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ORIGINAL ARTICLE

Hydrogen bonded assemblies of 1,8-naphthyridine derivatives: discrete or polymeric structures in the solid state

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Abstract A series of 1,8-naphthyridine derivatives, consist of a number of hydrogen bonding donor and acceptor sites, are found to exhibit interesting hydrogen bonded assemblies in the solid state. Placement of different types of functionalities around the 1,8-naphthyridine motif via simple synthetic methodologies can easily change the hydrogen bonding patterns involving naphthyridine as hydrogen bonding building block. Discrete or polymeric assemblies are observed while the substituents around the naphthyridine nucleus are varied. Water assisted dimeric structure is found in pyridine appended naphthyridine system and all the structures are determined by X-ray crystallographic analysis.

Keywords Self-assembly · Weak interaction · Molecular crystals · Naphthyridine · Polymeric structure · Water assisted dimer

Introduction

Engineering specific noncovalent interactions in the designed molecules with a number of hydrogen bond donors and acceptors to generate various types of hydrogen bonding structures is an interesting aspect in supramolecular chemistry [1–6]. In this regard, 1,8-naphthyridine, a simple heterocycle with two ring nitrogens as hydrogen bond acceptors, can serve as potential hydrogen bonding building block and has been widely used by various groups in the area of molecular recognition research [7, 8]. Zimmerman and co-workers [9] have used this motif with different functionalities in the dendrimer chemistry as well as in the supramolecular polymer chemistry. In their work, they have also established the folding and unfolding behaviors of 1,8-naphthyridinylureas. Goswami et al. [10] in their recent publications have shown that 2-oxo-1,8-naphthyridine system and 2,7-diamino-1,8-naphthyridine systems or their suitable derivatives form dimers as well as catemers with specific topologies. They have also exercised the possibility of tautomerization in the substituted 1,8-naphthyridines highlighting solid state structures and theoretical calculation. Interestingly, 1,8-naphthyridine based metallomacrocycles as reported by Tanase et al. [11] is excellent to trap silver(I) ion where the 1,8-naphthyridine ring nitrogens are deeply involved in coordination. Beside these, 1,8-naphthyridine systems have been used to construct hydrogen bonding receptor molecules of different architectures for encapsulation of biologically relevant molecules like urea [12], biotin [13], citric acid [14], etc. All these features of 1,8-naphthyridine system are interesting. During the course of our work in supramolecular chemistry we intended to explore the hydrogen bonding behavior of 1,8-naphthyridine when the substituents at the position 2 are varied. In this aspect, we report here the synthesis of substituted 1,8-naphthyridines, **1**–**5** and their self-assembling/hetero-assembling properties in the solid state.

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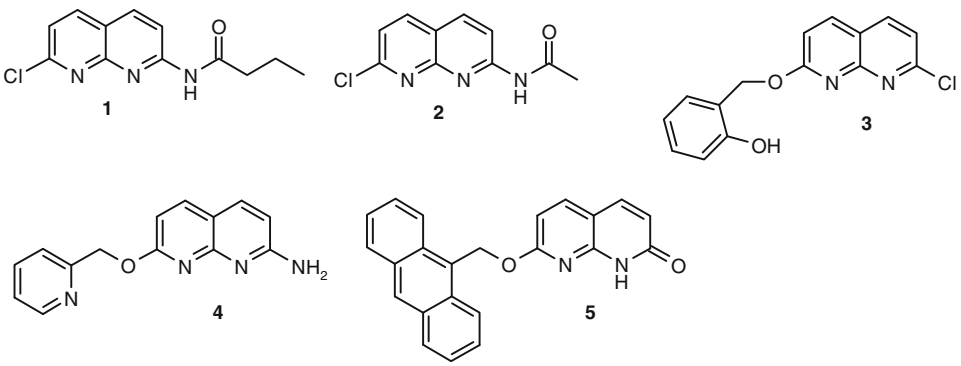


Materials and methods

All the solvents were dried by usual procedures prior to use. All the reactions were carried out under nitrogen atmosphere. IR spectra were recorded on Perkin Elmer model L120-00A. For ^{1}H spectra Bruker 400 and 500 MHz were used. Melting points were recorded in open capillaries and are uncorrected.

Synthesis of **1–5**

*N1-(7-chloro[1,8]naphthyridin-2-yl)butanamide (**1**)*

To a solution of 2-amino-7-chloro-1,8-naphthyridine [15, 16] (3 g, 0.017 mol) in dry CH_2Cl_2 (30 mL), butyryl chloride (2.1 mL, 0.02 mol) and triethylamine (2.9 mL, 0.02 mol) were added at room temperature. The resulting mixture was stirred at room temperature for 18 h. Excess acid chloride was neutralized with saturated $NaHCO_3$ solution. The crude product was extracted with $CHCl_3$ (3 × 30 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuum. The solid mass was purified through column chromatography using petroleum ether and ethyl acetate in 2:1 ratio as eluent. Compound 2-butaramido-7-chloro-1,8-naphthyridine **1** was isolated as white solid (2.5 g, 60%) and mp was noted as 182 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, $J = 8.9$ Hz, 1H), 8.41 (s, 1H), 8.18 (d, $J = 8.8$ Hz, 1H), 8.06 (d, $J = 8.2$ Hz, 1H), 7.39 (d, $J = 8.4$ Hz, 1H), 2.45 (t, 2H), 1.74–1.83 (m, 2H), 1.02 (t, 3H); FTIR ν_{max}/cm^{-1} (KBr) 3185 (m), 3055 (m), 2958 (m), 2873 (m), 1709 (s), 1615 (s), 1491 (s), 1327 (s), 1135 (s), 1013 (m), 928 (m), 851 (m), 797 (m).

*N1-(7-chloro[1,8]naphthyridin-2-yl)acetamide (**2**)*

A suspension of 2-amino-7-chloro-1,8-naphthyridine **7** (3 g, 0.017 mol) in dry CH_2Cl_2 (30 mL) with acetic anhydride (2.36 mL, 0.025 mol) was refluxed for 18 h. Excess anhydride was neutralized with saturated $NaHCO_3$ solution. The crude product was extracted with $CHCl_3$ (3 × 30 mL), dried over anhydrous Na_2SO_4 and removed under vacuum. The solid mass obtained was purified through column chromatography using petroleum ether and ethyl acetate in 3:1 ratio as eluent to give 2-acetamido-7-chloro-1,8-naphthyridine **2** as white solid (2.2 g, 60% yield) and mp was noted as 238 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (s 1H), 8.55 (d, $J = 8.84$ Hz, 1H), 8.19 (d, $J = 8.88$ Hz, 1H), 8.06 (d, $J = 8.48$ Hz, 1H), 7.39 (d, $J = 8.36$ Hz, 1H), 2.30 (s, 3H); FTIR ν_{max}/cm^{-1} (KBr) 3192 (m), 3058 (m), 1701 (s), 1606 (s), 1518 (s), 1487 (s), 1397 (s), 1320 (s), 1261 (s), 1107 (s), 984 (m), 849 (m), 794 (m).

*2-[(7-Chloro[1,8]naphthyridin-2-yl)oxy]methylphenol (**3**)*

A solution of 2-hydroxymethylphenol (0.5 g, 4.03 mmol) in dry THF (15 mL) was stirred with NaH (0.1 g, 4.2 mmol) under nitrogen atmosphere for 1 h under refluxing condition. The solution was cooled and 2,7-dichloro-1,8-naphthyridine **9** (0.8 g, 4.03 mmol) was added followed by catalytic amount of Cu_2O. The reaction mixture was stirred at room temperature for further 18 h. After completion of the reaction, THF was removed and water was added to the reaction mixture. The aqueous phase was extracted with $CHCl_3$ (3 × 30 mL). Evaporation of $CHCl_3$ afforded a crude mass which was purified by column chromatography using 25% ethyl acetate in petroleum ether as eluent. Compound **3** was obtained in very minimum amount (mp. 230 °C) and structure was confirmed by single crystal X-ray analysis.

*7-(2-Pyridylmethoxy)[1,8]naphthyridin-2-amine (**4**)*

To a stirred solution of 2-pyridylmethanol (0.6 g, 0.005 mol) in dry THF (20 mL), NaH (0.14 g, 0.006 mol) was added and the reaction mixture was refluxed for 3 h under nitrogen atmosphere. After cooling the solution, 2-acetamido-7-chloro-1,8-naphthyridine (1.45 g, 0.006 mol) was added followed by catalytic amount of Cu_2O. Reflux

was continued for further 18 h. The desired product was isolated after extraction with chloroform (3 × 30 mL) followed by purification through column chromatography using 35% ethyl acetate in petroleum ether as eluent. Product was obtained as solid, mp 156 °C, in 60% yield. It was then refluxed in the presence of 20% KOH in aqueous ethanol to cleave the amide bond. After extraction with chloroform, the corresponding product was isolated as white solid, mp 170 °C, in 85% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, $J = 4.36$ Hz, 1H), 7.80 (m, 2H), 7.69 (t, 1H), 7.48 (d, $J = 7.8$ Hz, 1H), 7.22 (m, 1H), 6.82 (d, $J = 8.56$ Hz, 1H), 6.61 (d, $J = 8.48$ Hz, 1H), 5.69 (s, 2H), 4.96 (s, 2H). FTIR ν_{max}/cm^{-1} (KBr) 3473 (s), 3364 (s), 3188 (s), 2913 (m), 1638 (s), 1595 (s), 1512 (s), 1432 (s), 1324 (s), 1246 (s), 1137 (m), 1038 (m), 839 (m), 799 (m).

*7-(9-Anthrylmethoxy)-1,2-dihydro[1,8]naphthyridin-2-one (**5**)*

To a solution of 9-hydroxymethylanthracene (0.52 g, 2.51 mmol) in dry THF (20 mL), NaH (0.066 g, 2.76 mmol) was added and the reaction mixture was refluxed for 1 h under nitrogen atmosphere. After 1 h, 2,7-dichloro-1,8-naphthyridine (0.2 g, 1.0 mmol) and a catalytic amount of Cu_2O were added at room temperature. The reaction mixture was refluxed for further 10 h. After evaporation of solvent, the residue was washed with water and extracted with $CHCl_3$ (3 × 30 mL). The crude mass was purified through column chromatography using 2% CH_3OH in $CHCl_3$ as eluent and the desired product was isolated as yellow solid (0.106 g, 30%), mp 208 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.55 (s, 1H), 8.42 (d, $J = 8.87$ Hz, 2H), 8.06 (d, $J = 8.37$ Hz, 2H), 7.76 (d, $J = 8.45$ Hz, 1H), 7.68 (d, $J = 9.46$ Hz, 1H), 7.56 (t, 2H), 7.51 (t, 2H), 6.57 (d, $J = 8.41$ Hz, 1H), 6.50 (d, $J = 9.41$ Hz, 1H), 6.45 (s, 2H), 10.74 (s, 1H). FTIR ν_{max}/cm^{-1} (KBr) 2922 (m), 2851 (m), 1733 (s), 1651 (s), 1594 (s), 1464 (s), 1258 (m), 1136 (m).

X-ray crystallography

Single crystals of compounds **1**, **2** and **5** were grown up by slow evaporation of chloroform and methanol mixture ($CHCl_3$:MeOH = 5:1 v/v) solvents. For compounds **3** and **4**, CH_2Cl_2 containing methanol (CH_2Cl_2:MeOH = 4:1 v/v) and ethyl acetate solvents were used respectively. X-ray intensity data sets were collected with Nonius Kappa CCD diffractometers, in case of Mo-radiation equipped with a rotating anode generator and the Cu-one with a normal

Table 1 Crystal data and structure refinement for **1**–**5**

	1	**2**	**3**	**4**	**5**
CCDC	704789	704790	704791	631313	704792
Empirical formula	$C_{12}H_{12}ClN_3O$	$C_{10}H_8ClN_3O$	$C_{15}H_{11}ClN_2O_2$	$C_{14}H_{12}N_4O*H_2O$	$C_{23}H_{16}N_2O_2$
Formula weight	249.70	221.64	286.71	270.29	352.38
Crystal system	Triclinic	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P-1$ (2)	$P2_1$/c (14)	$P2_1$/c (14)	$P2_1$/n (14)	$P-1$ (2)
a (Å)	7.698(1)	18.3242(5)	17.226(1)	6.596(1)	8.467(1)
b (Å)	11.610(1)	7.2393(1)	7.195(1)	14.676(1)	8.774(1)
c (Å)	26.951(1)	16.9758(3)	32.259(1)	13.796(1)	12.385(1)
α	96.76(1)				98.73(1)
β	93.74(1)	115.868(1)	91.04(1)	99.66(1)	94.90(1)
γ	90.04(1)				107.88(1)
V (Å^3)	2386.8(4)	2026.28(7)	3997.6(6)	1316.6(2)	856.93 16)
Z	8	8	12	4	2
μ (cm^{-1})	3.07	3.51	25.66	0.95	0.88
F(000)	1040 e	912 e	1776 e	568 e	368 e
Temperature (°C)	−75	−50	−50	20	−75
Wavelength (Å)	0.71073	0.71073	1.54178	0.71073	0.71073
Reflection collected	27027	12980	33153	7809	10476
Unique reflection	9612	4796	7095	2673	4086
R_{int}	0.058	0.052	0.079	0.062	0.065
Refined parameters	629	279	544	194	248
R_1 [$I > 2\sigma(I)$]	0.069	0.046	0.061	0.046	0.059
wR_2 (all data)	0.172	0.132	0.171	0.120	0.138

generator. The following Programs were used: data collection COLLECT [17], data reduction Denzo-SMN [18], absorption correction SORTAV [19–21], structure solution SHELXS-97 [22] and structure refinement SHELXL-97 [23], graphics SCHAKAL (Keller, E. 1997). The details and results of the analysis are presented in Table 1.

Results and discussion

Naphthyridine-based compounds having hydrogen bond donors and acceptors were prepared according to the Scheme 1. We started the syntheses of the compounds **1**, **2**, **3** and **4** from the starting compound 2-amino-7-chloro-1,8-naphthyridine **7** which was prepared by reported procedure [15, 16]. Chlorine atom at the position-2 in compound **7** is actually very labile and can be easily replaced by a number of nucleophiles. Oxygen atom containing nucleophiles of different types can be introduced in place of chlorine. In many cases, this has been done by carrying out the reaction in closed vessel under high pressure and temperature. Trace amount of water present in the solvent sometimes carries hydrolysis of C–Cl bond. In the present case, we have introduced various groups at the position 2 of naphthyridine under mild conditions.

All the naphthyridine-based compounds **1**–**5** contain an array of hydrogen bond donors and acceptors and therefore, they can follow different hydrogen bonding packing patterns in the solid state. They were explored in crystal engineering to gain an insight into their nature of hydrogen

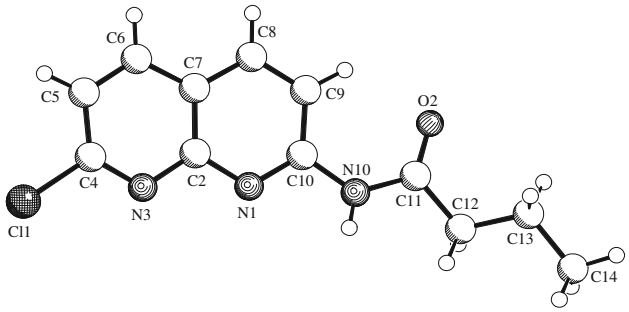
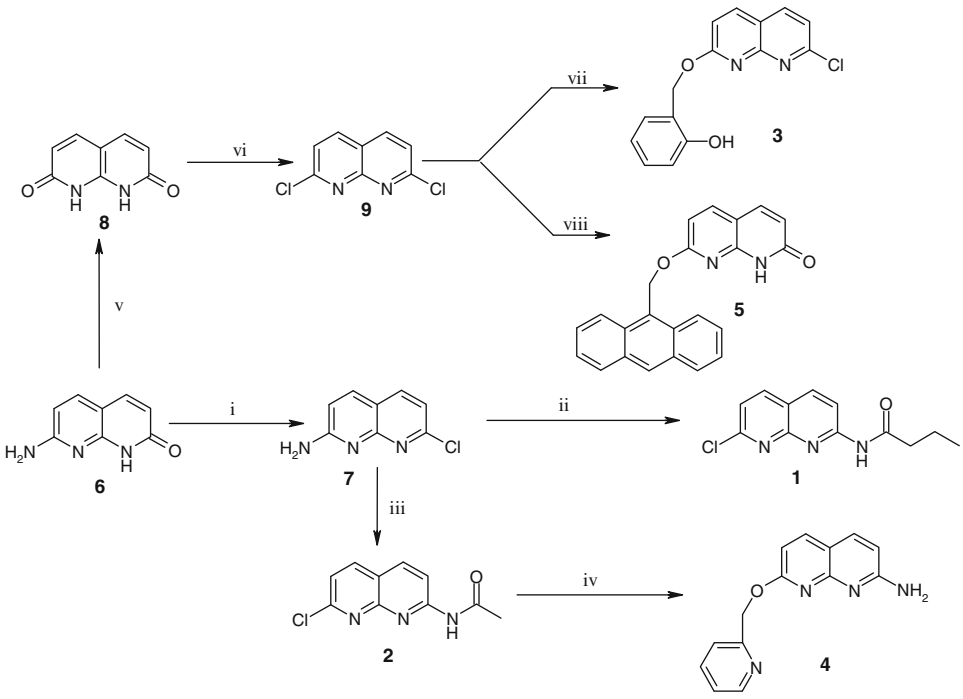


Fig. 1 SCHAKAL plot of one of the four independent molecules of **1** with atomic numbering scheme

bonding packing. In **1**, amide hydrogen is properly oriented with the ring nitrogens to form the assembly (Fig. 1). Four independent molecules of **1** form two pairs connected by hydrogen bonds, the hydrogen at N10 is accepted by the N1 of the other molecule [pair 1 (molecules A,B): N10A–H10A•••N1B: 0.84 Å, 2.18 Å, 172.8°; N10B–H10B•••N1A: 0.84 Å, 2.22 Å, 169.3°; pair 2 (molecules C, D): N1C–H10C•••N1D: 0.97 Å, 2.07 Å, 165.7°; N10D–H10D•••N1C: 0.88 Å, 2.17 Å, 170.1°]. These two pairs are connected via π–π interactions between the central six-membered rings (N1, C2, C7, C8, C9 and C10), the distances between B- and C-molecules and between A- and D-molecules are 3.60 and 3.59 Å, respectively. Figure 2a and b clearly show these features.

On contrary, compound **2**, which is very similar to **1**, exhibits different hydrogen bonding scheme due to the presence of shorter aliphatic chain in the amide part. The



Scheme 1 *i.* $POCl_3$, reflux; *ii.* butyryl chloride, Et_3N in dry CH_2Cl_2; *iii.* Ac_2O, reflux, dry CH_2Cl_2; *iv. a.* 2-hydroxymethylpyridine, NaH in dry THF, Cu_2O (cat.), reflux; *b.* 20% KOH in aq. EtOH; *v.* $NaNO_2$, conc. H_2SO_4; *vi.* PCl_5/$POCl_3$, reflux; *vii.* 2-hydroxymethylphenol, NaH in dry THF, Cu_2O (cat.), rt; *viii.* 9-hydroxymethyl anthracene, NaH in dry THF, Cu_2O (cat.), reflux

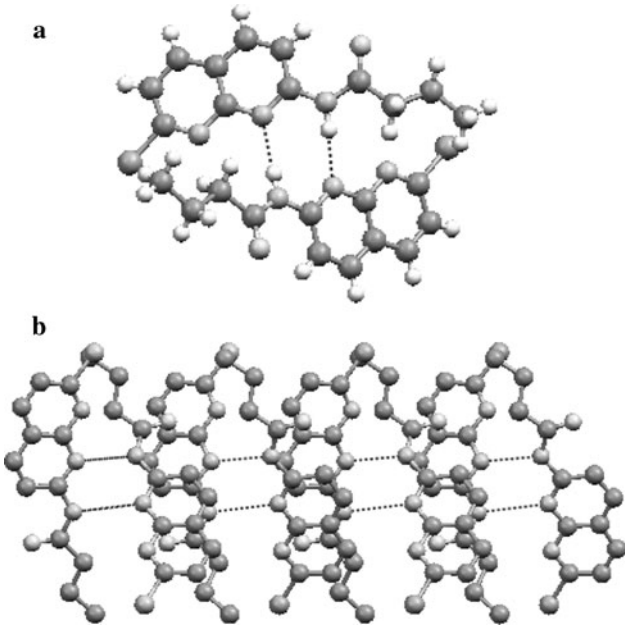


Fig. 2 **a** Hydrogen bonding scheme of **1**. **b** Packing of **1** along *a* axis (hydrogen atoms are omitted for clarity)

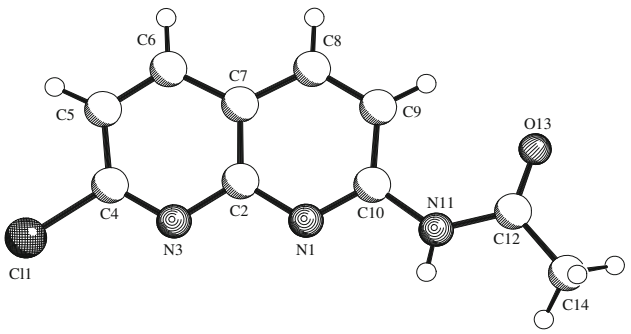


Fig. 3 SCHAKAL plot of one of the two independent molecules of **2** with atomic numbering scheme

structure is shown in Fig. 3 with atom numbering scheme. In this case, there are two independent molecules in the asymmetric unit, and each is forming an infinite chain by hydrogen bonding between H11 and N1* as shown in Fig. 4. The geometrical values for the hydrogen bond N11–H11•••N1* in molecules A and B are 0.81 Å, 2.28 Å, 170.0° and 0.87 Å, 2.21 Å, 163.6°, respectively. Interestingly, in the packing of **2** no π–π interactions are observed like **1**.

Instead of amide, the presence of other hydrogen bonding groups onto the naphthyridine motif controls the assembly in different ways. For example, in **3** dispositions of the three independent molecules in the asymmetric unit is dominated by strong intra-molecular hydrogen bond between H1 from O1 and N1. Figure 5 indicates the SCHAKAL plot of one of the three independent molecules of **3** with atom numbering scheme. The geometrical values

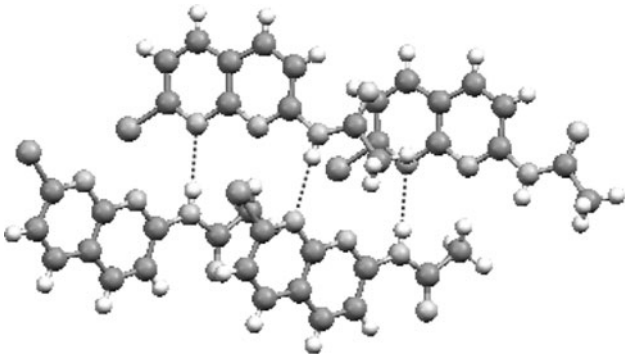


Fig. 4 Hydrogen bonding scheme of **2**

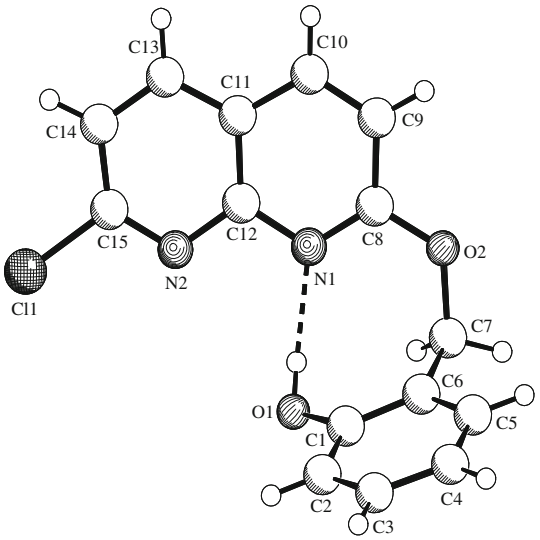


Fig. 5 SCHAKAL plot of one of the three independent molecules of **3** with atom numbering scheme and intra-molecular hydrogen bond

for these are O1–H1•••N1: 0.83 Å, 1.86 Å, 160.2° (molecule A); 0.83 Å, 1.87 Å, 168.5° (molecule B); 0.83 Å, 1.85 Å, 174.6° (molecule C). The molecules are again connected via π–π interactions between the two six-membered rings (N1, C8, C9, C10, C11, C12/N2, C12, C11, C13, C14, C15) (see Fig. 6). These are leading to two

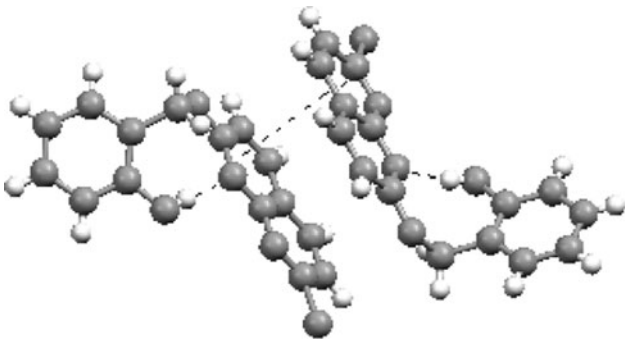


Fig. 6 π–π interactions between molecules B and C in **3**

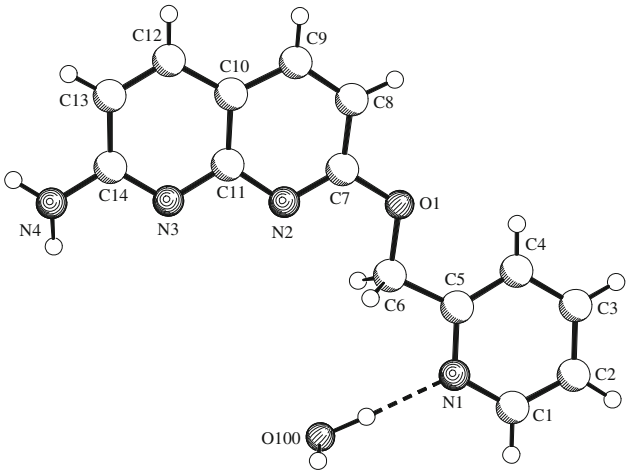


Fig. 7 SCHAKAL plot of **4** with atom numbering scheme and hydrogen bond to crystal water

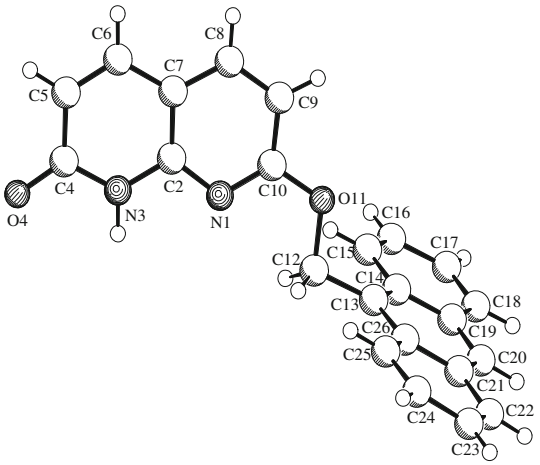


Fig. 9 SCHAKAL plot of **5** with atom numbering scheme

infinite stacks, molecule A is building up with symmetry equivalent molecules with distances of 3.65 and 3.73 Å, the second stack is build by B- and C-molecules, the distances are here 3.69 and 3.78 Å. Figure 6 shows the interaction for a pair of B- and C-molecules.

Similarly, introduction of pyridyl unit at the position 2 of 1,8-naphthyridine motif leads to a structure **4**, which has been found to be an useful hydrogen bonding building block for supramolecular interaction with other molecules of interest. We used this building block for the construction of a complete receptor [14] for citric acid. In the solid state, compound **4** exhibits hydrogen bonded polymeric assembly involving water molecule, coordinated to pyridine (Fig. 7) and the amine functionality. Figure 8 represents the hydrogen bonding network. The two hydrogen atoms of crystal water are accepted by N1 (O100–H102•••N1: 0.98 Å, 1.92 Å, 167.0°) and N3 (symmetry code: −x, −y+1, −z; O100–H101•••N3*: 0.80 Å, 2.31 Å, 164.0°), in addition O100 is an acceptor for the amine proton H4A (symmetry code: −x−1, −y+1, −z; N4–H4A•••O100*: 0.88 Å, 2.16 Å, 172.0°). The amine proton H4B is accepted by N3 (symmetry code: −x−1, −y+1, −z; N4–H4B•••N3*: 0.92 Å, 2.35 Å, 166.7°). No π–π interactions are observed in the assembly.

In order to observe the hydrogen bonding packing when a flat hydrophobic surface at the position 2 of 1,8-naphthyridine is present, we synthesized compound **5** (Fig. 9) from the intermediate compound **9**. During the synthesis one chlorine atom was replaced by anthacyl alcohol and another one was hydrolysed to give cyclic amide under the reaction condition. Due to the presence of very bulky anthracene group in **5** the possibility for setting up a hydrogen bonding scheme is very limited. Only strong bonded pairs are formed as shown in Fig. 10, the geometrical values are N3–H3•••O4*: 0.91 Å, 1.88 Å, 172.3° (symmetry code: −x+1, −y+1, −z+1). In addition, there are several π–π interactions, for example between the six-membered rings (N1, C2, C7, C8, C9, C10/C2, N3, C4, C5, C6, C7) with a distance of 3.61 Å and between the central rings of the anthracene (C13, C14, C19, C20, C21, C26) with a distance of 3.90 Å.

In summary, we have shown that the change in substituent around the 1,8-naphthyridine alters the hydrogen bonding patterns; discrete or polymeric structures. Change of aliphatic chain of the amide part induces different

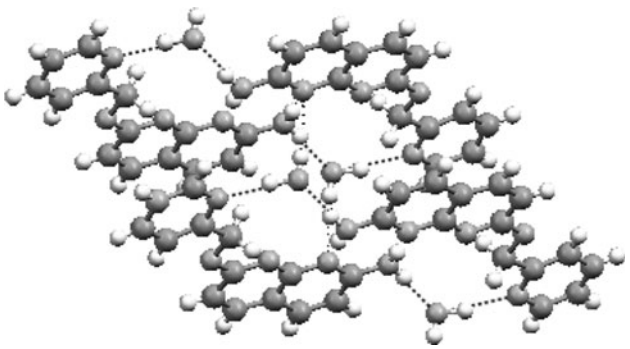


Fig. 8 Hydrogen bonding scheme of **4**

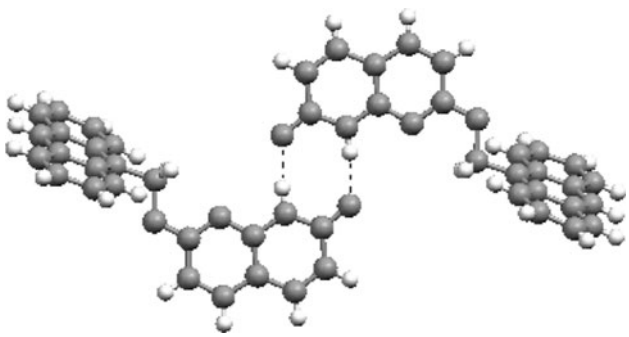


Fig. 10 Hydrogen bonded dimmer of **5**

hydrogen bonding packings. In addition, suitable hydrogen bond donor at the distal position from 1,8-naphthyridine ring is sometimes expected to be useful in making hetero association with the suitable guest partner. But involvement of naphthyridine ring nitrogen in the formation of intramolecular hydrogen bonding with the group at the distal position reduces the possibility of forming hetero association with suitable guest. This has been exemplified in compound **3** where intramolecular hydrogen bonding of phenolic −OH with naphthyridine ring nitrogen governs the hydrogen bonding possibilities and packing mode. The attachment of pyridine ring to the naphthyridine ring interestingly shows a water templated polymeric structure. In a similar way, the presence of anthracene which provides a flat hydrophobic surface around the naphthyridine influences a different packing. Compound **5** in this regime form a strong dimer. Therefore, the alteration of the groups around the naphthyridine has a significant effect in crystal packing. Such information is to be useful in the perspective of designing naphthyridine-based receptors for a specific task.

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