Electronic Supplementary Material (ESI) for Dalton Transactions

Supporting Information

Chiral Bis(phthalocyaninato) Yttrium Double-Decker Complexes. Synthesis, Structure, Spectroscopy, and Electrochemistry

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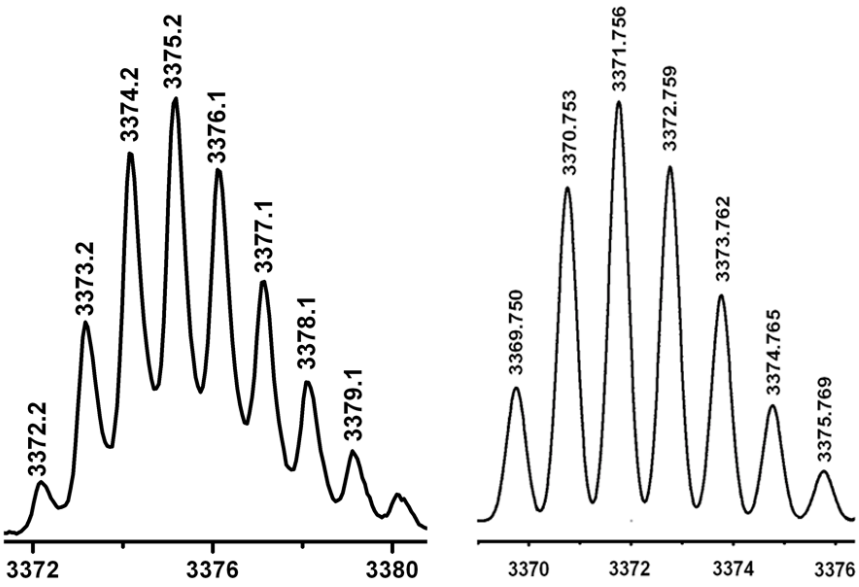

Fig. S1. Observed (left) and simulated (right) MALDI-TOF mass spectra of **1**.

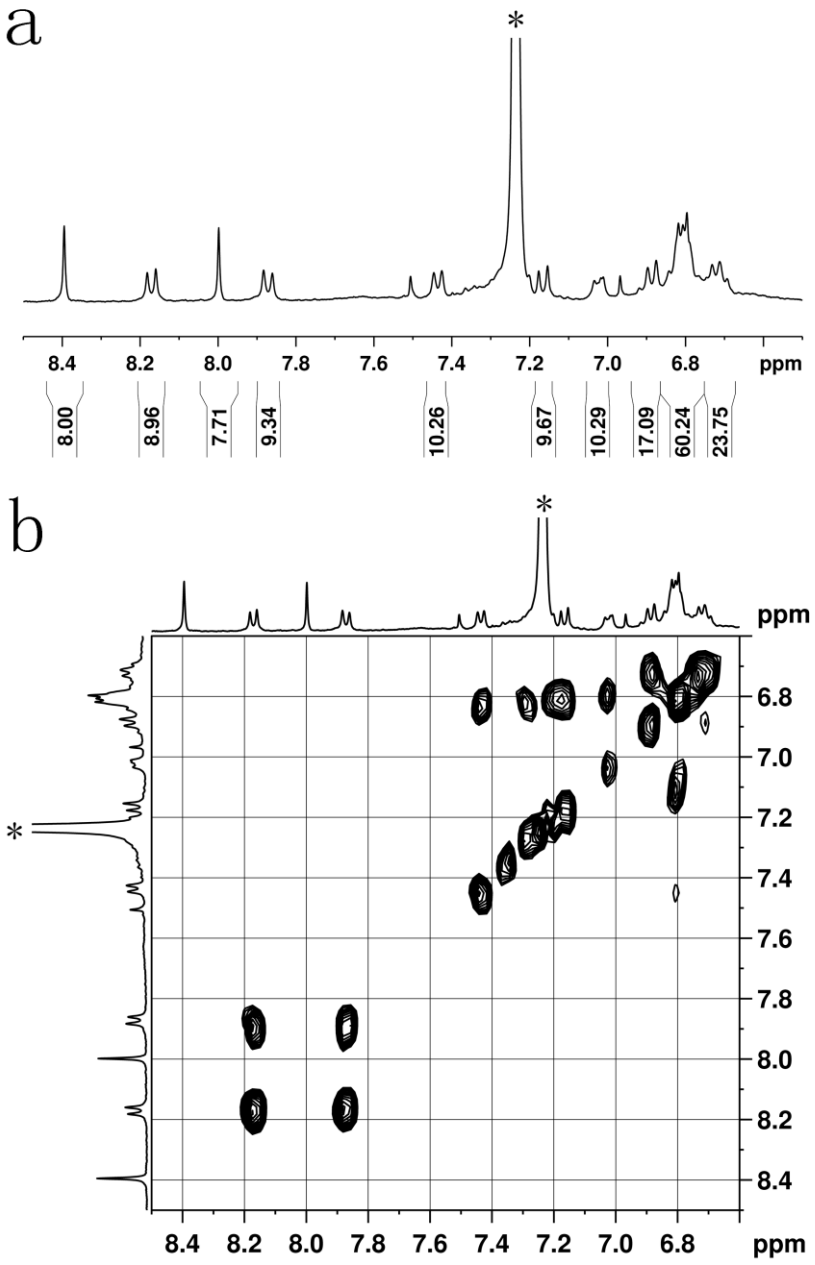

Fig. S2. (a) ^{1}H NMR and (b) ^{1}H-^{1}H COSY for **1** recorded in $CDCl_3$ in the presence of hydrazine hydrate. * denotes a solvent impurity.

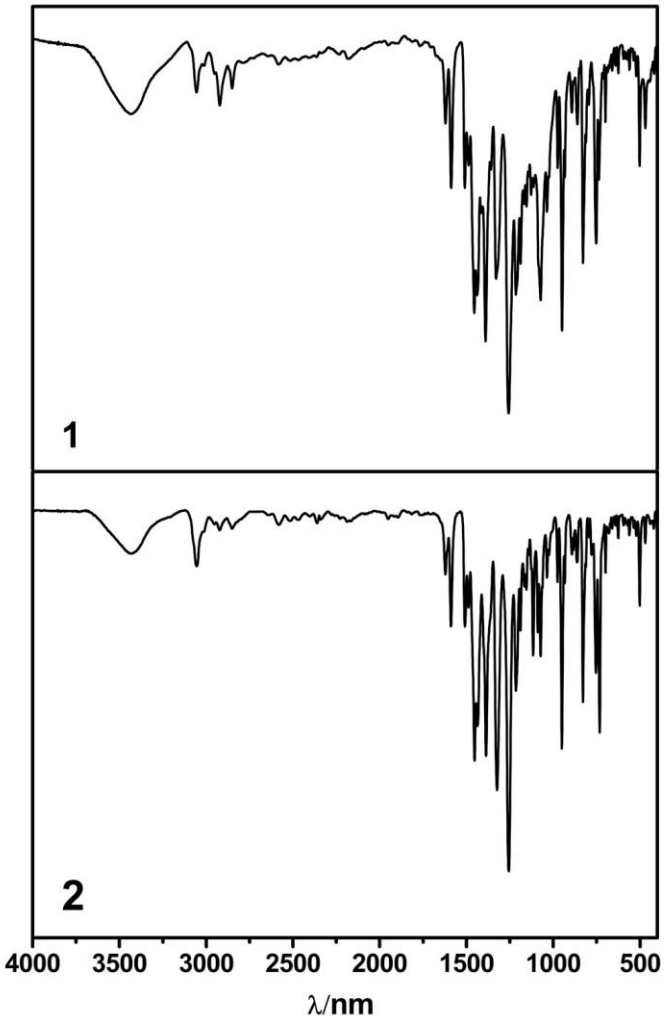

Fig. S3. IR spectra of **1** and **2** in the region of 400-4000 cm^{-1} recorded in KBr pellets.

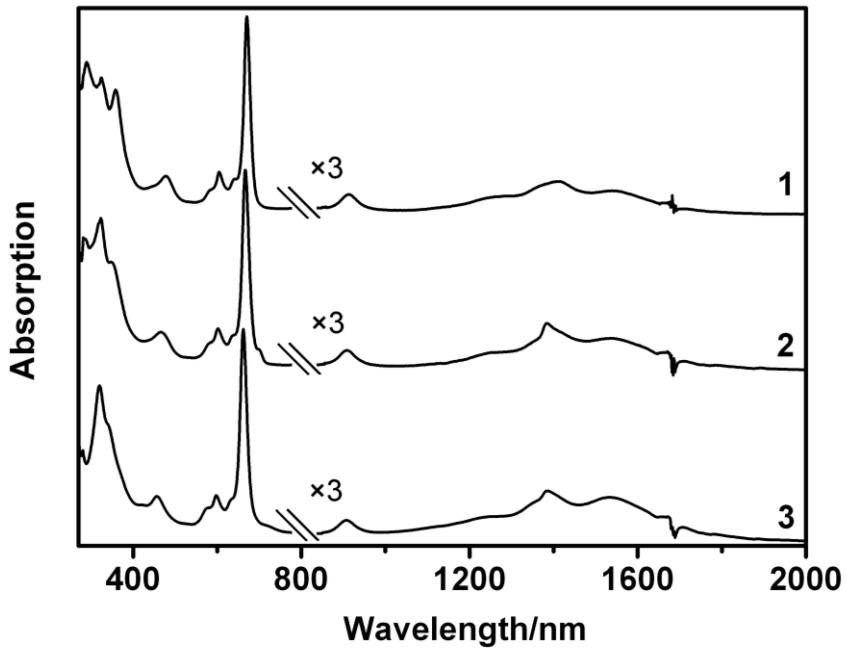

Fig. S4. Electronic absorption spectra of **1**, **2**, and $Y(Pc)_2$ in toluene.

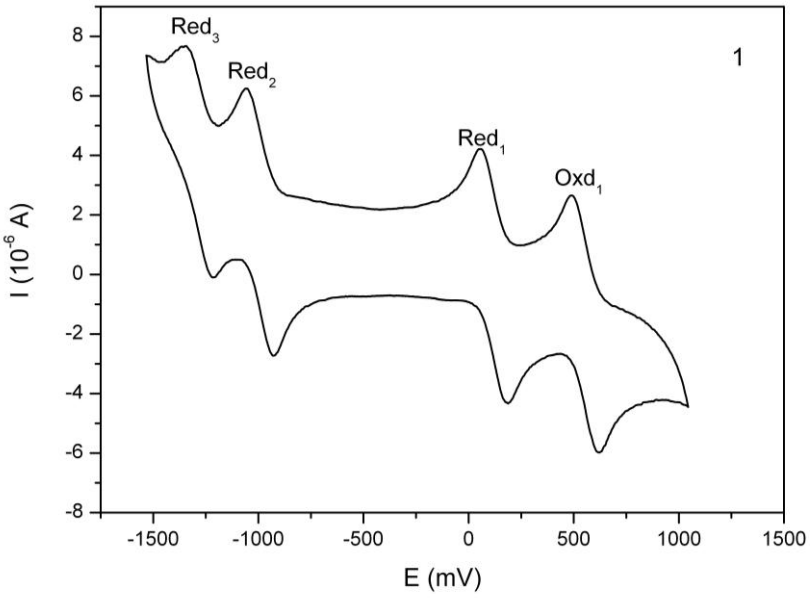

Fig. S5. Cyclic voltammogram of **1** recorded in CH_2Cl_2 containing 0.1 M $[NBu_4][ClO_4]$ at a scan rate 40 mV s^{-1}.

Table S1. ^{1}H NMR data (δ) for **1** and **2**.

compound	Signals for unsubstituted Pc		Signals for $Pc(OBNP)_4$		
	Pc-$H_{\alpha'}$	Pc-$H_{\beta'}$	H_{α}	inner set of binaphthyl substituents	outer set of binaphthyl substituents
1	–	–	8.40 (s, 8 H) 8.00 (s, 8 H)	8.17(d, 8 H) 7.43(d, 8 H) 7.87 (d, 8 H) 6.75~6.86 (m, 8 H)[a]	7.16 (d, 8 H) 7.00~7.07 (m, 16 H)[a] 6.68~6.93 (m, 40 H)[a]
2	9.07 (d, 4 H) 8.89 (d, 4 H)	8.39 ~ 8.42 (m, 4 H)[a] 8.28 ~ 8.32 (m, 4 H)	9.06 (s, 4 H) 8.73 (s, 4 H)	8.78 (d, 4 H) 8.39 (d, 4 H)[a] 8.51 (d, 4 H) 7.72 ~ 7.82 (m, 4 H)[a]	7.84(d, 4 H) 7.67 ~ 7.71 (m, 4 H) 8.00 ~ 8.05 (m, 8 H)[a] 7.72 ~ 7.82 (m, 8 H)[a] 7.51 ~ 7.58 (m, 8 H)[a]

[a] These signals overlap with each other.

Table S2. Comparison of the structural data for (*R*)- and (*S*)-**1**.

Structural parameter	(*R*)-**1**	(*S*)-**1**
Y-N_4(Pc) plane distance (Å)	1.383	1.390
N_4(Pc)-N_4(Pc) plane distance (Å)	2.77	2.78
dihedral angle between two N_4 planes(°)	0	0
average dihedral angle φ for the Pc ring(°)[a]	5.10	5.35
average twist angle(°)[b]	42.49	43.69

[a] The average dihedral angle of the individual isoindole rings with respect to the corresponding N_4 mean plane. [b] Defined as the rotation angle of one macrocycle away from the eclipsed conformation of the two macrocycles.

Table S3. Crystal data and structure refinements of (*R*)- and (*S*)-**1**.

compound	(*R*)-**1**[a]	(*S*)-**1**[a]
Formula	$C_{224}H_{108}N_{16}O_{16}Y$	$C_{224}H_{108}N_{16}O_{16}Y$
F.W.	3338.17	3368.17
system	tetragonal	tetragonal
space group	I_4	I_4
a	21.5507(5)	21.6634(5)
b	21.5507(5)	21.6634(5)
c	22.5192(14)	22.6033(14)
α	90	90
β	90	90
γ	90	90
Z	2	2
volume	10458.7(7)	10607.8(7)
F_{cald} / g cm^{-3}	1.070	1.056
*F*000	3462	3470
R_{int} $I>2\theta$	0.0636	0.0609
R_{w2} $I>2\theta$	0.1554	0.1507
R_{int} all	0.0676	0.0756
R_{w2} all	0.1593	0.1601
S	1.055	1.092
Absolute structure parameter	0.055(16)	0.036(19)
CCDC number	941795	941796

[a]In these structures, the unit cell includes a large region of disordered solvent molecules, which could not be modeled as discrete atomic sites. We employed PLATON/SQUEEZE to calculate the diffraction contribution of the solvent molecules and, thereby, to produce a set of solvent-free diffraction intensities. For (*R*)-**1**, the SQUEEZE calculations showed a total solvent accessible area volume of 1731 Å^3 and the residual electron density amounted to 481 electron per unit cell, corresponding to nearly 8 molecules of CH_2Cl_2 and 8 molecules of CH_3OH (about 2 CH_2Cl_2 and 2 CH_3OH molecules per asymmetric unit). As for (*S*)-**1**, the SQUEEZE calculations showed a total solvent accessible area volume of 1789 Å^3 and the residual electron density amounted to 335 electron per unit cell, corresponding to nearly 8 molecules of CH_2Cl_2 (about 2 CH_2Cl_2 molecules per asymmetric unit).