Supporting Information

Construction and fluorescence sensing performance of cucurbit[5]uril-based supramolecular assemblies incorporating amino-containing sulfonate ligand as structure inducer

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EXPERIMENTAL SECTION

Materials and General Methods. All reagents and solvents were commercially purchased and used as received without further purification except that Q[5] was synthesized according to the previously reported methods.[1] FT-IR spectra were recorded in the range of 400-4000 cm^{-1} on a Bruker FT-IR Spectrometer INVENIO using KBr pellets. Thermogravimetric analyses (TGA) were carried out on a Shimadzu DTG-60H microcomputer differential thermal balance under nitrogen with a heating rate of 10 °C min^{-1}. Powder X-ray diffraction (PXRD) data were collected at room temperature on bulk samples with Cu K_α radiation (1.5418 Å) on a Bruker D8 Advance X-ray diffractometer. The fluorescence spectra were measured on an F-280 fluorescence spectrophotometer at room temperature, using the

excitation and emission slit widths of 5 nm and photomultiplier tube voltage of 700 V.

X-Ray Crystallography. Single-crystal X-ray diffraction (SC-XRD) data of assemblies **1 - 4** were collected on a Bruker D8 Venture diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) using ω-scan technique. The integration of diffraction data and intensity corrections for the Lorentz and polarization effects were performed by using *SAINT* program.[2] Semi-empirical absorption corrections were applied using *SADABS* program.[3] The structures were solved by direct methods with SHELXT-2018, expanded by subsequent Fourier-difference synthesis, and all the non-hydrogen atoms were refined anisotropically on F^2 by the full-matrix least-squares technique using the SHELXL-2018 crystallographic software package.[4,5] All non-hydrogen atoms were refined anisotropically and all the hydrogen atoms except those of coordinated water molecules were introduced at the calculated positions. The free solvent molecules in the unit cell have been taken into account to SQUEEZE option of the PLATON program.[6] The final chemical formulas were obtained based on volume/count electron analysis and TG analysis. The reported refinements were of the guest-free structures obtained by the SQUEEZE routine and the results were attached to the CIF files. The squeezed water molecules are 22, 7, 8 and 12 for compounds **1 - 4**, respectively. The details of crystal parameters, data collection and refinements are listed in Table S1, and the selected bond lengths and angles are given in Table S2.

Table S1. Crystal Data and Structure Refinements for **1**-**4**.

Compound	**1**	**2**	**3**	**4**
chemical formula	$C_{80}H_{76}N_{86}Na_4O_{56}S_4$	$C_{40}H_{57}K_2N_{21}O_{26}S_2$	$C_{40}H_{57}N_{21}O_{27}Rb_2S_2$	$C_{80}H_{104}Cs_4N_{42}O_{47}S_4$
formula weight	2786.02	1390.27	1499.07	3065.81
λ (Å)	0.71073	0.71073	0.71073	0.71073
T (K)	296(2)	273(2)	273(2)	296(2)
crystal system	Triclinic	Orthorhombic	Orthorhombic	Monoclinic
space group	*P*-1	$P2_12_12_1$	$P2_12_12_1$	*Cc*
a /Å	17.4522(10)	12.0698(10)	12.428(4)	14.7761(18)
b /Å	18.3402(10)	15.9709(15)	15.927(4)	23.575(3)
c /Å	18.9286(12)	29.752(3)	29.711(9)	17.677(3)
α (°)	84.216(2)	90	90	90
β (°)	71.577(2)	90	90	110.898(4)
γ (°)	89.565(2)	90	90	90
Volume /Å^3	5716.9(6)	5735.1(9)	5881(3)	5752.6(14)
Z	2	4	4	2
D_c /g·cm^{-3}	1.387	1.464	1.551	1.644
μ /mm^{-1}	0.192	0.326	1.824	1.425
F (000)	2460	2608	2784	2832
reflections collected	44727	39718	37374	20121
Independent reflections	20074	10105	10322	7844
R_{int}	0.0595	0.0843	0.0644	0.0551
Data / restraints / parameters	20074 / 114 / 1515	10105 / 122 / 805	10322 / 110 / 824	7844 / 8 / 760
GOF	1.027	1.023	1.026	1.077
R_1, wR_2 $[I > 2\sigma(I)]$[a,b]	0.0738, 0.2079	0.0637, 0.1622	0.0629, 0.1616	0.0607, 0.1701
R_1, wR_2 (all data)	0.1201, 0.2398	0.1080, 0.1915	0.1084, 0.1862	0.0628, 0.1729
CCDC.NO.	2531079	2531080	2531081	2531082

[a]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b]$wR_2 = |\Sigma w(|F_o|^2 - |F_c|^2)|/\Sigma|w(F_o)^2|^{1/2}$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$. $P = (F_o^2 + 2F_c^2)/3$

Table S2. Selected Bond Lengths (Å) and Angles (°) for **1**-**4**.

1			
Na(1)-O(5)	2.371(4)	Na(1)-O(13)	2.380(5)
Na(1)-O(14)	2.410(4)	Na(1)-O(1W)	2.423(4)
Na(1)-O(4)	2.499(5)	Na(1)-O(21)	2.560(4)
Na(2)-O(11)	2.341(4)	Na(2)-O(1)	2.361(4)
Na(2)-O(1W)	2.407(4)	Na(2)-O(12)	2.479(5)
Na(2)-O(27)	2.506(4)	Na(2)-O(2)	2.520(5)
Na(3)-O(18)	2.721(5)	Na(3)-O(19)	2.806(4)
Na(3)-O(20)	2.806(4)	Na(3)-O(16)	2.854(4)
Na(3)-O(17)	2.872(5)	Na(4)-O(2W)	2.476(9)
Na(4)-O(6)	2.613(5)	Na(4)-O(9)	2.630(5)
Na(4)-O(7)	2.665(5)	Na(4)-O(10)	2.721(5)
Na(4)-O(8)	2.822(5)		
O(5)-Na(1)-O(13)	108.91(16)	O(5)-Na(1)-O(14)	169.86(18)
O(13)-Na(1)-O(14)	80.57(15)	O(5)-Na(1)-O(1W)	89.55(15)
O(13)-Na(1)-O(1W)	101.01(15)	O(14)-Na(1)-O(1W)	84.96(15)
O(5)-Na(1)-O(4)	78.71(15)	O(13)-Na(1)-O(4)	171.59(17)
O(14)-Na(1)-O(4)	92.08(15)	O(1W)-Na(1)-O(4)	82.27(15)
O(5)-Na(1)-O(21)	93.85(14)	O(13)-Na(1)-O(21)	83.94(14)
O(14)-Na(1)-O(21)	90.70(14)	O(1W)-Na(1)-O(21)	172.76(16)
O(4)-Na(1)-O(21)	92.12(14)	O(11)-Na(2)-O(1)	169.42(17)
O(11)-Na(2)-O(1W)	89.02(15)	O(1)-Na(2)-O(1W)	97.35(15)
O(11)-Na(2)-O(12)	79.22(15)	O(1)-Na(2)-O(12)	108.34(15)
O(1W)-Na(2)-O(12)	95.96(16)	O(11)-Na(2)-O(27)	91.55(14)
O(1)-Na(2)-O(27)	82.14(14)	O(1W)-Na(2)-O(27)	179.29(16)
O(12)-Na(2)-O(27)	83.74(14)	O(11)-Na(2)-O(2)	94.69(15)
O(1)-Na(2)-O(2)	77.35(14)	O(1W)-Na(2)-O(2)	86.52(15)
O(12)-Na(2)-O(2)	173.36(15)	O(27)-Na(2)-O(2)	93.85(14)
O(18)-Na(3)-O(19)	70.39(12)	O(18)-Na(3)-O(20)	134.71(13)
O(19)-Na(3)-O(20)	70.33(12)	O(18)-Na(3)-O(16)	131.87(14)
O(19)-Na(3)-O(16)	134.23(12)	O(20)-Na(3)-O(16)	68.53(12)
O(18)-Na(3)-O(17)	70.01(12)	O(19)-Na(3)-O(17)	135.64(14)
O(20)-Na(3)-O(17)	130.95(14)	O(16)-Na(3)-O(17)	66.76(12)
O(2W)-Na(4)-O(6)	81.48(19)	O(2W)-Na(4)-O(9)	119.95(19)
O(6)-Na(4)-O(9)	138.70(16)	O(2W)-Na(4)-O(7)	89.02(19)
O(6)-Na(4)-O(7)	72.62(15)	O(9)-Na(4)-O(7)	136.05(16)
O(2W)-Na(4)-O(10)	97.0(2)	O(6)-Na(4)-O(10)	70.94(14)
O(9)-Na(4)-O(10)	71.61(14)	O(7)-Na(4)-O(10)	141.66(16)

O(2W)-Na(4)-O(8)	110.1(2)	O(6)-Na(4)-O(8)	140.68(16)
O(9)-Na(4)-O(8)	68.97(13)	O(7)-Na(4)-O(8)	70.24(14)
O(10)-Na(4)-O(8)	139.56(15)		

2

K(1)-O(1)	2.745(6)	K(1)-O(2W)	2.784(10)
K(1)-O(4)	2.787(6)	K(1)-O(5)	2.792(6)
K(1)-O(2)	2.859(6)	K(1)-O(1W)	2.860(9)
K(1)-O(3)	2.930(7)	K(2)-O(3W)	2.698(8)
K(2)-O(10)	2.726(6)	K(2)-O(9)	2.803(6)
K(2)-O(6)	2.813(7)	K(2)-O(8)	2.831(7)
K(2)-O(7)	2.847(7)	K(2)-O(1W)#2	3.422(11)
O(1)-K(1)-O(2W)	131.1(3)	O(1)-K(1)-O(4)	127.70(19)
O(2W)-K(1)-O(4)	74.2(3)	O(1)-K(1)-O(5)	67.88(17)
O(2W)-K(1)-O(5)	89.8(3)	O(4)-K(1)-O(5)	67.65(19)
O(1)-K(1)-O(2)	70.00(16)	O(2W)-K(1)-O(2)	139.8(3)
O(4)-K(1)-O(2)	123.86(19)	O(5)-K(1)-O(2)	129.53(17)
O(1)-K(1)-O(1W)	74.9(3)	O(2W)-K(1)-O(1W)	68.6(4)
O(4)-K(1)-O(1W)	141.8(3)	O(5)-K(1)-O(1W)	103.3(3)
O(2)-K(1)-O(1W)	91.2(3)	O(1)-K(1)-O(3)	127.27(17)
O(2W)-K(1)-O(3)	101.0(3)	O(4)-K(1)-O(3)	67.16(18)
O(5)-K(1)-O(3)	128.20(18)	O(2)-K(1)-O(3)	63.67(16)
O(1W)-K(1)-O(3)	127.9(3)	O(3W)-K(2)-O(10)	89.4(2)
O(3W)-K(2)-O(9)	108.7(2)	O(10)-K(2)-O(9)	70.03(18)
O(3W)-K(2)-O(6)	95.0(3)	O(10)-K(2)-O(6)	69.33(18)
O(9)-K(2)-O(6)	132.21(19)	O(3W)-K(2)-O(8)	126.7(3)
O(10)-K(2)-O(8)	129.93(19)	O(9)-K(2)-O(8)	66.19(18)
O(6)-K(2)-O(8)	129.0(2)	O(3W)-K(2)-O(7)	116.9(2)
O(10)-K(2)-O(7)	129.63(19)	O(9)-K(2)-O(7)	128.8(2)
O(6)-K(2)-O(7)	66.46(19)	O(8)-K(2)-O(7)	68.50(19)
O(3W)-K(2)-O(1W)#2	74.7(3)	O(10)-K(2)-O(1W)#2	116.9(2)
O(9)-K(2)-O(1W)#2	59.5(2)	O(6)-K(2)-O(1W)#2	167.3(2)
O(8)-K(2)-O(1W)#2	56.8(2)	O(7)-K(2)-O(1W)#2	111.4(3)

3

Rb(1)-O(4)	2.867(7)	Rb(1)-O(3W)	2.902(10)
Rb(1)-O(3)	2.908(8)	Rb(1)-O(5)	2.909(7)
Rb(1)-O(2)	2.918(7)	Rb(1)-O(1)	2.957(7)
Rb(1)-O(4W)	3.387(11)	Rb(1)-O(5W)	3.54(3)

Rb(2)-O(10)#1	2.863(7)	Rb(2)-O(7)#1	2.873(7)
Rb(2)-O(6)#1	2.911(7)	Rb(2)-O(9)#1	2.957(7)
Rb(2)-O(2W)	2.979(14)	Rb(2)-O(8)#1	2.982(7)
Rb(2)-O(4W)	2.992(11)	Rb(2)-O(3W)	3.494(11)
O(4)-Rb(1)-O(3W)	89.6(2)	O(4)-Rb(1)-O(3)	67.4(2)
O(3W)-Rb(1)-O(3)	97.0(3)	O(4)-Rb(1)-O(5)	67.99(19)
O(3W)-Rb(1)-O(5)	110.4(3)	O(3)-Rb(1)-O(5)	126.6(2)
O(4)-Rb(1)-O(2)	124.9(2)	O(3W)-Rb(1)-O(2)	123.1(3)
O(3)-Rb(1)-O(2)	65.7(2)	O(5)-Rb(1)-O(2)	123.5(2)
O(4)-Rb(1)-O(1)	124.59(19)	O(3W)-Rb(1)-O(1)	132.2(3)
O(3)-Rb(1)-O(1)	125.0(2)	O(5)-Rb(1)-O(1)	64.28(19)
O(2)-Rb(1)-O(1)	67.0(2)	O(4)-Rb(1)-O(4W)	116.3(3)
O(3W)-Rb(1)-O(4W)	79.3(3)	O(3)-Rb(1)-O(4W)	174.6(2)
O(5)-Rb(1)-O(4W)	58.7(2)	O(2)-Rb(1)-O(4W)	112.9(3)
O(1)-Rb(1)-O(4W)	56.9(3)	O(4)-Rb(1)-O(5W)	102.8(5)
O(3W)-Rb(1)-O(5W)	60.4(5)	O(3)-Rb(1)-O(5W)	51.6(6)
O(5)-Rb(1)-O(5W)	167.8(4)	O(2)-Rb(1)-O(5W)	68.0(5)
O(1)-Rb(1)-O(5W)	127.5(5)	O(4W)-Rb(1)-O(5W)	123.0(6)
O(10)#1-Rb(2)-O(7)#1	123.3(2)	O(10)#1-Rb(2)-O(6)#1	66.42(18)
O(7)#1-Rb(2)-O(6)#1	66.3(2)	O(10)#1-Rb(2)-O(9)#1	68.26(17)
O(7)#1-Rb(2)-O(9)#1	120.17(19)	O(6)#1-Rb(2)-O(9)#1	125.17(18)
O(10)#1-Rb(2)-O(2W)	135.4(4)	O(7)#1-Rb(2)-O(2W)	76.6(4)
O(6)#1-Rb(2)-O(2W)	93.8(4)	O(9)#1-Rb(2)-O(2W)	140.7(4)
O(10)#1-Rb(2)-O(8)#1	122.47(18)	O(7)#1-Rb(2)-O(8)#1	66.08(19)
O(6)#1-Rb(2)-O(8)#1	124.15(19)	O(9)#1-Rb(2)-O(8)#1	62.09(18)
O(2W)-Rb(2)-O(8)#1	101.9(4)	O(10)#1-Rb(2)-O(4W)	79.2(3)
O(7)#1-Rb(2)-O(4W)	140.3(3)	O(6)#1-Rb(2)-O(4W)	102.3(3)
O(9)#1-Rb(2)-O(4W)	97.9(3)	O(2W)-Rb(2)-O(4W)	65.9(4)
O(8)#1-Rb(2)-O(4W)	133.1(3)	O(10)#1-Rb(2)-O(3W)	110.1(2)
O(7)#1-Rb(2)-O(3W)	116.8(3)	O(6)#1-Rb(2)-O(3W)	176.5(2)
O(9)#1-Rb(2)-O(3W)	52.28(19)	O(2W)-Rb(2)-O(3W)	88.5(4)
O(8)#1-Rb(2)-O(3W)	57.6(2)	O(4W)-Rb(2)-O(3W)	76.4(3)

4

Cs(1)-O(2)	2.943(12)	Cs(1)-O(3)	3.022(13)
Cs(1)-O(4)	3.055(11)	Cs(1)-O(1)	3.110(14)
Cs(1)-O(5)	3.204(13)	Cs(1)-O(1W)	3.253(17)
Cs(1)-O(15)	3.409(14)	Cs(2)-O(2W)	2.11(4)
Cs(2)-O(6)	2.930(14)	Cs(2)-O(8)	2.956(13)

Cs(2)-O(10)	2.991(13)	Cs(2)-O(9)	3.012(13)
Cs(2)-O(7)	3.022(12)	Cs(2)-O(1W)#1	3.401(17)
O(2)-Cs(1)-O(3)	62.5(3)	O(2)-Cs(1)-O(4)	115.1(3)
O(3)-Cs(1)-O(4)	65.3(3)	O(2)-Cs(1)-O(1)	63.0(3)
O(3)-Cs(1)-O(1)	116.1(3)	O(4)-Cs(1)-O(1)	113.7(3)
O(2)-Cs(1)-O(5)	115.1(3)	O(3)-Cs(1)-O(5)	116.2(3)
O(4)-Cs(1)-O(5)	60.6(3)	O(1)-Cs(1)-O(5)	63.4(3)
O(2)-Cs(1)-O(1W)	122.3(4)	O(3)-Cs(1)-O(1W)	155.4(3)
O(4)-Cs(1)-O(1W)	121.4(4)	O(1)-Cs(1)-O(1W)	84.2(4)
O(5)-Cs(1)-O(1W)	84.7(3)	O(2)-Cs(1)-O(15)	82.9(3)
O(3)-Cs(1)-O(15)	71.4(3)	O(4)-Cs(1)-O(15)	113.6(3)
O(1)-Cs(1)-O(15)	129.9(3)	O(5)-Cs(1)-O(15)	162.0(3)
O(1W)-Cs(1)-O(15)	84.9(4)	O(2W)-Cs(2)-O(6)	89.2(13)
O(2W)-Cs(2)-O(8)	142.5(13)	O(6)-Cs(2)-O(8)	117.8(3)
O(2W)-Cs(2)-O(10)	96.0(13)	O(6)-Cs(2)-O(10)	66.0(4)
O(8)-Cs(2)-O(10)	118.0(4)	O(2W)-Cs(2)-O(9)	125.0(12)
O(6)-Cs(2)-O(9)	118.0(3)	O(8)-Cs(2)-O(9)	67.1(3)
O(10)-Cs(2)-O(9)	60.9(4)	O(2W)-Cs(2)-O(7)	112.8(12)
O(6)-Cs(2)-O(7)	64.1(3)	O(8)-Cs(2)-O(7)	64.3(3)
O(10)-Cs(2)-O(7)	120.8(3)	O(9)-Cs(2)-O(7)	122.0(3)
O(2W)-Cs(2)-O(1W)#1	59.2(12)	O(6)-Cs(2)-O(1W)#1	92.2(4)
O(8)-Cs(2)-O(1W)#1	92.2(4)	O(10)-Cs(2)-O(1W)#1	148.2(4)
O(9)-Cs(2)-O(1W)#1	148.7(4)	O(7)-Cs(2)-O(1W)#1	61.6(3)

[a] Symmetry transformations used to generate equivalent atoms: #1 x+1, y, z; #2 x-1, y, z for **2**. #1 x-1, y, z; #2 x+1, y, z for **3**. #1 x-1/2, y-1/2, z; #2 x+1/2, y+1/2, z for **4**.

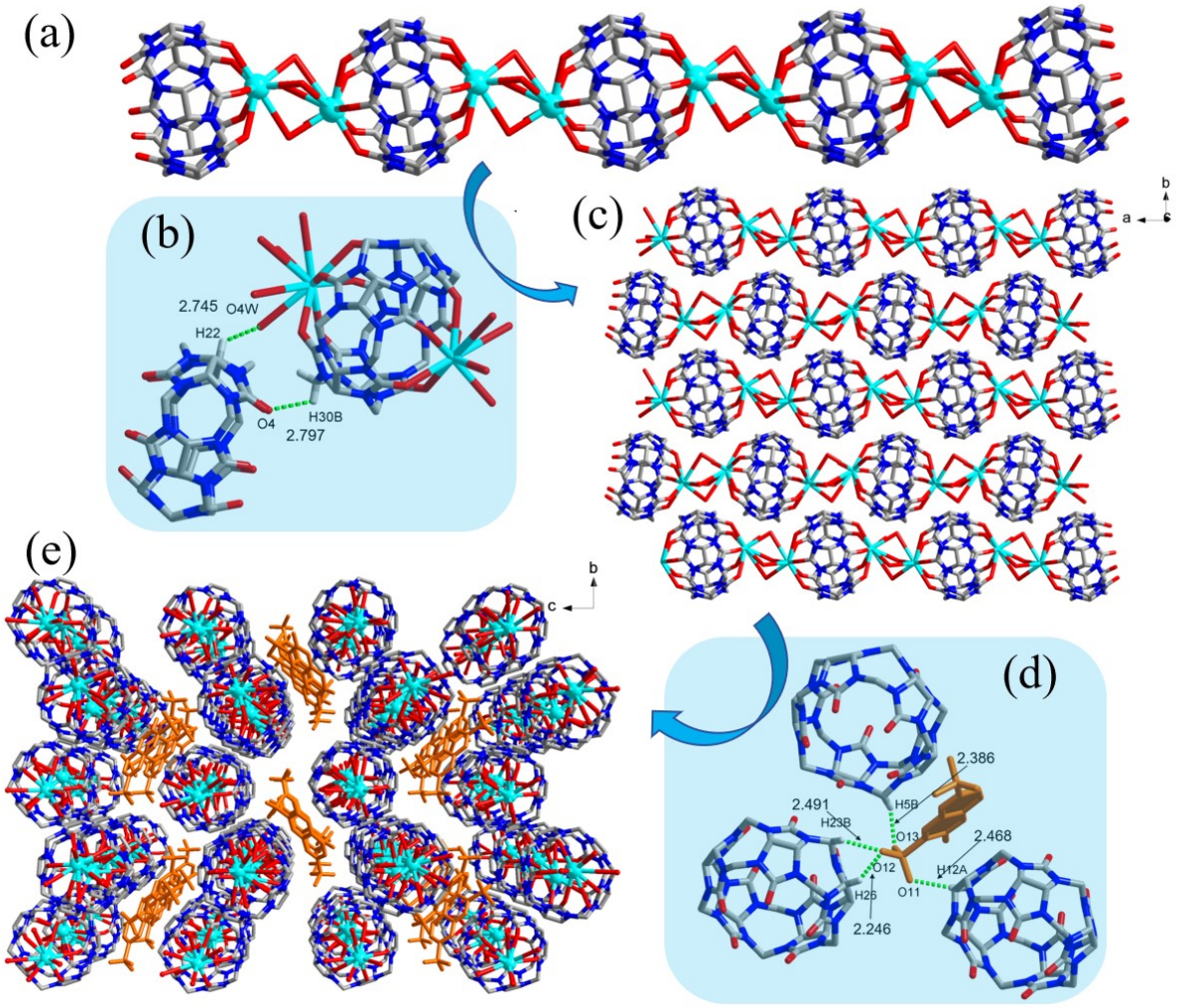
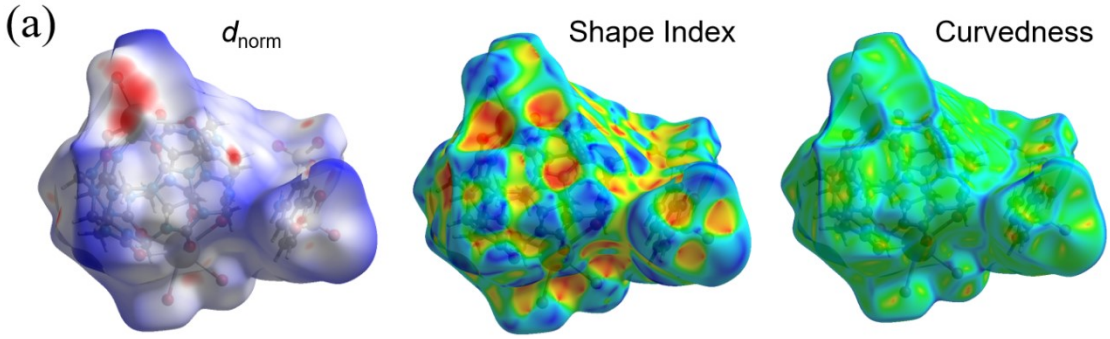


Figure S1. The structure of the 1D Q[5]-Rb^+ coordination chains (a), 2D Q[5]-K^+ supramolecular layers (c) and 3D Q[5]-K^+-$ADNS^{2-}$ framework (e). The hydrogen bonds between Q[5]-Rb^+ chains (b) or Q[5]-K^+ chains and $ADNS^{2-}$ anions (d).



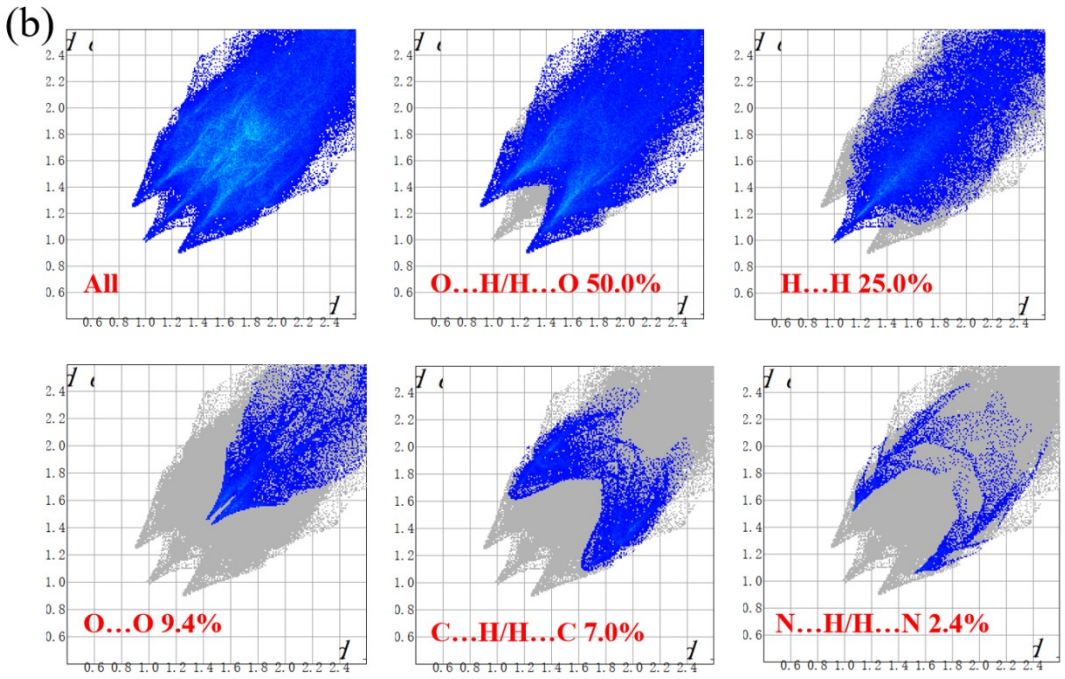
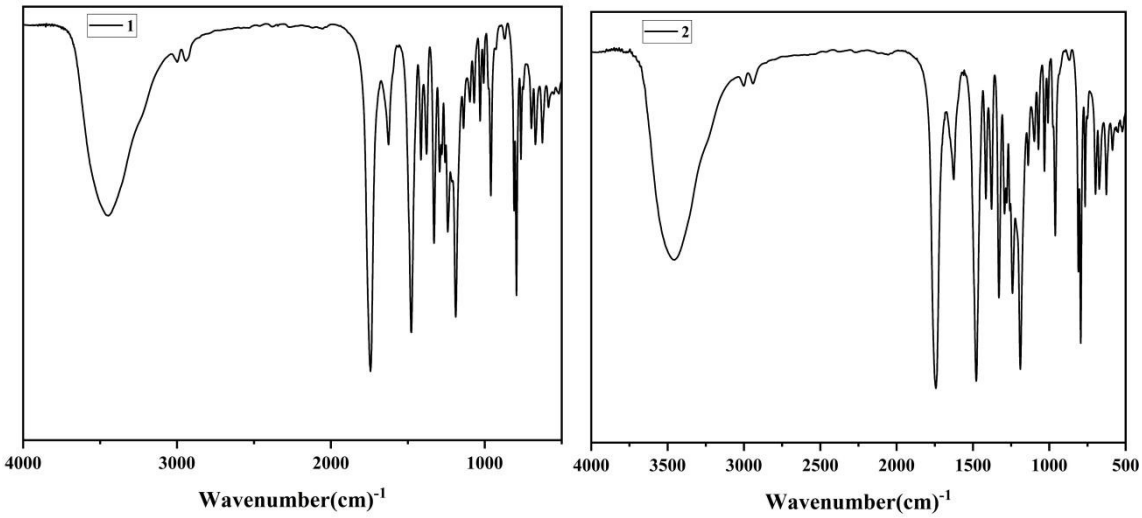


Figure S2. (a) Hirshfeld surfaces of assembly **2** mapped over d_{norm} (left), shape index (middle), and curvedness (right). (b) Fingerprint plot: Resolved into H…H, H…O/O…H, H…C/C…H, and O…O contacts contributed to the total Hirshfeld Surface area of the assembly **2**.



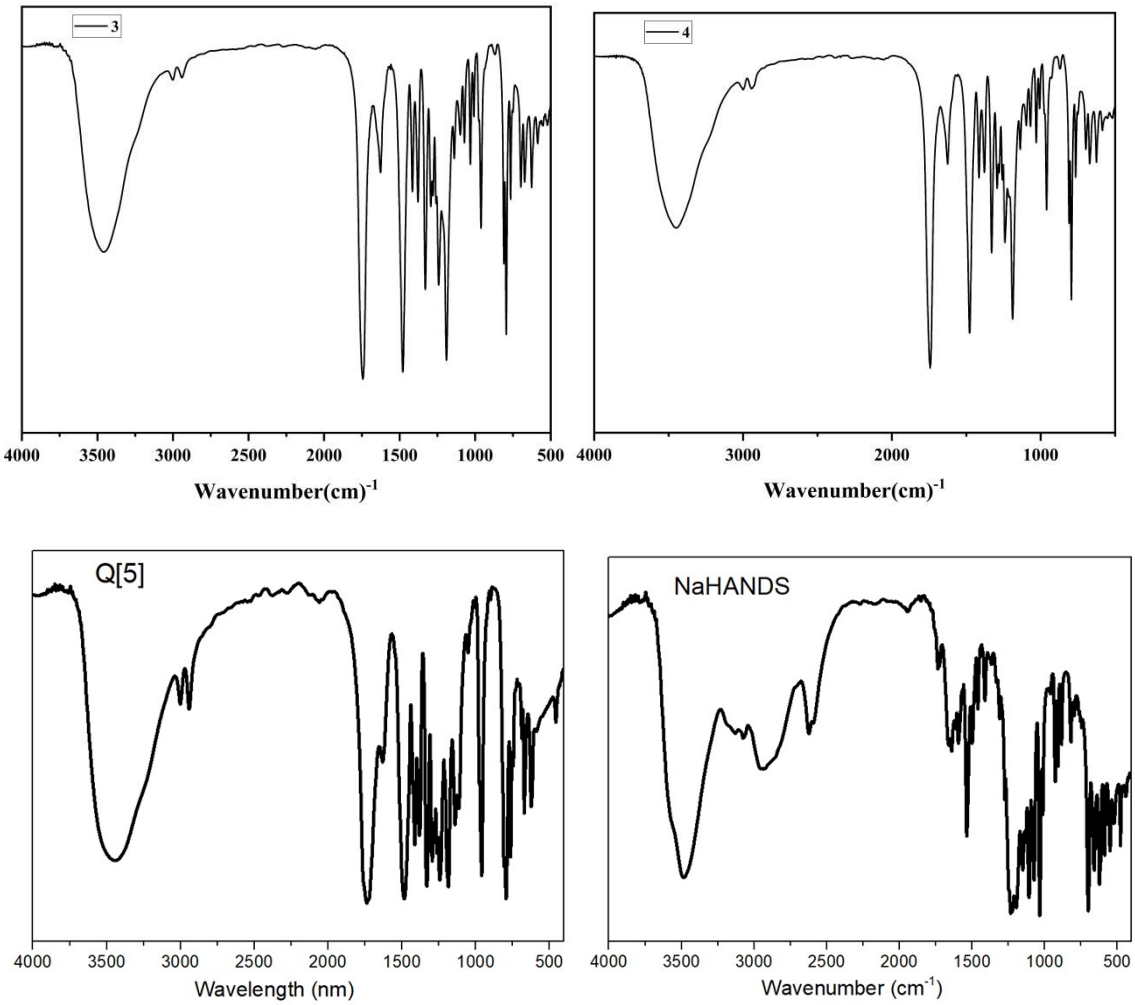
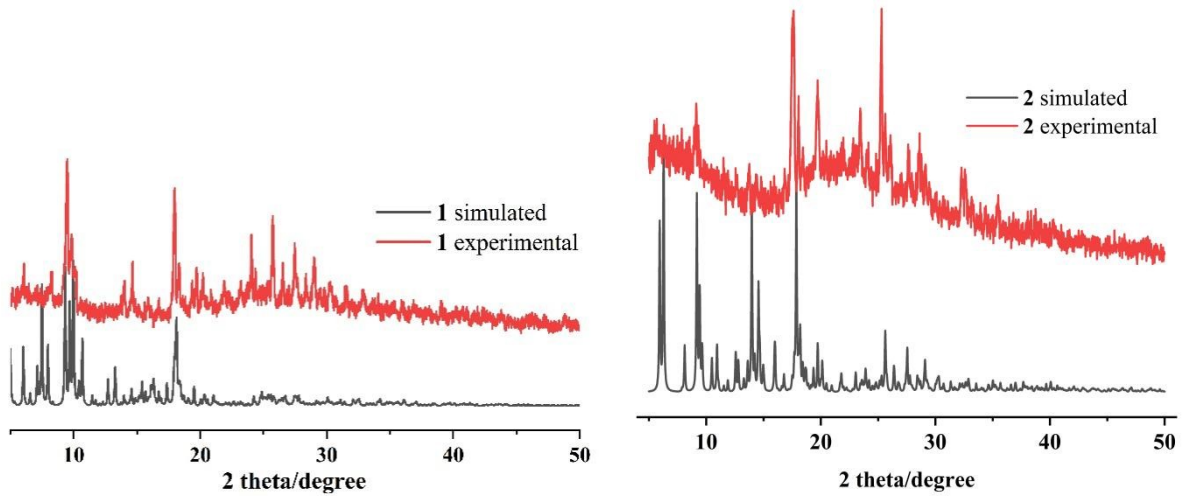


Figure S3. FT-IR spectra of assemblies **1-4**, Q[5] and ligand NaHANDS.



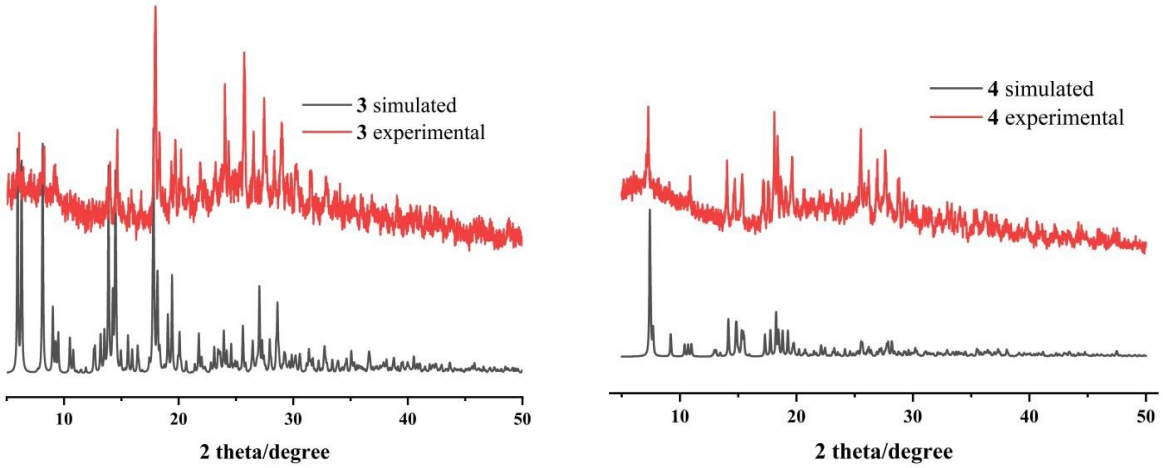


Figure S4. PXRD patterns of assemblies **1-4**.

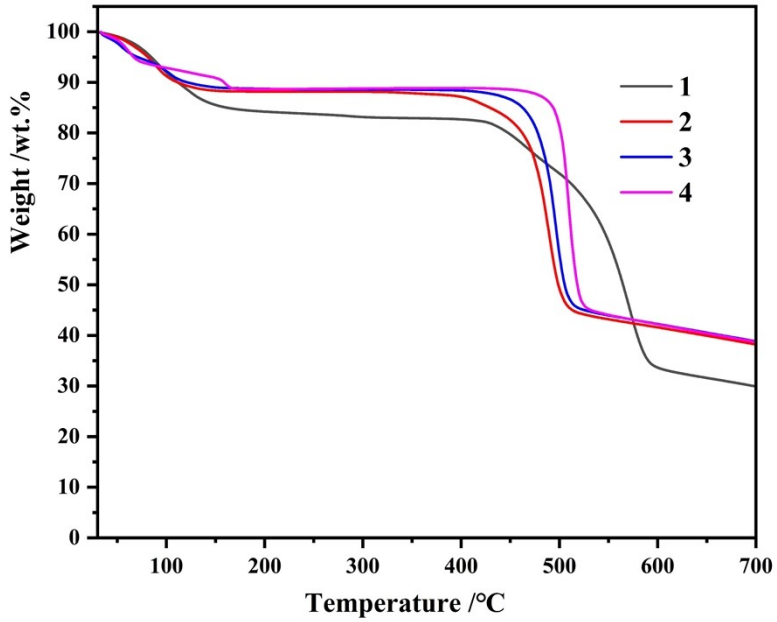


Figure S5. TG of assemblies **1-4**.

Table S3. Comparison on sensing performance of some previously reported sensors for CAP.

Sensor	Response model	LOD (μM)	*Ksv* (M^{-1})	References
Assembly **1**	Turn-off	6.12	1.418×10^4	This Work
MMIP-assisted biosensor	Turn-on	0.53	7.629×10^3	7
N,P-CDs	Turn-off	0.36	1.1×10^4	8
DNA aptamers	Turn-off	14	-	9
Eu-MOF	Turn-off	3.16	8×10^3	10
triangular gold nanoplates	colorimetric	5	-	11
Zn/Co-MOF	Turn-off	33.4	1.06×10^4	12
Zn/Ni-MOF	Turn-off	44.0	6.21×10^3	

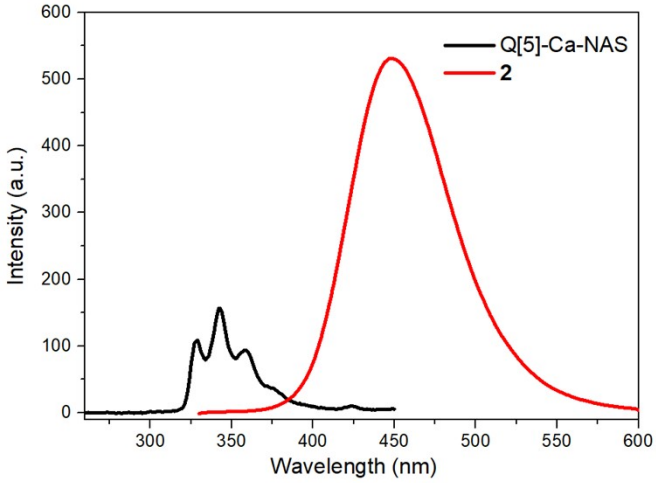


Figure S6. Fluorescence emission bands of assembly **2** and Q[5]-Ca-NAS.

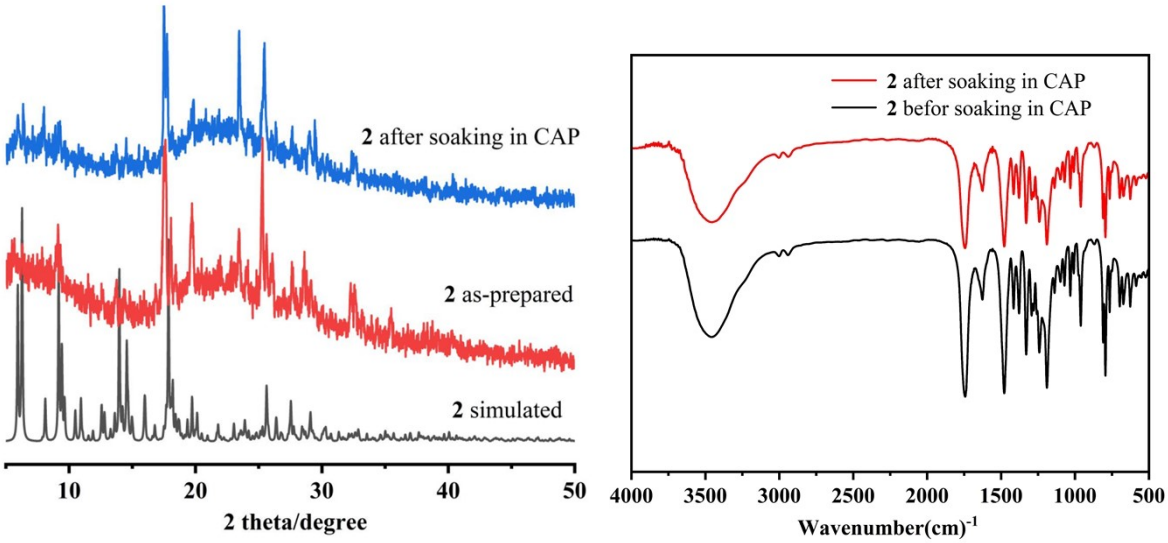


Figure S7. PXRD patterns (a) and FT-IR spectra (b) of assembly **2** before and after soaking in the solution of CAP.

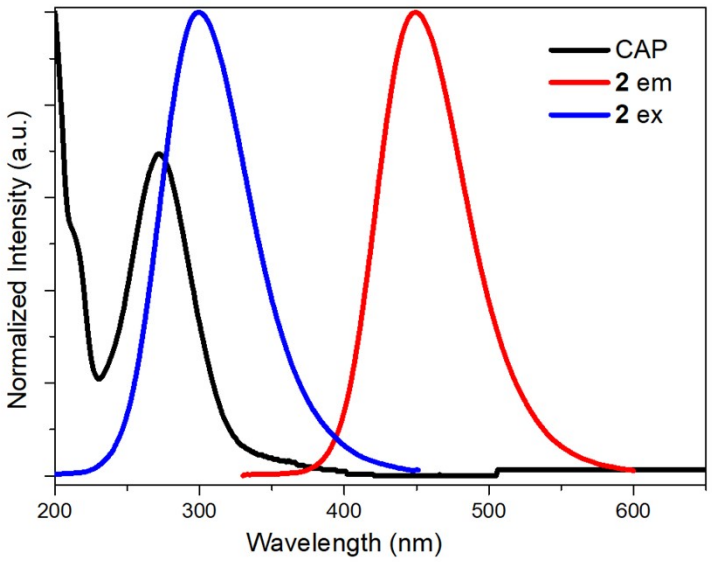


Figure S8. UV-vis absorption band of CAP, fluorescence excitation and emission bands of assembly **2**.

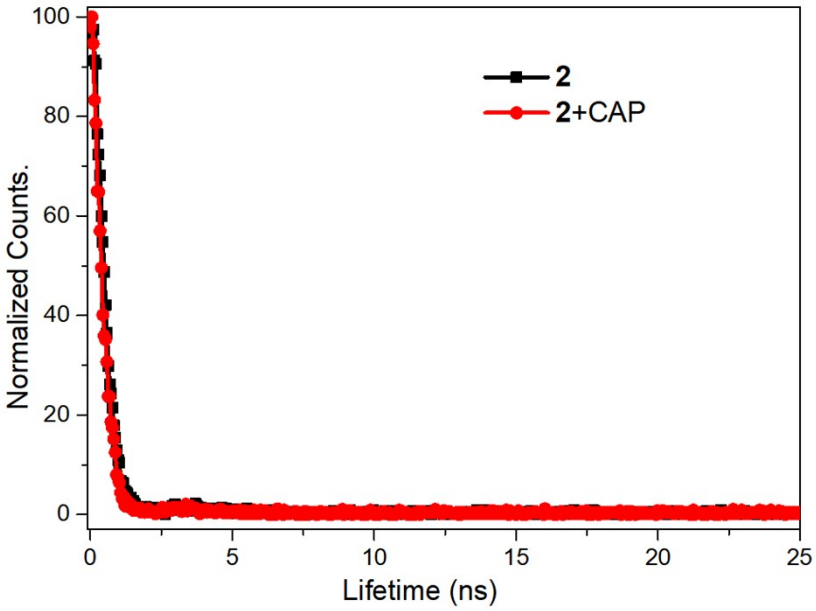


Figure S9. Fluorescence lifetimes of assembly **2** in the absence and presence of CAP.

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