Supporting Information

Slow Magnetic Relaxation in A Sandwich-Type Tetranuclear Dysprosium Complex with TMeQ[6] (TMeQ[6] = α, α', δ, δ'-Tetramethylcucurbit[6]uril)

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Details of the refinement of complexes 1 and 2:

All the structures were solved by direct methods using SHELXS-97, and further refined by full-matrix least-squares method on F^2 with anisotropic displacement parameters for the non-H atoms (except OH1, OH2, OH3, OH4, OH5, OH6, OH7, OH8, O6W, O34, N50, O32, O33, O12, O17, N4, N28, N27, N30, C046, C048, C066, C068, C074, C004, C070, N31, O14, C023, C75, C060, C069, C76, C77, N32, N35, N25, C052, C70', C053, C050, C059, C049, C071, O11, N31', N27', N30', C74', O12', C48' and C061 in **1** and OH1, OH6, OH5, OH7, OH4, OH8, O1, O16, C61, O1W, O4W, N41, C64, C6, C2, C23, C1, C5, N44, C66 and C67 in **2**) using SHELXL-97. Hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2 x Ueq of the attached atom (1.5 x Ueq for methyl hydrogen atoms). The severely disordered guest molecules in **1** and **2** were removed by SQUEEZE and other disordered atoms were refined with commands of ISOR. Totally 52 restraints have been applied in the structural refinement of **1**, 21 restraints for **2**.

Table S1: Selected Bond Distances (Å) and Angles (°) for 1.

Dy1-O16	2.196(8)	Dy2-O14	2.545(9)	Dy3-O13	2.198(8)
Dy1-O17	2.571(9)	Dy2-O15	2.203(7)	Dy3-O14$^{\#2}$	2.537(9)
Dy1-O36	2.335(12)	Dy2-O37	2.336(11)	Dy3-O29	2.337(11)
Dy1-O2W	2.566(7)	Dy2-O1W	2.527(9)	Dy3-O2W	2.596(7)
Dy1-O3W	2.539(9)	Dy2-O2W	2.641(7)	Dy3-O3W	2.543(9)
Dy1-OH5	2.346(19)	Dy2-OH6	2.302(15)	Dy3-OH5$^{\#2}$	2.318(17)
Dy1-OH8	2.354(16)	Dy2-OH7	2.355(16)	Dy3-OH6	2.360(14)
Dy1-OH7$^{\#2}$	2.341(17)	Dy2-OH8$^{\#2}$	2.328(17)	Dy3-OH7	2.240(16)
Dy4-O17$^{\#2}$	2.541(9)	Dy5-O5	2.115(11)	Dy6-O6	2.314(8)
Dy4-O22	2.227(7)	Dy5-O12	2.403(14)	Dy6-O7	2.457(7)
Dy4-O28	2.360(14)	Dy5-O31	2.321(14)	Dy6-O32	2.352(15)
Dy4-O1W	2.521(9)	Dy5-O4W	2.491(9)	Dy6-O5W	2.718(9)
Dy4-O2W	2.596(7)	Dy5-O5W	2.388(10)	Dy6-O6W	2.535(11)
Dy4-OH5	2.327(17)	Dy5-OH1$^{\#1}$	2.248(16)	Dy6-OH1	2.312(13)
Dy4-OH6$^{\#2}$	2.318(17)	Dy5-OH2	2.31(2)	Dy6-OH3	2.368(12)
Dy4-OH8	2.382(16)	Dy5-OH4	2.412(19)	Dy6-OH4$^{\#1}$	2.311(18)
Dy7-O11	1.954(12)	Dy8-O7$^{\#1}$	2.533(8)	Dy1-OH5-Dy4	101.7(7)
Dy7-O12' $^{\#1}$	2.329(19)	Dy8-O8	2.389(9)	Dy1-OH5-Dy3$^{\#2}$	106.1(7)
Dy7-O33	2.302(15)	Dy8-O34	2.308(14)	Dy3-OH7-Dy1$^{\#2}$	108.9(7)
Dy7-O5W	2.522(8)	Dy8-O4W	2.518(10)	Dy1-OH8-Dy4	99.9(6)
Dy7-O6W	2.563(10)	Dy8-O5W	2.635(9)	Dy1-OH8-Dy2$^{\#2}$	109.7(6)
Dy7-OH2	2.40(2)	Dy8-OH1	2.395(12)	Dy2-OH7-Dy1$^{\#2}$	109.2(6)
Dy7-OH3$^{\#1}$	2.334(16)	Dy8-OH2$^{\#1}$	2.20(2)	Dy2$^{\#2}$-OH8-Dy4	104.1(7)
Dy7-OH4	2.232(18)	Dy8-OH3	2.304(13)	Dy2-OH7-Dy3	103.2(6)
Dy3$^{\#2}$-OH5-Dy4	111.3(7)	Dy5-OH2-Dy7	100.4(8)	Dy8-OH1-Dy5$^{\#1}$	106.4(5)
Dy2-OH6-Dy4$^{\#2}$	107.0(6)	Dy6-OH3-Dy8	100.8(5)	Dy5-OH2-Dy8$^{\#1}$	111.0(9)
Dy7-OH2-Dy8$^{\#1}$	112.3(10)	Dy2-OH6-Dy3	101.2(5)	Dy8-OH3-Dy7$^{\#1}$	111.1(7)
Dy3-OH6-Dy4$^{\#2}$	110.1(6)	Dy7-OH4-Dy5	112.3(10)	Dy6-OH3-Dy7$^{\#1}$	104.3(5)
Dy7-OH4-Dy6$^{\#1}$	109.6(7)	Dy6-OH1-Dy8	99.8(5)	Dy5-OH4-Dy6$^{\#1}$	107.8(7)
Dy6-OH1-Dy5$^{\#1}$	113.6(7)				

#1 -x+2,-y+1,-z+1 #2 -x+1,-y,-z+2

Table S2: Selected Bond Distances (Å) and Angles (°) for 2

Tb1-O13	2.526(4)	Tb2-O15	2.122(4)	Tb3-O13$^{\#2}$	2.504(4)
Tb1-O14	2.294(3)	Tb2-O16	2.383(10)	Tb3-O17	2.275(3)
Tb1-O30	2.327(7)	Tb2-O31	2.318(6)	Tb3-O32	2.327(6)
Tb1-O4W	2.585(5)	Tb2-O5W	2.476(4)	Tb3-O5W	2.657(4)
Tb1-O5W	2.652(4)	Tb2-O6W	2.495(4)	Tb3-O6W	2.563(4)
Tb1-OH1	2.329(6)	Tb2-OH2	2.346(13)	Tb3-OH1	2.393(6)
Tb1-OH3	2.373(7)	Tb2-OH3$^{\#2}$	2.239(7)	Tb3-OH2$^{\#2}$	2.278(13)
Tb1-OH4$^{\#2}$	2.282(11)	Tb2-OH4	2.312(10)	Tb3-OH3	2.334(6)
Tb4-O16'$^{\#2}$	2.425(10)	Tb5-O1' $^{\#1}$	2.392(9)	Tb6-O3	2.257(3)
Tb4-O18	2.100(5)	Tb5-O4	2.138(4)	Tb6-O6$^{\#1}$	2.489(4)
Tb4-O33	2.353(6)	Tb5-O29	2.336(6)	Tb6-O26	2.315(6)
Tb4-O4W	2.504(4)	Tb5-O1W	2.513(5)	Tb6-O1W	2.571(5)
Tb4-O5W	2.515(4)	Tb5-O2W	2.524(4)	Tb6-O2W	2.690(4)
Tb4-OH1$^{\#2}$	2.257(7)	Tb5-OH5$^{\#1}$	2.230(7)	Tb6-OH5	2.353(7)
Tb4-OH2	2.308(12)	Tb5-OH6	2.317(9)	Tb6-OH7	2.343(6)
Tb4-OH4	2.310(10)	Tb5-OH8	2.307(13)	Tb6-OH8$^{\#1}$	2.282(13)
Tb7-O1	2.404(12)	Tb8-O5	2.311(3)	Tb1-OH1-Tb3	100.9(2)
Tb7-O2	2.127(4)	Tb8-O6	2.527(4)	Tb1-OH1-Tb4$^{\#2}$	107.9(3)
Tb7-O25	2.334(7)	Tb8-O28	2.333(6)	Tb2-OH1-Tb3	100.3(5)
Tb7-O2W	2.502(4)	Tb8-O2W	2.594(4)	Tb2-OH2-Tb3$^{\#2}$	107.2(5)
Tb7-O3W	2.490(4)	Tb8-O3W	2.555(4)	Tb2-OH2-Tb4	101.8(4)
Tb7-OH6	2.321(9)	Tb8-OH5	2.362(7)	Tb4-OH2-Tb3$^{\#2}$	113.9(5)
Tb7-OH7$^{\#1}$	2.275(7)	Tb8-OH6$^{\#1}$	2.256(10)	Tb1-OH3-Tb3	101.4(3)
Tb7-OH8	2.368(13)	Tb8-OH7	2.338(6)	Tb3-OH3-Tb2$^{\#2}$	108.9(3)
Tb1-OH3-Tb2$^{\#2}$	111.8(3)	Tb2-OH4-Tb1$^{\#2}$	112.5(4)	Tb2-OH4-Tb4	102.7(4)
Tb4-OH4-Tb1$^{\#2}$	107.7(4)	Tb6-OH5-Tb8	99.7(2)	Tb8-OH5-Tb5$^{\#1}$	112.7(3)
Tb6-OH5-Tb5$^{\#1}$	108.6(3)	Tb5-OH6-Tb7	103.5(3)	Tb5-OH6-Tb8$^{\#1}$	113.5(4)
Tb7-OH6-Tb8$^{\#1}$	108.5(4)	Tb6-OH7-Tb8	100.7(2)	Tb6-OH7-Tb7$^{\#1}$	112.5(3)
Tb8-OH7-Tb7$^{\#1}$	107.2(3)	Tb7-OH8-Tb5	102.4(5)	Tb7-OH8-Tb6$^{\#1}$	111.3(5)
Tb5-OH8-Tb6$^{\#1}$	108.4(5)				

#1 -x+1,-y,-z+1 #2 -x+2,-y+1,-z+2

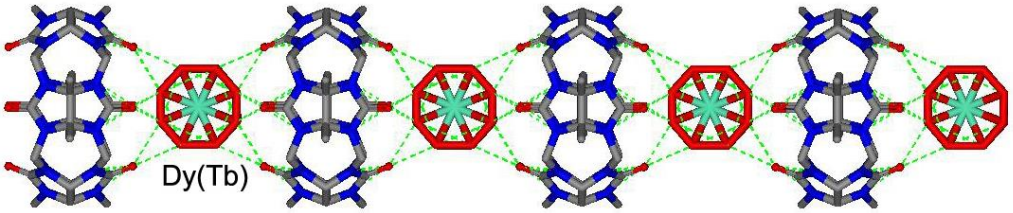

Fig.S1 One dimensional supramolecular chain constructed of TMeQ[6] and $Ln(H_2O)_8^{3+}$ complexes through hydrogen bonding. (Absence of 1H-[3-(4-Pyridyl) pyrazole]–acetic acid). This compound was prepared using the same procedure as described for the synthesis of complexes 1 or 2, only absence of 1H-[3-(4-Pyridyl) pyrazole]–acetic acid. Crystal data for **Dy**: Mr = 1343.49, Monoclinic, space group *c2/m*, *a* = 22.3936(8) Å, *b* = 12.4132(4) Å, *c* = 15.0550(9) Å, β = 116.855(4)°, *V* = 3733.6(3) Å^3, *Z* = 2, μ = 1.072 mm^{-1}, ρ_{calcd} = 1.195 g·cm^{-3}. Crystal data for **Tb**: Mr = 1339.92, Monoclinic, space group *c2/m*, *a* = 22.257(5) Å, *b* = 12.498(3)Å, *c* = 14.854(3) Å, β = 116.35(3)°, *V* = 3702.5(13)Å^3, *Z* = 2, μ = 1.027 mm^{-1}, ρ_{calcd} = 1.202g·cm^{-3}.

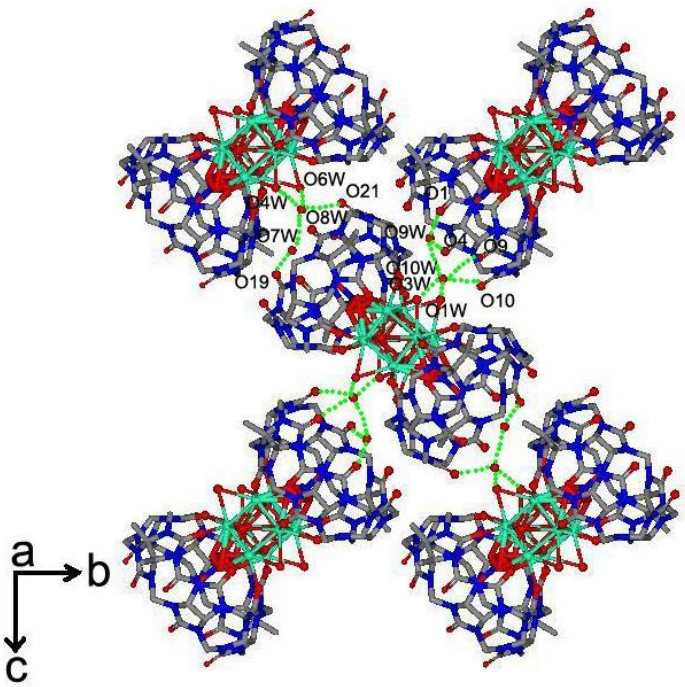

Fig.S2 Packing diagram of 1. The hydrogen atoms are omitted for clarity, and dashed lines indicate hydrogen-bonding interactions.

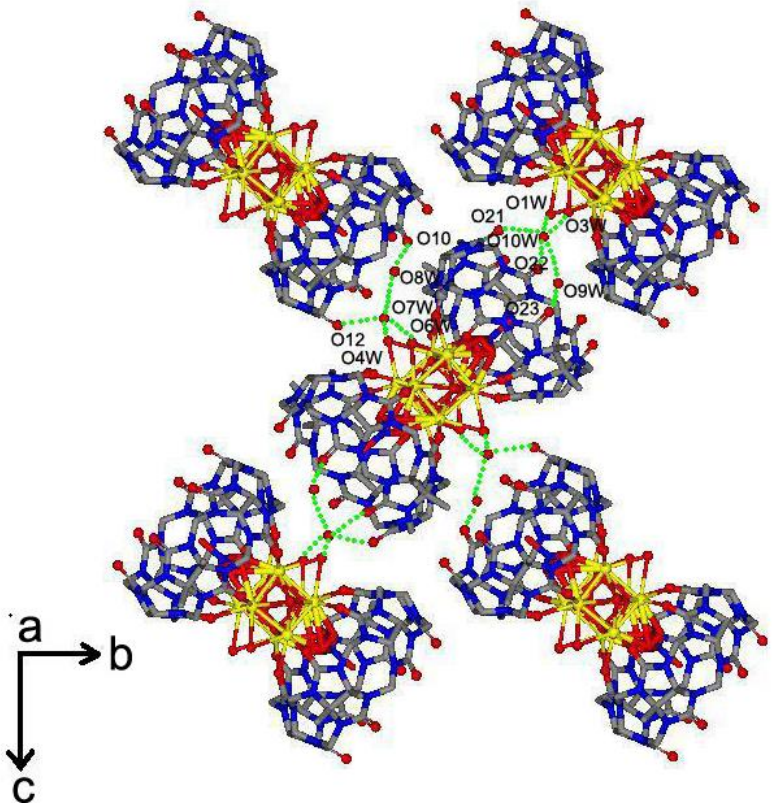

Fig.S3 Packing diagram of 2. The hydrogen atoms are omitted for clarity, and dashed lines indicate hydrogen-bonding interactions.

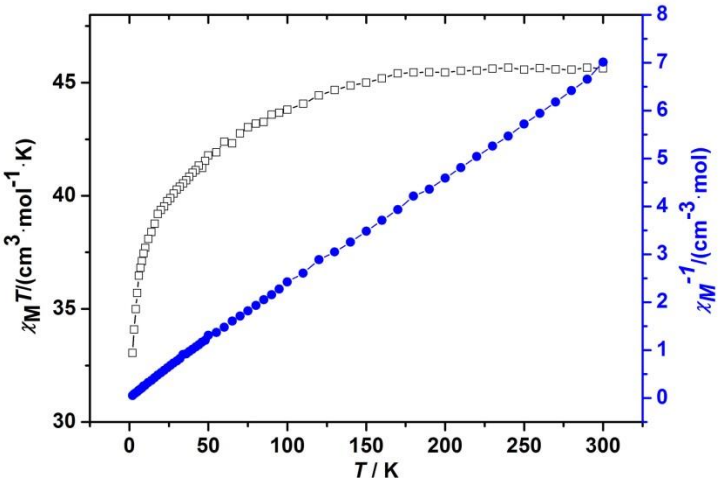

Fig.S4 Plots of temperature dependence of $\chi_M T$ vs T and χ_M^{-1} vs T for 2.

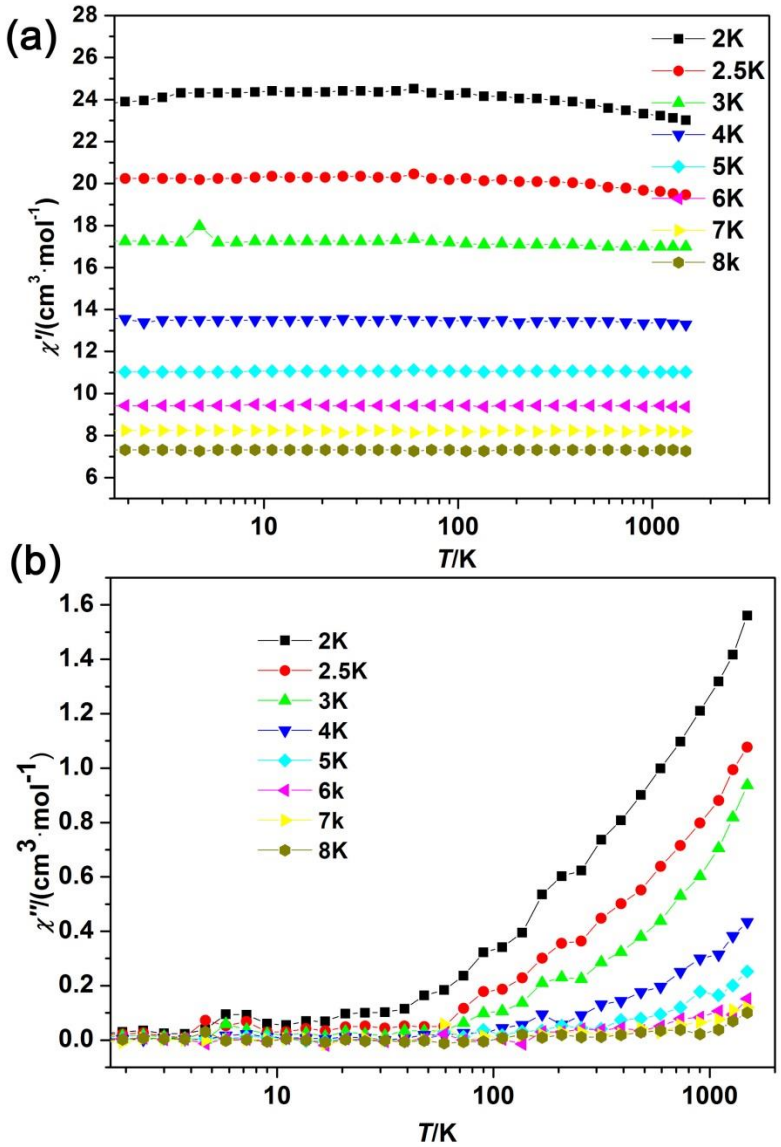

Fig.S5 Frequency dependence of in-of-phase (a) and out-of-phase (b) ac susceptibility of **1**

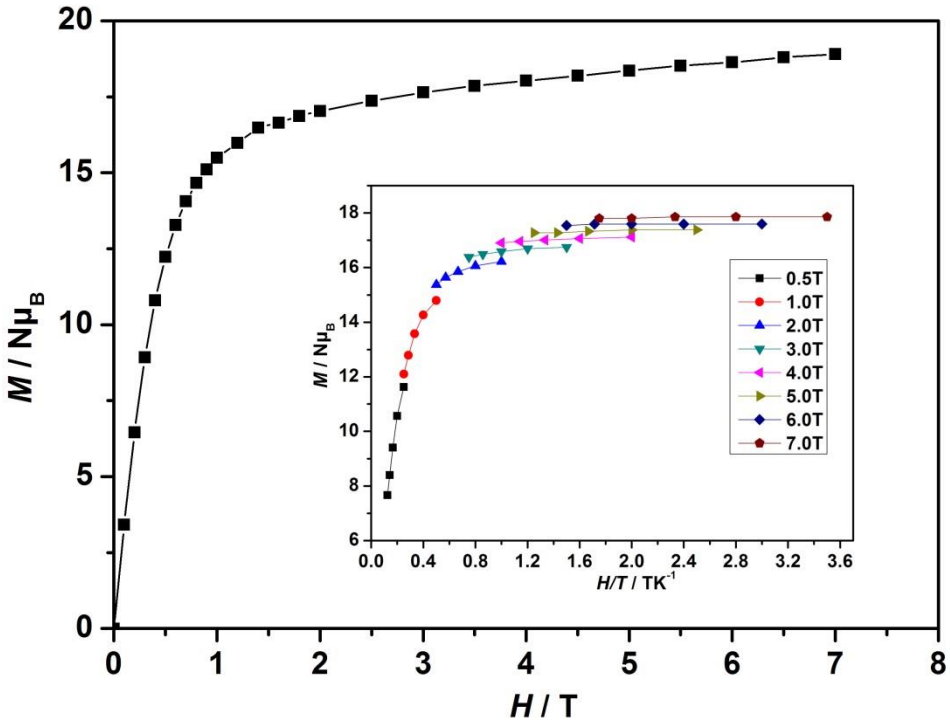

Fig.S6 *M* vs *H*/*T* plots for 2 measured in different fields below 7 T.

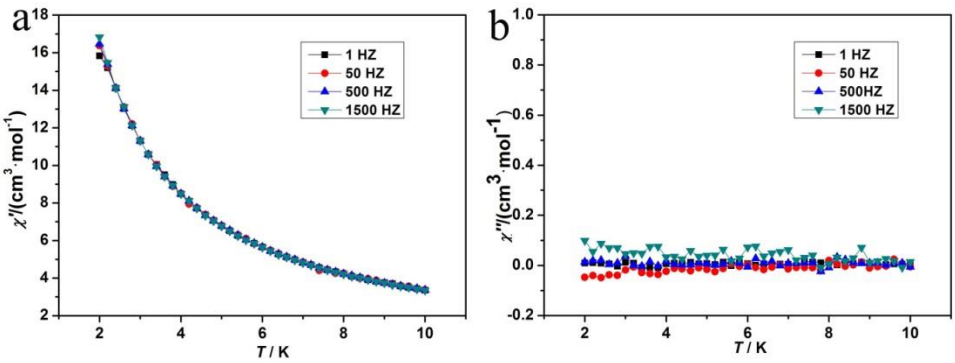

Fig.S7 Temperature dependence of the in-phase (a) the out-of-phase (b) ac susceptibilities at the indicated frequencies for 2 under zero dc field.

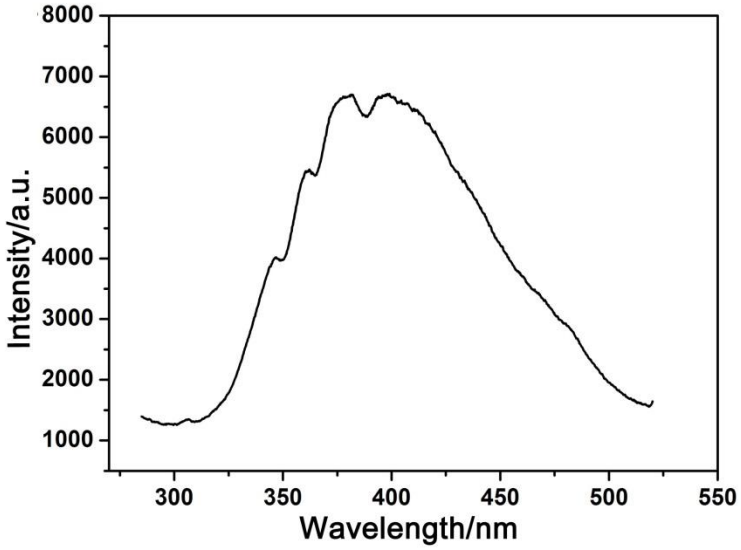

Fig.S8 Emission spectrum of 1 under 265 nm excitation in the solid state at room temperature.

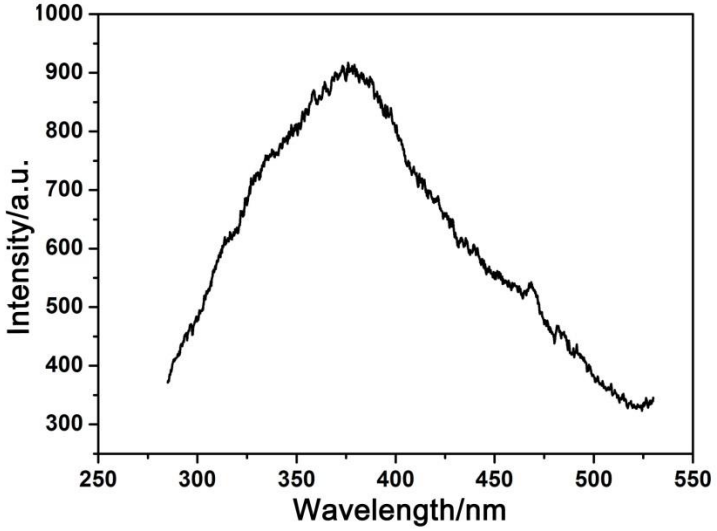

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Fig.S9 Emission spectrum of TMEQ[6] under 270 nm excitation in the solid state at room temperature.

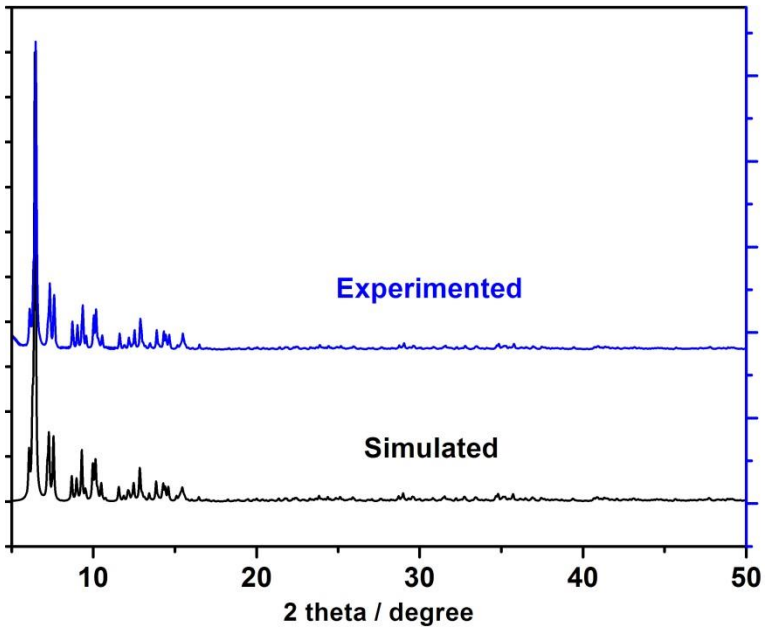

Fig.S10 The XRD patterns and the simulated one from the single-crystal diffraction data for 1

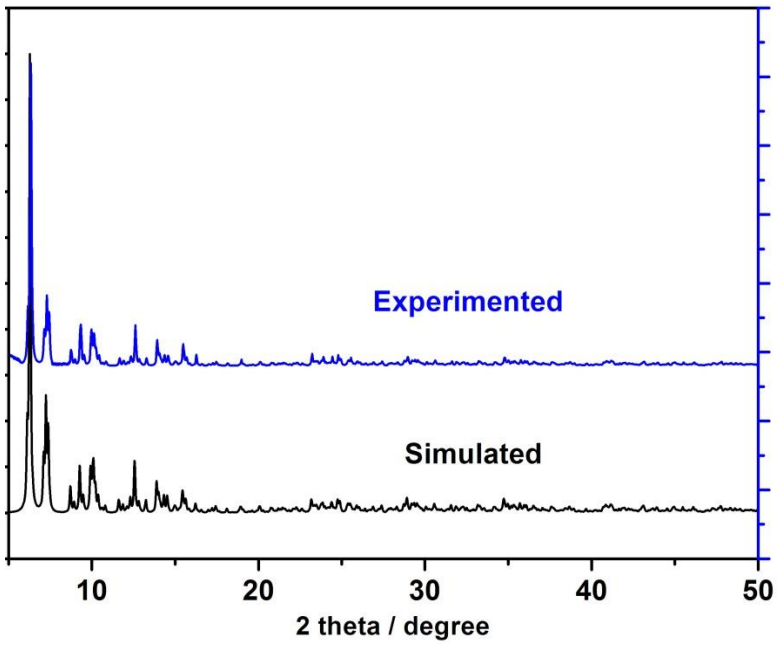

Fig.S11 The XRD patterns and the simulated one from the single-crystal diffraction data for 2