Supporting information

Ligand redistribution reactions in the syntheses of heterobimetallic rare-earth metal complexes with Co(II) and Fe(II)

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1. X-ray Crystallography

Diffraction was performed on a Bruker SMART APEX II CCD area detector diffractometer using graphite-monochromated Mo *Kα* radiation (λ = 0.71073 Å) and Bruker Platon II area detector diffractometer using graphite-monochromated Mo *Kα* radiation (λ = 0.71073 Å) for complexes, φ and ω scan technique. An empirical absorption correction was applied using the SADABS program.[1] All structures were solved by direct methods, completed by subsequent difference Fourier syntheses, and refined anisotropically for all non-hydrogen atoms by full-matrix least-squares calculations based on F^2 using the SHELXTL program package[2] and Olex2 program.[3] The hydrogen atom coordinates were calculated with SHELXTL by using an appropriate riding model with varied thermal parameters. All crystal structural pictures drawn by Olex 2 program.[3] Crystal parameters and refinement results are given in Table S1.

Table S1. Crystallographic and Refinement Data for **2, 3, 4** and **6**

Compound	**2**	**3**	**4**	**6**
Formula	$C_{108}H_{140}Cl_6Co_2N_4O_5P_4Y_2$	$C_{86}H_{115}ClCo_2KN_3O_8P_3$	$C_{70}H_{83}ClFe_2N_3OP_3$	$C_{76}H_{82}ClFe_2N_4O_3P_3$
Formula weight	2206.49	1604.12	1222.45	1339.51
Cryst size, mm	0.22 × 0.21 × 0.2	0.21 × 0.2 × 0.2	0.15 × 0.13 × 0.12	0.22 × 0.21 × 0.21
Crystal system	Trigonal	Monoclinic	Trigonal	Monoclinic
Space group	*P*-3	$P2_1/c$	*P*-1	$P2_1/n$
Temperature (K)	273(2)	273(2)	273(2)	273(2)
a (Å)	21.2208(10)	15.202(3)	10.3337(4)	17.770(2)

b (Å)	21.2208(10)	22.359(4)	12.3700(6)	12.4984(12)
c (Å)	20.8628(12)	27.450(5)	24.6238(12)	33.750(4)
α (°)	90	90	94.167(2)	90
β (°)	90	99.91(3)	97.369(2)	96.105(3)
γ (°)	120	90	92.465(2)	90
V (Å^3)	8136.3(9)	9191(3)	3109.0(2)	7453.1(14)
Z	3	4	2	4
D_{calcd}, g·cm^{-3}	1.351	1.159	1.306	1.194
F(000)	3450	3404	1292	2816
μ, mm^{-1}	1.619	0.538	0.633	0.536
θ range /°	2.929-27.754	2.835-25.058	2.895-27.629	2.783-28.516
Tot., uniq. data	476259, 12585	169853, 16144	117552, 14060	297703, 18089
R(int)	0.074	0.0677	0.0905	0.0829
Observed data [$I > 2\sigma(I)$]	9468	12743	9656	11060
Data/restrnts/params	12585/872/848	16144/2456/1558	14060/500/849	18089/1439/1186
R_1[a], wR_2[b] ($I > 2\sigma(I)$)	0.0519, 0.1328	0.0738, 0.2483	0.0766, 0.1484	0.0737, 0.1966
$R1$, wR_2 (all data)	0.0797, 0.1610	0.0946, 0.2248	0.1207, 0.1753	0.1280, 0.2369
GOF	1.145	1.075	1.062	1.079
$\Delta\rho_{max, min}$, e·Å^{-3}	0.945, -0.495	1.319, -0.721	0.866, -0.653	0.470, -0.492

[a] $R_1 = \Sigma||F_0|-|F_c||/\Sigma|F_0|$, [b] $wR_2 = \Sigma[w(F_0^2-F_c^2)^2]/\Sigma[w(F_0^2)^2]^{1/2}$.

2. References

[1] G. M. Sheldrick, SADABS: Program for Empirical Absorption Correction of Area Detector Data; University of Göttingen: Germany, 1996.

[2] G. M. Sheldrick, SHELXT-Integrated space-group and crystal structure determination, *Acta Crystallogr*., *Sect*. *C*., 2015, **7**, 3–8.

[3] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. J. Puschmann, *J*. *Appl*. *Crystallogr*., 2009, **42**, 339–341.