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# Alternate Synthetic Pathway Leading to Isolation of Dinuclear Single-Molecule Magnet

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Abstract: Using Schiff-base ligand, 1,5the Bis(salicylidene)carbohydrazide (H<sub>3</sub>bsc), an unique dinuclear [Dy<sub>2</sub>(µ-OH)(bsc) dysprosium(III) compound. (NO<sub>3</sub>)<sub>2</sub>(DMF)<sub>3</sub>(H<sub>2</sub>O)]·THF·2DMF (1.THF.2DMF), has been synthesized. The structural and magnetic properties are fully studied and single-molecule magnetic behaviour with an energy barrier (Ueff = 123 K) has been observed.

Single-Molecule Magnets (SMMs) observing superparamagnetlike behaviour generally result from the presence of large spin around state  $(S_7)$  and Ising-type magnetoanisotropy (D).<sup>1</sup> In this decade, pure lanthanide SMMs, in particular Dy<sup>III</sup>, have been intensively studied due to the large spin-orbit coupling, which increases the *D* value for the complex resulting in higher energy barriers.<sup>2</sup> The multinuclear complexes have been synthesized with nuclearity of the SMMs based on dysprosium(III) ranging from mononuclear to a Dy<sub>36</sub> cluster.<sup>3</sup> The high energy barrier of SMM might be governed by the interplay between the ligand field effect, the symmetry of metal ions and the hyperfine coupling and magnetic interaction.<sup>4</sup> In order to explore the real physical origin of this behaviour, the simplicity of the dinuclear lanthanide units has been applied for further evaluation. Complexes with different bridging motifs have been investigated due to the ability to probe magnetic interactions between lanthanide ions, such as N23-bridged Tb2 complex,5 phenoxide bridged  $Dy_2$  complex,<sup>6</sup> fluoro-bridged  $Dy_2$  complex<sup>7</sup> and sulfurbridged organometallic Dy<sub>2</sub> complexes.<sup>8</sup> Moreover, dinuclear complexes with two different local geometries of Dy<sup>III</sup> ions have also been synthesized. Asymmetric dinuclear molecules with two-step relaxation process of SMM properties are good examples for understanding the effect of D values.<sup>9,4e</sup> On the other hand, the presence of quantum tunneling of magnetization (QTM) negatively affects the magnetic bistability, which induces a reduction in the effective anisotropic barrier.<sup>10</sup> In pure lanthanide based SMM systems, the weak super-exchange interactions between the lanthanide ions can also be significant enough to suppress QTM.<sup>11</sup> With the aim to further understand lanthanide SMM system, more dinuclear complexes are necessary to be synthesized.

In our previous research, nonanuclear complex,  $[Dy_9(\mu_3\text{-}O)_4(\mu\text{-}OH)_6(hmc)_4(NO_3)_4(DMF)_4](OH)\cdot H_2O\cdot THF\cdot DMF$ 

 $(\mathbf{A}\cdot H_2 O \cdot THF \cdot DMF)$ , was synthesized by 1,5-Bis(2-hydroxy-3-methoxybenzylidene)carbonohydrazide (H<sub>2</sub>hmc), which has four potential coordination sites (Scheme 1, left). <sup>12</sup> In order to isolate

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the dinuclear unit, 1,5-Bis(salicylidene)carbohydrazide (H<sub>3</sub>bsc) was designed without the methoxyl group, which leads to only two potential coordination sites. An unexpected Dy<sub>6</sub> singlemolecule magnet, [Dy<sub>6</sub>(µ<sub>3</sub>-O)(bsc)<sub>3</sub>(CO<sub>3</sub>)<sub>3</sub>(MeOH)<sub>14</sub>(H<sub>2</sub>O)](OH)Cl<sub>3</sub>·2MeOH·2H<sub>2</sub>O (B-2MeOH-2H<sub>2</sub>O),<sup>13</sup> was synthesized, which exhibits slow relaxation of the magnetisation under zero applied dc field with a value of  $U_{eff}/k_B = 101.9$  K. The structure was assembled thereafter by three dinuclear units in a triangular arrangement, which were linked through CO<sub>3</sub><sup>2-</sup> bridges. With this in mind, we have focused our attention on synthesizing dinuclear or trinuclear units with the same ligand, H<sub>3</sub>bsc, whose magnetic property details may be further explored by simplifying the magnetic interaction or anisotropic axes alignments.



Dimethylformamide (DMF) was selected for the syntheses, since the coordination of the lanthanide ions in a polar aprotic solvent, such as DMSO or DMF, is a prevalent phenomenon that has been applied for crystallization due to its role as both terminal ligands and co-solvent.<sup>14</sup> Herein, the Dy<sup>III</sup><sub>2</sub> unit was successfully isolated by adding DMF in the solvent system. The details of magnetic properties have been studied, which show dinuclear complex exhibiting SMM properties with a value of  $U_{eff}/k_B = 123$  K.



Figure 1. Molecular structure of complex 1. Color code: Dy (yellow), O (red), N (blue), C(gray) and selected H(black). H atoms were omitted for clarity. The reaction conditions were described at the bottom.

## COMMUNICATION

To a solution of Dy(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O (0.50 mmol) in THF (15 ml) was added a solution of H<sub>3</sub>bsc (0.25 mmol) and ethylenediamine (EDA) (0.5 mmol) in DMF/THF (7 and 8 ml, respectively). Different dysprosium salts have been applied in the reactions, since no crystal of dinuclear complex was obtained by using DyCl<sub>3</sub>·6H<sub>2</sub>O which we selected before. The resulting pale yellow solution gave well-formed pale yellow crystals in 64% yield. The structure of the dinuclear complex, [Dy<sub>2</sub>(µ-OH)(bsc) (NO<sub>3</sub>)<sub>2</sub>(DMF)<sub>3</sub>(H<sub>2</sub>O)]·THF·2DMF (1·THF·2DMF), crystallized in the monoclinic  $P2_1/n$  space group (Figure 1). Since  $CO_3^{2^2}$  as a linker for dinuclear units was formed by strong base, EDA was selected as a weaker base. DMF was chose as a terminal solvent molecule to fill the coordination sites as well as to prevent the formation of a large metal cluster. Selected bond distances of two Dy<sup>III</sup> ions were shown in Table S1.

The pentadentate ligand coordinates to the Dy centers in the dinuclear unit by two designed coordination pockets via three O atoms (O2, O3 and O4) and two N atoms (N1 and N4). Two crystallographically independent DyIII ions are bridged by the enol group of the ligands and one µ-OH with a Dy1-O3-Dy2 and Dy1-O1-Dy2 angle of 110.62(15)° and 115.96(16)°, respectively. The Dy-Dy distance is 3.8422(4)Å. Both Dy<sup>III</sup> ions are eight coordinate and the remaining four coordination sites of each lanthanide ion are filled by one nitrate anion and two solvent molecules (O5 of the DMF molecule and O6 of the H<sub>2</sub>O for Dy1 as well as O10 and O11 of two DMF molecules for Dy2). Charge balance considerations for the molecule indicate that the ligand must have three negative charges resulting from the ligand being in the enol-form of two deprotonated phenoxides (O2 and O4) and one deprotonated enol (O3). This is surprising that complex 1 with enol form was different with the carbohydrazide derived Schiff base complexes A and B which were found to exhibit the keto form. The comparison of selected bond distances of ligand for complexes 1, A and B are presented in Scheme 2. Although the difference of bond distances of C2-N2 (1.324(7) Å) and C2-N3 (1.364(7) Å) in complex 1 are relatively insignificant, the difference (less than 0.04 Å) between double and single bonds of carbohydrazide derived ligands in 3d or 4f metal clusters were observed.<sup>15</sup> Moreover, difference of the central C-O distances of three complexes in Scheme 2,  $d_{C48-O8} = 1.260(15)$  Å,  $d_{C7-O3} = 1.276(11)$ Å, and  $d_{C2-O3} = 1.290(7)$  Å for complexes **B**, **A** and **1**, respectively, are also not very obvious. However, we can confirm the enol form of  $\ensuremath{\mathsf{bsc}}^{\ensuremath{\mathsf{3}^{-}}}$  with the ligands have been reported in its enol form with a  $d_{C-O} \le 1.29$  Å.<sup>1</sup>



Scheme 2. Top: Different deprotonated forms of keto and enol forms of H<sub>3</sub>bsc; Bottom: The selected bond distances of core structures of complexes 1 (right), A (middle) and B (left) in Å.

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Moreover, since the positions of terminal  $H_2O$  and DMF are very close (Dy1-O6 and Dy2-O11 bonds of 2.358(4) Å and 2.360(4) Å as well as O6-Dy1-Dy2 and O11-Dy2-Dy1 angles of 113.09(11)° and 110.13(11)°, respectively) and the bond distance differences of ligand as we mentioned above were insignificant, the bond distances and angles around the Dy center are also very close result in both Dy centers have similar coordination geometries. (Figure 2) The coordination geometries of Dy1 and Dy2 were both triangular dodecahedron with significant differences as determine by SHAPE 2.1. (Table S2) which could lead to different relaxations systems we mentioned below.



Figure 2. Top: The coordination polyhedra of two octa-coordinated Dy<sup>III</sup> ions. Bottom: The coordination polyhedral with molecular structure.



**Figure 3.** Temperature dependence of the  $\chi T$  product of complexes 1 at 1000 Oe. Inset: *M* vs. *HT*<sup>1</sup> plot at indicated temperatures for complex 1.

Direct current (dc) magnetic measurements were performed between 1.8 and 300 K, with an applied dc field of 1000 Oe. The temperature dependence of the  $\chi T$  product can be observed in Figure 3. At room temperature the observed  $\chi T$  value was 28.65 cm<sup>3</sup> K mol<sup>-1</sup> under 1000 Oe applied dc field. These values are consistent with the theoretical value of 28.34 cm<sup>3</sup> K mol<sup>-1</sup> for two non-interacting Dy<sup>III</sup> ions (<sup>6</sup>H<sub>15/2</sub>, *S* = 5/2, *L* = 5, *g* = 4/3,  $\chi T$  = 14.17 cm<sup>3</sup> K mol<sup>-1</sup>). The  $\chi T$  product displays a gradual decrease

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with temperature, until ~100 K where a more dramatic decrease is observed to 24.53 cm<sup>3</sup> K mol<sup>-1</sup> at 1.8 K, under 1000 Oe. The drop of  $\chi T$  at low temperature could be related to antiferromagnetic interactions, inherent magnetic anisotropy from Dy<sup>III</sup> ions, the Stark levels depopulation and/or the occurrence of SMM behavior.



**Figure 4.** Temperature dependence of the out-of-phase ( $\chi$ ") susceptibility of Complex **1** without applied dc field.

Isotherm magnetisation data was collected between 1.8 and 8 K (Figure S1). The *M* vs. *H* plot displays a rapid increase in the magnetisation at low magnetic fields, after which a gradual plateau beginning at 1 T forms. The *M* vs.  $HT^1$  data (Figure 3, inset) also displays that isotemperature lines to not overlay on a single master curve, thus further confirming the presence of non-negligible magnetic anisotropy and/or low-lying excited states. In order to assess the relaxation dynamics of the complex, alternating current (ac) susceptibility measurements were performed with an ac field of 3.5 Oe oscillating at frequencies up to 10000 Hz. We have measured the frequency-dependence of the in-phase ( $\chi'$ ) and out-of-phase ( $\chi''$ ) magnetic susceptibilities

under zero applied dc fields, where clear peaks are seen to shift to lower frequencies as the temperature is decreased, indicative of slow magnetic relaxation (Figure 4 and S2) The effective energy barrier and relaxation time were obtained through fitting the data of high temperature regime using the Arrhenius equation ( $\tau = \tau_0 \exp(U_{eff}/kT)$ ), which elicited a value of  $U_{eff} = 123$  K ( $\tau_0 = 7.60^{*}10^{-7}$  s), as can be seen in Figure S3. The presence of a full broad peak can be observed with a maximum located around 25 K at 1000 Hz without applied dc field which may result from two relaxation systems due to the different geometries and/or the direction of anisotropic axes on two Dy<sup>III</sup> ions. (Figure S4) Fitting the Cole–Cole plots (Figure S5) with a Debye model yields large values of the  $\alpha$  parameter ranging from 0.18 to 0.30 (Table S3), confirming the wide distribution of the relaxations.

Since the quantum tunneling of the magnetization (QTM) that can occur was suppressed by applying optimum dc fields, the find-field experiments were also performed.<sup>17</sup> (Figure S6) The addition of a static field was found to have only a little effect on the relaxation dynamics of **1**, and thus we may conclude that zero field quantum tunneling of the magnetization is not efficient

within this complex that the QTM is probably supressed by the exchange interaction between the Dy<sup>III</sup>.



**Figure 5.** Dinuclear core structures of complex **1** (top) and **B** (bottom) showing the direction of the anisotropy axes for all  $Dy^{III}$  ions. Atom labels are the same as cif files. Axes modelled using Magellan magnetic software.

In order to evaluate the anisotropies of complex 1, the alignment of the anisotropy axes was determined using Magellan magnetic software.<sup>18</sup> Electrostatic modeling of the lowest lying Kramers doublets for each Dy<sup>III</sup> ion reveals that the anisotropy axes of Dy<sup>III</sup> ions (Figure 5, top) are close to the Dy-O(phenoxide) bonds (The angle between the anisotropy axis of Dy2 and Dy2-O4 is 3.09°; the angle between the anisotropy axis of Dy1 and Dy1-O2 is  $3.11^{\circ}$  ). The broad peaks have been attributed to the anisotropic character of the coordination sphere around Dy<sup>III</sup> ions or different orientations of the anisotropic axes within multinuclear complexes.<sup>19</sup> Since the coordination environments of two Dy<sup>III</sup> ions are very similar, the possibility of the broad peaks observed in figure S4 may result from the non-parallel direction of the anisotropy axes of both Dy<sup>III</sup> ions. Moreover, comparing with the calculated anisotropic axes of complex B was shown in figure 5, bottom and S7. The directions of anisotropic axes between complexes 1 and B are slightly different due to the differences of the charge of the ligand and the bridging molecules.

In conclusion, we successfully synthesized the dinuclear Dy unit of Dy<sub>6</sub> single-molecule magnet by utilised DMF as a terminal solvent molecule which also exhibits SMM behavior with an anisotropic barrier of  $U_{eff}$  = 123 K ( $r_0$  = 7.6 x 10<sup>-7</sup> s). Since the coordination environments of two Dy<sup>III</sup> ions are similar which was not changed a lot by enol form of ligand, the non-parallel anisotropic axes directions have been also indicated the broad peak in high frequency are mainly result from anisotropic axes directions. This example demonstrated that the symmetry unit of multinuclear cluster may be also observed SMM properties which offered more information for understanding how to synthesize new SMMs with higher anisotropic barriers towards beneficial future applications.

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## COMMUNICATION

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Using the Schiff-base ligand, 1,5-Bis(salicylidene)carbohydrazide (H<sub>3</sub>bsc), a unique dinuclear dysprosium(III) compounds,  $[Dy_2(\mu - OH)(bsc)(NO_3)_2(DMF)_3(H_2O)] \cdot THF \cdot$ 2DMF, has been synthesized. The structural and magnetic properties are fully studied and single-molecule magnetic behaviour with an energy barrier has been observed.



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Page No. – Page No. Alternate Synthetic Pathway Leading to Isolation of Dinuclear Single-Molecule Magnet

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