

Synthesis and Single Crystal XRD of Hydroniumpentaaquabis(1,10-phenanthroline) europium(III)tetrachloride Hydrate [(H₃O⁺)(H₂O)₅(phen)₂Eu(III)]Cl₄.H₂O

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Authors' contributions

This whole work was carried out by the author SK.

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ABSTRACT

A novel europium(III)-1,10-phen complex (I) has been synthesized and characterized by single crystal XRD. Complex I crystallized in the monoclinic space group *C2/c* with unit cell dimensions $a = 30.8233(13)$ Å, $b = 9.5063(4)$ Å, $c = 21.9973(10)$ Å, $\alpha = 90^\circ$, $\beta = 113.616(2)^\circ$, $\gamma = 90^\circ$, and $Z = 4$. The structure involves several hydrogen bond donor and acceptor sites leading to the formation of a supramolecular architecture.

Keywords: Europium; 1,10-phenanthroline; hydronium ion; chloride; XRD.

1. INTRODUCTION

The chemistry of lanthanide coordination compounds is the subject of increasing interest in the recent years due to the wide variety of their chemical and biological applications [1,2]. Europium (III) and terbium (III) ions find an important place in the fluorescent studies as there is a suitable energy gap between the emissive excited state and the ground state as

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well as the large Stokes shift of emission. Hence, any overlap of the emission bands with the absorption bands is unlikely [3,4]. These ions have, therefore, been widely studied for applications such as fluoro-immunoassays, liquid-crystalline displays, and biomedical imaging [5]. However, since the f-f transitions are forbidden by spin and parity selection rules, the emission occurs through sensitization with coordinated ligands, which function as antennas [5]. The variety of ligands developed for this purpose ranges from simple aromatic acids [6] to more complicated multidentate ligand architectures capable of selectively binding different lanthanum ions [7]. Due to the potential applications in optoelectronics, catalysis, and magnetism [8,9], recently a significant increase in the research of coordination polymers in which metal ion centers are bridged through organic molecules has been observed. Europium (III)-phenanthroline complexes are known for their luminescence behaviour [10a-q]. Binuclear europium complexes exhibit interesting architecture [10r]. In continuation of our work [10s], the synthesis of a Eu(III)-1,10-phenanthroline complex I and its characterization by single crystal XRD have been reported.

2. EXPERIMENTAL DETAILS

2.1 Materials

1,10-Phenanthroline was purchased from Sigma-Aldrich. Europium nitrate was purchased from Merck. All other reagents and solvents were obtained from commercial sources and were of analytical grade.

2.2 Synthesis of Hydroneuropium(III) tetrachloride Hydrate (I)

1,10-Phenanthroline (2 mmol) and water-methanol mixture (1:1, 20 mL) were taken in a 100 mL RB flask. Europium nitrate (1 mmol) in water (10 mL) was added dropwise to the flask and the reaction mixture was stirred for 5 h. The precipitate formed was filtered off, washed several times with water followed by a little cold chloroform, and then dried in vacuo over anhydrous CaCl_2 . The yield was found to be 65%. This solid was dispersed in water and a few drops of dil. HCl were added to dissolve the precipitate. The solution obtained was kept at room temperature. After 6 weeks, colorless crystals suitable for single crystal study were obtained.

2.3 Single Crystal XRD Data Collection

A Leica MZ 75 microscope was used to identify a suitable colorless block with very well defined faces of dimensions (max, intermediate, and min) 0.04 mm × 0.03 mm × 0.03 mm from a representative sample of crystals of the same habit. The crystal mounted on a nylon loop was then placed in a cold nitrogen stream (Oxford) maintained at 110 K.

A BRUKER GADDS X-ray (three-circle) diffractometer was employed for crystal screening, unit cell determination, and data collection. The goniometer was controlled using the FRAMBO software, v.4.1.05 [11]. The sample was optically centered with the help of a video camera such that no translations were observed as the crystal was rotated through all positions. The detector was set at 5.0 cm from the crystal sample. The X-ray radiation employed was generated from a Cu sealed X-ray tube ($K\alpha = 1.5418 \text{ \AA}$ with a potential of 40 kV and a current of 40 mA) fitted with a graphite monochromator in the parallel mode (175 mm collimator with 0.5 mm pinholes).

180 Data frames were taken at widths of 0.50. These reflections were used to determine the unit cell using Cell_Now [12]. A suitable cell was found and refined by nonlinear least squares and Bravais lattice procedures. The unit cell was verified by examination of the $h k l$ overlays on several frames of data. No super-cell or erroneous reflections were observed.

After careful examination of the unit cell, a standard data collection procedure (9 sets) was initiated using omega and phi scans.

2.4 Data Reduction, Structure Solution, and Refinement

Integrated intensity information for each reflection was obtained by reduction of the data frames with APEX2 [13]. The integration method employed a three dimensional profiling algorithm and all data were corrected for Lorentz and polarization factors, as well as for crystal decay effects. Finally the data was merged and scaled to produce a suitable data set. SADABS [14] was employed to correct the data for absorption effects.

Systematic reflection conditions and statistical tests indicated the space group $C2/c$. A solution was obtained readily using SHELXTL (SHELXS) [15]. Five water molecules coordinated to Eu1 and a molecule and a half of hydration were found. Hydrogen atoms were placed in idealized positions and were refined using riding model. All non-hydrogen atoms were refined with anisotropic thermal parameters. The structure was refined (weighted least squares refinement on F^2) to convergence [15,16]. OLEX2 was employed for the structure plots [16].

3. RESULTS AND DISCUSSION

The crystal data is shown in the Table 1. The important bond lengths and bond angles are shown in the Tables 2 and 3. Complex I crystallizes in the monoclinic space group $C2/c$. The asymmetric unit of complex I is composed of one pentaquabis(1,10-phenanthroline)europium(III) cation, four chloride anions, one hydronium ion, and a lattice water molecule. The coordination number of Eu(III) ion in this complex is nine and the geometry around Eu(III) is found to be having slightly distorted and tricapped trigonal-prismatic coordination geometry [17-19]. Eu(III) is surrounded by five oxygen atoms furnished by five water molecules and four nitrogen atoms furnished by two bidentate 1,10-phenanthroline (phen) in the coordination sphere. The second sphere of Eu(III) is furnished by four chloride anions and four water molecules, and a hydronium ion which are located in the crystal lattice. ORTEP view and geometry of the complex I are shown in Figs. 1 and 2 respectively.

Table 1. Crystal data of complex I

Empirical formula	C ₄₈ H ₅₈ C ₁₇ Eu ₂ N ₈ O ₁₃
Formula weight	1507.09
Temperature	110(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 30.8233(13) Å ⁰ , α = 90°. b = 9.5063(4) Å ⁰ , β = 113.616(2)°. c = 21.9973(10) Å ⁰ , γ = 90°.
Volume	5905.7(4) Å ³
Z	4
Density (calculated)	1.695 Mg/m ³
Absorption coefficient	18.515 mm ⁻¹
F(000)	3004
Crystal size	0.04 x 0.03 x 0.03 mm ³
Theta range for data collection	3.13 to 60.00°.
Index ranges	-33<=h<=34, -10<=k<=10, -24<=l<=24
Reflections collected	18006
Independent reflections	4381 [R(int) = 0.0480]
Completeness to theta = 60.00°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.6066 and 0.5246
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4381 / 0 / 365
Goodness-of-fit on F ²	1.050
Final R indices [I>2sigma(I)]	R1 = 0.0312, wR2 = 0.0799
R indices (all data)	R1 = 0.0344, wR2 = 0.0812
Largest diff. peak and hole	0.859 and -1.221 e. Å ⁻³

Table 2. Bond lengths [Å⁰]

Eu(1)-O(1)	2.396(3)
Eu(1)-O(4)	2.431(3)
Eu(1)-O(2)	2.437(3)
Eu(1)-O(3)	2.447(2)
Eu(1)-O(5)	2.489(3)
Eu(1)-N(1)	2.582(3)
Eu(1)-N(4)	2.617(3)
Eu(1)-N(2)	2.632(3)
Eu(1)-N(3)	2.643(3)
N(1)-C(1)	1.342(5)
N(1)-C(5)	1.366(5)
N(2)-C(12)	1.335(5)
N(2)-C(9)	1.367(5)
N(3)-C(13)	1.331(5)
N(3)-C(17)	1.371(5)
N(4)-C(24)	1.336(5)
N(4)-C(21)	1.364(5)
O(55)-O(55)#1	1.382(15)

Table 3. Bond angles [°]

O(1)-Eu(1)-O(4)	138.33(9)
O(1)-Eu(1)-O(2)	137.46(10)
O(4)-Eu(1)-O(2)	71.12(9)
O(1)-Eu(1)-O(3)	72.15(9)
O(4)-Eu(1)-O(3)	139.14(9)
O(2)-Eu(1)-O(3)	105.22(9)
O(1)-Eu(1)-O(5)	69.54(9)
O(4)-Eu(1)-O(5)	68.90(9)
O(2)-Eu(1)-O(5)	128.25(9)
O(3)-Eu(1)-O(5)	126.51(9)
O(1)-Eu(1)-N(1)	82.75(10)
O(4)-Eu(1)-N(1)	81.44(10)
O(2)-Eu(1)-N(1)	71.30(10)
O(3)-Eu(1)-N(1)	137.45(9)
O(5)-Eu(1)-N(1)	71.47(9)
O(1)-Eu(1)-N(4)	86.09(9)
O(4)-Eu(1)-N(4)	82.19(9)
O(2)-Eu(1)-N(4)	134.82(10)
O(3)-Eu(1)-N(4)	72.43(9)
O(5)-Eu(1)-N(4)	69.10(9)
N(1)-Eu(1)-N(4)	140.48(10)
O(1)-Eu(1)-N(2)	71.86(9)
O(4)-Eu(1)-N(2)	131.62(9)
O(2)-Eu(1)-N(2)	66.60(9)
O(3)-Eu(1)-N(2)	75.81(9)
O(5)-Eu(1)-N(2)	123.27(9)
N(1)-Eu(1)-N(2)	63.78(10)
N(4)-Eu(1)-N(2)	145.65(9)
O(1)-Eu(1)-N(3)	134.18(10)
O(4)-Eu(1)-N(3)	73.34(10)
O(2)-Eu(1)-N(3)	74.40(10)
O(3)-Eu(1)-N(3)	66.78(9)
O(5)-Eu(1)-N(3)	121.76(10)
N(1)-Eu(1)-N(3)	142.45(10)
N(4)-Eu(1)-N(3)	63.15(9)
N(2)-Eu(1)-N(3)	114.97(10)
Eu(1)-O(1)-H(1A)	110.8
Eu(1)-O(1)-H(1B)	110.5
Eu(1)-O(2)-H(2A)	109.6
Eu(1)-O(2)-H(2B)	109.4
Eu(1)-O(3)-H(3A)	110.6
Eu(1)-O(3)-H(3B)	110.4
Eu(1)-O(4)-H(4A)	109.6
Eu(1)-O(4)-H(4B)	109.6
Eu(1)-O(5)-H(5A)	125.3
Eu(1)-O(5)-H(5B)	125.3
C(1)-N(1)-Eu(1)	121.6(3)
C(5)-N(1)-Eu(1)	120.3(2)
C(12)-N(2)-Eu(1)	124.5(3)
C(9)-N(2)-Eu(1)	118.4(2)
C(13)-N(3)-Eu(1)	123.9(3)
C(17)-N(3)-Eu(1)	119.0(2)
C(24)-N(4)-Eu(1)	122.5(3)
C(21)-N(4)-Eu(1)	120.5(2)

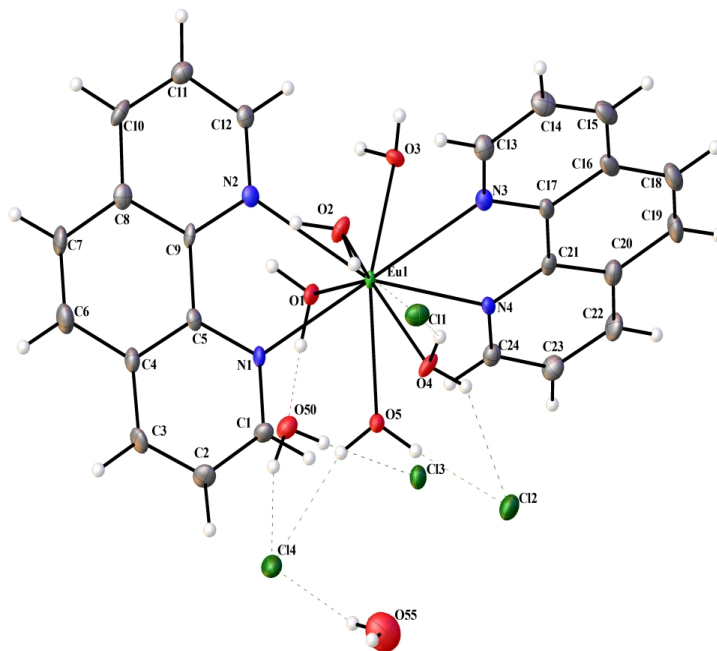


Fig. 1. ORTEP view of the asymmetric unit of complex I

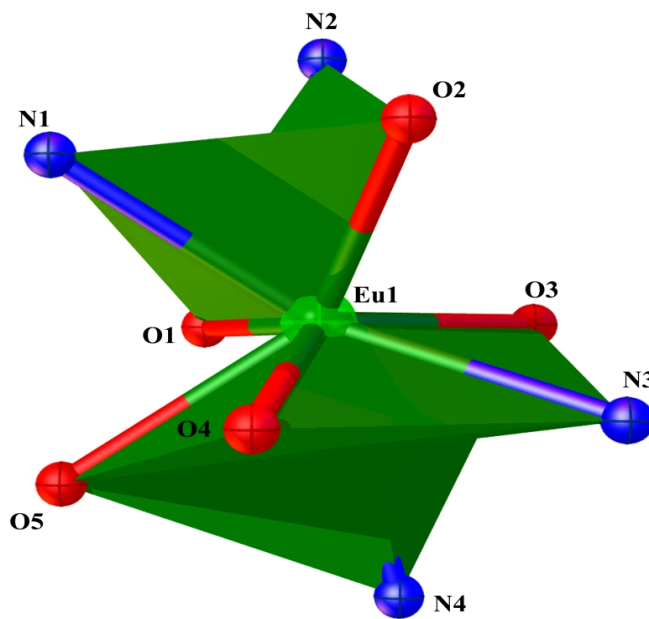


Fig. 2. The geometry environment of the nine-coordinated Eu(III) ion

The two coordinated phen molecules are noncoplanar having twisted at about 65.90° with respect to each other. The bond distances between Eu1 and the five coordinated water molecules (O1, O2, O3, O4, and O5) are found to be 2.396(3), 2.437(3), 2.447(2), 2.431(3), and 2.489(3) Å respectively. The distances between Eu1 and coordinated bidentated nitrogens (N1, N2, N3, N4) of phen are found to be 2.582(3), 2.632(3), 2.643(3), and 2.617(3) Å respectively. These values are found to be in good agreement with the reported values [20-22].

As this structure is rich in hydrogen bond donor and acceptor sites, almost all the donor and acceptor atoms involve in the formation of supramolecular architecture. Water molecules (both coordinated and non-coordinated) and chloride ions behave as both bifurcated hydrogen bond acceptors and donors. The supramolecular architecture is developed by several non-covalent interactions in which the coordinated and non-coordinated water molecules and chloride anions play a vital role in the construction of various supramolecular assemblies. Two different bifurcated $R_2^1(6)$ ring motifs are formed *via* two pairs of O-H...Cl hydrogen bonds. These two motifs interlink the coordinated water molecules (O2, O4, and O5) with Cl1 and Cl2. The generation of one $R_3^1(8)$ ring motif involves two coordinated water molecules (O1 and O5), one non-coordinated water molecule (O50), and a chloride ion (Cl4) which are interlinked *via* a pair of O-H...Cl and an O-H...O hydrogen bonds. In the asymmetric unit, each metal centre possesses these molecular self-assemblies made up of three ring motifs as shown in Fig. 3.

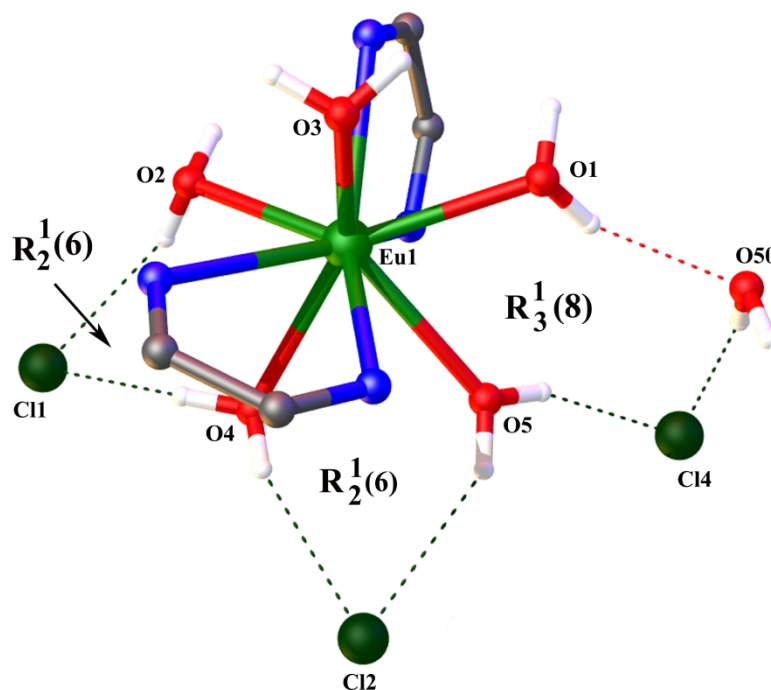


Fig. 3. Molecular self assemblies formed around the metal centre. Only coordinated atoms of 1,10-phen are shown for the sake of clarity

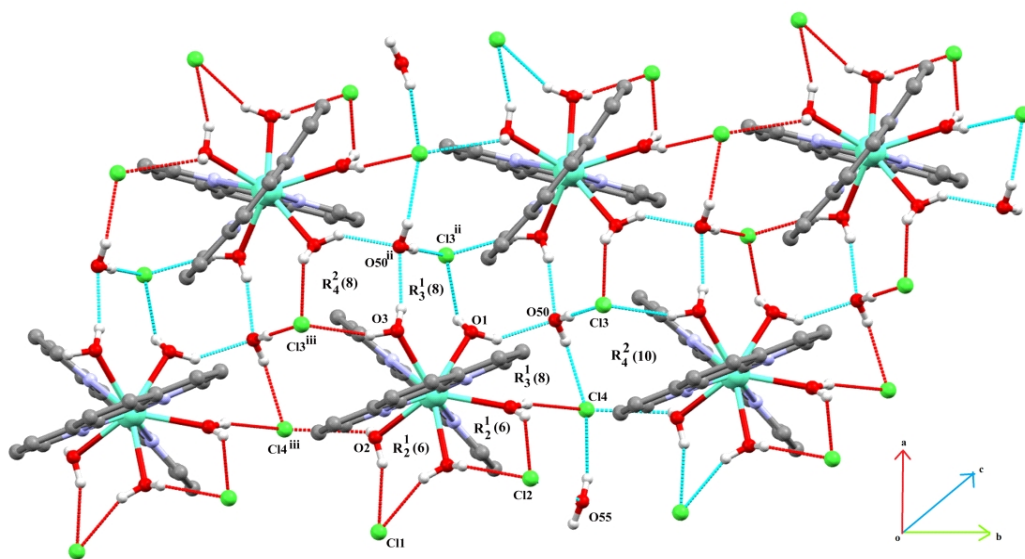


Fig. 4. Sheet-like architecture with multiple hydrogen bonded pattern extending along the b axis

Two such metal centered molecular self assemblies are further connected by three ring motifs $R_4^2(8)$, $R_3^1(8)$, and $R_4^2(10)$ which are formed via O-H...O and O-H...Cl hydrogen bonds. The generation of $R_4^2(8)$ motif involves, two inversion related molecular self assemblies, symmetry related lattice water molecule O50ii, and Cl3iii ion. The other $R_3^1(8)$ ring motif is formed by interlinking coordinated O1 and O3, symmetry related lattice water molecule O50ii, and Cl3ii ion. The alternative occurrence of these ring motifs link the molecular assemblies in adjacent plane intact and they develop multiple hydrogen bonded pattern extending along the b axis. The $R_4^2(10)$ ring motif connects the self assemblies present in the same plane through two pairs of O-H...Cl hydrogen bonds which involve symmetry related Cl3iii and Cl4iii ions. These interactions together have developed a sheet-like architecture propagating along the b axis (Fig. 4). Further, the structure is stabilized by several weak C-H...O and C-H...Cl hydrogen bonds.

3.1 Refinement

All the hydrogen atoms were positioned geometrically (C-H = 0.95 Å) and were refined using a riding model, with Uiso(H) = 1.2Ueq(C) for CH or 1.5Ueq(O) for water. It was not possible to locate the H atom for the hydronium ion in the difference Fourier map.

CCDC 955492 contains the supplementary crystallographic data for the complex I that can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Center.

4. CONCLUSION

This study provides the synthesis and single crystal XRD characterization of a rare earth complex I, hydroniumpentaquabis(1,10-phenanthroline)europium(III) tetrachloride hydrate.

The supramolecular architecture is developed by abundant non-covalent interactions leading to a sheet-like structure propagating along the b axis.

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COMPETING INTERESTS

This work was prepared in the research laboratory of SK. He proposed and drafted the work.

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