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Structures of *N*,*N*′,*N*″-tris(3-pyridinyl)-1,3,5-benzenetricarboxamide and its nano-scopic zinc(II) coordination ring and their interaction with calf thymus DNA

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ABSTRACT

N,*N*′,*N*″-tris(3-pyridinyl)-1,3,5-benzenetricarboxamide monohydrate 3-TPB·H_2O and an unprecedented cyclic dimeric oligoamide zinc complex $[Zn_2(3\text{-TPB})_2Cl_4]\cdot 2H_2O$ (**1**) have been synthesized and their crystal structure were reported. Different from anhydrous 3-TPB, which is *s*-conformation, 3-TPB·H_2O adopts *aas*-conformation. All amide hydrogen form H-bonds. Zn ion in dimeric $[Zn_2(3\text{-TPB})_2Cl_4]\cdot 2H_2O$ located in a tetrahedron coordination environment. Two 3-TPB ligands and two Zn(II) ions link alternatively to form a 28-membered ring. The Zn···Zn distance in the ring is 11.162(5) Å. One pyridine nitrogen atom is free. The nano-size macrocyclic framework of **1** is stable up to 390 °C. 3-TPB·H_2O and complex **1** have similar fluorescence emission ~450 nm. Fluorescence titration shows that calf thymus DNA can quench the fluorescence 3-TPB in aqueous solution. However, calf thymus DNA has no interaction with complex **1**.

1. Introduction

Organic Amides have been paid much attention not only for their relating to peptide, but also for the advantages on the assembly of supramolecular networks organized by hydrogen bond. [1–4]. Several pyridine-derived foldamers have been utilized to mimic the binding surfaces of protein helices [2]. In recent years significant progress has been made in the design of synthetic oligoamide nano-tube, representing potentially a new and important class of functional materials [5–8]. However, Study based on metal-containing cyclic amides is still rare. Puddephatt and coworkers reported a construction of a metal-containing molecular triangle taking advantage of dipyridylamides (*N*-pyridin-4-ylisonicotinamide) as a bridging ligand. The complex cation forms a dimeric/trimeric architecture by 2 + 2 or 3 + 3 self-assembly of rigid or flexible oligoamide with palladium(II), which is similar to that formed by cyclic peptides [9,10]. On the other hand, the design of synthetic compounds that can read the information in the nucleic acid in particular DNA duplex has been a central goal at the interface of chemistry and biology [11]. The interactions of some oligoamides and DNA have been exploited [11–13]. It was found that oligoamide compounds bind to DNA in the minor groove [12,13].

N,*N*′,*N*″-tris(3-pyridinyl)-1,3,5-benzenetricarboxamide (3-TPB) is a fascinating functional group that can serve as active base sites and which possesses two types of hydrogen bonding sites: the —NH moiety acts as a hydrogen donor and the —C=O group acts as a hydrogen acceptor. Palmans constructed hydrogen-bonded porous solid derived from 3-TPB [14]. Stang and coworkers have utilized 3-TPB to construct a Pd(II) coordination cage with the $(Pd_3L_2)^{6+}$ moiety [15]. Lah and coworkers reported $[Pd_6(3\text{-TPB})_8]^{12+}$ cages two years later [16]. $[Cu_6(3\text{-TPB})_8]^{12+}$ nano-cages can be formed in DMSO [17]. Mononuclear Cu(I), Ag(I) complexes of the *N*-methyl substituted 3-TPB have also been reported. All pyridine nitrogen atoms coordinated to metal ion [18]. In the reported complexes, all three pyridine nitrogen atoms were utilized to form monomer, cage, or polymer complex. There is no dimeric complex reported yet. We are also interested in pyridine-containing amide ligands and their complexes [19,20]. We report herein the solid-state structures of 3-TPB and its dimeric nano-size macrocyclic complex assembled from 3-TPB and $ZnCl_2$, luminescence properties of these two compounds and their interaction with calf thymus DNA.

2. Experimental

2.1. General information

All chemicals were of reagent grade and used as received without further purification. Calf thymus DNA (CT-DNA) was purchased from Sigma and was sonicated and purified as described preciously [21]. Elemental analyses were performed on a Vario EL III elemental analyzer. Infrared spectra were recorded with a Nicolet A370 FT-IR spectrometer by KBr pellets in the range 400–4000 cm^{-1}. Thermogravimetric analyses were completed on a Netzsch STA

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449C thermal analyzer at a heating rate of 10 °C min^{-1} in air. Fluorescent spectra were recorded on a Shimadzu RF-5301 spectrophotometer.

2.2. Synthesis of 3-TPB·H_2O

3-TPB was prepared in a one-step reaction between 3-aminopyridine and 1,3,5-benzene tricarboxylate chloride in dry THF in the presence of triethylamine following a reported procedure [14,22]. 3-TPB (0.438 g) was dissolved in 5 mL DMF. After filtration, the solution was allowed to stand at room temperature for a week, Light yellow crystals suitable for X-ray crystal analysis settled gradually in 35% Yield.

2.3. Synthesis of [Zn_2(3-TPB)$_2Cl_4$]·$2H_2O$

[Zn_2(3-TPB)$_2Cl_4$]·$2H_2O$ was readily achieved by reaction of 3-TPB with $ZnCl_2$ in the molar ratio of 1:1 solvothermally at 110 °C in ethanol. A mixture of $ZnCl_2$ (6.8 mg, 0.050 mmol), 3-TPB (21.9 mg, 0.050 mmol) and EtOH 10 ml was heated at 110 °C for 3 d. Light yellow crystals were obtained when the mixture was cooled to room temp at 10 °C h^{-1}, (74.4% yield based on 3-TPB). FTIR (KBr, cm^{-1}): 1686 (s), 1663 (m), 1584 (m), 1542 (vs), 1484 (m), 1419 (s), 1331(m), 1281 (m). Anal. Calcd for {[Zn(3-TPB)Cl_2]·H_2O} $C_{24}H_{20}Cl_2N_6O_4Zn$:C, 48.63; H, 3.40; N, 14.18. Found: C, 48.40; H, 3.54; N, 14.07.

2.4. Fluorescence titration experiments

The concentration of DNA was determined by visible absorbance measurement using extinction coefficient ε_{260} = 12824 M^{-1} cm^{-1}. The purity of the DNA samples were confirmed by a spectrophotometer, A_{260}/A_{280} ranges between 1.8 and 2.0. The 2.4 mM CT-DNA stock solution was always resuspended in BPE (bis-phosphate EDTA) buffer (6.0 mM Na_2HPO_4, 2.0 mM NaH_2PO_4, 1.0 mM EDTA, pH = 7.0) for the measurement. The 3-TPB and **1** were first dissolved in dimethylsulfoxide (DMSO) at 0.23 M (for 3-TPB) or 0.17 M (for **1**) and then further diluted with water. Stock solutions were kept at 4 °C and freshly diluted to the desired concentration prior to use. Titration experiments were carried out in aqueous solution unless noted otherwise. Fluorescence emission (excited at 287 nm) was monitored over the range 350–550 nm. 3-TPB (38 μM) aqueous solutions were used for titration. DNA concentration varies from 0.9 μM to 0.03 mM bp. In this case, volume change is less than 5% in the whole titration process.

2.5. X-ray crystallography

Single crystal diffraction data were collected on a Bruker Smart Apex-II CCD diffractometer with graphite monochromatic Mo $K\alpha$ radiation (λ = 0.71073 Å) at room temperature. Empirical absorption corrections were applied using SADABS program [23]. The structures were solved by direct method with SHELXS-97 program and refined by full-matrix least squares on F^2 with SHELXL-97 program [24,25]. All none-hydrogen atoms were refined anisotropically and hydrogen atoms were placed geometrically if possible. The crystal data and refinement results are given in Table 1. The selected bond distances and angles are presented in Table 2. H-bonds are listed in Table 3.

3. Results and discussion

3.1. Description of crystal structures

Fig. 1 shows the structures of 3-TPB·H_2O. In contrast to the transparent, hexagon-shaped crystals of anhydrous 3-TPB obtained from methanol, which was reported previously [14], the light yellow, rectangle-shaped 3-TPB·H_2O crystals were formed in DMF. According to the relative position of the amide oxygen and pyridyl nitrogen atom to amide nitrogen, 3-TPB adopts different conformations in crystal structures as illustrated in Chart 1. *s*-Conformation indicates the pyridine nitrogen atom and the amide oxygen atom are in the same side of amide nitrogen, while *a*-conformation indicates the two atoms are trans each other (Chart 1). The crystal structures demonstrate that anhydrous 3-TPB [14] is a highly symmetric molecule (*a*-conformation) while hydrated 3-TPB·H_2O is in *aas*-conformation. All three pyridine amide groups in 3-TPB are identical, thus all three pyridine amide are in *trans* position (*a*-conformation). In hydrated 3-TPB·H_2O however, the symmetry is much lower and the three pyridine amide are different. The three pyridine amides in 3-TPB·H_2O are *cis*, *cis*, trans, respectively, (*ssa*-conformation). The dihedral angles between amide CNO and benzene is in the ranges from 32.02(37)° to 34.74(38)°. While the dihedral angle between amide CNO and pyridine ranges from 26.4(5)° (N(4)-containing pyridine) to 60.63(37)° (N(6)-containing pyridine). 3-TPB·H_2O packed into a monolayer and the monolayer packed into 3D structure (Fig. S1). There are not much difference in bond distances and bond angles between 3-TPB·H_2O and 3-TPB, but the arrangement of the pyridine amide and dihedral angles are fundamental different. Additionally, different from the bilayer supramolecular structure assembled by anhydrous 3-TPB [14], the monolayer structure is formed (Fig. S1) by hydrated 3-TPB due to the different dihedral angles defined by pyridine ring and center benzene ring in anhydrous 3-TPB and hydrated 3-TPB.

Water molecule and all amide hydrogen atoms form H-bonds as expected. H-Bond distances and angles are listed in Table 3. H-bond links adjacent *N*,*N*′,*N*″-tris(3-pyridinyl)-1,3,5-benzenetricarboxamide in 3-TPB·H_2O (Fig. S2).

Table 1
Crystallographic data for 3-TPB·H_2O and **1**.

Compounds	3-TPB·H_2O	**1**
Formula	$C_{24}H_{20}N_6O_4$	$C_{24}H_{20}Cl_2N_6O_4Zn$
Formula weight	456.46	592.73
Temp. (K)	273(2)	296(2)
Crystal system	Monoclinic	monoclinic
Space group	Cc	$P_2(1)/n$
a (Å)	17.757(2)	13.871(8)
b (Å)	14.549(2)	14.633(9)
c (Å)	8.3497(10)	13.871(8)
α (°)	90	90
β (°)	99.961(2)	118.41
γ (°)	90	90
V (Å^3)	2124.6(5)	2476(3)
Z	4	4
Dc (mg m^{-3})	1.427	1.590
Abs coeff. (mm^{-1})	0.101	1.252
F(000)	952	1208
Reflns collections	5358	12693
Unique reflns	1878	4380
Params	211	342
GOF on F^2	0.892	1.013
R_1, wR [$I > 2\sigma(I)$]	0.0679, 0.1941	0.0466, 0.0951
R_1, wR (all data)	0.0694, 0.1981	0.0834, 0.1110
Largest diff. peak and hole (e Å^{-3})	0.414 and −0.536	0.372 and −0.410

Table 2
Selected bond length (Å) and angle (°) of complex **1**.

Zn(1)—N(4)#1	2.036(3)	Zn(1)—N(1)	2.041(3)
Zn(1)—Cl(2)	2.2032(17)	Zn(1)—Cl(1)	2.2358(14)
N(4)#1—Zn(1)—Cl(2)	106.36(9)	N(4)#1—Zn(1)—Cl(1)	101.64(10)
N(1)—Zn(1)—Cl(2)	104.98(9)	N(1)—Zn(1)—Cl(1)	110.85(10)
N(4)#1—Zn(1)—N(1)	112.78(13)	Cl(2)—Zn(1)—Cl(1)	120.35(6)

Table 3
Hydrogen bonds for 3-TPB·H_2O and complex **1** (Å) and angle (°).

D—H...A	d(D—H)	d(H...A)	d(D...A)	<(DHA)
3-TPB·H_2O				
N(2)—H(2N)—O(1W)	0.84(4)	1.94(5)	2.767(4)	166(4)
N(3)—H(3N)—O(2)	0.87(3)	1.95(3)	2.785(3)	161(3)
N(5)—H(5N)—O(3)	0.96(3)	2.13(3)	3.056(3)	163(3)
O(1W)—H(1AW)—N(4)	0.97	1.87	2.836(4)	173.3
O(1W)—H(1BW)—N(6)	0.89	2.00	2.866(4)	162.2
Symmetry transformations used to generate equivalent atoms: #1*x*, −*y* + 2, *z* − 1/2 #2*x*, − *y* + 2, *z* + 1/2.				
1				
O(4)—H(4B)...N(6)#2	0.852(10)	1.90(3)	2.734(6)	166(10)
N(5)—H(5A)...O(4)#3	0.86	1.99	2.801(6)	157.4
N(3)—H(3A)...Cl(1)#4	0.86	2.60	3.405(4)	157.5
N(2)—H(2A)...Cl(1)#5	0.86	2.47	3.253(4)	152.0
Symmetry transformations used to generate equivalent atoms: #1 −*x*, −*y*, −*z* #2*x*, *y*, *z* − 1 #3*x* − 1/2, −*y* + 1/2, *z* + 1/2, #4*x* + 1, *y*, *z* + 1 #5*x* + 1/2, −*y* + 1/2, *z* + 1/2.				

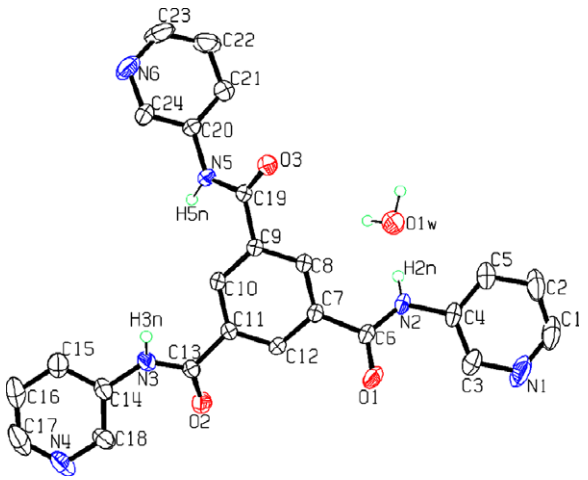

Fig. 1. ORTEP drawing of 3-TPB·H_2O with hydrogen atoms omitted for clarity (30% thermal ellipsoids probability).

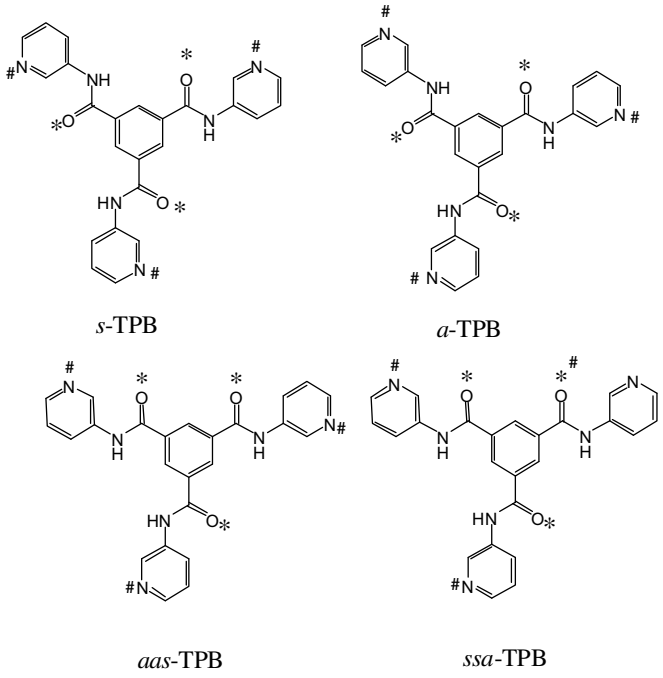

Chart 1. Conformations observed for 3-TPB in crystal structures.

In $[Zn_2(3\text{-TPB})_2Cl_4]\cdot 2H_2O$ (**1**), each Zn(II) ion is four-coordinated by two nitrogen atoms from different 3-TPB and two chloride anions to form a tetrahedron environment (Fig. 2). Two 3-TPB ligands and two Zn(II) ions link alternatively to form a 28-membered ring. The Zn···Zn distance in the ring is 11.162(5) Å, which is shorter compared with the Cu···Cu distances in a nano-cage of 13.940(3) Å [17]. This distance is much longer than a flexible oligoamide Zn complex of 3.572 Å [26]. In complex **1**, ligand 3-TPB adopts *ass*-conformation. The two coordinated pyridine amides are in *a*- and *s*-conformation, respectively, and the uncoordinated pyridine amide adopts *s*-conformation, which is quite different from the *a*- or *s*-conformation of the Cu(II) complex of 3-TPB and its Ag(I) and Pd(II) complex in *a*-conformation. Zn—N distance of 2.04 Å is comparable with that in Cu(II) complex of 2.03 Å [18], while Zn—Cl bond lengths are in the ranges from 2.20 to 2.24 Å.

Similar to other complexes based on amide/peptide ligands, hydrogen bonds interactions play very important role in crystal packing of complex **1** (Figs. S3 and S4). The uncoordinated pyridine nitrogen atom (N6) and the amide nitrogen atom (N5) outside the nano-ring both form hydrogen bonds with O4 atom from lattice water molecule with the distances of 2.804 and 2.734 Å, respectively, (Table 3). In addition, the amide nitrogen atom (N2) in the nano-ring also forms hydrogen bond with Cl1 anion and the Cl1...N2 distance is 3.253(4) Å. The distance of Cl1 and N3 is much longer with 3.405(4) Å (Table 3). The adjacent cyclic oligoamide complex molecules are packed to form bi-layered structure from *a*-axis (Fig. S3). Cl1...N2, N6...O4 H-bonds further assemble the bilayers into 3D supramolecular structure as shown in Fig. S4. All the H-bonds link adjacent bilayers. Intramolecular H-bonds does not exist in complex **1**. This phenomenon is quite different from that in dimeric complex of flexibly oligoamide [10]. The dihedral angle defined by pyridine ring and center benzene ring in hydrated 3-TPB·H_2O and **1** are different, 3-TPB·H_2O looks flatter than that of **1** (Fig. S5).

The thermal stability of complex **1** was examined by thermogravimetric analysis (TGA). The TGA curve (Fig. S6) shows that the first plateau (to 160 °C) lost 3.10% corresponding to the loss of one water molecule (calcd 3.03%). The framework remains unchanged up to 375 °C. The thermal stability of complex **1** is obviously higher than $[Cd(3\text{-TPB})_2Cl_2]_n$ reported by Tzeng et al. [27].

3.2. Solid-state emission spectra

The emission spectra of 3-TPB and complex **1** are investigated in solid-state at room temperature (Fig. 3). As expected, 3-TPB and its Zn(II) complex are both luminescent in the solid-state. Upon excitation at 389 nm, 3-TPB shows a broad emission with a maximum at ca. 450 nm, which is almost identical to excited at 300 nm [27]. Complex **1** displays a similar emission, but splits into two peaks at 450 and 471 nm, which is also quite similar to the 2D coordination polymer $[Ag(3\text{-TPB})PF_6]_n$ [27]. When excited at 287 nm, the emissions are essentially the same (Fig. 2, inset). Due to the close similarity of emissions between the ligand and its complex, these emissions can be ascribed to an intraligand transition [27].

3.3. DNA-binding affinity

The DNA-binding affinity was measured in aqueous solution using the intrinsic fluorescence of 3-TPB (Fig. 4). Fluorescence emission spectra were recorded with excitation at 287 nm and emission at 425 nm. Fig. 4 shows the fluorescence spectra (ex 287 nm) of 3-TPB in the absence and presence of CT-DNA. The presence of CT-DNA can quench the fluorescence emission of 3-TPB in aqueous solution. The steady-state fluorescence of free 3-TPB is quenched by double strand nucleic acid. Binding isotherms were obtained by measuring fluorescence of a fixed concentration

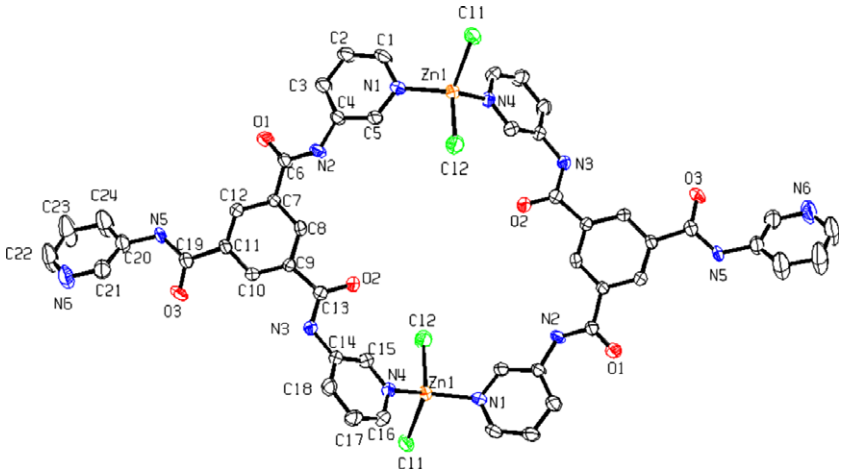

Fig. 2. ORTEP drawing of complex **1** with hydrogen atoms omitted for clarity (30% thermal ellipsoids probability).

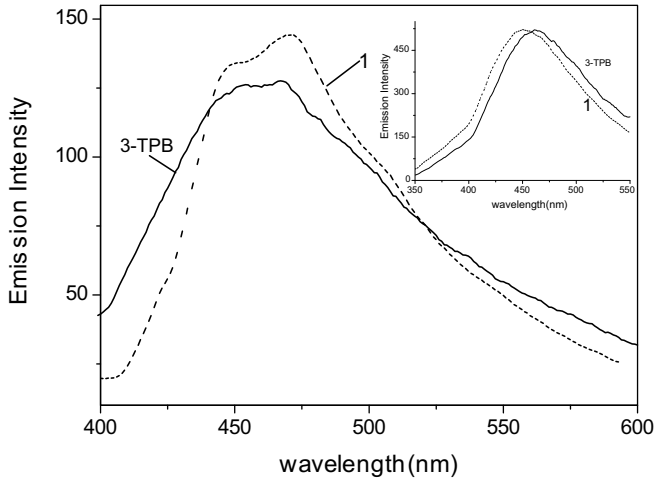

Fig. 3. Solid-state emission spectra of 3-TPB and complex **1** measured at room temperature with an excitation wavelength at 389 nm and 287 nm (inset).

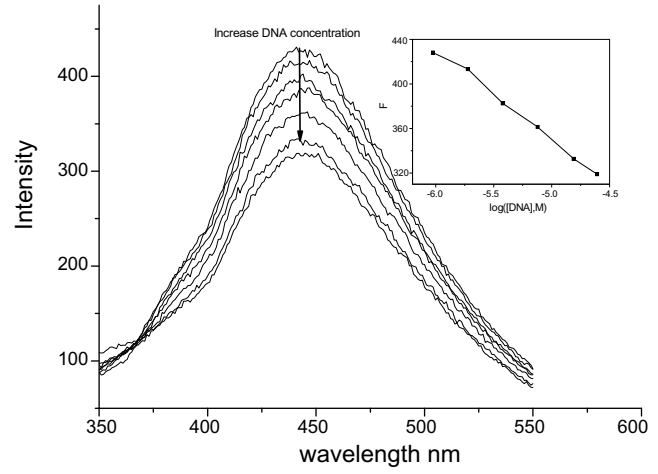

Fig. 4. Fluorescence titration of 38 μM 3-TPB aqueous solution with different concentration of calf thymus DNA (ex 287 nm).

of 3-TPB (38 μM) with various concentration of CT-DNA (Fig. 4). Oligoamides bind DNA in the minor groove mode [12,13], we also suggest 3-TPB binds to calf thymus DNA in the minor groove. We also examine the interaction of complex **1** with calf thymus DNA at the same condition, the result demonstrate that there no significant interaction, possibly due to bulky of the complex.

4. Conclusions

In summary, 3-TPB·H_2O and a nano-size cyclic oligoamide Zn(II) complex $[Zn_2(3\text{-TPB})_2Cl_4]\cdot 2H_2O$ was synthesized. H-bond play important role in crystal packing. The framework of the $[Zn_2(3\text{-TPB})_2Cl_4]$ can be stable up to 375 °C. Both 3-TPB and $[Zn_2(3\text{-TPB})_2Cl_4]\cdot 2H_2O$ exhibit intraligand emission peaks at ~460 nm upon excitation at 389 or 287 nm. Free 3-TPB can interact with CT-DNA in aqueous solution, but complex **1** cannot.

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Appendix A. Supplementary data

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Ref. Nos. 694563 (for monohydrate ligand) and 694564 (complex **1**). Copies of the data can be obtained free of charge from CCDC, 12 Union Road, Cambridge, CB2IEZ, UK. E-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2008.10.028.

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