

## 基于席夫碱配体的 Gd(III)/Dy(III)配合物的结构和磁性

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**摘要:** 以席夫碱 2-羟基-3-甲氧基-5-溴苯甲醛肟(HL)为配体, 合成了 2 例未见文献报道的席夫碱-稀土双核配合物  $[\text{Gd}_2(\text{L})_4(\text{HCOO})_2(\text{CH}_3\text{OH})_2]$  (**1**)和 $[\text{Dy}_2(\text{L})_4(\text{CH}_3\text{COO})_2(\text{CH}_3\text{OH})_2]$  (**2**)。X 射线单晶衍射分析表明配合物 **1** 和 **2** 中镧系金属均为对称的九配位双核结构。对配合物的磁性测试结果表明配合物 **1** 中双核 Gd(III)离子之间存在着弱的反铁磁相互作用, 交流磁化率测试结果表明, 配合物 **2** 显示出频率依赖。

**关键词:** 席夫碱; 双核; 稀土; 晶体结构; 磁性

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## Dinuclear Gd(III)/Dy(III) Complexes Based on Schiff base Ligands: Structures and Magnetic Properties

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**Abstract:** Two novel dinuclear Ln(III) complexes  $[\text{Gd}_2(\text{L})_4(\text{HCOO})_2(\text{CH}_3\text{OH})_2]$  (**1**) and  $[\text{Dy}_2(\text{L})_4(\text{CH}_3\text{COO})_2(\text{CH}_3\text{OH})_2]$  (**2**) (HL=2-hydroxy-3-methoxy-5-bromobenzaldehyde oxime) have been successfully synthesized and characterized by single crystal X-ray diffraction, IR spectroscopy and elemental analyses. Single crystal X-ray crystallographic analyses reveal that these two complexes have similar structures, in which two symmetric nine-coordinate Ln(III) ions are bridged by two phenoxide groups from two Schiff base ligands, two formate ions for **1** and two acetate ions for **2** respectively. Magnetic studies reveal that weak antiferromagnetic interaction between Gd(III) ions existing in complex **1**, while complex **2** shows slow relaxation of magnetization. CCDC: 1570377, **1**; 1570378, **2**.

**Keywords:** Schiff base; dinuclear; lanthanide; crystal structure; magnetic property

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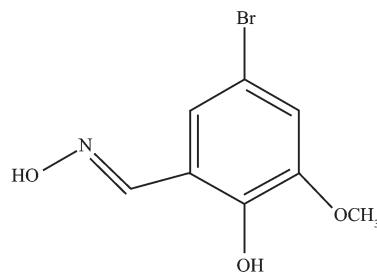
## 0 Introduction

Since the discovery of Single-molecule magnets (SMMs) during the 1990s, the study of SMMs has a booming development for its potential application in high-density information storage and quantum computing devices<sup>[1-4]</sup>. To achieve these applications, carefully designed structures of SMMs are required. Among all the central ions, owing to their significant magnetic anisotropy which arising from the large, unquenched orbital angular momentum, lanthanide-ions are widely used to construct SMMs<sup>[5-15]</sup>. For example, Zheng's group reported a monometallic Dy complex of magnetization reached an effective energy barrier to magnetic relaxation of  $U_{\text{eff}}=1\,815(1)\text{ K}$ <sup>[16]</sup>. Tong's group reported another Dy-based compound with a record anisotropy barrier up to  $1\,837\text{ K}$  in zero field and a record magnetic blocking temperature of  $60\text{ K}$ <sup>[17]</sup>. Notably, previous studies show that the magnetic interaction between centre metals and the alteration of coordination geometry on them are two important factors to adjust the relaxation dynamics of lanthanide-based SMMs<sup>[18-19]</sup>. For example, Tang's group reported an asymmetric dinuclear Dy(III) complex which shows the high axiality and Ising exchange interaction that can efficiently suppress quantum tunneling of magnetization and the exchange interaction between the Dy sites contribution leading to an increase of the relaxation time by 3 orders of magnitude<sup>[20]</sup>. In addition, the dinuclear SMMs are ideal configurations to study the single-ion effective anisotropic barriers versus the energy barriers which arising from the two interaction metal centers<sup>[21-22]</sup>. To date, various researches have been performed to study how the exchange coupling affects magnetic properties of lanthanide-based complexes and some innovative results have been reported<sup>[23-25]</sup>.

To construct dinuclear SMMs, the choice of ligands is very important. As ligands can influence the construction of coordination polymers and moreover, ligand field is a key factor to control the magnetic anisotropy of SMMs. Therefore, Schiff-base ligands with rich O,N-based multichelating sites have been regarded as excellent candidates to construct

lanthanide-based multinuclear coordination compounds. In addition, by modifying the terminal group of acylhydrazine derivatives, various ligand systems will be built in which ligand field will also affect the SMM properties of Ln(III) coordination compounds<sup>[26-28]</sup>.

Herein, we choose 2-hydroxy-3-methoxy-5-bromobenzaldehyde oxime (HL) as ligand in combination with methane acid and acetic acid to build new Ln-based coordination polymers. Two new complexes, namely,  $[\text{Gd}_2(\text{L})_4(\text{HCOO})_2(\text{CH}_3\text{OH})_2]$  (**1**) and  $[\text{Dy}_2(\text{L})_4(\text{CH}_3\text{COO})_2(\text{CH}_3\text{OH})_2]$  (**2**) have been constructed successfully. Their syntheses, crystal structures and magnetic properties are reported.



Scheme 1 Molecular structure of HL

## 1 Experimental

### 1.1 Materials and measurements

All reagents and solvents were purchased from Aladdin and used without further purification. The ligand HL was synthesized according to the literature<sup>[29]</sup>. Elemental analyses for C, H and N were performed on a Perkin-Elmer elemental analyzer model 240. IR spectra were recorded on a Nicolet IS10. IR spectrometer using KBr pellets in the range of  $4\,000\sim500\text{ cm}^{-1}$ . Variable temperature magnetic susceptibilities were measured on SQUID MPMSXL-7 magnetometer in the temperature range of  $2\sim300\text{ K}$ .

### 1.2 Synthesis of complex 1

Ligand HL (0.1 mmol) was dissolved in mixed solvent of  $10\text{ mL CH}_3\text{OH}$  and  $5\text{ mL CH}_3\text{CN}$  followed by addition of  $\text{GdCl}_3\cdot6\text{H}_2\text{O}$  (0.1 mmol) and sodium methoxide (0.2 mmol), which gave a clear solution after stirring for 2.5 h. Diethyl ether was diffused slowly into the solution at room temperature, and light-yellow single crystals were obtained in 5 days. Yield: 42.5% based on rare-earth. Elemental analysis

calculated for  $C_{40}H_{54}Br_4Gd_2N_4O_{22}(\%)$ : C 30.47; H 3.45; N 3.55. Found (%): C 30.61; H 3.52; N 3.61. FT-IR (KBr,  $cm^{-1}$ ): 1 689(w), 1 612(m), 1 568(m), 1 473(s), 1 312(w), 1 174(w), 1 072(m), 1 010(w), 952(w), 842(s), 753(m), 587(m).

### 1.3 Synthesis of complex 2

Ligand HL (0.1 mmol) was dissolved in mixed solvent of 10 mL  $CH_3OH$  and 5 mL  $CH_3CN$  followed by the addition of  $Dy(CH_3COO)_3 \cdot 4H_2O$  (0.1 mmol) and triethylamine (0.2 mmol). A clear solution was obtained after stirring for 3 h. Diethyl ether was used to diffuse slowly into this solution at room temperature, and light-yellow single crystals were obtained in 5 days. Yield: 44.5% based on rare-earth. Elemental analysis calculated for  $C_{40}H_{50}Br_4Dy_2N_4O_{20}(\%)$ : C 30.97; H 3.25; N 3.61. Found(%): C 30.81; H 3.21; N 3.51. FTIR (KBr,  $cm^{-1}$ ): 1 860(w), 1 601(m), 1 478(m), 1 380(s), 1 221(m), 1 110(s), 1 005(w), 941(w), 840(m), 734(s), 558(w).

### 1.4 X-ray crystallographic study

The suitable single crystals of complexes **1** and

**2**, with crystal size of 0.34 mm×0.34 mm×0.29 mm and 0.32 mm×0.31 mm×0.28 mm respectively, were used for X-ray diffraction data collection with a BRUKER SMART 1000 CCD, all using graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda=0.071\ 073\ nm$ ). The structures were solved primarily by direct methods and refined by the full-matrix least squares method. The computations were performed with the SHELX-97 program<sup>[30]</sup>. All non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were set in calculated positions and refined as riding atoms with a common fixed isotropic thermal parameter. The crystal data and refinement parameters of complexes **1** and **2** are summarized in Table 1.

CCDC: 1570377, **1**; 1570378, **2**.

## 2 Results and discussion

### 2.1 Crystal structures of complexes 1 and 2

The crystal structure of complex **1** is depicted in

Table 1 Crystal data and structure refinement for complexes **1** and **2**

Complex	<b>1</b>	<b>2</b>
Empirical formula	$C_{40}H_{54}Br_4Gd_2N_4O_{22}$	$C_{40}H_{50}Br_4Dy_2N_4O_{20}$
Formula weight	1 577.01	1 551.44
$T / K$	173(2)	173(2)
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/c$
$a / nm$	1.093 9(8)	1.188 5(6)
$b / nm$	1.128 8(9)	2.513 6(12)
$c / nm$	1.310 5(9)	0.835 8(5)
$\alpha / (^\circ)$	103.689(13)	
$\beta / (^\circ)$	100.567(15)	95.923(7)
$\gamma / (^\circ)$	116.970(12)	
$V / nm^3$	1.319 9(16)	2.484(2)
$Z$	1	2
$D_c / (g \cdot cm^{-3})$	1.984	2.075
$\mu / mm^{-1}$	5.598	6.283
$R_{int}$	0.033 2	0.056 1
$F(000)$	766	1 500
Reflection collected	9 747	17 798
GOF on $F^2$	1.016	1.015
$R_1^a [I > 2\sigma(I)]$	0.038 7	0.042 7
$wR_2^b [I > 2\sigma(I)]$	0.100 7	0.097 8

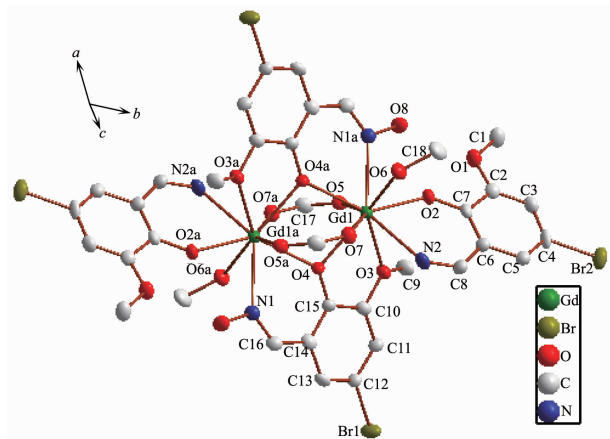
<sup>a</sup>  $R_1 = \sum (|F_o| - |F_c|) / \sum |F_o|$ ; <sup>b</sup>  $wR_2 = \{ \sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2 \}^{1/2}$ .

**Table 2** Selected bond lengths (nm) and angles (°) for complexes **1** and **2**

<b>1</b>					
Gd(1)-O(2)	0.228 6(4)	Gd(1)-O(7)a	0.244 0(4)	Gd(1)-O(3)	0.263 7(4)
Gd(1)-O(4)a	0.237 2(4)	Gd(1)-O(6)	0.245 3(4)	Gd(1)-Gd(1)a	0.376 7(3)
Gd(1)-O(5)	0.240 7(4)	Gd(1)-N(2)	0.259 2(6)	O(3)-C(10)	0.138 0(7)
Gd(1)-O(4)	0.241 8(4)	Gd(1)-N(1)a	0.259 3(6)	O(3)-C(9)	0.142 9(7)
O(2)-Gd(1)-O(4)a	139.32(1)	O(2)-Gd(1)-O(4)	135.76(1)	O(2)-Gd(1)-O(7)a	130.99(2)
O(2)-Gd(1)-O(5)	85.39(1)	O(4)a-Gd(1)-O(4)	76.31(1)	O(4)a-Gd(1)-O(7)a	76.04(2)
O(4)a-Gd(1)-O(5)	77.67(1)	O(5)-Gd(1)-O(4)	77.91(1)	O(5)-Gd(1)-O(7)a	143.18(2)
<b>2</b>					
Dy(1)-O(6)	0.229 0(4)	Dy(1)-O(4)	0.242 3(4)	Dy(1)-O(5)	0.265 0(4)
Dy(1)-O(4)a	0.236 8(4)	Dy(1)-O(7)	0.246 6(4)	Dy(1)-Dy(1)a	0.375 7(4)
Dy(1)-O(3)	0.237 9(4)	Dy(1)-N(1)a	0.257 8(5)	O(3)-C(10)	0.124 5(8)
Dy(1)-O(2)a	0.241 2(4)	Dy(1)-N(2)	0.262 9(6)	O(7)-C(18)	0.141 8(8)
O(6)-Dy(1)-O(4)a	140.38(1)	O(6)-Dy(1)-O(2)a	130.53(1)	O(6)-Dy(1)-O(4)	134.74(2)
O(6)-Dy(1)-O(3)	86.24(1)	O(4)a-Dy(1)-O(2)a	76.99(1)	O(4)a-Dy(1)-O(4)	76.70(2)
O(4)a-Dy(1)-O(3)	79.21(1)	O(3)-Dy(1)-O(2)a	141.49(1)	O(3)-Dy(1)-O(4)	75.67(2)

Symmetry codes: a:  $-x, -y, -z$  for **1**; a:  $-x+1, -y, -z+2$  for **2**.

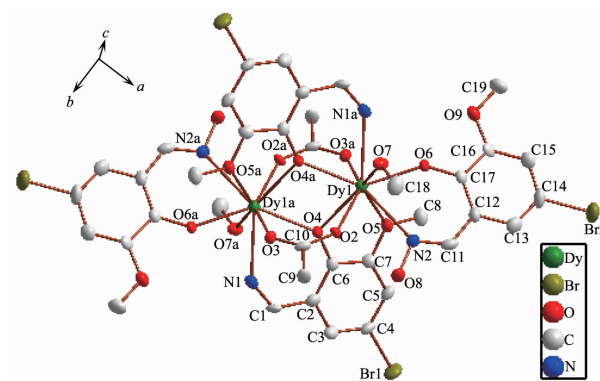
Fig.1. Complex **1** crystallizes in the space group of  $P\bar{1}$  in triclinic structure. Two symmetric nine-coordinated Gd(III) ions are bridged by two phenoxide groups from two Schiff base ligands and two molecules of formate ion with Gd-O<sub>phenoxide</sub> bond length of 0.241 7(5) and 0.237 4(3) nm, respectively. Then, each Gd(III) ion is further coordinated with two N ions, one O<sub>hydroxyl</sub> ion and one O<sub>methoxyl</sub> ion. The intramolecular Gd...Gd distance is 0.376 6(2) nm, as well as Gd-O-Gd angle of 103.65(3)°.



All hydrogen atoms are omitted for clarity; Symmetry codes: a:  $-x, -y, -z$

Fig.1 Crystal structure of complex **1** with thermal ellipsoids drawn at 30% probability

Complex **2** crystallize in monoclinic with space group of  $P2_1/c$  (Fig.2). The crystal structure of complex **2** is similar to complex **1** except that Dy(III) ions are bridged by two acetate ions instead of formate ions. The Dy-O and Dy-N distances are in the range of 0.228 9(4)~0.265 0(4) nm and 0.257 7(3)~0.262 9(2) nm, respectively. The intramolecular Dy...Dy distance is 0.375 7(1) nm with Dy-O-Dy angle of 103.30(5)°. Selected bond lengths and angles for two complexes are shown in Table 2.



All hydrogen atoms are omitted for clarity; Symmetry codes: a:  $-x+1, -y, -z+2$

Fig.2 Crystal structure of complex **2** with thermal ellipsoids drawn at 30% probability

## 2.2 Magnetic properties of complex 1

Variable-temperature magnetic susceptibilities of complexes **1** and **2** were measured from 300 to 2.0 K in an applied field of 1 kOe. As showed in Fig.3, the  $\chi_M T$  value for complex **1** at 300 K is  $15.67 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$ , which is in agreement with the expected value of  $15.76 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$  for two uncoupled Gd(III) ions ( $^8S_{7/2}$ ,  $S=7/2$ ). The  $\chi_M T$  value is almost constant until the temperature cooling to 30 K, after that, this value decreases rapidly, reaching a minimum value of  $11.04 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$  at 2.0 K. This phenomenon indicates the presence of a weak antiferromagnetic interaction between the Gd(III) ions.

To evaluate the coupling parameters, the magnetic data were analyzed by a theoretical expression<sup>[31]</sup>. The best fit from equation below gives  $g=1.99$ ,  $J=-0.1348$

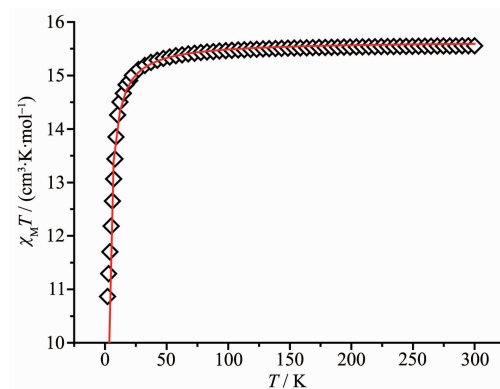
$$\chi_M = \frac{2Ng^2\beta^2}{kT} \cdot \frac{\exp(y) + 5\exp(3y) + 14\exp(6y) + 30\exp(10y) + 55\exp(15y) + 91\exp(21y) + 140\exp(28y)}{\exp(y) + 5\exp(3y) + 7\exp(6y) + 9\exp(10y) + 11\exp(15y) + 13\exp(21y) + 15\exp(28y)}$$

$$y = J/(kT)$$

## 2.3 Magnetic properties of complex 2

For complex **2** (Fig.4), at room temperature, the  $\chi_M T$  value ( $28.61 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$ ) is close to the theoretical value of  $28.34 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$  for two non-interacting Dy(III) ions ( $^6H_{15/2}$ ,  $g=4/3$ ). Upon cooling, the  $\chi_M T$  value decreases gradually in the temperature range of 300~50 K, then drops quickly and reaches  $6.8 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$  at 2 K. This observed decrease of  $\chi_M T$  value under high temperatures results from the depopulation of the excited Stark sublevels. However, the decrease behavior at low temperature mainly results from intramolecular antiferromagnetic interaction between Dy(III) ions<sup>[32]</sup>.

To investigate the dynamic magnetic properties of complex **2**, the temperature frequency dependencies of



Solid line represents the theoretical values based on the corresponding equation

Fig.3 Temperature dependence of  $\chi_M T$  for complex **1**

which confirms the weak antiferromagnetic interaction between the Gd ions.

magnetic susceptibility measurements under alternating current were measured in the range of 200~1 400 Hz. As shown in Fig.5, no maximum in-phase signal

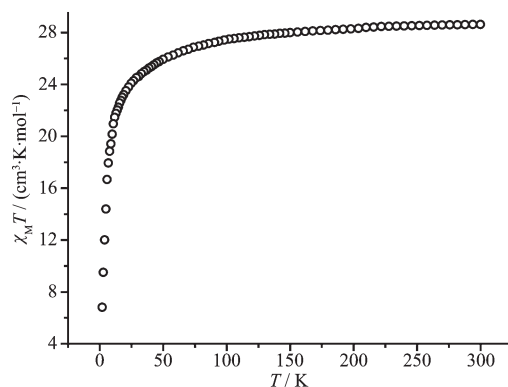


Fig.4 Temperature dependence of  $\chi_M T$  for complex **2**

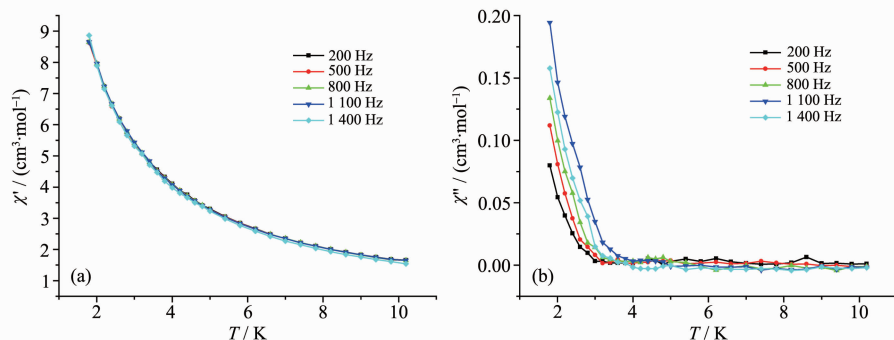


Fig.5 In-phase (a) and out-of-phase susceptibilities (b) of complex **2** at different frequencies under zero applied field

( $\chi'$ ) and out-of-phase signal ( $\chi''$ ) values are observed until 2 K which might arise from the quantum tunneling of magnetization (QTM). However, the values of in-phase signal ( $\chi'$ ) and out-of-phase signal ( $\chi''$ ) exhibit a frequency dependent character, indicating the slow relaxation of magnetization.

### 3 Conclusions

Two novel dinuclear Ln(III) based complexes have been successfully synthesized. These two complexes have similar structures, in which two symmetric nine-coordinated Ln(III) ions are bridged by two phenoxide groups from two Schiff base ligands, two molecules of formate ion for **1** and acetate ion for **2**. The magnetic studies show that weak antiferromagnetic interaction between the Gd ions existing in complex **1**, while complex **2** shows slow relaxation of magnetization.

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