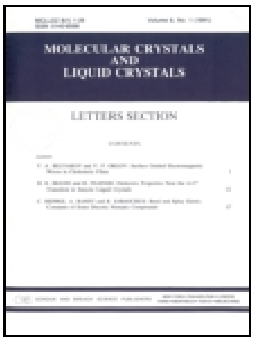

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Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: Yong-Hong Zhou, Xiao-Li Zhou, Su-Rong Zhou, Ling-Li Zhou, Zi-Han Wei, Jia-Yi Liang, Yu-Peng Tian, Jie-Ying Wu & Zhe-Yu Wang (2016) Synthesis, structures, and photophysical properties of two novel trinuclear Hg(II)complexes, Molecular Crystals and Liquid Crystals, 631:1, 187-195, DOI: 10.1080/15421406.2016.1170286

To link to this article: http://dx.doi.org/10.1080/15421406.2016.1170286

MOLECULAR CRYSTALS AND LIQUID CRYSTALS
2016, VOL. 631, 187–195
http://dx.doi.org/10.1080/15421406.2016.1170286

Synthesis, structures, and photophysical properties of two novel trinuclear Hg(II)complexes

Yong-Hong Zhou[a], Xiao-Li Zhou[a], Su-Rong Zhou[a], Ling-Li Zhou[a], Zi-Han Wei[a], Jia-Yi Liang[a], Yu-Peng Tian[b], Jie-Ying Wu[b], and Zhe-Yu Wang[c]

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ABSTRACT
Two novel trinuclear Hg(II) complexes, namely, $[Hg_3Cl_6(L^1)_2]$ (**1**) and $[Hg_3Cl_6(L^2)_2]$ (**2**), (L1 = trans-4-[(p-N,N-dimethylamino)styryl]-N-acetic-acidpyridinium, L2 = trans-4-[(p-N-2-hydroxyethyl-N-methylamino) styryl]-N-acetic-acid pyridinium) have been synthesized and characterized by IR spectroscopy, elemental analysis, and X-ray single-crystal diffraction. In both 1 and 2, the tri- and tetra-coordinated Hg(II) atoms are linked by the carboxyl groups, resulting in the formation of trinuclear structures. Their linear absorption, one-photon fluorescence, two-photon fluorescence, and two-photon pumped up-conversion lasing are investigated. Upon pumped by 1064 nm laser beam, complexes 1 and 2 exhibit excellent two-photon lasing at ~627 nm.

KEYWORDS
Two-photon absorption; complex; crystal structure; nonlinear optical properties

1. Introduction

In the last two decades, two-photon absorption (TPA) materials have gained much interest for their potential applications in fields of optoelectronics and photonics [1–5]. Among various TPA materials, organic compounds are the most frequently involved one [6–9]. Comprehensive researches have been carried out to design and synthesize various organic molecules with symmetry (D-π-D, A-π-A) or asymmetry (D-π-A) (D=donor, π = conjugated spacer, A = acceptor) structures. Many studies have been devoted to discuss the effects of conjugation length, donor-acceptor strength, chromophore number density and molecular planarity on the TPA activity [10–13].Recently, the scope of investigation on TPA materials has been largely extended by using metal complexes with the aim of combining the intrinsic photophysical properties of metal elements [large Stokes shifts, long lifetime emissions and sharp emission bands] with the unique advantage of TPA excitation [high transmission at low incident intensity and three dimensional spatial selectivity] [14–19]. Bharadwaj*et al.* reported the significant enhancement of TPA cross-section by binding Zn(II),Ca(II)orMg(II) ions to two organic compounds with D-π-A-π-D structural motif which possess low TPA cross-section [20]. Taki and co-workers demonstrated the use of benzoxazole-based Zinbo-type sensor to detect the Zn(II) ions in fibroblast cells with two-photon excitation (λ = 710 nm) [21]. More recently, due to their distinctive features such as sharp emission bands ranging from the visible region to the near-infrared region and long

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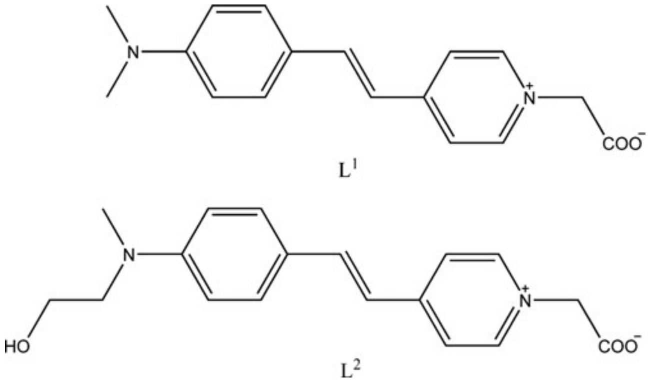

Figure 1. Structures of L1 and L2 ligands studied in this work.

excited-state lifetimes enabling time-delayed detection, two-photon microscopy imaging of cells by lanthanide complexes have also received considerable attention. Hu *et al.* reported the two-photon antenna effect of a europium(III) complex with functionalized β-diketonate and 1,10-phenanthroline as ligands. The complex was able to stain DNA in live cells [22]. Though complexes with intense TPA effect have been reported, examples of complexes with TPP (two-photon pumped) lasing properties are scarce, and their crystal structures have been rarely reported [23]. In fact, the crystal structures can offer information of molecular conformation, intra- and inter-molecular interactions, contributing to illustrate the relationship between the structures and properties. Based on these considerations, we have synthesized two new complexes $[Hg_3Cl_6(L^1)_2]$ (**1**) and $[Hg_3Cl_6(L^2)_2]$ (**2**), based on two D-π-A type chromophores, *trans*-4-[(p-N,N-dimethylamino)styryl]-N-acetic-acidpyridinium (L^1) and *trans*-4-[(*p*-*N*-2-hydroxyethyl-*N*-methylamino)styryl]-*N*-acetic-acid pyridinium (L^2) (Figure 1). L^1 and L^2 were chosen because of their ability to construct stable complexes exhibiting strong nonlinear optical properties. X-ray structure determinations for both **1** and **2** have been performed. The photophysical properties including linear absorption, one- and two-photon induced fluorescence and two-photon pumped lasing for **1** and **2** in solution are also reported.

2. Experimental section

Materials and general methods

All reagents were purchased from J&K chemical and used as received without further purification. L^1 and L^2 were synthesized using the literature method [23]. Elemental analyses for C, H, and N were obtained on a Vario EL III analyzer. Infrared spectra were collected with a Nicolet 510 FT-IR spectrometer in 4000–400 cm^{-1} range. Electronic absorption spectra were carried out on a Shimadzu UV3600 UV-Vis-NIR spectrophotometer. One-photon excited fluorescence spectra were measured with a RF-5301PC spectrophotometer in DMF solution at 298 K.

Synthesis of $[Hg_3Cl_6(L^1)_2]$ (1)

The aqueous solution of $HgCl_2$ (0.136 g, 0.5 mmol) was added slowly to a solution of L^1 (0.282 g, 1.0 mmol) in ethanol. The mixture was refluxed at 80°C for 6 h, and then cooled to room temperature. Dark red crystals of **1** were obtained from the mother liquid after 3 weeks

Table 1. Crystal data and structure refinement details for complexes 1 and 2.

Complex	**1**	**2**
Formula	$C_{34}H_{36}N_4O_4Cl_6Hg_3$	$C_{36}H_{40}N_4O_6Cl_6Hg_3$
Formula weight	1379.14	1439.19
Crystal system	triclinic	triclinic
Space group	P-1	P-1
V(Å^3)	2031.6(3)	2133.5(4)
Z	2	2
a(Å)	10.0394(6)	9.9027(12)
b(Å)	11.6830(9)	11.8421(15)
c(Å)	17.9608(14)	19.050(2)
α(°)	76.6540(10)	74.8560
β(°)	82.3900(10)	81.700
γ(°)	88.756(2)	88.772(3)
Reflections collected	10231	15433
Unique reflections (R$_{\text{int}}$)	7019	7519
R_1 [$I > 2\sigma(I)$]	0.0580	0.0855
wR_2	0.1441	0.1444
GOF	1.012	1.045

at room temperature in 53% yield based on $HgCl_2$. Anal. Calc. for $C_{34}H_{36}N_4O_4Cl_6Hg_3$: C 29.58, H 2.61, N 4.06%. Found: C 29.55, H 2.65, N 4.10%. IR (KBr, pellet, cm^{-1}): 1645 vs; 1601 vs; 1385 vs;1180 vs; 815 w; 542 w.

Synthesis of $[Hg_3Cl_6(L^2)_2]$ (2)

Complex 2 was synthesized following the same procedure described for 1 except L2 (0.312 g, 1.0 mmol) was used instead of L1 in 71% yield based on HgCl2. Anal. Calc. for $C_{36}H_{40}N_4O_6Cl_6Hg_3$: C 30.02, H 2.78, N 3.89%. Found: C 30.05, H 2.75, N 3.92%. IR (KBr, pellet, cm—1): 1640 vs; 1605 vs; 1386 vs;1182 vs; 816 w; 541 w.

Structure determination

The crystallographic data for complexes **1**and **2** were collected on a Bruker SMART APEX CCD diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). By using SADABS program [24], empirical absorption corrections were carried out. The structures were solved by direct process and refined anisotropically for all nonhydrogen atoms on F^2 by the full-matrix least-square method [24]. Detailed crystallographic data and refinement parameters for **1**and **2** are listed in Table 1, and selected bond lengths and angles are provided in Table 2. Crystallographic data for the structures reported here have been deposited with CCDC (Deposition No. CCDC-1063259 for **1** and 1063263 for **2**). These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html or from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, E-mail: deposit@ccdc.cam.ac.uk.

3. Results and discussion

Structure of $[Hg_3Cl_6(L^1)_2]$ (1)

Complex **1** crystallizes in triclinic system with the P-1 space group. The asymmetric unit of **1** has three Hg(II) atoms, six chlorine atoms and two L^1 ligands (Figure 2). The Hg(2) atom is four-coordinated by two chlorine atoms and two carboxylic oxygen atoms, locating in

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

Bond lengths (Å)	bond angles (°)
Complex **1**	
Hg(1)-Cl(1)= 2.298(4)	Cl(1)-Hg(1)-O(1)= 106.7(3)
Hg(1)-Cl(2)= 2.306(4)	Cl(4)-Hg(2)-Cl(3)= 160.35(17)
Hg(1)-O(1)= 2.448(10)	Cl(1)-Hg(1)-Cl(2)= 161.41(17)
Hg(2)-O(3)= 2.481(8)	Cl(2)-Hg(1)-O(1)= 90.9(3)
Hg(2)-O(2) =2.525(11)	Cl(4)-Hg(2)-O(3)= 94.0(2)
Hg(2)-Cl(3) =2.328(4)	Cl(4)-Hg(2)-O(2)= 98.2(3)
Hg(2)-Cl(4) =2.324(4)	O(3)-Hg(2)-O(2)= 79.5(3)
Hg(3)-Cl(5)= 2.305(4)	Cl(5)-Hg(3)-O(4)= 100.1(2)
Hg(3)-Cl(6)= 2.314(4)	Cl(3)-Hg(2)-O(3)= 99.1(2)
Hg(3)-O(4)= 2.410(8)	Cl(3)-Hg(2)-O(2)= 98.5(3)
	Cl(5)-Hg(3)-Cl(6)= 160.84(14)
	Cl(6)-Hg(3)-O(4)= 98.6(2)
Complex 2	
Hg(1)-Cl(1)= 2.346(6)	Cl(1)-Hg(1)-O(4)= 100.0(4)
Hg(1)-Cl(2)= 2.303(7)	Cl(2)-Hg(1)-O(1)= 96.5(4)
Hg(1)-O(1)= 2.464(13)	Cl(3)-Hg(2)-O(2)= 98.3(4)
Hg(1)-O(4)= 2.456(13)	Cl(3)-Hg(2)-Cl(4)= 160.1(2)
Hg(2)-Cl(3)= 2.318(6)	Cl(5)-Hg(3)-Cl(6)= 168.4(2)
Hg(2)-Cl(4)= 2.321(6)	Cl(6)-Hg(3)-O(5)= 87.5(4)
Hg(2)-O(2)= 2.392(12)	Cl(1)-Hg(1)-Cl(2)= 160.2(2)
Hg(3)-Cl(5)= 2.292(7)	Cl(2)-Hg(1)-O(4)= 98.1(4)
Hg(3)-Cl(6)= 2.297(7)	Cl(1)-Hg(1)-O(1)= 94.5(4)
Hg(3)-O(5)= 2.603(13)	O(4)-Hg(1)-O(1)= 79.5(4)
	Cl(4)-Hg(2)-O(2)= 100.6(4)
	Cl(5)-Hg(3)-O(5)= 102.7(4)

a distorted tetrahedron environment. However, Hg(1) and Hg(3) atoms are tri-coordinated to two chlorine atoms and one carboxylic oxygen atom, which is rare for Hg(II) complex. The Hg-O bond lengths are 2.410(8)–2.525(11)Å, being much shorter than that found in Cu(2-pyrazinecarboxylate)$_2$HgCl$_2$ (2.736(4) Å) [25], while the Hg-Cl bond lengths are close to that observed in related Hg(II) complexes [26]. In **1**, the carboxyl groups adopt bidentate bridging coordination mode, connecting adjacent Hg(II) atoms, forming a trinuclear [Hg$_3$(COO)$_2$] unit. The L^1 ligands are parallel attached to the [Hg$_3$(COO)$_2$] unit, as depicted in figure 2. Least-square plane calculations indicate that the benzene and pyridinium rings of L^1 possess perfect planarity and the maximum deviations to their corresponding planes

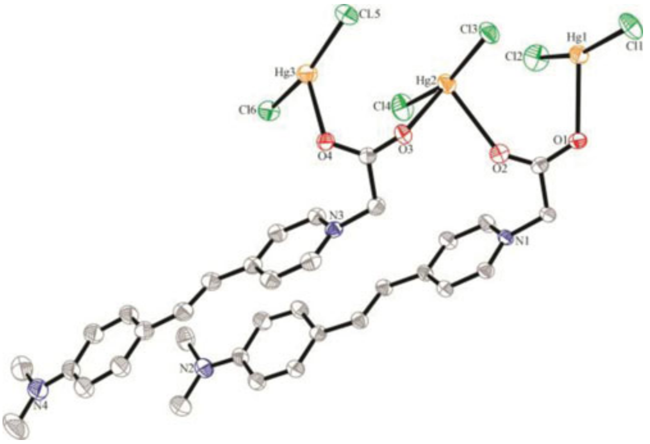

Figure 2. ORTEP view of the coordination sphere with the atom numbering scheme for complex 1. (30% probability factor for the thermal ellipsoids; H-atoms are omitted for clarity.)

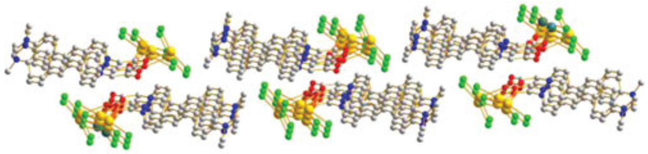

Figure 3. Packing diagram for complex 1.

are near 0.01 Å. Compared with the metal-free ligand, the bond lengths, bond angles and the planarity of L^1have no obvious differences upon Hg(II) ions binding. These results indicate that the metal ions exert few influences on the electron distribution in the ligands. The packing diagram shows that compound **1** stacks along the *a* axis to form polar sheets, and the adjacent sheets are arranged in a head-to-tail manner along the *c* direction (Figure 3).

Structure of [$Hg_3Cl_6(L^2)_2$] (2)

When L^2 was used instead of L^1 ligand, complex **2** was obtained. Complex **2** shares much structural similarity with complex **1**. The asymmetric unit consists of three crystallographically independent Hg(II) atoms, six chlorine atoms and two L^2 ligands. As depicted in figure 4, Hg1 atom exhibits a distorted tetrahedral coordination geometry, coordinated by two chlorine atoms and two oxygen atoms of two different L^2 ligands. Hg2 and Hg3 atoms adopt planar triangular geometry completed by two chlorine atoms and one oxygen atom from L^2 ligand. The Hg-O and Hg-Cl bond lengths are comparable with those of complex **1**. The Hg(II) atoms are bridged by two carboxyl groups, affording the trinuclear [$Hg_3(COO)_2$] motif. Two L^2 ligands are almost parallel arranged along one side of the unit with the dihedral angle between the two benzene rings being 4.8°. The packing diagram reveals that complex **2** stacks in a manner similar to that for complex **1**. However, the intermolecular hydrogen bonds involved the hydroxyl and the coordinated chlorine atom (O3-H3···Cl1, 3.421Å) connect the neighboring molecules to generate a 1D chain (Figure 5).

Recently, we reported the structure of [$Hg(L)Cl_2$]$_n$ (L= *trans*-4-[(*p*-*N*,*N*-dimethylamino) styryl]-*N*-(2-propanoic-acid)pyridinium) (**I**) [27], in **I**, the Hg(II) atoms locate in slightly

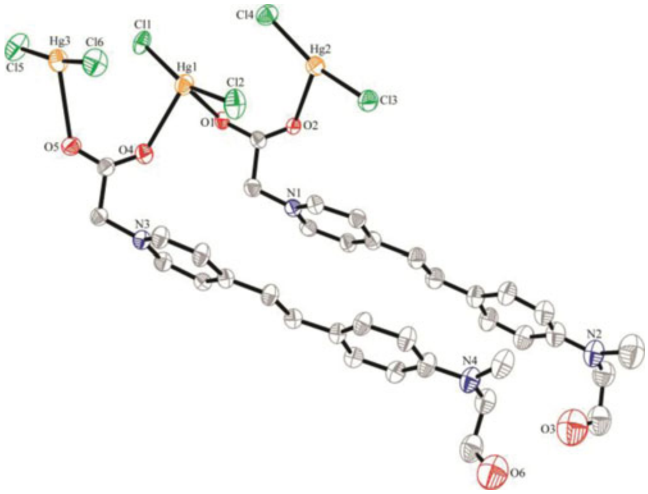

Figure 4. ORTEP view of complex 2 showing the coordination environment of the Hg(II) atoms. (30% probability factor for the thermal ellipsoids; H-atoms are omitted for clarity.)

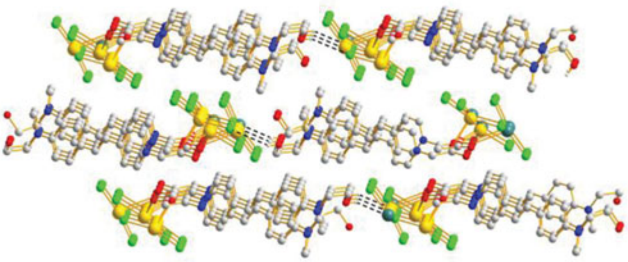

Figure 5. View of the 1D chain of complex 2 formed by the intermolecular hydrogen bonding interactions (black dashed lines).

distorted tetrahedral coordination geometry, completed by two chlorine atoms and two carboxylic oxygen atoms. However, in **1** and **2**, Hg(II) atom lies in distorted tetrahedronor planar triangle environments. Similar to that in **1** and **2**, the carboxyl group also adopts bidentate bridging coordination fashion in **I**, however, the carboxyl groups bridge symmetry-related Hg(II) atoms, forming a 1D $[Hg(COO)_2]_n$ chain.

Linear absorption and one-photon–induced fluorescence

The linear absorption spectra of the synthesized complexes were measured on a Shimadzu UV3600 UV-Vis-NIR spectrophotometer using samples of 10^{-5} mol/L in DMF. The absorption maxima are 460 and 465 nm for **1** and **2**, respectively. The linear absorption spectrum of complex **1** is depicted in figure6 as representative. It should be pointed out that there is no linear absorption in the range from 600 to 1100 nm. This implies that the emission excited by 1064 nm laser beam must be resulted from a multiphoton absorption process.

Figure7 shows the one-photon excited fluorescence spectra for**1** and**2** in dilute DMF solutions (10^{-6} mol/L) at the excitation of 460 nm. The fluorescence peaks for **1** and **2** are located at 590 nm and 605 nm, respectively. It is should be pointed out that there is a considerable overlap between the absorption and fluorescence spectra in the range of 500–550 nm.

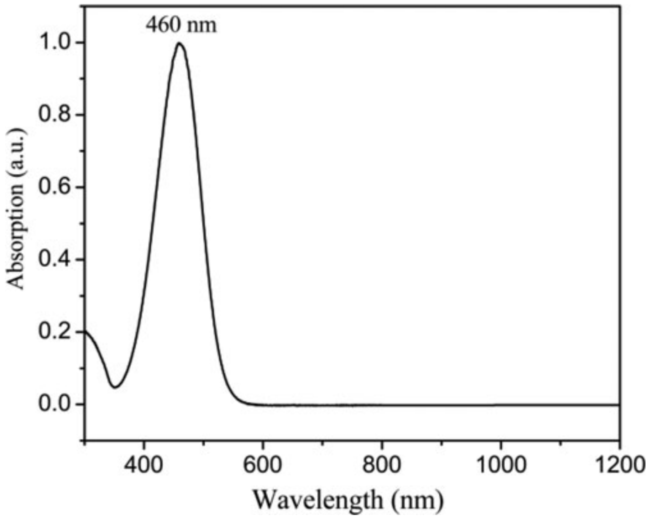

Figure 6. Linear absorption spectrum for complex 1 in DMF solution (1×10—5 mol/L).

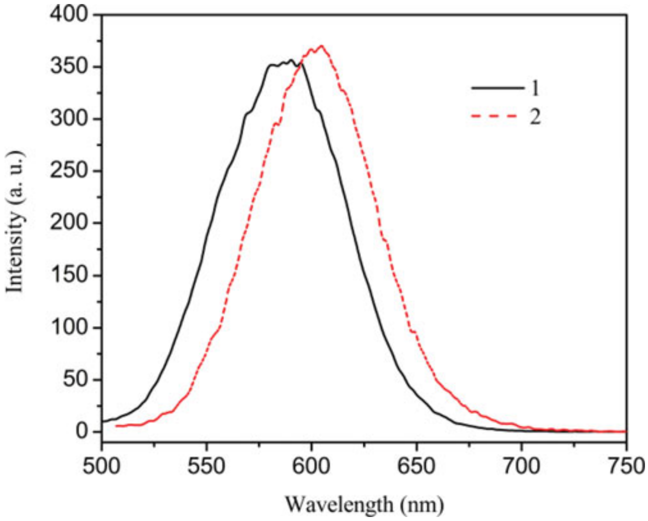

Figure 7. One-photon excited fluorescence spectra for 1 and 2 in DMF solution with concentration of 1 × 10—6 mol/L.

The two-photon-excited fluorescence (TPEF) spectra of 1 × 10^{-3}mol/L solutions for**1** and **2** in DMF are given in figures 8 and 9. These spectra were obtained with a streak camera as recorder and a passively mode-locked Nd:YAG laser as excitation source. Upon irradiation with the laser pulse at 1064 nm, intense fluorescence emission can be measured. The quadratic dependence of the emission intensity on the pump intensity, demonstrating the emission can be assigned to a two-photon absorption process.

The two-photon-excited fluorescence peak values for **1** and **2** are 625 nm and 622 nm, which are red-shifted 35 and 17 nm, relative to those of their one-photon fluorescence. The red-shift may arise from the reabsorption effect of the fluorescence in the solution. As shown in figures 6-9, the blue side of the fluorescence is overlapped on the red side of the linear absorption spectrum. In the case of one-photon excitation, a dilute solution (1 × 10^{-6}mol/L)

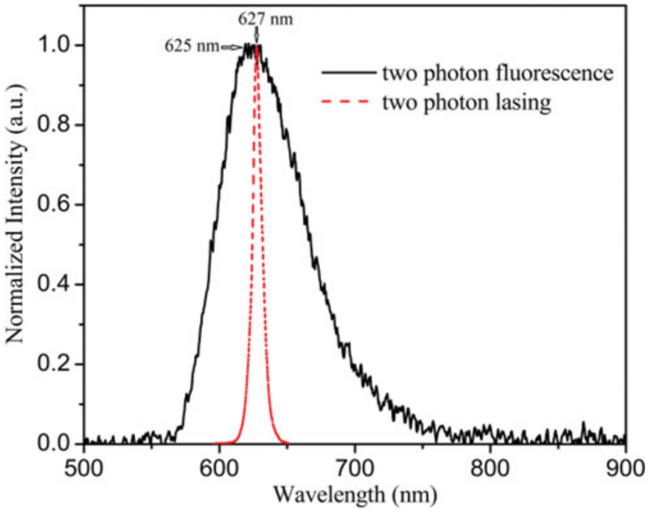

Figure 8. Two-photon excited fluorescence and lasing spectra for complex 1.

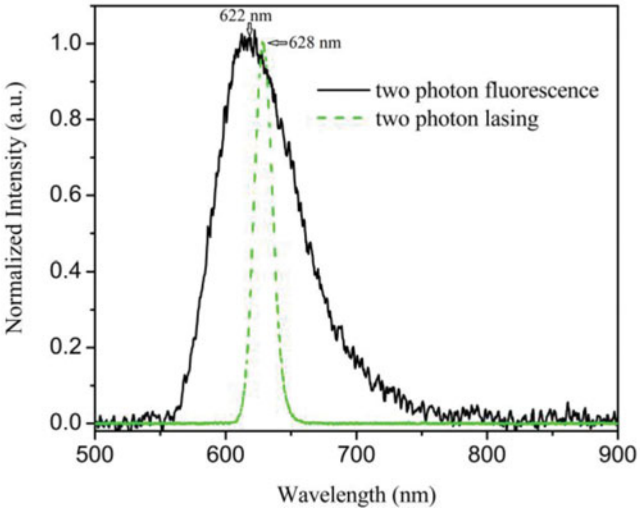

Figure 9. Two-photon excited fluorescence and lasing spectra for complex 2.

was used and the reabsorption effect of the fluorescence in the solution can be neglected. However, the TPEF experiments were carried out with concentrated solution (1×10^{-3}mol/L). The reabsorption effect increases significantly, which results an apparent red-shift of the fluorescence.

In the two-photon pumped lasing experiments, a mode-locked Nd:YAG laser was involved as pump source. The output wavelength, pulse duration, repetition rate are 1064 nm, 40 ps and 10Hz, respectively. Through $f = 15$ cm lens, the laser beam was focused onto the centre of solution sample filled in a 1 cm path quartz cell.

Figures8 and 9 display the sharp TPP lasing spectra of **1** and **2** in DMF solution. The central lasing wavelengths locate at 627 and 628 nm for **1** and **2**, respectively. Comparing figure7 with figures8 and 9, it can be seen that the TPP lasing has much narrower FWHM (9 nm for **1** and 14 nm for **2**) than their corresponding TPP fluorescence (75 nm for **1** and **2**) and one-photon fluorescence (71 nm for **1** and 67 nm for **2**). The peak positions of lasing are considerably red-shifted with respect to those of one-photon fluorescence. The results are in accordance with that reported in reference and can be explained by reabsorption effect [28].

Conclusion

Two new Hg(II) complexes based on D-π-A type chromophores have been synthesized and their structures have been determined. Their linear and nonlinear optical properties have been studied. Intense two-photon pumped fluorescence and lasing around 625 nm were found for both complexes. Our results suggest that stable complexes are promising as two-photon-based materials.

Acknowledgments

This work was financially supported by the National Natural Science Foundations of China (21401061), Scientific Research Foundation of Anhui Provincial Education Department (KJ2014A228) and Training Programs of Innovation and Entrepreneurship for Undergraduates (AH201410373039).

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