Electronic Supplementary Material for CrystEngComm

Electronic Supplementary Information (ESI)

The self-assembly of hetero-nuclear complex monitored with ESI-MS and fluorescence spectrophotometry

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Table S1. Details on crystal data collection and structure refinement.

Complex	**Ag-CDO**	**Tb-Ag-CDO**
Formula	$C_7H_5O_7Ag$	$C_{21}H_{24}O_{27}Ag_3Tb$
Fw	308.98	1190.93
Cryst size, mm	0.20 x 0.14 x 0.12	0.20 x 0.16 x 0.10
Temp, K	113(2)	113(2)
Cryst syst	Monoclinic	Triclinic
Space group	*P21/c*	*P-1*
a, Å	15.670(9)	8.740(2)
b, Å	3.5182(18)	11.111(2)
c, Å	15.036(8)	16.820(3)
α, deg	90	101.261(3)
ß, deg	104.523(7)	104.482(3)
γ, deg	90	98.162(3)
V, Å^3	747.9(4)	1519.6(5)
Z	4	2
D_c, g/cm^3	2.558	2.603
μ, mm^{-1}	2.528	4.319
no. of data/param	1901 / 136	7150 / 466
obs reflns	8303	9313
θ range, deg	2.69-27.88	1.91-25.02
F(000)	600	1144
R_{int}	0.0391	0.0391
R1 [$I > 2\acute{o}(I)$]	0.0260	0.0387
wR2 (all data)	0.0626	0.0909
max/min, e Å^{-3}	0.785 / -1.078	1.498/ -0.961

Table S2. Selected bond lengths (Å) and angles (deg) for **Ag-CDO** and **Tb-Ag-CDO**.

Complex **Ag-CDO**			
Ag(1)-O(6)#1	2.298(2)	O(6)#1-Ag(1)-O(7)	111.09(8)
Ag(1)-O(5)#2	2.339(2)	O(5)#2-Ag(1)-O(7)	71.16(8)
Ag(1)-O(7)#3	2.488(2)	O(7)#3-Ag(1)-O(7)	89.86(8)
Ag(1)-O(7)	2.494(2)	O(6)#1-Ag(1)-O(5)	112.41(7)
Ag(1)-Ag(1)#4	2.9901(15)	O(5)#2-Ag(1)-O(5)	89.28(8)
Ag(1)-Ag(1)#3	3.5182(18)	O(7)#3-Ag(1)-O(5)	66.23(8)
Ag(1)-Ag(1)#2	3.5182(18)	O(7)-Ag(1)-O(5)	130.69(8)
O(6)#1-Ag(1)-O(5)#2	140.20(7)	Ag(1)#3-O(5)-Ag(1)	89.28(8)
O(6)#1-Ag(1)-O(7)#3	92.62(8)	Ag(1)#2-O(7)-Ag(1)	89.86(8)
O(5)#2-Ag(1)-O(7)#3	127.09(8)		
Complex **Tb-Ag-CDO**			
Tb(1)-O(5)	2.275(5)	O(5)-Tb(1)-O(8)	125.57(18)
Tb(1)-O(20)	2.290(5)	O(20)-Tb(1)-O(8)	79.68(18)
Tb(1)-O(22)	2.322(5)	O(22)-Tb(1)-O(8)	72.04(18)
Tb(1)-O(24)	2.328(5)	O(24)-Tb(1)-O(8)	76.22(19)
Tb(1)-O(8)	2.337(5)	O(5)-Tb(1)-O(19)	74.7(2)
Tb(1)-O(19)	2.350(5)	O(20)-Tb(1)-O(19)	114.0(2)
Tb(1)-O(21)	2.360(5)	O(22)-Tb(1)-O(19)	138.5(2)
Tb(1)-O(23)	2.366(5)	O(24)-Tb(1)-O(19)	76.0(2)
Ag(1)-O(18)#1	2.161(4)	O(8)-Tb(1)-O(19)	73.03(18)
Ag(1)-O(1)	2.163(5)	O(5)-Tb(1)-O(21)	74.9(2)
Ag(1)-O(16)	2.444(5)	O(20)-Tb(1)-O(21)	138.44(19)
Ag(1)-Ag(2)	2.8479(16)	O(22)-Tb(1)-O(21)	110.4(2)
Ag(1)-Ag(1)#2	3.1654(16)	O(24)-Tb(1)-O(21)	68.4(2)
Ag(3)-O(12)#3	2.207(5)	O(8)-Tb(1)-O(21)	141.76(19)
Ag(3)-O(11)#4	2.217(5)	O(19)-Tb(1)-O(21)	84.6(2)
Ag(3)-O(4)#5	2.390(5)	O(5)-Tb(1)-O(23)	78.87(19)
Ag(3)-Ag(3)#6	2.7930(18)	O(20)-Tb(1)-O(23)	77.00(18)
Ag(2)-O(17)#1	2.199(5)	O(22)-Tb(1)-O(23)	72.13(19)
Ag(2)-O(2)	2.217(5)	O(24)-Tb(1)-O(23)	110.81(19)
Ag(2)-O(10)#7	2.419(5)	O(8)-Tb(1)-O(23)	139.72(18)
O(5)-Tb(1)-O(20)	75.1(2)	O(19)-Tb(1)-O(23)	146.97(19)
O(5)-Tb(1)-O(22)	145.68(19)	O(21)-Tb(1)-O(23)	69.52(19)
O(20)-Tb(1)-O(22)	80.8(2)	O(18)#1-Ag(1)-O(1)	165.6(2)
O(5)-Tb(1)-O(24)	134.6(2)	O(18)#1-Ag(1)-O(16)	108.73(18)
O(20)-Tb(1)-O(24)	149.7(2)	O(1)-Ag(1)-O(16)	85.61(19)

O(22)-Tb(1)-O(24)	74.5(2)

Symmetry transformations used to generate equivalent atoms, **Ag-CDO**: #1 -x+2, -y+1, -z+1; #2 x, y-1, z; #3 x, y+1, z; #4 -x+2, -y, -z+1. **Tb-Ag-CDO**: #1 -x, -y, -z+2; #2 -x+1, -y, -z+2; #3 x, y+1, z+1; #4 -x+1, -y, -z+1; #5 x-1, y, z; #6 -x+1, -y+1, -z+2; #7 x+1, y+1, z+1.

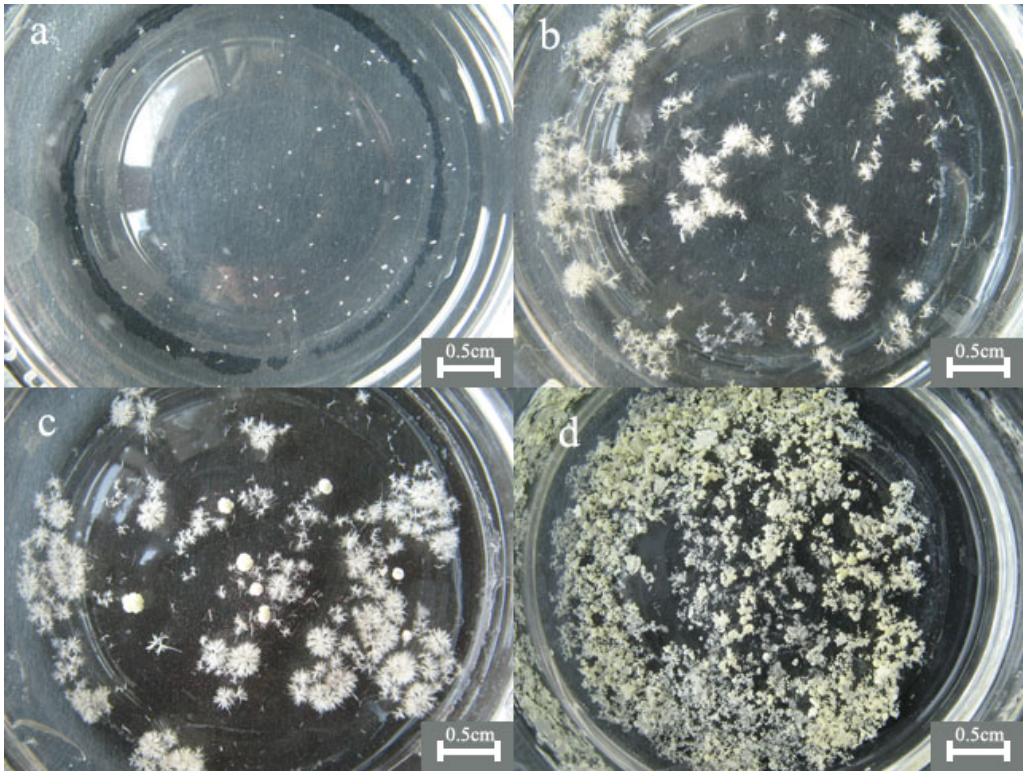

Figure S1. The visible sequential crystallization of complexes **Tb-Ag-CDO** and **Ag-CDO** recorded by a camera (photographs viewed in scale of 0.5 cm). (a) after 1h; (b) after 12 h; (c) after 24 h; (d) after two weeks.

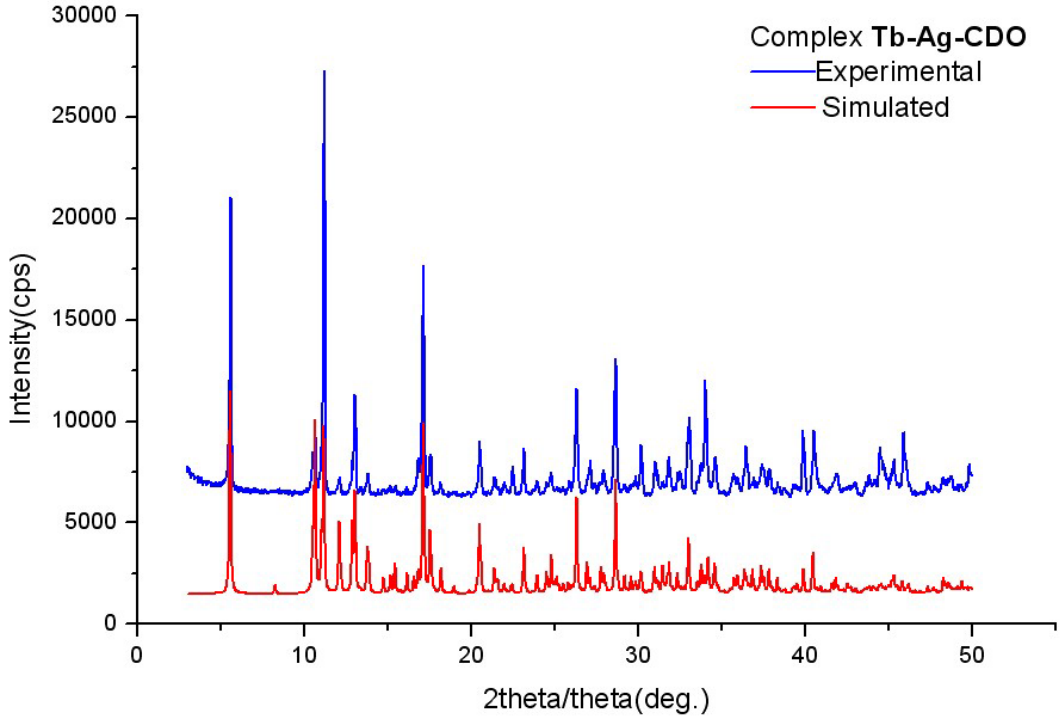

Figure S2. PXRD of colorless needle crystals selected after 12 h, together with the simulated PXRD of complex **Tb-Ag-CDO.**

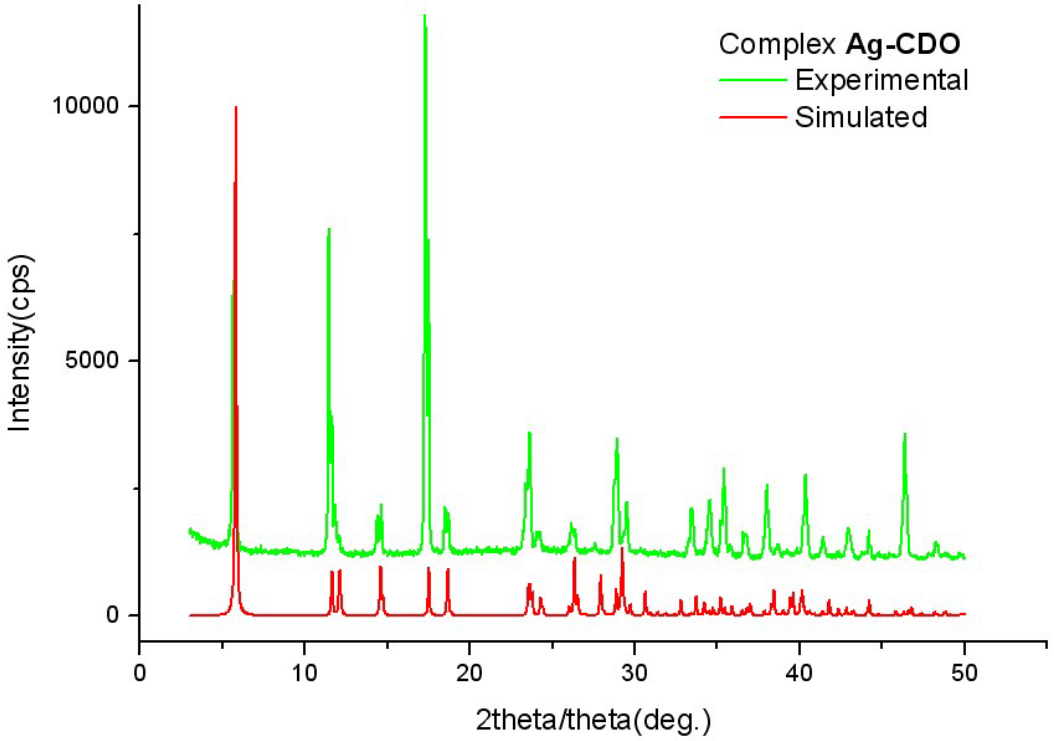

Figure S3. PXRD of crystals selected after 24 h, together with the simulated PXRD of complex **Ag-CDO**.

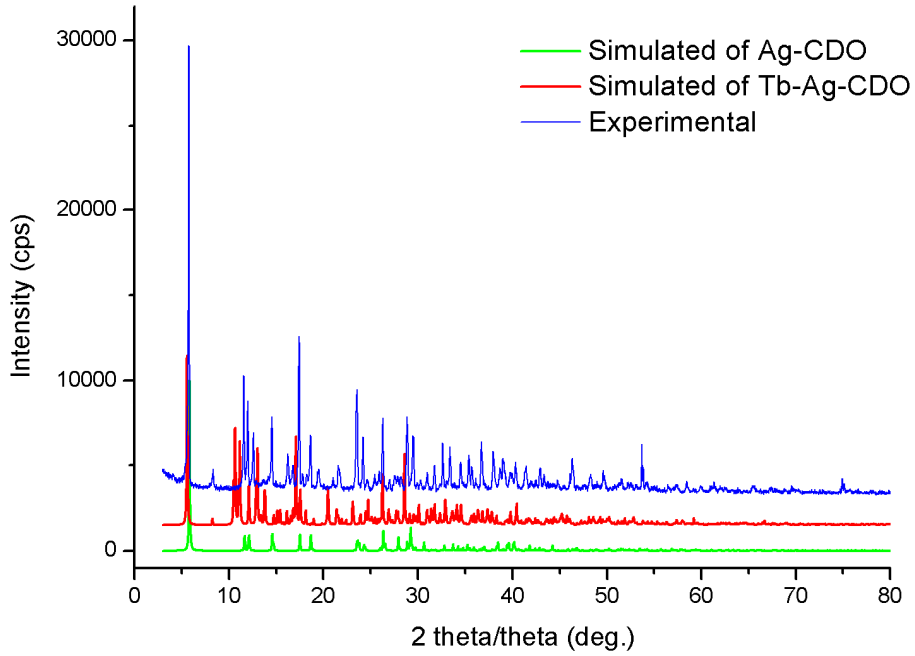

Figure S4. PXRD of crystals collected after two weeks, together with the simulated PXRD of complex **Ag-CDO** and **Tb-Ag-CDO**.

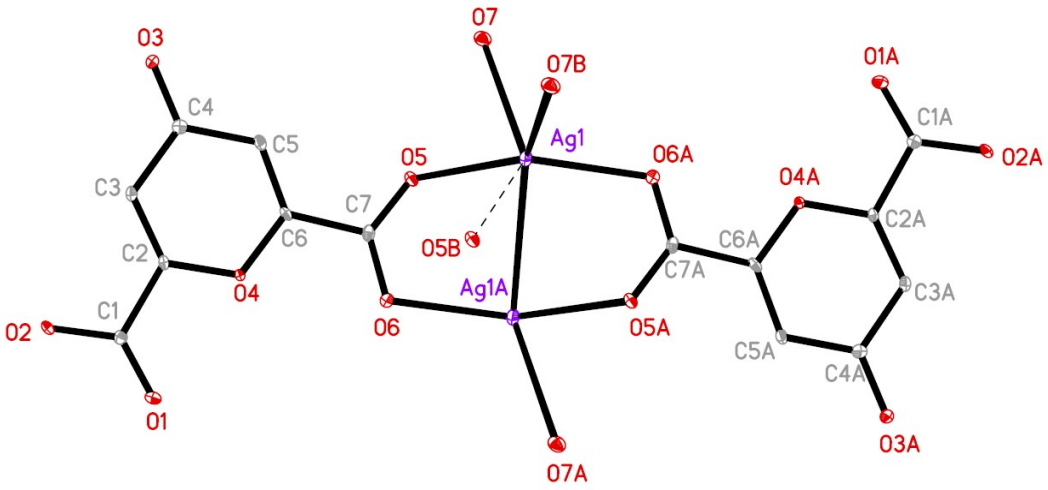

Figure S5. ORTEP representation of the coordinating environment of Ag atoms for **Ag-CDO** (30% probability ellipsoids) (A: x, 1+y, z; B: 2-x, -1-y, 1-z; C: 2-x, -y, 1-z).

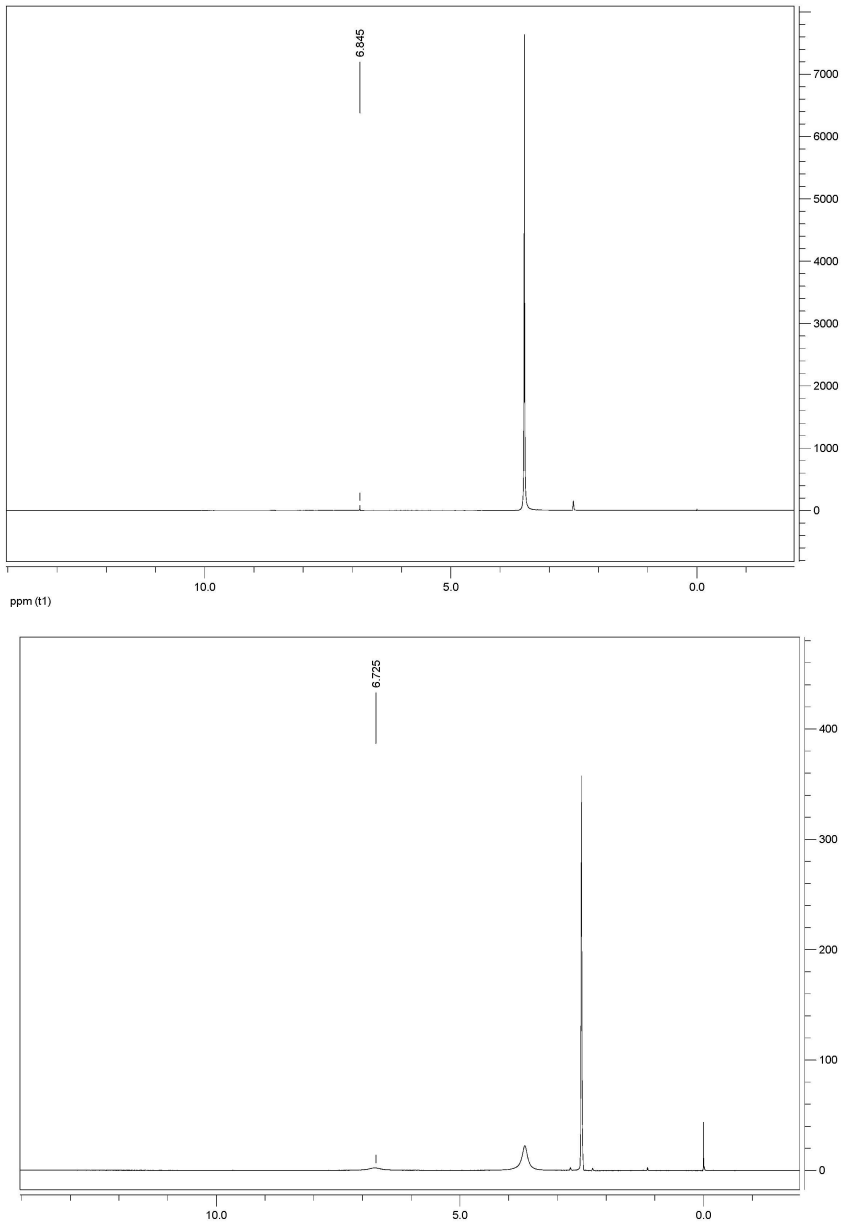

Figure S6. (Top) ^{1}H NMR spectrum of **Ag-CDO** in D_2O. (Bottom) ^{1}H NMR spectrum of **Tb-Ag-CDO** in DMSO.

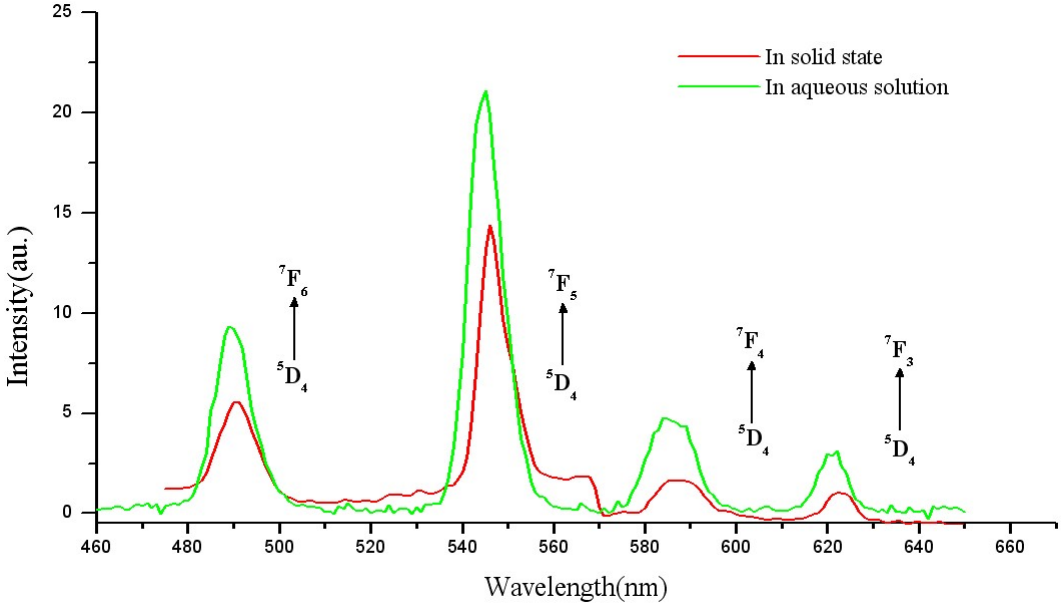

Figure S7. Emission spectra of complex **Tb-Ag-CDO** in solid state and in aqueous solution at RT. (solid state in red, aqueous solution in green).

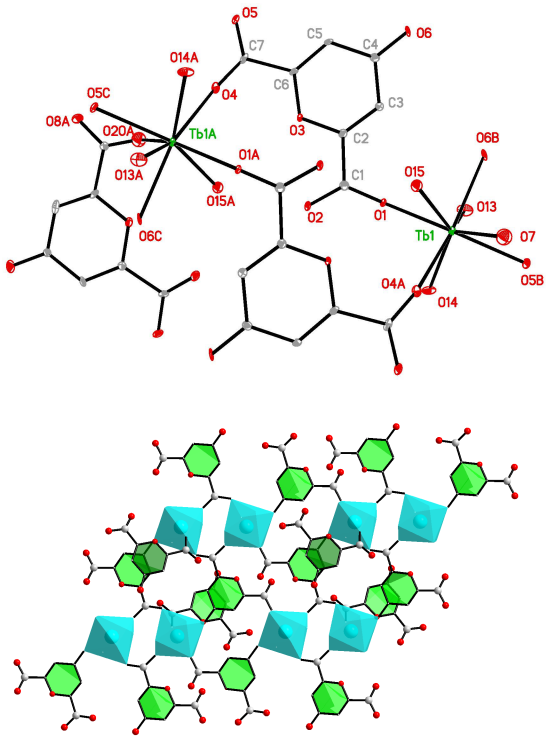

Figure S8. (Top) Coordination environment of Tb atom. (Bottom) 2D layer structure of **Tb-CDO**. Symmetry operator: A = x, y, z; B= 1+x, y, 1+z; C = -x, 1-y, 2-z.