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# Preparation and identification of new Chalcone- Azo ligand (SACAI) and its complexes.

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Keywords: - Chalcone - Azo , Azo compounds, amino chalcone, transition metal complexes.

**Abstract:** A new organic ligand (Chalcone-azo ligand), (2E)-1-(4-((4,5-diphenyl-1H-imidazol-2-yl)diazenyl)phenyl)-3-(2hydroxyphenyl) prop-2-en-1-one (SACAI) with its divalent (Co, Ni and Cu) complexes were synthesized. The azo-derivative was prepared via the preparation of amino chalcone first, then diazoating of this amine and coupling with (4, 5diphenylimidazole) component.

Three transition metal ion complexes were prepared from this legend, after the determination of (M: L) ratio beyond the ultraviolet-visible studies of the (ligand-ion) mixtures. The M: L ratios for all complexes were (1:2).

The amino-chalcone, new ligand (SACAI) and its complexes were identified with the available techniques such (Mass spectroscopy for the ligand, elementary analysis, Atomic Absorption, UV-Visible spectroscopy, F.T.I.R spectroscopy), electrical molar conductivity and Magnetic susceptibility for the solid complexes. From the results obtained, we can suggest the non-ionic octahedral geometry for all complexes. The new ligand acting as a bidentate chelating ligand that coordinating from the N3 imidazole atom and the far azo heterocyclic ring nitrogen atom. تحضير و تشخيص ليكاند آزو-جالكون الجديد مع بعض معقداته سعد مدلول مهدي قسم الكيمياء /كلية العلوم/ جامعة بابل

# الخلاصة:

*-(2E)*تم في هذه البحث تحضير ليكاند عضوي جديد لمركب الازو-جالكون *1-(4-(4,5-diphenyl-1H-imidazol-2-yl)diazenyl)phenyl)-3-(2hydroxyphenyl) prop-2-en-1-one* (SACAI) *من* معقداته لعناصر (الكوبلت والنيكل والنحاس) ثنائية الشحنة . حيث تم تحضير مركب (الكوبلت والنيكل والنحاس) ثنائية الشحنة . حيث تم تحضير مركب اولا ومن ثم ازواج ملح الديازونيوم للامـين مع (SAC)الجالكون الاميني في وسط قاعدي كحولي (4,5-Diphenyl imidazole)قاعـدة الازواج بدرجة الصفر المئوي.

حضرت المعقدات الصلبة الثلاث بعد تثبيت الظروف من التركيز الامثل والطول الموجي ، وحسـاب النسـبة المولية اللازمة لتحضير تلك المعقدات بعد دراسـة اطياف الاشـعة (الفوق بنفسـجية – المرئية) ، وكانت النسـبة مي (1:2,M:L).

ومعقداته (SACAI) والليكاند الجديد (SAC )شخص الجالكون الاميني بالتقنيات الممكنة كطيف الكتلة لليكاند الجديد وتقنيات ( التحليل العناصري الدقيق ومطيافية الاشعة الفوق بنفسجية – الحمراء ومطيافية الاشعة تحت الحمراء وطيف الانبعاث الذري) اضافة لاستخدام التقنيات اللاخرى ( كالتوصيلية المولارية والحساسية المغناطيسية ) للمعقدات الصلبة ، واثبتت خلاصة النتائج ومحصلتها امتلاك المعقدات جميعها الصفة اللاايونية واثبتت خلاصة النتائج ومحصلتها امتلاك المعقدات جميعها الصفة اللاايونية واشكل الهندسي ثماني السطوح خلال ارتباط الليكاند ثنائي المخلب وذرة نتروجين الازو البعيدة عن الحلقة Na

# Introduction:

The importance's of organic compounds lie in the type of constituent groups, utilized most manufacturing industries<sup>1</sup>. though how them in Azo compounds and their derivatives can regard one of them; due to they're entry in many manufacturers as (paints<sup>2</sup>, dyes<sup>3</sup>, pesticides<sup>4</sup>, food colorants<sup>5</sup> and in lubricant oil improvements<sup>6</sup>), as well as in the chemistry fields as a precursor for many organic compounds and the important organic chelating compounds, that have the ability of coordinating with a huge number of determination in different samples<sup>7</sup>. elements for trace amount

Azo compounds have a biological activity toward the microbial8; recent studies (Congo red and Evans blue) had been studied as an HIV inhibitor of viral replication, that believing in the bonding between the azo group and the protease & reverse transcriptase <sup>9</sup>. Chalcone the precursor of flavonoids<sup>10</sup>, pyrazoline<sup>11</sup> and other compounds, chalcones indeed their conjugation importantly, it acts as a non-azo dyes<sup>12</sup> compound and its one of the most important compounds that enter as drug source<sup>13</sup>, in pharma logical activities (antitumor<sup>14</sup>, anticancer<sup>15</sup> scavenger<sup>16</sup>). as and radical However, when the two components were meeting in one compound, we will see a dearth of sources, due to a lot of research working with this side<sup>17,</sup> <sup>18</sup>. This activated us to work hard in this field.

#### **Experimental**

The entire chemicals used in this work were of analytical reagent grade from (Fluke, BDH and Sigma), 4, 5-dipheny imidazole was a laboratory synthesized with a high grade of purification.



(2E) - 1 - (4 - ((4,5 - diphenyl - 1H - imidazol - 2 - yl)diazenyl) phenyl) - 3 - (2 - hydroxyphenyl) prop - 2 - en - 1 - one

#### Schem1.General steps in SACAN preparation.

- Mass Spectra was taken with MSD Direct Probe using Acq Method low energy. M, Metal analysis was performed by Shimadzu 6800AA (Flame Atomic Absorption Spectrophotometer. Elemental analysis (C.H.N) of compounds was carried out by (Micro Analytical unit, 1108 CHN Elemental analyzer), UV-Visible study and electronic spectra were recorded on a Shimadzu (UV-Visible

1650) spectrophotometer using a quartz cell. FTIR Spectra were recorded by Shimadzu FTIR8400 test scan series using the KBr disc in the wave number range (4000-400cm<sup>-1</sup>), magnetic susceptibility measurement was determined by Faraday's method using Magnetic Susceptibility Balance Model – M.S.B Auto. The molar conductivity was measured at room temperature in two solvents (ethanol and dimethyl formamide) in  $1 \times 10^{-3}$ M ligand concentration using (Info lab terminal). Melting points were determined using Saturates digital SPM30.

#### Synthesis of the amino chalcone<sup>19</sup>:

Amino chalcone (*E*)-1-(4-aminophenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one SAC) was prepared via reaction of ( p-aminoacetophenone, 0.01mole , 1.35gm) and ( salicyaldehyde, 0.01mole, 1.22 gm = 1.06ml) in absolute ethanol (30ml), then stirring and cooling at 5C°, within 30min. Dropping of (3M KOH) solution to the reaction component with stirring till the solution began more thickening, with continuing the stirring for 24hrs. Decant this thickened solution in iced bath and neutralized with dil. HCl, the yellow precipitant appear, filter and wash with deionized water several times to remove the trace of acid and salt formed, then air drying the precipitant, recrystallize the product with hot ethanol.

#### Synthesis of the Chalcone- azo (SACAI):

The Chalcone- azo was prepared according to Shibata method<sup>20</sup>, the ligand ((E)-1-(4-((Z)-(4,5-diphenyl-1H-imidazol-2-yl)diazenyl)phenyl)-3-(2hydroxyphenyl)prop-2-en-1-one SACAI) was synthesized by preparation of diazonium salt of the amino-chalcone (SAC) by dissolving (5mmole,1.195gm) of this amine in an acidic mixture (8ml conc. HCl + 15ml D.W) and cooling in  $0C^{\circ}$  water bath, then (5mmole, 0.35qm NaNO<sub>2</sub> in 10ml D.W ) solution was added carefully to the ammine with stirring at the same temperature, within 15min the diazonium salt was formed. The coupling component (4,5-diphenyl imidazole) was prepared by dissolving (5mmole, 1.1 gm) in alkaline alcoholic solution (2M NaOH). Then transferred in the ice cold bath, the daizonium salt was added dropwise to the coupling component with stirring at  $0C^{\circ}$ , a reddish-orange color was formed, complete the process until the end of daizonium solution, then neutralization of the pH value to complete the precipitation of the azo compound, the precipitant was filtered washed many times to remove the salt traces then drying and recrystallized with a hot ethanolic solution. A deep reddish-orange powder was collected.

#### Synthesis of the complexes

All the divalent transition metal element (Co, Ni and Cu) complexes were prepared with the same procedure, that:

In 100 ml rounded bottom flask 10 mmole of (SACAI) was dissolved in 40 ml absolute ethanol and refluxed with 5mmole of metal chloride (that dissolved in the minimum amount of absolute ethanol); they refluxed for 1hr. With stirring, then the solvent reduced to the quarter in hot water bath, then the hot solution, transferred to crash ice bath, the colored precipitant was developed, then collected, dried and recrystallized with hot ethanol.

Compound	M.Wt g/mol	Color	m.p C°	Yield
SAC	239.09	Yellow	116-118	77%
SACAI	470.17	Red-orange	120-122	72%
[Co(sacai) <sub>2</sub> Cl <sub>2</sub> ]	1069.22	Deep brawn	149-151	75%
[Ni(sacai) <sub>2</sub> Cl <sub>2</sub> ]	1068.22	Maroon	151-153	70%
[Cu(sacai) <sub>2</sub> Cl <sub>2</sub> ]	1075.49	Deep violet	172-174	78%

## Table.1 Physical characteristic of the ligands and its complexes

# Table.2 Elementary analysis for SACAI and its complexes

Compound	<b>C</b> %	H %	N %	M %
-	calculated	calculated	calculated	calculated
	(Found)	(Found)	(Found)	(Found)
SAC	75.30	5.48	5.85	
	(75.39)	(5.50)	(5.90)	
SACAI	76.58	4.71	11.91	
	(76.62)	(4.74)	(11.98)	
[Co(sacai) <sub>2</sub> Cl <sub>2</sub> ]	67.29	4.14	10.46	5.50
	(67.36)	(4.17)	(10.48)	(5.58)
[Ni(sacai) <sub>2</sub> Cl <sub>2</sub> ]	67.31	4.14	10.47	5.48
	(67.41)	(4.16)	(10.51)	(5.55)
[Cu(sacai) <sub>2</sub> Cl <sub>2</sub> ]	67.01	4.12	10.42	5.91
	(67.13)	(4.15)	(10.51)	(5.98)

#### **Results:-**

Because of the scientific novelty in the entrance of this subject, we found it necessary to go into these fields to prepare new organic legends that were many important functional groups approved the board outlook in the vital area and the others like (medical, pharmaceutical and industrial).

In this work a new chalcone- azo was synthesized by two steps:

Firstly the amino chalcone (SAC) was synthesized by the Cleasien – Schmidt condensation of the p-amino acetophenone with salicyladehyde in 10% alkaline alcoholic media to form the amino chalcone (SAC), the TLC (petroleum: ethyl acetate 4:1) with  $R_f = 0.28$  indicate the completion of the reaction.

The certainty of chalcone preparation can be enhanced with the FTIR spectral data, which the appearance of the functional group of the compound in their regions gives us confidence to the formation; the main bands are:

-The hydroxyl group of the salicyaldehyde gives str. band at 3396 cm<sup>-1</sup>, while the primary amine gives two adjacent str. bands at 3332 & 3226 cm<sup>-1</sup> respectively.

-The appearance of the chalcone bands clearly at (1647cm<sup>-1</sup> & 1514 cm<sup>-1</sup>) related to the carbonyl and methylene groups<sup>21</sup>, as shown in the following figure 1. :



# Fig. 1 FTIR Spectra for (SAC).

- The second step of the process, including the preparation of the azo compound (SACAI) as a brand new organic compound by the reaction of the diazonium salt of the amino chalcone (SAC) with the coupling component (4,5-diphenyl imidazole).

The brand new azo (SACAI), was identified with (CHN,FTIR and Mass spectroscopy ) to ensure the compound formation, the approaching of the found elementary(CHN) data to the theoretical calculated, with the assistance of the FTIR spectral data helps with reliability of preparation (as shown below):



Fig. 2 FTIR Spectra for (SACAI)

The disappearance of the amine bands and the appearance of the azo group (N=N) band in the 1452cm<sup>-1</sup> region<sup>22</sup>, the remaining of the other bands within the limit, i.e. that the formation of this azo, while the chalconic hydroxyl group was combined with the (N-H) imidazole group in the (3417cm<sup>-1</sup>) region, this will be shown with the ligand complexes, too.

**Mass spectroscopy**, is the analytical tools that used for compound identification, by their ability to sorts ions based on their mass (weight), one of the mass spectrum usage to determine the masses of the particles and of molecules, and to elucidate the chemical structure of the molecules<sup>23</sup>.

The mass fragmentation of (SACAI) gives clearly the site of the mother ion band at (470.1 m/e), this agreed with the molecular mass of (SACAI), other fragments of this azo compound was included within the following figure (as the 4,5-diphenyl imidazole fragment (220 m/e)).



## Fig.3 SACAI Mass Spectra

The new ligand (SACAI) was soluble in many solvents such (ethanol, methanol, DMF, DMSO, Acetone, chloroform, carbon tetrachloride, and acetonitrile) while its insoluble in water.

#### UV-Visible spectroscopy:-

A wide range of concentration will be done in this study to determine the optimal concentration for the ligand, and their divalent transition metal ion complexes ( $Co^{2+}$ ,  $Ni^{2+}$  and  $Cu^{2+}$ ), the concentration range ( $10^{-3}-10^{-4}$  M) gives out of range results, deep and condense colors that excluded from the study, while within( $10^{-5}$  M) ranges, gives clear solutions obey (beer-lambert law).

(SACAI) give an observable  $\lambda_{max}$  at (424nm) this related to the charge transfer via azo group, while the second absorption band to the  $(\pi - \pi^*)$  transition, a red shifting and vivid color observed when these ions aqueous solutions were

mixed with the alcoholic ligand solution, due to the coordination between the ions and ligand<sup>24</sup> take place, as shown:



Fig. 4 SACAI UV-Visible spectroscopic data

The UV-Visible study utilized to determine the no. of a mole used in the preparation of the complexes, by using the mole ratio method <sup>25</sup>, its suitable and easy method for soluble complex determination <sup>26</sup>.

From the results obtained, the mole ratio (M: L) was (1:2) for the three (SACAI) complexes. This will be shown in fig. below.



# Fig.4 Mole ratio curves for SACAI Complexes.

The mole ratio results deduced the stability of the complexes, by using the relation  $\alpha = \frac{A_m - A_s}{A_m}$ , where A<sub>s</sub>= the absorbance at the mole ratio, A<sub>m</sub> = the absorbance with a ligand component increment. a= dissociation constant.

 $\beta=\frac{1-\alpha}{4\alpha^3c^2}$  , for (1:2) ratio,  $\beta=$  stability constant and c = the

concentration of the two components (ligand &metal ion) in the study.

Metal ion Complex	A <sub>m</sub>	A <sub>s</sub>	a	β	Logβ
[Co(sacai) <sub>2</sub> Cl <sub>2</sub> ]	0.742	0.732	0.052	2.09X10 <sup>11</sup>	11.32
[Ni(sacai) <sub>2</sub> Cl <sub>2</sub> ]	0.740	0.732	0.108	5.43X10 <sup>11</sup>	11.73
[Cu(sacai) <sub>2</sub> Cl <sub>2</sub> ]	0.759	0.755	0.0052	7.18X10 <sup>12</sup>	12.85

# Table.3 complexes stability data

These results agreed with Irving-Williams Series<sup>27</sup> that the stability of complexes formed by divalent first-row transition metal ions generally increases across the period to a maximum stability of copper:

Mn (II) < Fe (II) < Co (II) < Ni (II) < Cu (II) > Zn(II).

The solid (SACAI) complexes were synthesized, according to their mole ratio results, and then identified with the elementary analyzer to determine their (CHN) component and atomic absorption to the metal percent in complex.

Their FTIR spectral data showed changes in two functional groups (the azo group N=N and the imidazole azomethine group C=N) by their shifting in their values due to coordination with these ions, and remaining the other groups within their limits. This will indicated that they were not participated in coordination), the M-Cl band doesn't appear due to the limitation of KBr disk working within 400cm<sup>-1</sup> while this band appear within 300 cm<sup>-1</sup> region<sup>28</sup>, and this well illustrated in the following table:

Compound.	v(N-H)	v(C- H)	v(C=N) imd	v(N=N)	M-N <sup>28</sup>
SACAI	3417	3061	1595	1458	
[Co(sacai) <sub>2</sub> Cl]	3417	3061	1598	1442	453
[Ni(sacai) <sub>2</sub> Cl]	3417	3061	1598	1450	449
[Cu(sacai) <sub>2</sub> Cl]	3417	3059	1585	1448	443

# Table.4 FTIR data for (SACAI) Complexes.

\*all values measured in cm<sup>-1</sup>.



And observed in the following figures:







() SHIMADZU



#### Electrical molar conductivity:-

Complex ionic formula can know by the electrical molar conductivity for them<sup>29</sup>, that the number of electrolyte species proportion with the conductivity<sup>30</sup>, and approach to zero value in non-electrolyte solutions.

All complexes have a non-ionic character due to the depression of their conductivity values in two solvent (ethanol and dimethyl formamide).

Table.5 conductivity values of the (SACAI) Complexes.

Metal ion	Conductivity in ethanol	Conductivity in DMF
Complex		
[Co(sacai) <sub>2</sub> Cl <sub>2</sub> ]	13	15
[Ni(sacai) <sub>2</sub> Cl <sub>2</sub> ]	19	22
[Cu(sacai) <sub>2</sub> Cl <sub>2</sub> ]	22	25

## Magnetic & electronic properties:-

Magnetic susceptibility data act as a complementary part to suggest the complexes geometry (in particular for TME's)  $^{31}$ , via the study influence of the partially filled outer orbitales, magnetic data supplied us with the electronic contribution and the metal oxidation state, that the number of odd electron(s) for the metal play a role which either complex in the high - spin state or low.<sup>32</sup>

- The cobalt complex has a 4.15 B.M value; this indicates with the abundance of three odd electrons of octahedral cobalt complexes <sup>33,</sup> the increment value returned to the orbital contribution.
- While the nickel complex gives a 3.02 B. M, this will agree with octahedral nickel complexes that have two odd electrons<sup>34</sup>.
- Copper susceptibility value was 1.89 B.M; this was tuned with the odd electron octahedral copper complexes as in literature<sup>35</sup>

The electronic spectral data shown in the following table:

Complex	Transition type	Transition value in
		Cm⁻¹
[Co(sacai) <sub>2</sub> Cl <sub>2</sub> ]	${}^{4}T_{1q(F)}$ ${}^{4}T_{1q(P)}$	18761
	${}^{4}T_{1q(F)} {}^{4}A_{2q(F)}$	16949
	${}^{4}T_{1q(F)} {}^{3}C_{2q(F)}$	9175
[Ni(sacai) <sub>2</sub> Cl <sub>2</sub> ]	${}^{3}A_{2g(F)}$ ${}^{3}T_{1g(P)}$	21928
	${}^{3}A_{2g(F)}$ ${}^{4}T_{1g(F)}$	14662
	${}^{3}A_{2q(F)}$ ${}^{4}T_{2q(F)}$	7444
[Cu(sacai) <sub>2</sub> Cl <sub>2</sub> ]	$^{2}E_{g}$ $^{2}T_{2g}$	14597
	_	

# Table.6 electronic spectral data for (SACAI) Complexes

These data agree with the literature octahedral complexes of the transition metal elements <sup>32</sup>.

From the study associative data, the (CHN, Mass spectra, FTIR) and the enhancement of (UV-Visible) to determine the ratio of solid complexation, Indeed to the (conductivity, magnetic and electronic spectra), give an evidence to suggest the octahedral geometry for all the three complexes for the new ligand (SACAI), acting as a bidentate

chelating ligand by the imidazole N3 atom and the far azo nitrogen atom.



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Some abbreviations mentioned in this research: **SAC**: salicyldehyde aminoacetophenone chalcone. **SACAI**: salicyldehyde aminoacetophenone chalcone AZO 4,5-Diphenyl imidazole.

The novelty of this work according to REAXYS.

