Trapping an ester hydrate intermediate in a π-stacked macrocycle by multiple hydrogen bonds

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Table S1. Crystallographic data of compound 1.

Compounds	**1**
Formula	$C_{159}H_{203.33}Cl_{10}Co_6N_{42}S_2O_{30.66}$
Formula weight	3805.60
Temp. (*K*)	200
Crystal System	Trigonal
Space group	$R\bar{3}$
a (Å)	22.8534(6)
b (Å)	22.8534(6)
c (Å)	28.8133(11)
a (°)	90
β (°)	90
γ (°)	120
V(Å^3)	13032.4(8)
Z	6
$\rho_{cal.}$(g cm^{-3})	1.423
μ	0.813
F(000)	5668.0
θ *range* (°)	2.176–27.494
Reflections ($I > 2\sigma$)	4323
$R_1(I > 2\sigma)$	0.0708
$wR_2(all)$	0.2171
GOF on F^2	1.027
CCDC#	2271453

[a] $R = \sum||F_0|-|F_c||)/\sum|F_0|$

[b] $wR = |\sum w(F_0-F_c)^2/\sum w(F_0^2)^2|^{1/2}$

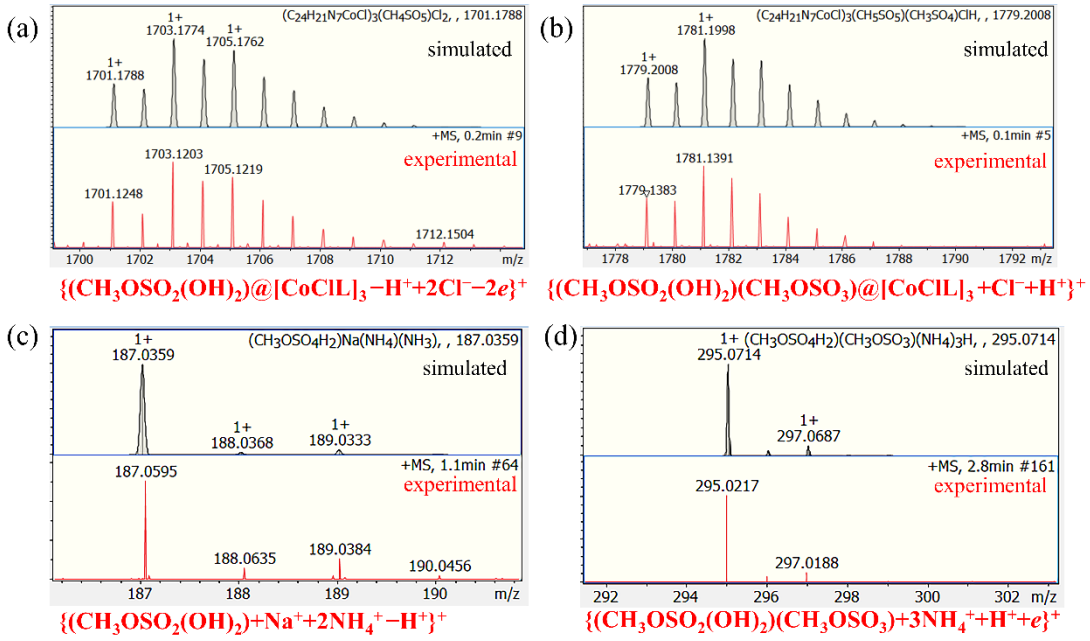

Figure S1. HR-MS of the reactant solution and the solution of compound **1** in methanol. (**a**) HR-MS of $\{(CH_3OSO_2(OH)_2)@[ClLCo^{II}]_3-H^++2Cl^--2e\}^+$ identified from the reactant solution. (**b**) HR-MS of $\{(CH_3OSO_2(OH)_2)(CH_3OSO_3)@[ClLCo^{II}]_3+Cl^-+H^+\}^+$ identified from the reactant solution. (**c**) HR-MS of $\{(CH_3OSO_2(OH)_2)+Na^++2NH_4^+-H^+\}^+$ identified from the solution of compound **1** in methanol. (**d**) HR-MS of $\{(CH_3OSO_2(OH)_2)(CH_3OSO_3)+3NH_4^++H^++e\}^+$ identified from the solution of compound **1** in methanol.

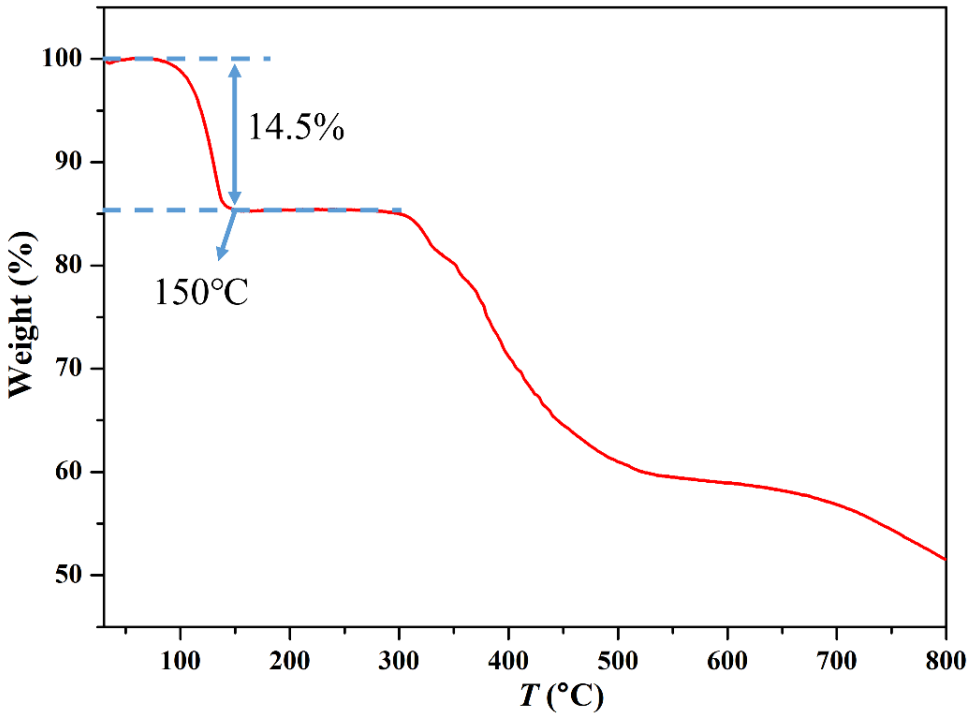

Figure S2. TGA for compound **1**.

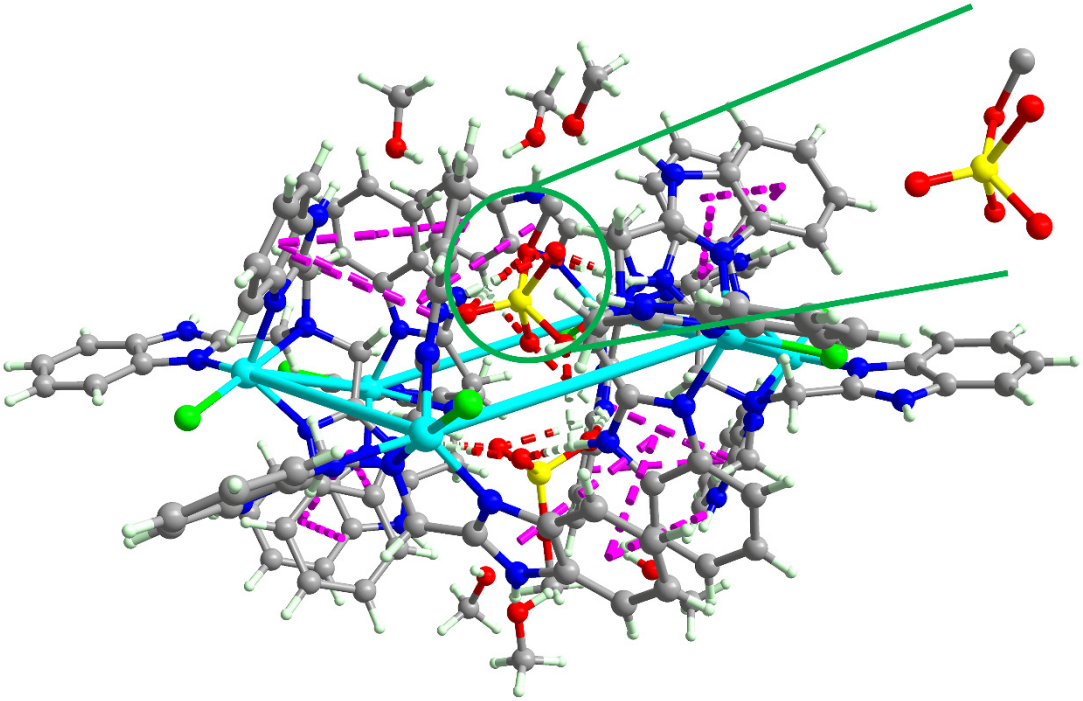

Figure S3. Side view of the structure of the boat-shaped macrocycle templating by $[CH_3OSO_2(OH)_2]^-$ and $[CH_3OSO_3]^-$ in compound **1**. (inset: structure of the ester hydrate $[CH_3OSO_2(OH)_2]^-$ in compound **1**.)

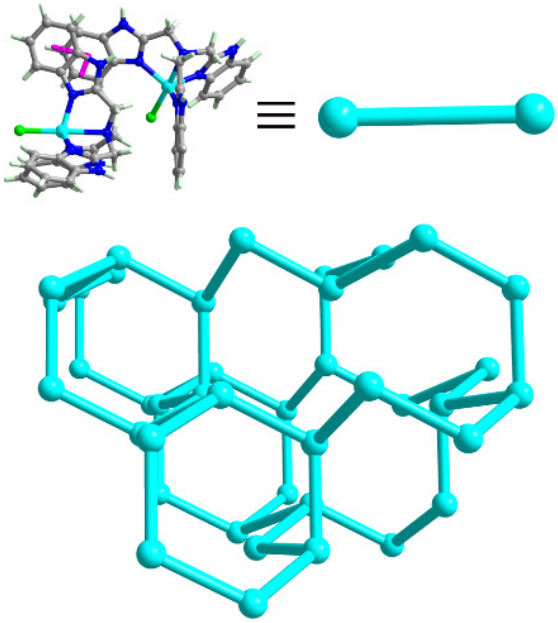

Figure S4. The pcu-h network of compound **1** when treating $[ClLCo^{II}]^+$ as node and the π-stacked pair of benzimidazoylmethyl arms as linker.

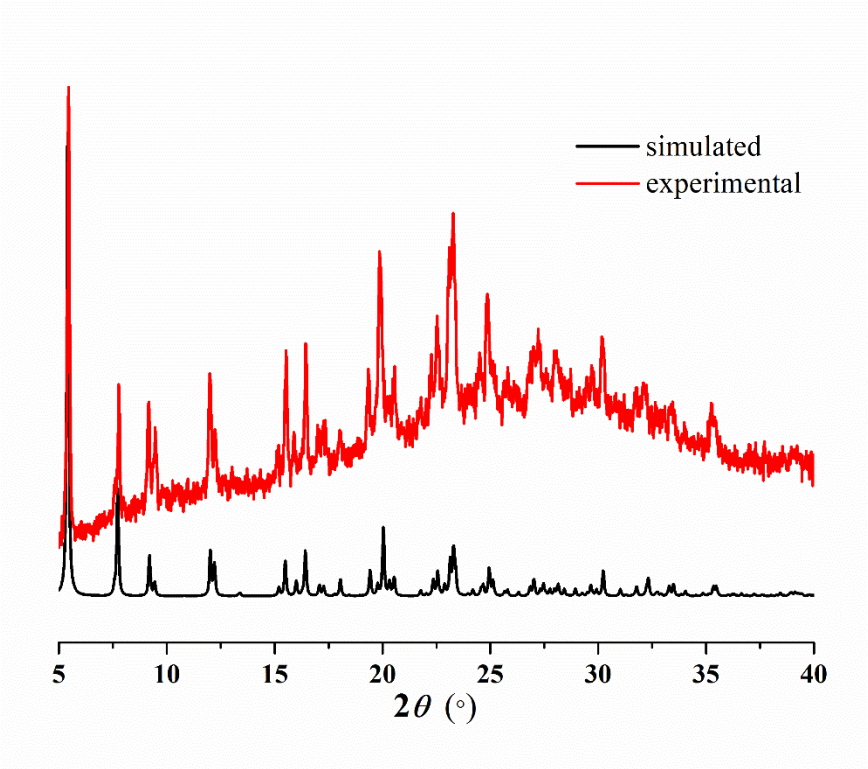

Figure S5. PXRD patterns for compound **1**.