Electronic Supplementary Information for CrystEngComm

Norfloxacin salts with benzenedicarboxylic acids: charge-assisted hydrogen-bonding recognition and solubility regulation

Xian-Feng Huang,[a,b,‡] Zhi-Hui Zhang,[a,‡] Qing-Qing Zhang,[a] Ling-Zhu Wang,[b] Ming-Yang He,[a] Qun Chen,*[,a] Guo-Qiang Song,*[,b] Lin Wei,[a] Fan Wang,[b] and Miao Du*[,c]

[a] *Key Laboratory of Fine Petrochemical Technology, Changzhou University, Changzhou 213164, P. R. China*

[b] *School of Pharmaceutical Engineering & Life Science, Changzhou University, Changzhou 213164, P. R. China*

[c] *College of Chemistry, Tianjin Key Laboratory of Structure and Performance for Functional Molecules, MOE Key Laboratory of Inorganic-Organic Hybrid Functional Material Chemistry, Tianjin Normal University, Tianjin 300387, P. R. China*

* Corresponding authors. Tel. & Fax: 86-519-86330251, E-mail: chenqunjpu@yahoo.com (Q. C.), drugs@vip.sina.com (G.-Q. S.); Tel. & Fax: 86-22-23766556, E-mail: dumiao@public.tpt.tj.cn (M. D.)

Table S1. Hydrogen bond metrics for compounds **1**–**3**.

Compound	D–H···A	H···A (Å)	D···A (Å)	D–H···A (°)
1	N1–H1*N*···O6^{i}	2.57	3.293 (6)	138
	N1–H1*N*···O7^{i}	1.78	2.653 (5)	164
	O2–H2···O3	1.74	2.503 (5)	154
	N1–H2*N*···O4	1.80	2.683 (5)	168
	O6–H6···O5	1.54	2.352 (4)	177
	C3–H3*B*···F1	2.26	2.872 (5)	120
	C4–H4*B*···O5^{ii}	2.59	3.277 (6)	128
	C16–H16*B*···O1^{iii}	2.37	3.150 (6)	137
	C23–H23···F1^{iv}	2.52	3.435 (6)	166
2	N1–H1*C*···O7^{i}	1.98	2.783 (4)	147
	N1–H1*D*···O7	1.79	2.681 (3)	170
	O2–H2···O1	1.71	2.480 (3)	154
	O4–H4···O3^{ii}	1.95	2.748 (3)	166
	C1–H1*A*···O2^{iii}	2.43	3.159 (4)	131
	C1–H1*B*···O6	2.39	3.098 (4)	129
	C2–H2*A*···F1	2.27	2.899 (4)	122
	C4–H4*A*···O3^{iv}	2.50	3.415 (5)	158
	C15–H15*B*···O6^{v}	2.59	3.457 (4)	148
	C20–H20···O5^{vi}	2.55	3.317 (4)	140
3	N1–H1*C*···O3^{i}	1.93	2.776 (3)	155
	N1–H1*D*···O4^{ii}	1.79	2.688 (3)	172
	O2–H2···O1	1.80	2.522 (3)	146
	O6–H6···O5^{iii}	1.86	2.667 (3)	167
	O8–H8···O5^{iv}	1.86	2.673 (3)	171
	C2–H2*B*···F1	2.16	2.846 (3)	126
	C4–H4*A*···O6^{v}	2.52	3.198 (3)	127
	C4–H4*B*···O2^{vi}	2.39	3.283 (4)	152
	C15–H15*B*···O7^{vii}	2.53	3.497 (4)	176
	C16–H16*B*···O8^{iv}	2.52	3.370 (4)	148

Symmetry codes: **1**, (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+2, -y, -z$; (iv) $x+1, y+1, z$. **2**, (i) $-x+1, -y+2, -z+1$; (ii) $x, y, z+1$; (iii) $-x+1/2, -y+2, z+1/2$; (iv) $x, -y+3/2, z+1/2$; (v) $x+1/2, y, -z+1/2$; (vi) $x-1/2, y, -z+3/2$. **3**, (i) $x, y, z-1$; (ii) $-x+2, -y+1, -z$; (iii) $x-1, y, z$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+1, -y+1, -z$; (vi) $-x+1, -y+2, -z+1$; (vii) $x+1, y, z$.

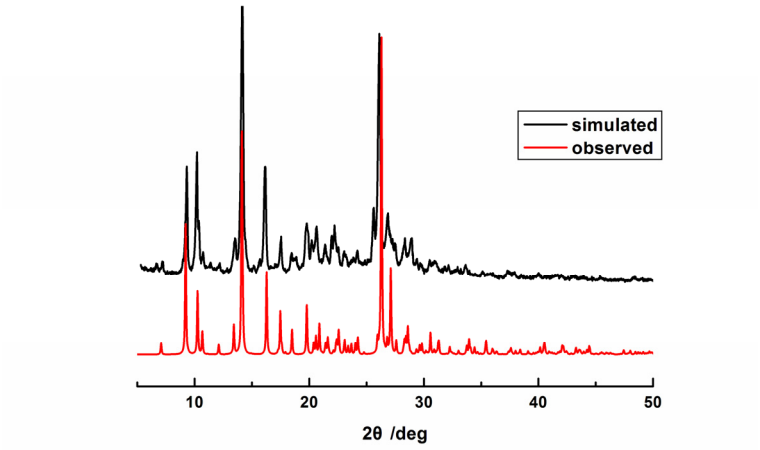
Table S2. Hydrogen bond metrics for compounds **4** and **5**.

Compound	D–H···A	H···A (Å)	D···A (Å)	D–H···A (°)
4	N1−H1*C*···O6^{i}	1.78	2.679 (2)	175
	N1−H1*D*···O7^{ii}	2.18	2.862 (2)	132
	N1−H1*D*···O5^{iii}	2.36	2.971 (2)	126
	O2−H2···O1	1.79	2.538 (3)	152
	N3−H3*D*···O5	2.04	2.693 (2)	132
	N3−H3*C*···O6	1.99	2.650 (2)	133
	O4−H4···O7^{ii}	1.84	2.589 (2)	152
	O8−H8*A*···O3^{iv}	2.36	2.978 (4)	133
	O8−H8*B*···O2^{v}	2.41	3.047 (5)	135
	C1−H1*A*···O5^{iii}	2.55	3.048 (3)	112
	C2−H2*A*···F1	2.26	2.915 (2)	124
	C4−H4*A*···O8	2.31	3.242 (4)	160
	C4−H4*B*···O4	2.37	3.251 (2)	150
	C15−H15*A*···O3^{vi}	2.49	3.369 (4)	150
5	N1−H1*C*···O5^{i}	1.87	2.763 (3)	172
	N1−H1*D*···O9^{ii}	1.84	2.734 (4)	172
	O2−H2···O1	1.75	2.500 (4)	151
	N3−H3*C*···O12^{iii}	2.29	3.105 (14)	157
	N3−H3*C*···O8	2.25	3.105 (5)	173
	O7−H7*A*···O5^{iv}	1.82	2.590 (3)	155
	O8−H8*A*···O10^{v}	2.41	3.062 (9)	137
	O9−H9*A*···O4^{i}	2.08	2.725 (5)	135
	O10−H10*A*···O11^{vi}	1.57	2.355 (17)	160
	O10−H10*B*···O8^{vii}	2.47	3.062(6)	130
	C2−H2*A*···F1	2.20	2.860 (4)	125
	C4−H4*A*···O2^{viii}	2.57	3.378 (4)	141

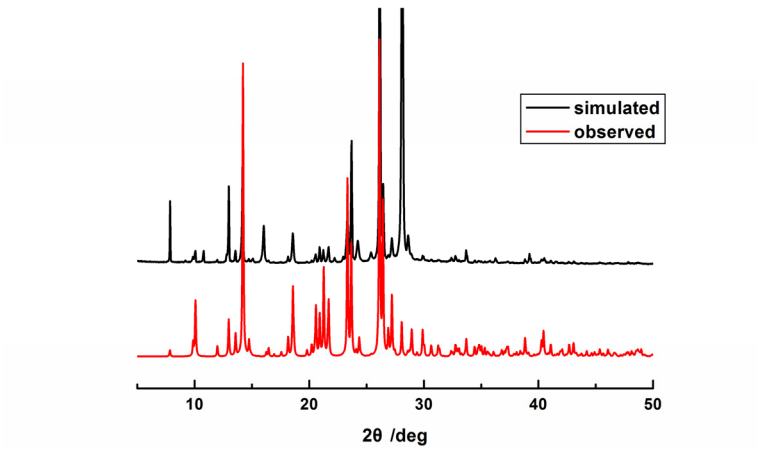
Symmetry codes: **4**, (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) *x*−1, *y*, *z*; (iii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iv) *x*−1, *y*−1, *z*; (v) −*x*+1, −*y*+2, −*z*; (vi) −*x*+2, −*y*+2, −*z*. **5**, (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*+1/2; (iv) −*x*, *y*−1/2, −*z*+1/2; (v) −*x*+1, *y*−1/2, −*z*+3/2; (vi) *x*, *y*, *z*+1; (vii) −*x*+1, *y*+1/2, −*z*+3/2; (viii) −*x*, −*y*+1, −*z*.

Table S3. Molar extinction coefficients of norfloxacin salts in various media (mL mg^{-1} cm^{-1}).

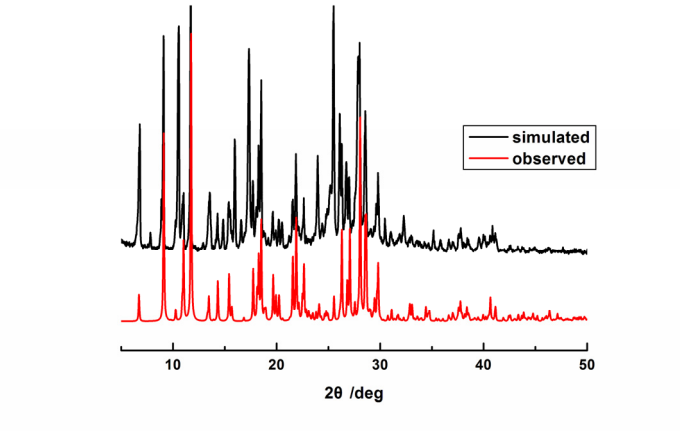
Medium	**Nf**	**1**	**2**	**3**	**4**	**5**
Pure water	107.9	86.1	51.6	93.4	44.9	64.2
0.1 M HCl	123.6	81.0	66.5	120.6	91.8	91.0



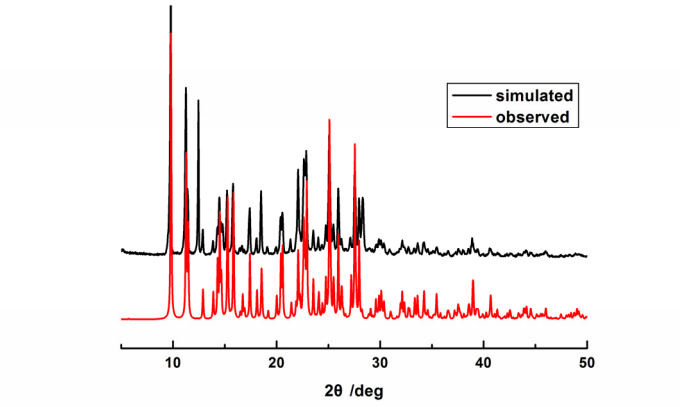
(a)



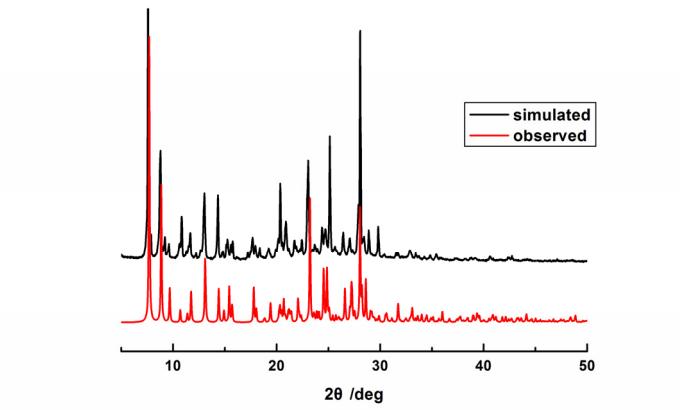
(b)



(c)

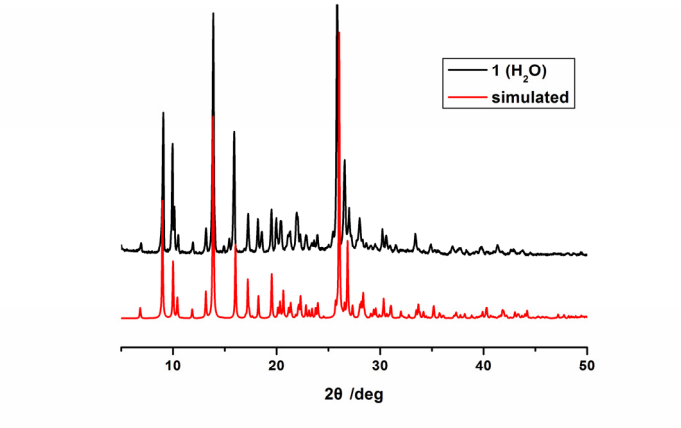


(d)

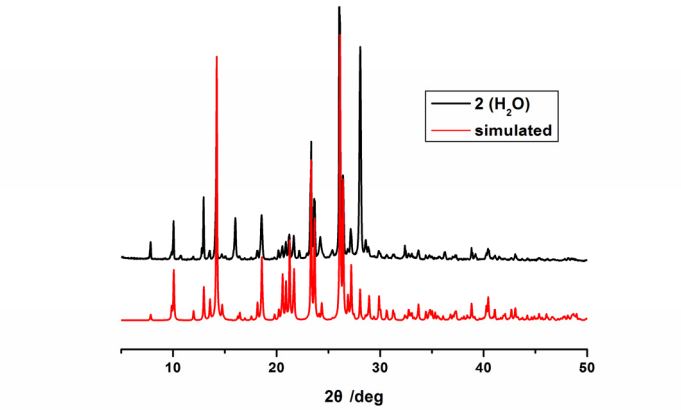


(e)

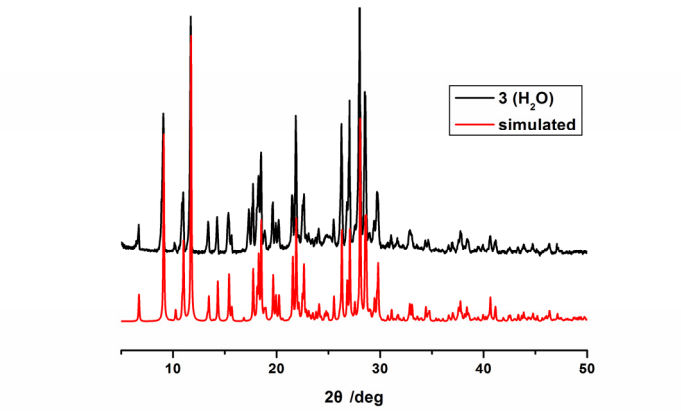
Fig. S1. Simulated (black) and observed (red) PXRD patterns for compounds **1**–**5 (a**–**e)**.



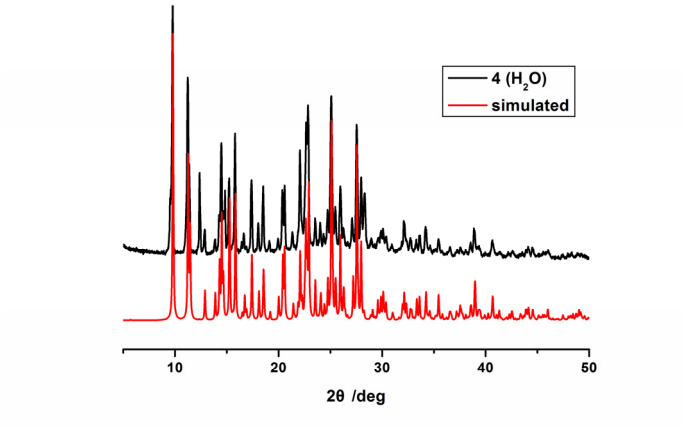
(a)



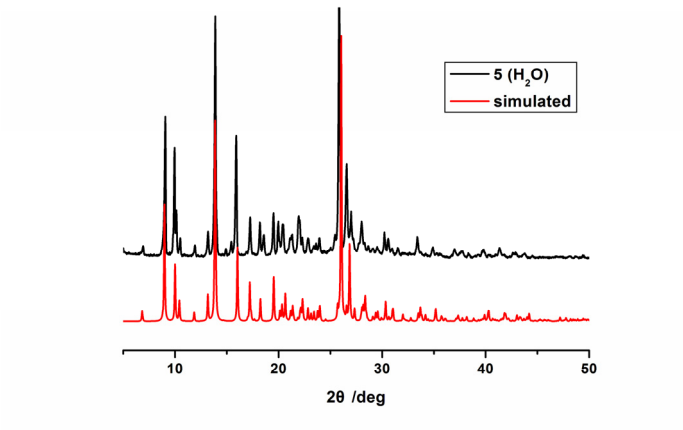
(b)



(c)

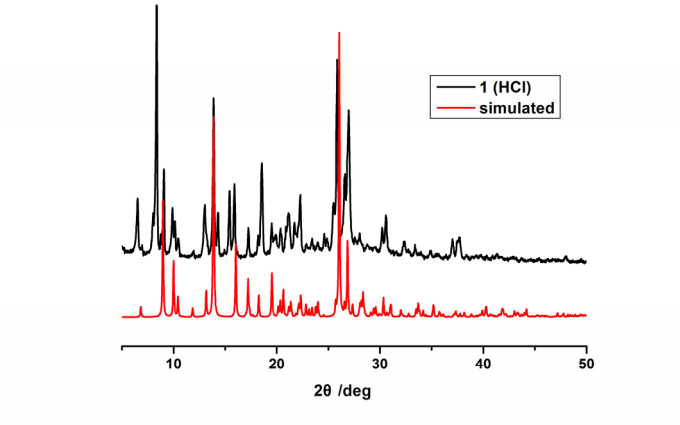


(d)

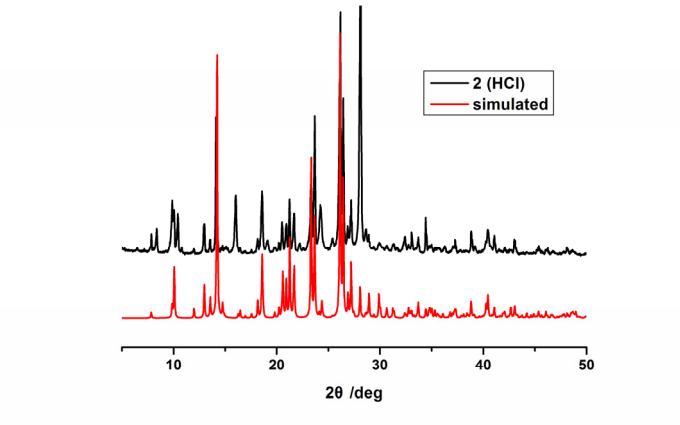


(e)

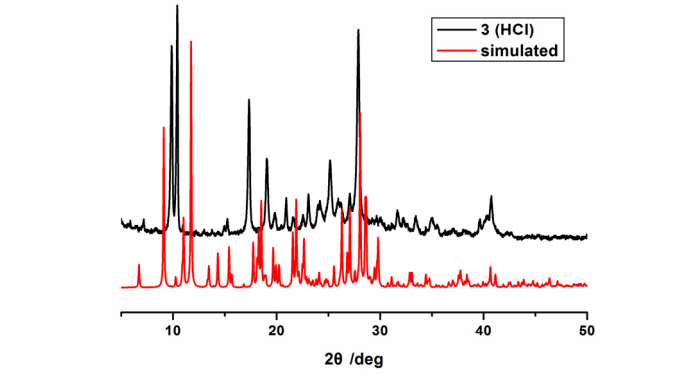
Fig. S2. Experimental (black) and simulated (red) PXRD patterns for compounds **1–5 (a–e)** after the dissolution experiment in pure water.



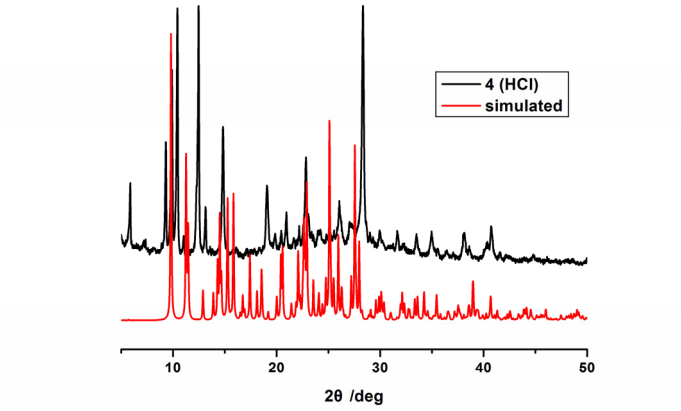
(a)



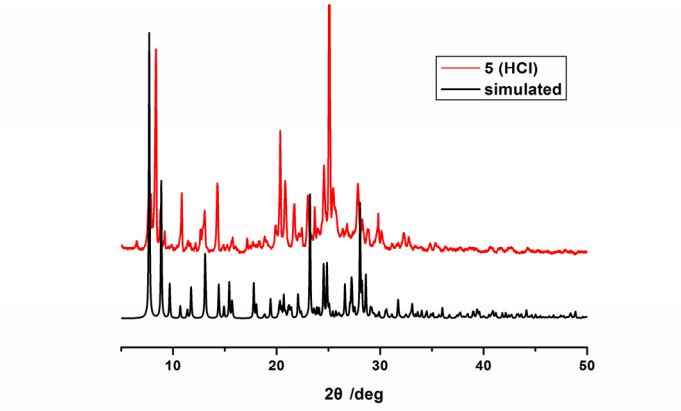
(b)



(c)

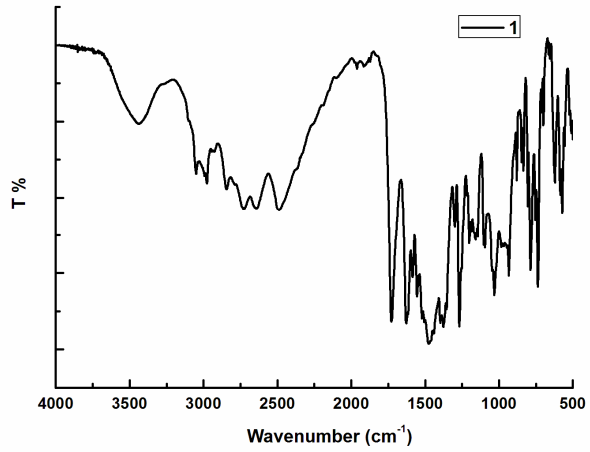


(d)

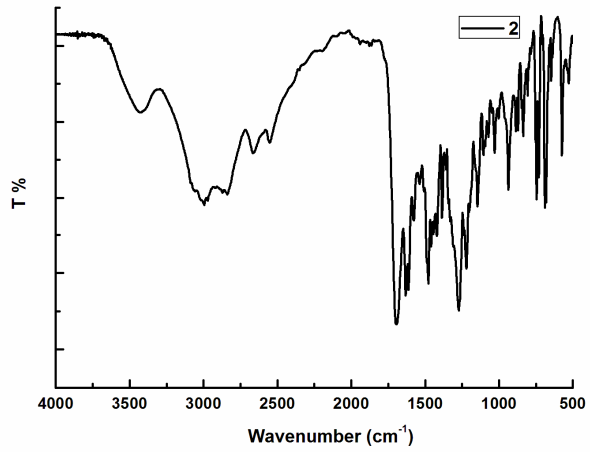


(e)

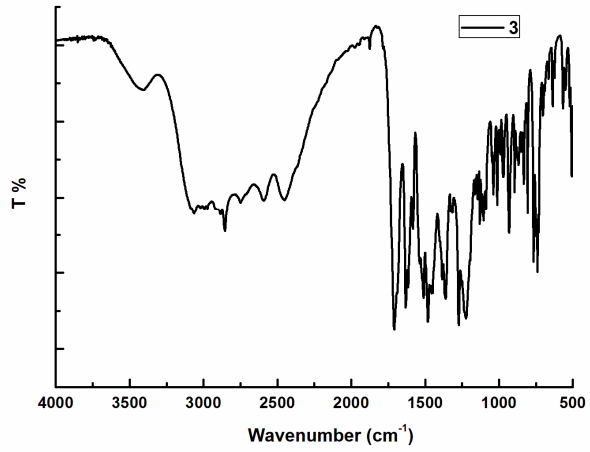
Fig. S3. Experimental (black) and simulated (red) PXRD patterns for compounds **1–5 (a–e)** after the dissolution experiment in 0.1M HCl.



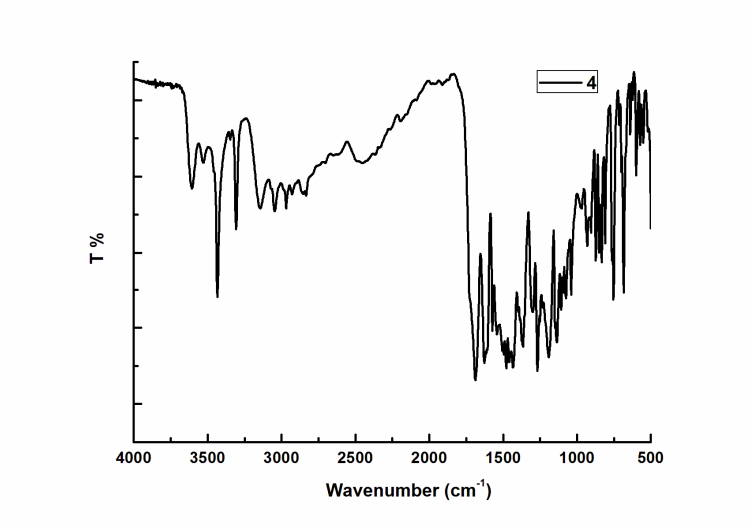
(a)



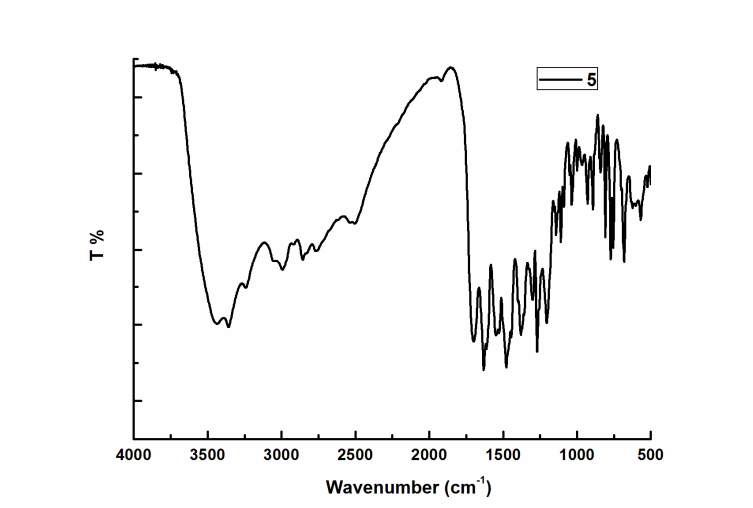
(b)



(c)



(d)



(e)

Fig. S4. Infrared spectra of norfloxacin salts **1**–**5 (a**–**e)**.

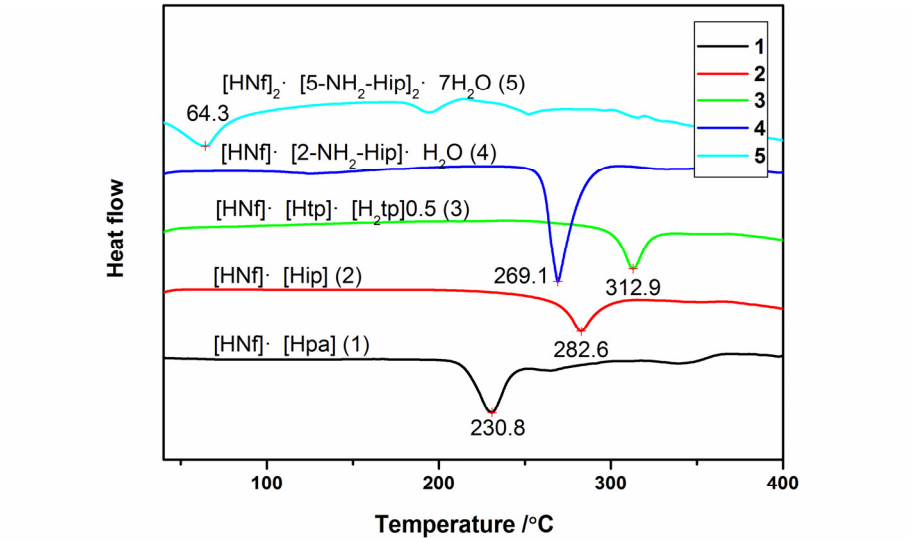


Fig. S5. DSC curves of norfloxacin salts **1**–**5**.