

Solvent extraction of copper (II) from aqueous solutions by new organic reagent 2-[(3-Hydroxy phenyl)azo]-4,5- diphenyl imidazole

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Abstract

This study include synthesis of new organic reagent 2-[(3-Hydroxy phenyl)azo]-4,5-diphenyl imidazole (3-HPADPI) and used it as complexing agent for extraction of Cu^{2+} ion from aqueous solutions. The results show the optimum $\text{pH}_{\text{ex}}=8$, optimum concentration of Cu^{2+} ion was $40\mu\text{g}$ in 5mL aqueous phase and the optimum shaking time was 10min . Stoichiometry study illustrated the complex structure was $1:1 \text{ Cu}^{2+}:(3\text{-HPADPI})$. Effect of cations interferences was studied. Synergism effect show MIBK gave increasing of distribution ratio (D). The study of organic solvents effect appear there is not any linear relation between dielectric constant (ϵ) of organic solvents used and distribution ratio (D). Thermodynamically appear that the reaction was Endothermic reaction with $\Delta H_{\text{ex}}=0.000062 \text{ KJ.mole}^{-1}$, $\Delta G_{\text{ex}}=-51.46 \text{ KJ.mole}^{-1}$, $\Delta S_{\text{ex}}= 159.31 \text{ J.mol}^{-1}.\text{K}^{-1}$.

1. Introduction

Today around 25% of the world's copper was recovered by using solvent extraction and solvent extraction was considered to be the lowest cost production route for the production of the quality cathode. Chelating complex was prepared for Cu(II) ions with organic reagent 4-(2-pyridyl azo)-resorcinol the UV.-Vis. spectrum of reagent solution in methanol show $\lambda_{\text{max}}= 410\text{nm}$ where the complex of Cu^{2+} show $\lambda=477\text{-}534\text{nm}$ [1]. Spectrophotometric determination of Cu(II) by using diamine-dioxime organic reagent, meso-HexaMethyl Propylene Amine Oxime (meso-HMPAO). A stable complex was formatted between Cu(II) and meso-HMPAO in aqueous solution 1:1 red-pink. Absorption measurements were carried at 497 nm , with $\epsilon= 338 \text{ Lmol}^{-1}\text{cm}^{-1}$. Beer's law was obeyed over the range of $0.5\text{-}370 \mu\text{g mL}^{-1}$ with a Sandell's sensitivity $0.18 \mu\text{g cm}^{-2}$. This method was applied for determination of Cu(II) in foodstuffs and pharmaceutical samples [2]. Spectrophotometric procedure for quantitation of Cu(II) based on the reaction between alizarin red S and boric acid in the solution of pH 8.5 to produce a red complex which place between organic and aqueous phases. In presence of Cu(II) the complex of alizarin red S and boric acid is dissociated and new complex between alizarin red S and Cu(II) ions formed. Beer's law obeys in the range $0.6\text{-}15 \mu\text{g.mL}^{-1}$. Effect of foreign ions was observed that all ions were interfered even at low concentrations because of formation a complex between alizarin red S and that ions [3]. A spectrophotometric method for determination of Fe(III), Cu(II) and $\text{UO}_2(\text{II})$ in aqueous solutions by the reaction of those cations with the reagent 2-ethanolimino-2-pentylidino-4-one (B3). Under the optimum reaction conditions, B3 reacts with these cations and forms colored complexes. The color reaction is rapidly completed

and the absorbance remains stable for at least a week at room temperature. The Fe(III) complex is detected at $\lambda_{\max}$ = 440nm and pH=3.5, the Cu(II) at $\lambda_{\max}$ =340nm and pH=6.0, while the UO₂(II) at $\lambda_{\max}$ =370 nm and pH=4.0. Bear-Lambert's law is obeyed in the range (2–17 mg/mL for Fe(III), 3–9 mg/mL for Cu(II) and 13–81 mg/mL for UO₂(II) complexes. The stoichiometries of the formed complexes are determined using different spectrophotometric methods[4]. A potentiometric and spectrophotometric study of Cu(II) mixed reagent complexes was involving hydroxamic acids as primary reagent and histamine or the amino acids glycine and histidine secondary reagent. All are potentially able to form chelate complexes with either five or six membered rings. It was evidenced that Cu-hydroxamate complexes do not favour significantly mixed reagent complex formation with amino acids having only the carboxylate and amino groups; however, these complexes are strongly formed with histidine, due to the presence of the imidazol group. [5]. Prepared a new chelating resin by coupling Amberlite XAD-4 with 1-amino-2-naphthole through an azo spacer. The resulting sorbent has been characterized by FT-IR, elemental analysis and thermogravimetric analysis and studied for preconcentrating of Cu (II) using FAAS for metal monitoring, and the optimum pH for sorption of the Cu²⁺ was 6.5[6]. The significant spectral overlap (Di)0.5 = 0.5667 which is about 75.3% overlapping of the UV/Vis. absorption spectra of Fe (II) and Cu(II) complexes necessitates chemometric assisted methods for analysis of these ions in the pharmaceutical mixture. These metal ions were analyzed simultaneously by UV/Vis. spectrophotometric method where 8-hydroxyquinoline was used as a chromogenic reagent. The methods were applied for analyzing synthetic mixtures and commercial pharmaceutical preparation [7]. The synergistic extraction of Cu (II) with N-phenyl benzohydroximic acid (PBHA) and tri-n-octyl phosphin oxide was investigated in CHCl₃. The effect found in presence of natural reagent is due to formation of the adduct Cu(PBHA)₂S in CHCl₃. The synergic coefficients were determined which have higher value in presence of TOPO than TBP, since TOPO has the higher basicity than TBP [8]. Determination of Cu(II) by using 2,3,4-trihydroxy acetophenone phenyl hydrazone (THAPPH) as a spectrophotometric method. The metal ion has formed 1:2 bluish green coloured complex with THAPPH in HCl-KCl buffer of pH 2.5. Beer's law was obeyed in the range 0.04-0.64 $\mu\text{g/mL Cu}^{2+}$ at $\lambda_{\max}$ =385 nm. ϵ =1.0053 $\times 10^5 \text{ L.mol}^{-1}\text{cm}^{-1}$ and Sandell's sensitivity (0.0006265 μgcm^{-2}). The interfering effect of various cations and anions was also studied. The reliability of the method was assured by analyzing the standard alloys, Brass, Bronze, and Phosphor-Bronze. This method was employed for the determination of Cu(II) in food and medicinal leafy samples and inter compared the experimental values using AAS [9].

2. Experimental

2.1. Apparatus

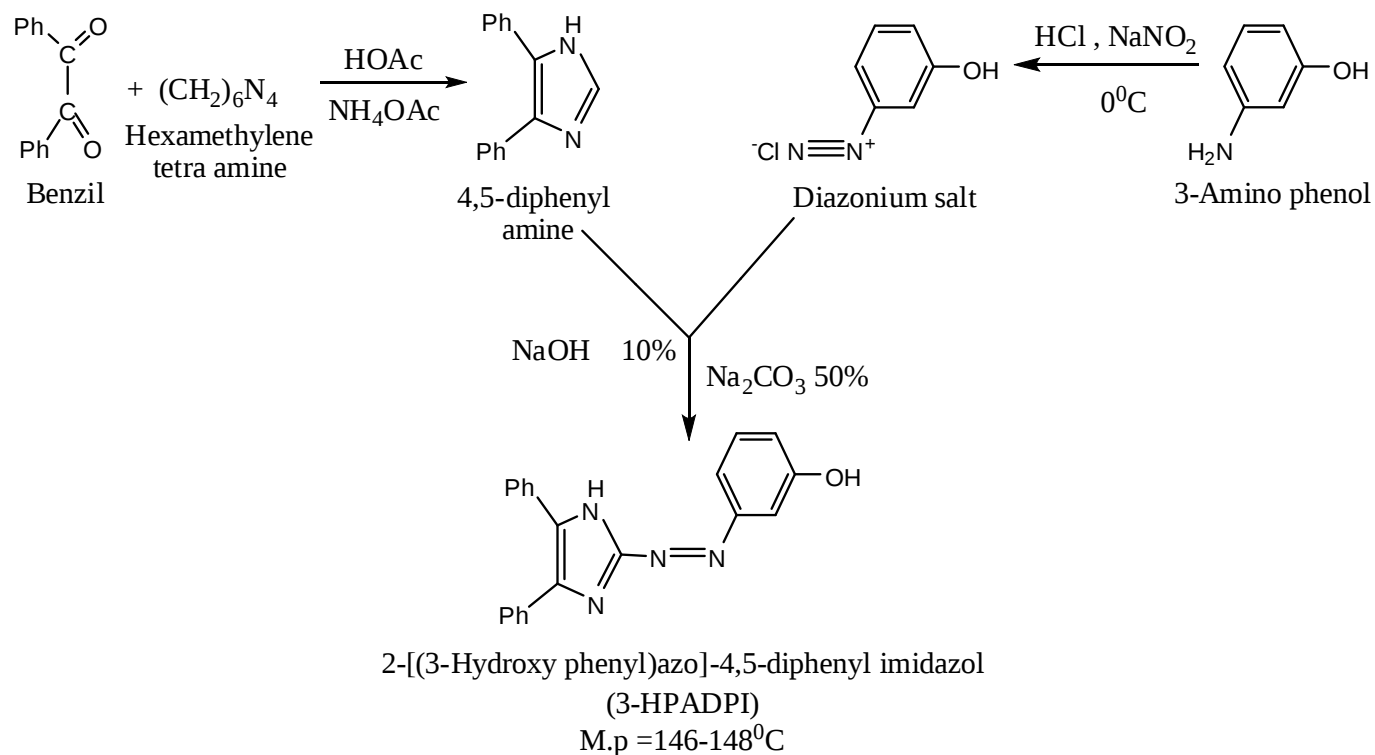
Spectrophotometric measurements used double beam (Uv.-Vis.) Spectrophotometer, Shimadzu UV.1700 (Japan), single beam (Uv.-Vis.) Spectrophotometer, Shimadzu (UV.100-02) ,(Japan), pH measurement carried out by pH –meter , WTW CE ,E163694, (Germany), Melting point measured by Stuart Scientific COLTD,220-240 (Britain). As well as for studied the structure of organic reagent prepared used FT-IR 8400 S(CE), Shimadzu corporation. Element analysis Carried out by Micro analytical unit, 1108 C.H.N elemental analysis.

2.2. Reagents

Stock solution 1mg/mL by dissolved 0.3928gm from $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Merck) in 100mL distilled water contain 1mL of concentration H_2SO_4 , $1 \times 10^{-2}\text{M}$ solution of dithiazone prepared by dissolved 0.0256gm in 10mL of carbon tetrachloride (B.D.H). The organic reagent solution $1 \times 10^{-2}\text{M}$ prepared by dissolving of 0.034gm in 10mL of chloroform(Merck). Other working solutions prepared by appropriate dilution with suitable solvent.

2.3. Synthesis of organic reagent

The first step of synthesis of organic reagent 3-HPADPI was prepared imidazole derivative by add 100mL of glacial acetic acid to mixture of 4.2gm of benzil and 1.4gm of hexamethylene tetra amine and 12gm ammonium acetate then reflux for 1hour, afterward add 400mL distilled water and chilling at last by add 40mL ammonium hydroxide to precipitate imidazole compound, then wash the precipitate with distilled water and reconstituted from pyridine, the second step preparation of diazonium derivative by dissolved 2.2gm from 3-Amino phenol in mixture of 2mL concentration HCl and 10mL distilled water and 10mL ethanol and chilling the mixture to 0°C , after that add 7mL from 10% sodium nitrite drop by drop with shaking and without rising temperature than 5°C , the third step preparation of organic reagent ,to solution of imidazole compound prepared by dissolved 2.2gm in 150mL ethanol and 50mL of 10% sodium hydroxide and 50mL of 50% sodium bicarbonate add solution of diazonium drop by drop at -5°C to precipitate the organic reagent and wash with distilled water and reconstituted in 1:1 ethanol: water.⁽¹⁰⁾



3. General procedure

Extraction experiments were carried out for Cu^{2+} ions in 5mL aqueous solutions by 5mL of organic reagent 3-HPADPI dissolved in chloroform at $1 \times 10^{-4}\text{M}$, and shaking the two layers for fixed time, after that separate the two layers and determine remainder quantity of Cu^{2+} in aqueous phase spectrophotometrically by (dithiazone method)[10], also determine Cu^{2+} ions transferred to the organic phase form complex, at later calculate distribution ratio (D).

4. Results and Discussion

4.1. Spectrophotometric studies

Table (1): Analytical ^a, UV-Vis. Spectra ^b [11, 12,13], IR spectral ^c [14,15,16]

Compound	Analysis %					UV-Vis	Infrared cm ⁻¹								
	C	H	N	O	Cu	λ nm	C-H	O-H	C=N	-N=N-		Cu-	SO ₄ ⁼	H ₂ O	
C ₂₁ H ₁₆ N ₄ O	74.10	4.74	16.46	4.70	-	422	2900 2980	3566	1600 1508	1450, 1475	696	-	-	3200- 3395	
[Cu ²⁺ (C ₂₁ H ₁₆ N ₄ O)] SO ₄ ⁼	62.44	3.99	13.87	3.96	15.73	435	2920 2854	3550	1643	1427 1371	600	700 - 800	1100	3387	

4.2. Effect of pH

The results show distribution ratio (D) enhanced with pH increasing as well acidic media are not suitable for extraction because the protonation of coordinate position in reagent molecule in addition basic pH value not suitable too. Where is the optimum value was $\text{pH}_{\text{ex}}=8$. As in Table (2) and Figure (5).

4.3. Effect of Metal ions concentration

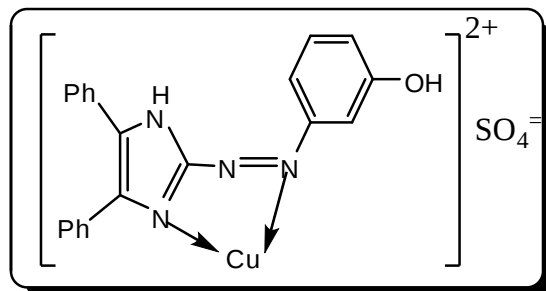
Decrease metal ion concentration in aqueous phase cause decreases in stability of ion pair complex this lead to decrease of distribution ratio (D). While the increase of metal concentration cause of increasing in distribution ratio (D) according to Lieshatlier principle. The results show optimum concentration of Cu^{2+} was $40\mu\text{g Cu}^{2+}$ in 5mL aqueous phase as in Table (3) and Figure (6).

4.4. Effect of Shaking Time

Kinetic study show distribution ratio (D) change as a function for shaking time, and appear 10min. was favorable shaking time which is giving higher distribution ratio (D) as in Table (4) and Figure(7).

4.5. Stoichiometry

Three methods (Slope analysis method, Mole ratio method, Continuous Variation Method) were applied to ascertain the stoichiometric composition of complex and the 1:1 $[\text{Cu}(\text{3-HPADPI})]^{2+}\text{SO}_4^{=}$ complex was indicated by these methods as in Tables (5,6,7) and Figure (8, 9, 10).



Probable structure of Cu^{2+} complex with organic reagent 3-HPADPI

4.6. Effect of foreign ions

Results in Table (8) show in presence of some cations in aqueous phase the distribution ratio (D) was decrease from its value in absence of these ions.

4.7. Synergism effect

Extracted $\text{Cu}(\text{II})$ from aqueous phase by organic reagent (3-HPADPI) in presence methyl isobutyl Keton (MIBK) the results in Table (9) and Figure (12) show distribution ratio (D) enhanced by presence MIBK also one molecule participate in coordination shell of complex $[\text{Cu}(\text{3-HPADPI})(\text{MIBK})]^{2+}\text{SO}_4^{=}$.

4.8. Organic Solvent effect

Results in Table (10) show there is not any linear relation between dielectric constant (ϵ) and distribution ratio (D) that there is an effect for organic solvent structure on extraction method.

4.9. Temperature effect

Thermodynamic study show the reaction between Cu^{2+} ion and organic reagent 3-HPADPI was endothermic, as the results in Table (11) and Figure (13, 14). After that calculate extraction constant K_{ex} by the relation below:

$$K_{\text{ex}} = \frac{D}{[\text{M}^{n+}]_{\text{aq.}} [\text{3-HPADPI}]_{\text{org.}}}$$

Latter plot $\log K_{\text{ex}}$ versus $1/T \text{ K} \times 10^{-3}$ from the slope of the straight line determine thermodynamic data according to [17]:

$$\text{Slope} = \frac{-\Delta H_{ex}}{2.303R}$$

$$\Delta G_{ex} = -RT \ln K_{ex}$$

$$\Delta G_{ex} = \Delta H_{ex} - T\Delta S_{ex}$$

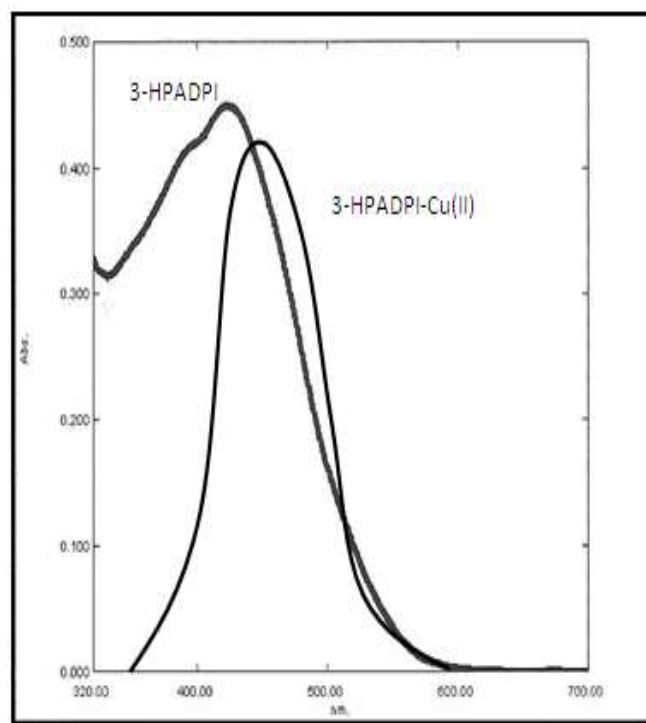


Figure (1): UV.-Vis. Spectrum of organic reagent 3-HPADPI & Cu^{2+} complex with organic reagent 3-HPADPI

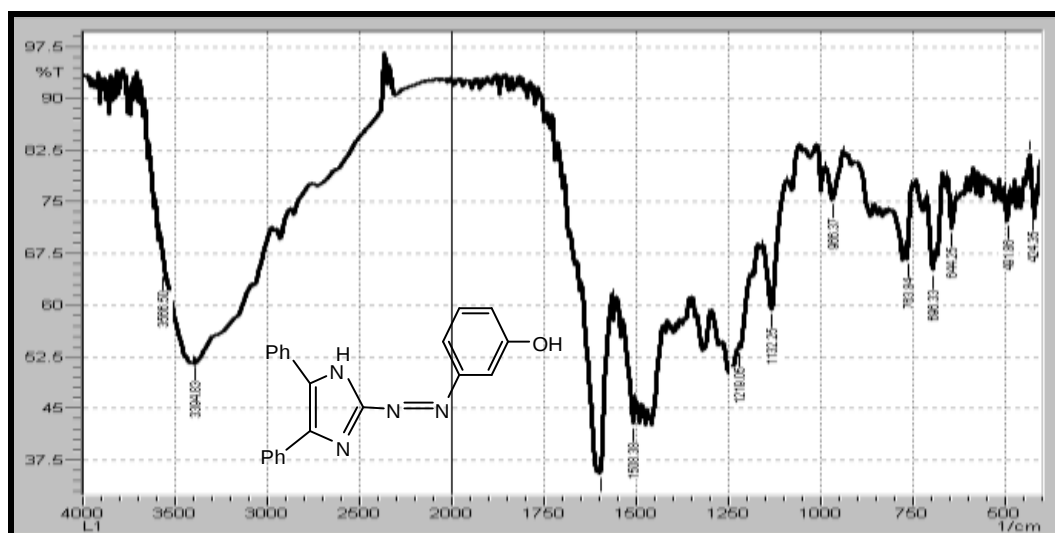


Figure (2): Infrared Spectrum of organic reagent 3-HPADPI

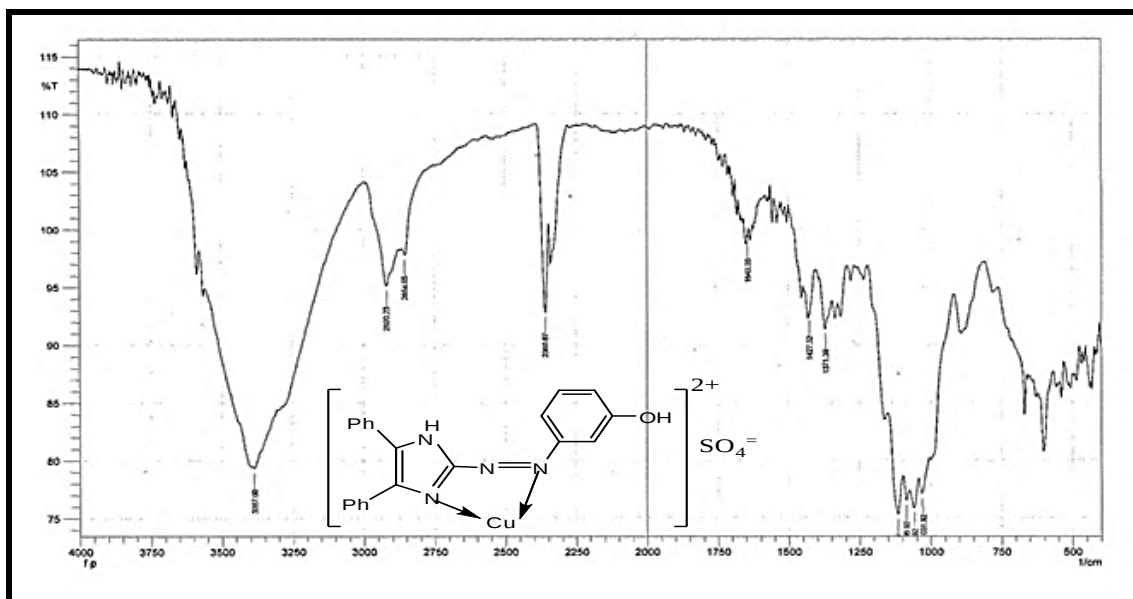


Figure (3): Infrared Spectrum of Cu^{2+} complex with organic reagent 3-HPADPI

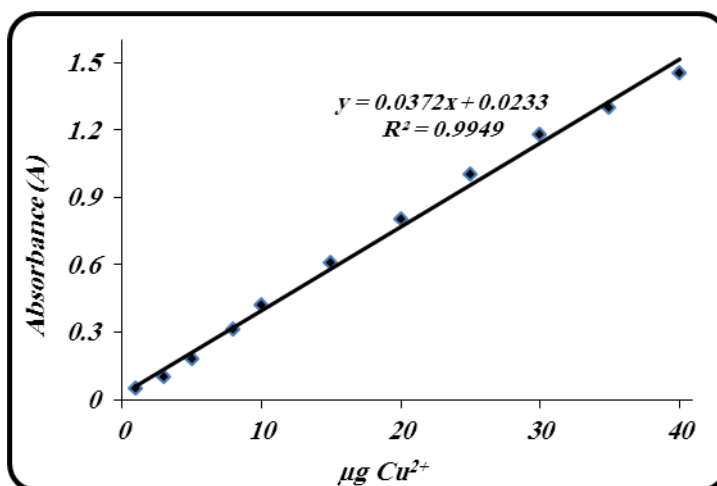


Figure (4): Calibration curve of Cu^{2+} with dithiazone

Table (2): Effect of pH on distribution ratio (D)

pH	D	%RSD	%E
5	5.01	0.19	83.36
6	6.31	0.15	86.32
7	9.00	0.11	90.00
8	19.00	0.05	95.00
9	9.00	0.10	90.00

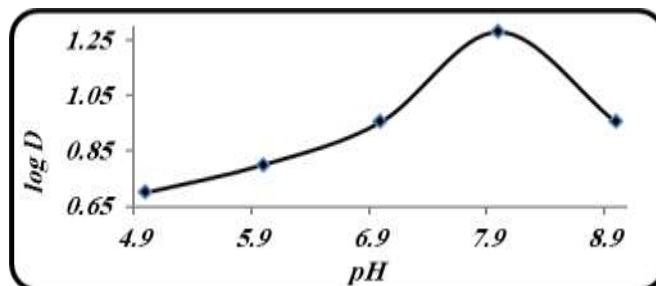


Figure (5): Effect of pH on distribution ratio (D)

$[Cu^{2+}] = 40 \mu g/5mL$, $[3-HPADPI] = 1 \times 10^{-4} M$ in $CHCl_3$, Shaking time=10min., Temp.=25°C

Table (3): Effect of Cu^{2+} concentration on distribution ratio (D)

$\mu g Cu^{2+}$	D	%RSD	%E
10	0.30	3.33	23.07
20	1.66	0.60	62.40
30	2.63	0.38	72.45
40	19.00	0.05	95.00
50	5.01	0.19	83.36
60	1.99	0.50	66.61

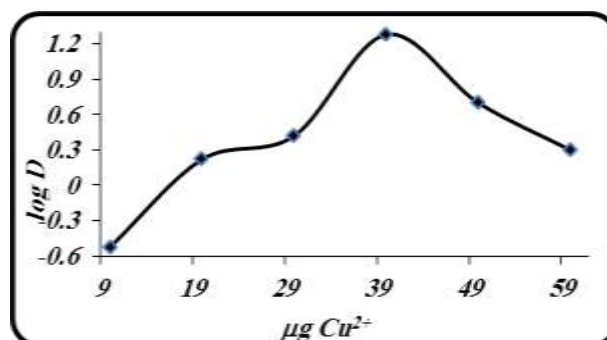


Figure (6): Effect of Cu^{2+} concentration on distribution ratio (D)

pH=8, $[3-HPADPI] = 1 \times 10^{-4} M$ in $CHCl_3$, Shaking time=10min., Temp.=25°C

Table (4): Effect of Shaking time on distribution ratio (D)

Time	D	%RSD	%E
5	4.00	0.25	80.00
10	19.00	0.05	95.00
15	5.01	0.19	83.36
20	2.51	0.39	71.50
25	2.00	0.5	66.66

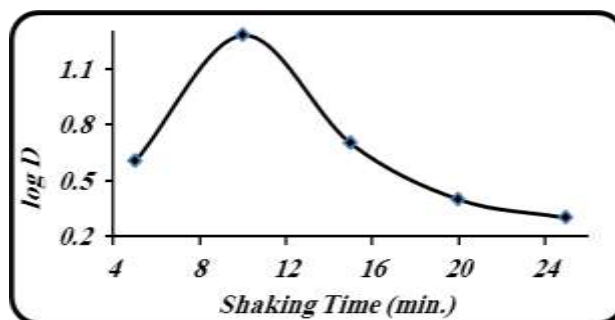


Figure (7): Effect of Shaking time on distribution ratio (D)

$[Cu^{2+}] = 40 \mu g/5mL$, pH=8, $[3-HPADPI] = 1 \times 10^{-4} M$ in $CHCl_3$, Temp.=25°C

Table (5): determination the stoichiometry of extracted complex (Slope analysis method)

[3-HPADPI]	D	%RSD	%E
5×10^{-3}	38.01	0.02	97.43
1×10^{-3}	30.20	0.03	96.79
5×10^{-4}	25.3	0.04	96.19
1×10^{-4}	19.00	0.05	95.00
5×10^{-5}	16.5	0.06	94.28
1×10^{-5}	10.46	0.09	91.27

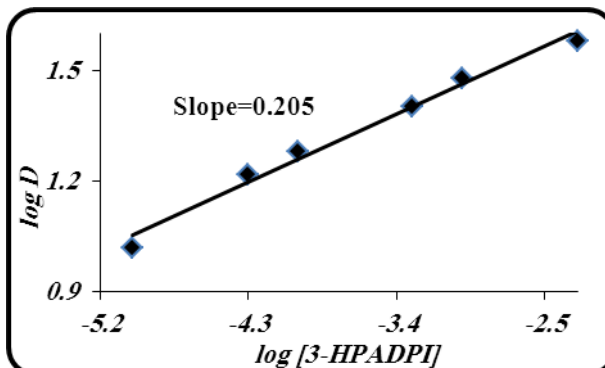


Figure (8): determination the stoichiometry of extracted complex(Slope analysis method)

$[\text{Cu}^{2+}] = 40 \mu\text{g}/5\text{mL}$, pH=8, Shaking time=10min., Temp.=25°C

Table (6): determination the stoichiometry of extracted complex (Mole ratio method)

C_L	C_L/C_M	Abs.	%RSD
1×10^{-6}	0.008	0.06	16.60
5×10^{-6}	0.040	0.20	5.00
1×10^{-5}	0.080	0.75	1.33
5×10^{-5}	0.400	0.90	1.11
1×10^{-4}	0.800	0.97	1.03
5×10^{-4}	4.000	1.10	0.90
1×10^{-3}	8.000	1.18	0.84

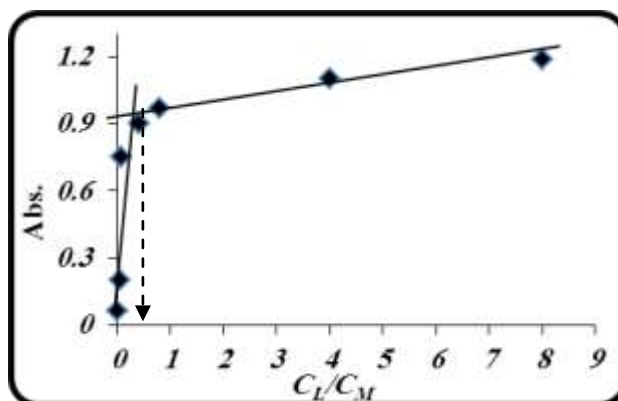


Figure (9): determination the stoichiometry of extracted complex(Mole ratio method)

$[\text{Cu}^{2+}] = 40 \mu\text{g}/5\text{mL}$, pH=8, Shaking time=10min., Temp.=25°C

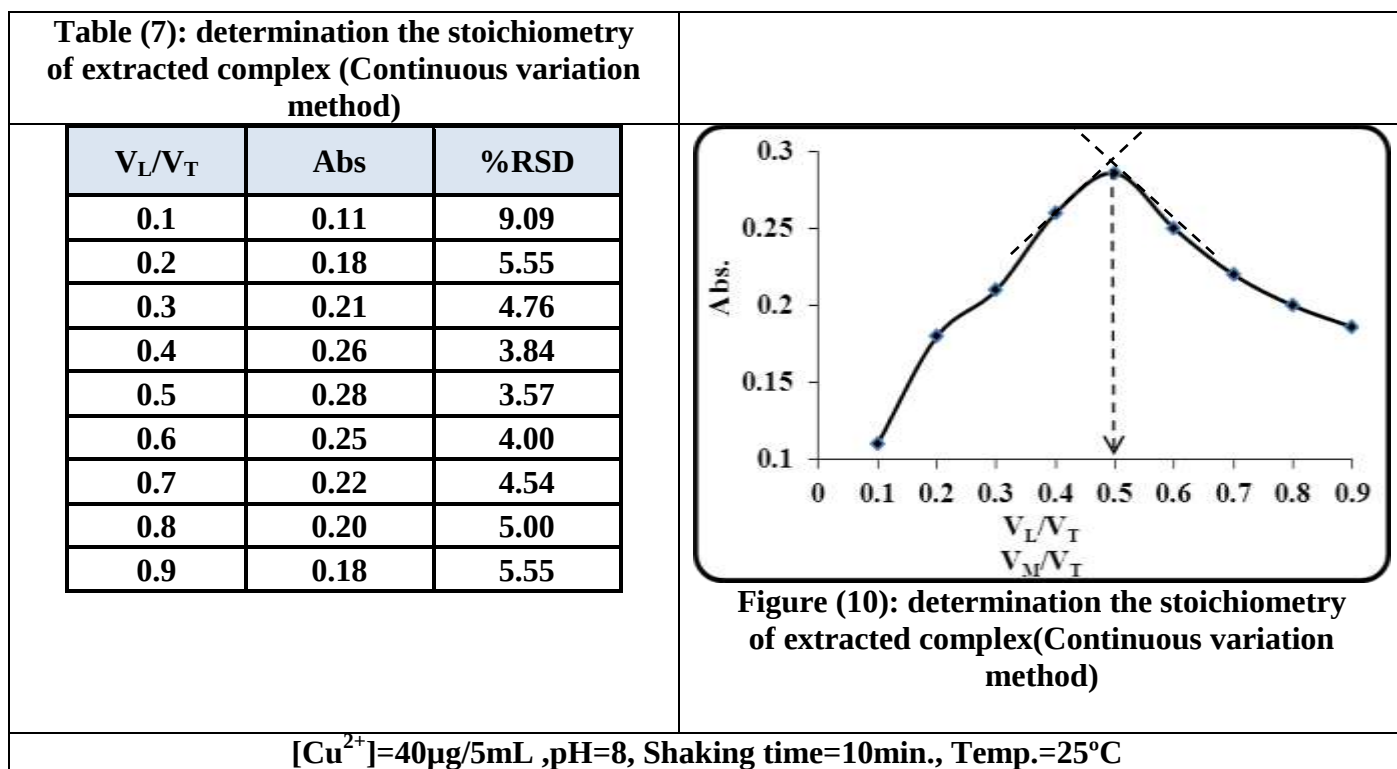


Table (8): Effect of foreign ions on distribution ratio (D)

Ions	Add as	D	%RSD	%E
Non	-	19.00	0.05	95.00
Cr^{3+}	$CrCl_3$	6.44	0.15	86.57
Ni^{2+}	$NiCl_2 \cdot 6H_2O$	11.42	0.08	91.95
Pb^{2+}	$PbCl_2$	16.77	0.05	94.37

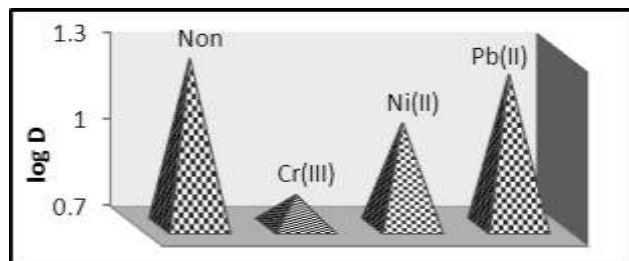


Figure (11): Effect of foreign ions on distribution ratio (D)

$[Cu^{2+}] = 40\mu g/5mL$, $[3-HPADPI] = 1 \times 10^{-4} M$ in $CHCl_3$, $[M^{n+}] = 40\mu g/5mL$, pH=8, Shaking time=10min., Temp.=25°C

Table (9): Synergism effect on distribution ratio (D)

[MIBK]	D	%RSD	%E
1×10^{-3}	39.00	0.025	97.50
5×10^{-4}	35.36	0.028	97.25
1×10^{-4}	32.33	0.030	97.00
5×10^{-5}	29.76	0.033	96.75
1×10^{-5}	24.00	0.041	96.00
1×10^{-6}	20.05	0.049	95.25

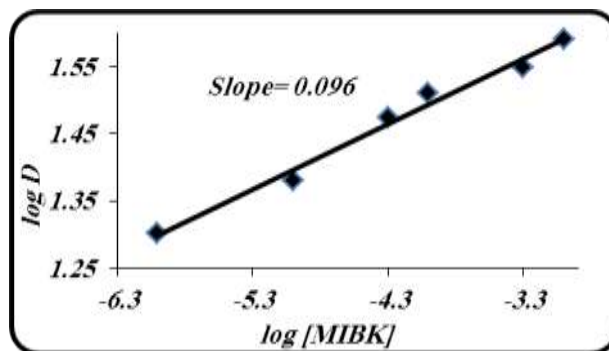


Figure (12): Synergism effect on distribution ratio (D)

$[\text{Cu}^{2+}] = 40 \mu\text{g}/5\text{mL}$, $[\text{3-HPADPI}] = 1 \times 10^{-4} \text{M}$ in CHCl_3 , $\text{pH}=8$,
Shaking time=10min., Temp.=25°C

Table (10): Effect of Organic Solvent on distribution ratio (D)

Organic Solvents	ϵ	D	%RSD	%E	ΔG_t	$K_A \times 10^4$ liter/mol	$K_{ex} \times 10^4$	ΔG_{ex} KJ/mol
Nitro benzene	35.74	0.98	1.02	49.52	-0.04	1.57	0.80	-20.31
Dichloro methane	9.08	0.85	1.17	46.02	-0.27	1.47	0.60	-19.68
Chloroform	4.806	19.00	0.05	95.00	-0.54	15.87	300.83	-33.71
Carbone tetrachl ride	2.38	0.67	1.49	40.12	-1.13	1.32	0.37	-18.59

$[\text{Cu}^{2+}] = 40 \mu\text{g}/5\text{mL}$, $[\text{3-HPADPI}] = 1 \times 10^{-4} \text{M}$ in different organic solvent, $\text{pH}=8$,
Shaking time=10min., Temp.=25°C

Table (11): Effect of Temperature on distribution ratio (D)

T°C	T K	1/T K $\times 10^{-3}$	D	% RSD	%E	$K_{ex} \times 10^8$	ΔH_{ex} KJ mole ⁻¹	ΔG_{ex} KJmole ⁻¹	ΔS_{ex} J mole ⁻¹ K ⁻¹
5	278	3.59	10.11	0.09	91.00	8.42	0.000062	-51.46	159.31
10	283	3.53	12.33	0.08	92.50	10.27			
15	288	3.47	13.28	0.07	93.00	11.07			
20	293	3.41	17.18	0.05	94.50	14.31			
25	298	3.35	19.00	0.05	95.00	15.83			
30	303	3.3	21.22	0.04	95.50	17.68			
40	313	3.19	29.76	0.03	96.75	24.80			
50	323	3.09	39.00	0.02	97.50	32.50			

$[\text{Cu}^{2+}] = 40 \mu\text{g}/5\text{mL}$, $[\text{3-HPADPI}] = 1 \times 10^{-4} \text{M}$ in CHCl_3 , $\text{pH}_{ex}=8$,
Shaking time=10min.,

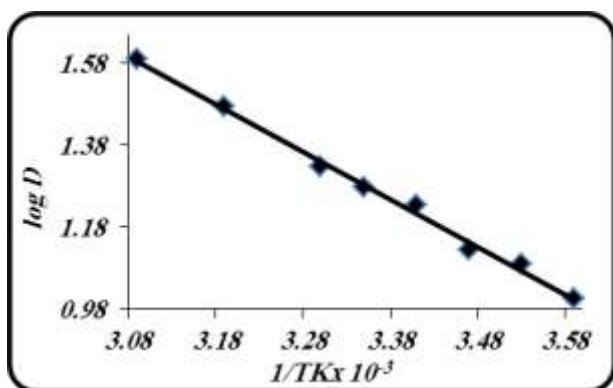


Figure (13): Effect of Temperature on distribution ratio (D)

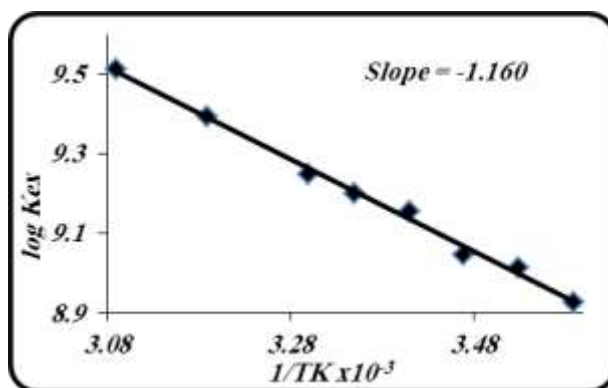


Figure (14): Effect of Temperature on distribution ratio (D)

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الاستخلاص المذيبى للنحاس (II) من المحاليل المائية باستعمال الكاشف العضوي الجديد

2-[(3-هيدروكسي فنيل) ازو]-5,4- ثنائي فنيل اميدازول

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الخلاصة

تضمنت هذه الدراسة تحضير الكاشف العضوي الجديد 2-[(3-هيدروكسي فنيل) ازو]-5,4- ثنائي فنيل اميدازول واستخدم كعامل تعقيد لاستخلاص ايونات Cu^{2+} من المحاليل المائية. أظهرت النتائج إن الدالة الحامضية المثلى لعملية الاستخلاص كانت ($\text{pH}_{\text{ex}}=8$) والتركيز الأمثل لايون Cu^{2+} كان $40\mu\text{g}$ في 5mL محلول مائي. أما بالنسبة لزمن الرج الأمثل فكان (10 دقائق). أما التركيب الأكثر احتمالا للصنف المستخلص فقد كان بنسبة 1:1 (Cu^{2+} : (3-HPADPI)). لم تظهر دراسة التداخل للايونات الموجبة أية زيادة لقيم نسب التوزيع (D) بوجود هذه الايونات. ان دراسة تأثير المشاركة Synergism على عملية الاستخلاص وباستعمال MIBK أشارت الى ارتفاع قيم نسب التوزيع (D) بوجود MIBK. دراسة تأثير المذيب العضوي أظهرت عدم وجود علاقة خطية بين ثابت العزل الكهربائي (ϵ) للمذيبات العضوية المستخدمة وقيم نسب التوزيع (D). أما الجانب الثرموديناميكي فقد اظهر إن التفاعل كان ماص للحرارة Endothermic reaction وان قيم الدوال الثرموديناميكية $\Delta H_{\text{ex}}=0.000062 \text{ KJ.mole}^{-1}$ ، $\Delta G_{\text{ex}}=-51.46 \text{ KJ.mole}^{-1}$ ، $\Delta S_{\text{ex}}= 159.31 \text{ J.mol}^{-1}.\text{K}^{-1}$.