Three lanthanide complexes with mixed salicylate and 1,10-phenanthroline: syntheses, crystal structures, and luminescent/magnetic properties

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ABSTRACT

Three new lanthanide complexes incorporating salicylate (HSA or SA) and 1,10-phenanthroline (phen), \(\text{Ln}_2(\text{HSA})_2(\text{SA})_2(\text{phen})_2\) [\(\text{Ln} = \text{Ho (1) and Er (2)}\)], and \(\text{Sm}_2(\text{HSA})_2(\text{SA})_2(\text{phen})_2\) (3), have been synthesized. X-ray structural analysis reveals that 1 and 2 are isostructural with a trinuclear pattern, and 3 exhibits a binuclear structure. Comparison of the structural differences between 1/2 and 3 suggests that the identity of metal plays an important role in construction of such complexes. The magnetic properties of 1 are discussed. Moreover, 2 and 3 are both photoluminescent materials, and their emission properties are closely related to their corresponding LnIII centers.

1. Introduction

Syntheses of lanthanide carboxylate materials attract attention due to their desirable structures [1] and potential applications arising from luminescence [2] and magnetic properties [3]. Organic ligands play a vital role in tuning the structural topology and functionality of such compounds [4]. The use of aromatic ligands is common as they are excellent in sensitizing the lanthanide luminescence by the well-known “antenna effect” [5]. A number of aromatic carboxylate ligands such as benzoate and modified benzoates are known [6]; salicylic acid (\(\text{H}_2\text{SA}\)), a type of aromatic carboxylate ligand with...
one carboxyl and one hydroxyl arranged in a 1,2-fashion around the central aromatic group, can form various coordination structures [7]. Carboxylate and hydroxy groups on the molecules can be partially or completely deprotonated to form versatile coordination models; the hydroxyl group may provide additional binding sites to generate more complicated structures in cooperation with the carboxyl groups. Furthermore, the phenyl can form C/O–H⋯O, C/O–H⋯π, and π⋯π interactions as steering forces in the control of molecular self-assembly. Chelating ligands such as 1,10-phenanthroline (phen) may inhibit expansion of the polymeric framework to give coordination polymers of low-dimensionality or zero-dimensional molecules [8].

Keeping these observations in mind, we have reported isomorphous DyIII and GdIII complexes with H₂SA and phen as ligands, in which a luminescent linear trinuclear DyIII complex exhibits slow magnetic relaxation of single-ion origin [7(d)]. These results indicate that H₂SA is an excellent ligand in the self-assembly of coordination complexes and further efforts are required to provide more information on its coordination behavior. As an extension of the above work, three new lanthanide complexes, Ln₃(HSA)₅(SA)₂(phen)₃ [Ln = Ho (1) and Er (2)], and Sm₂(HSA)₂(SA)₂(phen)₃ (3), based on HSA/SA and phen ligands were obtained. Herein, we report the syntheses, crystal structures, and luminescent properties of these complexes.

2. Experimental

2.1. Materials and general methods

All the starting reagents and solvents for synthesis were commercially available and used as received. Elemental analyses (C, H, and N) were performed on a Vario EL III Elementar analyzer. IR spectra were recorded from 4000 to 400 cm⁻¹ on a Tensor 27 OPUS (Bruker) FT-IR spectrometer with KBr pellets. Thermogravimetric analysis (TGA) experiments were carried out on a Perkin-Elmer diamond SII thermal analyzer from room temperature to 800 °C under nitrogen at a heating rate of 10 °C min⁻¹. The emission spectra in the visible region were tested on a F-7000 (HITACHI) spectrophotometer and those in near-infrared region were measured on an FLS-980 fluorescence spectrophotometer.

2.2. Synthesis of 1–3

All three complexes were prepared with similar methods. A general synthetic procedure is described as follows by 1 as an example.

2.2.1. Ho₃(HSA)₅(SA)₂(phen)₃ (1)
A mixture of Ho₂O₃ (0.2 mmol, 0.076 g), H₂SA (1.5 mmol, 0.207 g), phen (0.4 mmol, 0.079 g), and H₂O (15 mL) was placed in a Teflon-lined stainless steel vessel (23 mL), heated to 140 °C for 72 h, and then cooled to room temperature at a rate of 5 °C h⁻¹. Single crystals of 1–3 suitable for X-ray diffraction were obtained. Then, the obtained crystals were collected by filtration, washed with water and ethanol, and dried in air. Yield: 0.25 g (60% based on H₂SA). Elemental Anal. Calcd for C₈₅H₅₇Ho₃n₆O₂₁: C, 51.22; H, 2.88; N, 4.22. Found: C, 51.31; H, 2.75; N, 4.39%. The IR spectrum for 1 is shown in figure S1a.

2.2.2. Er₃(HSA)₅(SA)₂(phen)₃ (2)
The same synthetic method as that for 1 was used except that Ho₂O₃ was replaced by Er₂O₃ (0.2 mmol, 0.077 g). Yield: 0.24 g (55% based on H₂SA). Elemental Anal. Calcd for C₈₅H₅₇Er₃n₆O₂₁: C, 51.04; H, 2.87; N, 4.20. Found: C, 51.33; H, 2.95; N, 4.31%. The IR spectrum for 2 is shown in figure S1b.

2.2.3. Sm₂(HSA)₂(SA)₂(phen)₃ (3)
The same synthetic method as that for 1 was used except that Ho₂O₃ was replaced by Sm₂O₃ (0.2 mmol, 0.070 g). Yield: 0.31 g (60% based on H₂SA). Elemental Anal. Calcd for C₃₂H₁₉n₆O₅Pr: C, 55.39; H, 3.05; N, 6.06. Found: C, 55.21; H, 3.28; N, 6.29%. The IR spectrum for 3 is shown in figure S1c.
2.3. Crystal structure determinations of 1–3

X-ray single-crystal diffraction data for 1–3 were collected on an Xcalibur Gemini Eos CCD diffractometer at 294(2) K with Mo-Kα radiation (\(\lambda = 0.71073 \text{ Å}\)) by \(\omega\) scan mode. The program SAINT [9] was used for integration of the diffraction profiles. Semi-empirical absorption corrections were applied using SADABS [10]. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods with SHELXL [11]. Metal ions in each complex were located from the \(E\)-maps and other non-hydrogen atoms were located in successive difference Fourier syntheses and refined with anisotropic thermal parameters on \(F^2\). Hydrogens were geometrically positioned and refined using a riding model. Further, crystallographic data and structural refinement details for 1–3 are summarized in table 1. Selected bond lengths and angles are listed in tables S1–S3.

| Chemical formula | \(\text{C}_{85\text{H}}\text{Ho}_3\text{N}_0\text{O}_{21}\) | \(\text{C}_{85\text{H}}\text{Er}_3\text{N}_0\text{O}_{21}\) | \(\text{C}_{64\text{H}}\text{N}_0\text{O}_{12}\text{Sm}_2\) |
|------------------|---------------------------------|---------------------------------|---------------------------------|
| Formula mass     | 1993.16                         | 2000.15                         | 1387.74                         |
| Crystal system   | Monoclinic                      | Monoclinic                      | Monoclinic                      |
| Space group      | \(P2_1/c\)                      | \(P2_1/c\)                      | \(P2_1/c\)                      |
| \(a (\text{Å})\) | 14.2446(2)                     | 14.2353(3)                     | 10.9077(4)                     |
| \(b (\text{Å})\) | 23.2892(4)                     | 23.2985(5)                     | 28.5413(11)                    |
| \(c (\text{Å})\) | 23.7871(4)                     | 23.7963(6)                     | 18.3989(5)                     |
| \(\beta (^\circ)\) | 103.462(2)                     | 103.481(2)                     | 104.110                         |
| \(V (\text{Å}^3)\) | 7674.4(2)                      | 7674.9(3)                      | 5555.1(3)                      |
| \(t (\text{K})\) | 294(2)                         | 294(2)                         | 294(2)                         |
| \(Z\)            | 4                              | 4                              | 4                              |
| \(\mu (\text{mm}^{-1})\) | 3.144                     | 3.331                         | 2.165                          |
| \(R_p^{\text{H}} (I > 2\sigma(I))\) | 0.0245                    | 0.0277                         | 0.0509                         |
| \(R_p (\text{all data})\) | 0.0247                     | 0.0239                         | 0.0450                         |
| \(wR_p (\text{all data})\) | 0.0548                     | 0.0447                         | 0.1126                         |
| GOF              | 0.925                         | 0.827                         | 1.034                         |

\(R_p = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\).
\(wR_p = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2)^{1/2}\).

2.4. Magnetic measurements

Magnetic measurement for 1 was carried out in the Unitat de Mesures Magnètiques (Universitat de Barcelona) on polycrystalline samples (ca. 30 mg) with a Quantum Design SQUID MPMS-XL magnetometer equipped with a 5 T magnet. Diamagnetic corrections were calculated using Pascal’s constant and an experimental correction for the sample holder was applied.

3. Results and discussion

3.1. Descriptions of crystal structures

3.1.1. \(\text{Ho}_3(\text{HSA})_5(\text{SA})_2(\text{phen})_3\) (1) and \(\text{Er}_3(\text{HSA})_5(\text{SA})_2(\text{phen})_3\) (2)

Single-crystal X-ray structure analyses revealed that 1 and 2 are isostructural (figures 1 and S4); therefore, 1 is taken as an example to discuss in detail. Complex 1 crystallized in the monoclinic system with a \(P2_1/c\) space group. The asymmetric unit consists of three crystallographically unique HoIII ions, five HSA ligands, two SA ligands, and three phen ligands. Three HoIII centers adopt a nearly linear arrangement (figure 1) as confirmed by the Ho1–Ho3–Ho2 angle of 165.29(5)° and are all eight-coordinate, but surrounded by different donors (figure S2 and table S1).
O11, and O12) from four HSA/SA ligands and two nitrogens (N1 and N2) from one phen. For Ho2, the polyhedron is defined by four oxygens (O13, O17, O18, and O19) from three distinct HSA/SA ligands and four nitrogens (N3, N4, N5 and N6) from two phen ligands. Ho3 is coordinated to eight oxygens (O2, O8, O10, O11, O14, O16, O17, and O20) from six distinct HSA/SA ligands. Though all three ions show coordination geometries of a distorted triangular dodecahedron, they are slightly different. Ho3 is in a distorted triangular dodecahedron, while Ho1 and Ho2 are in different distorted bicapped trigonal...
prism geometries (figure S2). All bond lengths around the metal are comparable to those found in other reported HoIII complexes [12]. In the structure of 1, there are three types of coordination modes for the HSA/SA ligands: a $\mu_2$–$\mu_1$ : $\mu_1$ double monodentate bridging mode (scheme 1(A)), a $\mu_1$–$\mu_1$ : $\mu_1$ bidentate chelating mode (scheme 1(B)) and a $\mu_2$–$\mu_1$ : $\mu_2$ bridging mode linking two lanthanide ions (scheme 1(C)). Adjacent Ho ions are linked via the $\mu_2$-bridging carboxylate groups of HSA/SA to generate trinuclear building units. The structure of 1 contains numerous C–H⋯O hydrogen bonds and C–H⋯π supramolecular interactions (table S4), which link such Ho$_3$ motifs into a 1-D (figure 1(b)), 2-D (figure S3) and further an overall 3-D supramolecular framework (figure 1(c)).

3.1.2. Sm$_2$(HSA)$_2$(SA)$_2$(phen)$_3$
X-ray diffraction analysis revealed that the asymmetric unit of 3 consists of two crystallographically unique Sm ions, two HSA ligands, and two SA ligands. The coordination environment of the central Sm$^{III}$ is shown in figure 2(a) with atom numbering. The two Sm$^{III}$ ions are nine-coordinate but surrounded by different donors (figure S5). Sm1 is coordinated by five oxygens (O1, O3, O4, O5, and O7) from three HSA/AS ligands and four nitrogens (N1, N2, N3 and N4) from two phen ligands to form distorted tricapped trigonal prism geometry. Sm2 has seven oxygens (O1, O2, O4, O6, O8, O10, and O11) from four HSA/AS ligands and two nitrogens (N5 and N6) from one phen to form a distorted monocapped square antiprism (figure S5). All Sm–O and Sm–N bond lengths are comparable to those reported earlier for Sm complexes [13]. In the structure of 3, there are three types of coordination modes for the HSA/AS ligand as in 1: a $\mu_2$–$\mu_1$ : $\mu_1$ double monodentate bridging mode (scheme 1(A)), a $\mu_1$–$\mu_1$ : $\mu_1$ bidentate chelating mode (scheme 1(B)) and a $\mu_2$–$\mu_1$ : $\mu_2$ bridging mode (scheme 1(C)). Adjacent Sm ions are linked via $\mu_2$-bridging carboxylate groups of the HSA/AS ligands to generate a binuclear building unit. The binuclear units are further connected through C–H⋯π supramolecular interactions (table S5) to form a 2-D network structure along the $ab$ plane (figure 2(b)), extending to a 3-D supramolecular structure (figure 2(c)).

In comparison, the lanthanides in 1 and 2 are eight-coordinate, in which Ho1 and Ho2 have a distorted bicapped trigonal prism geometry, while Ho3 is a distorted triangular dodecahedron, but nine-coordinate in 3, in which the Sm1 has a distorted tricapped trigonal prism geometry, while Sm2 has a distorted monocapped square antiprism geometry. Complexes 1–3 contain the same coordination mode of the HSA/SA ligand. In 1 and 2, the $\mu_2$-bridging carboxylate groups of HSA/SA ligands linked the adjacent Ho ions to generate a trinuclear building unit. With respect to 3, adjacent Sm centers are linked to form a binuclear building unit. Comparison of the structural differences between 1/2 and 3 suggests that the identity of metal plays an important role in the construction of such complexes. The structures all contain numerous weak interactions, C–H⋯O hydrogen bonding interactions or/and C–H⋯π supramolecular interactions which further connect the trinuclear units of 1 and 2 as well as binuclear units of 3 to lead to the final 3-D supramolecular networks.
3.2. **X-ray powder diffraction results**

To confirm whether the crystal structures are truly representative of the bulk materials, X-ray powder diffraction (XRPD) experiments were carried out. The XRPD experimental and computer-simulated patterns of the corresponding complexes are shown in figure S6 in the Supporting Information. The bulk synthesized materials and the as-grown crystals are homogeneous for 1–3.

![Figure 2](image-url). Views of 3: (a) the unit structure, (b) the 2-D network, and (c) the 3-D supramolecular structure via C–H⋯π supramolecular interactions (black dashed lines).
3.3. Thermogravimetric analysis

To examine the thermal stabilities of 1–3, TGA experiments were performed (see figure S7). 1 is stable to ca. 200 °C. After that, the weight loss is sharp, indicating decomposition of organic ligands and collapse of the framework. Similar thermogravimetric traces with decomposition temperatures of 200–600 °C are observed for 2, suggesting that this complex possesses the same components and structure as 1 except for the different central lanthanide. With respect to 3, it is thermally stable to ca. 220 °C. Upon further heating, pyrolysis of the organic ligands occurs, which does not stop before the heating ends at 900 °C.

3.4. Magnetic properties

Magnetic susceptibility data for a crushed crystalline sample of 1 were collected at an applied field of 0.3 T from 2–300 K. The data are shown in figure 3(a) as a $\chi T$ versus $T$ plot (white circles). The $\chi T$ product has a value of 43 cm$^3$ K mol$^{-1}$ at 300 K, in agreement with the expected value for three isolated Ho(III) ions ($^5I_8$, $S = 2$, $L = 6$, $J = 8$ and $g_J = 10/8$). As temperature decreases, so does the $\chi T$ product, until below 50 K a sharp decrease to a $\chi T$ value of 23 cm$^3$ K mol$^{-1}$ is observed, indicating depopulation of the excited

![Figure 3](image-url)
stark sublevels. Magnetization versus field data for 1 are shown in figure 3(b) as a M/Nμ\textsubscript{B} versus field plot. The curves are typical of lanthanide ions with strong spin–orbit coupling, as expected for Ho(III) complexes. The data eventually reach 16.1 μ\textsubscript{B} at 1.8 K and 5 T. This value is lower than the expected saturation value for three Ho(III) ions, likely due to anisotropy and important crystal-field effects [14] at the Ln\textsuperscript{III} ion that eliminate the degeneracy of the ground state [15].

### 3.5. Luminescent properties

The luminescence spectra of 2 and 3 were investigated in the solid state at room temperature and exhibit clear emission spectra of the corresponding Er\textsuperscript{III} and Sm\textsuperscript{III} ions as shown in figure 4. For 2, the photoluminescent spectrum of Er\textsuperscript{III} in the near-infrared region was tested using excitation vis UV radiation. The Er\textsuperscript{III} complex displays an emission band at 1540 nm under excitation at 340 nm (figure 4).

![Solid-state emission spectra of (a) 2 and (b) 3.](image-url)
4(a)), and should be attributed to the transition of $^4I_{13/2} \rightarrow ^4I_{15/2}$ [16]. In the case of 3, under excitation at 340 nm, the spectrum exhibits four emission peaks at 565, 599, 646, and 701 nm (assignable to the $^4G_{5/2} \rightarrow ^4H_{j}$ ($j = 5/2, 7/2, 9/2, 11/2$) transitions), characteristic of Sm$^{III}$ and consistent with literature reports (figure 4(b)) [5(b), 17].

4. Conclusion

We synthesized three lanthanide complexes incorporating mixed salicylate and phen ligands, $\text{Ln}_3(\text{HSA})_2(\text{SA})_3(\text{phen})_3$ ($\text{Ln} = \text{Ho (1)}$ and Er (2)), and $\text{Sm}_2(\text{HSA})_2(\text{SA})_2(\text{phen})_3$ (3). Complexes 1 and 2 are isostructural with a trinuclear pattern, whereas 3 displays a binuclear structure. Comparison of the structural differences between 1/2 and 3 suggests that the identity of the metal plays an important role in such complexes. The magnetic properties of 1 were investigated and it exhibits the typical character of Ho ion. The Sm$^{III}$ complex shows luminescence in the visible region at excitation and the Er$^{III}$ complex displays its characteristic luminescence in the near-infrared region. Further work on the $\text{H}_2$SA-based coordination polymers with other rare-earth ions is underway in our laboratory for developing more interesting functional coordination polymers.

Disclosure statement

No potential conflict of interest was reported by the authors.

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