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Single-Molecule Magnetism in Dy₂ Cluster Based on a **Schiff Base Ligand**

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Ln^{III}-based Abstract. Two dinuclear clusters, namely $[Dy_2L_2(NO_3)_2(DME)_4]$ (1) and $[Gd_2L_2(NO_3)_2(DME)_4]$ (2) $[H_2L = (E)_2$ 2-((2-hydroxybenzylidene)amino)phenol] were obtained under hydrothermal condition. Two Ln^{III} ions are bridged by two phenolic hydroxyl oxygen atoms, and the distances of them are 3.829 Å (Dy1-

Introduction

Generally speaking, the single molecule magnet is composed of independent single molecules, which can maintain the magnetization intensity for a long time at low temperature and without external magnetic field. Its appearance makes it possible to develop memory devices with nanometer size magnetic complexes as basic units.^[1,2] In recent years, there is a growing interest in constructing novel SMMs with the paramagnetic lanthanide ions (especially Dy^{III}, Ho^{III}, Er^{III}, and Tb^{III}). Because the large anisotropy barrier is crucial for SMM, lanthanide ions with their advantages of large ground-state spin and significant intrinsic anisotropy can meet this demand.^[3] Many lanthanide SMMs with varying ranges of nuclearities have been prepared.^[4–10] Among all the lanthanide-based SMMs, [Ln2]SMMs have attracted significant attention of the chemists and physicists in the world.^[11-40] There are many reasons for this, for example, (i) compared with other polynuclear clusters, the synthesis of $[Ln_2]$ is easier to control; (ii) the structureproperty relationships of the dinuclear systems could be easily investigated by modifying the functional groups of the ligands; (iii) in studying the magnetic interaction between two lanthanide atoms, they represent the simplest structural units; and (iv) it is relatively convenient to determine the possible orientation of anisotropy in the dinuclear system. We believe that Ln₂ is the most basic structure in explaining the nature of single molecule magnets (such as the relationship between single ion relaxation and molecular entity relaxation; the influence of magnetic interaction on magnetization relaxation, the possible orientation of anisotropy).

Dy1A) and 3.860 Å (Gd1-Gd1A). Two Dy1-O-Dy1A and Gd1-O-Gd1A angles are 109.4° and 109.8°, respectively. Magnetic studies reveal a weak antiferromagnetic interaction between Gd ions in complex 2, and single-molecule magnet behavior for 1 with $U_{\text{eff}} = 49.9 \text{ K}$ and $\tau_0 = 1.54 \times 10^{-6}$ s.

In this work, we have used O-hydroxybenzaldehyde Schiffbase (E)-2-((2-hydroxy- benzylidene)amino)phenol (H₂L) in combination with N,N-dimethylacetamide (DME) for the synthesis of two dinuclear complexes, and the Dy2 complex exhibited SMM behavior. Herein, we present the syntheses, crystal structures and magnetic properties of these two complexes.

Results and Discussion

Description of the Structures 1 and 2

The molecular structures of 1 are depicted in Figure 1. Single crystal X-ray diffraction reveals that complexes 1 and 2 are isomorphous, herein only the structure of representative complex 1 will be described in detail. Complex 1 is in the monoclinic space group $P2_1/n$. The asymmetric unit consists of one Dy^{III} ion, one L²⁻ ligand one NO₃⁻ and two DME ligands. The Dy1 atom is coordinated by three phenolate oxygen atoms from two L²⁻ ligands and one nitrogen atoms from one L²⁻ ligand, two oxygen atoms from two DME ligands, and two oxygen atoms from NO3-. The Dy1 and Dy1A are bridged by two µ-phenolate oxygen atoms(O2), and the distance of them is 3.829 Å. Two Dy1–O–Dy1A angles are both 109.4(1)° (Figure 1a). The L^{2-} anions is in $\mu^2{:}\eta^2{:}\eta^1{:}\eta^1$ mode as shown in Figure 1. The eight-coordinated DyIII center has an O7N sphere and displays a distorted triangular dodecahedron arrangement. The bond lengths of Dy1-O1, Dy1-O2, Dy1-O2A, Dy1-O3, Dy1-O4, Dy1-O6 and Dy1-O7 are 2.197(2), 2.367(2), 2.323(2), 2.599(2), 2.443(2), 2.377(2) and 2.316(2) Å, respectively. The length of Dy-N bond is found to be 2.495(2) Å.

The program SHAPE 2.1 was used to analyze the exact geometry of the octa-coordinated dysprosium ion,^[43] and close analysis of the selected resulting data are listed in Table S1 (Supporting Information). The triangular dodecahedron (D_{2d}) geometry for 1 with a minimum CshM value of 5.49 was determined, which indicates the coordination environment of Dy1 ion is close to a distorted triangular dodecahedron configuration.

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Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/zaac.202000089 or from the author.

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Figure 1. (a) The molecular structure of 1. (b) Coordination polyhedron of the Dy^{III} ion in 1.

Although complex 1 joins a big family of Dy_2 clusters,^[11–40] none of the complexes crystallized from the DME solution, and the DME oxygen atom also serves as a coordination site in 1. These features indicate the improvement of the synthesis method and the structural novelty of complexes 1 and 2.

Magnetic Studies

Direct current (dc) magnetic properties were investigated on polycrystalline samples for clusters **1** and **2** in the 1.8–300 K range under an applied magnetic field of 1000 Oe. As shown in Figure 2, at room temperature, the $\chi_M T$ value of **1** is 25.97 cm³ mol⁻¹ K, which is slightly lower than the theoretical value of 28.34 cm³·mol⁻¹·K for non-interacting spins of two Dy^{III} ions (⁶ $H_{15/2}$, S = 5/2, L = 5, g = 4/3). Upon cooling, the value of $\chi_M T$ decreases gradually and then more sharply reaches a maximum value of 6.82 cm³ mol⁻¹ K at 1.8 K. This magnetic behavior indicates that weak antiferromagnetic exchange interaction may exist. For complex **2**, the $\chi_M T$ value is 16.12 cm³·mol⁻¹·K at room temperature, which is closed to the value expected for two magnetically isolated Gd^{III} (⁸ $S_{7/2}$, S =7/2, L = 0, g = 2) of 15.75 cm³·mol⁻¹·K. With the decrease of temperature, the $\chi_M T$ values decrease gradually and then rapidly below 10 K. This phenomenon indicates weak antiferromagnetic interactions between two Gd^{III} ions.



Figure 2. Temperature dependence of the $\chi_M T$ product in the range of 1.8–300 K at 1.0 kOe for **1** and **2**. The solid red line of **2** correspond to the best fits in the range of 1.8–300 K.

In order to obtain more insight into the magnetic coupling between the Gd^{III} ions, we fitted the magnetic data of **2** with the isotropic spin Hamiltonian $H = -2JS_{Gd1}S_{Gd1}A$. The magnetic data in the temperature range of 1.8–300 K can be well fitted and the best-fitting parameters for **2** are J = -0.043 cm⁻¹, g = 2.03. The negative J value confirms the weak antiferromagnetic interactions between two Gd^{III} centers in **2**.

The field-dependent magnetization measurements performed for **1** and **2** at fields of 0–70 kOe under 2 K are shown in Figure 3. For **1**, the magnetization increases very rapidly at low magnetic fields, then a slow linear increase in high-field region without saturation even at 70 kOe. The magnetization value at 70 kOe is 10.50 N β , which is lower than the theoretical saturation value of 20.00 N β . This behavior may be caused by the significant magnetic anisotropy and/or possibly lowlying excited states in all systems.^[44–46] For **2**, the magnetization value at 70 kOe is 13.79 N β , which is close to the expected theoretical value of 14.00 N β for two Gd^{III} ions.



Figure 3. Field dependence of the magnetization of 1 and 2 obtained at 2 K.



The dynamic magnetic properties of 1 were probed to further understand its magnetism. The temperature dependence of alternating current (ac) susceptibilities for 1 was performed in the temperature range of 22–2 K at the indicated frequencies of 1, 10, 32, 100, 320, 666, 780, and 1000 Hz under an applied zero dc field. As shown in Figure 4, the ac magnetic susceptibilities of 1 display the temperature and frequency dependent characters in both the in-phase (χ_M') and out-of-phase (χ_M''), which are the typical features associated with the single molecule magnet behavior. Temperature dependence of 1 was also confirmed by the frequency dependence of χ_M' and χ_M'' in the range 2–10 K (Figure 5). The peaks of the frequency-dependent χ_M'' susceptibilities shift to a broad range under a zero dc field.





Figure 4. Temperature dependence of the in-phase $[\chi_{M}', (a)]$ out-of-phase $[\chi_{M}'', (b)]$ ac susceptibility under an applied zero Oe field for **1**.

The Cole-Cole plots of complex 1 show one semicircular shape from 2 to 10 K (Figure 6), which confirms the presence of one relaxation process. These plots are well fitted using a generalized Debye Model. The obtained values of α are in the range 0.004 to 0.16, indicating a moderate distribution of magnetic relaxation times, especially at low temperature. At high temperature, the relaxation time obeys the Arrhenius law: $\tau = \tau_0 \exp(U_{\text{eff}}/k_{\text{B}}T)$ [Equation (1)] (Figure 7). The best fitting results give energy barriers $U_{\text{eff}} = 49.9$ K and pre-exponential



Figure 5. Frequency dependence of the in-phase $[\chi_{M}', (a)]$ out-of-phase $[\chi_{M}'', (b)]$ ac susceptibility under an applied zero Oe field for **1**.

factors $\tau_0 = 1.54 \times 10^{-6}$ s and the obtained τ_0 value is consistent with the expected for a SMM $(10^{-6}-10^{-11} \text{ s}).^{[47]}$



Figure 6. Cole-Cole plots measured at 2–10 K in zero-dc field; the solid lines are the best fit to the experimental data.

At low temperature, $\ln \tau$ vs. 1/*T* plots indicate that other relaxation pathways may exist. As processes including spinlattice relaxation of Quantum Tunneling of the Magnetization (QTM), Raman and Orbach are considered, The magnetic data of **1** were fitted with equation: $1/\tau = \tau_{OTM}^{-1} + CT^{n} + \tau_{0}^{-1}$ Ilgemeine Chemi

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Figure 7. Relaxation time $\ln \tau$ vs. T^{-1} plot for **1** under zero-dc field. The blue solid lines represent data fits to Equation (1) and the red solid lines represent data fits to Equation (2).

 $\exp(-U_{\rm eff}/k_{\rm B}T)$ [Equation (2)].^[48,49] The best fit is in good agreement with the data over the entire temperature range and gives the parameters of $U_{\rm eff} = 56.5$ K, $\tau_0 = 2.11 \times 10^{-7}$ s, $\tau_{\rm QTM} = 1.59 \times 10^{-3}$ s, C = 0.75 s⁻¹·K^{-4.56}, n = 4.56.

Two Dy2 complexes, $[Dy_2(NO_3)_2(L)_2(DMF)_4]$ and $[Dy_2(L)_2(NO_3)_2(MeOH)_4]$ have been reported with the same ligand^[50,51] but using different auxiliary ligands. To our surprise, their magnetic properties are totally different indicating that the structural change could lead to variation of the magnetic properties.

Conclusions

In conclusion, we have successfully obtained two isostructural dinuclear lanthanide clusters using a Schiff-base ligand under solvothermal conditions. The dinuclear framework is composed of two ligands which are doubly-deprotonated, two chelated nitrate anions and four coordinated DME molecules. Both the metal centers are surrounded by NO7 environments in distorted triangular dodecahedron (D_{2d}) arrangement with a minimum CshM value of 5.49. The magnetic properties of them are investigated, and cluster **1** displays slow magnetic relaxation behavior with $U_{\text{eff}} = 49.9$ K, $\tau_0 = 1.54 \times 10^{-6}$ s. This work demonstrates the potential applications of the Schiff-base ligands in the construction of SMMs.

Experimental Section

General: H_2L was prepared by mixing salicylaldehyde and 2-aminophenol in 1:1 molar ratio in hot methanol. The reagents and chemicals were obtained from Sinopharm Chemical Reagent Co., Ltd, China and used as purchased without further purification. IR spectra were recorded in the range 4000–400 cm⁻¹ on a Nicolet 470 FT-IR spectrophotometer. Powder XRD patterns were recorded on a Rigaku D/Max-2500 diffractiometer at 40 kV and 100 Ma with a Cu-target tube and a graphite monochromator. Elemental analyses for C, H and N were carried out on a Perkin–Elmer 2400 analyzer. Magnetic susceptibility data were collected on a Quantum Design MPMS-7 SQUID magnetometer.

Synthesis of [**Dy**₂**L**₂(**NO**₃)₂(**DME**)₄] (1): A mixture of H₂L (0.0214 g, 0.1 mmol), Dy(NO₃)₃·6H₂O (0.0456 g, 0.1 mmol), NaOH (0.0080 g, 0.2 mmol), EtOH (1.5 mL) and DME (0.5 mL) was sealed in a Pyrextube (10 mL). The tube was heated for 72 h at 353 K. After cooling to room temperature, yellow crystals of **1** were obtained in a yield of 36% (0.0218 g based on Dy). $C_{42}H_{54}N_8O_{14}Dy_2$: calcd. C, 41.35; H, 4.46; N, 9.19%; found: C, 41.52; H, 4.38; N, 9.03%. **IR** (KBr, selected data): $\tilde{v} = 1636$ (s), 1612 (s), 1585 (m), 1481 (m), 1467 (s), 1450 (m), 1406 (w), 1384 (m), 1350 (w), 1310 (m), 1280 (m), 1262 (w), 1171 (w), 1147 (w), 1029 (w), 918 (w), 830 (w), 745 (m), 601 (w), 488 (w) cm⁻¹.

Synthesis of [Gd₂L₂(NO₃)₂(DME)₄] (2): Complex 2 was prepared using the same method as that of 1 except that Dy(NO₃)₃·6H₂O was replaced by Gd(NO₃)₃·6H₂O. Yield: 29% (0.0175 g based on Gd). C₄₂H₅₄N₈O₁₄Gd₂: calcd. C, 41.71; H, 4.50; N, 9.27%; found: C, 41.81; H, 4.42; N, 9.13%. **IR** (KBr, selected data): $\tilde{v} = 1633$ (s), 1612 (s), 1581 (m), 1481 (m), 1466 (s), 1449 (m), 1407 (w), 1385 (m), 1349 (w), 1309 (m), 1279 (m), 1261 (w), 1171 (w), 1147 (w), 1029 (w), 918 (w), 829 (w), 745 (m), 600 (w), 487 (w) cm⁻¹.

X-ray Diffraction Crystallography: Crystals suitable for X-ray structure analysis were carefully collected. Measurements were made on Bruker Smart Apex II diffractometer with graphite-monochromated Mo- K_{α} radiation ($\lambda = 0.71073$ Å) using the ω - 2θ scan technique. Semiempircal multi-scan absorption corrections were applied by SADABS and integration of diffraction profiles were applied by SAINT. The crystal structures were solved by direct methods and refined by full-matrix least-squares technique using the SHELXTL refinement package.^[41,42] The crystallographic data for complexes **1** and **2** are listed in Table 1. The selected bond lengths and angles are listed in Table 2.

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

	1	2	
Empirical formula	C ₄₂ H ₅₄ N ₈ O ₁₄ Dy ₂	C42H54N8O14Gd2	
Formula weight	1219.94	1209.45	
Crystal system	monoclinic	monoclinic	
Space group	$P2_1/n$	$P2_1/n$	
a /Å	11.808(2)	11.765(2)	
b /Å	14.180(2)	14.142(3)	
c /Å	15.195(3)	15.196(3)	
a /°	90	90	
β /°	107.561(3)	107.225(3)	
γ /°	90	90	
$V/Å^3$	2425.7(7)	2414.9(7)	
Z	2	2	
$Dc /g cm^{-3}$	1.670	1.663	
$_{\circ}^{\theta}$ range for data collection $_{\circ}^{\circ}$	1.93 ~ 27.52	1.94 to 27.72	
μ /mm ⁻¹	3.127	2.793	
Reflections collected / unique	14492/ 5511	14173 / 5495	
$R_{\rm int}$ reflections	0.0220	0.0188	
Data / restraints / param- eters	5511 / 0 / 355	5495/1/355	
<i>F</i> (000)	1212.6	1205.4	
Goodness-of-fit	1.024	1.030	
Final <i>R</i>	$R_1 = 0.0217, wR_2$	$R_1 = 0.0208, wR_2$	
	= 0.0478	= 0.0456	
R indices (all data)	$R_1 = 0.0290, wR_2$ = 0.0506	$R_1 = 0.0296, wR_2$ = 0.0495	
$\Delta \rho(\text{max, min})$ /e·Å ⁻³	0.48 and -0.79	0.76 and -0.55	

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Zeitschrift für anorganische und allgemeine Chemie Table 2. Selected bond lengths /Å and angles /° for complexes 1 and 2. 1 Bond Bond Dy1-07 2.316(2) Dy1-03 2.599(2)Dy1-N1 Dy1-02 2.495(2)2.367(2)Dy1-06 2.377(2) Dy1-04 2.443(2)Dy1-O2#1 Dy1-01 2.197(2)2.323(2)Angle Angle O4-Dv1-O6 76.43(7) O2-Dy1-N1 66.00(6) O2#1-Dy1-O4 04-Dy1-01 130.88(7) 142.07(6) O2-Dy1-O4 O4-Dy1-O3 50.33(7) 79.24(6) 06-Dy1-07 72.18(8) O4-Dy1-N1 79.94(7) 01-Dy1-07 81.75(8) O2-Dy1-O7 152.97(7) O2#1-Dy1-O7 01-Dy1-06 143.33(7) 104.47(8) O3-Dy1-O7 73.41(9) 02-Dy1-06 80.89(6) 02^{#1}–Dy1–O6 O3-Dy1-O6 113.72(8) 76.58(7) O3-Dy1-O1 O2-Dy1-O1 123.20(7)81.61(8) O2#1-Dy1-O1 N1-Dy1-O7 138.02(8) 85.79(7) N1-Dy1-O6 142.27(7)O2-Dy1-O3 117.05(7) N1-Dy1-O1 O2#1-Dy1-O3 73.54(7) 167.40(7) O2#1-Dy1-N1 N1-Dy1-O3 69.87(7) 106.89(6) O4-Dy1-O7 92.01(8) 2 Bond Bond Gd(1)-N(1) Gd(1)-O(7) 2.332(2)2.522(2)Gd(1)-O(4)2.599(2)Gd(1) - O(6)2.402(2)Gd(1)-O(1) 2.219(2)Gd(1) - O(2)2.383(2)Gd(1)-O(2)#1 Gd(1)-O(3) 2.462(2)2.339(2) Angle Angle O4-Gd1-N1 69.88(7)O6-Gd1-O1 143.73(7)O1-Gd1-N1 72.88(7) O6-Gd1-O3 76.52(7) 82.05(7) O1-Gd1-O4 O6-Gd1-O7 72.06(8) O3-Gd1-N1 80.00(7) O2#1-Gd1-N1 106.59(6) 49.93(7) O3-Gd1-O4 O2-Gd1-N1 65.25(6) O3-Gd1-O1 130.80(7) O2#1-Gd1-O4 168.37(7)O7-Gd1-N1 138.16(8) O2-Gd1-O4 116.10(7) 122.22(6) O7-Gd1-O4 73.87(8) O2-Gd1-O1 82.19(8) O2#1-Gd1-O1 86.32(6) O7-Gd1-O1 O2#1-Gd1-O3 O7-Gd1-O3 92.39(8) 141.39(6) O6-Gd1-N1 142.48(7)O2-Gd1-O3 78.97(6) O6-Gd1-O4 113.45(7) O2#1-Gd1-O7 104.69(8) O2#1-Gd1-O6 O2-Gd1-O7 153.67(7) 76.33(6) O2-Gd1-O6 81.71(6) Crystallographic data (excluding structure factors) for the structures in

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-1985549 for 1 and CCDC-1985550 for 2 (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http://www.ccdc. cam.ac.uk).

Supporting Information (see footnote on the first page of this article): Including structural characterization information, some magnetic data information and the information of examples of O-bridged dinuclear dysprosium complexes having SMM properties in the literatures.

Acknowledgements

We gratefully acknowledge the financial support from the National Science Foundation of China (Grant No: 21771061), the Project of Shandong Province Higher Educational Science and Technology Program (J12LD54), the Youths Science Foundation of Jining University (no. 2017BSZX09), and the Talent Culturing Plan for Leading Disciplines of University in Shandong Province.

Keywords: Single molecule magnet; Schiff-base ligand; Lanthanides; Dinuclear clusters; Energy barriers

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Received: February 21, 2020 Published Online: ■ Zeitschrift für anorganische und allgemeine Chemie

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Single-Molecule Magnetism in Dy_2 Cluster Based on a Schiff Base Ligand

