



Crystal Structure and Luminescence Properties of a New Two-Dimensional Gd(III) Complex

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Abstract

Gd(III) ions display fascinating luminescence properties. Therefore, a new two dimensional polymeric Gd(III) complex, $\{[\text{Gd}(2\text{-stp})\cdot 3(\text{H}_2\text{O})]\cdot (\text{H}_2\text{O})\}_n$, [2-stp = 2-sulfoterephthalate] has been synthesized under hydrothermal conditions and characterized by elemental analysis, FT-IR, UV–visible and single-crystal X-ray diffraction and solid state photoluminescence measurements. The crystal structure of the Gd complex shows that Gd atom is coordinated to eight oxygen atoms by four symmetry-related 2-stp ligands and three coordinated water molecules to form a distorted square-antiprismatic geometry. The 2-stp ligand coordinates to four different Gd atoms and acts as a μ_4 -bridging ligand, end up with a two-dimensional layer structure. Moreover, 2D layer structure with hydrogen bonding interactions may develop the decisiveness of the crystal structure of Gd complex and achieve a 3D architecture. In addition, the solid state photoluminescence spectra show that Gd complex exhibited a strong green emission when it is excited under UV light at 349 nm.

Keywords Gd complex · 2D polymer · Luminescence · Structural analysis

Introduction

In recent years, the construction of the lanthanide coordination complexes has become popular due to its widespread use in gas storage and separation, catalysis, luminescent and magnetic properties with various topological networks, versatile architectures [1–11]. The luminescence properties of lanthanide elements depend on the narrow emission and high color purity produced by these ions [12], hence, luminescent lanthanide complexes have important potential applications in fluorescence and electroluminescent devices, and as fluorescence probes and labels in various biological systems [13–21]. The relationship between the energies of the ligand triplet level and the emission levels of the lanthanide ions depends on the presence and intensity of the lanthanide-localized

luminescence bands in the emission spectrum [22, 23]. Due to their easily identified and emitted strong green light, Gd(III) complexes have potential technology applications for green light emitting OLEDs and photoactive materials [24, 25].

In this article, 2-sulfoterephthalate (2-stp) ligand has been selected because this ligand has two –COOH and one –SO₃H potentially coordinating groups which causes various coordination modes in different lanthanide complexes [26]. In addition, the sulfonate group has a very different coordination ability compared to the carboxylate group, so 2-stp ligand is quite suitable for creating new versatile networks. In the Cambridge Structured Databases (CSD version 5.39, November 2017 updates), only 5 different coordination modes for 2-stp were observed in all lanthanide complexes in which the 2-stp ligand was used. In recent communications, our research group and others have studied the synthesis, structure, magnetic and luminescence properties of some Ln(III) complexes with 2-stp ligand [27–31]. In this context, in view of the importance luminescence properties of Gd(III) complexes and in an effort to enlarge the library of such complexes, the synthesis of a new 2D polymeric Gd(III) complex along with single-crystal X-ray diffraction, FT-IR, solid state UV–visible and photoluminescence studies is presented here. To the best of

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our knowledge, the solid state photoluminescence properties of the Gd(III) complex containing 2-stp ligand is described here for the first time.

Experimental Section

Materials, Physical Measurements and X-ray Structure Determination

All chemicals and solvents were purchased from TCI America or Sigma-Aldrich. Elemental (C, H) analyses were carried out with a LECO, CHNS-932 analyzers. The FT-IR spectra were measured with a Perkin-Elmer Spectrum 65 instrument (4000–600 cm^{-1}). The solid state UV–visible spectra were determined by Ocean Optics Maya 2000Pro Spectrometer (300–600 nm). PXRD measurements were recorded on a Bruker-AXS D8-Advance diffractometer by using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) in the range $5^\circ < 2\theta < 50^\circ$ in θ – θ mode with a step ns ($5 \text{ s} < n < 10 \text{ s}$) and step width of 0.03° . The room temperature solid state luminescence spectra in the visible region were measured using an ANDOR SR500i-BL Photoluminescence Spectrometer, which equipped with a triple grating and an air-cooled CCD camera as detector for UV– and visible region. The luminescence measurements were done using the excitation source (349 nm) of a Spectra-Physics Nd:YLF laser with a 5 ns pulse width and 1.3 mJ of energy per pulse as the source.

Single crystal X-ray data was collected on a Xcalibur, Eos diffractometer using $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) at room temperature. The structure was solved by direct methods with SHELXS [32] and refined by full-matrix least-squares based on $|F_{\text{obs}}|^2$ with SHELXL [32], a via the Olex2 [33]. The non-hydrogen atoms were refined as anisotropic and the hydrogen atoms were generated using idealized geometry which were made to “ride” on their parent atoms and used in the structure factor calculations. Details of the supramolecular π -interactions were calculated PLATON 1.17 program [34]. Molecular drawings were obtained using MERCURY [35].

Synthesis

Gd(III) complex was synthesized from mixture of $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.1 mmol, 0.0451 g) and 2-sulfoterephthalic acid monosodium salt (0.1 mmol, 0.0268 g) in 40 ml distilled water. The mixture was stirred for 1 h at room temperature and sealed into a bomb equipped with a Teflon liner (45 ml) and then heated at 140°C for 3 days. The final pH value of this reaction media was close to 4.0. The resultant single crystals were collected and washed with distilled water. Analysis calculated for $\text{C}_8\text{H}_{11}\text{GdO}_{11}\text{S}$

(yield 65%): C 20.34, H 2.35%. Found: C 20.39, H 2.32%. For the ligand (2-stp); IR (cm^{-1}): $\nu(\text{COOH}) = 1792$, $\nu(\text{SO}_3\text{H}) = 1692$. UV–Vis: $\lambda_{\text{max}}/\text{nm}$: 374. For the Gd(III) complex; IR (cm^{-1}): $\nu_{\text{as}}(\text{COO}^-) = 1560$, $\nu_{\text{s}}(\text{COO}^-) = 1394$, $\nu(\text{O-H}) = 3510$. UV–Vis: $\lambda_{\text{max}}/\text{nm}$: 352, 438.

Results and Discussion

Crystal Structure Description

Single crystal X-ray diffraction analysis reveals that Gd(III) complex crystallize in monoclinic system with space group $\text{P2}_1/\text{n}$, forming a two-dimensional coordination polymer (Table 1). The asymmetric unit compose of one Gd(III) ion, one 2-stp ligand, three coordinated and one lattice water molecules. The crystal structure of the complex with the atomic labelling is shown in Fig. 1. Gd atom is coordinated to eight oxygen atoms by four symmetry-related 2-stp ligands and three coordinated water molecules to form a distorted square-antiprismatic geometry. The Gd–O bond distances are in the range of 2.307(5)–2.763(5) \AA , and the O–Gd–O angles are in the range of $50.64(14)^\circ$ – $147.26(18)^\circ$. All angles and bond distance can be compare with similar structures [36, 37] (Table 2).

The 2-stp ligand is deprotonated and utilizes the carboxylate and sulfonate groups to coordinated four Gd atoms (Scheme 1). The carboxylate groups (O8–C8–O9 and O3–C1–O4) adopt a bridging-bidentate modes, in which O8 and O9 bind to the same Gd atom, O3 and O4 atoms bind to two different Gd atoms, respectively. Furthermore, the sulfonate group functions as a μ_2 -bridging dentate mode, in which O5 and O7 atoms coordinated to two different Gd atoms. The 2-stp ligand coordinates to four different Gd atoms and acts as a μ_4 -bridging ligand, end up with a two-dimensional layer structure (Fig. 2a). Moreover, 2D layer structure with hydrogen bonding interactions may develop the decisiveness of the solid-state structure of Gd(III) complex and achieve a three-dimensional architecture (Fig. 2b and Table 3).

Before proceeding to the spectroscopic and photoluminescence studies, we note that experimental X-ray powder diffraction traces for Gd(III) complex are well compatible with those of simulated traces on the basis of single crystal structure of the complex (Figure S1).

Photophysical Properties

UV–Visible Spectra in Solid State

Figure 3 demonstrates the solid state UV–Visible spectra of the ligand (2-stp) and Gd(III) complex. The absorption spectra of the Gd(III) complex exhibited different

Fig. 1 The molecular structure of Gd(III) complex. Lattice water molecule is omitted for clarity

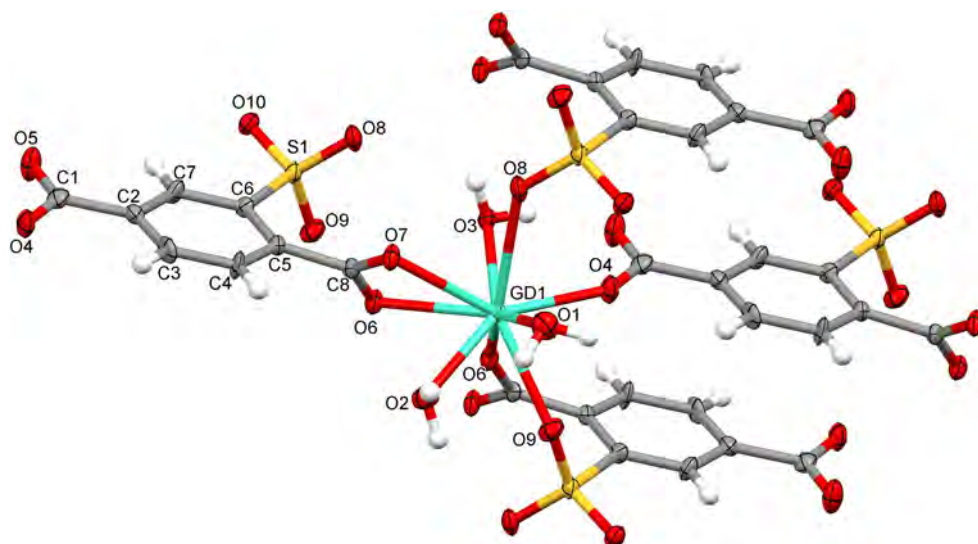


Table 1 Details of the data collection and refinement parameters for Gd(III) complex

Empirical formula	C ₈ H ₁₁ GdO ₁₁ S
Formula weight	472.48
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> /Å	6.9597(3)
<i>b</i> /Å	15.4994(7)
<i>c</i> /Å	11.4449(4)
α /°	90
β /°	98.198(2)
γ /°	90
Volume/Å ³	1221.96(10)
<i>Z</i>	4
ρ_{calc} g/cm ³	2.568
μ /mm ⁻¹	5.659
Θ range for data collection/°	6.37–52.742
Index ranges	– 8 ≤ <i>h</i> ≤ 8, – 17 ≤ <i>k</i> ≤ 19, – 14 ≤ <i>l</i> ≤ 7
Reflections collected	4866
Independent reflections	2493
Data/restraints/parameters	2493/0/196
Goodness-of-fit on F ²	1.072
Final R indexes [<i>I</i> ≥ 2σ (<i>I</i>)]	R ₁ = 0.0374, wR ₂ = 0.0944

absorption patterns as compared to the ligand. Two broad absorption bands were detected at 352 and 438 nm in the spectrum of Gd(III) complex while a broad absorption band comes out at $\lambda_{\text{max}} = 374$ nm for free ligand 2-stp, which may be corresponding to the π – π^* transitions of the ligand [38, 39]. The shifting of the absorption bands in the spectra of the complex means the Gd(III) ion coordination with the ligand (2-stp) [40].

Photoluminescence Properties in Solid State

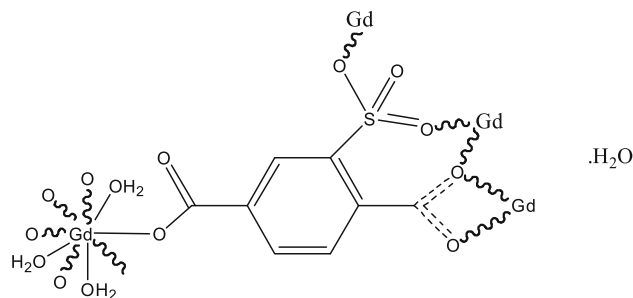
The solid-state photoluminescence features of the ligand (2-stp) and Gd(III) complex were examined at room temperature in the visible region upon excitation at $\lambda_{\text{ex}} = 349$ nm (Fig. 4). The free ligand displays three emission peaks at $\lambda_{\text{max}} = 479, 512$ and 557 nm which may be corresponding to the $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ intra-ligand charge transfer (ILCT) [41, 42]. As seen in Fig. 4, Gd(III) complex has a broad green emission band at 493 nm, the same

Table 2 Selected bond distance (Å) and bond angles (°) for Gd(III) complex

Gd1–O1	2.422 (5)	Gd1–O6 ⁱⁱ	2.598 (5)
Gd1–O2	2.431 (5)	Gd1–O7 ⁱⁱ	2.473 (5)
Gd1–O3	2.373 (4)	Gd1–O8 ⁱⁱⁱ	2.725 (5)
Gd1–O4	2.307 (5)	Gd1–O9 ⁱ	2.463 (5)
Gd1–O6 ⁱ	2.368 (5)		
O1–Gd1–O2	72.84 (18)	O4–Gd1–O6 ⁱ	82.48 (17)
O1–Gd1–O6 ⁱⁱ	136.86 (17)	O4–Gd1–O6 ⁱⁱ	136.18 (15)
O1–Gd1–O7 ⁱⁱ	91.04 (17)	O4–Gd1–O7 ⁱⁱ	141.92 (17)
O1–Gd1–O8 ⁱⁱⁱ	66.96 (16)	O4–Gd1–O8 ⁱⁱⁱ	79.01 (17)
O1–Gd1–O9 ⁱ	76.36 (17)	O4–Gd1–O9 ⁱ	77.39 (17)
O2–Gd1–O6 ⁱⁱ	74.82 (16)	O6 ⁱ –Gd1–O1	147.26 (18)
O2–Gd1–O7 ⁱⁱ	69.18 (17)	O6 ⁱ –Gd1–O2	99.98 (17)
O2–Gd1–O8 ⁱⁱⁱ	116.62 (16)	O6 ⁱ –Gd1–O3	71.06 (16)
O2–Gd1–O9 ⁱ	69.16 (17)	O6 ⁱ –Gd1–O6 ⁱⁱ	66.23 (19)
O3–Gd1–O1	134.19 (18)	O6 ⁱ –Gd1–O7 ⁱⁱ	116.78 (15)
O3–Gd1–O2	140.62 (16)	O6 ⁱⁱ –Gd1–O8 ⁱⁱⁱ	104.50 (15)
O3–Gd1–O6 ⁱⁱ	66.43 (15)	O6 ⁱ –Gd1–O8 ⁱⁱⁱ	138.89 (15)
O3–Gd1–O7 ⁱⁱ	80.75 (16)	O6 ⁱ –Gd1–O9 ⁱ	71.34 (16)
O3–Gd1–O8 ⁱⁱⁱ	68.81 (15)	O7 ⁱⁱ –Gd1–O6 ⁱⁱ	50.64 (14)
O3–Gd1–O9 ⁱ	135.56 (17)	O7 ⁱⁱ –Gd1–O8 ⁱⁱⁱ	64.91 (15)
O4–Gd1–O1	85.45 (18)	O9 ⁱ –Gd1–O6 ⁱⁱ	117.01 (15)
O4–Gd1–O2	143.39 (17)	O9 ⁱ –Gd1–O7 ⁱⁱ	138.33 (16)
O4–Gd1–O3	75.02 (17)	O9 ⁱ –Gd1–O8 ⁱⁱⁱ	137.51 (15)

region as the ligand. The reason for the blue shift in the spectrum of the complex is that the Gd(III) ion is coordinated with the ligand (2-stp) [43–45].

The intra molecular energy transfer efficiency is known to be closely related to the energy gap between the lowest triplet energy level (T) of the ligand and the lowest excited

**Scheme 1** Coordination modes of 2-stp ligand for Gd(III) complex

state levels of the Ln⁺³ ion (Fig. 5) [46–48]. The triplet state of the ligand (2-stp) lie about 20,900 cm⁻¹. Whereas, the lowest excited energy level (⁶P_{7/2}) for the Gd(III) ion is found at 31,000 cm⁻¹. As a result, ligand-to-metal energy transfer cannot be observed and the observed luminescence for the Gd(III) complex is clearly ligand-oriented [49–51].

Conclusions

In this work, the synthesis, single-crystal X-ray diffraction, FT-IR, UV–visible and solid state photoluminescence characterization of a new 2D polymeric Gd(III) complex is presented. The solid-state photoluminescence measurements display remarkable green emission for Gd(III) complex, which is attributable to the n → π or π → π* intra-ligand charge transfer (ILCT). The suitability of the energy gap between the ligand triplet state and the metal-centered emissive states is a critical factor for the sensitization of lanthanide luminescence. Since the triplet state of the 2-stp ligand is lower than the lowest excited energy level of Gd(III) ion, the observed luminescence for the

Table 3 Hydrogen bond geometry (Å, °) of Gd(III) complex

D–H...A*	D–H	H...A	D...A	D–H...A	Symmetry
O1–H1A...O8	0.87	2.43	3.067	131	– 1 + x, y, z
O1–H1A...O6	0.87	2.44	2.804	106	– x, 1 – y, 1 – z
O2–H2A...O8	0.89	1.88	2.733	159	– 1 + x, y, z
O2–H2B...O5	0.89	1.93	2.790	163	– x, – y, 1 – z
O1–H1A...O7	0.87	2.42	2.886	114	– x, 1 – y, 1 – z
O1–H1A...O10	0.87	2.27	2.922	133	– x, 1 – y, 1 – z
O2–H2A...O11	0.85	2.07	2.741	135	1/2 + x, 1/2 – y, 1/2 + z
O2–H2B...O8	0.85	2.37	2.819	113	1/2 – x, – 1/2 + y, 3/2 – z
Cg(I)...				Cg...	
Cg(1)...				4.366(3)	1 – x, – y, 2 – z

D donor, A acceptor, Cg(I) plane number I (= ring number in () above), Cg–Cg distance between ring Centroids (Å), Cg (I) = C2–C3–C4–C5–C6–C7

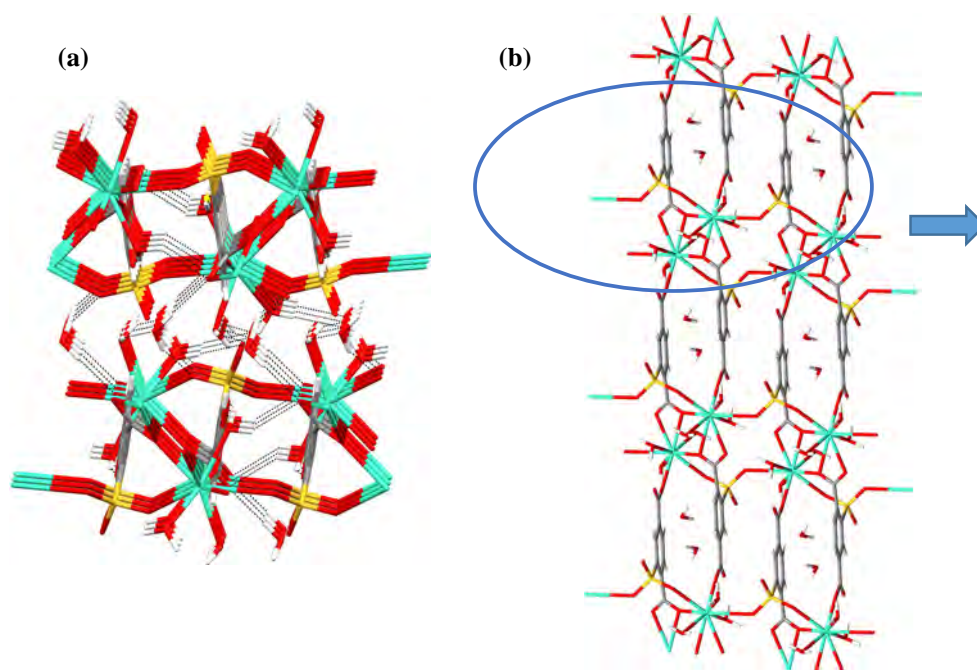


Fig. 2 **a** Hydrogen bonded 3D structure of Gd(III) complex. **b** 2-D layer framework of Gd(III) complex

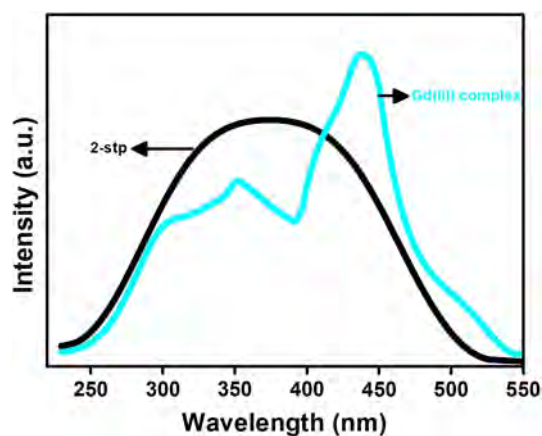


Fig. 3 Solid state UV-Visible spectra of the free ligand 2-stp and Gd(III) complex

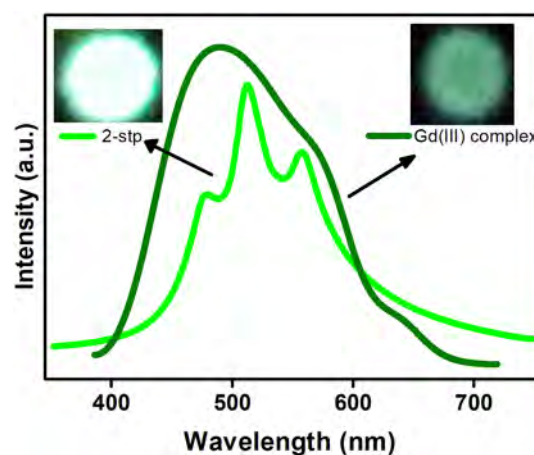


Fig. 4 Solid-state photoluminescence spectrum of the ligand (2-stp) and Gd(III) complex. The upper-left photo is a photoluminescent image of the ligand while the upper-right photo is the complex ($\lambda_{exc} = 349$ nm)

Gd(III) complex is ligand-oriented. Furthermore, Gd(III) complex exhibits a strong green luminescence emission in the solid state at room temperature, and hence the complex may be a promising green OLED developing electroluminescent material for flatted panel display applications.

Supplementary Material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 1834440 for Gd(III) complex. Copies of the

data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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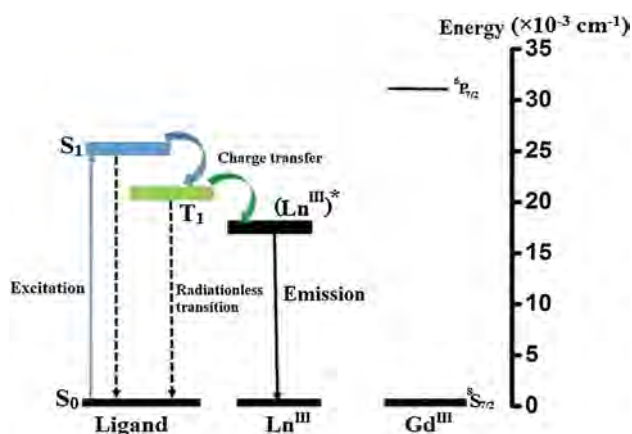


Fig. 5 The energy level diagram of the Gd(III) complex

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