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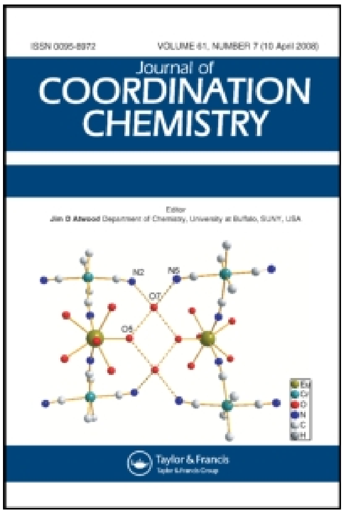

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Synthesis, structure and nonlinear optical properties of three dimensional compounds

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Synthesis, structure and nonlinear optical properties of three dimensional compounds

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The combination of metal ions with H_3tbba has resulted in the formation of two three dimensional coordination compounds $\{[Zn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**1**) and $\{[Mn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**2**) (H_2tbba = 2-thiobarbituric acid anion). Compounds **1** and **2** are isostructural with metal ions bridged by four hydroxyl oxygens from four different H_2tbba ligands to form a 3D network. H_2tbba exhibits bidentate coordination with both hydroxyl oxygens participating in coordination, a new coordination mode. Nonlinear absorption and refraction of **1** and **2** in DMF are studied by using *Z*-scan measurement technique at 532 nm. **1** and **2** possess nonlinear optical absorption and self-focusing.

Keywords: Coordination polymer; Zinc; Manganese; Nonlinear optics; *Z*-scan

1. Introduction

The design and synthesis of metal-organic coordination compounds from bridging ligands and transition metal ions have been rapidly expanding owing to the diversity of network topologies and applications as functional porous materials [1–8]. The coordination chemistry of heterocyclic thione ligands has also developed rapidly due to fascinating structural diversity and potential applications [9]. We choose H_3tbba for the following reasons: first, the hydroxyl groups on the ligand may be completely or partially deprotonated, second, the ligand has many coordinate sites to form diverse structures and third, it can act as hydrogen bond donors and acceptors, making it a wonderful candidate for construction of supramolecular networks. H_2tbba as a bridging ligand only by its hydroxyl oxygen atoms has not been reported previously, though it can adopt many coordination environments, summarized in chart I [10–19].

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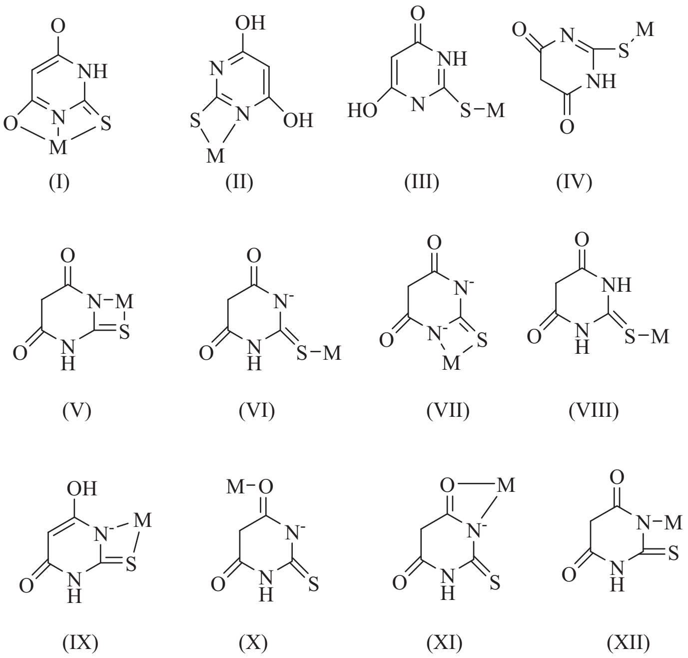

(I) (II) (III) (IV)

(V) (VI) (VII) (VIII)

(IX) (X) (XI) (XII)

Chart I. Coordinating modes of H_3tbba ligand.

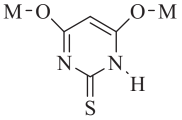

Chart II. Coordinating modes of H_3tbba in title compounds.

Two new coordination polymers, $\{[Zn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**1**) and $\{[Mn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**2**), bridged by both hydroxyl oxygen atoms of H_2ttba were successfully synthesized and structurally characterized. H_2ttba adopts a coordination mode illustrated in chart II. Herein we report the synthesis, solid state structure and nonlinear optical (NLO) properties of these two new compounds.

2. Experimental

2.1. *General methods*

All chemicals were of reagent grade from commercial sources and were used without further purification. The IR absorption spectra of the compounds were recorded on a VECTOR22 spectrometer in the range 400–4000 cm^{-1}. C, H, and N analyses were carried out with a Perkin-Elmer 240C elemental analyzer.

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Table 1. Crystallographic data and structure refinement details for **1** and **2**.

Compound	**1**	**2**
Formula	$C_{14}H_{20}N_4O_7S_2Zn$	$C_{14}H_{20}MnN_4O_7S_2$
Formula weight	485.83	475.40
Temperature (K)	293	293
Crystal system	Tetragonal	Tetragonal
Space group	I-42d	I-42d
a	16.9560(10)	17.094(2)
b	16.9560(10)	17.094(2)
c	7.5571(8)	7.6687(14)
V (Å)	2172.7(3)	2240.9(6)
Z	4	4
Crystal size (mm^3)	0.30 × 0.25 × 0.15	0.30 × 0.25 × 0.10
D_c ($g\,cm^{-3}$)	1.485	1.409
$\mu(mm^{-1})$	1.363	0.815
$F(000)$	1000	980
Total number of measured intensities	5752	5971
Number of unique data	1075	1107
Observed reflections [$I > 2.0\sigma(I)$]	924	900
R(int)	0.066	0.053
λ(Mo-Kα) (Å)	0.71073	0.71073
R_1, wR_2	0.0513, 0.1031	0.0583, 0.1296
GOF on F^2	1.090	1.051
Min. and max. resd. dens. (e $Å^3$)	−0.633, 0.702	−0.277, 0.342

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2.2. *Synthesis of [Zn(H_2tbba)$_2$(H_2O)]•2(OC_3H6) (1)*

A suspension of $ZnSO_4 \cdot 7H_2O$ (0.50 mmol, 0.144 g) and H_3tbba (1.0 mmol, 0.144 g) in DMF (10 mL) was stirred at room temperature for 10 h. After the mixture was filtered, acetone (10 mL) was layered onto the filtrate; a few weeks later yellow block crystals of **1** were isolated in a yield of 0.204 g (85%, based on Zn). Elemental analysis for $C_{14}H_{16}N_4O_7S_2Zn$ (481.80): calcd. C, 34.90; H, 3.35; N, 11.63. Found C, 34.85; H, 3.40; N, 11.62. IR (KBr, cm^{-1}): 3348(m), 3109(m), 1654(m), 1464(m), 1339(m), 1293(m), 1149(s), 936(s), 834(m), 771(s), 627(m), 544(s), 496(s), 450(s).

2.3. *Synthesis of [Mn(H_2tbba)$_2$(H_2O)]•2(OC_3H_6) (2)*

The preparation of **2** was similar to that of **1**, but $MnSO_4 \cdot H_2O$ (0.50 mmol, 0.084 g) was substituted for $ZnSO_4 \cdot 7H_2O$; yellow block crystals of **2** were isolated with a yield of 80%. Elemental analysis for $C_{14}H_{20}MnN_4O_7S_2$ (475.40): calcd. C, 35.37; H, 4.24; N, 11.79. Found C, 35.36; H, 4.27; N, 11.82. IR (KBr, cm^{-1}): 3452(m), 3111(m), 1656(m), 1462(m), 1336(m), 1291(m), 1148(s), 938(s), 832(s), 771(s), 625(s), 542(s), 496(s), 447(s).

2.4. *X-ray crystallographic determinations for 1 and 2*

Data were collected at room temperature using epoxy-coated crystals mounted on a glass fiber. All measurements were made on a Smart CCD diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The relevant crystallographic data are presented in table 1 and selected bond lengths and angles are given in table 2. The raw data were reduced and corrected for Lorentz and polarization

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

1		**2**	
Zn1–O1	2.127(3)	Mn1–O1	2.187(3)
Zn1–O2	2.032(7)	Mn1–O2	2.160(5)
Zn1–O1_a	2.127(3))	Mn1–O1_a	2.187(3)
Zn1–O2_a	2.032(7)	Mn1–O2_a	2.160(5)
Zn1–O1_b	2.127(3)	Mn1–O1_b	2.187(3)
Zn1–O1_c	2.127(3)	Mn1–O1_c	2.187(3)
S1–C3	1.661(7)	S1–C1	1.639(9)
O1–Zn1–O2	93.39(9)	O1–Mn1–O1_a	90.179(11)
O1–Zn1–O1_a	90.200(11)	O1–Mn1–O2	93.21(10)
O1–Zn1–O2_a	86.61(9)	O1–Mn1–O2_a	86.79(10)
O1–Zn1–O1_b	173.23(18)	O1–Mn1–O1_b	173.6(2)
O1–Zn1–O1_c	90.200(11)	O1–Mn1–O1_c	90.179(11)
O1_a–Zn1–O2	86.61(9)	O1_a–Mn1–O2	86.79(10)
O2–Zn1–O2_a	180.00	O2–Mn1–O2_a	180.00
O1_b–Zn1–O2	93.39(9)	O1_b–Mn1–O2	93.21(10)
O1_c–Zn1–O2	86.61(9)	O1_c–Mn1–O2	86.79(10)
O1_a–Zn1–O2_a	93.39(9)	O1_a–Mn1–O2_a	93.21(10)
O1_a–Zn1–O1_b	90.200(11)	O1_a–Mn1–O1_b	90.179(11)
O1_a–Zn1–O1_c	173.23(18)	O1_a–Mn1–O1_c	173.6(2)
O1_b–Zn1–O2_a	86.61(9)	O1_b–Mn1–O2_a	86.79(10)
O1_c–Zn1–O2_a	93.39(9)	O1_c–Mn1–O2_a	93.21(10)
O1_b–Zn1–O1_c	90.200(11)	O1_b–Mn1–O1_c	90.179(11)

Translation of symmetry code to equiv pos. a = y, $1-x$, $-z$; b = $1-x$, $1-y$, z; c = $1-y$, x, $-z$ (**1**). a = $1+y$, $1-x$, $-z$; b = $2-x$, $-y$, z; c = $1-y$, $-1+x$, $-z$ (**2**).

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effects using the SAINT program and for absorption using SADABS. The structures were solved by direct methods using SHELXS-97, and all non-hydrogen atoms were refined anisotropically by full-matrix least-squares based on $F2$ values [20]. All the non-hydrogen atoms were located from the trial structure and then refined anisotropically with SHELXTL using full-matrix least-squares procedures. The hydrogen atom positions were fixed geometrically at calculated distances and allowed to ride on the parent atoms. Hydrogen atoms on hydroxyl oxygen atoms and nitrogen atoms of pyrimidine are disordered into two positions, and each position has a site occupancy factor of 0.5 in both compounds.

2.5. *Optical measurement*

A 2.60×10^{-4} M DMF suspension of ground sample was placed in a 5 mm quartz cuvette for NLO measurements. Their NLO properties were measured with an 8 ns pulse at 532 nm generated from a Q-switched frequency-doubled Nd-YAG laser. The spatial profiles of the optical pulses were nearly Gaussian after passing through a filter. The pulsed laser was focused onto the sample cell with a 30 cm focal length mirror. Incident and transmitted pulse energies were measured simultaneously by two energy detectors (RJP-735 energy probers, laser precision). The NLO properties of the sample were determined by performing Z-scan measurements. The sample was mounted on a translation stage that was controlled by the computer to move along the Z-axis with respect to the focal point. An aperture of 0.5 mm radius was placed in front of the transmission detector. The transmittance was recorded as a function of the sample

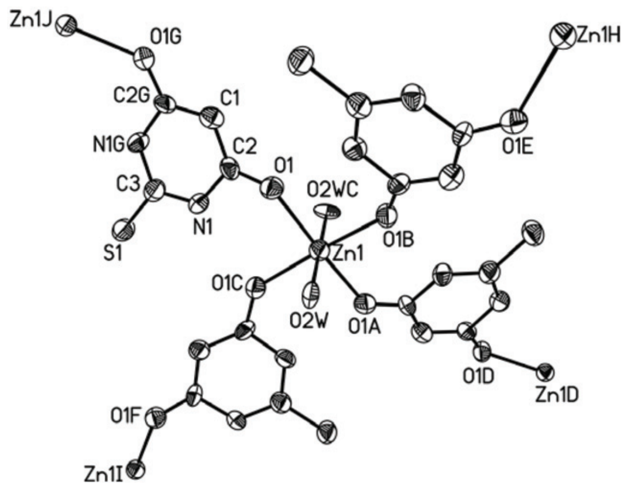

Figure 1. An ORTEP drawing of **1** showing 50% ellipsoid probability (hydrogen atoms and solvent molecules are omitted for clarity). Atoms labeled with the suffixes A, B, C, D, E, F, G, H, I and J are at the symmetry positions (A) $1-x$, $1-y$, z; (B) $1-y$, x, $-z$; (C) y, $1-x$, $-z$; (D) $1-x$, $y-0.5$, $0.25-z$; (E) $y-0.5$, x, $z-0.25$; (F) $1.5-y$, $1-x$, $z-0.25$; (G) x, $1.5-y$, $0.25-z$; (H) $0.5-x$, y, $-0.25-z$; (I) $1.5-x$, y, $-0.25-z$; (J) $1-x$, $0.5+y$, $0.25-z$.

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position on the Z-axis (closed aperture Z-scan). For measuring the NLO absorption, the Z-dependent sample transmittance was taken without the aperture (open aperture Z-scan).

3. Results and discussion

3.1. *Crystal structures*

Single-crystal X-ray diffraction analyses reveal that compounds **1** and **2** are isostructural, possessing three-dimensional networks. Using **1** as the example, as shown in figure 1, the Zn(II) is octahedral with two water oxygen and four hydroxyl oxygen atoms coming from four H_2tbba ligands. Four hydroxyl oxygen atoms (O1, O1A, O1B, and O1C) from four different H_2tbba ligands constitute the octahedral equatorial plane with Zn1–O1 lengths of 2.125(3)Å and an average angle of 90.16, while the oxygen atoms of the water molecules (O2W, O2WC) occupy the axial positions with Zn1–O2W lengths of 2.040(8)Å, and O2W–Zn1–O2WC is 180.000(1)°. H_3tbba has many possible tautomeric structures, as illustrated in chart III [10, 15–18, 21–30].

The distance of C3–S1 (in **1**) and C1–S1 (in **2**) are 1.661(7) and 1.639(9)Å, much shorter than the theoretical value of a C–S single bond (1.78 Å), indicating a C=S double bond in the compounds. The IR spectra of both compounds show the presence of strong ν(N–H) bands in the 3200–3000 cm^{-1} region [21], thus H_3tbba adopts tautomeric structure (C) in chart III. The metal ions are bridged by H_2tbba and extended into a three dimensional network with cavities. H_2tbba ligands in **1** and **2** are bridges through two hydroxyl oxygens, forming a M_6L_6 36-membered parallelogram (figure 2). These macro-rings form large parallelogram-shaped channels with a dimensionality of about 8.68×16.57 Å^2 for **1** and 8.76×17.09 Å^2 for **2**

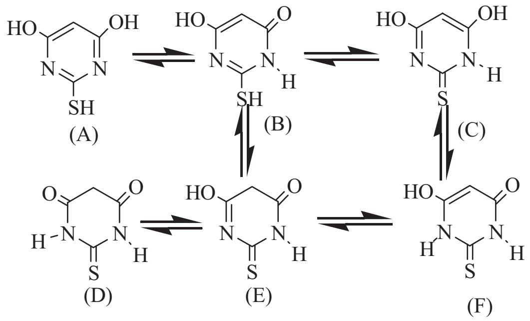

Chart III. Some possible tautomers of H_3tbba ligand.

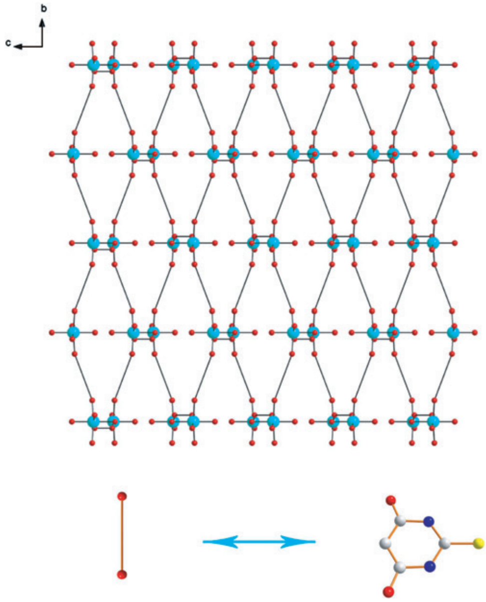

Figure 2. 3D framework structure of **1** and **2** viewed along the *c* axis. Color scheme online: M (Zn or Mn), green; O, red. C, H, N, and S atoms and solvent molecules have been omitted for clarity.

[defined by lengths of the parallelogram, as illustrated in figure 3(a) and figure 3(b)]. Packing of the acetones is head-to-head with oxygen atoms or carbon atoms of acetone molecules [as illustrated in figure 4(a)]. To further illustrate the packing style, solvate acetone molecules are classified into two kinds represented by yellow and purple. The purple acetone molecules locate in the 36-membered M_6L_6 parallelogram macro-rings viewed along the *a* axis [figure 4(b)]; it seems like the yellow acetone molecules locate in the small aromatic-rings, but actually they also locate in the macro-ring channels when viewed along the *b* axis [figure 4(c)]. We conclude that acetone molecules are located in the 36-membered M_6L_6 parallelogram macro-ring channels. Solvate volumes are $978.3\,Å^3$ per unit cell volume $2172.7\,Å^3$ for **1** and $1011.6\,Å^3$ per unit cell volume $2240.8\,Å^3$ for **2**, 45.0% and 45.1% of the unit cell volumes, respectively, calculated by PLATON program.

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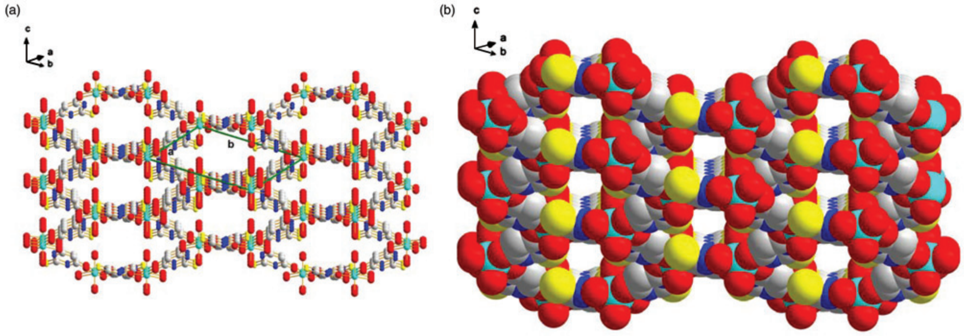

Figure 3. (a) View of six-metal-constructed macro-ring. Color scheme online: M (Zn or Mn), green; O, red; C, grey; N, blue; S, yellow. Crystallization acetone molecules and hydrogen atoms have been omitted for clarity; (b) Space-filling view shows macro-ring. Color scheme online: M (Zn or Mn), green; O, red; C, grey; N, blue; S, yellow. Crystallization acetone molecules and hydrogen atoms have been omitted for clarity.

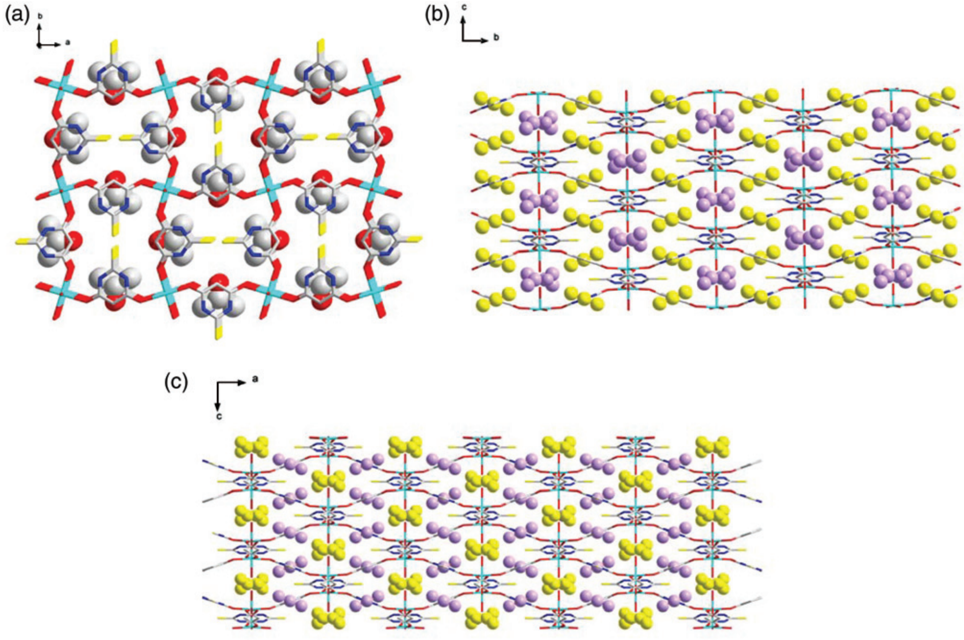

Figure 4. (a) View of the channels filled with acetone molecules (in space-filling mode) along the c axis. Color scheme online: M (Zn or Mn), green; O, red; C, grey; N, blue; S, yellow; hydrogen atoms omitted for clarity; (b) View of the channels filled with acetone molecules (in space-filling mode, represented by yellow and purple, respectively, for clarity) along the *a* axis. Color scheme online: M (Zn or Mn), green; O, red; C, grey; N, blue; S, yellow; hydrogen atoms omitted for clarity; (c) View of the channels filled with acetone molecules (in space-filling mode, represented by yellow and purple, respectively, for clarity) along the *b* axis. Color scheme online: M (Zn or Mn), green; O, red; C, grey; N, blue; S, yellow; hydrogen atoms omitted for clarity.

3.2. *Nonlinear optical properties of 1 and 2*

The third-order nonlinear optical (NLO) properties of **1** and **2** were carried out by the *Z*-scan method in 2.60×10^{-4} M DMF solutions [31]. Compounds **1** and **2** exhibit both nonlinear optical refraction and nonlinear optical absorption. The nonlinear

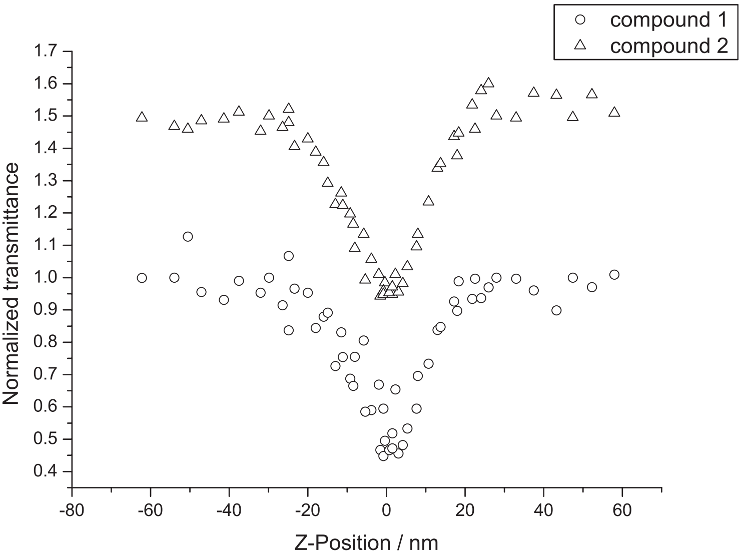

Figure 5. *Z*-scan data of **1** and **2** in 2.60×10^{-4} M DMF solution. The data were collected under an open aperture configuration and the data for **2** (∇) are vertically displaced by 0.5 for clarity.

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absorption component was evaluated under an open aperture configuration (figure 5). The open-aperture *Z*-scan curves have a deep valley, and the transmittance drops to the lower value of ca. 57%. Those results suggest that the compounds have a strong nonlinear absorption; the NLO absorption data obtained can be described by equations (1) and (2):

$$T(Z) = \frac{1}{\sqrt{\pi}q(Z)} \int_{-\infty}^{\infty} \ln[1 + q(Z)]e^{-\tau^2} \mathrm{d}\tau \tag{1}$$

$$q(Z) = \alpha_2 I_i(Z) \frac{1 - e^{-\alpha_0 L}}{\alpha_0} \tag{2}$$

which is derived to describe a third-order NLO absorptive process where α_0 and α_2 are linear and effective third-order NLO absorption coefficients, L the optical length and τ is the time. Light transmittance (T) is a function of the sample's *Z*-position.

The nonlinear refraction properties of **1** and **2** were assessed by dividing the normalized *Z*-scan data obtained under the closed aperture configuration by the normalized *Z*-scan data obtained under the open aperture configuration. The valley peak pattern of the normalized transmittance curve shows characteristic self-focusing behavior of propagating light in the sample. The valley and peak occur at equal distances from the focus with the valley-peak separation $\Delta Z_{v-p} = 16\,\mathrm{mm}$, fitting equation (3), where ω_0 is the laser beam waist radius ($35 \pm 5\,\mu\mathrm{m}$) and λ is the laser wavelength (532 nm). Those results suggest that observed optical nonlinear properties have a third order dependence on the incident electric field.

$$\Delta Z_{V-P} = 1.72 \frac{\pi \omega_0^2}{\lambda} \tag{3}$$

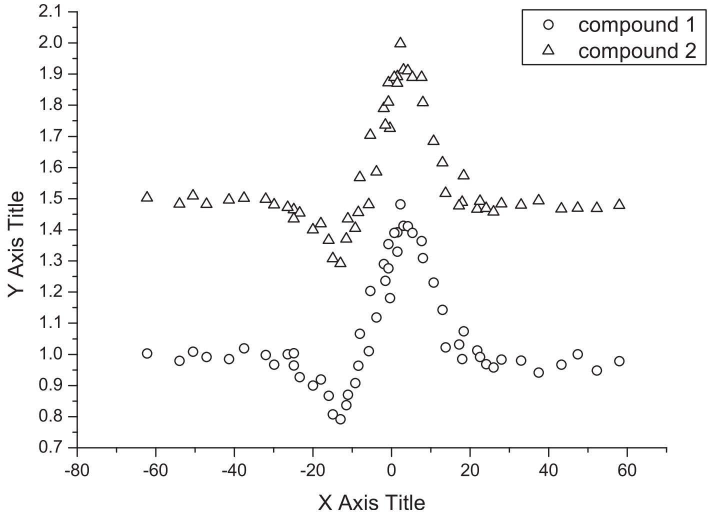

Figure 6. *Z*-scan data of **1** and **2** in 2.60×10^{-4} M DMF solution. The data were obtained by dividing the normalized *Z*-scan measured under a closed aperture configuration by the normalized *Z*-scan data of an open aperture configuration, and the data for **2** (∇) are vertically displaced by 0.5 for clarity.

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The difference between normalized transmittance values at valley and peak positions, ΔT_{v-p}, is related to nonlinear refractive index n_2 by equations (4) and (5), where $\Delta\phi_0$ and I_0 are the on axis phase shift and on axis irradiance, both at focus, respectively,

$$\Delta T_{V-P} = 0.406|\Delta\Phi_0| \tag{4}$$

$$\Delta\Phi_0 = \frac{2\pi}{\lambda} I_0 \frac{1 - e^{-\alpha_0 L}}{\alpha_0} n_2 \tag{5}$$

and α_0 and L are the linear absorption coefficient and optical path of the sample. The n_2 values calculated from figure 6 are 1.68×10^{-11} esu and 1.70×10^{-11} esu for **1** and **2**. The positive n_2 values show that **1** and **2** exhibit self-focusing. Experiments with varied I_0 show that n_2 so measured is indeed independent of I_0, consistent with the notion that $n = n_0 + n_2 I$ and the observed NLO phenomenon is third order in nature. The polymers **1** and **2** exhibit similar nonlinear optical effects to the ligand, and the n_2 values of H_3tbba, **1** and **2** are about the same, indicating effects of metal ions on the third-order NLO properties of **1** and **2** are weak, and the effects of ligand are crucial. This suggests that special ligands may control the third-order NLO properties of coordination polymers.

4. Conclusions

We synthesized two three-dimensional coordination compounds $\{[Zn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**1**) and $\{[Mn(H_2tbba)_2(H_2O)] \cdot 2(OC_3H_6)\}_n$ (**2**) bridged by both hydroxyl oxygen atoms of H_2ttba, the first example of H_2tbba as a bridging ligand with only hydroxyl oxygens taking part in coordination. Compounds **1** and **2** exhibit both nonlinear optical refraction and nonlinear optical absorption. Such activity could be further exploited in the future.

Supplementary material

CCDC 650494 and 650495 for **1** and **2** contain the supplementary crystallographic data for this article. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK. (Fax: (+44) 1223-336-033; or Email: deposit@ccdc.cam.ac.uk.)

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