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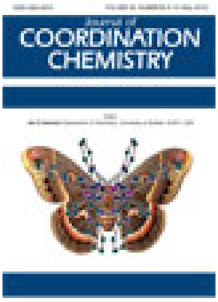

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Synthesis, structures, and fluorescence of azido-bridged dinuclear and thiocyanato-bridged tetranuclear cadmium(II) complexes derived from N-isopropyl-N′-(1-pyridin-2-ylethylidene)ethane-1,2-diamine

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Synthesis, structures, and fluorescence of azido-bridged dinuclear and thiocyanato-bridged tetranuclear cadmium(II) complexes derived from *N*-isopropyl-*N*′-(1-pyridin-2-ylethylidene)ethane-1,2-diamine

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A new azido-bridged dinuclear cadmium(II) complex $[Cd_2L_2(\mu_{1,1}\text{-}N_3)_2(ONO_2)_2]$ (**1**) and a new thiocyanato-bridged tetranuclear cadmium(II) complex $[Cd_4L_4(\mu_{1,3}\text{-}NCS)_4(NCS)_4]$ (**2**), where L is the tridentate Schiff base *N*-isopropyl-*N*′-(1-pyridin-2-ylethylidene)ethane-1,2-diamine, were prepared and characterized. X-ray single crystal structure determination reveals that **1** is a centrosymmetric end-on azido-bridged dinuclear cadmium(II) compound and **2** is a rarely seen centrosymmetric end-to-end thiocyanato-bridged tetranuclear cadmium(II) compound. Cd in each complex is in distorted octahedral coordination. In the crystal structure of **1**, the dinuclear cadmium(II) molecules are linked through N–H ··· O hydrogen bonds to form a 3-D network. In the crystal structure of **2**, the tetranuclear cadmium(II) complex molecules are linked through N–H ··· S hydrogen bonds to form 1-D chains. The thermal stability and preliminary fluorescence properties of the complexes were investigated.

Keywords: Synthesis; Crystal structure; Schiff base; Cadmium complex; Fluorescence

1. Introduction

Polymeric complexes containing bridging ligands have received attention because of their interesting molecular topologies and the fact that they may be designed with specific functionalities [1–3]. Azide and thiocyanate act as bridging ligands between metallic centers [4–8]. A major obstacle to a more comprehensive study of such azido-based or thiocyanato-based polymeric complexes is the lack of rational synthetic procedures, since with the present state of knowledge it is hardly possible to determine which coordination mode will be adopted by the azide or thiocyanate and whether the sought-after alternating polymeric structure will finally be formed. Recently, we reported the synthesis, structures, and luminescence properties of two azido- and thiocyanato-bridged dinuclear cadmium(II) complexes derived from the tridentate Schiff base *N*-isopropyl-*N*′-(1-pyridin-2-ylethylidene)ethane-1,2-diamine (L; scheme 1) [9]. In this work, two new cadmium(II) complexes, $[Cd_2L_2(\mu_{1,1}\text{-}N_3)_2(ONO_2)_2]$ (**1**) and

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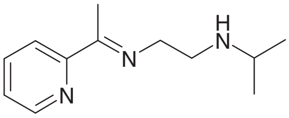

Scheme 1. The Schiff base ligand L.

$[Cd_4L_4(\mu_{1,3}\text{-}NCS)_4(NCS)_4]$ (**2**), were prepared by using the same starting materials and synthetic procedure as those reported previously, but with different molar ratio of L, cadmium nitrate, and bridging groups.

2. Experimental

2.1. *General methods and materials*

2-Acetylpyridine and *N*-isopropylethane-1,2-diamine were purchased from Aldrich Chemical Company Inc. and used as-received. All other reagents were of analytical grade. Elemental analyses (C, H, and N) were performed using a Perkin-Elmer 240 elemental analyzer. Infrared (IR) spectra were recorded on JASCO FT/IR-480 PLUS Fourier transform spectrophotometer with pressed KBr pellets from 200 to 4000 cm^{-1}. The Schiff base L was prepared according to the literature method [9]. Luminescence spectra were reported on a JASCO FP-6500 spectrofluorimeter (solid) from 200 to 850 nm. Thermal stability analysis was performed on a Perkin-Elmer Pyris Diamond TG-DTA thermal analyses system.

Caution: Sodium azide is potentially explosive, only small quantity should be used and handled with great care.

2.2. *Synthesis of $[Cd_2L_2(\mu_{1,1}\text{-}N_3)_2(ONO_2)_2]$ (1)*

To a methanol solution (10 mL) of L (0.1 mmol, 20.5 mg) and sodium azide (0.1 mmol, 6.5 mg) was added a methanol solution (10 mL) of $Cd(NO_3)_2 \cdot 4H_2O$ (0.1 mmol, 30.8 mg) with stirring. The mixture was stirred for 30 min at room temperature to give a clear colorless solution. Upon keeping the solution in air for a few days, colorless block-shaped crystals, suitable for X-ray diffraction, were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated by filtration, washed thrice with cold methanol, and dried in air. Yield: 53% on the basis of sodium azide. Anal. Calcd for $C_{24}H_{38}Cd_2N_{14}O_6$ (%): C, 34.2; H, 4.5; N, 23.2. Found (%): C, 34.0; H, 4.6; N, 23.3. IR data (cm^{-1}): 3228 (m, sh), 2050 (vs), 1657(s), 1588 (m), 1478 (w), 1439 (m), 1380 (w), 1333 (s), 1312 (s), 1250 (w), 1163 (m), 1132 (m), 1065 (m), 1010 (m), 961 (m), 899 (w), 809 (w), 788 (s), 747 (w), 653 (w), 580 (w), 412 (w).

2.3. *Synthesis of $[Cd_4L_4(\mu_{1,3}\text{-}NCS)_4(NCS)_4]$ (2)*

A procedure identical to that described for the preparation of **1**, but with sodium azide replaced by ammonium thiocyanate (0.1 mmol, 7.6 mg), produced colorless single

Table 1. Crystallographic data for **1** and **2**.

Complex	**1**	**2**
Empirical formula	$C_{24}H_{38}Cd_2N_{14}O_6$	$C_{56}H_{76}Cd_4N_{20}S_8$
Formula weight	843.5	1735.4
Temperature (K)	298(2)	298
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	P–1
Unit cell dimensions (Å, °)		
a	10.962(2)	10.833(2)
b	14.868(3)	12.612(3)
c	11.044(2)	14.073(3)
α	90	95.527(2)
β	112.721(2)	91.964(2)
γ	90	108.022(2)
Volume (Å^3), Z	1660.3(5), 2	1815.9(7), 1
Calculated density (g cm^{-3})	1.687	1.587
Absorption coefficient (mm^{-1})	1.341	1.435
F(000)	848	872
Reflections collected	13,232	14,706
Independent reflections	3603 [R(int) = 0.0236]	7700 [R(int) = 0.0185]
Observed reflections [$I \geq 2\sigma(I)$]	3007	6077
Restraints/parameters	0/211	0/403
Final R indices [$I \geq 2\sigma(I)$]	$R_1 = 0.0235$, $wR_2 = 0.0524$	$R_1 = 0.0267$, $wR_2 = 0.0493$
R indices (all data)	$R_1 = 0.0324$, $wR_2 = 0.0578$	$R_1 = 0.0404$, $wR_2 = 0.0546$

crystals of **2**. Yield: 37% on the basis of ammonium thiocyanate. Anal. Calcd for $C_{56}H_{76}Cd_4N_{20}S_8$ (%): C, 38.8; H, 4.4; N, 16.1. Found (%): C, 38.7; H, 4.5; N, 16.3. IR data (cm^{-1}): 3231 (m, sh), 3190 (m, sh), 2118 (vs), 2078 (vs), 2062 (vs), 1657 (s), 1589 (m), 1463 (w), 1441 (m), 1381 (s), 1331 (w), 1309 (m), 1249 (w), 1174 (w), 1135 (m), 1083 (w), 1067 (m), 1010 (m), 961 (m), 898 (w), 806 (w), 789 (s), 764 (w), 749 (w), 656 (w), 632 (w), 577 (w), 557 (w), 465 (w), 409 (w), 391 (w).

2.4. *X-ray data collection and structure determination*

Diffraction intensities for the complexes were collected at 298(2) K using a Bruker APEX II CCD area-detector with Mo-Kα radiation ($\lambda = 0.71073$ Å). The collected data were reduced using SAINT [10] and empirical absorption corrections were performed using SADABS [11]. The structures were solved by direct methods and refined against F^2 by full-matrix least-squares using the SHELXTL package [12]. All non-hydrogen atoms were refined anisotropically; hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. The crystallographic data for the complexes are summarized in table 1. Selected bond lengths and angles are summarized in table 2. Hydrogen bonds are listed in table 3.

3. Results and discussion

The molar ratio of bridging ligands to metal salts is an important factor to construct polymeric complexes with different topologies. In previously publication [9], two

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

1			
Cd1–N1	2.370(2)	Cd1–N2	2.3123(19)
Cd1–N3	2.334(2)	Cd1–N5	2.253(2)
Cd1–N5$^{\mathrm{i}}$	2.539(2)	Cd1–O1	2.357(2)
N5–Cd1–N2	151.22(8)	N5–Cd1–N3	119.17(8)
N2–Cd1–N3	75.05(7)	N5–Cd1–O1	84.05(8)
N2–Cd1–O1	116.65(8)	N3–Cd1–O1	108.51(9)
N5–Cd1–N1	94.32(8)	N2–Cd1–N1	68.66(7)
N3–Cd1–N1	143.69(7)	O1–Cd1–N1	87.20(9)
N5–Cd1–N5$^{\mathrm{i}}$	74.98(8)	N2–Cd1–N5$^{\mathrm{i}}$	80.81(7)
N3–Cd1–N5$^{\mathrm{i}}$	88.90(8)	O1–Cd1–N5$^{\mathrm{i}}$	157.59(8)
N1–Cd1–N5$^{\mathrm{i}}$	86.61(8)		
2			
Cd1–N1	2.403(2)	Cd1–N2	2.319(2)
Cd1–N3	2.396(2)	Cd1–S2	2.6208(10)
Cd1–S3$^{\mathrm{ii}}$	2.8448(9)	Cd1–N10	2.248(3)
Cd2–N4	2.356(2)	Cd2–N5	2.329(2)
Cd2–N6	2.335(2)	Cd2–N7	2.437(3)
Cd2–N8	2.229(3)	Cd2–N9	2.325(3)
N1–Cd1–S2	95.38(5)	N1–Cd1–S3$^{\mathrm{ii}}$	93.88(6)
N2–Cd1–N1	68.54(7)	S2–Cd1–S3$^{\mathrm{ii}}$	76.83(3)
N2–Cd1–N3	74.32(7)	N2–Cd1–S2	159.43(6)
N3–Cd1–N1	142.76(7)	N3–Cd1–S2	119.97(6)
N10–Cd1–N1	94.29(9)	N10–Cd1–N2	97.50(9)
N10–Cd1–N3	93.05(9)	N10–Cd1–S2	96.41(7)
N10–Cd1–S3$^{\mathrm{ii}}$	169.85(7)	N2–Cd1–S3$^{\mathrm{ii}}$	91.08(6)
N3–Cd1–S3$^{\mathrm{ii}}$	84.04(6)	N8-Cd2-N9	92.09(10)
N8–Cd2–N5	167.74(10)	N9–Cd2–N5	88.09(9)
N8–Cd2–N6	119.21(10)	N9–Cd2–N6	95.85(9)
N5–Cd2–N6	72.92(8)	N8–Cd2–N4	98.61(10)
N9–Cd2–N4	93.38(9)	N5–Cd2–N4	69.15(8)
N6–Cd2–N4	140.55(8)	N8–Cd2–N7	87.23(10)
N9–Cd2–N7	178.00(10)	N5–Cd2–N7	92.97(9)
N6–Cd2–N7	82.85(9)	N4–Cd2–N7	88.58(8)

Symmetry transformations used to generate equivalent atoms: **1**: (i) $1-x$, $2-y$, $-z$; **2**: (ii) $-x$, $2-y$, $1-z$.

Table 3. Geometrical parameters for hydrogen bonds.

Hydrogen bonds	D–H (Å)	H $\cdots$ A (Å)	D $\cdots$ A (Å)	D–H $\cdots$ A (°)
1				
N3–H3 $\cdots$ O3$^{\mathrm{iii}}$	0.91	2.45	3.315(3)	159
2				
N6–H6 $\cdots$ S1	0.91	2.84	3.593(3)	141
N3–H3 $\cdots$ S4$^{\mathrm{iv}}$	0.91	2.72	3.589(2)	159

Symmetry codes: **1**: (iii) $3/2-x$, $-1/2+y$, $1/2-z$; **2**: (iv) $-1+x$, $-1+y$, z.

azido- and thiocyanato-bridged dinuclear cadmium(II) complexes were prepared by the reaction of L, cadmium nitrate, and sodium azide or ammonium thiocyanate in a molar ratio of 1 : 1 : 3. In an attempt to investigate the influence of the molar ratio of the starting materials on the final structures of the complexes, in this article, two new cadmium(II) complexes were prepared by the reaction of L, cadmium nitrate, and sodium azide or ammonium thiocyanate in a molar ratio of 1 : 1 : 1. The complexes

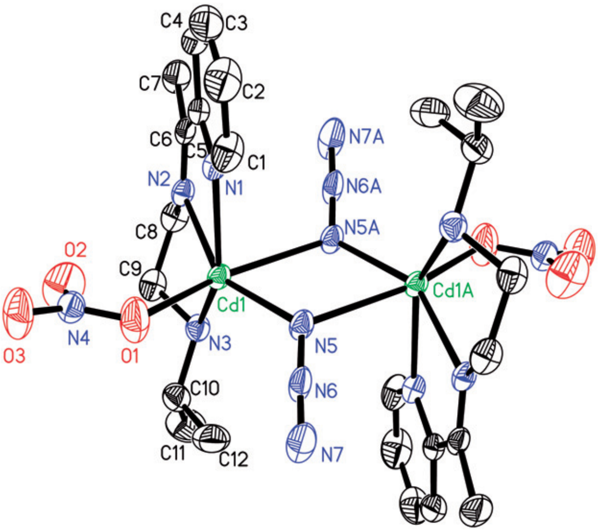

Figure 1. The structure of **1** showing the atom-numbering scheme. Atoms labeled with the suffix A or unlabeled atoms are at the symmetry position 1: $-x$, $2-y$, $-z$.

are soluble in common polar organic solvents, such as DMSO, DMF, methanol, ethanol, and acetonitrile, etc., but insoluble in water.

3.1. *Crystal structure description of 1*

Figure 1 gives the perspective view of **1** together with the atomic labeling system. The complex is a double end-on azido-bridged centrosymmetric dinuclear Schiff base cadmium(II) compound. The inversion center is located at the midpoint of the two Cd's. The complex contains two CdL(ONO_2) units connected to each other by two end-on azide bridges. The Cd ⋯ Cd distance is 3.806(1) Å. The Cd is in octahedral coordination, six-coordinated by the NNN donor set of the Schiff base and by one terminal N of one bridging azide, defining the equatorial plane, and by one terminal N of the other bridging azide and one nitrate, occupying the axial positions. The Cd1–N5 bond [2.253(2) Å] is much shorter than the Cd1–N5A bond [2.539(2) Å; symmetry code for A: $1-x$, $2-y$, $-z$], which might be caused by hindrance effects of the two CdL units. The O1–Cd1–N5A bond angle [157.6(1)°] in the complex severely deviates from the ideal value of 180°, also due to the same hindrance effects. The coordinate bond lengths in the complex are comparable to those observed in other Schiff base–cadmium(II) complexes [9, 13–16] and, as expected, the bond involving the amine nitrogen [2.334(2) Å] is longer than that involving the imine [2.312(2) Å]. The bridging azide groups are nearly linear and show bent coordination mode with Cd.

In the crystal structure of the complex, the dinuclear cadmium molecules are linked through N–H ⋯ O hydrogen bonds to form a 3-D network, as shown in figure 2.

3.2. *Crystal structure description of 2*

Figure 3 gives the perspective view of **2** together with the atomic labeling system. The complex is a rarely seen end-to-end thiocyanato-bridged centrosymmetric tetranuclear

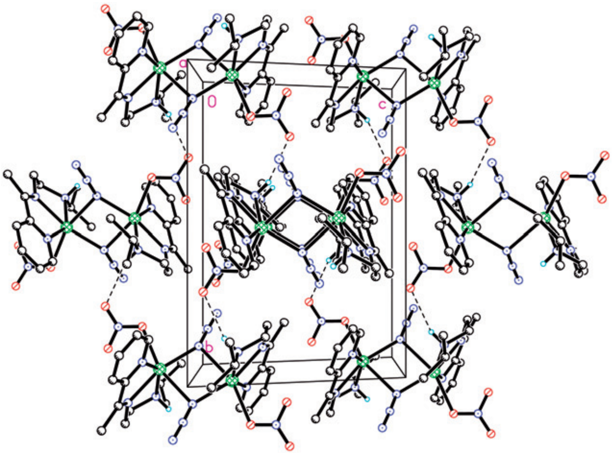

Figure 2. The molecular packing of **1**. Hydrogen bonds are shown as dashed lines.

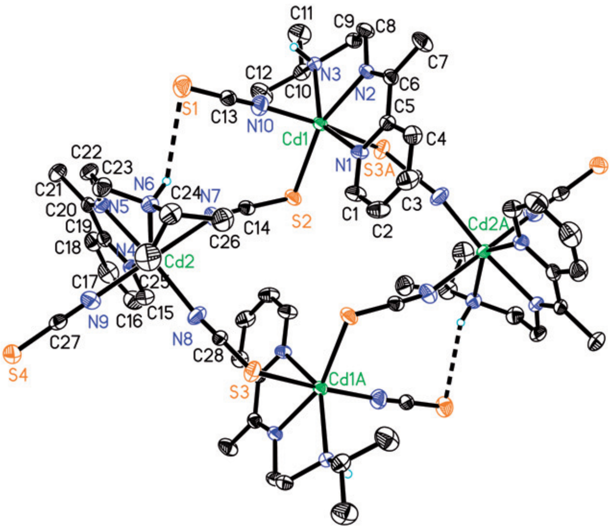

Figure 3. The structure of **2** showing the atom-numbering scheme. Atoms labeled with the suffix A or unlabeled atoms are at the symmetry position $-x$, $2-y$, $1-z$.

Schiff base–cadmium(II) compound. The inversion center is located at the midpoint of the parallelogram defined by the four Cd's. The molecule contains four CdL(NCS) units, connected to each other by four other end-to-end thiocyanates. The Cd ··· Cd distances are 6.741(1) and 6.527(1) Å. Cd1 in the complex is octahedral by the NNN donor set of the Schiff-base ligand and by one S of a bridging thiocyanate, defining the equatorial plane, and by one N of a terminal thiocyanate, and one S of another bridging thiocyanate, occupying axial positions. Cd2 in the complex is also six-coordinate by the NNN donor set of the Schiff base and by one N of a bridging thiocyanate, defining the equatorial plane, and by one N of a terminal thiocyanate, and one N of another bridging thiocyanate, occupying the axial positions. Coordinate bond lengths in the complex are comparable to those in **1** and also comparable to those observed in other

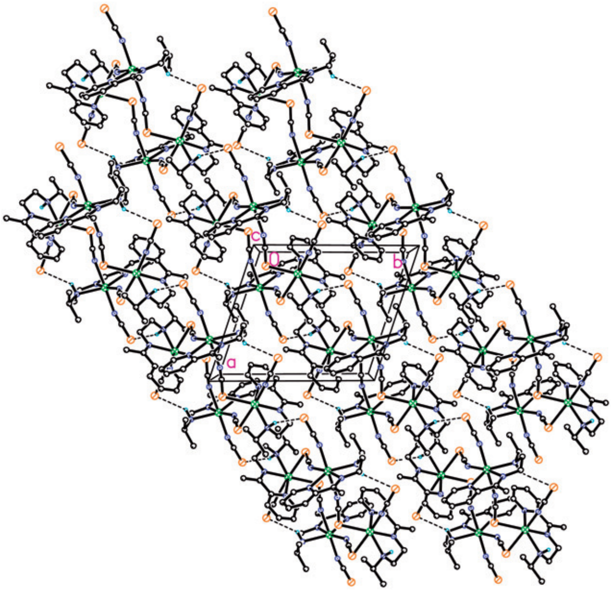

Figure 4. The molecular packing of **2**. Hydrogen bonds are shown as dashed lines.

Schiff base–cadmium(II) complexes [9, 13–16]. The bridging and terminal thiocyanate groups are nearly linear and show bent coordination with Cd.

In the crystal structure of the complex, there are two N–H $\cdots$ S hydrogen bonds in the tetranuclear unit. The tetranuclear cadmium molecules are further linked through N–H $\cdots$ S hydrogen bonds to form 1-D chains, as shown in figure 4.

3.3. *IR spectra*

IR spectra of L and the two complexes provide information about the metal–ligand bonding. Assignments are based on typical group frequencies. The medium and sharp absorptions at 3190–3250 cm^{-1} for L and the complexes can be assigned to ν(N–H) [17, 18]. In **2**, two different peaks at 3231 and 3190 cm^{-1} are assigned to N–H vibrations, which agree with the X-ray single crystal structure determination that there are two different types of N–H$\cdots$S hydrogen bonds. The intense absorption at 2050 cm^{-1} in **1**, and those at 2118, 2078, and 2062 cm^{-1} in **2**, are assigned to stretching vibrations of azide and thiocyanate, respectively [19, 20]. The strong absorption band at 1635 cm^{-1} in the spectrum of L is assigned to the azomethine, ν(C = N) [18], shifted to higher wavenumbers (1657 cm^{-1}) in the complexes. The shift of the absorption indicates coordination of the azomethine N to Cd [21]. Bands indicating nitrate in **1** are at 1440 and 1315 cm^{-1} [18]. In both complexes, the Schiff base coordination to Cd is substantiated by weak bands at 570–380 cm^{-1} [22, 23].

3.4. *Thermal stability*

Thermal gravimetric analyses (TGA) were conducted to examine the stability of the complexes (figure 5). The TGA trace shows that **1** has two-step thermal decomposition from 183°C to 622°C. The first mass loss of 25% started at 183°C and was completed

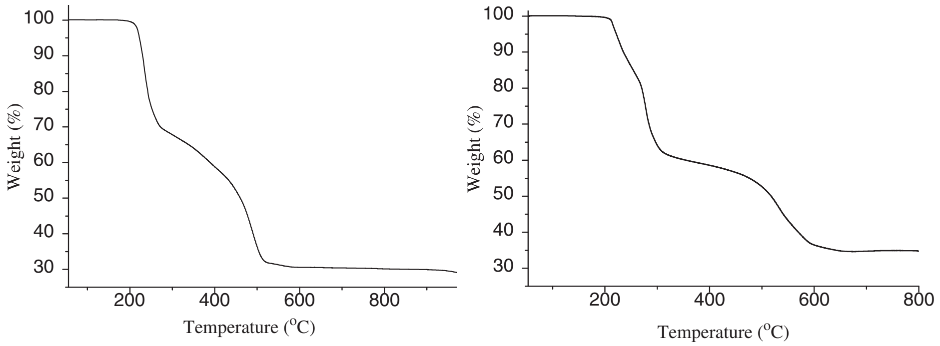

Figure 5. TGA curves of the complexes in air at a heating rate of 10°C min^{-1} (**1**; left) and (**2**; right).

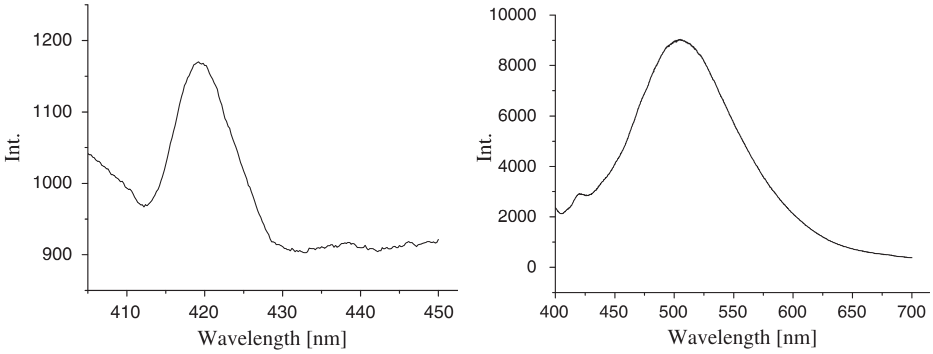

Figure 6. Emission spectra of (**1**; left) and (**2**; right).

at 251°C, corresponding to the loss of azide and nitrate (Calcd 24.7%). The second mass loss of 45% started at 251°C and was completed at 622°C, corresponding to the loss of the Schiff base. The final residue of 30.2% (Calcd 30.4%) at 622°C corresponds to CdO. The TGA trace shows that **2** also has a two-step thermal decomposition from 180°C to 652°C. The first of 27% started at 180°C and was completed at 280°C, corresponding to the loss of thiocyanate (Calcd 26.8%). The second mass loss of 42% started at 280°C and was completed at 652°C, corresponding to the loss of the Schiff base. The final residue of 30.7% (Calcd 29.6%) at 652°C corresponds to CdO.

3.5. *Fluorescence character description of the complexes*

The fluorescence properties of the complexes were studied at ambient temperature in the solid state. Figure 6 is the emission spectra of **1** and **2**; they exhibit different fluorescence, although constructed from the same Schiff base and metal. The emission band of **1** is from 410 to 430 nm, with $\lambda_{max} = 419$ nm ($\lambda_{ex} = 372$ nm). Complex **2** exhibits bands ranging from 405 to 650 nm with $\lambda_{max} = 505$ nm ($\lambda_{ex} = 375$ nm). The red-shift of the fluorescence of the complexes was also observed in other cadmium complexes [24]. For cadmium(II) complexes, no emission originating from metal-centered MLCT/LMCT excited states are expected, since Cd(II) is difficult to oxidize or reduce [25].

Thus, the emission observed in the complexes is tentatively assigned to the π–π^* intraligand fluorescence [13, 26, 27]. The co-ligands are different between the two complexes causing the different fluorescence properties of **1** and **2**.

4. Conclusions

Two new centrosymmetric polymeric cadmium(II) complexes with pseudohalide ligands were prepared and structurally characterized. In both complexes, Cd is distorted octahedral. The Schiff base *N*-isopropyl-*N*′-(1-pyridin-2-ylethylidene)ethane-1,2-diamine coordinates to Cd through three nitrogen atoms. The molar ratio of the bridging ligands to the metal salts influences the final structures of the Schiff base–cadmium complexes. Fluorescence measurements show that **1** and **2** emit fluorescent bands at 419 and 505 nm, respectively, which agree with those reported in the literature.

Supplementary material

CCDC-855768 (**1**) and 855769 (**2**) contain the supplementary crystallographic data for this article. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/const/retrieving.html or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-336033 or E-mail: deposit@ccdc.cam.ac.uk

Acknowledgments

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