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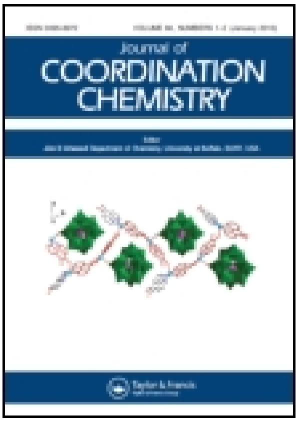

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Syntheses, structures, and luminescent properties of lanthanide complexes with N,N′-bis(p-methoxyphenyl)-3-oxapentanediamide

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Published online: 07 Sep 2011.

To cite this article: Yong-Hong Wen , Hui-Ling Wen , Lei Wang & Xiao-Hui Wu (2011) Syntheses, structures, and luminescent properties of lanthanide complexes with N,N′-bis(p-methoxyphenyl)-3-oxapentanediamide, Journal of Coordination Chemistry, 64:17, 3022-3030, DOI: 10.1080/00958972.2011.611242

To link to this article: http://dx.doi.org/10.1080/00958972.2011.611242

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Journal of Coordination Chemistry
Vol. 64, No. 17, 10 September 2011, 3022–3030

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Syntheses, structures, and luminescent properties of lanthanide complexes with N,N′-bis(*p*-methoxyphenyl)-3-oxapentanediamide

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(*Received 22 January 2011; in final form 25 July 2011*)

An amide-type acyclic polyether, *N,N*′-bis(*p*-methoxyphenyl)-3-oxapentanediamide (L), and its three lanthanide coordination compounds, $[Ln(NO_3)(L)_2(H_2O)_2] \cdot (NO_3)_2$ [Ln = Pr (**1**), Nd (**2**), and Dy (**3**)], have been synthesized and characterized by elemental analysis, IR spectroscopy, thermal analysis, and X-ray diffraction. Single crystal X-ray structure analyses reveal that **1** and **2** have the same structure and crystallize in the monoclinic crystal system with *C*2/*c* space group. The coordination geometry around the central atom is a distorted bicapped square antiprism. Extensive intermolecular hydrogen bonds in **1** and **2** result in 3-D supramolecular networks. Complex **3** exhibits luminescence in the visible region upon excitation with UV-rays.

Keywords: Acyclic polyether; Lanthanide complex; Crystal structure; Luminescence

1. Introduction

Acyclic polyether compounds receive attention because of their complexing ability with high selectivity to metal ions and their potential application in supramolecular chemistry and materials chemistry [1–3]. Diamide-type acyclic polyethers that have been used as the active materials for ion-selective electrodes and extractants for metal ions [4–9] are of particular interest. To further explore the relationship between structures and properties of rare earth complexes with such ligands, a flexible acyclic polyether, *N,N*′-bis(*p*-methoxyphenyl)-3-oxapentanediamide (L), was synthesized. Herein we report the synthesis, structural characterization, and luminescence of lanthanide nitrate complexes with L $[Ln(NO_3)(L)_2(H_2O)_2] \cdot (NO_3)_2$ [Ln = Pr (**1**), Nd (**2**), and Dy (**3**)].

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2011 Taylor & Francis
DOI: 10.1080/00958972.2011.611242

2. Experimental

2.1. *Materials and measurements*

All commercially available reagents and chemicals were of analytical grade and used without purification. Deionized water was obtained by passing distilled water through a Barnstead E-pure 3-Module system. C, H, and N contents were determined by using an Elementar Vario EL III analyzer. Infrared spectra were recorded from KBr pellets by a Nicolet 510 P FT-IR spectrometer. ^{1}H NMR spectra were recorded with a Bruker Advance 500 MHz spectrometer with TMS as an internal standard. Melting points were determined on a RY-1 melting point apparatus and are uncorrected. Fluorescence measurements were made using a Hitachi F-4500 fluorescence spectrophotometer. Thermogravimetric analysis was recorded on a Mettler TG-50 thermal analyzer under an atmosphere of air at a heating rate of 10°C min^{-1}. Powder X-ray diffraction (XRD) patterns were recorded (Bragg-Brentano) on a Siemens D5005 diffractometer by using Cu-Kα radiation ($\lambda = 1.5418$ Å) with a graphite monochromator. The step size was 0.02° and the count time was 4s.

2.2. *Synthesis*

2.2.1. Synthesis of N,N′-bis(p-methoxyphenyl)-3-oxapentanediamide (L). L was synthesized by the reaction of oxydiacetic acid and *p*-methoxyaniline according to the literature method [10]. Yield: 82%; m.p.: 145–147°C. ^{1}H NMR (500 MHz, CDCl$_3$, δ, ppm): 8.17 (s, 2 H, 2 × NH), 7.48 (d, $J = 8.7$ Hz, 4 H, ArH), 6.88 (d, $J = 8.7$ Hz, 4 H, ArH), 4.25 (s, 4 H, 2 × CH$_2$), 3.80 (s, 6 H, 2 × CH$_3$). IR data (KBr pellet, cm^{-1}): 3431m, 3268m, 2932w, 2854w, 1666s, 1602w, 1556m, 1513s, 1463w, 1417w, 1299w, 1273w, 1236s, 1181w, 1132m, 1031m, 832m, 781w, 726w, 658w, 598w, 525m. Anal. Calcd for $C_{18}H_{20}N_2O_5$: C, 62.78; H, 5.85; N, 8.13. Found: C, 62.85; H, 5.79; N, 8.06.

2.2.2. Synthesis of $[Pr(NO_3)(L)_2(H_2O)_2]\cdot(NO_3)_2$ (1). A solution of $Pr(NO_3)_3 \cdot 6H_2O$ (0.1335 g, 0.25 mmol) in 10 mL ethanol was added dropwise with stirring to a solution of L (0.1722 g, 0.5 mmol) in 10 mL ethyl acetate. The resulting mixture was stirred at room temperature for 8 h. The precipitated solid was filtered, washed with ethanol, and dried *in vacuo* over P_2O_5 for 48 h. Yield: 0.1867 g, 71%. IR data (KBr pellet, cm^{-1}): 3433bs, 3064w, 2961w, 2924w, 1637vs, 1564m, 1513m, 1384vs, 1316m, 1298w, 1129w, 1042w, 965w, 819m, 741w, 506w, 440w. Anal. Calcd for $C_{36}H_{44}N_7O_{21}Pr$: C, 41.11; H, 4.22; N, 9.32. Found: C, 41.20; H, 4.16; N, 9.27. Colorless column crystals suitable for XRD analysis of **1** were obtained by slow evaporation of a DMF-ethanol (1 : 2, v/v) solution of the solid complex for 20 days.

2.2.3. Synthesis of $[Nd(NO_3)(L)_2(H_2O)_2]\cdot(NO_3)_2$ (2). Synthesis of **2** was similar to **1**. Yield: 0.1740 g, 66%. IR data (KBr pellet, cm^{-1}): 3456bs, 3056w, 2964w, 2922w, 2867w, 1635vs, 1564m, 1514m, 1456w, 1383vs, 1317w, 1130m, 1044w, 967w, 819m, 744w, 600w, 506w, 442w. Anal. Calcd for $C_{36}H_{44}N_7O_{21}Nd$: C, 40.98; H, 4.20; N, 9.29. Found: C, 40.89; H, 4.17; N, 9.25. Colorless column crystals suitable for XRD analysis of **2** were obtained by slow evaporation of a DMF-ethanol (1:2, v/v) solution of the solid complex for 30 days.

2.2.4. Synthesis of $[Dy(NO_3)(L)_2(H_2O)_2]\cdot(NO_3)_2$ (3). Synthesis of **3** was similar to **1**. Yield: 0.1582 g, 59%. IR data (KBr pellet, cm^{-1}): 3434bs, 3039w, 2923w, 2871w, 1636vs, 1565m, 1514m, 1454w, 1383vs, 1316w, 1273w, 1132m, 1045w, 968w, 819m, 777w, 744w, 685w, 592w, 561w, 503w, 443w. Anal. Calcd for $C_{36}H_{44}N_7O_{21}Dy$: C, 40.29; H, 4.13; N, 9.14. Found: C, 40.19; H, 4.08; N, 9.20. No crystal suitable for XRD analysis of **3** was obtained using a procedure similar to that reported for **1** and **2**.

2.3. *X-ray crystallography*

Single crystal XRD data of **1** and **2** were collected with a Bruker SMART 1000 CCD diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) using ω scan mode at 293(2) K. Intensity data were corrected for *Lp* factors and empirical absorption. The structure was solved by direct methods and expanded by using Fourier differential techniques with SHELXTL [11]. All non-hydrogen atoms were located with successive difference Fourier syntheses. The structure was refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms. Atomic scattering factors and anomalous dispersion corrections were taken from International tables for X-ray crystallography [12]. A summary of the key crystallographic data and structural refinements for **1** and **2** is presented in table 1. Selected bond distances and angles are illustrated in table S1 in the "Supplementary material".

3. Results and discussion

3.1. *Spectral studies*

The IR spectrum of **L** shows bands at 1666 and 1132 cm^{-1}, which may be assigned to ν(C=O) and ν(C–O–C), respectively. In IR spectra of **1–3**, these bands shift by *ca* 30 and 3 cm^{-1} to lower wavenumbers, respectively; this indicates that all carbonyl and ether oxygens coordinate to the metal and that the Ln–O (carbonyl) bonds are stronger than Ln–O (ether) bonds. Absorption bands at *ca* 3440 and 967 cm^{-1} indicate that coordinated water molecules exist in **1–3**. The strong absorptions at *ca* 1383 cm^{-1} are assigned to uncoordinated nitrate [13]. Compounds **1–3** were too insoluble, even in DMSO, to obtain NMR data.

3.2. *X-ray crystal structures*

Single-crystal XRD analysis reveals that the structure of **1** is composed of $[PrL_2(NO_3)(H_2O)_2]^{2+}$, two NO_3^- counter anions, and two C_2H_5OH molecules linked by hydrogen bonds and *van der Waals*' forces. The molecular structure of **1** with atomic numbering is shown in figure 1. Complex **1** crystallizes in monoclinic space group *C2/c* with one half-molecule in the asymmetric unit. The central Pr(III) is coordinated with 10 oxygens, six from two tridentate L's, two from a bidentate nitrate, and the remaining

Table 1. Crystal data and structure refinement information of **1** and **2**.

Compounds	**1**	**2**
Empirical formula	$C_{40}H_{56}N_7O_{23}Pr$	$C_{40}H_{56}N_7O_{23}Nd$
Formula weight	1143.83	1147.16
Crystal system	Monoclinic	Monoclinic
Space group	*C2/c*	*C2/c*
Temperature (K)	293(2)	293(2)
Unit cell dimensions (Å, °)		
a	13.1676(11)	13.142(2)
b	10.7667(9)	10.750(2)
c	35.176(3)	35.153(8)
α	90	90
β	96.236(1)	96.206(4)
γ	90	90
Volume (Å^3), *Z*	4957.5(7), 4	4937.2(2), 4
Calculated density (g cm^{-3})	1.533	1.543
Absorption coefficient (mm^{-1})	1.071	1.141
Crystal size (mm^3)	0.16 × 0.12 × 0.11	0.23 × 0.14 × 0.08
θ range for data collection (°)	2.33–25.67	2.33–25.51
Reflections collected	13218	12980
Unique reflections	4700 [R(int) = 0.0344]	4582 [R(int) = 0.0606]
Data/parameters	4700/329	4582/329
Goodness-of-fit on F^2	1.107	1.025
R indices (all data)	$R_1 = 0.0516$, $wR_2 = 0.0985$	$R_1 = 0.0723$, $wR_2 = 0.0951$
Final R indices [$I = 2\sigma(I)$]	$R_1 = 0.0432$, $wR_2 = 0.0948$	$R_1 = 0.0495$, $wR_2 = 0.0882$

$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR_2 = [\Sigma w(F_o^2 - F_c^2)/\Sigma w(F_o^2)^2]^{1/2}$.

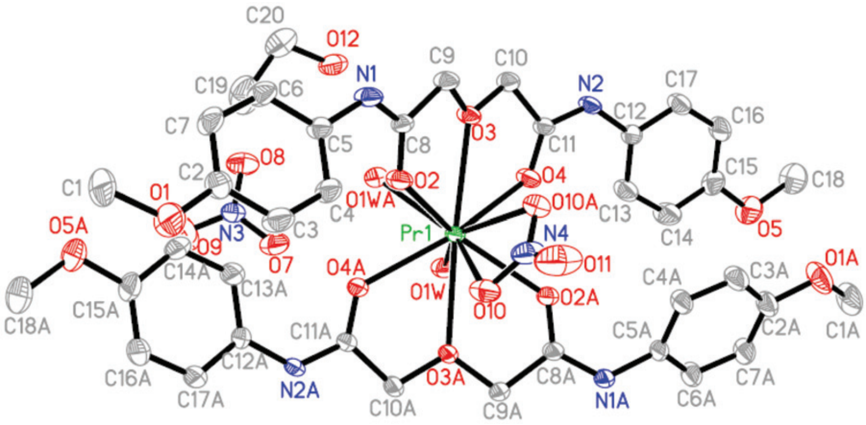

Figure 1. The molecular structure of **1**. Hydrogens are omitted for clarity. Thermal ellipsoids are at 30% probability. Compound **2** has an identical molecular structure and atom numbering scheme with **1**.

two from two water molecules. The coordination geometry around Pr is a distorted bicapped square antiprism (figure 2), with the bicapped positions occupied by O(3) and O(3A). The Pr–O lengths are in the ranges 2.476(3)–2.664(3) Å and comparable to those reported for other lanthanide oxygen-donor complexes [14–17]. The shorter distance of Pr–O$_{(C=O)}$ [Pr(1)–O(2) = 2.503(3), Pr(1)–O(4) = 2.476(3) Å] than Pr–O$_{(C\text{-}O\text{-}C)}$ [Pr(1)–O(3) = 2.664(3) Å] suggests stronger complexing ability of the carbonyl oxygen to Pr

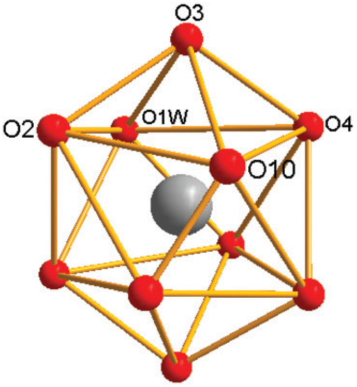

Figure 2. The coordination polyhedron of Pr in **1**.

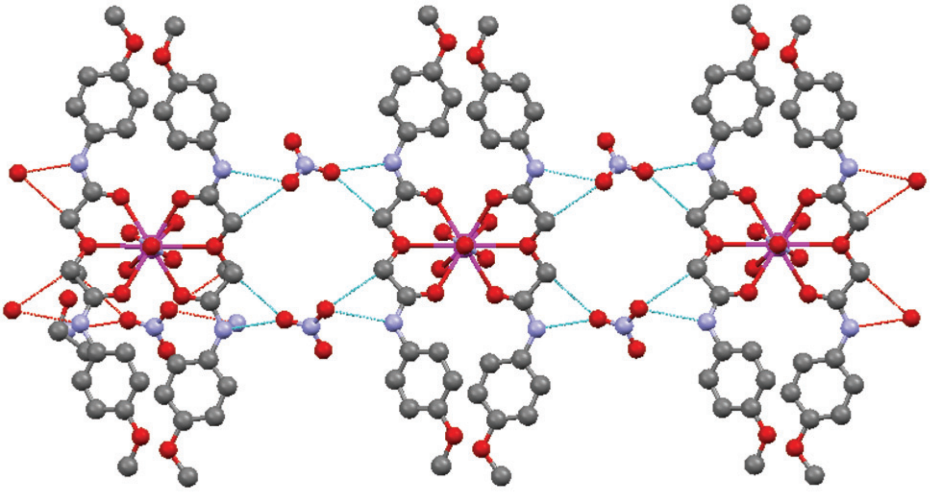

Figure 3. The 1-D infinite chain structure of **1**, formed by hydrogen bonds of uncoordinated nitrates, and viewed down the *b* axis.

than ether oxygen. In **1**, L is a tridentate forming two stable five-membered rings; the two five-membered rings are essentially planar, with a dihedral angle of 9.96(11)°. The dihedral angle between two benzene rings is 41.0(2)°. One nitrate coordinates bidentate with Pr and the other two nitrates were not coordinated and balance the charge.

There exist different types of hydrogen bonds in **1**. Uncoordinated nitrates as hydrogen bond acceptors form intermolecular hydrogen bonds, N(1)–H(1) ⋯ O(7), C(9)–H(9A) ⋯ O(7), N(2)–H(2) ⋯ O(8), and C(10)–H(10A) ⋯ O(8), connecting the complex into 1-D infinite chains (figure 3 and table 2). Ethanols as hydrogen bond donors and acceptors form intermolecular O(12)–H(12) ⋯ O(10) and O(1W)–H(1WB) ⋯ O(12) hydrogen bonds with coordinated nitrate and water (figure 4 and table 2). Finally, there exist different types of C–H ⋯ O hydrogen bonds in **1**; hydrogen

Table 2. Hydrogen bond data (Å) and (°) for **1** and **2**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠DHA
Complex **1**				
N(1)–H(1)···O(7)[a]	0.86	2.09	2.926(4)	164
N(2)–H(2)···O(8)[b]	0.86	2.03	2.808(5)	150
O(1W)–H(1WA)···O(7)[c]	0.85(4)	2.05(4)	2.870(4)	165(4)
O(1W)–H(1WB)···O(12)[c]	0.85(4)	1.93(3)	2.767(4)	166(3)
O(12)–H(12)···O(10)[a]	0.82	2.01	2.813(4)	165
C(4)–H(4)···O(2)	0.93	2.57	2.975(5)	107
C(6)–H(6)···O(9)[a]	0.93	2.58	3.411(7)	150
C(9)–H(9A)···O(7)[a]	0.97	2.69	3.125(5)	108
C(10)–H(10A)···O(8)[c]	0.97	2.33	3.023(6)	128
C(13)–H(13)···O(4)	0.93	2.33	2.909(5)	120
C(19)–H(19C)···O(8)	0.96	2.58	3.489(9)	157
C(20)–H(20A)···O(9)[a]	0.97	2.56	3.521(7)	170
Complex **2**				
N(1)–H(1)···O(7)	0.86	2.10	2.938(6)	164
N(2)–H(2)···O(8)[c]	0.86	2.04	2.815(7)	150
O(1W)–H(1WA)···O(7)[b]	0.85(3)	2.07(4)	2.870(5)	157(4)
O(1W)–H(1WB)···O(12)[d]	0.85(3)	1.93(3)	2.762(5)	166(3)
O(12)–H(12)···O(10)	0.82	1.99	2.807(5)	172
C(4)–H(4)···O(9)	0.93	2.58	3.410(7)	149
C(9)–H(9B)···O(7)	0.97	2.69	3.121(6)	108
C(10)–H(10B)···O(8)[b]	0.97	2.32	3.020(7)	128
C(17)–H(17)···O(4)	0.93	2.33	2.909(6)	120
C(6)–H(6)···O(2)	0.93	2.56	2.971(6)	107
C(19)–H(19A)···O(8)[e]	0.96	2.59	3.499(9)	158
C(20)–H(20B)···O(9)[f]	0.97	2.57	3.532(8)	170

Symmetry codes: [a]$-1/2+x, -1/2+y, z$; [b]$1/2-x, -1/2+y, 1/2-z$; [c]$1-x, y, 1/2-z$; [d]$-1/2-x, -1/2+y, 1/2-z$; [e]$-1+x, y, z$; [f]$-1/2+x, 1/2+y, z$.

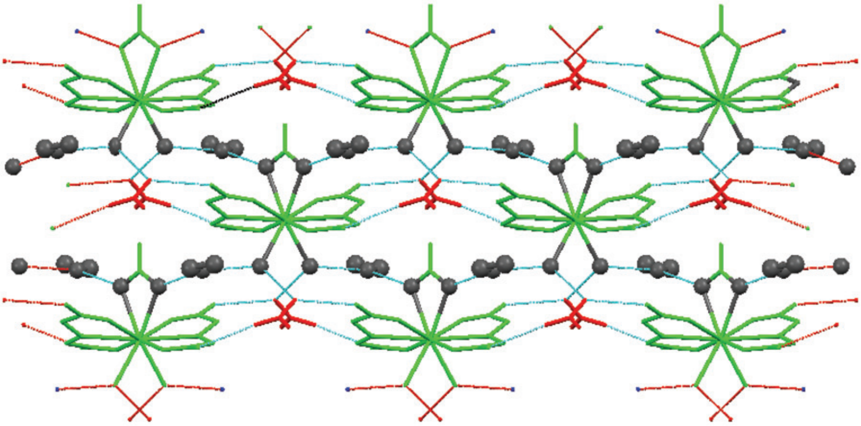

Figure 4. The hydrogen bonds among C_2H_5OH and coordinated nitrates and water molecules in **1**, viewed down the *c* axis. The involved hydrogen bonds are shown in ball and stick style, and the hydrogens and methoxyphenyl groups are omitted for clarity.

bond data are shown in table 2. The crystal packing exhibits weak π–π stacking interactions with mean distance of 3.6316 Å. The angle of the two phenyl rings is 15.5°. These hydrogen bonds and weak π–π stacking interactions connected the complex into a 3-D supramolecular structure (figure 5).

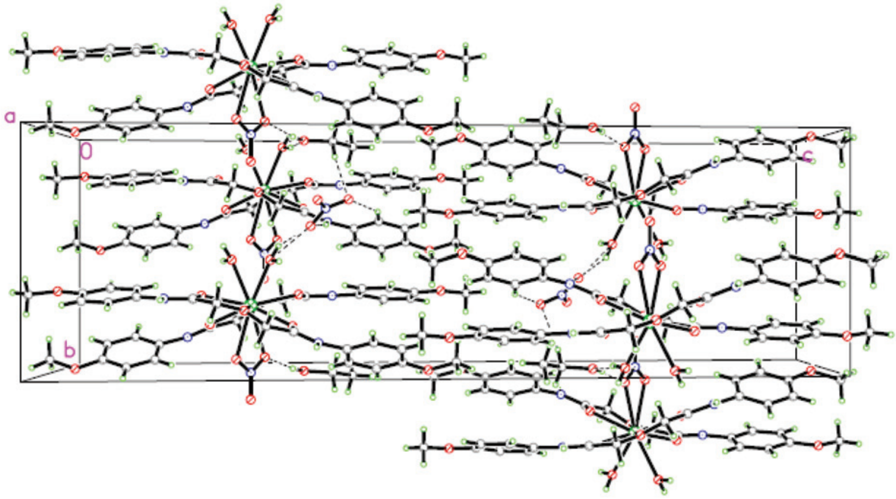

Figure 5. The 3-D supramolecular structure of **1**, viewed down the *a* axis. The dashed lines show hydrogen bonds.

The structure of **2** is identical to that of **1**, including coordination geometry around lanthanide, the coordination mode of ligand, and the connecting modes of the hydrogen bonds. All Nd–O lengths (average value 2.541 Å) are shorter than the corresponding Pr–O ones (average value 2.555 Å, see table 2) with the decrease in the lanthanide ion radius, in agreement with the lanthanide contraction.

3.3. *Thermogravimetric analyses*

Thermogravimetric analyses (TGA) for **1–3** were carried out from room temperature to 900°C with a heating rate of 10°C $\min^{-1}$ (Supplementary material). Complex **1** shows four weight loss steps. Weight loss of 2.43% from 50°C to 140°C is attributed to the loss of NO of coordinated nitrate (Calcd 2.85%). The weight loss of 3.34% between 180°C and 250°C corresponds to the loss of two coordinated water molecules (Calcd 3.42%). The weight loss of 77.21% from 250°C to 600°C is attributed to the loss of two ligands and remaining nitrate (Calcd 77.50%). The residual percentage weight (observed 17.02%) at the end of the decomposition is consistent with Pr_6O_{11} (Calcd 16.19%).

Complex **2** loses 5.93% from 50°C to 150°C, corresponding to the removal of one coordinated nitrate (calcd. 5.88). The weight loss of 3.39% between 160°C and 250°C corresponds to the loss of two coordinated water molecules (Calcd 3.41%). The weight loss of 73.66% from 250°C to 600°C is attributed to the loss of ligands and two uncoordinated nitrates (Calcd 74.72%). The residue (observed 17.22%) is Nd_2O_3 (Calcd 16.00%).

Complex **3** loses 2.90% from 50°C to 100°C, corresponding to the removal of NO of coordinated nitrate (Calcd 2.80%) and the weight loss of 3.31% between 100°C and 250°C corresponds to the loss of two coordinated water molecules (Calcd 3.36%). The weight loss of 74.04% from 250°C to 750°C is attributed to the loss of two ligands and

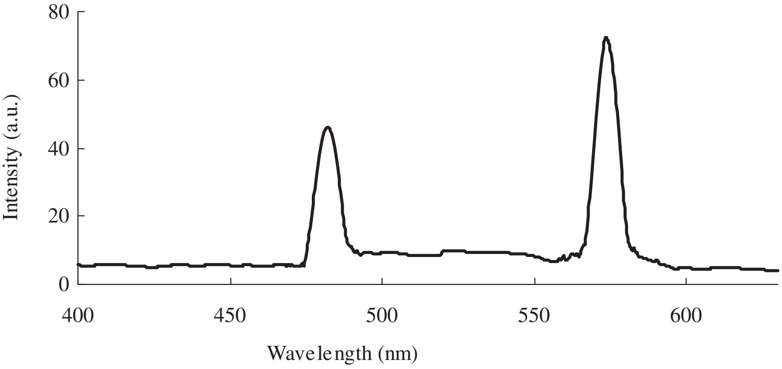

Figure 6. Fluorescence spectra of **3** in solid state at room temperature.

remaining nitrate (Calcd 76.42%). The residue (observed 19.75%) is Dy_2O_3 (Calcd 17.38%).

3.4. *Powder XRD*

Experimental powder XRD patterns of **1** and **2** are in good agreement with the simulated ones on the basis of the single-crystal structures (Supplementary material), suggesting the phase purity of the as-synthesized samples. The experimental powder XRD pattern of compound **3** is very similar to **1** and **2**, suggesting that **3** has the same structure as **1** and **2**.

3.5. *Luminescence of 3*

The solid state photoluminescence spectrum of **3** was recorded under excitation at 310 nm at room temperature (figure 6). Two emitting peaks at 482 and 574 nm are assigned to the transitions $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ of Dy(III), respectively [18, 19]. The relatively strong emission intensity of **3** is probably because the ligand absorbed the excitation energy and transferred the energy to the Dy(III), showing the characteristic emissions of Dy(III) [20–22].

4. Conclusion

The amide-type acyclic polyether *N*,*N*′-bis(*p*-methoxyphenyl)-3-oxapentanediamide and its three lanthanide coordination compounds have been prepared and structurally characterized. Pr and Nd complexes have the same structure; the coordination geometry around the metal is a distorted bicapped square antiprism. Extensive intermolecular hydrogen bonds in Pr and Nd compounds result in 3-D supramolecular networks. The fluorescence suggests that the Dy complex could be a potential fluorescent material.

Supplementary material

Crystallographic data for the structural analyses of **1** and **2** have been deposited with the Cambridge Crystallographic Data Center (CCDC). CCDC reference numbers 808365 and 808366 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 20971076) and the Science and Technology Projects (No. BS2010NJ004, 2009ZRB019KH) of Shandong Province, China.

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