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Syntheses, characterization and crystal structures of diamine-bridged dinuclear 5,14-dihydro-6,8,15,17-tetramethyldibenzo[*b*,*i*][1,4,8,11]tetraazacyclotetradecinatozinc(II) complexes

Xuan Shen, Daisuke Hirayama, Kazunori Sakata *, Mamoru Hashimoto

Department of Applied Chemistry, Faculty of Engineering, Kyushu Institute of Technology, Tobata-ku, Kitakyushu 804-8550, Japan

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Abstract

A *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TEMED)-bridged dinuclear tmtaa zinc(II) complex, Zn(tmtaa)(μ-TEMED)(tmtaa)Zn (**1**) (H_2tmtaa = 5,14-dihydro-6,8,15,17-tetramethyldibenzo[*b*,*i*][1,4,8,11]tetraazacyclotetradecine), a *N*,*N*′-dimethylethylenediamine (DEMED)-bridged dinuclear tmtaa zinc(II) complex, Zn(tmtaa)(μ-DEMED)(tmtaa)Zn (**2**) and an ethylenediamine (EDA)-bridged dinuclear tmtaa zinc(II) complex, Zn(tmtaa)(μ-EDA)(tmtaa)Zn (**3**), were prepared by the reactions of $Zn(CF_3SO_3)_2$, H_2tmtaa and TEMED, DEMED or EDA in acetonitrile. **1**, **2** and **3** have been characterized by IR spectra, UV spectra, mass spectra, NMR spectra and elemental analyses, as well as DSC measurements. Crystal structures of **1** and **2** were determined by X-ray diffraction. The structures reveal that **1** and **2** are dinuclear tmtaa zinc(II) complexes bridged with TEMED or DEMED. Two zinc(II) ions in **1** or **2** are coordinated through four nitrogen atoms of tmtaa and a nitrogen atom of the diamine to form two five-coordinated dinuclear complexes of square-pyramidal geometry. The spectra of **3** are very similar to those of **1** and **2**, so the structure of **3** should be analogous to those of **1** and **2**. The coordination behavior of the diamine at the axial sites the tmtaa zinc(II) complexes is also discussed.

Keywords: Zinc(II) complexes; Macrocycles; Five-coordinated; Diamine-bridged; Dinuclear complexes; Crystal structures

1. Introduction

5,14-Dihydro-6,8,15,17-tetramethyldibenzo[*b*,*i*][1,4,8,11]tetraazacyclotetradecine, abbreviated as H_2tmtaa (Fig. 1), has a structure and properties resembling porphyrins and phthalocyanines, but has a smaller N_4 coordination cavity ‘hole size’ and typically possesses a non-planar, saddle-shaped conformation. It has been shown that H_2tmtaa is the organometallic and coordination chemist’s flexible friend. It is tunable, easily prepared and readily introduced. It provides a reliable and a generally robust framework that continues to allow new main group and transition metal chemistry to flourish. Syntheses, properties and structures of mononuclear tmtaa complexes were compiled by Cotton and Czuchajowska [1] and Mountford [2]. Octacoordinated $M(tmtaa)_2$ (M = Ti, Zr, Hf, Ce, Y, Pr, Nd, Sm, Gd, Tb, Er, Yb) complexes which have sandwich structures have been investigated [3–6]. Furthermore, dinuclear metal–metal bonded complexes $[M(tmtaa)]_2$ (M = Ru, Rh, Mo, Cr, Sm) [7–15] and dinuclear bridged complexes [M(tmtaa)–X–M(tmtaa)] (M = Fe or Ti, X = O; M = Fe, X = S; M = Sn, X = CH_2; M = V, X = N; M = Ge, X = Cl) [16–21] have also been synthesized and characterized. To our knowledge, tmtaa zinc(II) complexes were reported for the first time in 1975 [22]. After that, no tmtaa zinc(II) complexes were reported until we synthesized and characterized five-coordinated mononuclear tmtaa zinc(II) complexes several years ago [23,24]. Up until now, all metallic bridged tmtaa dimers that have been reported are linked by one atom or a

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* Corresponding author. Tel.: +81-93-884-3307; fax: +81-93-884-3300.

E-mail address: sakata@che.kyutech.ac.jp (K. Sakata).

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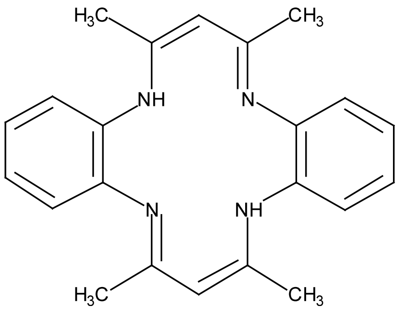

Fig. 1. Suggested structure of H_2tmtaa.

simple alkyl [16–21]. Furthermore, bridged dinuclear tmtaa zinc(II) complexes have never been discussed. In this paper, we present the syntheses, crystal structures and spectral properties of three five-coordinated dinuclear tmtaa zinc(II) complexes bridged with a long size ligand (compared with former works): diamine.

2. Experimental

2.1. Reagents and physical measurements

All solvents were distilled and all chemicals were reagent grade. Elemental analyses were performed on a Yanaco CHN Corder MT-3. IR spectra (4000–400 cm^{-1}) were recorded on a JASCO FT/IR-400 spectrophotometer in KBr pellets at room temperature. Ultraviolet and visible spectra were recorded on a Shimadzu UV-200S double beam spectrophotometer in benzene at room temperature. DSC data were obtained using a Shimadzu DSC-50 differential calorimeter. FAB^+ mass spectra were carried out with a JEOL JMS-SX102A gas chromatograph-mass spectrometer in a 3-nitrobenzyl alcohol matrix. ^1H NMR measurements were taken on a BRUKER AVANCE-400 in dimethyl sulfoxide (DMSO)-d_6, and chemical shifts were given in ppm relative to tetramethylsilane as an internal reference standard.

2.2. Preparation of Zn(tmtaa)(μ-TEMED)(tmtaa)Zn (1)

A hot solution of H_2tmtaa (0.689 g, 2.0 mmol) in acetonitrile (100 ml) was added to a solution of $Zn(CF_3SO_3)_2$ (0.727 g, 2.0 mmol) in acetonitrile (50 ml) under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 10 min. The colour of the solution turned from yellow to red and the product precipitated immediately from the solution as orange crystals as soon as *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TEMED) (0.581 g, 5.0 mmol) was added dropwise to the reaction mixture. The orange crystals were collected by filtration and washed with acetonitrile. Yield: 0.820 g (88%). *Anal.* Found: H, 6.27; C, 64.66; N, 14.87. Calc. for $C_{50}H_{60}N_{10}Zn_2$: H, 6.49; C, 64.44; N, 15.03%. M.p. 396–399 °C (DSC). UV (C_6H_6, λ_{max}, nm): 376, 420 (sh). IR (KBr, cm^{-1}): 1614(w), 1554(s), 1476(s), 1456(s), 1419(s), 1365(m), 1276(m), 1189(s), 1023(m), 737(s). FAB^+ MS: $m/z = 406$ $[Zn(tmtaa)]^+$.

2.3. Preparation of Zn(tmtaa)(μ-DEMED)(tmtaa)Zn (2)

H_2tmtaa (0.689 g, 2.0 mmol) and $Zn(CF_3SO_3)_2$ (0.727 g, 2.0 mmol) reaction solutions were prepared in a similar manner as above. When *N*,*N*′-dimethylethylenediamine (DEMED) (0.441 g, 5.0 mmol) was added dropwise to the reaction solution, the colour of the solution turned from yellow to red, precipitating no product initially. After the reaction solution was kept at room temperature for 12 h, red crystals were deposited. The crystalline product was collected by filtration and washed with acetonitrile. Yield: 0.325 g (36%). *Anal.* Found: H, 6.53; C, 63.89; N, 15.67. Calc. for $C_{48}H_{56}N_{10}Zn_2$: H, 6.25; C, 63.79; N, 15.50%. M.p. 380–384 °C (DSC). UV (C_6H_6, λ_{max}, nm): 376, 414 (sh). IR (KBr, cm^{-1}): 1618(m), 1553(s), 1474(s), 1454(s), 1418(s), 1356(m), 1276(m), 1188(s), 1026(m), 743(s). FAB^+ MS: $m/z = 406$ $[Zn(tmtaa)]^+$.

2.4. Preparation of Zn(tmtaa)(μ-EDA)(tmtaa)Zn (3)

H_2tmtaa (0.689 g, 2.0 mmol) and $Zn(CF_3SO_3)_2$ (0.727 g, 2.0 mmol) reaction solutions were prepared in a similar manner as above. When ethylenediamine (EDA) (0.300 g, 5.0 mmol) was added dropwise to the reaction solution, the colour of the solution did not change immediately. After the reaction solution was refluxed for 10 h, an orange solution was obtained. A red gummy product was collected after filtrating and evaporating the reaction solution, which was then fully washed with methanol. An orange powdery product was collected by filtration and washed with methanol again. Yield: 0.499 g (57%). *Anal*. Found: H, 6.22; C, 63.35; N, 15.82. Calc. for $C_{46}H_{52}N_{10}Zn_2$: H, 5.98; C, 63.09; N, 15.99%. M.p. 375–378 °C (DSC). UV (C_6H_6, λ_{max}, nm): 374, 420 (sh). IR (KBr, cm^{-1}): 1617(m), 1550(s), 1472(s), 1453(s), 1420(s), 1362(m), 1275(m), 1187(s), 1025(m), 746(s). FAB^+ MS: $m/z = 406$ $[Zn(tmtaa)]^+$.

2.5. X-ray crystal structure analysis

Crystals of **1** and **2** are both stable in air. An orange crystal of **1** was measured on a Rigaku AFC7R diffractometer equipped with a rotating anode generator using monochromated Cu Kα radiation ($\lambda = 1.54178$ Å). A red crystal of **2** was also measured on a Rigaku AFC7R diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71069$ Å) and a rotating anode generator. The structures of **1** and **2** were solved by direct methods [25] and expanded using the Fourier technique [26]. The non-hydrogen atoms were refined anisotropically. Crystal data and details associated with data collection of **1** and **2** are summarized in Table 1.

3. Results and discussion

3.1. Synthesis

$Zn(CF_3SO_3)_2$ and H_2tmtaa were dissolved in hot acetonitrile. Crystals of the TEMED-bridged dinuclear tmtaa zinc(II) complex **1**, which were suitable for X-ray structure analysis, separated out as soon as TEMED was added into the mixture of the above two solutions. The product was obtained in a high yield (88%). Crystals of the DEMED-bridged dinuclear tmtaa zinc(II) complex **2**, which were suitable for X-ray structure analysis, appeared 12 h later from the mixture of the above two solutions to which was added DEMED. The product was obtained in a low yield (36%). However, no crystals of the EDA-bridged dinuclear tmtaa zinc(II) complex **3**, separated out from the solution directly, though we acquired a powdery product at a yield of 57%. Thus, the reaction between $Zn(CF_3SO_3)_2$ and H_2tmtaa in the presence of tertiary diamine (TEMED), secondary diamine (DEMED) or primary diamine (EDA) leads to the corresponding diamine-bridged dinuclear complexes **1**, **2** or **3**. Judging from pK_{a1} and pK_{a2} data of TEMED, DEMED and EDA, which are 5.85, 8.97 [27]; 7.40, 10.16 [28] and 6.97, 9.97 [29], respectively, we consider the coordination behaviors of the tmtaa zinc(II) complex with the three diamines to be approximately similar. However, because of different solubility of the products, **1**, **2** and **3** were obtained in different ways.

Table 1
Crystal data, data collection and refinement parameters for **1** and **2**

Complex	**1**	**2**
Empirical formula	$C_{50}H_{60}N_{10}Zn_2$	$C_{48}H_{56}N_{10}Zn_2$
Formula weight	931.85	903.80
Crystal colour, habit	Orange, prismatic	Red, chunk
Crystal dimensions (mm)	0.05 × 0.10 × 0.20	0.10 × 0.10 × 0.20
Crystal system	monoclinic	triclinic
Space group	$C2/c$ (#15)	$P\bar{1}$ (#2)
No. of reflections used for unit	25	19
cell determination (2θ Range)	(46.4–57.0°)	(20.4–29.7°)
Lattice parameters		
a (Å)	10.024(3)	11.482(4)
b (Å)	22.146(4)	12.701(6)
c (Å)	21.091(3)	9.216(3)
α (°)	101.84(2)	100.86(3)
β (°)		105.42(3)
γ (°)		113.58(3)
Z	4	1
$F(0\ 0\ 0)$	1960.00	474.00
D_{calc} (g cm^{-3})	1.351	1.341
Radiation	Cu Kα ($\lambda = 1.54178$ Å) graphite monochromated	Mo Kα ($\lambda = 0.71069$ Å) graphite monochromated
Temperature (°C)	20.0	20.0
Scan type	$\omega - 2\theta$	$\omega - 2\theta$
$2\theta_{max}$ (°)	120.1	56.0
No. of reflections measured	Total: 3752	Total: 4436
	Unique: 3415	Unique: 4432
	($R_{int} = 0.017$)	($R_{int} = 0.086$)
Residuals: R; Rw	0.073; 0.134	0.241; 0.227
Residuals: R_1	0.044	0.079
Goodness of fit indicator	1.42	1.27
Max shift/error in final cycle	0.004	0.001

Because of the non-planar and saddle-shaped conformation of H_2tmtaa, only metallic tmtaa dimers bridged with one atom or a simple alkyl have been prepared [16–21]. According to this work, we plan to change the central metal zinc(II) into metals in the +IV oxidation state and use similar long size diamine bridging ligands to overcome the steric hindrance between tmtaa units and make an attempt at preparing six-coordinated metallic tmtaa oligomers or polymers in our future works.

3.2. Characterization

Complexes **1**, **2** and **3** were characterized by IR spectra, UV spectra, mass spectra, NMR spectra and elemental analyses, as well as DSC measurements. **1**, **2** and **3** have very analogous characteristics.

The IR spectra of **1**, **2** and **3** are similar to each other. IR bands assigning to C=N, C=C stretching modes of **1** appear at 1614(w), 1554(s), 1476(s) and 1456(s) cm^{-1}, while C=N, C=C stretching bands of **2** and **3** likewise appear at 1618(m), 1553(s), 1474(s), 1454(s) cm^{-1} and 1617(m), 1550(s), 1472(s), 1453(s) cm^{-1}, respectively. This proves that **1**, **2** and **3** should have analogous configurations and that interactions between the bridged-diamine and tmtaa units are also similar.

Electronic absorption spectra for **1**, **2** and **3** were measured in benzene solution at room temperature. The spectra of **1**, **2** and **3** are similar to each other with two absorption bands at ca. 375 and 420 (shoulder) nm. Comparing the maximum absorption band (348 nm) of H_2tmtaa with those of **1**, **2** and **3** shows ca. 30 nm shift to longer wavelength, which may be attributed to $\pi \rightarrow \pi^*$ transitions within the ligand molecule and CT transitions from metal to ligand [23].

FAB$^+$ mass spectra of **1**, **2** and **3** all show major fragments at m/z 406 corresponding to $[Zn(tmtaa)]^+$. No molecular ion peak was observed. This suggests that the coordination bond between the central zinc(II) ion

Table 2
^{1}H NMR data for **1**, **2** and **3**

Complex	Macrocyclic skeleton				Diamine		
	Methyl	Methine	Aromatic		Methyl	Ethylene	N–H
1	2.14 (s)	4.60 (s)	6.84 (m)	7.01 (m)	2.11 (s)	2.32 (s)	
2	1.98 (s)	4.44 (s)	6.68 (m)	6.85 (m)	1.95 (s)	2.10 (s)	12.29 (s)
3	2.13 (s)	4.58 (s)	6.82 (m)	6.99 (m)		2.33 (s)	12.44 (s)

Chemical shifts are given in ppm for TMS. Measured in DMSO-d_6. Multiplicity of a proton signal is given parentheses after δ-value: s = singlet, m = multiplet.

and the nitrogen atom of diamine would be cleaved easily in FAB$^+$ mass spectral conditions.

1, **2** and **3** gave well-resolved ^{1}H NMR spectra. ^{1}H NMR data for **1**, **2** and **3** are listed in Table 2. Because of bad solubility compared with mononuclear five-coordinate tmtaa zinc(II) complexes, ^{1}H NMR spectra of **1**, **2** and **3** were measured in DMSO-d_6. The proton signals of **1**, **2** and **3** are similar to each other. The signals of methyl, ethylene, methine and aromatic protons are singlet, singlet, singlet and multiplet. However, in ^{1}H NMR spectra of **2** and **3**, singlet signals are found at 12.29 and 12.44 ppm, respectively, which are assigned to N–H (diamine).

Not only spectra measured in solution (UV and ^{1}H NMR spectra), but also spectra measured in solid state (MS, IR spectra and DSC measurements) suggest that the structure of **3** may be analogous to those of **1** and **2**.

3.3. Description of the structure

The crystal structures of **1** and **2** are similar to each other. Fig. 2 and Fig. 3 display the molecular structures of **1** and **2**. Selected bond lengths and bond angles of **1** and **2** are listed in Table 3 and Table 4.

The crystal structures of **1** and **2** show good symmetry. Only half of the formula unit was measured with X-ray diffraction. The structures reveal that **1** and **2** are dinuclear complexes bridged with TEMED or DEMED. The two zinc(II) ions are coordinated through four nitrogen atoms of tmtaa and one nitrogen atom of diamine to form two five-coordinated dinuclear complexes of square-pyramidal geometry. The two N_4 planes in the two tmtaa units are almost parallel to each other. This is different from other bridged dinuclear tmtaa complexes in which two tmtaa units in the

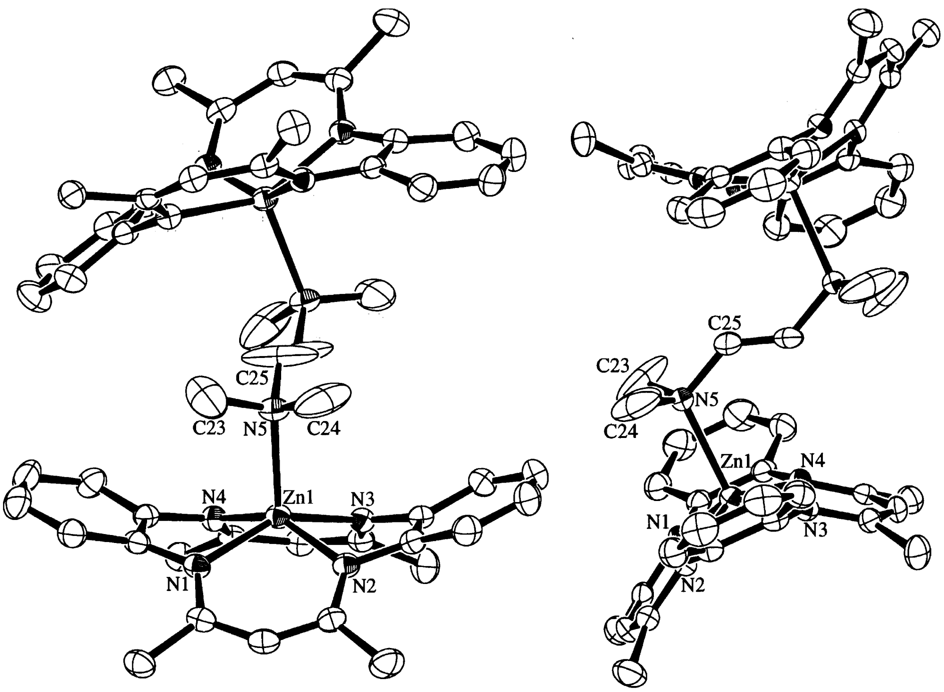

Fig. 2. ORTEP diagram of Zn(tmtaa)(μ-TEMED)(tmtaa)Zn (**1**) with atom labelling scheme showing 50% probability thermal ellipsoids for all non-hydrogen atoms.

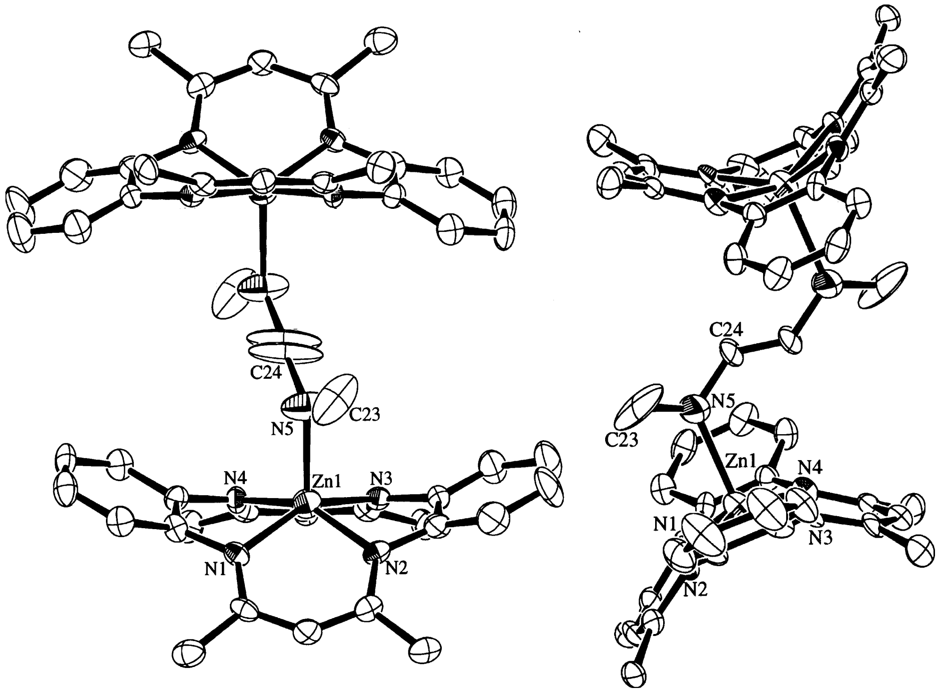

Fig. 3. ORTEP diagram of Zn(tmtaa)(μ-DEMED)(tmtaa)Zn (**2**) with atom labelling scheme showing 50% probability thermal ellipsoids for all non-hydrogen atoms.

Table 3
Selected bond lengths (Å) and bond angles (°) of **1**

Zn(1)–N(1)	2.037(3)	N(5)–C(23)	1.445(7)
Zn(1)–N(2)	2.047(3)	N(5)–C(24)	1.436(7)
Zn(1)–N(3)	2.040(3)	N(5)–C(25)	1.424(6)
Zn(1)–N(4)	2.044(3)		
Zn(1)–N(5)	2.207(3)		
Zn(1)···N_4 least squares plane: 0.533			
N(1)–Zn(1)–N(2)	92.1(1)	N(1)–Zn(1)–N(3)	149.0(1)
N(2)–Zn(1)–N(3)	80.3(1)	N(2)–Zn(1)–N(4)	150.5(1)
N(3)–Zn(1)–N(4)	91.3(1)	Zn(1)–N(5)–C(23)	109.2(3)
N(4)–Zn(1)–N(1)	80.5(1)	Zn(1)–N(5)–C(24)	110.9(3)
N(5)–Zn(1)–N(1)	104.2(1)	Zn(1)–N(5)–C(25)	113.9(3)
N(5)–Zn(1)–N(2)	103.0(1)	C(25)–N(5)–C(23)	103.3(6)
N(5)–Zn(1)–N(3)	106.9(1)	C(25)–N(5)–C(24)	112.2(6)
N(5)–Zn(1)–N(4)	106.4(1)	C(23)–N(5)–C(24)	106.8(6)

Table 4
Selected bond lengths (Å) and bond angles (°) of **2**

Zn(1)–N(1)	2.035(7)	N(5)–C(23)	1.320(1)
Zn(1)–N(2)	2.026(6)	N(5)–C(24)	1.370(1)
Zn(1)–N(3)	2.031(7)		
Zn(1)–N(4)	2.036(7)		
Zn(1)–N(5)	2.175(9)		
Zn(1)···N_4 least squares plane: 0.496			
N(1)–Zn(1)–N(2)	91.2(3)	N(1)–Zn(1)–N(3)	151.5(3)
N(2)–Zn(1)–N(3)	82.1(3)	N(2)–Zn(1)–N(4)	152.1(3)
N(3)–Zn(1)–N(4)	91.8(3)	Zn(1)–N(5)–C(23)	117.4(8)
N(4)–Zn(1)–N(1)	81.2(3)	Zn(1)–N(5)–C(24)	118.7(8)
N(5)–Zn(1)–N(1)	103.8(3)	C(24)–N(5)–C(23)	122.0(1)
N(5)–Zn(1)–N(2)	102.8(3)		
N(5)–Zn(1)–N(3)	104.7(3)		
N(5)–Zn(1)–N(4)	105.2(3)		

complexes orient at some angle between the two N_4 planes [16–21]. This may be due to the long size bridging diamine ligands that reduce the steric hindrance between the two tmtaa units in this work. Both structures of **1** and **2** show disorder in the bridging ligands, because several atoms (C23, C24 and C25 in **1**; C23 and C24 in **2**) show large ellipsoids. Furthermore, maybe because of this disorder, the *R*-value of **2** is a little high (Table 1), though it is still in the normal scope.

The mean Zn–N bond distances in the N_4 planes are 2.042 Å in **1** and 2.034 Å in **2**. They are shorter than the Zn–N bond distances between the nitrogen atoms of diamine and the central zinc(II) ion, which are 2.207 and 2.177 Å, respectively. The mean Zn–N bond distances are slightly different from those of other mononuclear five-coordinated tmtaa zinc(II) complexes (2.050 Å in Zn(tmtaa)N(C_2H_5)$_3$ [23] and 2.024 Å in Zn(tmtaa)-OPPh$_3$ [24]). The two zinc atoms in **1** and **2** are displaced from the N_4 planes toward the benzenoid faces of the saddle-shaped tmtaa ligands, as commonly observed in other mononuclear five-coordinated tmtaa zinc(II) complexes. The displacements are 0.533 Å in **1** and 0.496 Å in **2**, respectively, which are shorter than that of the mononuclear five-coordinated tmtaa zinc(II) complex, Zn(tmtaa)N(C_2H_5)$_3$ (0.563 Å) [23]. This suggests that the coordinating ability of mononuclear tmtaa zinc(II) complexes is a little stronger than that of the dinuclear tmtaa zinc(II) complexes. Furthermore, judging from the displacements from the N_4 planes, we find that the coordinating ability of the bridged-ligand and the zinc tmtaa complex in **1** is stronger than that in **2**. On the other hand, there are two methyl groups above the tmtaa unit in **1** and there is only one methyl group above the tmtaa unit in **2**. This may result in weaker steric hindrance in **2** than in **1**. Bringing the above factors into a state of balance, we consider that **1** and **2** have similar characteristics of spectra, structures and coordination behaviors.

Crystals of **3**, suitable for X-ray structure analysis were not obtained in our work. However, because of similar spectra compared with **1** and **2**, the structure of **3** should be analogous to those of **1** and **2**.

4. Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center. Copies of the data (CCDC-164442 for complex **1** and CCDC-185363 for complex **2**) can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@chemcrys.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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