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Synthesis, structure, magnetic and luminescent properties of two dysprosium Single-Molecule Magnets based on phenoxide dye ligands.

Jérôme Long*,^a Alexander N. Selikhov,^{b,c} Ekaterina Mamontova,^a Konstantin A. Lyssenko,^{c,d} Yannick Guari,^a Joulia Larionova,^a and Alexander A. Trifonov*,^{b,c}

We report here two dysprosium complexes based on phenoxide-based emissive ligands 1-(2,4-dimethylphenylazo)-2-naphtholate (L1) and anthracenolate (L2). While 1 is a dinuclear homoleptic complex $[\text{Dy}_2(\text{L1})_6] \cdot 2\text{C}_7\text{H}_8$ exhibiting a zero-field slow relaxation of the magnetization, 2 is a mononuclear complex $[\text{Dy}(\text{L2})_3(\text{py})_3]$ (py = pyridine) showing a field-induced relaxation.

Lanthanide-based complexes have been extensively studied for the last fifteen years due to their impressive magnetic properties that give rise to the appearance of a slow relaxation of the magnetization and so-called Single-Molecule Magnet (SMM) behaviour, which finds potential applications in high-density information storage or quantum computing.¹⁻⁴ Yet, such expectations require to efficiently optimize the magnetic properties of the complexes by controlling the magnetic anisotropy through a modulation of the lanthanide ions' coordination environment. Indeed, in such systems, the high magnetic anisotropy of certain lanthanide ions may be exacerbated in a specific ligand environment/symmetry to provide a large crystal-field splitting and in turn generate a large anisotropic barrier, Δ , separating two opposite magnetic states. Hence, the presence of dominated thermally activated relaxation (or Orbach relaxation) might result in the appearance of the SMM behaviour. One of the main challenges in the field is dedicated to increase the anisotropic barriers and the blocking temperatures.⁵⁻⁶ Despite the fact that other processes, such as the Quantum Tunnelling of the Magnetization (QTM), direct or Raman relaxations may create their own relaxation pathways therefore decreasing the effective energy barrier, simple electrostatic considerations provide a synthetic strategy based on the optimal alliance between the electronic density of specific 4f lanthanide ions (oblate or prolate) and the crystal-field imposed by the ligand environment (axial or equatorial).⁷ Following this approach, numerous lanthanide complexes with high anisotropic barriers of few thousands of wavenumbers have been recently reported,^{5, 8-11} while a series of dysprosium metallocene complexes has demonstrated the best magnetic properties with the highest energy barriers and hysteresis^{10, 12-13} at temperatures that even exceed liquid nitrogen boiling temperature.¹⁴ The majority of the aforementioned examples belong to organometallic complexes involving cyclopentadienyl derivatives (Cp^{R}) providing linear $\text{Cp}^{\text{R}}\text{-Dy-Cp}^{\text{R}}$ angles, short Dy- Cp^{R} distances in axial positions and the lack of ligands in equatorial positions, which generates a strong axial

crystal-field maximizing the anisotropy.¹²⁻¹⁴ Using such route with coordination ligands involving nitrogen or oxygen-lanthanide bonds appears however more intricate.

On the other hand, although exchange interactions between lanthanide ions in SMMs are commonly considered as weak with respect to the dominating single-ion anisotropy, their role is not yet clearly understood.¹⁵ Different literature examples have demonstrated a decrease of the anisotropy barrier in polynuclear Dy^{3+} based complexes due to the mixing of the states and/or QTM increase.¹⁶ However, several cases show also a positive impact of the magnetic interactions caused by an exchange bias effect that shifts the usual zero-field QTM relaxation,¹⁷⁻²⁰ that could eventually lead to a large magnetic coercivity.²¹ Obviously, dinuclear complexes represent therefore the simplest model to study the influence of exchange interactions over the slow relaxation dynamics.²² In this sense, one strategy consists in employing bulky phenoxide ligands in the axial position that ensure Ln-O short distances,^{8, 23-25} and potentially bridge lanthanide ions. In addition, such ligands may be appropriate to implement additional functionalities, such as luminescence.²⁶⁻²⁸ With this in mind, numerous dyes incorporating phenoxide moieties may be used as ligands for the design of luminescent SMMs.^{26, 28-29}

Following this idea, we report in this communication the synthesis, structure, magnetic and luminescent studies of two dysprosium complexes showing different nuclearities and based on two phenoxide dye ligands. Remarkably, the dinuclear complex reveals a zero-field slow relaxation reflecting a genuine SMM behaviour, while the mononuclear exhibits a field-induced slow relaxation.

Firstly, the reaction of $\text{Dy}[\text{N}(\text{SiMe}_3)_2]_3$ with three equivalents of 1-(2,4-dimethylphenylazo)-2-naphthol (orange dye Sudan-II), in toluene at 60 °C expectedly affords a homoleptic dinuclear naphtholate complex $[\text{Dy}_2(\text{L1})_6] \cdot 2\text{C}_7\text{H}_8$ (**1**) (Scheme 1). Recrystallization from cold toluene resulted in the isolation of the complex as bright-orange moisture and air sensitive **1** crystals in 90% yield. Dissolution of **1** in THF does not lead to its dissociation and the dimeric structure of the complex remains retained after recrystallization from a mixture of THF-hexane. Secondly, reaction of anhydrous DyCl_3 with potassium anthracenolate was carried out in a pyridine solution at 80 °C (Scheme 1). Yellow crystals of complex $[\text{Dy}(\text{L2})_3(\text{py})_3]$ (**2**) were

obtained by slow diffusion of hexane into a solution of the complex in pyridine.

Scheme 1. Synthesis of complexes **1** and **2**.

The single-crystal X-ray diffraction studies reveal that complex **1** crystallizes in the triclinic $P\bar{1}$ space group with 2 molecules of toluene ($1 \cdot 2\text{C}_7\text{H}_8$) as a solvate (Table S1). The asymmetric unit contains two crystallographically independent dinuclear complexes (one of those is shown in Fig. 1 and another in Fig. S1, ESI) both located in the center of symmetry. Each dysprosium is coordinated by three N,O - κ^2 chelating naphtholate ligands; two of them are terminal, while one is μ^2 -bridging two Dy^{3+} centers *via* phenolate oxygen. Hence, each Dy^{3+} ion is seven-coordinated and surrounded by four naphtholate oxygen atoms of four ligands and three nitrogen atoms of $-\text{N}=\text{N}-$ azo groups. Each dysprosium site was analyzed by the SHAPE³⁰ software that indicates a capped octahedron geometry (Table S2). The intramolecular Dy1-Dy1 and Dy2-Dy2 distances are equal to 3.8442(11) and 3.818(1) Å respectively, while the bridging angles Dy-O-Dy are 110.2(2)° and 109.0(2)°. These data are in a good agreement with the distances found in the previously described dinuclear phenolate dysprosium complexes with a similar structure and coordination environment.^{17, 31}

Fig. 1. a) Molecular structure of one of the two crystallographically independent dinuclear complexes in **1**. b) View of one of the centrosymmetric dinuclear core in **1**. c) molecular structure of the mononuclear complex **2**. Colour code: orange, Dy; blue, N; grey, C; Hydrogen atoms have been omitted for clarity.

The O1-Dy1-O1A angle is equal to 150° with short distances of $\text{Dy1-O1} = 2.144(6)$ Å/ $\text{Dy1-O1A} = 2.151(8)$ Å. These latter are expectedly shorter compared to the equatorial distances involving the μ^2 -bridging oxygen with Dy1-O1B distance of 2.325(8) Å. The second crystallographic complex exhibits comparable distances and bond lengths with a O1C-Dy2-O1E angle of 147° and distances $\text{Dy2-O1C} = 2.157(6)$ Å/ $\text{Dy2-O1E} = 2.160(6)$ Å in the axial direction, while the equatorial Dy2-O1D distance is equal to 2.365(9) Å (Fig. S1). In contrast, **2** could be described as a mononuclear complex with a unique crystallographic complex. This structure is similar to that of the previously described 2,6-diisopropyl- and 2,6-dimethyl-phenoxide complexes of Dy.³² The Dy^{3+} adopts a distorted octahedron geometry (Table S2) and is coordinated by three monoanionic anthracenolate ligands and three pyridine

molecules. The aryloxy ligands and pyridine localization yields to the *meridional* isomer of the octahedral complex. The Dy-O bond lengths are 2.131(3), 2.144(3), 2.164(3) Å, with an average value of 2.146 Å, which is in good agreement with the values described for other Dy³⁺ phenoxide derivatives.³² Noticeably, the two shortest Dy-O1 and Dy-O3 distances are located in the axial direction (in *trans* position) and forms an angle O1-Dy-O3 of 161.1(1)°. The lengths of the Dy-N bonds are largely longer with values ranging from 2.490(4) to 2.591(4) Å. For both complexes, the crystal packing analysis reveals that the shortest intermolecular Dy-Dy distances are equal to 13.328(3) and 9.2020(7) Å, for **1** and **2**, respectively (Fig. S2). These values suggest that whereas the dinuclear complexes are relatively well isolated, the presence of intermolecular interactions may be expected for **2**.

The magnetic properties were investigated in both, static (direct current (dc)) and dynamic (alternating current (ac)) modes by using a SQUID MPMS-XL. At room temperature, the χT values of 27.84 and 13.30 cm³·K·mol⁻¹ for **1** and **2**, respectively, are in a relatively good accordance with the expected values of 28.34 cm³·K·mol⁻¹ and 14.17 cm³·K·mol⁻¹ (⁶H_{15/2}) for either two non-interacting Dy³⁺ ions or a single Dy³⁺ ion. Lowering the temperature results in a monotonous decrease of χT due to the thermal depopulation of the m_j levels (Fig. S3). Yet, the pronounced χT decrease for **1** rather suggests antiferromagnetic interactions between the Dy³⁺ ions. The field dependence of the magnetization at 1.8 K reveals a rapid increase of the magnetization as a function of field with the values of 12.64 and 5.53 N β under a 70 kOe field for **1** and **2**, respectively (Fig. S3).

The occurrence of a slow relaxation of the magnetization was investigated by ac measurements. Under a zero-dc field, both **1** and **2** exhibit a significant out-of-phase susceptibility (χ'') component but only **1** shows a maximum of χ'' in our experimental conditions (Fig. S4). The frequency dependence of χ'' for **1** reveals the presence of a temperature dependent single peak (Fig. 2). Despite the presence of two crystallographic dysprosium sites for **1**, the fitting of the Cole-Cole plots (Fig. S5) with a generalized Debye model yields to relatively low values of the α parameter (*i.e.* < 0.3) at low temperature, indicating a moderate distribution of relaxation times (Table S3). From these data, the relaxation time, τ , could be extracted in order to analyse the relaxation dynamics. The $\ln \tau$ vs. T plot (Fig. 3) reveals a deviation from the linearity at low temperature indicating a divergence from a thermally activated process. The whole data range could be modelled using the following equation: $\tau^{-1} = \tau_0^{-1} \exp(-\Delta/kT) + CT^m + \tau_{\text{QTM}}^{-1}$ (Eq. 1).³³ The first term accounts for a thermally activated process, while the second and third ones stand for two-phonon Raman and QTM, respectively. To avoid over-parameterization, the m coefficient was fixed to different values until getting the best correlation coefficient. It turns out that the magnetization relaxes through a combination of these three relaxation processes with a modest Δ value of 24 ± 5 cm⁻¹ (Table 1). It could be noticed that a poor fitting in the low temperature range is observed with a large deviation for the QTM constant. This may be ascribed either to the presence of

two dysprosium sites or the effect of the magnetic interactions between Dy³⁺ ions. Besides, a strong contribution from the Raman process should be underlined.

Fig. 2. Frequency dependence of χ' and χ'' for **1** under a zero dc-field.

With the aim to shortcut the QTM, ac measurements were performed under static dc-fields for both complexes **1** and **2** (Fig. S6). The field dependence of the relaxation time for **2** (Fig. S7) could be modelled with the usual equation $\tau^{-1} = DH^4T + B_1/(1+B_2H^2) + K$ (Eq. 2), for which the first term accounts for the direct process (for Kramers-ion), the second one stands for the QTM, while the K constant accounts for the field-independent Raman and thermally activated processes (Table S4). For **1**, the optimum field could be estimated at 500 Oe. However, it could be noticed that the relaxation time is almost field-independent up to 2000 Oe and it was not possible to model the field dependence of the relaxation time which may be related to the presence of two dysprosium sites. This field-independent character may also suggest that magnetic interactions could shortcut in some extent the QTM as observed in other dinuclear complexes.¹⁷ On the contrary, Eq. 2 permits to nicely fit the τ vs. H curve for **2** showing an optimal magnetic field between 1000 and 2000 Oe (Fig. S7). The frequency dependence of the ac susceptibilities and related Cole-Cole plots under the corresponding optimal dc fields still show a single relaxation process for both complexes (Fig. S8-S9, Table S5-S6). The temperature dependence of τ was also modelled by taking into account an additional direct process: $\tau^{-1} = \tau_0^{-1} \exp(-\Delta/kT) + CT^m + AT^n$ (Eq. 4, Table 1, Fig. 3) giving the Δ values of 34 ± 1 and 30 ± 1 cm⁻¹ for **1** and **2**, respectively.

Further insights could be obtained by determining the direction of the anisotropic axes of the ground doublet in both complexes with the MAGELLAN package.³⁴ For the two dinuclear complexes in **1** and owing to the centrosymmetric character, the anisotropic axes of both Dy sites are found identical and are almost collinear to the two shortest Dy-O distances involving the terminal phenoxide moieties (Fig. S10) with a deviation of 8° from the shortest Dy1-O1 bond of 2.144(6) Å and 14° from the second shortest Dy1-O1A bond of 2.151(8) Å. The second dinuclear complex shows comparable deviation angle of 9.85 and 14.75°. Hence the electrostatic contributions are mainly dominated by these two negatively charged oxygens, while the bridging phenoxide oxygens located at longer distances (ranging from 2.311 to 2.375 Å) do not induce a strong deviation of the anisotropic axis. This results in the presence of an axial crystal-field suitable to stabilize the oblate electronic density of each Dy³⁺ sites⁷ and, probably with a non-detrimental or positive impact of the magnetic interactions between the two Dy³⁺ ions that can explain the appearance of a slow magnetic relaxation in zero-field. Nevertheless, the absence of an opening in the hysteresis loop at 1.8 K in our experimental conditions (Fig. S11) for **1**, suggests that the magnetic interactions between the Dy(III) ions are not strong enough to provide an exchange bias system that may greatly shortcut the zero-field QTM.

As regards **2**, the anisotropic axis is almost collinear to the two shortest Dy-O3 and Dy-O1 bonds located in *trans* position (distances of 2.131 and 2.144 Å, Fig. S10) with deviation angles of 6.63 and 7.23°. On the other hand, the equatorial oxygen O2 is located almost perpendicularly (O1-Dy-O2 = 97.8° and O3-Dy-O2 = 98.8°) with a relatively short distance of 2.164 Å. This explains the occurrence of a transverse component leading to a strong QTM and in turn the need to use a static dc field to reveal the maximum of χ'' . It could be however highlighted that despite the presence of relatively short Dy-O distances, the anisotropic barriers for **1** and **2** are found relatively weak, suggesting a major influence of the Raman relaxation process as deduced from the fitting of the temperature dependence of the relaxation time.

Moreover, phenolate-based ligands are also appropriate to implement luminescence properties in order to design luminescent SMMs.²⁶⁻²⁷ At room temperature and solid-state, both, **1** and **2** exhibit a broad ligand-based emission band located at with different emissions located at 615 and 550 nm, respectively (Fig. S12).

Table 1: Fit parameters of the temperature dependence of the relaxation time for **1** and **2**.

Compound	Δ (cm ⁻¹)	τ_0 (s)	m	C (s ⁻¹ .K ^{-m})	τ_{QTM} (s)	A (s ⁻¹ .K ⁻¹)
1 (0 Oe)	24 ± 5	(3 ± 2) × 10 ⁻⁴	4.8*	0.00184 ± 0.00004	0.08 ± 0.14	-
1 (500 Oe)	34 ± 1	(4.6 ± 0.5) × 10 ⁻⁶	6.5*	(6.4 ± 0.1) × 10 ⁻⁷	-	-
2 (1000 Oe)	30 ± 1	(2.7 ± 0.4) × 10 ⁻⁶	5*	0.0045 ± 0.0001	-	1 ± 1

* fixed parameter

Fig. 3. Temperature dependence of the relaxation time for **1** (0 and 500 Oe) and **2** (1000 Oe) using the ac susceptibility data. The solid line represents the fit with Eq. 1 (0 Oe) and dashed lines with Eq. 3.

In summary, we have described in this communication two dysprosium complexes using different phenoxide-based dyes. Thus, in the homoleptic dinuclear complexes **1**, the relatively short Dy-O distances in the axial position and a relatively wide O-Dy-O angle of 147-150° create an axial crystal-field for each Dy³⁺ site. This promising axiality and the non-deleterious impact of the magnetic interactions between the two Dy³⁺ are suitable to provide a zero-field slow relaxation of the magnetization observed for this compound and offers a nice example of a dinuclear Dy-based genuine SMM behaviour. On the other hand, the mononuclear complex shows only a field-induced SMM behaviour due to the presence of a phenolate ligand with the short Dy-O bond in the equatorial plane, which certainly decreases the axiality. In both cases, the influence of the Raman relaxation seems to be important and explain the presence of a relatively low energy barriers. Depending on the ligand's nature, a drastic change in the emission properties of the resulting complexes is observed. Consequently, the large tunability of such ligands in (steric and electronic) should provide facile experimental means to adjust and improve both the slow relaxation features and optical properties.

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Conflicts of interest

There are no conflicts to declare.

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