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Cocrystals of Quinoline Derivatives with 5-Aminoisophthalic Acid

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The structures of cocrystals of 5-aminoisophthalic acid (AIA) with various quinoline derivatives are investigated. The cocrystals of AIA with isoquinoline are comprised of dimers of carboxylic acids end capped by isoquinoline molecules. The repeat units have length 30.05 Å each. The hydrated cocrystals of AIA with quinoline and 8-hydroxyquinoline are structurally characterized and they have four and six coordination environment around water molecules respectively. The cocrystal of 4-hydroxyquinazoline with AIA has sheet like structure with repeated dimeric carboxylic acids possessing $R^2_2(14)$ type of H-bond patterns. In thermogrvimetry, the cocrystals of 5-aminophthalic acid with quinoline or 8-hydroxyquinoline loses the quinoline counterparts followed by decarboxylation of the parent acid, the decarboxylation takes place at 275°C–450°C.

Keywords 5-Aminoisophthalic acid; cocrystal; end capping; quinoline derivatives; tetra and hexa coordinated water; water assisted assembly

Introduction

Due to close correlation to nature, arrangements of small molecules in cocrystals have received considerable attention [1–8]. Identification of new synthons from such studies will make crystal engineering much more easier and predictable [9]. In this regard, the assemblies derived from carboxylic acid-aromatic amine are studied frequently to construct extended solid state architectures [10–18]. Most common among such assemblies are the carboxylic acid-pyridine or quinoline assemblies; which consist of primary O–H··N hydrogen bond interactions along with or without C–H··O interactions. Although, p*K*a, is used to predict formation of cocrystal or salt, the synthons having partial proton transfer causes complicacies [19, 20]. It is difficult to predict the formation of particular H-bonded motif; which leaves enough scope to study weak interactions in carboxylic acids, and aromatic heterocyclic systems. Other facet of such study is to gather information on the directional properties of weak interactions leading to new motifs. We choose to study here quinolines and 5-aminoisophthalic acid (AIA) cocrystals, as quinoline derivatives have relevance to nature and are relatively bigger size [21–23] with multiple aromatic units as compared to pyridine. On the other hand, AIA is an amino carboxylic acid, has well known ability to form supramolecular assemblies [24–27]. Thus, it can act as nodes having resemblance to natural amino acids in terms of functionality. The AIA may lead

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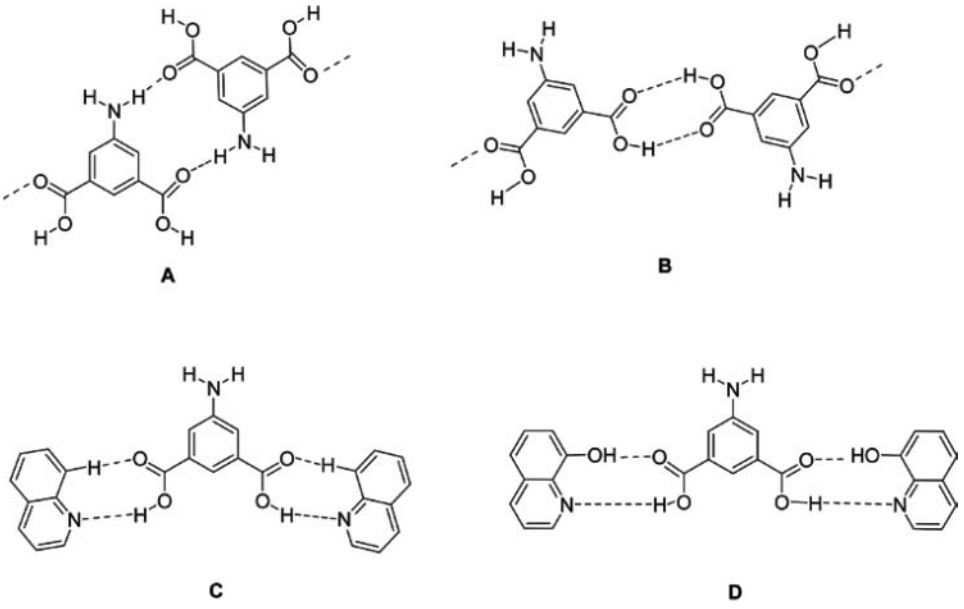

Scheme 1. Some homo and hetero-assemblies of 5-aminoisophthalic acid (AIA).

to self assembly through numbers of hydrogen bond patterns; two of which are illustrated in Scheme 1 (A, B). These assemblies may interact with other guest molecules such as aromatic amines either retaining a portion of its original character or through complete reorganization of the original interaction schemes. Representative cases that can happen are shown in Scheme 1 (C, D). In this study, structures of cocrystals of AIA with quinoline (**1**), isoquinoline (**2**), 8-hydroxyquinoline (**3**), and 4-hydroxyquinazoline (**4**) as shown in the chart 1 are studied. These led to interesting end terminated assemblies, and water assisted assemblies.

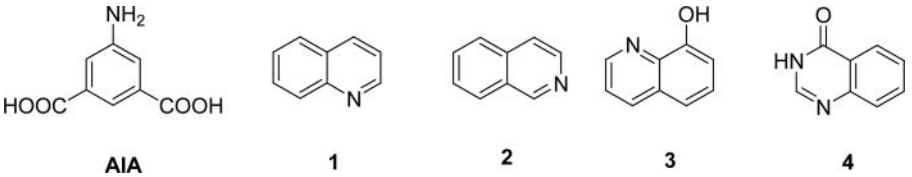

Chart 1. Structure of the molecules used in cocrystals and salt formation.

Results and Discussion

Quinoline forms cocrystal with AIA as $(AIA)_2.(quinoline)_2.\ H_2O$ (**5**); it crystallizes in monoclinic space group, C2/c. The asymmetric unit of **5** contains one quinoline molecule, one AIA along with half of a water molecule. One of the two acidic protons from one carboxylic acid group of AIA transfers to the amine group and the other carboxylic acid group remains intact. Thus, AIA exits as zwitterions; contains both COO^- and NH_3^+ group. The key structural feature of this cocrystal **5** is the involvement of the water molecule in

simultaneous H-bond with COO^- and NH_3^+ of the amino acid as shown in the Fig. 1(b). It can be mentioned here that a single water molecule holds four independent AIA molecules together. Another weak interaction stabilizing the cocrystal is C–H$\cdot\cdot\pi$ interaction between the aromatic ring of the acid molecule and C9–H on the quinoline molecule. When viewed along *b*-crystallographic axis it shows an alternate layered structure of acid, quinoline and water molecules. The acid molecules posses a layered structure in which the quinoline and

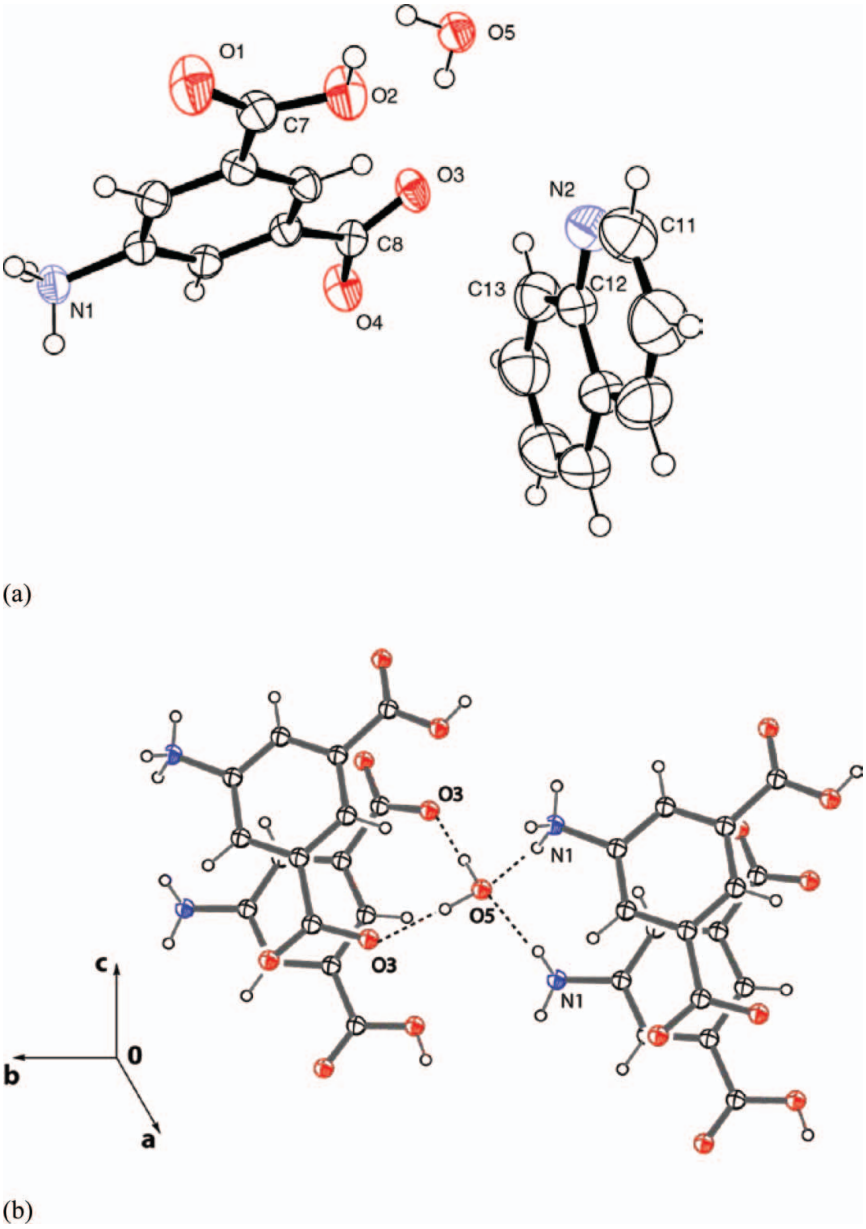

Figure 1. (a) Asymmetric unit of **5** (water has half occupancy); (b) Water assisted assembly among AIA molecules.

Table 1. Hydrogen bond parameters of **5**

D–H··A	$d_{D–H}$ (Å)	$d_{H··A}$ (Å)	$d_{D··A}$ (Å)	< D–H··A(^{0})
O(2)–H..O(3) [i]	0.874 (16)	1.621 (16)	2.4933 (15)	175.3 (17)
N(1)–H..O(4) [ii]	0.915 (15)	2.109 (17)	2.9273 (19)	148.3 (17)
N(1)–H..N(2) [iii]	0.924 (17)	1.861 (17)	2.772 (2)	168.3 (16)
N(1)–H..O(5)	0.90 (2)	1.94 (2)	2.8359 (18)	171.9 (19)
O(5)–H..O(3) [iv]	0.861 (17)	2.044 (17)	2.8976 (15)	171 (2)

[i] = $x, -y, -1/2 + z$; [ii] = $x, 1 - y, -1/2 + z$; [iii] = $1/2 - x, 1/2 + y, 1/2 - z$; [iv] = $x, 1 + y, z$.

water molecules are held between the layers. Selected H-bonding parameters contributing to the layers are shown in the Table 1. While forming water assisted hydrogen bonded assembly, amino acids generally transform to zwitterionic forms [28–30]. Thus, our system may be compared to the water assisted assemblies that have zwitterionic forms and found in nature.

The cocrystal (AIA).(isoquinoline) **6** crystallizes in triclinic space group, P-1 and the asymmetric unit contains one AIA molecule and one isoquinoline molecule [Fig. 2(a)].

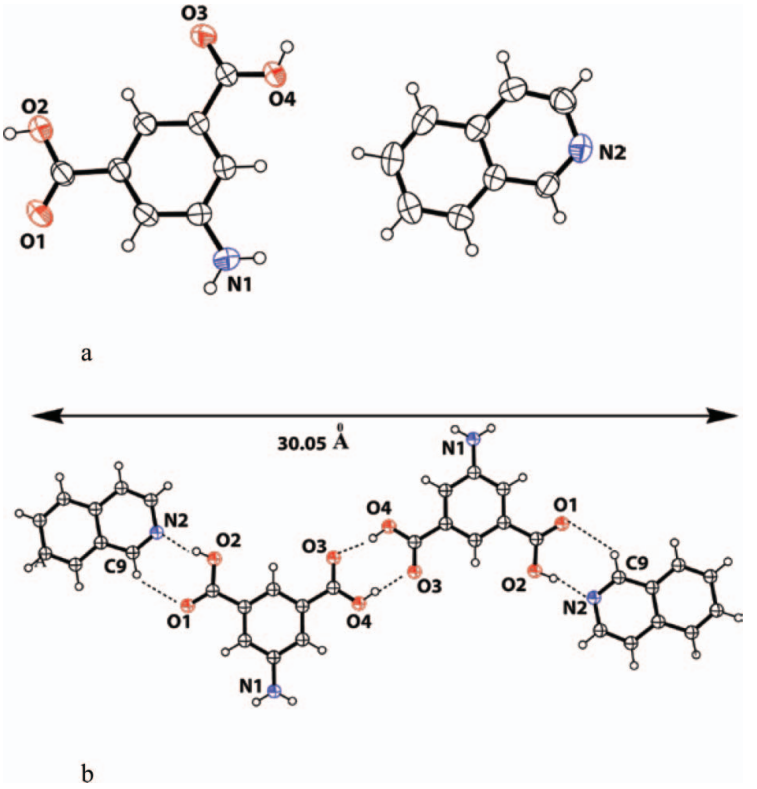

Figure 2. (a) Asymmetric unit of the cocrystal **6**; (b) End capped dimer of AIA.

Table 2. Hydrogen bond parameters of **6**

D–H··A	$d_{D–H}$ (Å)	$d_{H··A}$ (Å)	$d_{D··A}$ (Å)	< D–H· A(^{0})
O(2)–H..N(2) [i]	0.91 (3)	1.74 (3)	2.648 (2)	176 (3)
O(4)–H..O(3) [ii]	0.92 (3)	1.70 (3)	2.615 (2)	171 (4)
C(9)–H..O(1)	0.93	2.51	3.208 (3)	132

[i] = $1 + x, y, -1 + z$. [ii] = $1 - x, -y, 1 - z$.

The crystal structure shows repeated motifs comprising of two acids and two isoquinoline molecules [Fig. 2(b)]. The interesting feature of the molecule is that here the AIA molecules remain as dimeric pairs. Extensions of such dimers are blocked by two isoquinoline molecules positioned at two ends through weak interactions [Fig. 2(b)]. Two acid molecules are held together by cyclic $R^2_2(8)$ type of hydrogen bonds. The other two ends are held by C9–H···O1 and O2–H···N2 interactions making cyclic $R^2_2(7)$ type of structures. The dimeric dicarboxylic acids capped by isoquinoline, has 30.05 Å length between two ends. The selected H-bonding parameters are listed in the Table 2.

The cocrystal of AIA with 8-hydroxyquinoline has a composition $(AIA)_2$.(8-hydroxyquinoline)$_2$·H_2O (**7**). It crystallizes in monoclinic space group, C2/c. The asymmetric unit contains one 8-hydroxyquinoline molecule, one AIA along with half of a water molecule. Here, also the amino acid exists as zwitterion which contains both COO^- and NH_3^+ as in the case of cocrystal **5** and the involvement of the water molecule in simultaneous H-bonding with COO^- and NH_3^+ of the amino acid is observed. Unlike in cocrystal **5**, in cocrystal **7** six independent acid molecules instead of four AIA molecules are held together by a single water molecule [Fig. 3(b)]. In this case, i.e., cocrystal **7** one may call the assembly to comprise of N_2O_4 environment, whereas in the case of **5** one may call as N_2O_2 environment. This classification would lead to imagination and description of types of such environments to make a new chemistry altogether around water molecules.

The C—H . . . π interaction between the aromatic ring of the acid molecule and C9–H on the 8-hydroxyquinoline is observed in lattice of cocrystal **7**. This cocrystal shows layered structure as in the case of **5** which incorporates water and 8-hydroxyquinoline molecules between the acid layers. The selected H-bonding parameters are shown in the Table 3.

A 1:2 cocrystal obtained from AIA and 4-hdroxyquinazoline **8** crystallizes in triclinic space group, P-1 and the asymmetric unit contains one AIA and two 4-hydroxuquinazoline molecule (A and B). The self-assembly of the cocrystal may be described as assemblies of repeated dimeric carboxylic acids with a $R^2_2(14)$ type of H-bond pattern. There are two different types of 4-hydroxquinazoline molecules present in the lattice which are linked as dimeric structures generated through $R^2_2(8)$ type interactions among the keto form of the 4-hydroxyquinazoline [Fig. 4(b)]. Each set of these dimers are held by O–H . . . N interactions to form sheet like structure. The other weak interaction seen in this structure is the π-π stacking interaction among the 4-hydroxyquinazoline marked as A in Fig. 4(a). The parallel plane to plane π-π distance is found to be 3.38 Å. Again, between the two molecules A and B of 4-hydroxyquinazoline a C22—H . . . π (A) and C14–H . . . O6 interaction appears whose distances are found to be 2.88 Å and 2.47 Å respectively. Selected H-bond parameters are shown in the Table 3.

Thermogravimetry of the samples **5** and **7** have similar trends in decomposition (Fig. 5). The cocrystal **5** loses weight in two steps on heating, in the first step between 80°C and 170°C, 42.9% weight loss occurs due to the loss of two molecules of quinoline and one

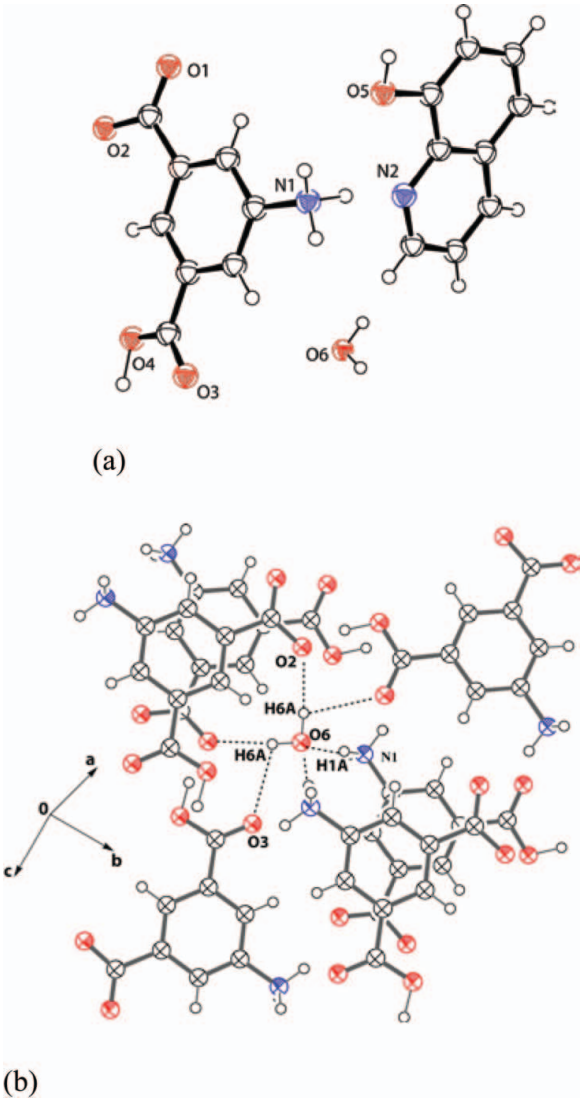

Figure 3. (a) Asymmetric unit of the cocrystal **7** (occupancy of water molecule is half); (b) Water assisted assembly of six molecules of AIA molecules.

molecule of water (theoretical weight loss 41%). In the second step between temperatures range 275°C–450°C, 69% weight loss occurs from parent composition (theoretical weight loss 68.6%) due to the decarboxylation of AIA and after that a continuous degradation occurs. The cocrystal **7** is stable up to 100°C. When heated further, it loses 44% weight in temperature range 100°C–175°C, due to the loss of two molecules of 8-hydroxyquinoline and one molecule of water (theoretical weight loss 46.0%). In the second step between temperatures 275°C–450°C the decarboxylation of the AIA takes place and this weight loss is 72% of weight loss from parent composition (theoretical weight loss 71%) and after that a continuous degradation occurs.

Table 3. Hydrogen bond parameters of **7** and **8**

Cocrystal	D–H··A	$d_{D—H}$ (Å)	$d_{H··A}$ (Å)	$d_{D··A}$ (Å)	< D–H··A(^{0})
7	N(1)–H..O(6)	0.92 (3)	1.97 (3)	2.889 (2)	178 (2)
	N(1)–H..O(1)[i]	0.94 (2)	2.15 (3)	2.978 (2)	146 (2)
	N(1)–H..O(5)	0.97 (2)	2.46 (2)	2.978 (2)	113.1 (15)
	N(1)–H..N(2)	0.97 (2)	1.82 (2)	2.785 (2)	171.9 (18)
	O(5)–H..O(2) [ii]	0.82	2.01	2.794 (2)	160
	O(4)–H..O(2) [iii] (intra)	0.82	1.71	2.5197 (19)	172
	O(6)–H..O(2) [ii]	0.90 (3)	2.19 (3)	3.0788 (17)	169 (3)
	O(6)–H..O(3) [iv]	0.90 (3)	2.50 (3)	2.9285 (14)	110 (2)
	C(6)–H..O(1)	0.93	2.26	3.050 (2)	143
8	N(1)–H..O(5) [v]	0.86	2.32	3.143 (3)	161
	N(1)–H..O(3) [vi]	0.86	2.20	3.032 (4)	164
	O(2)–H ..N(4)	0.87 (3)	1.91 (4)	2.767 (4)	170 (4)
	N(3)–H..O(5) [vii]	0.94 (4)	1.94 (4)	2.847 (4)	163 (3)
	O(4)–H(4A)..N(2)[ii]	0.87 (2)	1.83 (2)	2.690 (3)	172(3)
	N(5)–H(5N)..O(6) [viii]	0.97 (4)	1.87 (4)	2.834 (4)	174 (3)

Symmetry code: [i] = x, $1-y$, $-1/2+z$. [ii] = x, $-1+y$, z. [iii] = x, $2-y$, $-1/2+z$; [iv] = x, $1-y$, $1/2+z$. [v] = $1+x$, y, z. [vi] = $2-x$, $-y$, $-z$. [vii] = $-x$, $1-y$, $-z$, [viii] = $-x$, $1-y$, $1-z$.

In conclusion, we have shown formation of 30.05 Å repeated blocks of dimeric pair of AIA molecules end capped by isoquinoline molecules, through H-bond interactions. It is also shown that the water of crystallization helps in formation new types of assemblies with hexa-coordinate or tetra-coordinated water in different environments. This opens further scope to make various types of coordination arrangements around water molecules to discern them. Such study not only will enrich the understanding of new water environments;

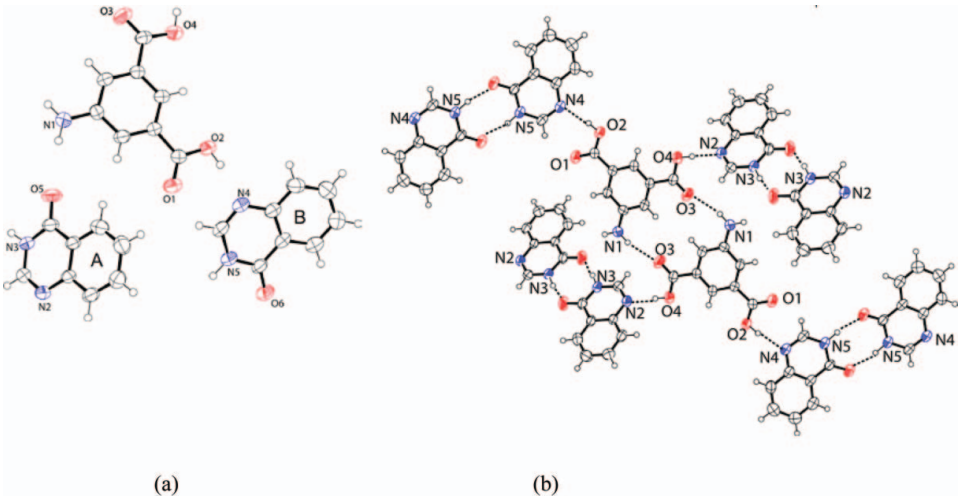

Figure 4. (a) Asymmetric unit of the cocrystal **8**; (b) hydrogen bonds in lattice of **8** (30% thermal ellipsoid)

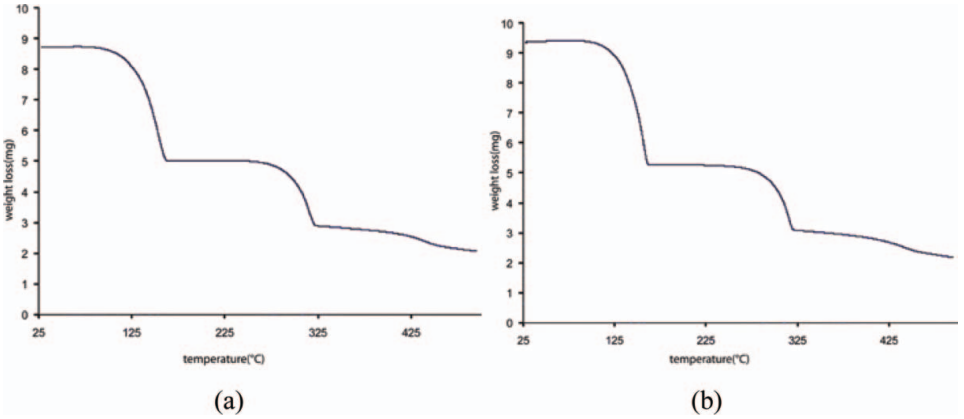

Figure 5. TGA of cocrystals of 5-aminoisophthalic acid (AIA) with (a) quinoline (**5**); (b) 8-hydroxyquinoline (**7**) (Heating rate 20°C min^{-1})

but also it would provide means for designating water environments commonly encountered in supramolecular chemistry.

Experimental

Preparation of Cocrystals

Cocrystal **5** and **6** were prepared in similar procedures. AIA (0.276 g, 2 mmol) and quinoline (0.236 ml, 2 mmol) or isoquinoline (0.235 ml, 2 mmol) were dissolved in methanol (10 ml). The solutions were then heated to 50°C in a conical flask for 10 min and kept undisturbed at room temperature for crystallization. After 3–4 days colorless needle like crystals of **5** and yellow colored needle like crystal of **6** were observed. The crystals were collected by filtration and then washed with little amount of methanol. Cocrystal **7** and **8** were prepared in similar procedures. AIA (0.276 g, 2 mmol) and of 8-hydroxyquinoline (0.290 g, 2 mmol) or 4-hyroxyquinazoline (0.584 g, 4 mmol) were dissolved in DMSO (10 ml). The solutions were then heated up to 70°C for 10 min and kept undisturbed at room temperature for crystallization. After one week light yellow block for **7** and light brown block for **8** crystals were observed. The solution was filtered out to get the crystals and finally we washed the crystals with little amount of dimethylsulphoxide.

IR data for cocrystals of AIA with:

Quinoline (**5**) (KBr, cm^{-1}): 3427 (bs), 3082 (s), 2925 (s), 1707 (s), 1629 (s), 1593 (s), 1375 (s), 1315 (s), 1262 (s), 1220 (s), 1123 (s), 998 (s), 809 (s), 783 (m), 752 (m). Isoquinoline (**6**) (KBr, cm^{-1}): 3415 (s), 3346 (s), 2925 (w), 1683 (s), 1629 (s), 1456 (s), 1424 (s), 1331 (s), 1277 (s), 1172 (s), 1037 (s), 963 (m), 881 (m), 863 (m), 824 (s), 760 (s), 738 (s), 697 (m), 661 (m), 690 (m), 671 (m), 624 (m). 8-Hydroxyquinoline (**7**) (KBr, cm^{-1}): 3436 (bs), 1692 (s), 1626 (s), 1593 (s), 1519 (s), 1470 (s), 1434 (s), 1380 (s), 1335(s), 1275 (s), 1227 (s), 1162 (s), 1095 (s), 1061 (s), 925 (s), 822 (m), 774 (m), 754 (m), 720 (s), 689 (s), 584 (s). 4-hydroxyquinazoline (**8**) (KBr, cm^{-1}): 3438 (bs), 2922 (w), 1709 (s), 1678 (s), 1440 (s), 1389 (s), 1332 (s), 1278 (s), 1241 (s), 1124 (s), 1019 (s), 952 (m), 926 (m), 868 (m).

Table 4. Crystallographic parameters for cocrystals **5–8**

Compound no.	**5**	**6**	**7**	**8**
Formulae	C_{34} H_{30} N_4 O_9	C_{17} H_{14} N_2 O_4	C_{34} H_{30} N_4 O_{11}	C_{24} H_{19} N_5 O_6
CCDC No.	829470	827763	827764	827761
Mol. wt.	638.62	310.30	670.62	473.44
Crystal system	Monoclinic	Triclinic	Monoclinic	Triclinic
Space group	C2/c	P-1	C2/c	P-1
Temperature /K	296(2)	293 (2)	296 (2)	296 (2)
Wavelength /Å	0.71073	0.71073	0.71073	0.71073
a /Å	25.990 (3)	3.7779 (3)	27.2188 (19)	3.7984 (3)
b /Å	8.7640 (9)	14.0430 (10)	8.7246 (6)	14.4180 (14)
c /Å	13.9254 (15)	14.9689 (11)	14.2446 (15)	19.4399 (18)
α/°	90.00	66.852 (5)	90.00	88.442 (6)
β/°	110.081 (12)	89.421 (6)	116.518 (7)	88.099 (6)
γ/°	90.00	86.837 (5)	90.00	89.836 (5)
V/ Å^3	2979.1 (6)	729.04 (9)	3026.8 (4)	1063.65 (17)
Z	4	2	4	2
Density/Mgm^{-3}	1.424	1.404	1.472	1.478
Abs. Coeff. /mm^{-1}	0.105	0.102	0.112	0.109
Abs. correction	None	None	None	None
F(000)	1336	320	1400	492
Total no. of reflections	2569	2588	2618	3255
Reflections, $I > 2\sigma$ (*I*)	2077	1783	2189	1943
Max. θ/°	25.25	25.25	25.00	25.24

Ranges (h, k, l)	$-27 \leq h \leq 28$ $-10 \leq k \leq 10$ $-16 \leq l \leq 16$	$-4 \leq h \leq 4$ $-16 \leq k \leq 16$ $-17 \leq l \leq 17$	$-32 \leq h \leq 32$ $-10 \leq k \leq 10$ $-16 \leq l \leq 16$	$-4 \leq h \leq 4$ $-16 \leq k \leq 17$ $-22 \leq l \leq 21$
Complete to 2θ (%)	94.9	97.2	98.1	84.00
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/Restraints/ parameters	2569/14/233	2588/2/216	2618/1/240	3255/2/332
Goof (F^2)	1.094	0.962	1.054	0.979
R indices [$I > 2\sigma(I)$],	0.0371	0.0496	0.0385	0.0525
R indices (all data),	0.0488	0.0752	0.0465	0.0952

The X-ray single crystal diffraction data were collected with MoK_α radiation (λ = 0.71073 Å) using a Bruker Nonius SMART CCD diffractometer equipped with a graphite monochromatic. The SMART software was used for data collection and also for indexing the reflections and determining the unit cell parameters; the collected data were integrated using SAINT software. The structures were solved by direct methods and refined by full-matrix least-squares calculations using SHELXTL software. All the non-H atoms were refined in the anisotropic approximation against F^2 of all reflections. A part of H-atoms attached to nitrogen and oxygen were located in difference Fourier maps and refined with isotropic displacement coefficients, other H-atoms were placed at their calculated positions.

The crystal parameters of the crystals are shown in Table 4.

Supplementary Materials

The CIFs are deposited to the Cambridge Crystallographic database and have the CCDC Nos 829470, 827761, 827763, and 827764.

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