

Supramolecular Assemblies of (±)-2,2'-Dihydroxy-1,1'-binaphthyl with 2,2'-Bipyridine and Naphthodiazine[†]

Ji, Bao-Ming^{*a}(吉保明) CHEN, Hua-Ting^a(陈花婷)
DU, Chen-Xia^b(杜晨霞) DING, Kui-Ling^{*c}(丁奎岭)

^aDepartment of Chemistry, Luoyang Teacher's College, Luoyang, Henan 471022, China

^bDepartment of Chemistry, Zhengzhou University, Zhengzhou, Henan 450052, China

^cState Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Shanghai 200032, China

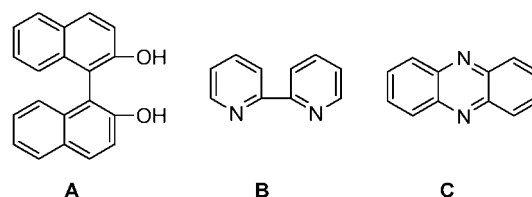
Supramolecular assemblies of (±)-2,2'-dihydroxy-1,1'-binaphthyl (BINOL, **A**), with aza donor molecules including 2,2'-bipyridine (**B**) and naphthodiazine (**C**), have been synthesized and characterized by single-crystal X-ray diffraction methods. Two inclusion complexes crystallize in the triclinic system with *P*-1 space group. In the inclusion complex between **A** and **B**, two molecules of **A** and two molecules of **B** are linked each other by intermolecular hydrogen bonds with two molecules of water as the bridges, forming a centrosymmetric dimer with formula of $A_2(H_2O)_2B_2$; while in the inclusion complex between **A** and **C**, the molecule **C** virtually acts as a bridge to link molecules **A** through intermolecular O—H···N hydrogen bonds, forming a short-chain supramolecular block with a formula of $A_2 \cdot C_3$. Besides the hydrogen bonding interaction between the host and guest molecules, π - π stacking interactions also play an important role in the solid-state packing of these two inclusion complexes. The structural information disclosed on the complex between dihydroxy compound and aza hydrogen bond acceptors in this work would be particularly important for the rational design of supramolecular organic functional materials.

Keywords 2,2'-dihydroxy-1,1'-binaphthyl, 2,2'-bipyridine, naphthodiazine, hydrogen bonding, supramolecular structure

Introduction

Optically active 1,1'-bi-2-naphthol (BINOL) and its derivatives have been widely used as chiral ligands of catalysts for asymmetric reactions and effective host compounds for the isolation or optical resolution of a wide range of organic guest molecules through the formation of crystalline inclusion complexes.^{1,2} The wide-ranging and important applications of these compounds in organic synthesis have stimulated great interest in developing efficient methods for their preparation.³ The preferential crystallization of a conglomerate, which is an equimolecular mixture of two crystalline enantiomers, could afford one of the enantiomers in optically active form, and this represents one of the most economical methods for optical resolution of racemic compounds due to the fact of without using chiral resolving agents.⁴ Despite of the well-established resolution of racemic BINOL (**A**) for preparation of optically pure BINOL,^{5,6} the preparation of optically pure binaphthol by the formation of inclusion complexes with different achiral guest molecules through the

formation of conglomerates has not yet been successful.⁷ Therefore, we made an attempt to use the selective formation of inclusion complexes with achiral guest molecules such as 2,2'-bipyridine (**B**) and naphthodiazine (**C**) for the efficient resolution of the racemic binaphthol (**A**). Unfortunately, we were not successful in resolving racemic binaphthol utilizing this idea. In spite of the nonsuccess of this approach, the interesting interaction patterns in the inclusion complexes of racemic binaphthol with 2,2'-bipyridine and naphthodiazine promoted us to disclose the synthesis and crystal structural features of both molecular complexes in this paper, which might be useful for the elucidation of inclusion mechanism.



* E-mail: kding@mail.sioc.ac.cn; lyhxxjbm@eyou.com

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Experimental

General methods

All the starting materials used were commercial products the physical constants of which were in agreement with reported values. 1,1'-Bi-2-naphthol (**A**) was synthesized following the literature method.⁸ The C, H and N microanalyses were carried out with an elemental Foss-Heraeus VARIO EL apparatus. FT-IR spectrum was recorded in KBr pellet in the range of 4000—400 cm⁻¹ on a Nicolet Avatar-360 spectrometer. Melting point was measured on a WC-1 apparatus and uncorrected.

General procedure for the preparation of inclusion complexes

Equimolar amounts of binaphthol (**A**) and 2,2'-bipyridine (**B**) or naphthodiazine (**C**) were dissolved in acetonitrile and then stirred at room temperature for 1 h. The undissolved materials were removed by filtration. The filtrate was set aside to crystallize, providing the title inclusion crystals.

A₂(H₂O)₂B₂: Yield 92%; m.p. 190—191 °C; Anal. calcd for C₆₀H₄₈N₄O₆: C 78.24, H 5.25, N 6.08; found C 78.30, H 5.19, N 6.12. IR ν : 3254, 3059, 1619, 1590, 1507, 1453, 1338, 1276, 1145, 845, 816, 757, 737, 543 cm⁻¹.

A₂•C₃: Yield 89%; m.p. 188—189 °C; Anal. calcd for C₇₆H₅₂N₆O₄: C 82.02, H 4.68, N 7.55; found C 82.21, H 4.80, N 7.39. IR ν : 3257, 3052, 1619, 1515, 1430, 1262, 1195, 1140, 1124, 816, 754, 744 cm⁻¹.

X-ray crystallography

A colorless single crystal of the title inclusion complexes suitable for X-ray structural analysis was selected from the crystals obtained above. All measurements were made on a Rigaku-IV imaging plate area detector with graphite monochromated Mo K α radiation ($\lambda=0.71073$ Å). The intensity data were corrected for Lorentz-polarization factors as well as for absorption. Structure was solved by direct methods and expanded using Fourier techniques. All hydrogen atoms were located in difference electron density maps but generally placed in idealized riding positions while hydroxy hydrogen atoms were refined freely. In the final cycle of full-matrix refinement using SHELXTL-97, all non-hydrogen atoms were refined anisotropically and hydrogen atoms isotropically. The main crystallographic data are given in Table 1. The final positional parameters are listed in Table 2. The selected bond lengths and angles are given in Table 3. Primary intermolecular hydrogen bonds are listed in Table 4.

Results and discussion

1,1'-Bi-2-naphthol (**A**) is a strong double donors of hydrogen bonds, while 2,2'-bipyridine (**B**) or naphtho-

diazine (**C**) is a strong acceptor of hydrogen bonds. When the hydrogen bond donor **A** was mixed with hydrogen bond acceptor **B** or **C** in acetonitrile solution,

Table 1 Crystallographic data and structure refinement parameters

Inclusion complex	A₂(H₂O)₂B₂	A₂•C₃
Empirical formula	C ₃₀ H ₂₄ N ₂ O ₃	C ₃₈ H ₂₆ N ₃ O ₂
Formula weight	460.52	556.62
Temperature/K	291(2)	291(2)
Wavelength ($\lambda/\text{Å}$)	0.71073	0.71073
Crystal system	triclinic	triclinic
Space group	<i>P</i> -1	<i>P</i> -1
<i>a</i> /Å	9.849(2)	10.447(2)
<i>b</i> /Å	10.204(2)	10.517(2)
<i>c</i> /Å	13.161(3)	13.301(3)
α (°)	103.62(3)	99.21(3)
β (°)	106.45(3)	101.89(3)
γ (°)	92.55(3)	93.71(3)
<i>Z</i>	2	2
<i>D</i> _{calc} /(g•cm ⁻³)	1.244	1.316
Volume/Å ³	1224.1(4)	1404.4(5)
μ/mm^{-1}	0.081	0.082
<i>F</i> (000)	480	582
Crystal size/mm	0.40×0.40×0.30	0.40×0.40×0.30
θ range for data collection/(°)	2.07—27.51	1.59—27.47
Index range	$-12 \leq h \leq 12$ $0 \leq k \leq 13$ $-17 \leq l \leq 16$	$-13 \leq h \leq 13$ $0 \leq k \leq 13$ $-17 \leq l \leq 17$
Unique data	2984	3814
Absorption correction range	0.976—0.868	0.976—0.967
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	2984/0/325	3814/0/405
Goodness-of-fit on <i>F</i> ²	1.085	1.064
Final <i>R</i> indices [<i>I</i> >2 σ (<i>I</i>)]: <i>R</i> ₁ , <i>wR</i> ₂	0.0607, 0.1397	0.0428, 0.0978
<i>R</i> indices (all data): <i>R</i> ₁ , <i>wR</i> ₂	0.0938, 0.1505	0.0587, 0.1045
Extinction coefficient	0.020(2)	0.0208(15)
Largest diffraction peak and hole/(e ⁻ •Å ⁻³)	0.285 and -0.383	0.135 and -0.137

Table 2 The final atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for non-hydrogen atoms

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}	Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
A₂(H₂O)₂B₂									
O(1)	3454(2)	7871(2)	1729(1)	52(1)	C(14)	6476(2)	5080(2)	3936(2)	47(1)
O(2)	5985(2)	5943(2)	1329(1)	57(1)	C(15)	6443(2)	6356(2)	4644(2)	41(1)
O(3)	7053(2)	3712(2)	528(1)	59(1)	C(16)	6529(2)	6523(3)	5763(2)	55(1)
N(1)	2595(2)	4157(3)	1284(2)	89(1)	C(17)	6491(3)	7749(3)	6416(2)	60(1)
N(2)	2405(2)	6369(2)	2970(2)	52(1)	C(18)	6383(2)	8892(3)	5988(2)	52(1)
C(1)	4687(2)	8711(2)	1928(2)	38(1)	C(19)	6290(2)	8781(2)	4923(2)	44(1)
C(2)	4517(2)	9769(2)	1389(2)	48(1)	C(20)	6297(2)	7514(2)	4204(2)	36(1)
C(3)	5662(2)	10643(3)	1490(2)	52(1)	C(21)	2539(3)	3139(3)	329(2)	72(1)
C(4)	7035(2)	10509(2)	2144(2)	44(1)	C(22)	1267(3)	2436(4)	-329(2)	86(1)
C(5)	8262(3)	11386(3)	2249(2)	62(1)	C(23)	67(3)	2761(4)	-37(3)	93(1)
C(6)	9580(3)	11231(3)	2867(2)	75(1)	C(24)	44(2)	3684(2)	838(2)	46(1)
C(7)	9744(3)	10209(3)	3414(2)	70(1)	C(25)	1280(2)	4371(2)	1491(2)	49(1)
C(8)	8595(2)	9328(3)	3324(2)	52(1)	C(26)	1282(2)	5399(2)	2515(2)	45(1)
C(9)	7199(2)	9452(2)	2686(2)	38(1)	C(27)	209(2)	5323(3)	2999(2)	57(1)
C(10)	5995(2)	8540(2)	2566(2)	36(1)	C(28)	288(3)	6230(3)	3966(2)	62(1)
C(11)	6152(2)	7360(2)	3066(2)	37(1)	C(29)	1453(3)	7215(3)	4447(2)	60(1)
C(12)	6169(2)	6084(2)	2418(2)	41(1)	C(30)	2463(3)	7237(3)	3919(2)	59(1)
C(13)	6350(2)	4936(2)	2864(2)	48(1)					
A₂•C₃									
O(1)	1427(1)	2291(1)	1719(1)	67(1)	C(18)	5775(2)	2914(1)	2488(1)	46(1)
O(2)	3268(1)	-463(1)	2050(1)	66(1)	C(19)	5995(2)	1727(1)	1912(1)	53(1)
N(1)	10875(1)	8504(1)	2480(1)	50(1)	C(20)	5155(2)	646(2)	1790(1)	54(1)
N(2)	9024(1)	6332(1)	2288(1)	53(1)	C(21)	4046(2)	681(1)	2243(1)	46(1)
N(3)	292(1)	4164(1)	731(1)	56(1)	C(22)	3792(1)	1795(1)	2828(1)	40(1)
C(1)	8781(2)	7248(1)	1696(1)	48(1)	C(23)	2601(1)	1806(1)	3287(1)	39(1)
C(2)	7561(2)	7132(2)	962(1)	64(1)	C(24)	2637(1)	1525(1)	4307(1)	39(1)
C(3)	7288(2)	8028(2)	353(2)	73(1)	C(25)	3803(2)	1263(1)	4961(1)	47(1)
C(4)	8219(2)	9103(2)	437(2)	74(1)	C(26)	3803(2)	978(2)	5926(1)	59(1)
C(5)	9392(2)	9255(2)	1122(1)	64(1)	C(27)	2633(2)	907(2)	6286(1)	64(1)
C(6)	9716(2)	8341(1)	1789(1)	49(1)	C(28)	1500(2)	1166(1)	5687(1)	56(1)
C(7)	10173(2)	6508(1)	2989(1)	49(1)	C(29)	1457(2)	1503(1)	4693(1)	44(1)
C(8)	10470(2)	5594(2)	3663(2)	64(1)	C(30)	292(2)	1804(1)	4066(1)	52(1)
C(9)	11593(2)	5766(2)	4399(2)	72(1)	C(31)	279(2)	2099(1)	3106(1)	53(1)
C(10)	12508(2)	6866(2)	4517(2)	69(1)	C(32)	1438(2)	2092(1)	2710(1)	46(1)
C(11)	12278(2)	7755(2)	3884(1)	61(1)	C(33)	3091(2)	7005(2)	478(2)	75(1)
C(12)	11106(2)	7604(1)	3097(1)	47(1)	C(34)	3387(2)	6174(2)	1212(2)	78(1)
C(13)	4669(1)	2942(1)	2959(1)	42(1)	C(35)	2479(2)	5254(2)	1303(2)	70(1)
C(14)	4463(2)	4129(1)	3538(1)	51(1)	C(36)	1202(2)	5100(2)	653(1)	52(1)
C(15)	5303(2)	5225(2)	3648(1)	61(1)	C(37)	-902(2)	4058(1)	89(1)	52(1)
C(16)	6383(2)	5193(2)	3174(1)	63(1)	C(38)	-1882(2)	3106(2)	146(2)	66(1)
C(17)	6618(2)	4066(2)	2611(1)	57(1)					

Table 3 Selected bond lengths (Å) and bond angles (°) with their standard deviations

A₂(H₂O)₂B₂			
C(1)—O(1)	1.378(3)	C(12)—O(2)	1.364(3)
O(1)—H(1A)	0.96(3)	O(2)—H(2A)	1.07(3)
N(1)—C(25)	1.414(3)	N(1)—C(21)	1.415(4)
N(2)—C(30)	1.335(3)	N(2)—C(26)	1.346(3)
C(1)-O(1)-H(1A)	111.9(17)	C(12)-O(2)-H(2A)	111.6(17)
C(25)-N(1)-C(21)	116.2(2)	C(30)-N(2)-C(26)	117.3(2)
C(10)-C(1)-O(1)	123.9(2)	C(2)-C(1)-O(1)	114.74(18)
C(11)-C(12)-O(2)	118.2(2)	C(13)-C(12)-O(2)	120.5(2)
C(25)-C(26)-N(2)	116.8(2)	C(26)-C(25)-N(1)	118.5(2)
C(22)-C(21)-N(1)	120.0(3)	C(29)-C(30)-N(2)	124.5(2)
A₂•C₃			
C(32)—O(1)	1.3653(19)	C(21)—O(2)	1.3646(17)
O(1)—H(1C)	0.893(19)	O(2)—H(2C)	0.940(2)
C(6)—N(1)	1.343(2)	C(12)—N(1)	1.3506(19)
C(7)—N(2)	1.342(2)	C(1)—N(2)	1.3430(19)
C(37)—N(3)	1.346(2)	C(36)—N(3)	1.354(2)
C(32)-O(1)-H(1C)	112.5(14)	C(21)-O(2)-H(2C)	114.4(11)
C(6)-N(1)-C(12)	116.98(13)	C(7)-N(2)-C(1)	116.71(13)
C(37)-N(3)-C(36)	117.69(13)	C(2)-C(1)-N(2)	119.15(14)
C(6)-C(1)-N(2)	121.70(14)	C(5)-C(6)-N(1)	120.38(14)
C(1)-C(6)-N(1)	121.44(14)	C(8)-C(7)-N(2)	119.39(14)
C(12)-C(7)-N(2)	122.02(14)	C(11)-C(12)-N(1)	119.92(14)
C(7)-C(12)-N(1)	121.11(14)	C(38)-C(37)-N(3)	119.74(15)
C(36)#1-C(37)-N(3)	121.26(15)	C(35)-(36)-N(3)	120.31(15)
C(37)#1-C(36)-N(3)	121.06(15)		

#1 $-x, -y+1, -z$ **Table 4** Primary hydrogen bonds in inclusion complexes **A₂(H₂O)₂B₂** and **A₂•C₃**

D—H···A	$d(\text{D—H})/\text{Å}$	$d(\text{H}\cdots\text{A})/\text{Å}$	$\angle\text{DHA}/(^{\circ})$	$d(\text{D}\cdots\text{A})/\text{Å}$
A₂(H₂O)₂B₂				
O(1)—H(1A)···N(2)	0.96(3)	2.09(3)	134(2)	2.842(3)
O(2)—H(2A)···O(3)	1.07(3)	1.64(3)	159(2)	2.667(2)
A₂•C₃				
O(1)—H(1C)···N(3)#1	0.893(19)	1.906(19)	154.5(19)	2.7388(18)
O(2)—H(2C)···N(1)#2	0.94(2)	2.05(2)	143.6(16)	2.8629(19)

Symmetry transition of **A**: #1 $-x, -y+1, -z$; #2 $x-1, y-1, z$.

the molecular crystals were formed spontaneously through hydrogen-bonding interaction between them. The elemental analyses indicated that racemic BINOL forms 1 : 1 or 1 : 1.5 inclusion complex with 2,2'-bipyridine (**B**) and naphthodiazine (**C**) in these two molecular complexes, respectively. The IR spectra of these two inclusion complexes between **A** and **B** as well as **A** and **C** showed strong OH signals at 3254 and 3257 cm^{-1} , respectively, which can be considered as the

characteristics of strong intermolecular hydrogen-bonding interactions.

The structural determination of molecular crystal between **A** and **B** revealed that the inclusion complex exists as a hydrate with the constituents in the asymmetric unit being in a 1 : 1 : 1 ratio. Arrangement of the molecules in the inclusion complex between **A** and **B** is shown in Figure 1a, which demonstrated that two molecules of **A** and two molecules of **B** are linked each

other through intermolecular hydrogen bonding with two molecules of water as the bridges, forming a centro-symmetric dimer. Therefore, the self-assembly formed between **A** and **B** can be expressed as the formula of $A_2(H_2O)_2B_2$. The atom distances of $O(1A)\cdots N(2A)$, $O(1A)\cdots O(3B)$ and $O(2A)\cdots O(3A)$ are 2.842(3), 2.917(3) and 2.667(2) Å, respectively. It is obvious that the interaction between $O(2A)$ and $O(3A)$ is stronger than that between $O(1A)$ and $O(3B)$ because the former distance is significantly shorter. The bond angles of $O(1A)-H(1A)\cdots N(2A)$ and $O(2A)-H(2A)\cdots O(3A)$ are $134.0(2)^\circ$ and $159.0(2)^\circ$, respectively. These results indicated that the hydrogen bonding network in this inclusion complex is in such a way as to match the strongest donor and the strongest acceptor first.⁹ The dihedral angle of two naphthalene rings of **A** is 80.0° . As shown in Figure 2a, two pairs of adjacent naphthalene rings are nearly parallel to each other and partially overlapped. The center-to-center distances between them are 3.101 and 2.976 Å, respectively. The face-to-face distance between two adjacent

2,2'-bipyridine rings is 3.231 Å. These results indicated that there are $\pi-\pi$ stacking interactions between adjacent aromatic rings in the inclusion complex $A_2(H_2O)_2B_2$. Assembly of host-guest molecules by means of intermolecular hydrogen bonds, $\pi-\pi$ stacking interactions and Van der Waals force further constitutes a three-dimensional steric structure.

As shown in Figure 1b, arrangement of the molecules in the inclusion complex between **A** and **C** distinctly differs from that of the molecules in the inclusion complex between **A** and **B** with respect to the linking pattern of host-guest molecules. Hydrogen bonding interactions between the nitrogen atoms of naphthodiazine and the OH groups of binaphthol are responsible for making an inclusion complex. The hydrogen bonding interactions shown in Figure 2b suggest that the naphthodiazine molecule virtually acts as a bridge to link binaphthol molecules each other through intermolecular $O-H\cdots N$ hydrogen bonds, forming one dimensional chain structure. The atom distances of $O(1)\cdots N(3)$ and $O(2)\cdots N(1)$ are 2.739(18) and 2.863(19) Å,

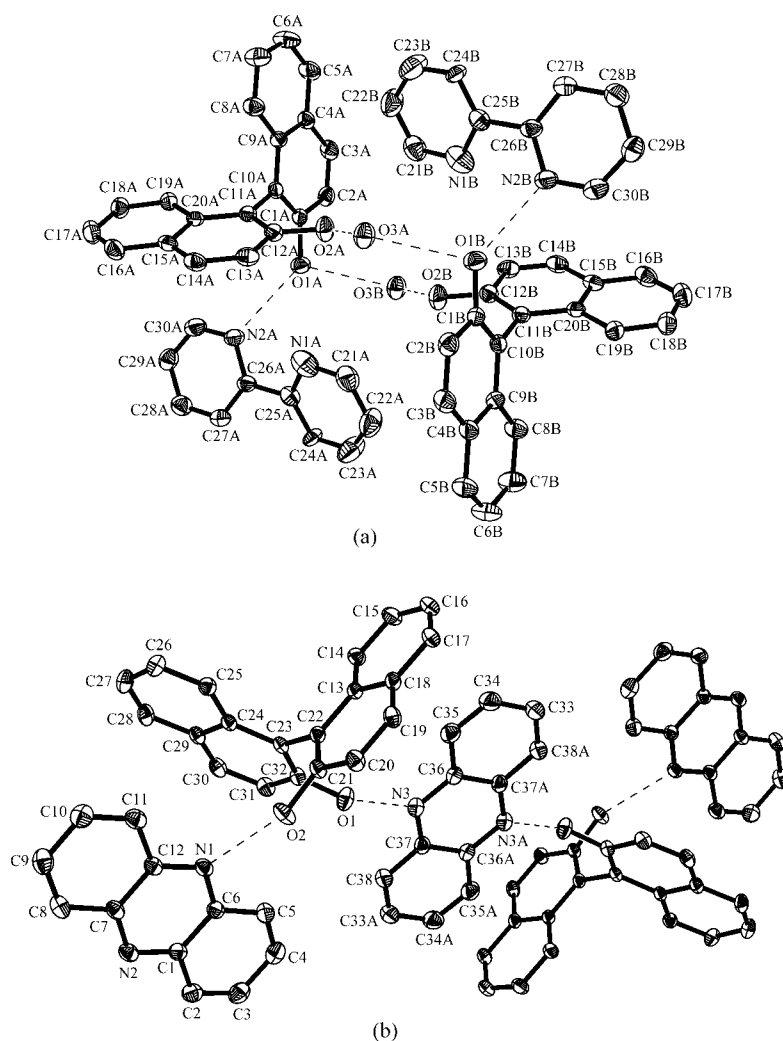


Figure 1 ORTEP diagram of inclusion complexes $A_2(H_2O)_2B_2$ (a) and $A_2\cdot C_3$ (b).

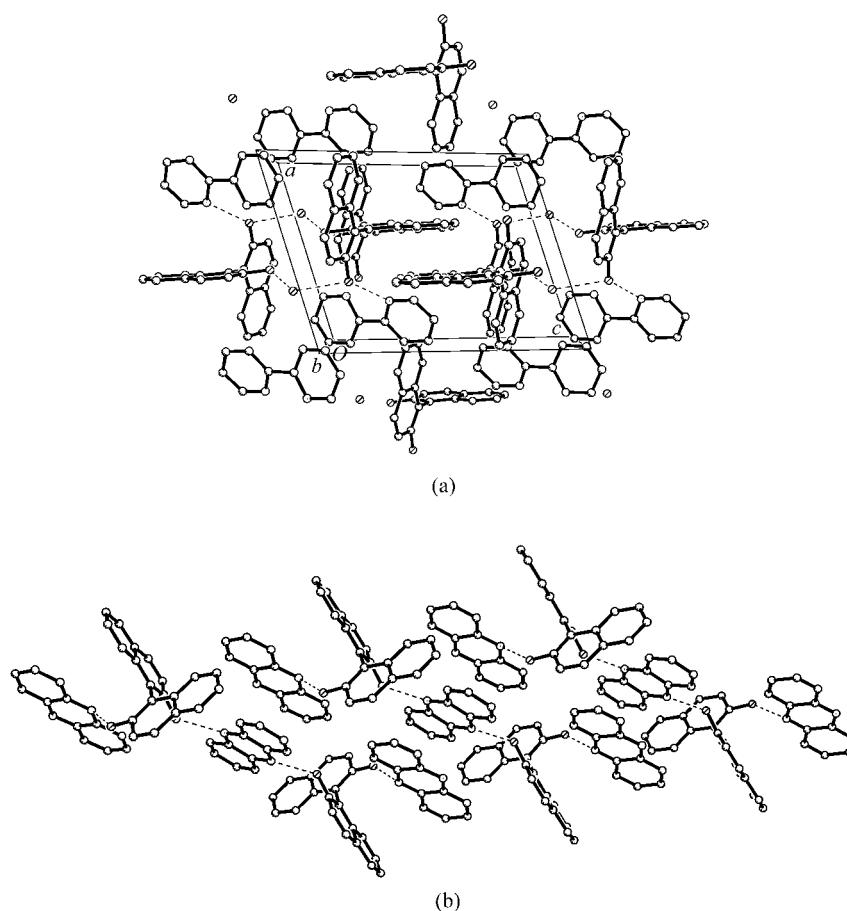


Figure 2 Packing diagram of inclusion complexes $A_2(H_2O)_2B_2$ (a) and A_2C_3 (b).

respectively. The dihedral angle of two naphthalene rings of **A** is 88.9° , which indicates an almost perpendicular arrangement of two naphthyl rings. The bond angles of $O(1)-H(1C)\cdots N(3)$ and $O(2)-H(2C)\cdots N(1)$ are $154.5(19)^\circ$ and $143.6(16)^\circ$, respectively. In addition, there are $\pi-\pi$ stacking interactions in inclusion complex between **A** and **C** (Figure 2b). The least-squares planarity analysis shows that two pairs of adjacent naphthalene rings are nearly parallel to each other and partially overlapped. The center-to-center distances between partially overlapped naphthalene rings are 4.3 and 5.1 Å, respectively. Two adjacent naphthodiazine rings are also almost parallel to each other (dihedral angle 9.2°) and partially overlapped. The face-to-face distance between partially overlapped naphthodiazine rings is 3.2–3.5 Å. These results indicated that there are also $\pi-\pi$ stacking interactions between adjacent aromatic rings in the inclusion complex between **A** and **C**. Under such circumstance, **A** and **C** form a short-chain supramolecular block with a formula of A_2C_3 via $O-H\cdots N$ hydrogen bonds. The $\pi-\pi$ stacking interaction of aromatic rings between adjacent blocks and Van der Waals force facilitate the assembly of them into a three-dimensional steric structure.

In conclusion, we found that racemic BINOL could spontaneously form supramolecular assemblies with aza

hydrogen bonding donor molecules, such as 2,2'-bipyridine (**B**) and naphthodiazine (**C**). The supramolecular structures of molecular crystals were highly dependent on the structures of the guest molecules. In the case of 2,2'-bipyridine (**B**) as the hydrogen bond acceptor, a self-assembly of $A_2(H_2O)_2B_2$ has been formed. On the other hand, interaction between racemic BINOL and naphthodiazine **C** resulted in the formation of a short-chain supramolecular block. Besides the hydrogen bonding interaction between the host and guest molecules, $\pi-\pi$ stacking interactions also play an important role in the solid-state packing of these two inclusion complexes. The structural information on the complexes between dihydroxy compound and aza hydrogen bond acceptors obtained in this work will be particularly important for the rational design of supramolecular organic functional materials.

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