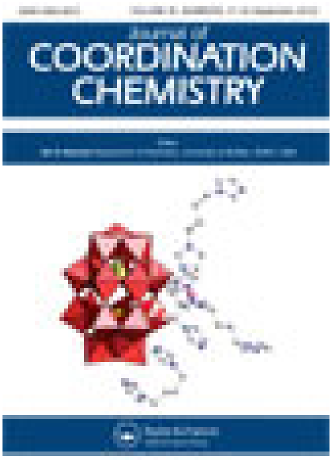

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Coordination-driven synthesis of Ag(I) compounds based on a double emission ligand consisting of 1,3,4-oxadiazole and cyclotriphosphazene units

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Coordination-driven synthesis of Ag(I) compounds based on a double emission ligand consisting of 1,3,4-oxadiazole and cyclotriphosphazene units

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A new ligand (**L**) which consists of cyclotriphosphazene and 1,3,4-oxadiazole units is reported. Two new Ag(I) coordination compounds $\{[Ag(L)SO_3CF_3]_n$ (**1**) and $Ag_2L_2(NO_3)_2$ (**2**)$\}$ based on **L** and Ag(I) salts are obtained. Compound **1** features a 1-D chain, in which the ligand **L** adopts a divergent *trans*-conformation, whereas **2** is a discrete binuclear Ag(I) molecule in which **L** adopts convergent *cis*-conformation. Compounds **1** and **2** are fully characterized by ^{1}H-NMR, Infrared, elemental analysis, X-ray powder, and single-crystal diffraction. Luminescent properties of **1** and **2** are investigated.

Keywords: 1,3,4-Oxadiazole; Cyclotriphosphazene; Ag(I); Coordination compounds

1. Introduction

Interest has focused on coordination compounds with potential applications in sensing, photoluminescence, ion exchange, separations, gas storage, and catalysis [1, 2]. Recently, effort has been dedicated to functional inorganic rings and cages as scaffolds for assembling supramolecular compounds. For example, cyclotriphosphazenes, as an important class of inorganic nondelocalized cyclic units [3, 4], have been used to construct organic or metal-organic supramolecular systems [5, 6]. Supramolecular compounds based on both cyclotriphosphazenes and five-membered heterocycles are relatively rare. We have longstanding interest in coordination chemistry of bent organic ligands bridged by five-membered heterocycles such as oxadiazole and triazole, especially in how different types of linkages and orientations impact the structures of various coordination-driven supramolecular compounds [7–9]. In this contribution, we present a new cyclotriphosphazene ligand (**L**) containing 1,3,4-oxadiazole,

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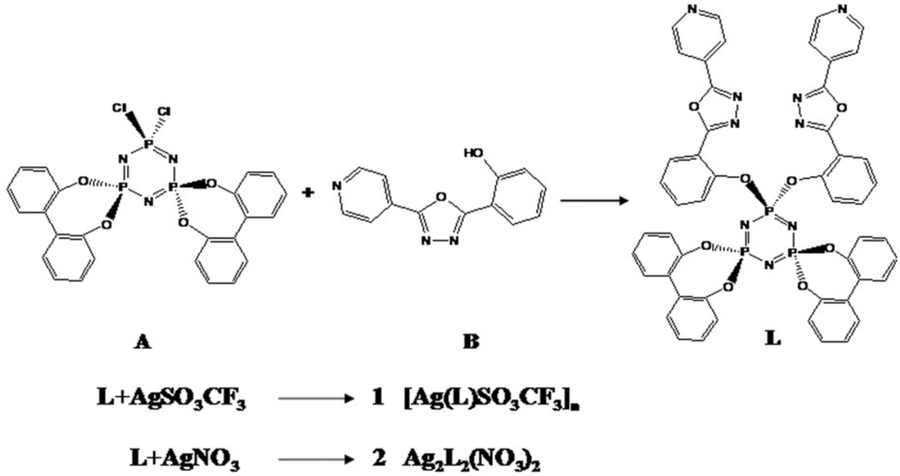

Scheme 1. Synthesis of **1** and **2**.

bis(2,2′-dioxybiphenyl)bis(2-phenoxy-5-(4-pyridyl)-1,3,4-oxadiazole)cyclotriphosphazene and two new Ag(I) coordination compounds (**1** and **2**) based on it (scheme 1). Compounds **1** and **2** have been fully characterized by ^{1}H-NMR, Infrared (IR), elemental analysis, X-ray powder and single-crystal diffraction. In addition, the photoluminescence of **1** and **2** are investigated in the solid state.

2. Experimental

2.1. *Physical measurements*

$AgSO_3CF_3$ and $AgNO_3$ (Acros) were used as obtained without purification. IR samples were prepared as KBr pellets and spectra were obtained in the 400–4000 cm^{-1} range using a Perkin-Elmer 1600 FTIR spectrometer. Elemental analyses were performed on a Perkin-Elmer model 2400 analyzer. ^{1}H-NMR data were collected using a Bruker AM-300 spectrometer. Chemical shifts are reported in δ relative to TMS. All fluorescence measurements were carried out on a Cary Eclipse spectrofluorimeter (Varian, Australia) equipped with a xenon lamp and quartz carrier at room temperature. XRD patterns were obtained on a D8 ADVANCE X-ray powder diffractometer (XRD) with Cu-Kα radiation ($\lambda = 1.5405$ Å).

2.2. *X-ray structural studies*

Suitable single crystals of **1** and **2** were selected and mounted in air onto thin glass fibers. X-ray intensity data of **1** and **2** were measured at 173 K on a Bruker SMART Apex CCD-based diffractometer (Mo-Kα radiation, $\lambda = 0.71073$ Å). The raw frame data for **1** and **2** were integrated into SHELX-format reflection files and corrected for

Table 1. Crystallographic parameters of **1** and **2**.

Parameters	**1**	**2**
Empirical formula	$C_{51}H_{32}AgF_3N_9O_{11}P_3S$	$C_{100}H_{64}Ag_2N_{20}O_{22}P_6$
Formula weight	1236.70	2299.27
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	8.8900(11)	10.6079(19)
b	14.9601(19)	14.672(3)
c	18.655(2)	15.413(3)
α	87.538(2)	88.441(3)
β	86.282(2)	80.379(3)
γ	86.973(2)	88.504(3)
Volume (Å^3), Z	2470.4(5), 2	2363.7(8), 1
Calculated density (g cm^{-3})	1.663	1.615
Absorption coefficient (mm^{-1})	0.633	0.604
$F(000)$	1248	1164
Crystal size (mm^3)	$0.35 \times 0.30 \times 0.16$	$0.26 \times 0.16 \times 0.12$
Reflections collected	13,109	12,482
Independent reflection	9046 [R(int) = 0.0183]	8625 [R(int) = 0.0251
Goodness-of-fit on F^2	1.025	1.014
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0506$, $wR_2 = 0.1289$	$R_1 = 0.0538$, $wR_2 = 0.1303$
R indices (all data)	$R_1 = 0.0641$, $wR_2 = 0.1394$	R1 = 0.0759, $wR_2 = 0.1444$

Lorentz and polarization effects using SAINT. Corrections for incident and diffracted beam absorption effects were applied using SADABS. The crystals showed no evidence of crystal decay during data collection. The structures were solved by a combination of direct methods and difference Fourier syntheses and refined against F^2 by full-matrix least squares. Crystal data, data collection parameters, and refinement statistics for **1** and **2** are listed in table 1.

2.3. *Synthesis of bis(2,2′-dioxybiphenyl)bis(2-phenoxy-5-(4-pyridyl)-1,3,4-oxadiazole)cyclotriphosphazene (L)*

To solution of **A** (2-phenoxy-5-(4-pyridyl)-1,3,4-oxadiazole (0.50 g, 2.1 mmol) in 30 mL THF) was added NaH (50.4 mg, 2.1 mmol) and the mixture was stirred for 1 h at room temperature, then **B** ([N_3P_3(2,2′-dioxybiphenyl)$_2Cl_2$] (0.574 g, 1 mmol) in 20 mL THF) was added. The mixture was heated at reflux over 3 h, allowed to cool, the precipitate separated by filtration, washed several times with water, and the residue was purified on a silica gel column using CH_2Cl_2 : MeOH = 20 : 1 as the eluent to afford **L** as a white crystalline solid (yield: 0.62 g, 60.8%). IR (KBr pellet, cm^{-1}): 1608(m), 1540(w), 1477(m), 1435(m), 1410(w), 1276(m), 1233(m), 1175(s), 1090(m), 1033(m), 932(w), 890(s), 818(w), 785(m), 750(m), 716(m), 701(m), 630(w), 608(m), 532(m), 409(w). ^{1}H-NMR (300 MHz, 25°C, DMSO-d$_6$, TMS, ppm): 8.41 (d, 2H, –C_5H_4N), 8.26 (d, 1H, –C_6H_4O), 8.02 (d, 2H, –C_5H_4N), 7.76(d, 1H, –C_6H_4O), 7.51–7.39 (m, 3H, –C_6H_4O, –$C_{12}H_8O_2$), 7.28–7.26 (m, 5H, –C_6H_4O, –$C_{12}H_8O_2$), 7.03 (d, 2H, –$C_{12}H_8O_2$). Anal. Calcd for $C_{50}H_{32}N_9O_8P_3$: C, 61.29; H, 3.30; N, 12.86. Found: C, 61.33; H, 3.24; N, 12.89.

2.4. *Synthesis of [Ag(L)SO$_3$CF$_3$]$_n$ (1)*

The solution of $AgSO_3CF_3$ (5.20 mg, 0.02 mmol) in THF (7 mL) was layered onto a solution of **L** (19.6 mg, 0.02 mmol) in MeOH (7 mL). The system was left for one day at room temperature and colorless crystals of **1** were obtained (yield: 15.6 mg, 63.1%). IR (KBr pellet, cm^{-1}): 1608(w), 1539(w), 1475(w), 1437(w), 1414(w), 1275(m), 1232(m), 1176(s), 1094(m), 1038(w), 976(w), 934(m), 890(s), 830(w), 786(m), 758(m), 712(w), 610(m), 531(m). ^{1}H-NMR (300 MHz, 25°C, DMSO-d_6, TMS, ppm): 8.52 (d, 4H, p-C_5H_4N), 8.25 (d, 2H, p-C_6H_4O), 7.78 (d, 4H, o-C_5H_4N), 7.74 (d, 4H, –C_6H_4O), 7.62 (d, 4H, –$C_{12}H_8O_2$), 7.48 (t, 2H, m-C_6H_4O), 7.43–7.04 (m, 8H, –$C_{12}H_8O_2$), 7.07 (d, 4H, –$C_{12}H_8O_2$). Anal. Calcd for $C_{51}H_{32}AgF_3N_9O_{11}P_3S$: C, 49.53; H, 2.61; N, 10.19. Found: C, 45.46; H, 2.56; N, 10.25.

2.5. *Synthesis of Ag$_2$L$_2$(NO$_3$)$_2$ (2)*

The solution of $AgNO_3$ (3.40 mg, 0.02 mmol) in THF (7 mL) was layered onto a solution of **L** (19.6 mg, 0.02 mmol) in MeOH (7 mL). The system was left for three days at room temperature and colorless crystals of **2** were obtained (yield: 16.32 mg, 71%). IR (KBr pellet, cm^{-1}): 1658(s), 1613(w), 1542(w), 1476(w), 1439(w), 1414(w), 1268(m), 1259(s), 1230(m), 1172(s), 1096(m), 1035(w), 979(w), 930(m), 892(s), 782(m), 758(m), 712(w), 610(m), 531(m). ^{1}H-NMR (300 MHz, 25°C, DMSO-d_6, TMS, ppm): 8.49 (d, 4H, p-C_5H_4N), 8.25 (d, 2H, p-C_6H_4O), 7.77 (d, 4H, o-C_5H_4N), 7.74 (d, 4H, –C_6H_4O), 7.62 (d, 4H, –$C_{12}H_8O_2$), 7.49 (t, 2H, m-C_6H_4O), 7.43-7.04 (m, 8H, –$C_{12}H_8O_2$), 7.07 (d, 4H, –$C_{12}H_8O_2$). Anal. Calcd for $C_{100}H_{64}Ag_2N_{20}O_{22}P_6$: C, 52.24; H, 2.81; N, 12.18. Found: C, 52.29; H, 2.73; N 12.09.

3. Results and discussion

3.1. *Description of the crystal structure of 1*

L was prepared from 2-phenoxy-5-(4-pyridyl)-1,3,4-oxadiazole (**A**) and N_3P_3(2,2′-dioxybiphenyl)$_2Cl_2$ (**B**). Compounds **1** and **2** were obtained by combination of **L** with AgX [X = $SO_3CF_3^-$ (**1**) and NO_3^- (**2**)] in mixed solutions (THF : CH_3OH = 1 : 1) under ambient conditions.

Compound **1** crystallizes in the triclinic space group $P\bar{1}$. As shown in figure 1 two arms, which are located at the same phosphorus (P3) in each ligand, adopt divergent *trans*-conformation to coordinate to two Ag(I) centers. Each Ag(I) is two-coordinate with a long non-bonding Ag···O contact (O from one $SO_3CF_3^-$); the coordination sites are occupied by two pyridyl N-donors from two ligands. The Ag–N distances range from 2.175(3) to 2.187(3) Å, while the Ag···O distance is 2.597(4) Å. The Ag–N and Ag···O distances are comparable to reported values [10, 11].

The Ag(I) centers in **1** are connected to each other by **L** to form a 1-D chain, and these chains are further linked by Ag···Ag interactions to form infinite 1-D double chains (figure 2). The corresponding Ag···Ag distance is 3.076(4) Å (figure 3a) which is consistent with reported values [12]. These parallel double chains are joined to generate

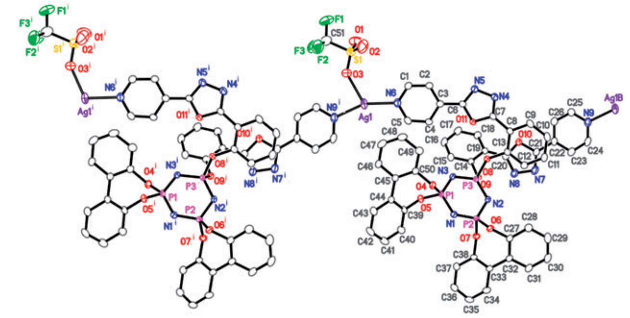

Figure 1. The structure of **1** with hydrogen atoms removed for clarity.

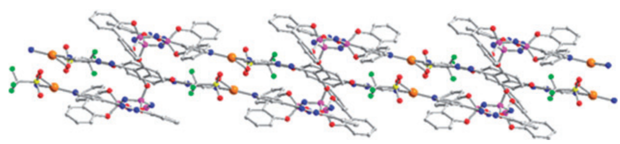

Figure 2. The 1-D double chain of **1**.

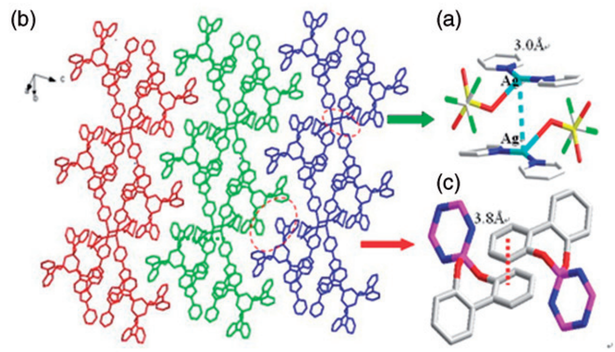

Figure 3. (a) The metal-metal bond between two Ag(I)'s from two adjacent chains. (b) The 2-D supramolecular network arranged by **1**. (c) π–π interactions between two adjacent 2,2′-biphenol molecules.

2-D sheets (figure 3b) *via* interchain π–π interactions ($d_{\pi-\pi} = \sim 3.8$ Å). The interchain π–π interactions are the result of the two adjacent 2,2′-biphenol molecules (figure 3c).

3.2. *Description of the crystal structure of 2*

In order to study the impact of various counterions on self-assembly based on **L**, NO_3^- was used instead of ellipsoidal $SO_3CF_3^-$. Compound **2** was crystallized with **L** and $AgNO_3$ in the same solvent system at room temperature. Similar to **1**, **2** crystallizes in the triclinic space group $P\bar{1}$. The two arms adopt convergent *cis*-conformations, different from those in **1**. Like in **1**, Ag(I) is two-coordinate with a long non-bonding Ag···O contact (O comes from one NO_3^-); the coordination positions are occupied by

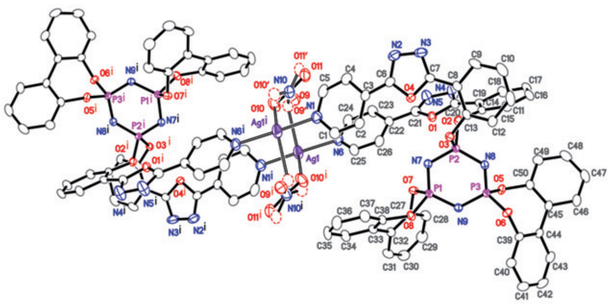

Figure 4. The structure of **2** with hydrogen atoms removed for clarity.

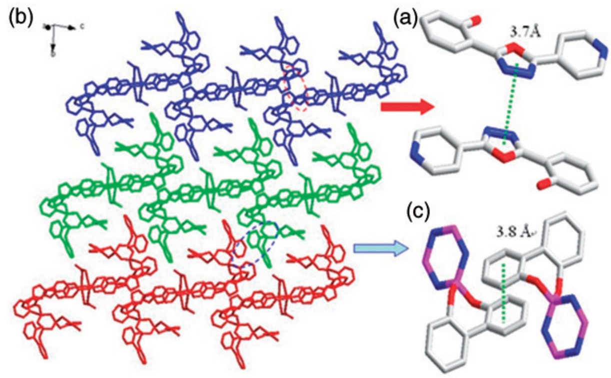

Figure 5. (a) π-π interactions between two 1,3,4-oxadiazole rings from two $Ag_2\mathbf{L}_2(NO_3)_2$ molecules. (b) The 2-D supramolecular network arranged by **2**. (c) π-π interactions between two adjacent 2,2′-biphenol molecules.

two pyridyl N-donors from two ligands (figure 4). The pyridyl rings of two arms in each **L** are partly parallel, the dihedral angle between two pyridyls is 32.064(8)° and the distance of pyridyl N(1)–N(6) is 3.765(8) Å. The Ag–N distances (ranging from 2.184(6) to 2.201(5) Å) are slightly longer than those of **1**, while the Ag···O distance (2.593(1) Å) is slightly shorter than that of **1**.

Compared to **1**, two Ag(I) centers in **2** are connected by **L** to form a discrete binuclear $Ag_2\mathbf{L}_2(NO_3)_2$; these discrete molecules are linked through π–π interactions ($d_{\pi-\pi} = \sim 3.7$ Å) between two 1,3,4-oxadiazole rings from two different molecules (figure 5a) into an infinite 1-D chain. Parallel chains stack to form a 2-D network (figure 5b) through π–π interactions ($d_{\pi-\pi} = \sim 3.8$ Å) from two adjacent 2,2′-biphenol molecules (figure 5c).

3.3. *XRPD patterns of 1 and 2*

Compounds **1** and **2** are obtained as pure phases, confirmed by X-ray powder diffraction. As shown in figure 6, XRPD patterns of **1** and **2** obtained from the bulk crystalline solids are identical to those of simulated ones based on single crystals.

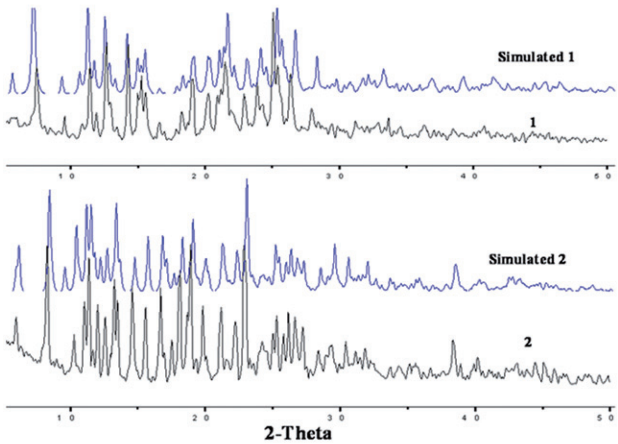

Figure 6. The XRPD patterns (black lines) obtained from the as-synthesized solids of **1** and **2** and the simulated XRPD patterns (blue lines) from single crystals of **1** and **2**.

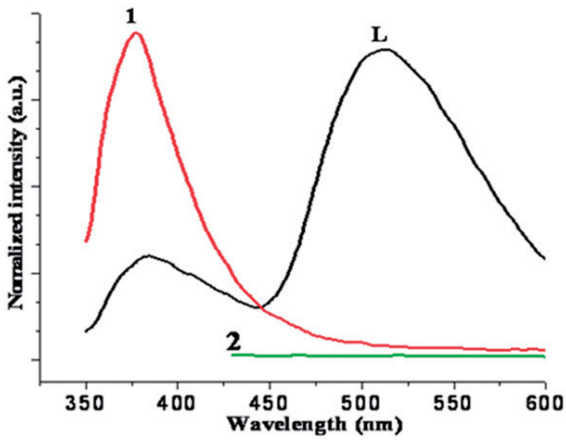

Figure 7. Solid-state photoinduced emission spectra of **1** and **2** at room temperature.

3.4. *Fluorescence of 1 and 2*

Synthesis of organic–inorganic coordination compounds by judicious choice of conjugated organic spacers and metal centers have proven to be an efficient method for obtaining new luminescent materials [13]. The photoluminescence properties of these two new compounds as well as the free ligand have been examined in the solid state at room temperature. **L** excitation spectra show a lambda-max at 337 nm, **L** exhibits two emission maxima at 377 and 510 nm, and **1** which has the same maximum excitation wavelength as **L**, exhibits a fluorescence enhancement at 377 nm and a fluorescence quenching at 510 nm. While **2** excitation spectra show a lambda-max at 409 nm, the fluorescence of **2** is completely quenched (figure 7). To understand different fluorescences of **1** and **2**, we study the coplanarities of three rings of each arm in **L**. In **1**, the dihedral angle between oxadiazole ring (C6, C7, N4, N5, O11) and phenyl ring

(C8, C9, C10, C11, C12, and C13) is 2.57(14)°, and the dihedral angle between oxadiazole ring and pyridyl ring (C1, C2, C3, C4, C5, and N6) is 4.74(12)°. In **2**, the dihedral angle between oxadiazole ring (C20, C21, N4, N5, and O1) and phenyl ring (C14, C15, C16, C17, C18, and C19) is 4.03(15)°, bigger than that of **1**, and the dihedral angle between oxadiazole ring and pyridyl ring (C22, C23, C24, C25, C26, and N6) is 13.02(15)°, also bigger than that of **1**. So the coplanarity of the three rings in **1** is better than that of **2**, which may lead to their different fluorescence properties [14].

4. Conclusions

Two new coordination compounds have been synthesized on the basis of a new double emission ligand, bis(2,2′-dioxybiphenyl)bis(2-phenoxy-5-(4-pyridyl)-1,3,4-oxadiazole)-cyclotriphosphazene (**L**) and AgX [X = $SO_3CF_3^-$ (**1**) and NO_3^- (**2**)]. $[Ag(\mathbf{L})SO_3CF_3]_n$ (**1**) shows a 1-D chain structure and **L** adopts divergent *trans*-conformation, whereas $Ag_2\mathbf{L}_2(NO_3)_2$ (**2**) is a discrete molecule, in which **L** adopts convergent *cis*-conformation. Compounds **1** and **2** are formed at room temperature compared with 2-D and 3-D silver-organic coordination polymers $[AgL]_n{}^{\bullet}nASF_6$, $[AgL]_n{}^{\bullet}nSbF_6$ [15] and $[Ag(NO_3)(APZ)]_n$, $\{[Ag(APZ)_2][CF_3COO]^{\bullet}H_2O\}_n$ [16] under ambient conditions. Furthermore, **1** exhibits strong fluorescence at 377 nm but **2** is completely quenched. So, we conclude that the different counterions $SO_3CF_3^-$ and NO_3^- in **1** and **2** result in their distinct structures and fluorescence properties. We are currently expanding the results presented here by preparing new ligands of this type with different substituted organic functional groups. We anticipate this approach to be useful for construction of a variety of new coordination compounds with interesting fluorescent properties.

Supplementary material

Additional crystallographic details and complete bond lengths and angles, coordinates and displacement parameters have been deposited at the Cambridge Crystallographic Data Center (CCDC) as supplementary publication no: CCDC 2155347 for **1** and CCDC 800579 for **2**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: (+44)1223-336-033; E-mail: deposit@ccdc.cam.ac.uk).

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