

# Synthesis, structures, and magnetic properties of Fe<sub>4</sub>-Ln<sub>2</sub> (Ln = Tb, Ho, and Er) clusters with *N, N, N', N'*-tetrakis-(2-hydroxyethyl)ethylenediamine

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## ABSTRACT

Three isostructural heterometallic {Fe<sup>III</sup><sub>4</sub>Ln<sup>III</sup><sub>2</sub>} coordination clusters are reported with general formula; [Fe<sup>III</sup><sub>4</sub>Ln<sup>III</sup><sub>2</sub>(μ<sub>4</sub>-O)<sub>2</sub>(Hedte)<sub>2</sub>(piv)<sub>6</sub>(NO<sub>3</sub>)<sub>2</sub>]•2MeCN•2CH<sub>2</sub>Cl<sub>2</sub> {Ln = Tb(1), Ho(2) and Er(3), where Hedte = *N,N,N,N'*-tetrakis-(2-hydroxyethyl)ethylenediamine; piv = pivalic acid}. These clusters are obtained by reacting the FeCl<sub>2</sub>•4H<sub>2</sub>O, pivalic acid, Ln(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O, and H<sub>4</sub>edte in MeCN/CH<sub>2</sub>Cl<sub>2</sub>. X-ray measurements revealed that 1–3 clusters exhibit a “butterfly” core shape. Solid-state *dc*-magnetic susceptibility measurement indicates the ferromagnetic interactions in 1 while 2 and 3 demonstrates dominated antiferromagnetic interactions.

## 1. Introduction:

In the last decade, mixed metal 3*d*-4*f* clusters have become an increasing research field due to exciting structural motifs and diversity in applications and one of them is molecular nanomagnetism [1,2]. Since the first 3*d*-4*f*SMM, [Cu<sup>II</sup>Tb<sup>III</sup>] was reported in 2003 [3], attention has been devoted to synthesizing and characterizing various clusters having 3*d*-4*f* metal ions [2,4,5]. In the heterometallic class, multinuclear 3*d*-4*f* complexes are considered important fields due to their diversity in spin carriers which make them able to produce optimized materials with dominant properties that can deliver useful applications in nanomagnetism and material science [6–9]. Despite the fact, stimulating development is carried out concern to the theoretic and practical vision [10] but the construction of such types of clusters is not an easy task because *d* and *f* orbitals exhibit different electron clouds, coordination geometries, and coordination favors to donor atoms in a method of synthesis. A subcategory of these compounds is iron- and manganese-based clusters in which a special work has been turned to obtain Fe<sup>III</sup>-Ln<sup>III</sup> systems. These reported complexes exhibited remarkable magnetic properties [11–13]. Furthermore, types of 3*d*-4*f* clusters such as [Mn Ln] [5,14–20], [Cu Ln] [5,7,21,22], [Ni Ln] [5,23–25], and [Co Ln], [5,26–28] and Fe Ln [5,11–13,29–33] have been found in literature. In respect to the selection of the ligand, aminopolyalcohols

are a class of ligands with N- and O- donor coordination sites and seem ideal in 3*d*-4*f* based coordination chemistry. Furthermore, normally the nitrogen of aminoalcohols is preferred to coordinate with 3*d* metal ions while oxygen of alkoxy groups with 4*f* metal ions [5,34,35]. Along with *N*-substituted diethanolamine, the use of carboxylates as co-ligands seems to be more interesting as it has different types of coordination modes that deliver some interesting magnetic properties [36–38]. In literature, aminoalcohols have been used previously to construct 3*d*-4*f* polynuclear complexes but, edteH<sub>4</sub> which can be considered either as a double diethanolamine explored rarely in 3*d* [39–41] and 3*d*-4*f* in the nanomagnetism field of research [14,33,42], therefore, we have been explored this ligand previously in Fe-Ln (Ln = Y, Dy, and Gd) chemistry [33]. As a continuation of our previous work herein, we are presenting coordination compounds of remaining lanthanides (Tb, Ho, and Er) along with magnetic properties to know the effects of other lanthanides on magnetic properties.

## 2. Experimental

### 2.1. Materials and general methods

FTIR spectra were obtained on the Perkin Elmer Spectrum One spectrometer with KBr discs. Elemental analyses were done by using an

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Elementar Vario EL analyzer. The data for 1–3 were conducted at 100 K on a Bruker SMART Apex diffractometer with graphite-monochromated Mo-K $\alpha$  radiation. The structures of these compounds were solved through direct methods and refined by SHELXTL program suite [43]. Crystals for 3 are formed very small weakly-diffracting needles. No significant intensity could be evaluated at angles corresponding to a resolution better than 0.95 Å, and the dataset was truncated at this point. As a consequence of the low resolution of the dataset (and possibly also unresolved twinning), anisotropic refinement of many of the atoms of the ligands and lattice solvent molecules was problematic (and it proved necessary to apply rigid-bond restraints to the thermal parameters of many of these atoms). Magnetic susceptibility data were taken on Quantum Design model MPMS-XL instrument, in a 0.1 T applied magnetic field. Magnetization isotherms were collected at 2–8 K between 0 and 7.0 T. The sample holder correction was performed and Pascal's constants are used to evaluate the diamagnetism of the samples.

## 2.2. Preparation of complexes 1–3

### 2.2.1. [Fe<sup>III</sup><sub>4</sub>Tb<sub>2</sub>] (1)

FeCl<sub>2</sub>·4H<sub>2</sub>O (1.00 mmol, 0.20 g), Tb(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O (0.5 mmol, 0.23 g) and pivalic acid (5.00 mmol, 0.50 g) were dissolved in 20 mL of acetonitrile. To this stirred solution, added edteH<sub>4</sub> (1.00 mmol, 0.24 g) along with 10 mL of dichloromethane. The gained light red coloured solution was further stirred 1 h, and then stand this solution at room temperature for crystallization. After one day, light red crystals were gained, washed with acetonitrile. Yield: 31% (based on Fe). Anal. Calcd. for C<sub>56</sub>H<sub>104</sub>Cl<sub>4</sub>Fe<sub>4</sub>N<sub>8</sub>O<sub>28</sub>Tb<sub>2</sub>: C 33.29, H 5.19, N 5.54. Found: C, 33.11; H, 5.04; N, 5.41. IR data (cm<sup>-1</sup>): 3407 (w), 2960 (s), 2925 (m), 2867 (m), 1560 (vs), 1524 (m), 1484 (vs), 1460 (m), 1425 (vs), 1378 (m), 1361 (m), 1307 (m), 1228 (m), 1075 (m), 1033 (w), 929 (w), 897(m), 814 (w), 790 (w), 746 (w), 723 (w), 650 (m), 603 (m), 577 (m), 510 (s), 417 (w).

### 2.2.2. [Fe<sup>III</sup><sub>4</sub>Ho<sub>2</sub>] (2)

The synthesis was done by the same procedure to that of 1 but using Ho(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O Yield: 21% (based on Fe). Anal. Calcd. for C<sub>56</sub>H<sub>104</sub>Cl<sub>4</sub>Fe<sub>4</sub>Ho<sub>2</sub>N<sub>8</sub>O<sub>28</sub>: C, 33.09; H, 5.17; N, 5.51; Found: C, 33.02; H, 4.99; N 5.21. IR data (cm<sup>-1</sup>): 3420 (w), 2959 (s), 2919 (m), 2870 (m), 1561 (vs), 1528 (m), 1484 (vs), 1461 (m), 1425 (vs), 1389 (m), 1362 (m), 1311 (m), 1228 (m), 1075 (m), 1034 (w), 929 (w), 898(m), 814 (w), 790 (w), 750 (w), 722 (w), 650 (m), 603 (m), 578 (m), 512 (s), 419 (w).

### 2.2.3. [Fe<sup>III</sup><sub>4</sub>Er<sub>2</sub>] (3)

Same procedure was adopted for this synthesis but using Er(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O Yield: 25% (based on Fe). Anal. Calcd. for C<sub>56</sub>H<sub>104</sub>Cl<sub>4</sub>Er<sub>2</sub>Fe<sub>4</sub>N<sub>8</sub>O<sub>28</sub>: C, 33.02; H, 5.14; N, 5.50. Found: C, 32.95; H, 5.01; N, 5.33. IR data (cm<sup>-1</sup>): 3419 (w), 2960 (s), 2927 (m), 2869 (m), 1563 (vs), 1532 (m), 1484 (vs), 1459 (m), 1426 (vs), 1378 (m), 1361 (m), 1313 (m), 1228 (m), 1079 (m), 1029 (w), 928 (w), 899(m), 814 (w), 790 (w), 749 (w), 721 (w), 650 (m), 602 (m), 580 (m), 515 (s), 419 (w).

## 3. Results and discussion

Isostructural compounds 1–3 were obtained by the reaction of FeCl<sub>2</sub>·4H<sub>2</sub>O, Ln(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, piv, and H<sub>4</sub>edte in MeCN / CH<sub>2</sub>Cl<sub>2</sub>.

### 3.1. Crystal structures 1–3

Coordination clusters 1–3 are isomorphs with two independent (but isostructural) half molecules in the asymmetric unit. The X-ray data of these compounds are presented in Table 1 and bond distances are depicted in Table S1. The description of 1 is illustrated here (Fig. 1a and 1c) which is comparable to 2 and 3.

The core of the complex exhibits a [Fe<sup>III</sup><sub>4</sub>Tb<sub>2</sub>(μ<sub>4</sub>-O)<sub>2</sub>]<sup>14+</sup> framework and the four Fe<sup>III</sup> ions organized in a “butterfly” motif, and the Fe centers are coplanar. The geometry of terbium centers is (BTPR-8) biaugmented trigonal prism (Fig. 1b) confirmed by Continuous Shape Measures (CSM) (Table S2) implemented in Shape 2.1 [44].

In this structure, peripheral ligation is given by one nitrate and one pivalato ligand chelating Tb1, four pivalic acid exhibits syn, syn bridging mode between the Tb and the Fe centers, and two triply-deprotonated (Hedte)<sup>3-</sup> anions. Each Fe<sup>3+</sup> triangle, encompassing one wing- and two body-irons is attached to a capping Tb<sup>III</sup> via a μ<sub>4</sub>-O bridge. Thus O<sub>1</sub> bridges between Fe<sub>2</sub>, Fe<sub>1</sub> and Fe<sub>1</sub> [1-x,1-y,1-z] and to Dy<sub>1</sub>. Each Hedte<sup>3-</sup> chelates Fe<sub>2</sub> by using its two nitrogens and imparts three μ-alkoxo oxygen bridges Tb<sub>1</sub> to Fe<sub>1</sub> and Fe<sub>1</sub> [1-x,1-y,1-z], respectively. Each Hedte<sup>3-</sup> triply degenerates and the fourth arm remains protonated and produces an intermolecular hydrogen bond (Table S3) to generate 1D chain through pivalato oxygen of a neighboring molecule. The “Butterfly” central tetranuclear [Fe<sub>4</sub>O<sub>2</sub>]<sup>8+</sup> core has been previously observed in many homonuclear compounds [45–49]. The main difference between these compounds and those reported in this paper is that here O<sub>1</sub> forms a μ<sub>4</sub>-O bridge instead of μ<sub>3</sub>-O, with additional coordination to the lanthanide ion. Due to this additional coordination, O<sub>1</sub> is displaced by 0.987(4) Å out of the Fe<sub>2</sub>-Fe<sub>1</sub>-Fe<sub>1</sub> [1-x,1-y,1-z] plane, as

**Table 1**  
X-ray measurement data for 1–3.

	1	2	3
Molecular formula	C <sub>56</sub> H <sub>104</sub> Cl <sub>4</sub> Fe <sub>4</sub> N <sub>8</sub> O <sub>28</sub> Tb <sub>2</sub>	C <sub>56</sub> H <sub>104</sub> Cl <sub>4</sub> Fe <sub>4</sub> Ho <sub>2</sub> N <sub>8</sub> O <sub>28</sub>	C <sub>56</sub> H <sub>104</sub> Cl <sub>4</sub> Er <sub>2</sub> Fe <sub>4</sub> N <sub>8</sub> O <sub>28</sub>
Formula weight	2020.51	2032.53	2037.19
Crystal system	monoclinic	monoclinic	monoclinic
Space group	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n	P2 <sub>1</sub> /n
a/Å	22.1736(9)	22.1656(14)	22.1508(14)
b/Å	15.9444(6)	15.9174(10)	15.8778(10)
c/Å	24.0880(10)	23.9089(15)	23.8072(15)
β, °	107.326(1)	106.887(1)	106.863(1)
Volume/Å <sup>3</sup>	8129.8(6)	8071.8(9)	8013.1(9)
Z	4	4	4
ρ <sub>calc</sub> (g/cm <sup>3</sup> )	1.651	1.673	1.689
μ/mm <sup>-1</sup>	2.620	2.847	2.987
F(000)	4088	4104	4112
Reflections collected	47875	82337	32966
Independent reflections	15841 [R <sub>int</sub> = 0.0525 R <sub>sigma</sub> = 0.0630]	20830 [R <sub>int</sub> = 0.0643 R <sub>sigma</sub> = 0.0689]	9790 [R <sub>int</sub> = 0.0961 R <sub>sigma</sub> = 0.0907]
Data/restraints/parameters	15841/35/931	20830/40/930	9790/245/921
Goodness-of-fit on F <sup>2</sup>	1.065	1.024	1.028
Final R indexes [I > 2σ(I)]	R <sub>1</sub> = 0.0487, wR <sub>2</sub> = 0.1077	R <sub>1</sub> = 0.0537, wR <sub>2</sub> = 0.1385	R <sub>1</sub> = 0.0578, wR <sub>2</sub> = 0.1341
Final R indexes [all data]	R <sub>1</sub> = 0.0765, wR <sub>2</sub> = 0.1182	R <sub>1</sub> = 0.0832, wR <sub>2</sub> = 0.1501	R <sub>1</sub> = 0.1050, wR <sub>2</sub> = 0.1521
CCDC	2145538	2145539	2145540

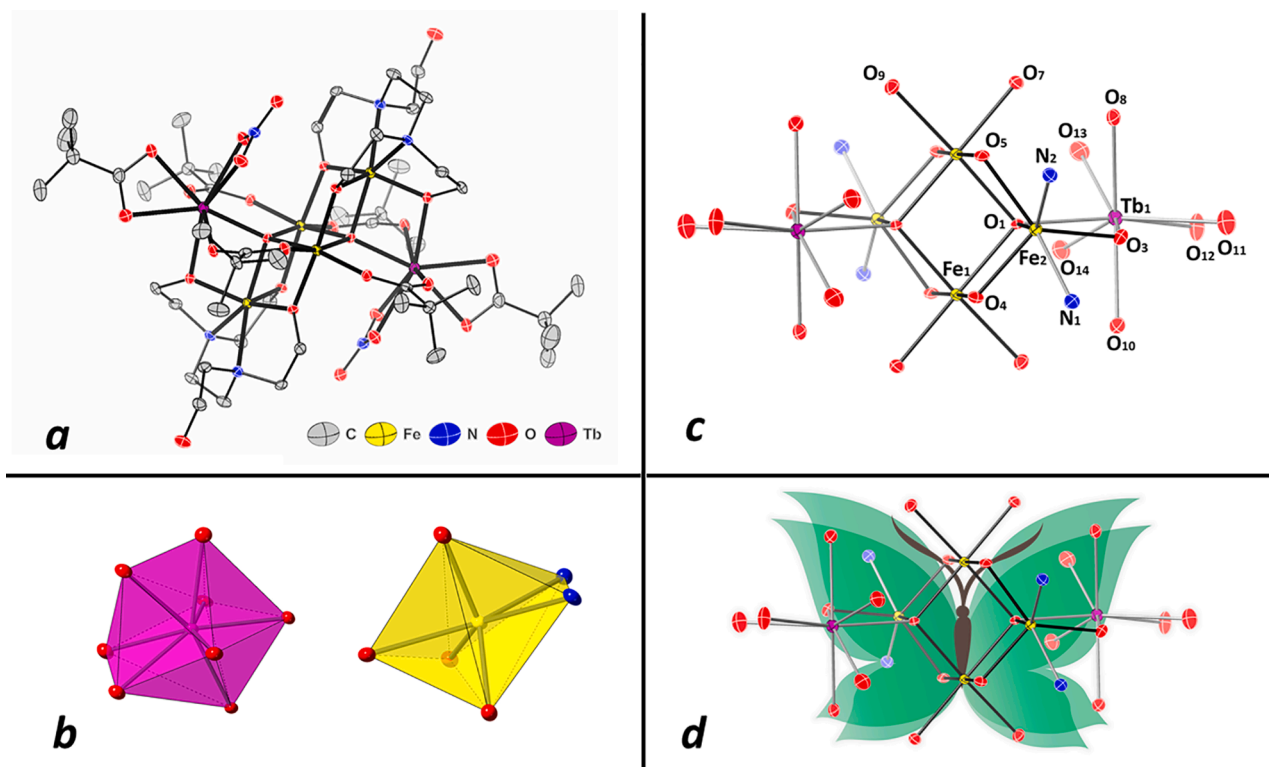


Fig. 1. (a) Thermal ellipsoid of **1** at 30%, (b) Polyhedral view for  $\text{TbO}_8$  (BTPr-8) and  $\text{FeN}_2\text{O}_4$  (distorted octahedron), (c) Labeled molecular structure for **1**, showing only the core and (d) Butterfly picture overlapping with the crystal structure of **1**.

opposed to being close to coplanar in the homonuclear systems. Furthermore, it is reflected in the sum of the Fe-O-Fe angles at  $\text{O}_1$ ,  $295.8^\circ$ , much smaller than in the previously reported  $\text{Fe}_4$  butterfly complexes ( $342\text{--}360^\circ$ ). The atom  $\text{O}_1$  lies closer,  $0.348(3) \text{ \AA}$  to its  $\text{Fe}_2\text{Tb}$  triangle than to the  $\text{Fe}_3$  triangle. The shortest intermolecular Tb-Tb distance is  $11.128 \text{ \AA}$ , and only few hydrogen bonding (Table S3) among other weak interactions are found in **1** (Figure S1). Therefore, the lanthanide centers can be considered as isolated from a magnetic point of view.

### 3.2. Magnetic measurements

The  $\chi_M T$  product for the hexanuclear heterometallic **1–3** compounds under an applied magnetic field of  $0.1 \text{ T}$  is demonstrated in (Fig. 2, left). In case of **1**, on lowering the temperature, the  $\chi_M T$  product of **1** first decreases progressively from  $36.33 \text{ cm}^3\text{Kmol}^{-1}$  at room temperature to  $26.44 \text{ cm}^3\text{Kmol}^{-1}$  around  $26 \text{ K}$  and then rises to a value of

$34.84 \text{ cm}^3\text{Kmol}^{-1}$  at  $3 \text{ K}$  and after a decrease to  $34.04 \text{ cm}^3\text{Kmol}^{-1}$  at  $1.8 \text{ K}$ . The value at room temperature is lower than the expected value of  $41.26 \text{ cm}^3\text{Kmol}^{-1}$  for non-interacting four  $\text{Fe}^{\text{III}}$  ( ${}^6\text{A}_{1g}$ ,  $S = 5/2$ ,  $g = 2$ ) and two  $\text{Tb}^{\text{III}}$  ( ${}^7\text{F}_6$ ,  $g = 3/2$ ) ions. The increasing slope of the  $\chi_M T$  dependence in the  $3\text{--}26 \text{ K}$  range of temperature and maximal values of  $\chi_M T$  then two uncoupled  $\text{Tb}^{\text{III}}$  ( $23.76 \text{ cm}^3\text{Kmol}^{-1}$ ,  ${}^7\text{F}_6$ ,  $g = 3/2$ ) ions indicate the ferromagnetic interaction, that is similar to the previously reported compounds;  $\text{Fe}_4\text{Gd}_2$  and  $\text{Fe}_4\text{Dy}_2$  [33]. The  $\chi_M T$  at room temperature is close to the sum of  $\text{Fe}_4$  and two uncoupled (noninteracted with  $\text{Fe}_4$  core) Tb ions ( $37.26 \text{ cm}^3\text{Kmol}^{-1}$ ) which indicated that the character of magnetic interactions in  $\text{Fe}_4$  core is also similar to  $\text{Fe}_4\text{Dy}_2$  and  $\text{Fe}_4\text{Y}_2$ . The lowering of  $\chi_M T$  below  $3 \text{ K}$  may be imputed to weak intermolecular interactions.

In **2** and **3** the  $\chi_M T$  product are lower ( $38.54$  and  $30.61 \text{ cm}^3\text{Kmol}^{-1}$ ) than the expected value of  $45.64$  and  $40.46 \text{ cm}^3\text{Kmol}^{-1}$  for non-interacting four  $\text{Fe}^{\text{III}}$  ( ${}^6\text{A}_{1g}$ ,  $S = 5/2$ ,  $g = 2$ ) and two  $\text{Ho}^{\text{III}}$  ( ${}^5\text{I}_8$ ,  $g = 5/4$ ) and two  $\text{Ho}^{\text{III}}$  ( ${}^4\text{I}_{15/2}$ ,  $g = 6/5$ ) ions. The low temperature  $\chi_M T$  products of

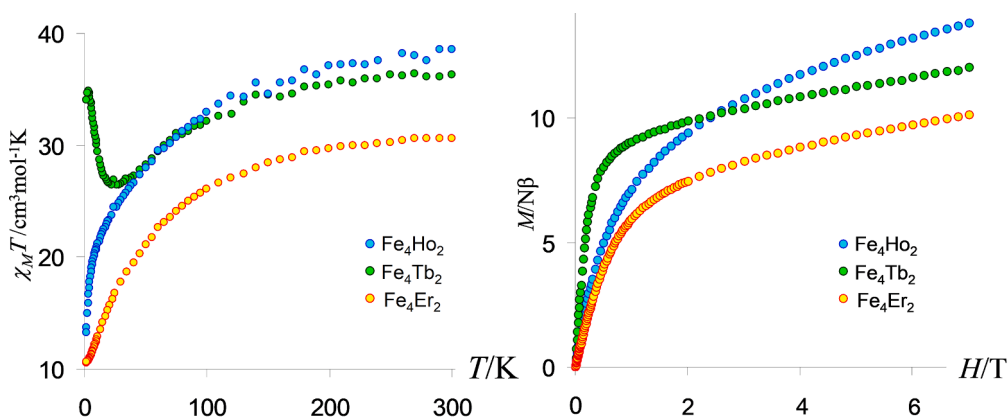


Fig. 2.  $\chi_M T$  versus  $T$  plots at  $0.1 \text{ T}$  and the magnetization  $M$  vs applied field  $H$  at  $2 \text{ K}$ ; for **1–3**.

2 and 3 are continuously decreasing to 13.26 and 10.64 cm<sup>3</sup>·K·mol<sup>-1</sup> which indicates the dominated antiferromagnetic interaction in these compounds. The shape of the curves showing the magnetization to the magnetic field is typical for the presence of anisotropy in 1–3 (Fig. 2, right).

#### 4. Conclusion

We have prepared three isostructural heterometallic {Fe<sup>III</sup><sub>4</sub>Ln<sup>III</sup><sub>2</sub>} complexes by using a mixed ligands approach; pivalic acid and edteH<sub>4</sub>. All these compounds contain a similar structure and central core; [Fe<sup>III</sup><sub>4</sub>Tb<sub>2</sub>(μ<sub>4</sub>-O)<sub>2</sub>]<sup>10+</sup> with the four Fe<sup>III</sup> ions are organized in the “butterfly” shape. Magnetic study shows that compound 1 displays ferromagnetic interactions but no ac signals were seen while compounds 2 and 3 reveal dominated antiferromagnetic interactions. The use of edteH<sub>4</sub> ligand will be useful for further work in the coordination chemistry of 3d-4f metal ions.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ica.2022.120920>.

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