

# Synthesis and Biological Activity of Dysprosium Lanthanide Complex with Azo Ligand

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## Abstract

**Background:** The present study describes complex reaction between 4-(2-Thiazolylazo)resorcinol (TAR) and Dy(III) metal ion. The TAR compound is known to form coloured chelates with various lanthanide metal ions. Researches done in the field of azo complexes is relatively little.

**Objective:** To Dysprosiumchloride was selected as a metallic salt, coordination complex of Dy(III) with TAR ligand were prepared, Dysprosium metal ion get chelates as it react with TAR. It's been recognized for along interval of time that the azo group has the ability to develop a coordinate fitting with metal ions. Dy(III) complex was identified via FT-IR, UV-Vis. spectroscopy and measuring the electrical properties. The purpose of the research applied in the field of dyes

**Patients and Methods:** The complex [Dy(TAR)<sub>2</sub>].Cl have been characterized by elemental analysis (CHN). TAR ligand and its complex were further studied using <sup>1</sup>H-NMR and <sup>13</sup>C-NMR techniques. Spectra of this complex shows that the TAR compound behaves as a tridentate manner via (ONN) atoms.

**Results:** The antibacterial activity of free ligand and so Dy(III) complex were investigated. The composition of the chelate was found to be 1:2 (M:L). Dy(III) metal ion was six coordinate the analytical results of the complex show the ratio of stoichiometry and complex showed octahedral geometry around metal ion.

**Conclusion:** All results indicates that the Dy complex have the chemical formula [Dy(TAR)<sub>2</sub>].Cl The overall number of points of attachment to the central element is termed the coordination number. The study of biological activity shows that the complex is more effective than the ligand.

**Keywords:** Trivalent metal ion, spectral studies, chelate azo reagent lanthanide, antibacterial activity.

## OPEN ACCESS

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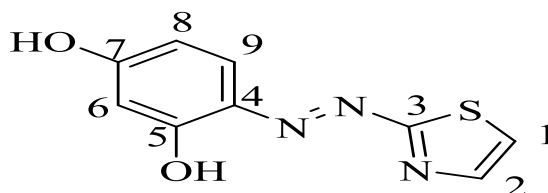
## Introduction

The term azo dyes is used to those synthetic aromatic organic colourants that are characterized by the existence of the chromophoric azo group (-N=N-). Most of these compounds are monoazo compounds such as 4-(2-Thiazolylazo)resorcinol. Substance. The TAR ligand has essential role

in preparation of many complexes [1-3], the azo ligand have additionally been extensively searched. The reaction products are heavily tinted as well as show a particular form of complicated dye stuff that is extensively explored for its distinct (mainly optical) residential or commercial buildings.

Suggested that TAR ligand [4] behaves as a spectrophotometric reagent[5,6], because it creates dark red coloured chelates with

Dysprosium (III) metal ion and also some lanthanide metal ions in basic medium [7], Figure(1) illustrate TAR ligand structure.



**Figure(1):** 4-(2-Thiazolylazo)resorcinol (TAR) ligand

TAR has actually been used in the spectrophotometric resolution of many metals as a colour-forming representative. Just recently, the development of brand-new reactive colour structures has distressed passion, as well as abundant brand-new frameworks valuable in a commercial application, azo compounds have received much attention due to their versatile use in many important industries such as textile dyeing silk, wool, and cotton, along with their blends with various other fibers, in addition to other areas as in cosmetics industries, food coloring and electronic devices[8]. The lanthanides have a number of chemical qualities that distinguish them from the d-group metals. The aspects have greater power than the modification metals. The 4f orbitals in the lanthanide(III) ions are well secured by the 5S<sub>2</sub> and 5P<sub>6</sub> orbitals, so they do not involve straight in bonding[9-11]. Their spectroscopic and magnetic properties are thus greater unaffected by the ligand. These compounds are important to understand the mechanism of the magnetic interaction between 3d and 4f orbitals cores [12]. The many-sided ligational behaviour of azo compounds had excite considerable interest in the past.

The construction of a synchronization complex that can be used as an improvement

thinks about dyeing techniques is plainly one of the most essential elements to enhance the add-on, along with communications in between the azo-dyes as well as the fabric. Premetallized dyes with a number of colour bits coordinated with a metal ion are known as metal challenging dyes. Azo-Dye fragments are composed of added teams like hydroxyl, carboxyl, or amino, which can create a limited coordination facility with change metal ions. Metal ions have been used as a mordant in dyeing techniques. In synchronization applications, the principles of complexation, as well as mordent, are similar, other than that complexation needs chelating the metals in the dye structure, before application which is described as premetallized colour[12-15]. The present investigation aims to synthesize one of the lanthanide complexes and determine the coordination number of a Dy(III) central metal ion in the prepared complex, since the coordination numbers of these metals between 6-12, with chelating ligands a good example of this type of ligands is TAR compound. The benefit from knowing the coordination number which determines the type of hybridization and the geometrical shape of the complex. Which leads us to know the stability of the complex by other futuristic studies.

## Patients and Methods

### Instruments

UV-Vis. spectra were obtained in methanol on (Shimadzu-1800 UV-Vis. spectrophotometer double beam). The conductivity of the complex was measured in Dimethylsulphoxide sing (pH / conductivity meter ) at room temperature. IR- spectra were taken on SHIMADZU, Fourier Transeform Infrared Spectroscopy FT-IR spectroscopy Mod IR Affinity-1CE, in which solid samples were taken as a disc KBr special for spectroscopy in 4000-400 cm<sup>-1</sup> range . The studies were measured in the Faculty of Education Chemistry Department Salahaddin University – Erbil city, Iraq. Melting points determined by an Electrothermal melting point device 9100LTD(UK) and are uncorrected. The antibacterial studies were conducted in the College of Education Salahaddin University – Biology Department-Erbil-Iraq. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR were measured on a broker 400MHz Ultra-shield with Tetramethylsilane(TMS) as internal references, in Jordan University of Science and Technology- P.O.Box3030 Irbid 22110 Jordan.

### Materials

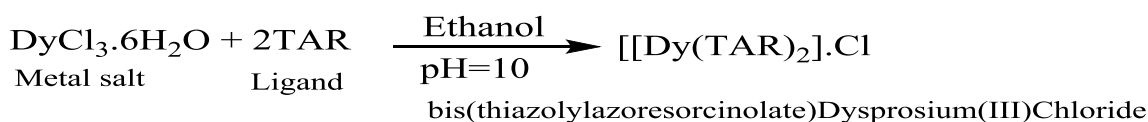
All reagents were commercially available and used as received from suppliers, Dysprosium used in the form of chloride salt without further purification .The azodye is currently available for purchase from BDH, organic solvents were used as received, absolute ethanol, methanol, DMSO, Sigma-

Aldrich Co. Ammonium acetate, ammonia solution and distilled water were used when preparing a buffer solution.

### Synthesis of Dysprosium complex with (TAR) Ligand

A solution of ligand 4-(2-Thiazolylazo) resorcinol TAR 2-mmol (0.442g) in 10 ml ethanol was added in the form of drops with stirring to the 1mmol metal salt DyCl<sub>3</sub>.6H<sub>2</sub>O.(0.376g) dissolved in the buffer solution pH equal to 10. The resulted mixture was protected at room temperature for 24 hrs ,a deep red precipitate was acquired , collected by filtration and washed with diethyl ether many times and dried in desiccator to give 79% yield of the output complex (m.p. 280oC ).Anal. Cal. For DyC<sub>18</sub>H<sub>12</sub>N<sub>6</sub>O<sub>4</sub>S<sub>2</sub>.Cl: C, 33.85; H, 1.88; N, 13.16 , Found: C,33.77; H,1.88; N, 13.11, Dy,25.47. The molar conductivity is 155 S.cm<sup>2</sup>.mol<sup>-1</sup> suggesting 1:1 electrolyte nature of Dysprosium complex. The percentage of Dy(III) metal ion was determined by volumetric analysis using EDTA[16,17] .

For determining the stoichiometry of the complex Job's method were used[18] .The ratio of metal to the ligand is 1:2, a series of metal salt and ligand solutions which were prepared from their 10<sup>-3</sup> mol/L in ethanol their absorbances were measured by UV-Vis. technique which agreed with CHN analysis. Scheme (1) expresses chemical reaction of complex formation .



Scheme.1. Reaction equation

## Results

### IR spectra

The IR data of ligand and dysprosium complex summarized in Table (1) shows the i.r data of the TAR ligand and its metal complex in the 4000-400  $\text{cm}^{-1}$  range. The TAR reagent is tridentate bind to the metal ion Dy(III) with three donor positions of the resorcinol OH, azo N, and thiazole N, [19,20].C=N stretching frequency of the thiazole nitrogen is noted as a high strength band at  $1631\text{cm}^{-1}$  in the HL ligand, according to IR spectra. The C=N band in Dysprosium complexes is then moved to lower frequencies  $1589\text{cm}^{-1}$ , suggesting it was influenced by coordination to Dysprosium ions.

The shift of the C-O stretching band, which was noted at  $1269\text{cm}^{-1}$  in the HL ligand, to  $(1209-1193)\text{cm}^{-1}$  in the dysprosium complex, confirms the participated of the deprotonated phenolic hydroxyl group in chelation. The band corresponding to the OH

group at  $3412\text{cm}^{-1}$  shows that the ligand contain hydroxyl group. The -N=N- stretching band in the TAR molecule is detected at  $1519\text{cm}^{-1}$ ; however, this band is moved to a lower vibration  $(1508-1487)\text{cm}^{-1}$ , implying azo group coordination [21,22]. The complex peaks found at  $(420-452)\text{cm}^{-1}$  in the IR spectra of Dysprosium complex be appointed to the M-N stretching frequency of the nitrogen azo and thiazole nitrogen, respectively. The infrared spectra of the ligand (TAR) are thoroughly interpreted, as is the effect of bounding with Dy (III) ion on the vibrational frequencies of the TAR reagent. The displacement of the C-O stretching band, noted at  $1238\text{cm}^{-1}$  in the totally TAR ligand, for  $(1209-1193)\text{cm}^{-1}$  in the Dysprosium facility, validates the participation of the deprotonated phenolic hydroxyl group in binding, Figure (2) and (3) shown TAR and Dysprosium complex i.r spectra respectively.

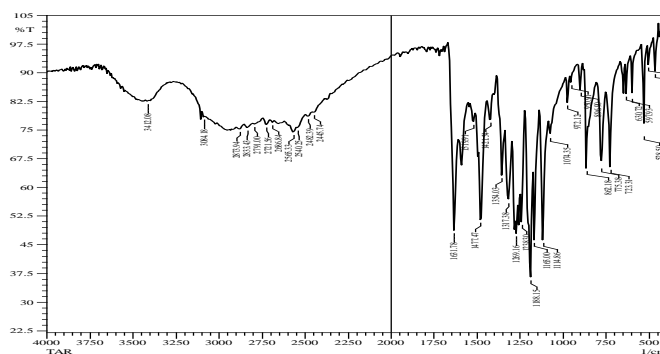
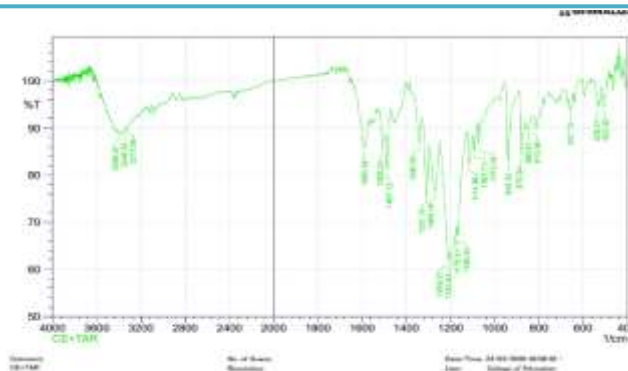


Figure (2): Infrared spectrum of 4-(2-Thiazolylazo)resorcinol TAR Ligand



**Figure (3):** Infrared spectrum of bis-(thiazolazoresorcinolate)Dysprosium(III)chloride [Dy(TAR)<sub>2</sub>].Cl complex

**Antibacterial Activity study**

Inhibition zone including the disc diameter was measured for each treatment after 5 days of incubation . The metal complex [Dy(TAR)<sub>2</sub>].Cl were tested against the bacterial species Staphylococcus aureus (Sa) and Escherichia Coli (E.Coli). Chloramphenicol as a standard antibacterial agent or reference was evaluated for their antibacterial activity and the result was compared with its Dy(III) complex. The comparison of the biological activities of the synthesized complex and free ligand shows that the azo ligand (TAR) and its metal

complex have higher antibacterial effect than that of the standard and Dy(III) complex have more biological activity than the ligand and standard , such increased activity of the metal chelates can be explained based on chelation theory . On chelation the polarity of the metal ion will be reduced to greater extent due to the overlap of the ligand orbitals and partial sharing of the positive charge of the metal ion with donor groups , and possible electron delocalization over the whole chelate ring [31,32].The results of biological activity are given in Table ( 2 ).

**Table (1):** Infrared data of the TAR ligand and it’s metal complex Dy(III) in cm<sup>-1</sup>

Compounds	C=N	N=N	C-O	M-N	M-O
1- TAR ligand	1631	1587	1278	-----	-----
2- Dy(III) complex	1589	1508	1209-1193	420-452	503-536

**Table( 2 ):**Effect of varied concentration of TAR ligand and Dy(III)complex on the mean radial growth (in cms)

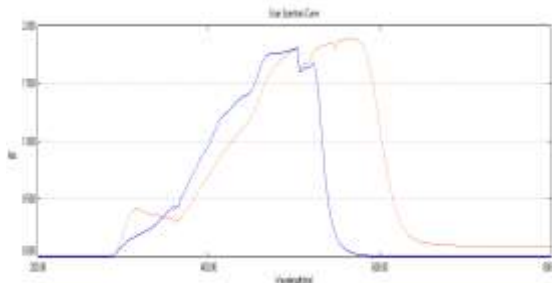
Compound	<i>Staphylococcus</i>			<i>Escherichia Coli ( E.Coli)</i>			
	40mg/10ml	30mg/10ml	20mg/10ml	40mg/10ml	30mg/10ml	20mg/10ml	
TAR	0.6	0.9	1		0.7	1	1
[Dy(TAR) <sub>2</sub> ].Cl	0.8	1.3	1.8		0.9	1.2	1.5

## Discussion

### Electronic Spectra

In absolute ethanol, the UV-Visible spectra of the HL reagent as well as the Dy(III) complex were measured in  $1 \times 10^{-3}$  methanol. In the ultraviolet region, HL's electronic spectra shows four bands. The band forms in

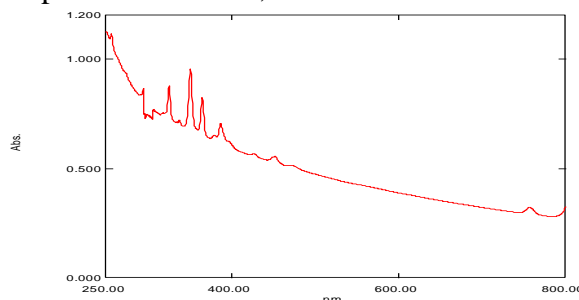
the 350-358 nm range due to the HLs  $\pi-\pi^*$  transformation. The band seen in the visible region between 550 and 545 nm, as well as 450 nm (shoulder), is attributed to n-  $\pi^*$  digital change[23-25].Figure (4). Shown combined spectra of TAR & Dy<sup>3+</sup> complex.



**Figure (4):** Combined UV-Vis. spectra of TAR and Dy(III) complex in  $1 \times 10^{-3}$  M methanol solution. A- Red Dy(III) complex B-Blue TAR Ligand

The 4f-electrons of lanthanides output two types of transitions such as f-d and f-f transitions. The f-f transitions which appear sharp, narrow bands, absorption spectra of the lanthanide elements are thus typically sharp as opposed to the broad absorptions of the transition metals[26], comparatively weak intensities which are Laporte forbidden,

whereas allowed f-d transitions are relatively wide and intense, however, the 4f orbitals in the lanthanides are placed deep within the atom and the broadening effect of ligand vibrations is minimized [27]. The absorption spectrum of Dy<sup>3+</sup> cation is shown in Figure(5).



**Figure (5):** UV-Vis. spectrum of Dyspronsium(III)metal ion

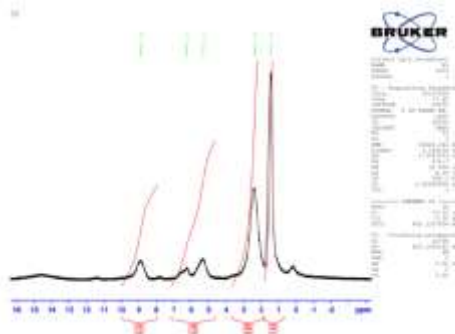
### <sup>1</sup>H-NMR and <sup>13</sup>C-NMR For TAR Ligand

The <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra of TAR ligand record in DMSO-d<sub>6</sub>, further substantiates the mode of coordination suggested by the electronic and (i.r) spectral studies. The <sup>1</sup>H-NMR spectrum of the ligand

a distinct singlet signal at  $\delta$  11.29-10.87 ppm displays to the presence of proton of hydroxyl group. The multiplets of aromatic proton in TAR ligand appeared within the range  $\delta$  7.95-7.64 ppm. Chemical shift for protons linked to the thiazole ring and

resorcinol ring appears in the range at  $\delta$  6.46-7.95 ppm [28]. The signals of the  $^1\text{H-NMR}$  spectra as well as the important bands in the IR spectra are having been and discussed in contact to molecular structure. When comparing the nmr spectrum of complex with

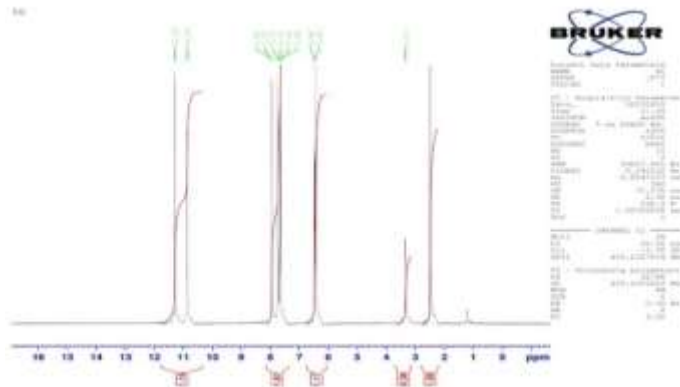
ligand it is observed that the signal of  $-\text{OH}$  group disappears in the complex spectrum this indicates the participation of the oxygen atom of hydroxyl group in the coordination after deprotonation as shown in Figure (6).



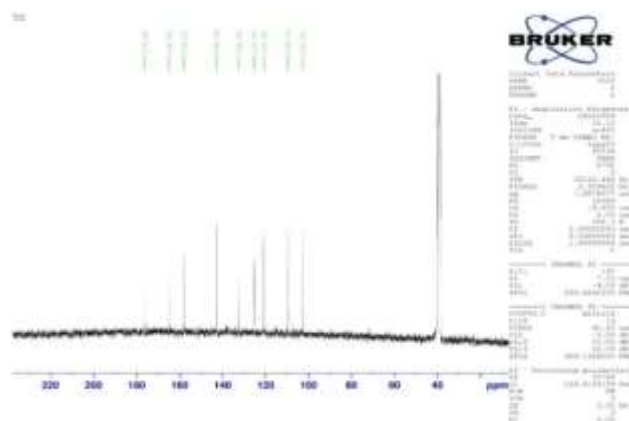
**Figure (6):**  $^1\text{H-NMR}$  spectrum of Dy(III) complex with TAR ligand

$^{13}\text{C-NMR}$ . Phenyl ring carbons gives signals with chemical shift values from 100 to 200 ppm.  $^{13}\text{C-NMR}$  spectrum shows nine signals corresponding to nine types of carbon atom were fitted to. ( $\text{C}_4$ :102.91,  $\text{C}_6$ :109.74,  $\text{C}_8$

:120.85,  $\text{C}_1$ :125.40,  $\text{C}_9$ : 132.50,  $\text{C}_2$ :142.96,  $\text{C}_5$ :158.05,  $\text{C}_7$ :164.91 and  $\text{C}_3$ :176.28) [29,30].  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectra of the TAR molecule are given in Figure (7&8) respectively.



**Figure (7):**  $^1\text{H-NMR}$  spectrum of TAR Ligand

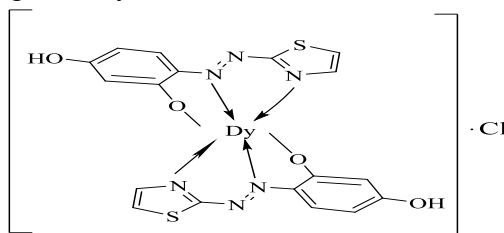


**Figure (8):** <sup>13</sup>C-NMR spectrum of TAR Ligand

### Conclusions

All results indicates that the Dy complex have the chemical formula [Dy(TAR)2].Cl The overall number of points of attachment to the central element is termed the coordination number .The coordination number of a complex is influenced by the relative sizes of the metal ion and the ligands and by electronic factors, such as charge which is dependent on the electronic configuration of the metal Dy= [Xe]6S24f10 the 4f orbitals transfuse the xenon core considerably . Because of this, they cannot overlap with ligand orbitals and therefore do not share safely in bonding. The Dy(III) complex possess octahedral geometry around

the central metal ion, chloride ion behave as anion counterion to balance the charge of the complex which agreed with molar conductivity value of prepared complex. Lots of research on lanthanide metals and their complexes have been done and numerous research articles are published until to day. Lanthanide ions and their compounds received the industrial and academic attention due to their great medicinal significance. In the most of cases, the inhibition capacity increase on using the [Dy(TAR)2].Cl complex rather than the TAR free ligand . Figure (9) shown the geometrical shape of 4-(2-Thiazolylazo) resorcinol complex.



**Figure (9):** Proposed structure of the Dy(III) complex with TAR ligand

### Recommendations

Azo complexes are used to make a number of azo dyes and pigments which are used in a

variety of industries. These commercial dyes have the benzene group or its derivatives attached instead of the usual nitrogen. Azo



dyes give bright, high intensity colours, much more so than the next most common dye class (anthraquinones). They have fair to good fastness properties, but not so good as the carbonyl and phthalocyanine classes. Azo dyes are widely used in the food, pharmaceutical, cosmetic, textile, and leather industries. Azo dyes represent the largest production amount of dye chemistry today, and their relative importance may even increase in the future. They play a crucial role in the governance of the dye and printing market. These dyes are synthesized from a simple method of diazotization and coupling. Different routes and modifications are made to obtain the desired color properties, yield and particle size of the dye for improved dispersibility.

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**Ethical clearance:** Ethical approval was obtained from the College of Medicine / University of Diyala ethical committee for this study.

**Conflict of interest:** Nil

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## تخليق و الفعالية البيولوجية لمعدد دايسبراسيوم اللانثانيد مع الليكاند الأزو

كامران بشير حسين<sup>1</sup>، سنكر صباح صابر<sup>2</sup>

### الملخص

**خلفية الدراسة:** الدراسة الحالية تبين تفاعل التنسيق بين ٤- (٢- ثايوزول أزو) ريزورسينو TAR و الايون الفلزي Dy(III). من المعروف ان المركب TAR يكون معدد مخلبي مع مختلف الايونات الفلزية للثنائات .  
**اهداف الدراسة:** تم اختيار الملح الفلزي كلوريد الدايسبروسيوم و تحضير معدد تناسقي بين Dy(III) و TAR.  
**المرضى والطرائق:** تم اختيار الملح الفلزي كلوريد الدايسبروسيوم و تحضير معدد تناسقي بين Dy(III) و TAR.  
من المعروف منذ زمن طويل ان مركبات الأزو لديها القدرة على تطوير مركبات تناسقية مع الأيونات الفلزية . تم تشخيص المعدد بواسطة الأشعة تحت الحمراء و الأشعة فوق البنفسجية – المرئية وقياس الخواص الكهربائية . تم دراسة المعدد [Dy(TAR)<sub>2</sub>].Cl بواسطة التحليل الدقيق للعناصر CHN ، تمت دراسة الليكاند و المعدد أيضا باستخدام تقنيات <sup>1</sup>H-NMR و <sup>13</sup>C-NMR

**النتائج:** تظهر الدراسات الطيفية ان المركب TAR يتصرف كليكاند ثلاثي السن عبر ذرات (ONN) ،الفعالية الب بيولوجية لمعدد Dy(III) و الليكاند الحرتم انجازها .النسبة المولية للمعدد ١:٢ (M:L) النتائج التحليلية ونسبة اتحاد العناصر للمعدد تبين شكل هندسي ثماني السطوح حول الأيون الفلزي .

**الاستنتاجات:** تشير جميع النتائج إلى أن مركب Dy له الصيغة الكيميائية [Dy(TAR)<sub>2</sub>].Cl ويسمى العدد الإجمالي لنقاط الارتباط بالعنصر المركزي رقم التنسيق. وتبين دراسة النشاط البيولوجي أن المركب أكثر فعال.

**الكلمات المفتاحية:** الأيون الفلزي ثلاثي التكافؤ ، دراسات طيفية ، كاشف الأزو المخلبي ، اللانثانات، الفعالية البيولوجية

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