

## *Spectroscopic Studies and Biological Activity of Cerium (IV) 8-hydroxyquinoline Complex*

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**Abstract:** Spectrophotometric analysis of the complexation of 8-hydroxyquinoline (8-HQ) with cerium(IV) has been performed. The Ce-8-HQ complex was prepared and characterized using FT-IR and UV-VIS absorption spectral data. According to the spectral data, both oxygen and nitrogen atoms from 8-HQ were involved in the complex formation. The stoichiometry and formation constant (Kf) of 8-HQ ligand with cerium(IV) ion were determined using the mole-ratio method at constant temperature, with the molar ratio and Kf of this complex being 1:2 (metal:ligand) and  $1.308 \times 10^{14}$ , respectively. Beer's law was followed with a molar absorptivity ( $\epsilon$ ) of  $22089 \text{ l}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$  at  $\lambda_{\text{max}} = 253 \text{ nm}$  in the range  $2.0 \times 10^{-5}$ - $5.0 \times 10^{-5} \text{ M}$ . In vitro antimicrobial effects of the ligand and complex were also investigated. The biological study data confirmed that each of the investigated compounds, i.e. ligand and complex, has the best pastime against all of the microorganisms studied. However, comparing the spots between concentrations of cerium (IV) complex with 8-hydroxy quinoline indicates a more potent pastime with maximum Gram-terrible.

**Keywords:** Cerium (IV), 8-hydroxyquinoline, Spectroscopic investigation, Antibacterial activity

### Introduction

The chemistry of heterocyclic compounds is a constantly evolving field in organic chemistry. Despite incredible advances in medicinal chemistry, infectious diseases caused by microorganisms such as bacteria, fungi, viruses, and parasites continue to pose a serious threat to human health, with widespread drug resistance, particularly multidrug resistance, emerging [1-5].

Donor chelating bidentate ligands are usually (N,N), (N,O), (O,S), or (O,O) molecules that can be cytotoxic. Among the bioactive ligands that have received extensive research are 8-hydroxyquinoline (HQ) and its derivatives. [6-10]. The chemical formula for the organic compound 8-hydroxyquinoline (Figure 1) is  $\text{C}_9\text{H}_7\text{NO}$ . It is a derivative of the heterocyclic quinolone due to the addition

of an OH group to the carbon atom at position 8. This pale yellow substance is commonly found in industry. [11,12], which is also known in medicinal chemistry as a "privileged structure" [13,14]. Because of its ability to arrange with different metallic ions as bidentate via the nitrogen atom of the quinoline ring and the oxygen atom after deprotonation of the hydroxyl group, hydroxyquinoline is a necessary compound in many applications [15-19]. In the literature, there are numerous examples of 8-hydroxyquinoline (HQ) and its derivatives transition polymetallic complexes [20]. The derivatives of heterocyclic nitrogen compounds play an important role in many biological processes and as synthetic medications. [21,22].

Recently, metabolism research and the development of slow-release or longact-

ing medications have both increased the importance of metal complexes. Metal ions are known to hasten drug action [23,24]. Antibacterial, anticancer, and antiviral properties of rare earth ions [25-27]. Lanthanides have piqued the interest of organic chemists in recent decades due to their high oxo-philicity [28,29]. Molecular cerium compounds, unlike other lanthanides, have easy access to the trivalent and tetravalent oxidation states [30]. Liddell et al., for example, have recently reported the preparation of cerium methanediide complexes [31].

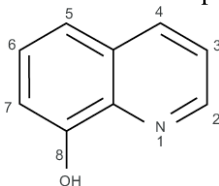


Figure 1. 8-hydroxyquinoline structure

The current research was designed to prepare and characterize a Cerium (IV) metal chelate with 8-hydroxyquinoline ligand, as well as investigate its structure and stoichiometry using IR and UV-VIS spectroscopic methods. Antibacterial activity has also been studied.

### Materials and methods:

#### Reagents and solvents

All reagents and chemicals were of analytical grade and used exactly as received, with no further purification. Sigma-Aldrich provided 8-hydroxyquinoline (C<sub>9</sub>H<sub>7</sub>NO, 99.0%). Merck supplied the Cerium (IV) Sulfate [Ce(SO<sub>4</sub>)<sub>2</sub>·4H<sub>2</sub>O] (> 99.8%), ethanol, acetonitrile, and methanol (99%). All experiments were performed using double-distilled water.

#### Preparation of metal complex

The complex was synthesized using the method described in the literature [32]. To prepare the ligand and metal ion solutions, (99%) ethanol, methanol, and double distilled water were used as solvents. 1 mmol 0.4 g Cerium (IV) Sulfate in 10 ml double distilled water

was slowly added to an equimolar amount of 8-hydroxyquinoline 1 mmol 0.145 g in 10 ml Methanol. In an open flask, the mixture was stirred for half an hour at room temperature. The temperature was gradually raised, and the reaction was then refluxed in a water bath for four hours, until the color was dark brown. The solution was then filtered through Whatman filter paper No. 1, and the precipitate was washed with ethanol before being washed with acetone and finally dried in a vacuum desiccator over anhydrous CaCl<sub>2</sub> (Figure 1).

#### Spectral analysis

The UV-visible absorption spectra of 8-HQ, the complex in their solutions, were measured using a Cary 60 spectrophotometer from Agilent Technologies. Fourier transform-infrared spectroscopy (FTIR) of 8-HQ and synthesized cerium complex was performed using a Perkin Elmer FT-IR Spectrometer with a resolution of 1 cm<sup>-1</sup>. All spectra were measured at 25°.

By using a spectrophotometric technique that had already been published by Joe and Yones [33-35], the stoichiometry of the cerium-8-hydroxyquinoline complex was ascertained. While varying the Ce concentrations, a group of solutions with a constant 8-HQ concentration of 2.00x10<sup>-6</sup> mol/l were created. The complex's stoichiometric composition and the values of the K<sub>f</sub> formation constant and molar absorptivity were calculated in accordance with the observed results.

#### Source of Microbial Cultures

Cultures were obtained from the Moafa Lab and Alalamia Lab in Misuratua, Libya's microbiology department. They are made up of four different bacteria strains (Staphylococcus aureus, Escherichia coli, Klebsiellaspp, pseudomonas spp).The bacterial cultures were kept in their respective agar at a temperature of about 5°C throughout the investigation and used as stock cultures.

### In Vitro Antibacterial Activity Testing

The in vitro antimicrobial activities of Cerium (IV) complex with 8-hydroxy quinoline were investigated using four strains of different bacteria (*Staphylococcus aureus*, *Escherichia coli*, *Klebsiella* spp, *Pseudomonas* spp) by applying the well diffusion technique was performed according to the standard method by Thitilertdecha et al., 2008 [36]. Microorganism cell suspension at 0.5 McFarland standard was used. Four concentrations; 0.5, 1, 2 and 5 mg/mL for compound was prepared by dissolving the cerium (IV) complex with 8-hydroxy in acetonitrile, were used for the assay. The sample volumes applied to the agar wells were approximately 20  $\mu$ l of each solution was inoculated onto wells were made in the spread plate culture of each microbial isolates (The plates were carried out in triplicates). All plate of the tested organisms was then allowed to incubate at 37°C for overnight. After 18- 24 h of incubation, each solution was noted for zone of inhibition for all isolates. The diameters of the zone of inhibitions were measured by measuring scale in millimeter (mm). While standard antibiotic drugs were used Ciprofloxacin (conc. 5 mg/mL) as a control.

### Results and discussion: Infrared Spectra

Figures 2 and 3 show the IR spectral bands of the 8-hydroxyquinoline ligand and its cerium complex. The IR spectral for ligand (8-HQ) (Figure 1) shows a broad band in 3050 - 3060  $\text{cm}^{-1}$ , which has been qualified into stretching vibration for  $\nu(\text{OH})$  phenol; however, this band vanished into the spectra (Figure 3) for generated complex, which referred coordination this band for metal ion. The absence of a band around 3000  $\text{cm}^{-1}$  due to the O-H stretching vibration of the free O-H group of 8-HQ is an important feature of the infrared spectra of metal complexes. This observation leads to the assumption that complex formation

occurs via deprotonation of the 8-HQ moiety's hydroxyl group. The  $\nu(\text{C}=\text{N})$  mode observed at 1621  $\text{cm}^{-1}$  in the spectrum of free HQ ligand is found to be shifted to 1637  $\text{cm}^{-1}$  in the complex spectra, indicating co-ordination via the tertiary nitrogen donor of HQ. A strong  $\nu(\text{CO})$  band observed in the complex spectra in the range of 1100-1110  $\text{cm}^{-1}$  indicates the presence of the complex's 8-hydroxyquinolate group coordinating through its oxygen atoms as uninegative bidentate ligand.

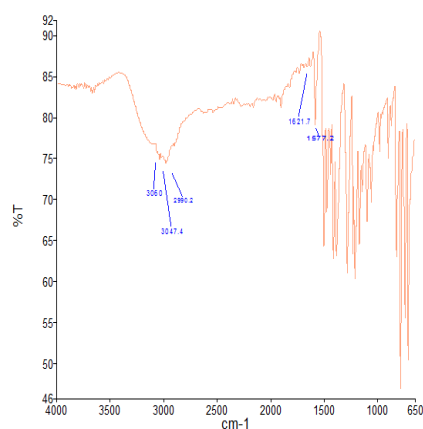


Figure 2. IR spectrum of 8-hydroxy quinoline

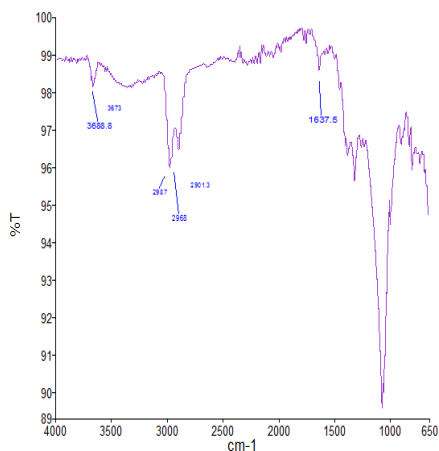


Figure 3. IR spectrum of cerium complex

### Electronic Spectra

In acetonitrile solution, UV-Visible spectra of the 8-hydroxyquinoline ligand and its cerium complex were measured in the wavelength range 200-450 nm as

shown in Figure 4. The ligand's UV-Visible spectrum shows two bands at 241 and 308 nm, which may be attributed to  $\pi-\pi^*$  and  $n-\pi^*$  transitions. The first band in the metal complexes was slightly redshifted to 253 nm, while the second band became more intense due to the coordination between cerium ion and 8-HQ ligand. However, a new band at 288

nm has been discovered, which can be attributed to metal-ligand charge transfer excitation.

Figure 5 depicts the change in complex absorption as a function of the concentration of the metal ion at the two absorption peaks. The increase in complex absorption is observed until the end point is reached.

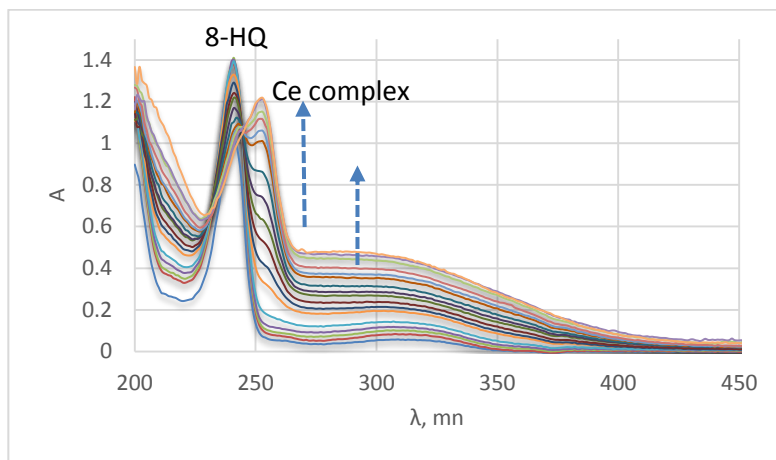


Figure 4. UV-VIS spectra of 8-hydroxy quinoline (8-HQ) and Ce complex

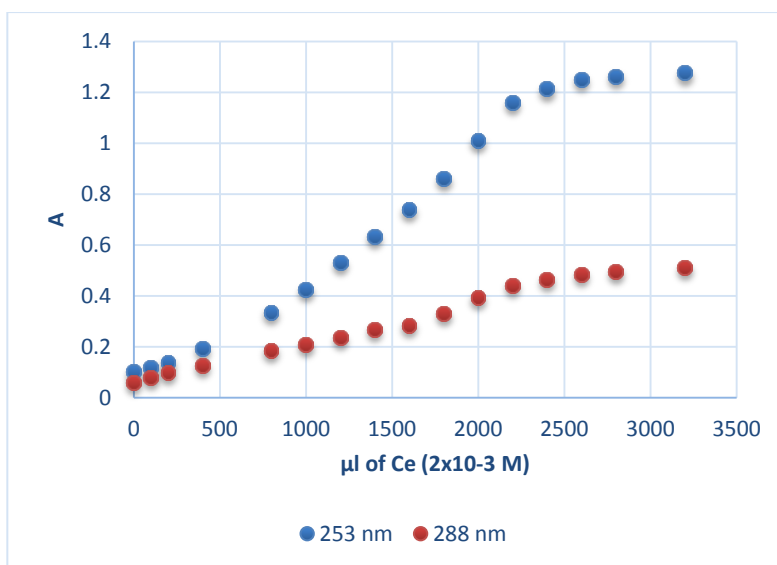


Figure 5. Titration of 8-HQ with Ce ion solution

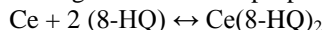
### Stoichiometry and Formation constant

The stoichiometric ratio of Ce(IV) ions to 8-HQ in the complex was

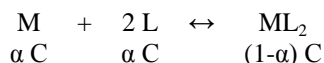
determined using the mole ratio method of [37,38]. The mole ratio plot (Figure 6) reached a maximum value at a mole ratio (L/M) of 2.0, confirming that the

complex's molar ratio between Ce(IV) ions and 8-HQ is 1:2.

The metal-ligand interaction proposed is:



The complex's formation constant  $K_f$  was calculated using the following equations:



Where C is the total molar concentration of the complex assuming no dissociation,  $\alpha$  is degree of dissociation and  $\alpha \text{C}$  and  $(1-\alpha) \text{C}$  are equilibrium concentrations of the metal, ligand, and complex, respectively.

The formation constant was calculated by the following equation:

$$K_f = \frac{[\text{ML}_2]}{[\text{M}]^x [\text{L}]^2}$$

$$K_f = \frac{(1-\alpha)}{4x\alpha^3 C^2}$$

The molar absorptivity ( $\epsilon$ ) of the complex and the absorbance of the complex's dissociated part ( $A_\alpha$ ) can be used to calculate a value of degree of dissociation  $\alpha$ .

A series of Ce(IV) standard solutions were prepared and complexed with 8-HQ at their optimal  $\lambda_{\text{max}}$ . Each standard solution's absorbance is measured. The obtained results show a linear relationship between absorbance and concentration in the  $1.0 \times 10^{-5}$  -  $5.0 \times 10^{-5}$  M range. The linear part of Beer's law plot is used to calculate molar absorptivity (Figure 7). Table 2 shows the values of complex stoichiometry, formation constant, degree of dissociation, and molar absorptivity.

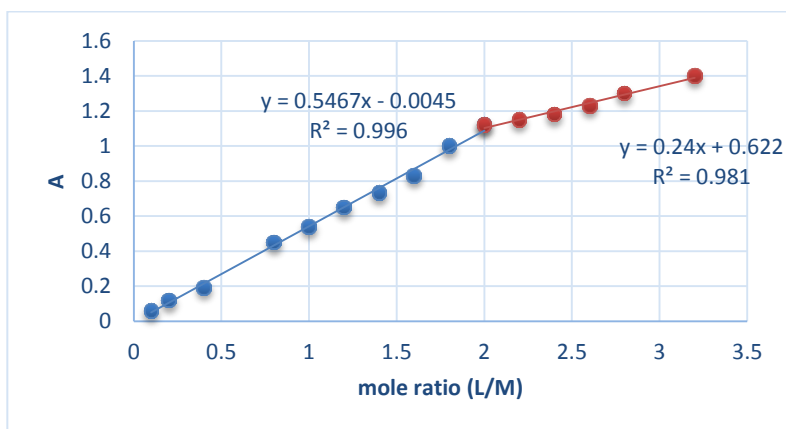


Figure 6. Mole ratio plot for the complex

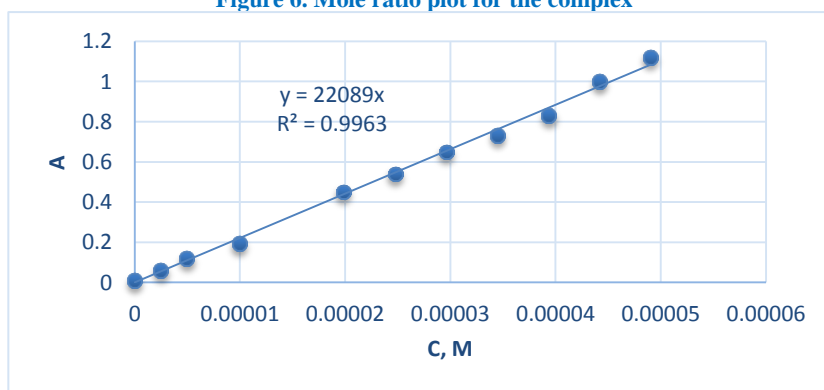


Figure 7. Calibration curve of the complex

**Table 1. Several parameters calculated for Ce(IV) complex**

Stoichiometry (M:L)	$A_{\alpha}$	$\alpha$	$\lambda_{\max}$ (nm)	$\epsilon$ ( $L \cdot mol^{-1} \cdot cm^{-1}$ )	$R^2$	Linear range ( $10^{-5}$ M)	$K_f$
1:2	0.010	0.009	253	22089	0.9963	2.0 – 5.0	$1.308 \times 10^{14}$

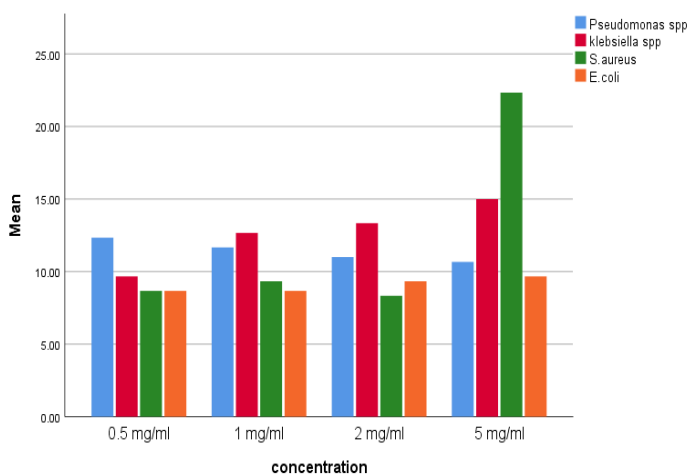
### Antimicrobial investigations

As shown in Table 2, the effects discovered a variable sensitivity of compound in the direction of microorganisms. The data confirmed that each of the investigated compounds has the best pastime against all of the investigated microorganisms. The complex of cerium (IV) with 8-hydroxy quinoline (conc. 5 mg/mL) provided the most effective activity against Gram-positive bacteria *S.aureus* and Gram-negative bacteria *Klebsiella* spp. Both (conc. 1-2 mg/mL) demonstrated maximum activity against *Klebsiella*spp,

while the 8-HQ with Four concentrations demonstrated lower inhibition zones against *Pseudomonas* spp., including (conc. 0.5 mg/mL), which demonstrated maximum inhibition zone (Figure 8). In conclusion, an evaluation of the spots among concentrations of cerium (IV) complex with 8-hydroxy quinoline suggests a more potent pastime with maximum Gram-terrible. Furthermore, statistical analysis of the results using ANOVA ONE WAY revealed no significant differences except for *Staphylococcus aureus*.

**Table 2. Analysis of variance (ANOVA) table for antibacterial activity**

		Sum of Squares	df	Mean Square	F	Sig.
E.coli	Between Gr.	2.25	3	0.75	0.47	0.71
	Within Gr.	12.67	8	1.58		
	Total	14.92	11			
S.aureus	Between Gr.	415.00	3	138.33	28.621	0.00
	Within Gr.	38.67	8	4.83		
	Total	453.67	11			
klebsiellaspp	Between Gr.	44.67	3	14.89	1.985	0.20
	Within Gr.	60.00	8	7.50		
	Total	104.67	11			
Pseudomonas spp	Between Gr.	4.92	3	1.64	0.25	0.86
	Within Gr.	52.00	8	6.50		
	Total	56.92	11			



**Figure 8. Graph of finding in relation to effective the cerium complex and 8-hydroxy quinoline**

### Conclusion

FT-IR and UV-VIS spectroscopy were used to characterize a cerium (IV) complex containing 8-HQ. According to IR spectra, the complexation between cerium ion and the ligand occurred via the oxygen and nitrogen atoms of 8-HQ. Furthermore, Ce(IV) ions reacted with 8-HQ in a molar ratio of 1:2 (M:L), as determined by the mole ratio method.

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The formation constant value ( $K_f = 1.308 \times 10^{14}$ ), which was determined using a spectrophotometric method, indicated that the formed complex was stable. Finally, cerium complex demonstrated the most potent antibacterial activity against Gram-positive bacteria *S.aureus* and Gram-negative bacteria *Klebsiella spp.*

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