

A Linear Ce^{III} Complex Based on *in-situ* Generated N-hydroxy-1,8-naphthalenedicarboximide^①

LIU Yang^{a, b, c} CAO Xue-Li^{a, b, c} WANG Wei^{b, c}
 LI Guo-Ling^{b, c} LI Shun^{b, c} HUANG You-Gui^{b, c②}

^a (College of Chemistry, Fuzhou University, Fuzhou 350108, China)

^b (CAS Key Laboratory of Design and Assembly of Functional Nanostructures, and Fujian Provincial Key Laboratory of Nanomaterials, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou 350002, China)

^c (Xiamen Institute of Rare Earth Materials, Haixi Institutes, Chinese Academy of Sciences, Xiamen 361021, China)

ABSTRACT A Ce^{III} linear complex of N-hydroxy-1,8-naphthalenedicarboximide (HL) has been synthesized with acenaphthoquinone dioxime (acndH₂) under solvothermal conditions, in which L is generated by a series of *in-situ* reactions starting from acndH₂. X-ray crystallography reveals the coordination chains of Ce^{III} ions are assembled into a 3D supramolecular framework via $\pi \cdots \pi$ interactions. The title complex has been characterized by IR and UV-Vis spectra and thermogravimetric analysis (TGA). Furthermore, the fluorescent properties study demonstrated a ligand-based emission of the Ce^{III} complex with ~6 nm blue-shift compared with the emission of the HL ligand.

Keywords: N-hydroxy-1,8-naphthalenedicarboximide, acenaphthoquinone dioxime, rare earth, fluorescence complex, X-ray crystallography; DOI: 10.14102/j.cnki.0254-5861.2011-3190

1 INTRODUCTION

Rare earth coordination complexes have attracted much attention because of their potential applications on catalysis^[1, 2], luminescence^[3], magnetism^[4], and bio-imaging or sensing^[5, 6]. As hard Lewis acids, rare earth ions can easily coordinate with multidentate hard base ligands containing O atoms^[7, 8]. Therefore, various multidentate O-containing ligands such as phenol^[9], quinine^[10], carboxylic acid^[11–15], and oxime^[16, 17] have been used to prepare rare earth complexes, and numerous complexes with structural diversities ranging from discrete molecule to three-dimensional (3D) framework have been obtained^[18–21]. As an O-containing multidentate ligand, acenaphthoquinone dioxime (acndH₂) comprising four oximate-based donor sites is a promising ligand for rare earth ions, and several 3d-4f complexes based on this ligand have been synthesized^[22, 23]. For example, a series of structures based on the {Cu^{II}₆Ln^{III}₂} building unit have been

reported by Stamatatos et al^[23]. However, homometallic rare earth coordination complexes of acndH₂ are still elusive. Therefore, we intend to obtain such rare earth complexes, and Ce attracts our interest because of its two stable oxidation states of Ce^{III} and Ce^{IV}^[24, 25]. Surprisingly, acndH₂ undergoes a series of reactions under solvothermal condition. The solvothermal reaction of CeCl₃ and acndH₂ in the presence of MnCl₂·4H₂O and Co(SCN)₂ leads to a Ce^{III} chain complex [Ce(L)₂Cl]_n·nEtOH (**1**) (EtOH = ethanol) with N-hydroxy-1,8-naphthalenedicarboximide (HL) as ligand which is *in-situ* generated from acndH₂. A reasonable route of the formation of L ligand is proposed. To support the proposed route, crystals of 1,8-naphthalimide were also obtained through crystallization of the resulting filtrate from the solvothermal reaction. Herein, we report the structure of complex **1** and propose a possible generation mechanism of L ligand from acndH₂. The thermal stability and the UV-Vis and IR spectra of the complex were investigated. Further-

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② Corresponding author. E-mail: yghuang@fjirsm.ac.cn

more, the fluorescent properties were also studied.

2 EXPERIMENTAL

2.1 Materials and methods

All reagents were purchased from commercial suppliers and used without further purification unless otherwise specified. IR spectrum was recorded on a Thermo Nicolet is 50 FT-IR spectrometer using the KBr pellet method in the range of 400~4000 cm⁻¹ and processed with the OMNIC 6.0 software package. Powder X-ray diffraction (PXRD) pattern was performed on a Rigaku Miniflex 600 diffractometer with Cu-K α radiation using flat plate geometry: the scan rate was 5 °/min and the 2 θ scan range is 5~50 °. Thermogravimetric analysis (TGA) was performed on a Mettler-Toledo TGA/DSC-1 at 30~800 °C in argon atmosphere at a heating rate of 10 °/min. UV-vis spectrum was recorded on a Cary 5000 at room temperature. Fluorescence spectra were measured at room temperature on a FLS980 system.

2.2 Synthesis of the title complex

A mixture containing acndH₂ (10.0 mg, 0.047 mmol), CeCl₃ (25.0 mg, 0.101 mmol), MnCl₂ 4H₂O (30.0 mg, 0.152 mmol), Co(SCN)₂ (10.0 mg 0.057 mmol), ethanol (10 mL), and distilled H₂O (0.5 mL) was sealed in a 25 mL Teflon-lined autoclave and heated to 160 °C in 5 h. After maintaining at 160 °C for 20 h, the autoclave was cooled to room temperature in 8 h. Yellow crystals of complex **1** were obtained (yield: 42% based on acndH₂). Crystallization of

the green filtrate led to the crystals of 1,8-naphthalimide (**2**).

2.3 X-ray structure determination

The single-crystal X-ray diffraction data were collected at 200 K on a Bruker D8 Venture diffractometer with MoK α (λ = 0.71073 Å) radiation. Data reduction, integration, and scaling were performed with a Bruker APEX3 software package. Crystal structures were solved by direct methods and refined by full-matrix least-squares technique on F^2 with the SHELXTL 2014 program^[26, 27]. Hydrogen atoms on C atoms were located using the geometric method, while those on O and N atoms were found by Fourier map. Non-hydrogen atoms were refined with anisotropic thermal parameters.

3 RESULTS AND DISCUSSION

3.1 Crystal structure

Single-crystal X-ray analysis reveals that complex **1** crystallizes in triclinic space group $P\bar{1}$, with a = 8.3852(3), b = 12.1240(6), c = 12.1565(6) Å, α = 101.993(2)°, β = 91.757(2)° and γ = 102.412(2)°. Its asymmetric unit contains a Ce^{III} ion, a Cl⁻ ion, two L ligands, and a lattice ethanol molecule. Each Ce^{III} ion is nono-coordinated by one Cl⁻ ion and four L ligands in a tricapped trigonal prismatic geometry (Fig. 1a). The Ce–Cl bond length is 2.819 Å and the Ce–O bond distances fall in the range of 2.427~2.670 Å (Table 1). The L ligand adopts a $\eta^1:\eta^2:\eta^1:\mu_3$ binding mode bridging two Ce^{III} ions (Fig. 1b).

Table 1. Selected Bond Lengths (Å) and Bond Angles (°)

Bond	Dist.	Bond	Dist.	Bond	Dist.
Ce(1)–Cl(1)	2.8188(8)	Ce(1)–O(3)	2.670(2)	Ce(1)–O(6)#2	2.668(2)
Ce(1)–O(5)	2.427(2)	Ce(1)–O(4)	2.583(2)	Ce(1)–O(2)#1	2.475(2)
Ce(1)–O(2)	2.450(2)	Ce(1)–O(5)#2	2.507(2)	Ce(1)–O(1)#1	2.558(2)
Angle	(°)	Angle	(°)	Angle	(°)
O(5)#2–Ce(1)–O(1)#1	69.20(6)	O(2)–Ce(1)–O(2)#1	59.82(7)	O(5)–Ce(1)–O(2)#1	134.64(7)
O(5)#2–Ce(1)–O(4)	116.35(6)	O(5)–Ce(1)–O(5)#2	62.46(7)	O(2)–Ce(1)–O(5)#2	156.68(7)
O(1)#1–Ce(1)–O(4)	72.97(7)	O(5)–Ce(1)–O(4)	61.95(6)	O(2)#1–Ce(1)–O(1)#1	61.46(6)
O(5)–Ce(1)–O(6)#2	121.74(6)	O(2)#1–Ce(1)–O(3)	116.21(6)	O(5)–Ce(1)–O(1)#1	79.93(6)
O(2)–Ce(1)–O(6)#2	100.22(6)	O(5)#2–Ce(1)–O(3)	126.12(6)	O(2)–Ce(1)–O(1)#1	119.40(6)
O(5)#2–Ce(1)–O(6)#2	59.77(6)	O(1)#1–Ce(1)–O(3)	145.22(7)	O(2)–Ce(1)–O(4)	86.86(7)
O(1)#1–Ce(1)–O(6)#2	73.77(7)	O(4)–Ce(1)–O(3)	72.33(7)	O(1)#1–Ce(1)–Cl(1)	141.22(5)
O(4)–Ce(1)–O(6)#2	145.00(7)	O(6)#2–Ce(1)–O(3)	140.62(7)	O(4)–Ce(1)–Cl(1)	142.12(6)
O(5)–Ce(1)–O(3)	81.98(6)	O(5)–Ce(1)–Cl(1)	101.55(5)	O(3)–Ce(1)–Cl(1)	71.65(5)
O(2)–Ce(1)–O(3)	60.73(6)	O(2)–Ce(1)–Cl(1)	85.67(5)	O(2)#1–Ce(1)–O(5)#2	117.59(6)
O(2)#1–Ce(1)–O(6)#2	71.29(7)	O(2)#1–Ce(1)–Cl(1)	123.28(5)	O(6)#2–Ce(1)–Cl(1)	72.86(5)
O(5)–Ce(1)–O(2)	137.80(6)	O(5)#2–Ce(1)–Cl(1)	77.22(5)	O(2)#1–Ce(1)–O(4)	83.47(7)

Symmetry codes: (#1) $-x+1, -y+1, -z+1$; (#2) $-x, -y+1, -z+1$

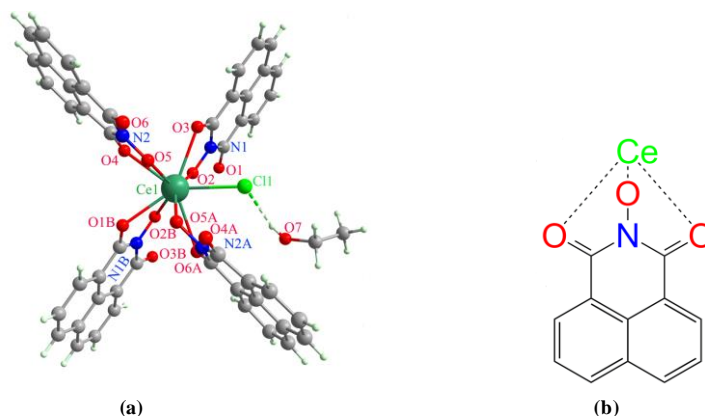


Fig. 1. (a) Coordination environment of Ce^{III} in complex 1. Symmetry codes: A: $-x, 1-y, 1-z$; B: $1-x, 1-y, 1-z$. (b) Coordination mode of L ligand in complex 1

The Ce^{III} ions are doubly bridged by the L ligands, resulting in a linear structure with alternative Ce–Ce distances of 4.220 and 4.269 Å (Fig. 2a), respectively. Each chain associates with its four neighbors through two types of strong offset $\pi \cdots \pi$ stacking interactions. The two interassociated naphthalene moieties are parallel with each other with centroid-centroid distances of 3.701 and

3.678 Å, respectively (Fig. 2b). As a result of $\pi \cdots \pi$ interactions, a three-dimensional (3D) porous supramolecular structure with channels along the $[1\ 0\ 0]$ direction is generated. The lattice ethanol molecules hydrogen-bond to the Cl^- ions ($\text{OH} \cdots \text{Cl}$: 2.370 Å) and fill the channels (Fig. 2c).

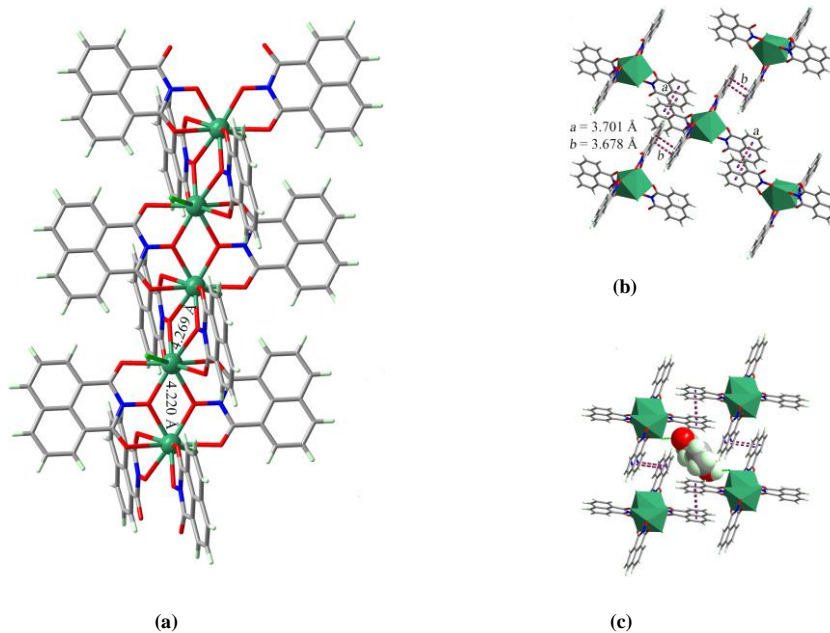


Fig. 2. (a) Linear Ce^{III} chain of complex 1. (b) Structural illustration of the $\pi \cdots \pi$ interactions between adjacent chains of complex 1. (c) 3D porous supramolecular structure showing the ethanol filled channel

3.2 PXRD, TGA, UV-Vis, and FT-IR analyses

The powder X-ray diffraction pattern (PXRD) of the synthesized sample of complex 1 matches well with the simulated one, confirming the phase purity of the as-synthesized sample (Fig. 3a). Thermogravimetric analysis (TGA) demonstrates an initial weight loss of ~6.57% from 30 to 180 °C which corresponds to the departure of lattice ethanol molecules (calcd. weight loss: 7.12%) (Fig. 3b).

UV-Vis spectrum of the solid sample of complex 1 reveals a broad absorption at ~370 nm (Fig. 3c). The FT-IR spectrum of complex 1 was also recorded in the 4000~400 cm^{-1} range. The absorption at ~3329 cm^{-1} is characteristic of $\nu_{(\text{OH})}$, confirming the existence of ethanol in the crystal structure. The absorption at ~1653 and 1585 cm^{-1} is attributed to the C=O groups in the L ligand (Fig. 3d).

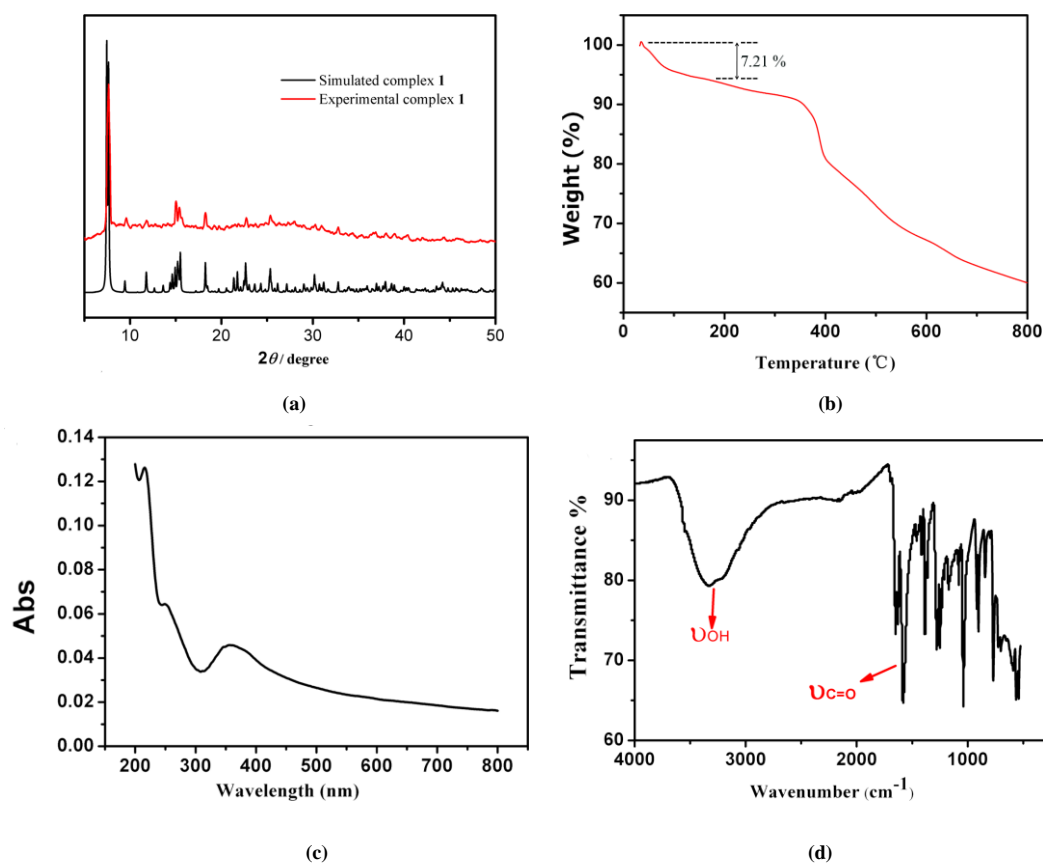


Fig. 3. (a) PXRD patterns, (b) TGA, (c) UV-Vis spectrum, and (d) IR spectrum of complex 1

3.3 Fluorescent properties

Although Ce^{III} is nonfluorescent, as a 1,8-naphthalimide derivative, the ligand HL is expected to be luminescent^[28]. To give insights into whether the coordination of Ce^{III} influences the luminescence of HL, the fluorescence of both the ligand and complex 1 was studied at room temperature. Excited at 248 nm, the HL ligand yields blue fluorescence with a broad emission band at 480 nm. Excited at the same

wavelength, complex 1 exhibits a broad emission band at 474 nm, which is weaker and ~6 nm blue-shifted compared with that of the HL ligand. The blue luminescence behaviors of the HL ligand and complex 1 are due to the $\pi \rightarrow \pi^*$ transition of the naphthalene core^[29], while the blue-shift can be attributed to the destabilizing effect on the π^* orbitals of the naphthalene core induced by Ce^{III} ions, therefore increasing the energy of the intraligand $\pi \rightarrow \pi^*$ transition^[29].

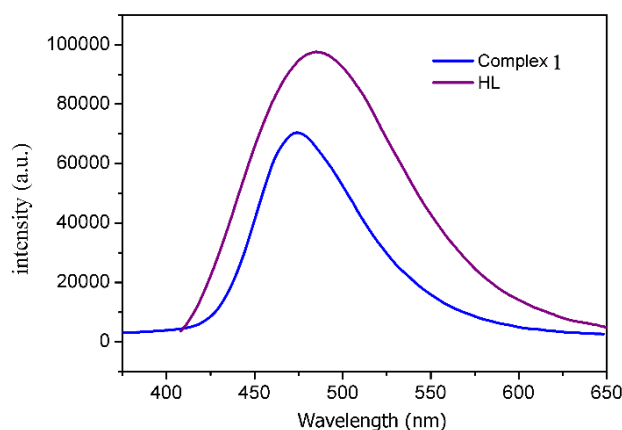
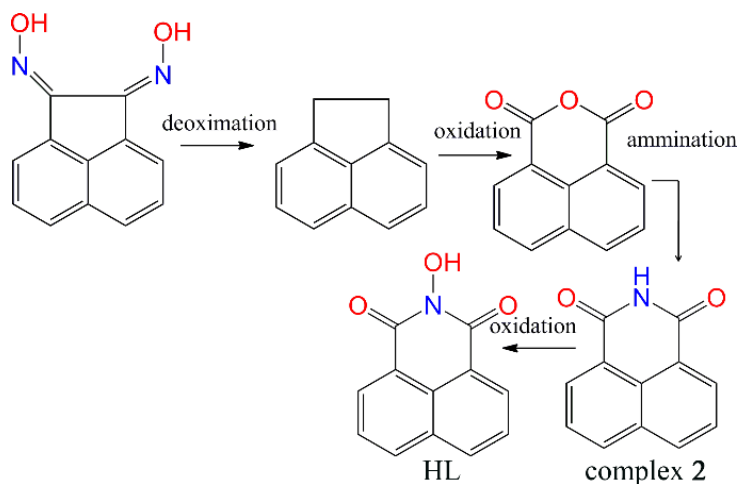


Fig. 4. Photoluminescent spectra of the ligand (HL) and complex 1 ($\lambda_{ex} = 248$ nm)

3.4 Proposed route of the generation of L ligand

The most surprising is the finding of L ligand in the structure of complex **1**, considering that the added ligand is acndH_2 . Therefore, acndH_2 must undergo a series of *in-situ* reactions under the hydrothermal condition. The proposed route of the generation of L ligand is illustrated in Scheme 1. Initially, acndH_2 underwent deoxygenation forming acenaphthene, which then was further oxidized into 1,8-naphthalic anhydride^[30]. The generating 1,8-naphthalic anhydride could

undergo ammination^[31] and then be oxidized leading to the ligand L^[32]. To support the proposed route, we tried to obtain some intermediate states. Fortunately, some crystals of 1,8-naphthalimide (**2**) molecules were isolated from the filtrate after the solvothermal reaction for the synthesis of complex **1**. Complex **2** crystallizes in space group of $P2_1/n$ (Table 1), wherein 1,8-naphthalimide molecules exist as hydrogen bonded dimers, as found previously^[33].



Scheme 1. Proposed route of the synthesis of ligand HL (N-hydroxy-1,8-naphthalenedicarboximide) using acndH_2 as the reactant

4 CONCLUSION

In summary, a linear Ce^{III} complex based on *in-situ* generated L ligand has been synthesized using acndH_2 under solvothermal conditions. The complex exhibits a ligand-based emission with a broad band at 474 nm, which is ~6 nm blue-shifted compared with the emission of the HL ligand. It was proposed that acndH_2 undergoes a series of

in-situ reactions of deoxygenation/oxidation/amination/oxidation forming L ligand which further coordinates with Ce^{III} ions in the resulting linear structure. Our study demonstrates a new avenue for coordination complexes based on N-hydroxy-1,8-naphthalenedicarboximide. Following this strategy, some new coordination complexes based on N-hydroxy-1,8-naphthalenedicarboximide can be obtained.

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