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Research paper

Modulating SMM behaviors in phenoxo-O bridged Dy_2 compounds via different β-diketonate

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ABSTRACT

Two new Dy_2 compounds, $[Dy(TTA)_2L]_2$ **(1)** and $[Dy(dbm)_2L]_2$ **(2)** (TTA = 2-thenoyltrifluoroacetone, and HL = 2-[(4-chlorophenyl)imino]methyl]-8-hydroxyquinoline and dbm = dibenzoylmethane), were synthesized via solvent evaporation method, and the structures and magnetic properties of them have been characterized. The X-ray structural analysis show that both **1** and **2** are binuclear dysprosium complexes with similar structure, and the two central Dy atoms are bridged by two phenoxo-O atoms of two 8-hydroxyquinoline Schiff base ligands. The only difference is the coordinate β-diketonate. Magnetic measurements indicated that the two Dy_2 compounds display different single-molecular magnets (SMMs) behaviors with U_{eff}/k_B = 29.4 K for **1** and U_{eff}/k_B = 43.5 K for **2**.

1. Introduction

Over the past two decades, the design and construction of Ln(III)-based single-molecule magnets (SMMs) have attracted much interest and attention due to their potential applications for using in high-density magnetic information storage, quantum computing devices, and molecular spintronics [1]. The compounds exhibit slow relaxation of magnetization on molecular level, and they normally display in-phase and out-of-phase ac signals and/or magnetic hysteresis-loops below the blocking temperature (T_B), which are so-called single-molecule magnets (SMMs) [2]. In 2003, the first Ln(III)-based single molecule magnet (SMM), $[Pc_2Ln]^{-}\cdot TBA^{+}$(Ln = Tb and Dy, *Pc* = dianion of phthalocyanine; $TBA^{+} = N(C_4H_9)_4{}^{+}$), was discoveried by Naoto Ishikawa group [3]. Whereafter, lots of Ln(III)-based compounds showing slow relaxation of magnetization have been reported [4]. For a favourable single-molecule magnet (SMM), large effective energy barrier (U_{eff}) and high blocking temperature (T_B) are two key and significant factors [5]. Focus on the two key factors, a lot of outstanding and interesting works for Ln (III)-based SMMs have been reported [6]. Throughout these studies, Gao, Tong, Zheng, and Layfield groups have conducted pioneering work in the design and synthesis of Dy(III)-based SMMs [7]. It seems that and mononuclear and binuclear Ln(III)-based compounds can display excellent SMMs behaviors. Here, a number of works focus on how to enhance energy barriers (U_{eff}/k_B) of Ln(III)-based SMMs [8]. In 2016, the Tong group reported a stable pentagonal bipyramidal Dy(III)-based SMM, namely [Dy(bbpen)Br], it has a record effective energy barrier over 1000 K (U_{eff}/k_B = 1025 K) [9]. Later on, Zheng group reported a nearly perfect pentagonal bipyramidal Dy(III)-based SMM with formula $[Dy(OtBu)_2(py)_5][BPh_4]$ having a larger U_{eff}/k_B of 1815 K [10]. In 2018, research of this aspect has achieved a breakthrough, Layfield group reported a dysprosium metallocene cation $[(Cp^{/Pr5})Dy(Cp^{*})]^{+}$ ($Cp^{/Pr5}$ = penta-isopropylcyclopentadienyl, Cp* = pentamethylcyclopentadienyl) with a largest effective energy barrier (U_{eff}/k_B) of 2219 K and blocking temperature T_B = 80 K [11]. It is the largest effective energy barrier of all the reported Dy(III)-based SMMs at present. These excellent and outstanding works attracted much more inorganic and materials chemistry researchers for focusing the deep study of Ln(III)-based SMMs.

Attempts to seek efficient synthetic strategies to assemble Dy(III)-based SMMs and further to explore the outstanding magnetic properties, in the paper, we designed and obtained two dinuclear Dy(III)-based compounds, $[Dy(TTA)_2L]_2$ **(1)** and $[Dy(dbm)_2L]_2$ (**2**). The two Dy_2 compounds were synthesized by using an 8-hydroxyquinoline Schiff base ligand (HL) and two β-diketones (Schemes 1 and 2). The structures and magnetism of **1** and **2** have been deeply studied. Complexes **1** and **2** show very similar structural features with a Dy_2 core, however, we find that by adjusting the coordination environment of Dy (III) ion of **1** and **2** via two *β*-diketones yielded different single-

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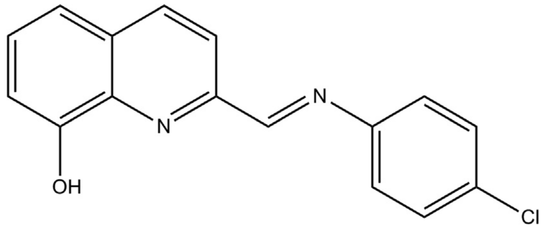


Scheme 1. The structure of ligand HL.

molecular magnets (SMMs) behaviors.

2. Experimental section

2.1. Synthesis of 1 and 2

$Dy(TTA)_3·2H_2O$ (0.04 mmol) was added to a solution of 15 mL *n*-heptane and refluxed for 3 h, then the solution was cooled to 60 °C and 5 mL CH_2Cl_2 containing HL (0.04 mmol) was added. The mixed solution was stirred at 60 °C for about 1 h. After reaction finished, the mixed solution cooled to room temperature. The mixture was filtered and the filtrate was kept at room temperature, red and needlelike crystals of **1** were obtained after about one week, yield about 41% (based on Dy $(TTA)_3·2H_2O$). Elemental analysis (%) calcd for $C_{64}H_{36}Cl_2Dy_2F_{12}N_4O_{10}S_4$ (M_W = 1773.10): C 43.31, H 2.03, N 3.16. Found: C 43.45, H 1.91, N 3.27. IR (KBr, cm^{-1}): 1651(s), 1598(w), 1552(w), 1485(m), 1258(s), 1202(s) , 1148(m), 1096(w), 907(w), 842(m), 765(m) , 562(m).

$Dy(dbm)_3·2H_2O$ (0.03 mmol) was added to a solution of HL (0.03 mmol) in 20 mL of CH_3OH/CH_2Cl_2 (v/v = 3:1), and the mixture was stirred at 80 °C for 4 h. Then the mixed solution cooled to room temperature and filtered. The filtrate was kept at room temperature, red and needlelike crystals of **2** were obtained after about 10 days. Yield about 47% (based on $Dy(dbm)_3·2H_2O$). Elemental analysis (%) calcd for $C_{94}H_{70}Cl_4Dy_2N_4O_{11}$ (M_W = 1898.34): C 59.42, H 3.69, N 2.95; Found: C 59.46, H 3.58, N 2.83. IR (KBr, cm^{-1}): 1652(s), 1599(w), 1553(w), 1486(m), 1259(s), 1203(s), 1145(m), 1098(w), 907(w), 840(m), 760(m), 561(m).

2.2. Single-crystal X-ray diffraction measurements

Single crystal X-ray diffraction data of **1** and **2** was collected on a computer-controlled Rigaku Saturn CCD area detector diffractometer, equipped with confocal monochromatized Mo Kα radiation with a radiation wavelength of 0.71073 Å using the ω-φ scan technique. The structures were solved by direct methods and refined with a full-matrix least-squares technique based on F^2 using the SHELXS-2016 and SHELXL-2016 programs [12]. Anisotropic thermal parameters were assigned to all non-hydrogen atoms. Crystallographic data and structural refinement parameters are listed in Table 1. CCDC 1061001 for **1** and 1973326 for **2** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

3. Results and discussion

3.1. Structure descriptions of 1 and 2

In this paper, the reactions of ligand HL and $Dy(TTA)_3·2H_2O$/Dy $(dbm)_3·2H_2O$ in a mixture of *n*-heptane/CH_2Cl_2 or CH_3OH/CH_2Cl_2, red and needlelike crystals of **1** and **2** suitable for single-crystal X-ray diffraction were obtained, respectively. X-ray diffraction analyses reveal that both compounds **1** and **2** possesse a dinuclear structure, however, compound **1** crystallizes in the triclinic space group $P\bar{1}$ with $Z = 1$, and compound **2** crystallizes in the monoclinic space group $P2_1/n$ with $Z = 2$ (Table 1). For **1** (Fig. 1), the dinuclear dysprosium complex is composed of two Dy^{3+} ions, four TTA^- and two L^-, and each central Dy^{3+} ion is eight-coordinated with a N_2O_6 coordination environment. According to the computational result by using SHAPE 2.0 software (see Table S1), each central Dy^{3+} ion possess a distorted triangular dodecahedron geometrical configuration. The coordination mode of HL and TTA of are shown in Figs. S1 and S2. The ligand HL serves as a tridentate ligand and chelates the central Dy1 atom through one phenoxide oxygen atom (O1), one pyridyl ring nitrogen atom (N1) and one imine nitrogen atom (N2), and the TTA^- adopts a bidentate mode to chelate the central Dy1 atom. The two central Dy^{3+} ions are bridged by two μ_2-phenoxide oxygen atoms (O1 and O1a) of two ligands HL. The two Dy atoms and two μ_2-phenoxide oxygen atoms form a parallelogram Dy_2O_2 core with a Dy–O–Dy angle of 108.77(8)°, and the Dy···Dy distance is 3.8319(15) Å. Moreover, in **1**, the lengths of the Dy–O bonds are in the range of 2.306(5)–2.408(5) Å, the Dy1–N1 and Dy1–N2 bond distances are 2.461(5) and 2.661(6) Å, respectively; the O–Dy–O angles are in the in the range of 70.17(17)–150.62(18)°. These bond distances and angles of **1** are comparable to those of the reported Ln(III)-based complexes [13].

As shown in Fig. 2, the molecular structure for **2** is composed of two Dy^{3+} ions, four dbm^- and two L^-. The coordination environment and geometrical configuration of central Dy^{3+} ion are almost the same as these of **1** (see Table S1), the only difference is the Dy–O and Dy–N bond distances. The Dy–O bond distances are in the range of 2.294(2)–2.434(2) Å, the Dy1–N1 and Dy1–N2 bond lengths are 2.489(3) and 2.672(3) Å, respectively. Furthermore, in **2**, the distance of the two neighbouring Dy^{3+} ions is 3.8776(7) Å and the Dy–O–Dy bone angle is 109.06(8) °, which are slight bigger than these in **1**.

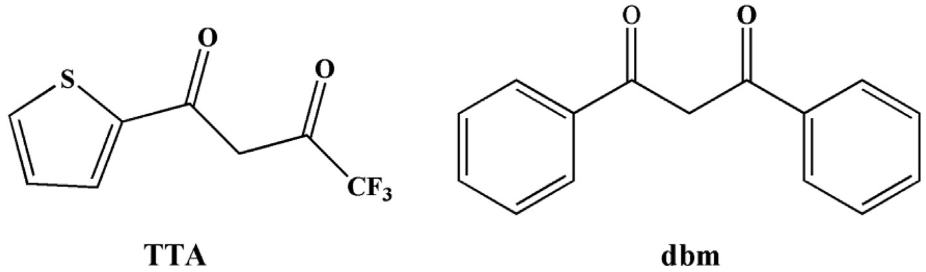
3.2. Powder X-ray diffraction

In order to verify phase purities of compounds **1** and **2**, the crystalline products of them have been characterized by Powder X-ray diffraction (PXRD) at room temperature (Figs. S3 and S4). The experimental PXRD patterns are in good agreement with the simulated ones from the single crystal data, indicating the high purities of the synthesized samples of **1** and **2**.

3.3. Magnetic properties

Variable-temperature direct-current (dc) magnetic susceptibility for compounds **1** and **2** were measured under the field of 1000 Oe and during the temperature range 300–2.0 K. The plots of $\chi_M T$ versus T for **1** and **2** are shown in Fig. 3. At 300 K, the $\chi_M T$ values are 28.32 cm^3 K mol^{-1} for **1** and 28.37 cm^3 K mol^{-1} for **2**, respectively; which are good agree with the expected value of 28.34 cm^3 K mol^{-1} for two free Dy(III) ions ($^6H_{15/2}$, $g = 4/3$). As the temperature is decreased, the $\chi_M T$ values of **1** and **2** gradually decrease from 300 to 50 K and then rapidly drop to a minimum value of 8.90 **(1)**, 8.55 (**2**) cm^3 K mol^{-1} at 2.0 K. Such magnetic behaviors generally due to one or a combination of the following two phenomena: (1) the progressive depopulation of the excited Stark sub-levels of the Dy^{III} ions; (2) weak antiferromagnetic interactions between the two adjacent Dy^{III} ions of **1** and **2** [14].

Alternating-current (ac) magnetic susceptibility of compounds **1** and **2** were undertaken at 3.0 Oe ac field with H_{dc} = 0 field in order to investigate the dynamics magnetic behavior. As shown in Fig. 4, complexes **1** and **2** exhibit pronounced frequency-dependent out-of-phase (χ'') signals and evident peaks are observed, indicating slow magnetic relaxation with the characteristic of SMM behavior [15]. However, the χ'' peaks were observed during the temperature range 4.5–9.0 K for **1**; and the shoulder peaks of χ'' signals were appeared during the temperature range 8.5–13.0 K between 711 and 3111 Hz for **2**. For further study the dynamics magnetic behavior of **1** and **2**, the frequency dependence of ac susceptibilities were also measured under different temperature for **1** (2.0–10.0 K) and for **2** (2.0–14.0 K) (Figs. S5 and



Scheme 2. The structures of TTA and dbm.

Table 1
Crystallographic data and structure refinement summary for **1** and **2**.

Complexes	1	2
Empirical formula	$C_{64}H_{36}Cl_2Dy_2F_{12}N_4O_{10}S_4$	$C_{94}H_{70}Cl_4Dy_2N_4O_{11}$
*M*r	1773.10	1898.34
T (K)	113(2)	113(2)
Crystal system	triclinic	monoclinic
Space group	*P*1	$P2_1/n$
a/Å	10.797(2)	11.126(2)
b/Å	12.891(3)	23.786(5)
c/Å	13.362(3)	15.314(3)
α/°	69.90(3)	90
β/°	71.01(3)	101.15(3)
γ/°	79.98(3)	90
V/Å^3	1647.5(6)	3976.2(14)
Z	1	2
D_{calcd}/g cm^{-3}	1.787	1.586
μ/mm^{-1}	2.553	2.066
θ/°	2.00 to 25.02	1.60 to 27.89
F(0 0 0)	866	1900
Reflections collected	13,792	38,855
Unique reflns	5787	9451
R_{int}	0.0498	0.0516
GOF (F^2)	1.050	1.103
R_1, wR_2 ($I > 2\sigma(I)$)	0.0506, 0.1220	0.0341, 0.0834
R_1, wR_2 (all data)	0.0574, 0.1275	0.0430, 0.0921

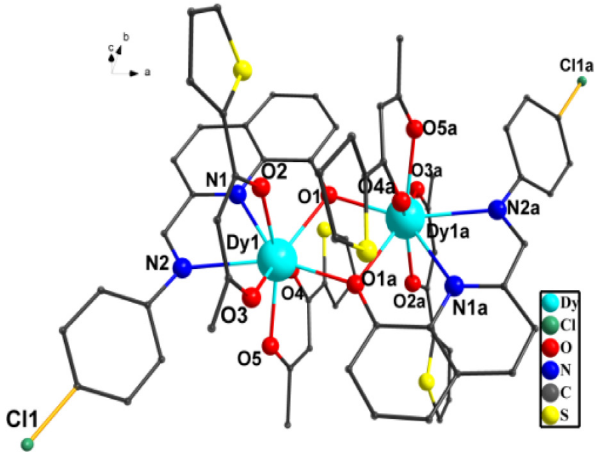


Fig. 1. Molecular structure for **1** (hydrogen atoms are omitted for clarity).

S6). The out-of-phase (χ'') signals of the ac susceptibility of **1** and **2** show temperature dependences, which further confirms the SMM behaviors in **1** and **2**.

What is noteworthy that the plots of ln(τ) versus $1/T$ for compounds **1** and **2** under zero dc field exhibit obvious curvature, which indicates that perhaps another relaxation pathway is also operative. So, we fitted the magnetic data with the Eq. (1) [16]:

$$ln\tau = -ln[AT + B + CT^n + \tau_0{}^{--1}exp(-\Delta E/K_BT)] \quad (1)$$

where $AT + B$, CT^n and $\tau_0{}^{-1}\exp(\Delta E/k_BT)$ represent direct, Raman and Orbach relaxation processes, respectively. The best fit was obtained for $n = 5.08$, $A = 0.3764\ s^{-1}\ K^{-5.08}$, $C = 0.08811$, $U_{eff}/k_B = 30.7$ K and $\tau_0 = 3.98 \times 10^{-6}$ s for **1**, and $n = 4.40$, $A = 0.4773\ s^{-1}\ K^{-4.40}$, $C = 0.4227$, $U_{eff}/k_B = 45.7$ K and $\tau_0 = 1.29 \times 10^{-6}$ s for **2** (Fig. 5). The important parameter values of U_{eff}/k_B and τ_0 for **1** and **2** are comparable to those of reported Dy_2 SMMs [17]. The small *A* and *C* value indicate that the relaxation process can dominate by the Orbach mechanism at the high temperature and the Raman mechanism at the low temperature.

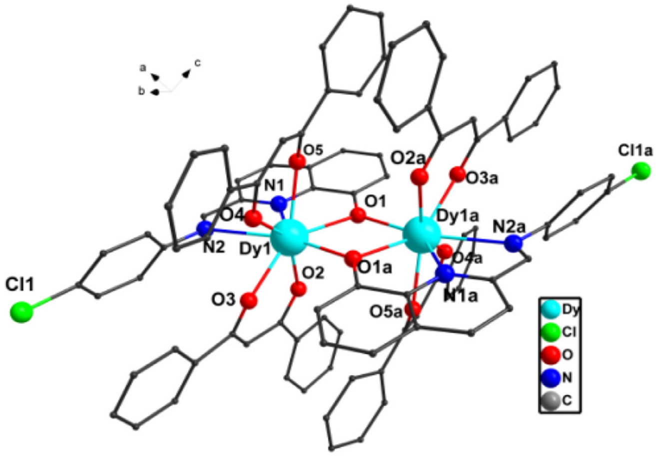


Fig. 2. Molecular structure for **2** (hydrogen atoms are omitted for clarity).

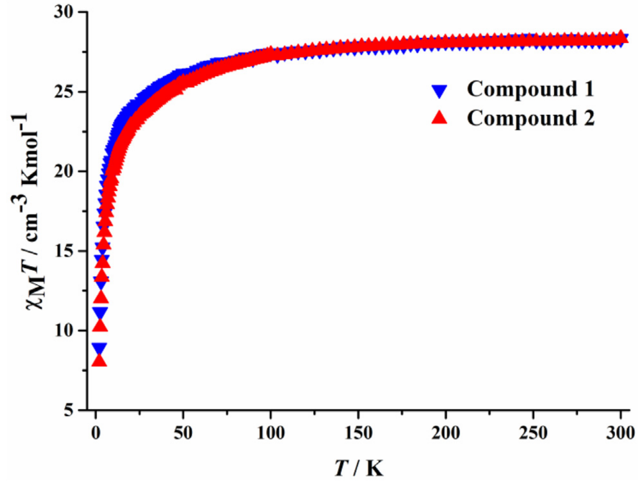


Fig. 3. Temperature dependence of the χ_MT products at 1.0 kOe for **1** and **2**.

The Cole–Cole plots of χ'' *vs.* χ' for **1** and **2** are shown in Fig. 6, for **1**, it is a relatively symmetrical and semicircle shape, and the best fits the Cole–Cole curves in the range of 2.0–10.0 K were obtained with $\alpha = 0.13$–0.24 using the generalized Debye model. For **2**, it shows an asymmetric shape, and we fitted it by using Debye model and obtained

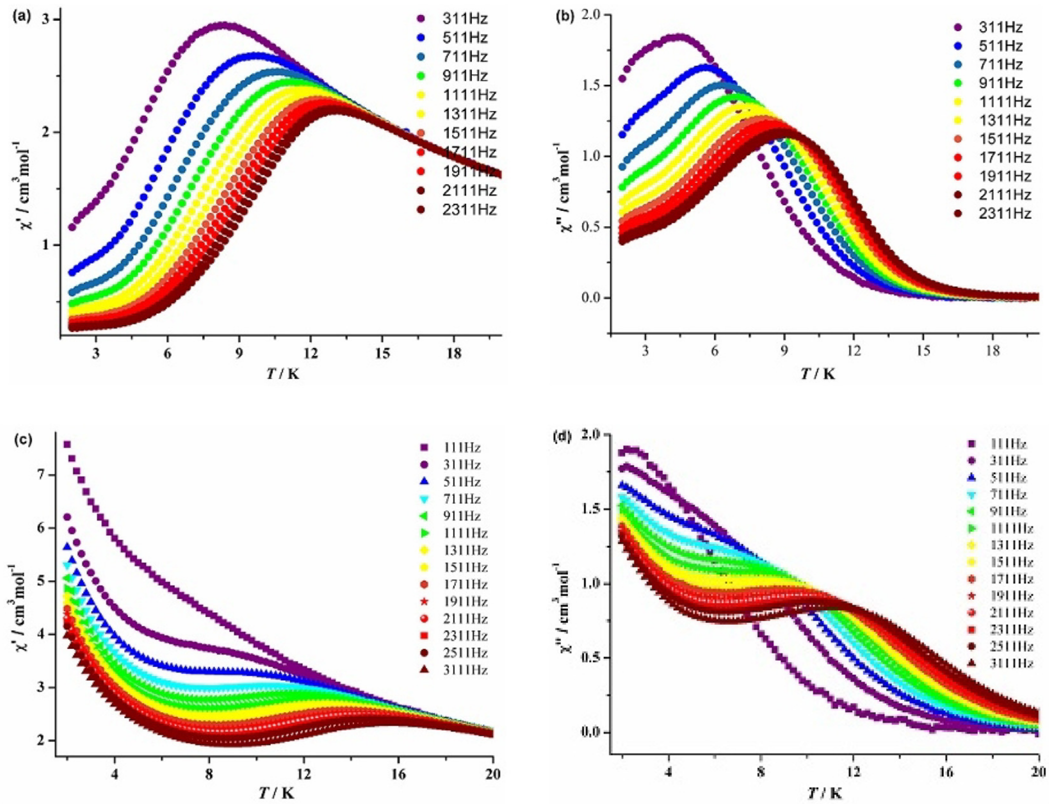


Fig. 4. Temperature dependence of the in-phase and out-of-phase components of the ac magnetic susceptibility for **1** and **2** under $H_{dc} = 0$ Oe field with an oscillation of 3.0 Oe.

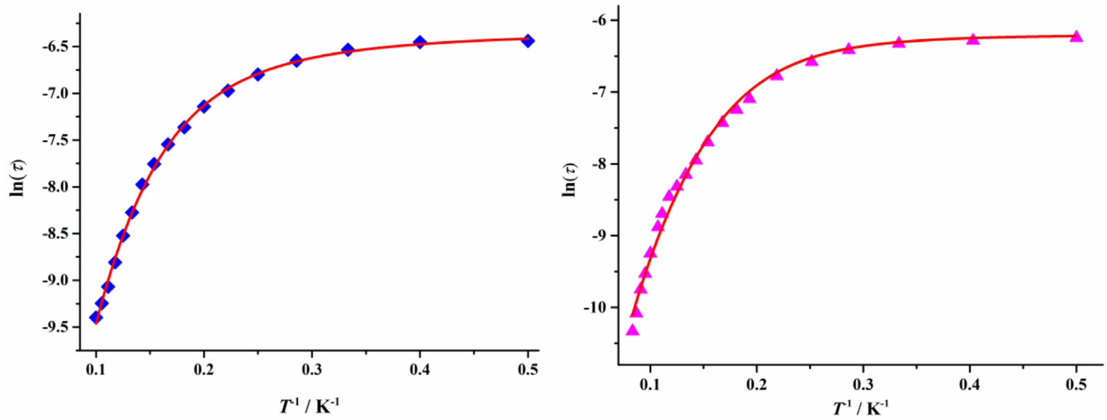


Fig. 5. Plots of ln(τ) versus T^{-1} fitting to the Arrhenius law for complexes **1** (left) and **2** (right) under $H_{dc} = 0$ Oe field.

$\alpha = 0.17$–0.32. The α value of **2** is larger than that of **1**, which imply a wider distribution of relaxation time in **2**.

4. Conclusion

In summary, two new dinuclear dysprosium compounds (**1** and **2**) based on an 8-hydroxyquinoline Schiff-base ligand have been synthesized. The structures and magnetic properties of them have been deeply studied. Although the two phenoxo-O bridged binuclear complexes with similar structures, ac susceptibility study show that compounds **1** and **2** exhibit different single-molecular magnets (SMMs) behaviors.

CRediT authorship contribution statement

Yin Ling Hou: Writing - original draft, Software. **Ting-Ting Yang:** Visualization, Investigation, Data curation. **Wen-Xin Zhao:** Data curation, Software. **Chen-Juan Fan:** Data curation. **Li-Li Yan:** Software. **Xiao-Fen Guan:** Supervision. **Jia Ji:** Validation, Funding acquisition. **Wen-Min Wang:** Conceptualization, Methodology.

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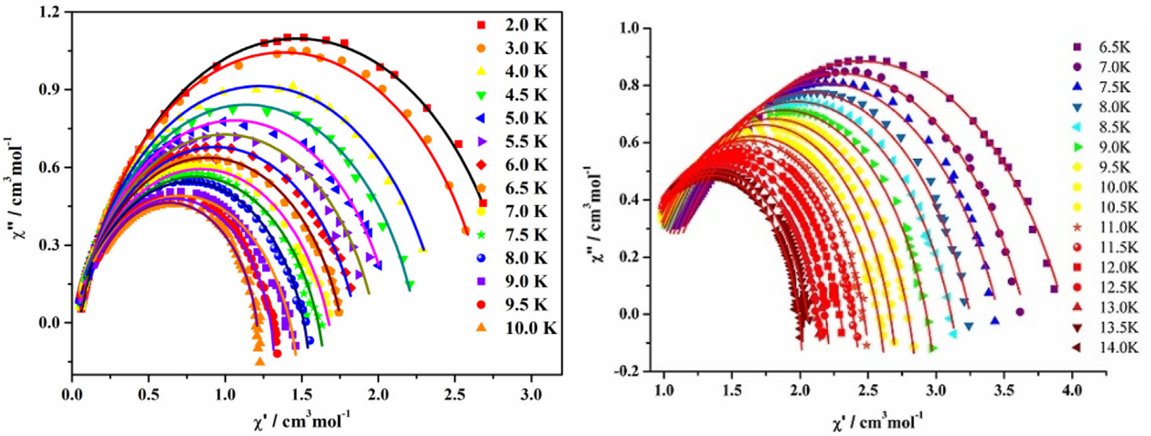

Fig. 6. Cole–Cole plots for **1** (left) and **2** (right) measured in zero-dc field. The red solid lines are the best fit to the experimental data, obtained with the generalized Debye model with $\alpha = 0.13–0.24$ for **1** and $\alpha = 0.17–0.32$ for **2**. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ica.2020.119595.

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