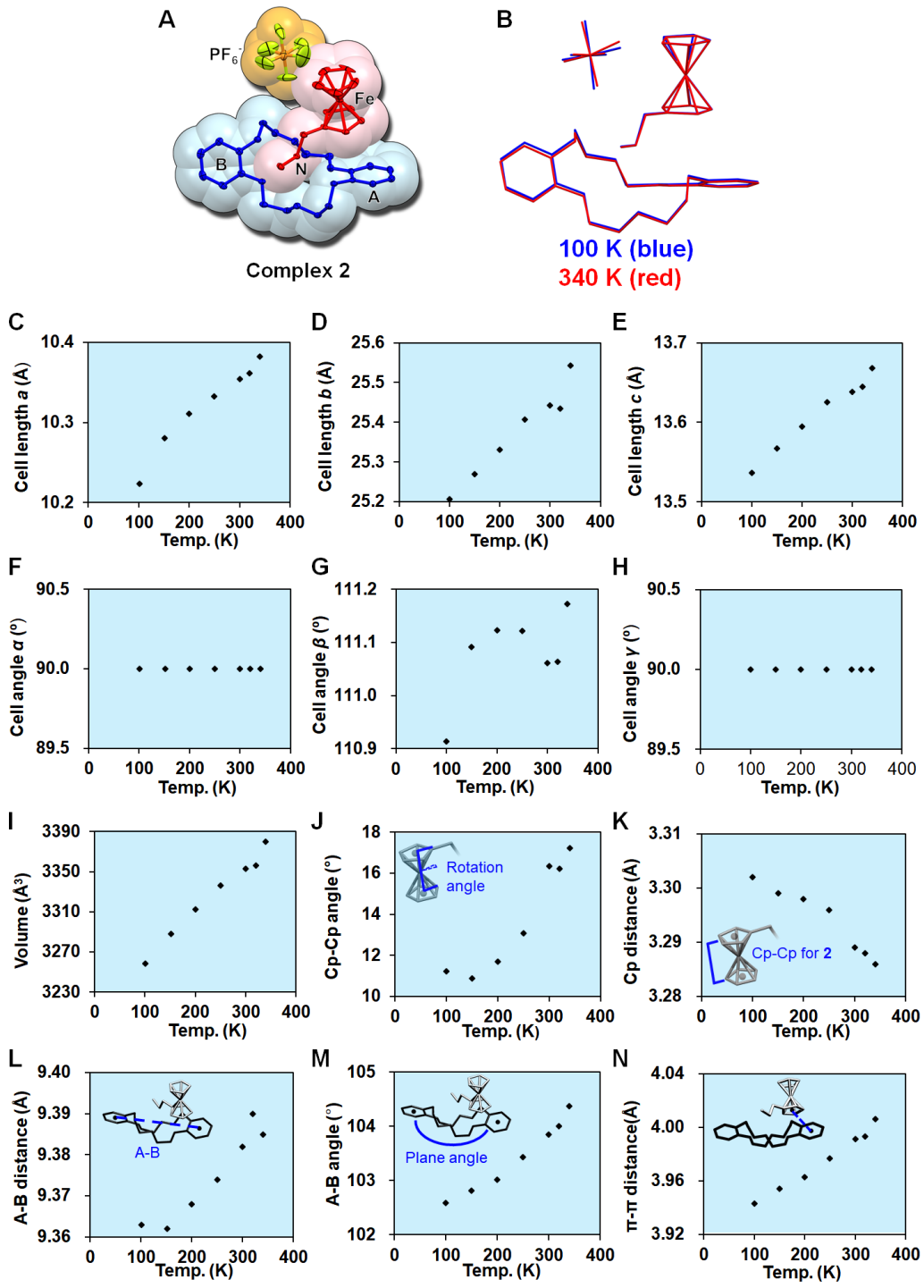

Figure S8 (A) Molecular structure of **2** at 100 K. (B) Overlay of the molecular structures of **2**. Temperature dependence of (C-I) unit cell parameters and volume, (J) internal rotation-angle of Cp rings, (K) distance between Cp rings, (L) distance between catechols A and B, (M) plane angle between catechols A and B, and (N) π -π distance.

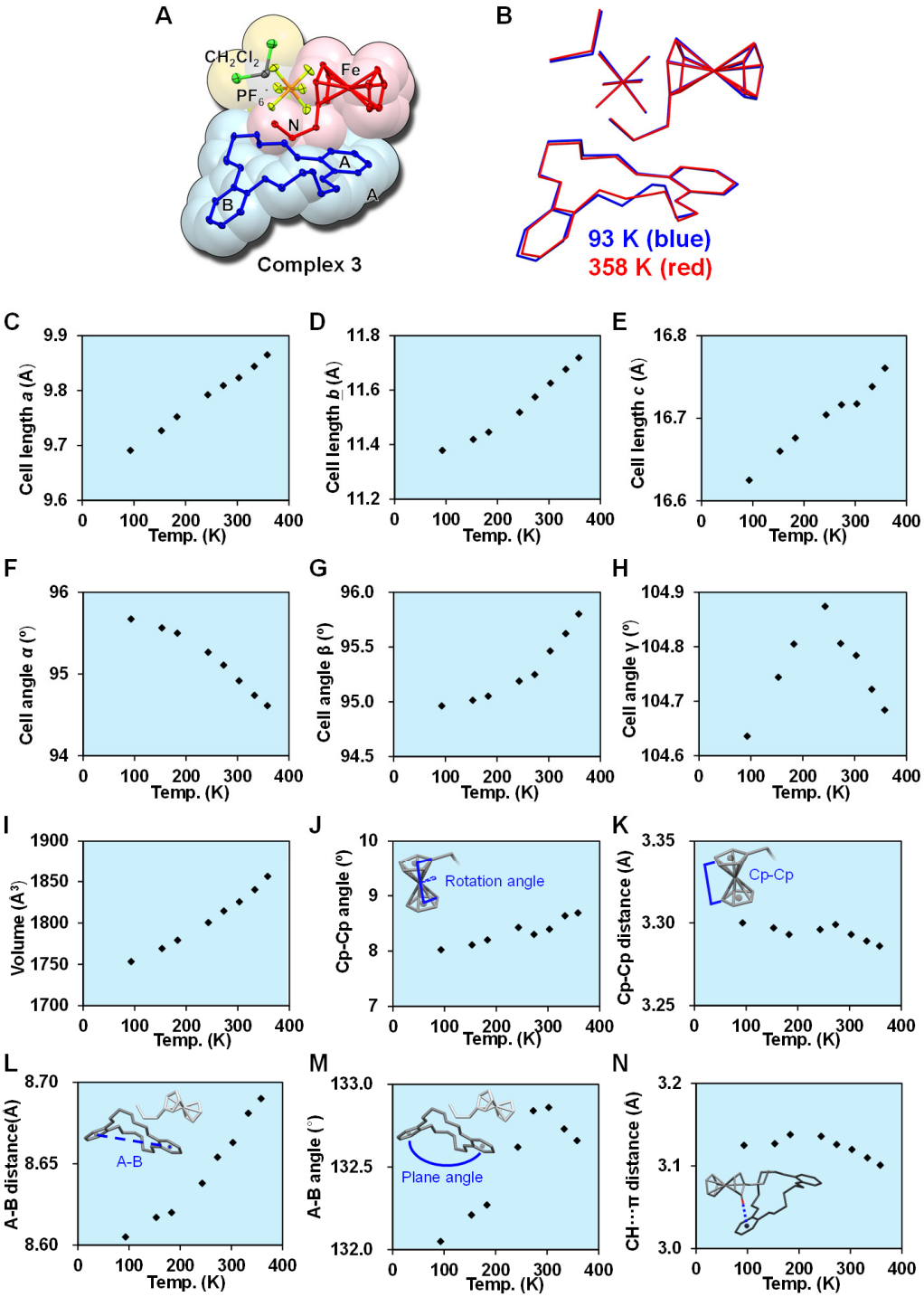

Figure S9 (A) Molecular structure of **3** at 93 K. (B) Overlay of the molecular structures of **3**. Temperature dependence of (C-I) unit cell parameters and volume, (J) internal rotation-angle of Cp rings, (K) distance between Cp rings, (L) distance between catechols A and B, (M) plane angle between catechols A and B, and (N) C–H···π distance.

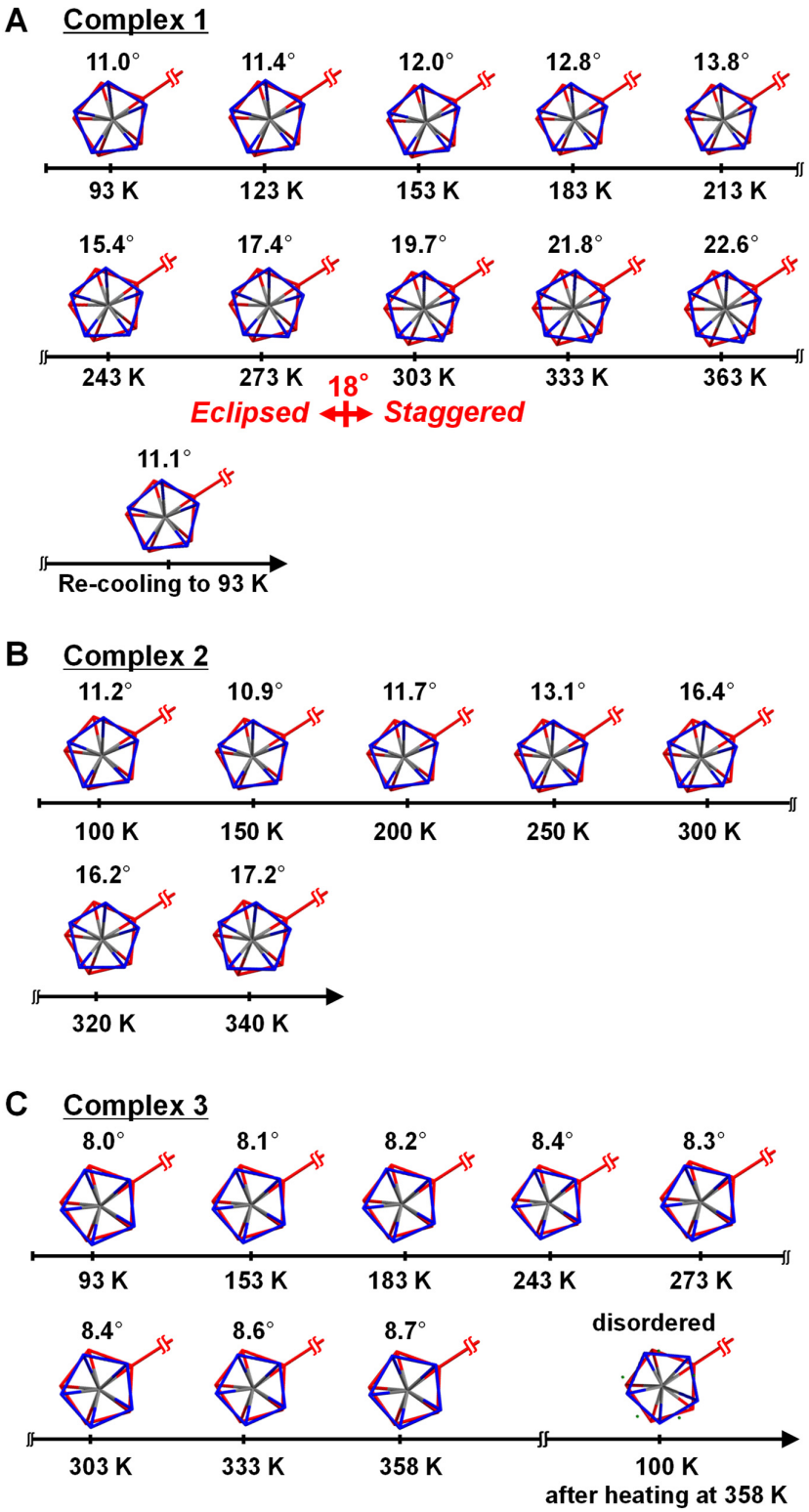

Figure S10 Top view of ferrocenyl group of (A) **1**, (B) **2**, and (C) **3** at various temperatures.

Table S1 Crystal data and details structure refinement of complex **1** at various temperatures.

	a	**b**	**c**	**d**
Temp./K	93	123	153	183
Formula	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$
molecular weight	1001.79	1001.79	1001.79	1001.79
crystal system	Triclinic	Triclinic	Triclinic	Triclinic
space group	P1bar	P1bar	P1bar	P1bar
a/Å	11.2754(3)	11.2781(2)	11.2892(2)	11.2958(3)
b/Å	11.2786(2)	11.2893(2)	11.3208(3)	11.3414(3)
c/Å	19.1955(4)	19.2130(3)	19.2519(4)	19.2872(5)
α/deg	83.501(2)	83.432(1)	83.330(2)	83.238(2)
β/deg	81.762(2)	81.586(1)	81.460(2)	81.374(2)
γ/deg	76.134(2)	76.335(1)	76.478(2)	76.703(2)
V/Å^3	2337.60(9)	2343.29(7)	2357.22(9)	2368.53(11)
Z	2	2	2	2
μ(MoKα)/mm^{-1}	3.623	3.614	3.593	3.576
F(000)	1052	1052	1054	1052
D/g cm^{-3}	1.423	1.420	1.411	1.405
crystal size/mm^3	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06
reflections collected	31660	31533	32523	31546
independent reflections	8923	8917	8895	8932
R	0.0323	0.0310	0.0354	0.0370
*R*w	0.0837	0.0872	0.0988	0.0990
GOF	1.076	0.871	0.8828	1.075

Table S1 Continued

	e	**f**	**g**	**h**
Temp./K	213	243	273	303
Formula	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$
molecular weight	1001.79	1001.79	1001.79	1001.79
crystal system	Triclinic	Triclinic	Triclinic	Triclinic
space group	P1bar	P1bar	P1bar	P1bar
a/Å	11.3088(3)	11.3259(3)	11.3327(2)	11.3456(3)
b/Å	11.3689(3)	11.3917(3)	11.4193(2)	11.4668(3)
c/Å	19.3307(4)	19.3628(5)	19.3877(4)	19.4140(5)
α/deg	83.100(2)	83.024(2)	82.951(2)	82.907(2)
β/deg	1.166(2)	80.932(2)	80.729(2)	80.580(2)
γ/deg	76.913(2)	77.121(2)	77.436(2)	77.690(2)
V/Å^3	2382.63(10)	2395.24(11)	2406.89(8)	2424.07(11)
Z	2	2	2	2
μ(MoKα)/mm^{-1}	3.555	3.536	3.519	3.494
F(000)	1052	1052	1054	1054
D/g cm^{-3}	1.396	1.389	1.382	1.372
crystal size/mm^3	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06
reflections collected	33397	33262	26322	32171
independent reflections	33397	8740	8255	8295
R	0.0408	0.0439	0.0597	0.0572
*R*w	0.1070	0.1185	0.1678	0.1736
GOF	1.029	1.058	1.066	1.0292

Table S1 Continued

	i	j	k
Temp./K	333	363	93 (second cooling)
Formula	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$	$C_{47}H_{62}F_6FeNO_{10}P$
molecular weight	1001.79	1001.79	1001.79
crystal system	Triclinic	Triclinic	Triclinic
space group	P1bar	P1bar	P1bar
a/Å	11.3611(2)	11.3733(2)	11.2780(2)
b/Å	11.5447(3)	11.6068(3)	11.2872(3)
c/Å	19.4228(5)	19.4270(5)	19.2076(4)
α/deg	82.910(2)	83.003(2)	83.456(2)
β/deg	80.543(2)	80.579(2)	81.762(2)
γ/deg	77.861(2)	77.986(2)	76.218(2)
V/Å^3	2446.24(10)	2464.26(10)	2342.05(9)
Z	2	2	2
μ(MoKα)/mm^{-1}	3.462	3.437	3.616
F(000)	1052	1052	1052
D/g cm^{-3}	1.360	1.350	1.421
crystal size/mm^3	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06	0.42 x 0.24 x 0.06
reflections collected	25365	26909	32572
independent reflections	7518	7270	8957
R	0.0658	0.0727	0.0334
*R*w	0.2053	0.2296	0.0881
GOF	1.0375	1.0302	1.064

Table S2 Intra-/intermolecular distances and angles of complex **1** before and after heating.

	1 (a, first cooling**)**	**1 (k**, second cooling**)**
Temp./K	93	93
internal rotation-angle of Cp rings/°	11.0	11.1
distance between Cp rings/Å	3.30	3.30
C–H···π distance/Å	3.26	3.27
distance between catechols A and B/Å	7.50	7.51
plane angle between catechols A and B/°	56.3	56.3

Table S3 Crystal data and details structure refinement of complex **2** at various temperatures.

	a	**b**	**c**	**d**
Temp./K	100	150	200	250
Formula	$C_{32}H_{40}F_6FeNO_6P$	$C_{32}H_{40}F_6FeNO_6P$	$C_{32}H_{40}F_6FeNO_6P$	$C_{32}H_{40}F_6FeNO_6P$
molecular weight	735.47	735.47	735.47	735.47
crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
space group	P 21/n	P 21/n	P 21/n	P 21/n
a/Å	10.2236(7)	10.2800(6)	10.3108(6)	10.3328(7)
b/Å	25.2061(18)	25.2690(16)	25.3315(17)	25.4063(17)
c/Å	13.5364(10)	13.5675(8)	13.5949(9)	13.6255(9)
α/deg	90	90	90	90
β/deg	110.914(2)	111.0919(13)	111.1227(13)	111.1223(14)
γ/deg	90	90	90	90
V/Å^3	3258.5(4)	3288.2(3)	3312.2(4)	3336.6(4)
Z	4	4	4	4
μ(MoKα)/mm^{-1}	0.591	0.586	0.582	0.577
F(000)	1528	1528	1528	1528
D/g cm^{-3}	1.499	1.486	1.475	1.464
crystal size/mm^3	0.25 x 0.20 x 0.12	0.25 x 0.20 x 0.12	0.25 x 0.20 x 0.12	0.25 x 0.20 x 0.12
reflections collected	28089	28296	28561	28777
independent reflections	6894	6940	6990	7047
R	0.0712	0.0607	0.0640	0.0668
*R*w	0.1930	0.1737	0.1905	0.2085
GOF	1.058	1.063	1.052	1.067

Table S3 Continued

	e	f	g
Temp./K	300	320	340
Formula	$C_{32}H_{40}F_6FeNO_6P$	$C_{32}H_{40}F_6FeNO_6P$	$C_{32}H_{40}F_6FeNO_6P$
molecular weight	735.47	735.47	735.47
crystal system	Monoclinic	Monoclinic	Monoclinic
space group	P 21/n	P 21/n	P 21/n
a/Å	10.354(3)	10.361(4)	10.3821(19)
b/Å	25.443(7)	25.435(10)	25.543(5)
c/Å	13.638(4)	13.645(6)	13.668(3)
α/deg	90	90	90
β/deg	111.061(5)	111.064(8)	111.172(4)
γ/deg	90	90	90
V/Å^3	3353.0(15)	3356(2)	3380.0(12)
Z	4	4	4
μ(MoKα)/mm^{-1}	0.575	0.574	0.570
F(000)	1528	1528	1528
D/g cm^{-3}	1.457	1.456	1.445
crystal size/mm^3	0.25 x 0.20 x 0.12	0.25 x 0.20 x 0.12	0.25 x 0.20 x 0.12
reflections collected	28918	28457	28760
independent reflections	7060	7082	7104
R	0.0712	0.0804	0.0866
*R*w	0.2328	0.3065	0.3215
GOF	1.071	0.977	1.009

Table S4 Crystal data and details structure refinement of complex **3** at various temperatures.

	a	**b**	**c**	**d**
Temp./K	93	153	183	243
Formula	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$
molecular weight	820.39	820.42	820.42	820.42
crystal system	triclinic	triclinic	triclinic	triclinic
space group	P1bar	P1bar	P1bar	P1bar
a/Å	9.6910(3)	9.7269(3)	9.7522(2)	9.7922(2)
b/Å	11.3787(3)	11.4193(3)	11.4459(2)	11.5185(3)
c/Å	16.6251(4)	16.6601(4)	16.6761(3)	16.7041(4)
α/deg	95.673(2)	95.565(2)	95.500(2)	95.266(2)
β/deg	94.963(2)	95.014(2)	95.052(2)	95.190(2)
γ/deg	104.636(2)	104.744(2)	104.805(2)	104.874(2)
V/Å^3	1753.22(8)	1769.03(9)	1779.05(6)	1800.49(8)
Z	2	2	2	2
μ(MoKα)/mm^{-1}	5.971	5.917	5.884	5.814
F(000)	848.0	850.6	850.6	850.6
D/g cm^{-3}	1.554	1.540	1.531	1.513
crystal size/mm^3	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37
reflections collected	21543	23236	22171	21920
independent reflections	6566	6505	6400v	6290
R	0.0596	0.0511	0.0617	0.0660
*R*w	0.1709	0.1469	0.1755	0.1893
GOF	1.041	1.046	1.006	1.019

Table S4 Continued

	e	f	g	h
Temp./K	273	303	333	358
Formula	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$	$C_{33}H_{42}Cl_2F_6FeNO_6P$
molecular weight	820.42	820.39	820.42	820.42
crystal system	triclinic	triclinic	triclinic	triclinic
space group	P1bar	P1bar	P1bar	P1bar
a/Å	9.8091(3)	9.8233(3)	9.8440(3)	9.8649(3)
b/Å	11.5747(4)	11.6256(3)	11.6767(3)	11.7188(4)
c/Å	16.7136(5)	16.7174(5)	16.7383(5)	16.7606(5)
α/deg	16.7136(5)	94.917(2)	94.740(2)	94.613(3)
β/deg	95.249(2)	95.464(2)	95.623(2)	95.802(3)
γ/deg	104.806(3)	104.784(3)	104.722(2)	104.684(3)
V/Å^3	1814.42(10)	1825.52(9)	1840.28(9)	1853.35(11)
Z	2	2	2	2
μ(MoKα)/mm^{-1}	5.769	5.734	5.665	5.648
F(000)	850.6	848.0	850.6	850.6
D/g cm^{-3}	1.502	1.492	1.480	1.470
crystal size/mm^3	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37	0.15 x 0.35 x 0.37
reflections collected	23738	18742	18575	18185
independent reflections	6429	5899	5571	5174
R	0.0544	0.0546	0.0708	0.0810
*R*w	0.1535	0.1587	0.2253	0.2553
GOF	1.006	1.085	1.007	1.002

Table S5 Crystal data and details of structure refinement of complex **3** after heating at 358 K.

	3 (after heating at 358 K)
Temp./K	100
Formula	$C_{32}H_{40}F_6FeNO_6P$
molecular weight	735.47
crystal system	Monoclinic
space group	P21/n
a/Å	10.2753(9)
b/Å	25,224(2)
c/Å	13.5486(11)
α/deg	90
β/deg	111.171(2)
γ/deg	90
V/Å^3	3274.6(5)
Z	4
μ (MoKα)/cm^{-1}	5.88
F(000)	1528
D/g cm^{-3}	1.492
crystal size/mm	0.55 x 0.19 x 0.14
Unique reflections	51880
Used reflections [I>2.0σ (I)]	5801
R	0.0767
*R*w	0.1887
GOF	1.022

Reference

1. M. Horie, Y. Suzaki, D. Hashizume, T. Abe, T. Wu, T. Sassa, T. Hosokai, K. Osakada, *J. Am. Chem. Soc.* **2012**, *134*, 17932-17944.