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Electronic Supporting Information

Solvent-induced Reversible High-temperature Phase Transition in Crown Ether Clathrates

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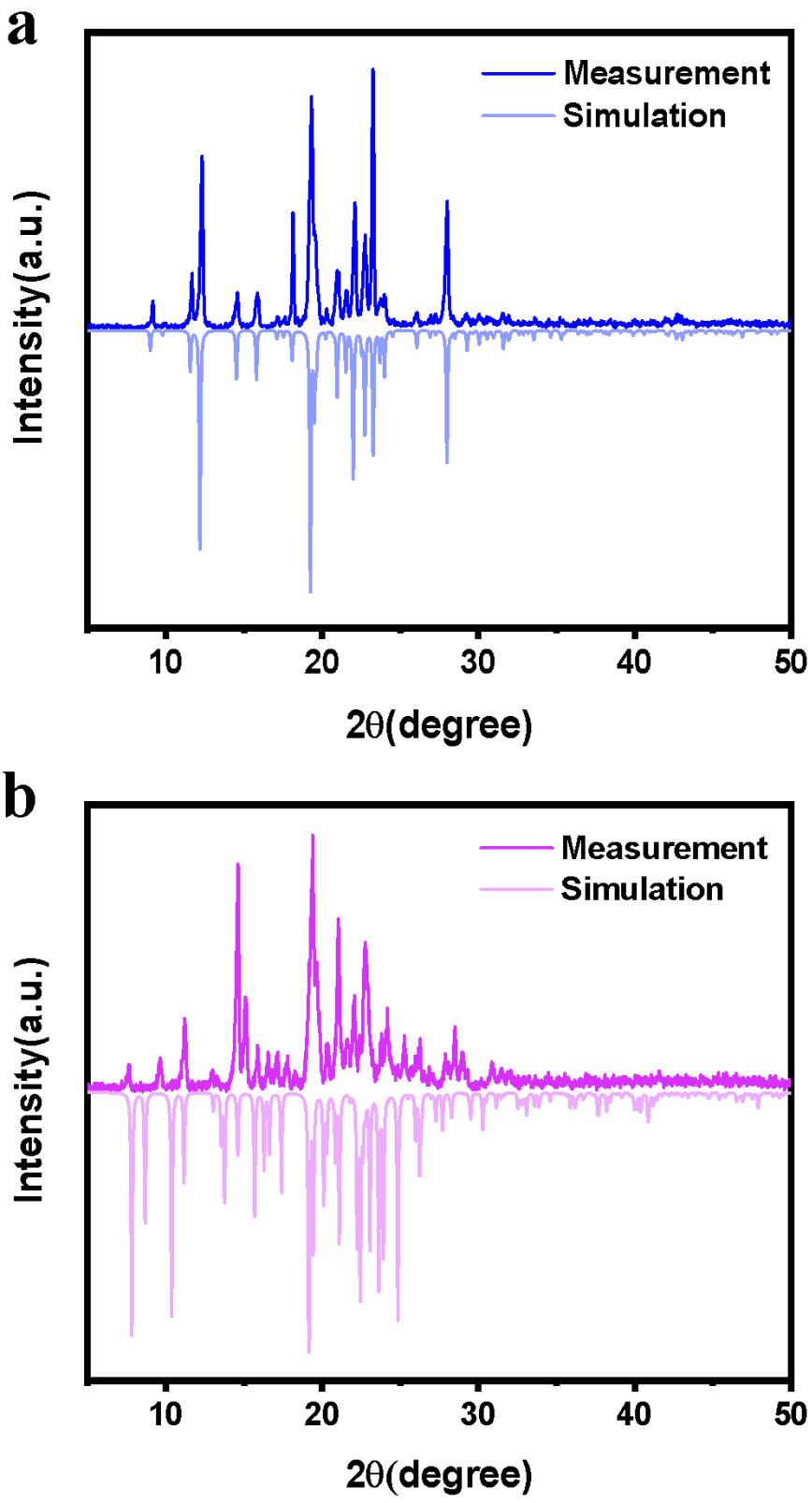

Fig. S1 The experimental and simulated PXRD patterns of compound 1 (a) and 1·ACN (b) at 298K.

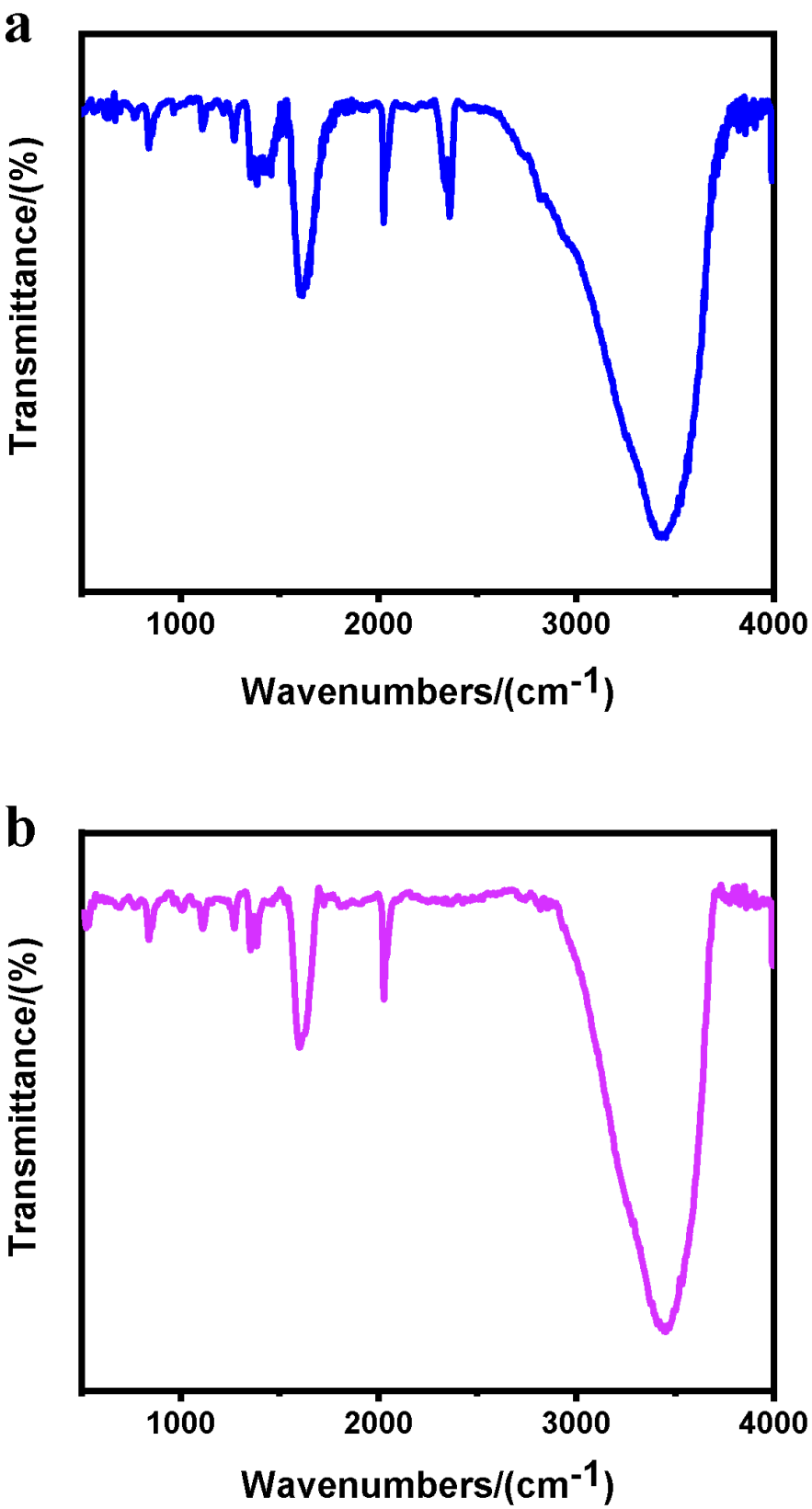

Fig. S2 The IR spectrum of compound **1** (a) and **1·ACN** (b).

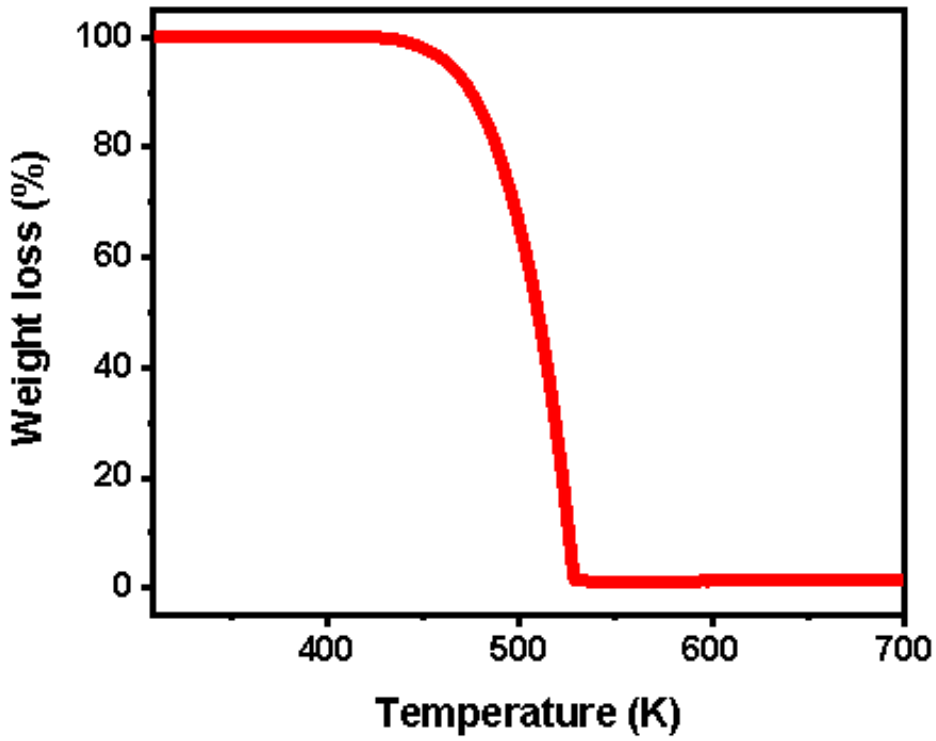

Fig. S3 TGA curve of compound **1·ACN.**

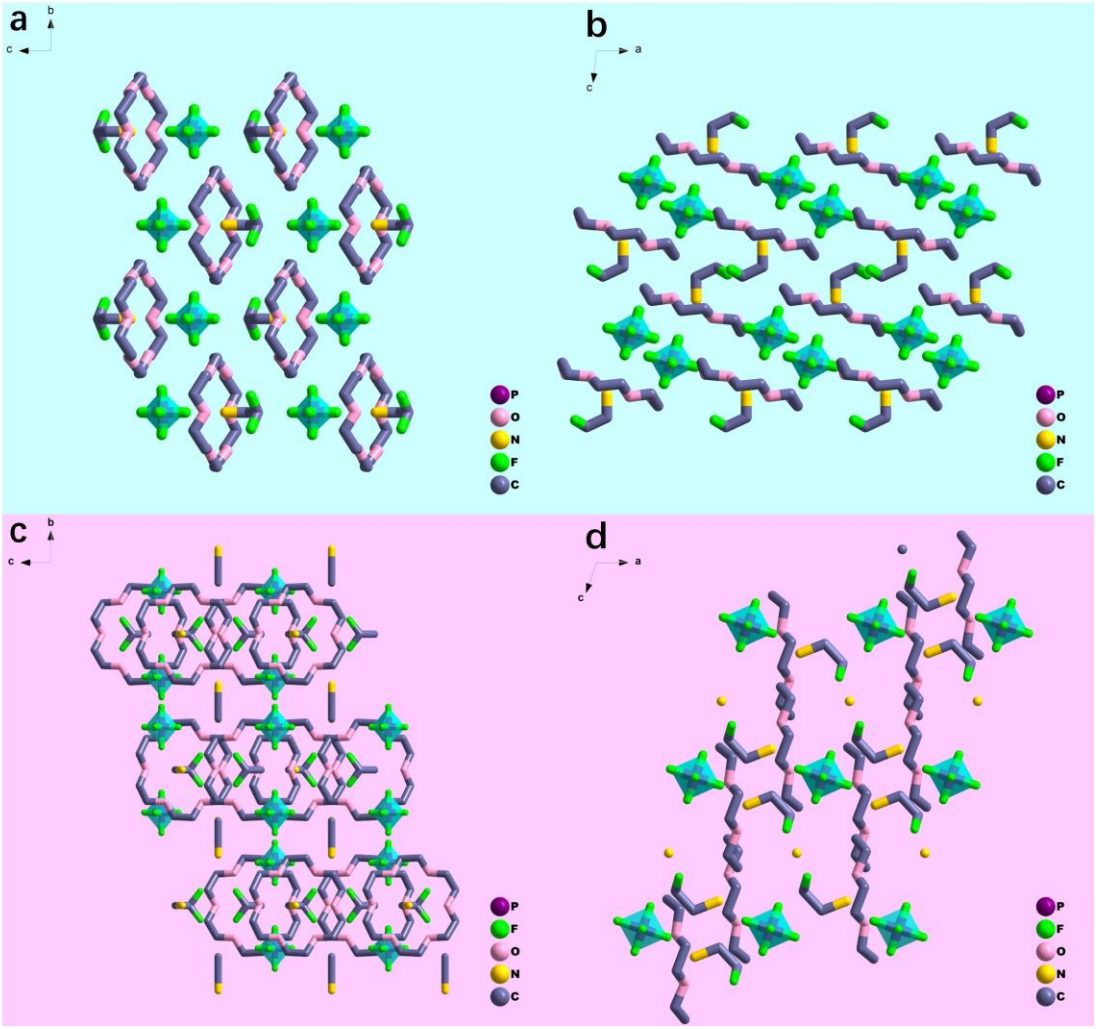

Fig. S4 The diagrams of the packing structures of compound **1** along the *a*-axis (a) and along the *b*-axis (b). The diagrams of the packing structures of compound **1·ACN** along the *a*-axis (c) and along the *b*-axis (d). Hydrogen atoms are omitted for clarity.

Tab. S1 Crystal data of compound **1** at 223 K and compound **1·ACN** at 208 K

	1	**1·ACN**
Formula	491.35	532.41
T (K)	223	208
Crystal system	monoclinic	monoclinic
Space group	*P*21/*m*	*I*2/*m*
a/Å	9.0833(9)	9.0149(4)
b/Å	12.1944(10)	20.3713(7)
c/Å	9.8627(10)	14.2323(6)
α (deg)	90	90
β (deg)	96.197(9)	107.599(5)
γ (deg)	90	90
V/Å^3	1086.06(18)	2491.36(19)
Z	2	4
F(000)	512.0	1112.0
radiation type	Mo Kα	Mo Kα
GOF	0.998	0.960
R1	0.0968	0.0915
wR2	0.2005	0.1706

Tab. S2 Selected bond lengths and bond angles for compound **1** at 223 K

Temperature	Bond lengths [Å]		Bond angles [°]	
223 K	P1—F5	1.533(4)	F3—P1—F6	178.0(3)
	P1—F3	1.498(4)	F6—P1—F2	96.9(3)
	P1—F6	1.522(4)	F5—P1—F2	178.7(2)
	O4—C6	1.409(4)	F3—P1—F2	85.0(2)
	O3—C5	1.421(4)	C4—O3—C5	111.9(3)
	O3—C4	1.417(4)	C2—O2—C3	113.7(3)
	O2—C3	1.432(5)	O4—C6—C5	109.3(3)
	O2—C2	1.429(4)	O3—C5—C6	109.0(3)
	O1—C1	1.424(5)	O3—C4—C3	109.0(3)
	F1—C7	1.358(4)	O1—C1—C2	110.6(3)
	N1—C8	1.478(5)	O2—C3—C4	108.5(3)
	C8—C7	1.466(6)	O2—C2—C1	107.8(3)
	C6—C5	1.479(5)	C7—C8—N1	114.4(3)
	C4—C3	1.485(6)	F1—C7—C8	110.4(3)

Tab. S3 Selected bond lengths and bond angles for compound **1·ACN** at 208 K and 296 K

Temperature	Bond lengths [Å]		Bond angles [°]	
208 K	P1—F3	1.493(7)	F3—P1—F1	180
	P1—F1	1.520(7)	F4—P1—F3	89.9(3)
	P1—F4	1.476(5)	F2—P1—F3	90.4(5)
	N1—C1	1.484(8)	F4—P1—F1	90.1(3)
	C10—C9	1.489(8)	F2—P1—F1	89.6(5)
	C8—C7	1.495(8)	C8—O3—C9	112.2(4)
	F5—C2	1.356(5)	C6—O2—C7	113.0(5)
	O3—C8	1.426(6)	C2—C1—N1	111.6(5)
	O3—C9	1.429(6)	F5—C2—C1	109.8(4)
	O1—C5	1.429(7)	O3—C9—C10	108.7(4)
	O2—C7	1.420(7)	O2—C6—C5	108.6(5)
296 K	P1—F3	1.527(3)	F6—P1—F3	87.2(2)
	P1—F5	1.508(5)	F4—P1—F6	180
	P1—F6	1.491(5)	F4—P1—F3	92.8(2)
	N1—C7	1.495(7)	C2—O2—C3	113.4(4)
	F1—C8	1.352(5)	C5—O3—C4	113.7(4)
	C10—N2	1.139(10)	C8—C7—N1	111.5(5)
	O4—C6	1.414(7)	O1—C1—C2	109.5(5)
	O3—C4	1.412(7)	O3—C4—C3	108.5(5)
	O2—C3	1.433(6)	O2—C3—C4	109.5(4)
	O1—C1	1.406(6)	O2—C2—C1	109.0(4)
	C4—C3	1.472(8)	O4—C6—C5	108.5(5)
	C6—C5	1.505(9)	O3—C5—C6	108.8(5)