



STUDIES ON THE BINARY AND TERNARY COMPLEXES OF MERCURY (II) WITH GALLIC ACID AND ADRENALINE

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ABSTRACT:

The interaction of mercury(II) ions with gallic acid (3,4,5-trihydroxybenzoic acid) (GAL) and adrenaline (epinephrine) (1-(3,4-dihydroxyphenyl)-2-methylaminoethanol) (ADR) were investigated by potentiometric and optical means. The stoichiometries and stability constants of the binary and ternary complexes have been determined pH-metrically in aqueous solution at 25° C and in ionic strength 0.1 M (NaNO₃). The complex formation equilibria involving adrenaline were characterized.

The equilibrium $\text{Hg}(\text{GAL})_2 + \text{Hg}(\text{ADR})_2 \rightleftharpoons 2\text{Hg}(\text{GAL})(\text{ADR})$, the corresponding constant is $\log K = 4.22$. The constant due to $\Delta \log k_{\text{Hg}} = \log k_{\text{Hg}}^{\text{Hg}(\text{GAL})} - \log k_{\text{Hg}}^{\text{Hg}(\text{GAL})(\text{ADR})}$ is 0.75. The results indicate that the overall ratio of the ternary complex $\text{Hg}(\text{GAL})(\text{ADR})$ is 1:1:1 and the mixed-ligand complex is more stable than one expected from purely statistical reasons. UV-Vis spectroscopy gave additional support to the results. There is a strong evidence from FT-IR spectrum that adrenaline chelates by the phenolic groups.

INTRODUCTION:

The use of mercury in a variety of products and industrial processes has been declining due to concerns about its hazardous effects on the environment. Mercury in different forms is introduced to the natural environment from a variety of sources and converted into more toxic form, i. e. methylmercury chloride by aquatic organism, and accumulated in the tissue of fish and birds^[1]. The majority of mercury that accumulates in our bodies the “silver” amalgam fillings in our teeth. In fact, these filling contain

53 percent or more mercury along with other dangerous and toxic metals. Mercury poisons many systems in the body leading to dozens symptoms and an equal number of disease states originating in all parts of the body. Mercury is toxic to our cells because it paralyzes the respiratory enzymes, these cells enable to use oxygen. Mercury exists in three states in the body: the mercuric ion Hg(II); the mercurous ion Hg(I) and metallic mercury Hg. The demand for rapid and sensitive methods for studying and the determination of chemical forms of toxic elements in environmental

samples is increasing. The interaction of toxic metal ions with biological molecules provides one of the most fascinating areas of coordination chemistry. Investigation of the stability of ternary complexes will help toward understanding the driving forces that lead to the formation of such complexes in biological systems. This paper presents results of the equilibrium and spectral studies of the interaction of addressed adrenaline (ADR) and/or gallic acid with mercury(II) systems as well as of studies of the complexation reactions in binary and ternary systems of these ligands with Hg(II). Adrenaline was the first hormone to be identified, and was successfully synthesized in 1904. It is part of a family known as biogenic amines, which includes serotonin and histamine, among others. Epinephrine is the compound commonly also called adrenaline (ADR). Its specific compound group is the catecholamine group, which also includes norepinephrine and dopamine. Sustained high levels of catecholamines in the blood are a good indicator of chronic stress. Adrenaline is a hormone produced by the adrenal gland in the body of many animals. When it is produced in the body it stimulates the heart-rate, dilates blood vessels and air passages, and has a number of more minor effects. Adrenaline is very potent vasoconstrictor and cardiac stimulant^[2]. Adrenaline ($C_9H_{13}NO_3$) (ADR) is one of the catecholamines that plays quite an important role in physiology as neurotransmitter in the central nervous system, CNS, along with other catecholamines such as the dopamine and noradrenaline. This makes them critical in

maintaining the body's homeostasis and in responding to acute and chronic stress, through an orchestration of cardiovascular, metabolic and visceral activities^[3]. Adrenaline (epinephrine) accounts for 5%-10% of the total catecholamines in the central nervous system (CNS), there is a suggestion that CNS adrenaline is involved in the central control of blood pressure^[4,5], respiration^[6] and pituitary hormone secretion^[7]. Adrenaline is the favored treatment for anaphylactic shock, and should be administered immediately if a person begins exhibiting severe allergic reactions. Green tea is honoured drink, is used medicinally and as a refreshment after meals. A study suggests a correlation between the natural anti-oxidants found in green tea and overall good health^[8]. The two largest components of green tea are carbohydrates, including cellulosic fiber and protein both of which are water insoluble. The next largest group comprises the polyphenols which are water soluble and may constitute up to 40% from the dry weight of green tea. Polyphenols are useful in the fight against numerous diseases. Tea inhibits the activity of several enzymes related to tumour promotion and cell proliferation including ornithine decarboxylase, protein kinase c, cyclooxygenase and lipoxigenase. It can also inhibit the formation of lung cancer in mice arising from NNK, a common tobacco carcinogen. Gallic acid is one of the polyphenols which are water soluble of green tea. Gallic acid (GAL) (3,4,5-trihydroxybenzoic acid), which contains ortho-diphenolic groups, has been described as a degradation product of lignin and humic

acid^[9]. Gallic acid have been used as chelating agent in ternary complexes^[10]. The dissociation constants of gallic acid and its ternary copper(II)^[11] and thorium(IV)^[12,13] complexes have been reported. The microscopic constants for side-chain amonium and phenolic hydroxy groups of adrenaline have been determined from spectrophotometric and potentiometric data^[14,15]. The dissociation constants of adrenaline were determined from potentiometry^[16,17] and spectrophotometry titrations^[18]. The last literature includes different stability constants values of adernaline of various studies. Several studies on some metal-ions complexes formed with adrenaline were pупlished^[13-16,19-21]. The reaction of L-adrenaline with mercury(II) in ternary system containing gallic acid has not been yet reported. The aim of the present work is to establish the stoichiometric compositions and stability constants of the species formed in the mercury(II)-adrenaline or gallic and mercury(II)-adrenaline-gallic systems. In addition, an attempt is to obtain information on the bonding modes in the complex formed and on the participation of the side-chain of adrenaline in complex formation. The complexation equilibria of mono and biligand systems in solution were also studied. The basic characteristics of the mixed-ligand complex of mercury(II) with ADR and gallic acid (GAL) in a 1:1:1 molar ratio were investigated potentiometrically. The Irving and Rossotti pH-titration technique^[22] and its related modification^[23,24] were employed. FT-IR spectrum is convincing evidence that adrenaline

tend to coordinate with mercury(II) via the oxygen atoms of the catechol phenolic groups.

EXPERIMENTAL:

1-Reagents:

All chemicals were of analytical grade. L-adrenaline (L-epinephrine) and gallic acid were purchased from Fluka and were used without further purification. Mercuric chloride, nitric acid, sodium nitrate, sodium hydroxide and potassium hydrogen phthalate were purchased from Sigma-Aldrich Chemicals Co., (USA) and were used as received. Doubly distilled water were used for the preparation of the solutions. All ligand solutions of initial concentration $C_L = 2.5 \times 10^{-3}$ M were prepared by direct weighing and dissolution in bidistilled water before use. The stock solution of mercuric chloride (5×10^{-2} M) was prepared in deionized water and standardized complexometrically^[25]. The working solutions were prepared by accurate dilution. The acidity of solutions investigated was adjusted by the addition of either dilute nitric acid or sodium hydroxide solution. The ionic strength was maintained constant at $I = 0.1$ M (NaNO_3).

2-Equipment:

pH measurements were carried out using a Corning 215 pH meter with a combined glass electrode. The glass electrode was calibrated before each titration with two Merck standard buffer solutions, first with the pH 7.0 followed by a pH 4.0 at 25°C by coupling the titration cell with a thermostatic bath set at this temperature.

The elemental analyses were done on a Perkin-Elmer 240 C instrument. The electronic spectra of ligand solutions (GAL or ADR) and its different mercury complex were recorded on a Perkin-Elmer (Lambda 35) computerized spectrophotometer equipped with 1 cm matched quartz cells. The infrared spectra were performed by a fourier transform infrared spectrometer (FT-IR) in the region 400-4000 cm^{-1} with Jasco 480 spectrometer using the potassium bromide disk technique.

3-Syntheses of Hg-ADR binary system:

The coordination complex was prepared by mixing a suitable aliquot of a solution of Hg^{II} containing 2.0 mmol in of doubly distilled water with aqueous solution of the ligand adrenaline (ADR) and the pH value was adjusted at 8.3 by adding aqueous sodium hydroxide. The colour of reaction mixture was changed and then the mixture was stirred for two hours under reflux on a water bath at a constant temperature of 70 $^{\circ}\text{C}$ then concentrated by evaporation to its half volume and left to cool to room temperature overnight. The complex was filtered off on a water pump and the obtained metal chelates washed several times with doubly distilled water and preserved in desiccator over P_4O_{10} , yield 82%, m.p.:320 $^{\circ}\text{C}$.

Analysis calculated for $\text{Hg}(\text{C}_9\text{H}_{11}\text{NO}_3)_2\cdot\text{C}$, 38.29%; H, 3.90%; N, 4.96%.

Found: C, 38.13%; H, 3.72%; N, 4.98%.

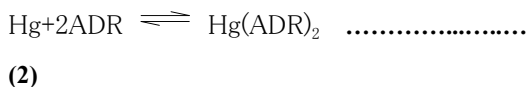
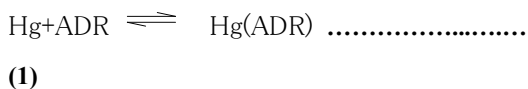
4-Calculations:

The acid base properties of GAL in water-ethanol media have been discussed

previously^[11]. Using the potentiometric and the absorption spectra data obtained for each method, estimation of the acidity constants of L-adrenaline and the complex formation constants were determined using the SUPERQUAD program^[26]. The program has been used to calculate acidity constants in systems previously studied^[27]. The absorbance vs. pH graphs were analyzed graphically as described previously^[11]. Timberlake^[28] showed that adrenaline has four weakly acidic function groups, the first ionisation is relatively strong and is attributed to the amine group, the second and third ones to catechol phenolic groups and the fourth to the alcoholic group, respectively. The dissociation constants of ADR were determined in aqueous solution by potentiometric titration of 50 ml of 2.5×10^{-3} M HNO_3 and NaNO_3 ($I=0.1$ M) in the presence and absence of the ligand (1×10^{-4} M) with standard carbonate free NaOH solution (1.08×10^{-2} M). The differences in NaOH consumption between such a pair of titration were used for calculation. The dissociation constant of ADR corresponding to the ionisation of imino hydrogen, pK_1 was found to be 8.52 ± 0.20 . The dissociation constants for hydroxyl catechol groups are listed in Table 1, and agree well with that reported by Grgas-Kuznar *et al.*^[16]. The final results for pK values are the average of six pairs of independent titrations. The dissociation constants of ADR and GAL as obtained from the titration graphs are given in Table 1. The stability constants of the complexes (Eqs. 1 and 2) were determined under the same conditions as used in the experiments for the

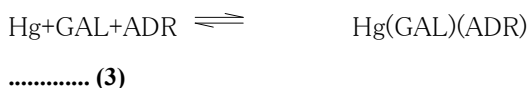
acidity constants. The titrations were carried out at four ADR/Hg^{II} ratios. The ligand/metal ratio was varied from 4:1 to 1:1. The stability constant for the binary Hg-ADR complexes were calculated from titration curves in which the metal to ligand ratio was 1:2 and the concentration of Hg(II) was 8×10⁻⁵ M. The final results given for the overall stability constant (Table 1) are always the averages of at least five independent pairs of titrations.

For the following equilibria in binary systems containing ADR:

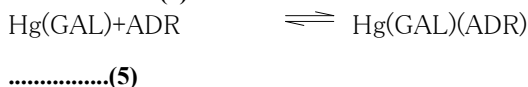


The equilibrium constants for the ternary systems were calculated from titration curves obtained for a 1:1:1 molar ratio of Hg-GAL-ADR and multi-titrations (usually six) were carried out with Hg(II)-GAL-ADR ternary system under the same experimental conditions of binary systems.

For ternary systems, the formation constant for the equilibrium



The stepwise formation constants for the equilibria



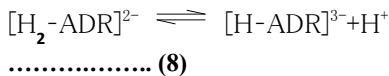
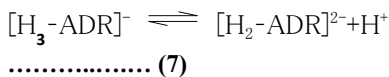
were calculated considering the relevant data or the acid dissociation constants and the cumulative binary and ternary constants.

RESULTS AND DISCUSSION:

1-Acid-base properties of the reagents:

Our previous work^[11] on the acid-base properties of GAL in water-organic solvent mixtures in the pH range 2-11.5 indicated that the ligand GAL exists in four different forms neutral (GAL-H₄), monoanionic (GAL-H₃), dianionic (GAL-H₂)²⁻ and trianionic (GAL-H)³⁻. The potentiometric titration graph for ADR in the neutral (H₄-ADR) shows a steep inflection at a=3 (where a is the number of moles of base added per mole of ligand). The constant corresponding to deprotonation of the fourth alcoholic group not determined under our experimental condition.

For the secondary ligand H₄-ADR, the constants corresponding to the following equilibria were also determined under our experimental condition:

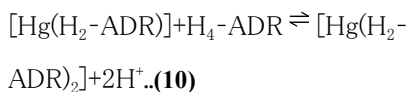
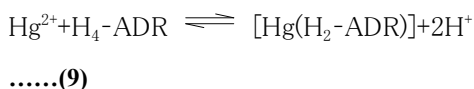


2-Stability constants of Hg(II) binary complexes:

The stoichiometry of the complexes formed during the interaction of Hg(II) with ADR (H₄-ADR) was established from the magnitude of the proton displacement, which was determined by

titrating solutions containing the ligand against standard alkali in the absence and presence of different molar quantities of Hg(II). The titration graph for a system containing a 1:2 molar ratio of Hg(II) and ADR exhibits two inflections at m=2 and 4 (m=moles of base added per mole of metal ion), indicating the formation of mono and bis-binary complexes.

The corresponding equilibria may be represented as follows:



The stability constants determined in this study are listed in Table 1.

3-Stability of mixed ligand complexes Hg(GAL)(ADR):

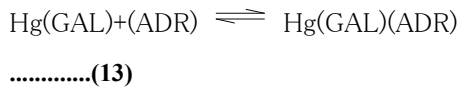
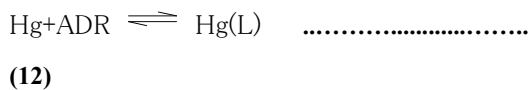
For the ternary complexes composed of L-adrenaline (ADR), Hg(II) and gallic acid (GAL) the experimental data show that formation of the ternary complexes shifts the buffer region of the ligands to lower pH values, which indicates that the ternary complexes are more stable than the binary complexes. The potentiometric titration curves for the ternary systems containing Hg(II), GAL and ADR in a 1:1:1 molar ratio exhibit a single steep inflection at m=4. The composite curve drawn by adding the horizontal distance of the Hg-GAL titration curve to the ADR curve is not superimposable with the mixed ligand titration curve, thereby confirming the formation of the Hg-GAL-ADR complex. The stability constants for the ternary

systems were computed from the titrations in which the concentrations of Hg(II):GAL:ADR were kept in the ratio 1:1:1, listed in Table 2. According to the results, the complex equilibria of Hg-GAL-ADR can be represented by the following:

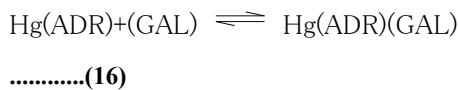
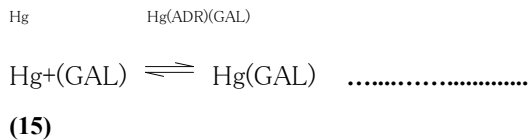


In order to compare the stabilities of the ternary complex species with those of the parent binary complexes the value $\Delta \log K$, the difference between the stabilities of the binary and the ternary complexes were determined.

The parameter $\Delta \log K$ is determined by Equations 11 to 16^[29]:

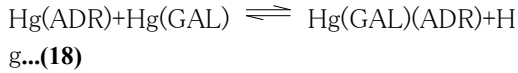


$$\Delta \log K = \log K^{\text{Hg}(\text{GAL})} - \log K^{\text{Hg}(\text{ADR})} \dots\dots(14)$$



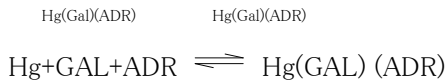
$$\Delta \log K = \log K^{\text{Hg}(\text{ADR})} - \log K^{\text{Hg}(\text{GAL})} \dots\dots(17)$$

The value of $\Delta \log K_{Hg}$ is the logarithm of the equilibrium constant due to equitation (18):

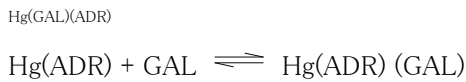


The overall constant, $\beta_{Hg(GAL)(ADR)}$, which was determined experimentally as in (Table 2) is connected with

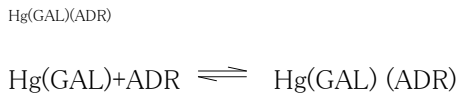
$K_{Hg(GAL)(ADR)}$ and $K_{Hg(GAL)}$ by equations (16)&(17).



$$\beta_{Hg} = \frac{[Hg(GAL)(ADR)]}{[Hg][GAL][ADR]} \dots (19)$$



$$K_{Hg(ADR)(GAL)} = \frac{[Hg(ADR)(GAL)]}{[Hg(ADR)][GAL]} \dots (20)$$



$$K_{Hg(GAL)(ADR)} = \frac{[Hg(GAL)(ADR)]}{[Hg(GAL)][ADR]} \dots (21)$$

$$\log K_{Hg(ARD)} = \log \beta_{Hg(ADR)(GAL)} - \log K_{Hg(GAL)(ADR)}$$

$$\log K_{Hg(GAL)} = \log \beta_{Hg(GAL)} - \log K_{Hg(GAL)(ADR)}$$

The other approach commonly used to quantify the stability of a ternary complex is based on the equilibrium constant, X , as defined by equation (24)^[30,31], $\log X$ may be calculated according to equation (25).

$$M(GAL)_2 + M(ADR)_2 \rightleftharpoons 2(M(GAL)(ADR))$$

$$X = \frac{[M(GAL)(ADR)]^2}{[M(GAL)_2][M(ADR)_2]} \dots (24)$$

$$\log X = 2 \log \beta_{M(GAL)(ADR)} - \left[\log \beta_{M(ADR)(GAL)} + \log \beta_{M(ADR)} \right]$$

$$= \left[\log K_{M(ADR)(GAL)} - \log K_{M(ADR)} \right] - \left[\log K_{M(ADR)} - \log K_{M(GAL)} \right] \dots (25)$$

M(GAL)(ADR)

M(ARD)(GAL)

Comparing the curves resulting from the titration of Hg(II) and ADR in a molar ratio of 1:1 with that where, in addition, GAL was present (ratio 1:1:1) observed that the deprotonation of ADR at a lower pH. This means that the ternary complex is more stable

than the corresponding binary one, the results obtained for the formation of the Hg-GAL-ADR ternary complex are given in Table 2. By using the data given in Table 2, the values of $\Delta \log K$ and $\log X$ calculated.

Table 1: Negative Logarithms of the acidity constants of the ligands and Logarithms of its stability constants of the binary Hg(II) complexes

Ligand (L)	$\text{pK}_{\text{H}_3\text{L}}^{\text{H}}$	$\text{pK}_{\text{H}_2\text{L}}^{\text{H}}$	$\text{pK}_{\text{HL}}^{\text{H}}$	$\log K_{\text{HgL}}^{\text{Hg}}$	$\log K_{\text{HgL}_2}^{\text{HgL}}$	$\log \beta_{\text{HgL}_2}^{\text{Hg}}$
GAL	8.51	10.70	—	9.03	7.35	16.38
ADR	8.52	10.04	11.95	8.20	7.16	15.36

Table 2: Logarithms of the stability constants of the ternary Hg(II)-GAL-ADR complexes and some related data [I=0.1, water media, 25°C]

$\log \beta_{\frac{\text{Hg}}{\text{Hg}(\text{GAL})(\text{ADR})}}$	$\log K_{\frac{\text{Hg}(\text{ADR})}{\text{Hg}(\text{ADR})(\text{GAL})}}$	$\log K_{\frac{\text{Hg}(\text{GAL})}{\text{Hg}(\text{GAL})(\text{ADR})}}$	$\Delta \log K_{\frac{\text{Hg}(\text{ADR})}{\text{Hg}(\text{GAL})(\text{ADR})}}$	$\log X$
17.98	9.78	8.95	0.75	4.22

4-Spectrophotometric study of binary and ternary complex of Hg(II) with GAL and ADR:

Absorption spectra and optimum pH:

The absorption spectra of L-adrenaline solution (1×10^{-4} M) in acid medium, pH range 2.5-6.5 exhibits absorption maximum band at $\lambda=280$ nm. At higher pH values, there was a bathochromic shift in nature up to a 296 nm at pH 11.2. The UV-visible spectra of GAL exhibits absorption band at $\lambda=340$ nm within the pH range 2-7.5. This band undergoes a reasonable bathochromic shift to shorter wavelengths on adding a Hg(II) solution. The spectra of the Hg(II)-GAL 1:1 complex with the reagent as reference are characterized by an

absorption band at $\lambda=395$ nm. A L-adrenaline solution (1×10^{-4} M) exhibits absorption band at $\lambda=294$ nm in the pH rang 2.5-12. In the pH range 4.2-9, the absorption spectrum of the Hg(II)-ADR 1:1 complex was characterized by an absorption band at $\lambda=380$ nm. The solution containing equimolar concentration of GAL and ADR undergoes a change in colour to pale yellow when mixed with the Hg(II) solution. The spectrum of the reaction mixture against a blank solution containing the same concentration of the two ligands shows a band at $\lambda=408$ nm. The latter band is unambiguously due to the formation of a mixed -ligand complex of Hg(II).

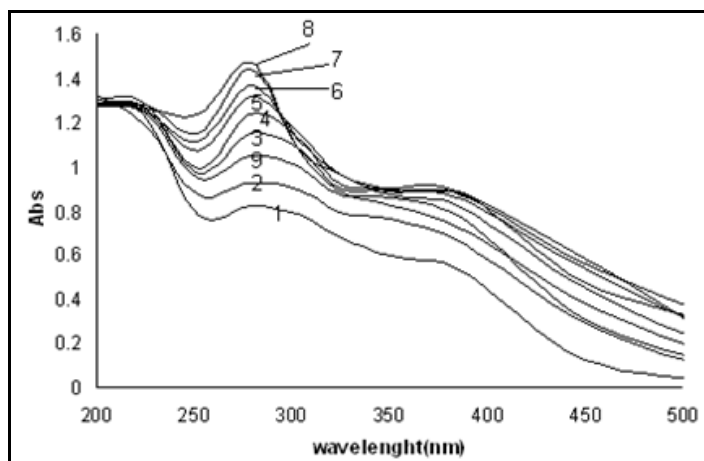


Fig. 1: Absorption spectrum of 1:1 Hg(II)-ADR complex at different pH values, $C_L=C_M=2.5 \times 10^{-4}$ M; pH (1) 2, (2) 3, (3) 4, (4) 5, (5) 6, (6) 7, (7) 8, (8) 8.5, (9) 9.5

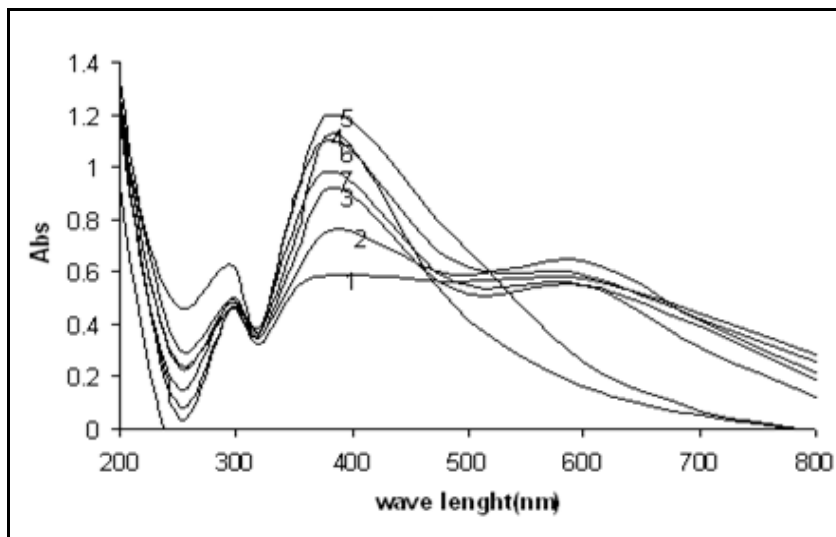


Fig. 2: Absorption spectrum of 1:1 Hg(II)-GAL complex at different pH values, $C_L=C_M=2.5 \times 10^{-4}$ M; pH (1) 6, (2) 6.5, (3) 7, (4) 7.5, (5) 8, (6) 8.5, (7) 9

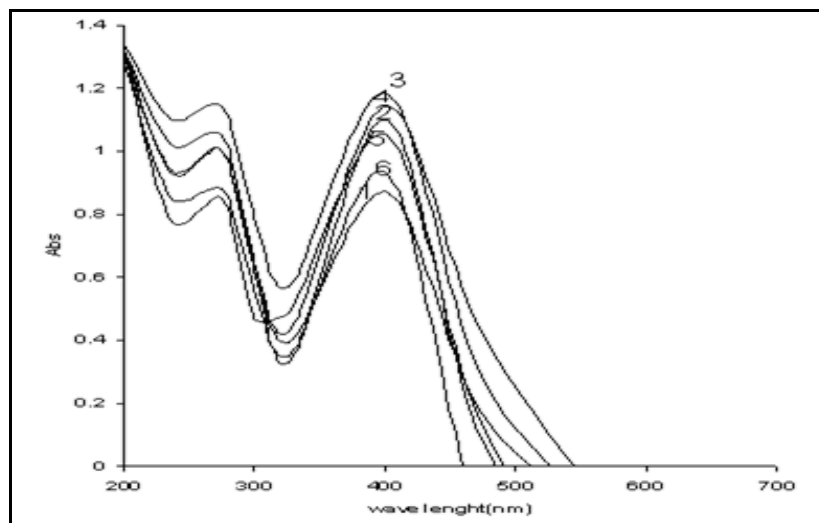


Fig. 3: Absorption spectrum of 1:1:1 Hg(II)-GAL-ADR complex at different pH values, $C_L=C_M=2.5 \times 10^{-4}$ M; pH (1) 6.5, (2) 7, (3) 7.5, (4) 8, (5) 8.5, (6) 9

Development attained in the in the pH range 3.5-7.2. Job's method of continuous variation^[32,33] was applied to establish the composition of the ternary Hg-GAL-ADR complex. The molar fraction of two of the component were varied continuously, keeping their combined concentration constant and

keeping their component in a large excess for all solutions in the series. The results indicate that the overall Hg-GAL-ADR complex has a 1:1:1 composition at the pH of the study. The stoichiometry of the ternary system was also determined by applying the mole-ratio method^[34].

5-FT-IR Spectra of mercury–adrenaline complex:

L-adrenaline compound has various potential donor sites. The FT-IR spectra of L-adrenaline was recorded (Fig. 4). The solid 1:1 chelates of mercury ion with ADR binary complex were isolated and characterized (Fig. 5). The aim is to explore the possibility that adrenaline tend to coordinate with mercury(II) via the oxygen atoms of the catechol phenolic groups. A comparison between the FT-IR spectra of adrenaline with mercury complex provide evidence regarding the bonding sites in adrenaline complex.

The FT-IR spectrum of adrenaline shows characteristic band at 3331 cm^{-1} and 1340 cm^{-1} corresponding to stretching phenolic and bending $\nu_{\text{(OH)}}$ of the catechol bonded groups. This band disappeared in the spectrum of the binary complex confirms that the two phenolic hydrogen atoms is replaced by the metal and

consequently coordination through oxygen of phenolic catechol groups. The C-O stretching mode of catechol observed at 1267 cm^{-1} is shifted to lower frequency by 21 cm^{-1} in the complexed ligand. The FT-IR spectrum of the free ligand adrenaline exhibits bands around 3100 , 3030 and 1590 cm^{-1} which may assigned to $\nu_{\text{as(NH)}}$, $\nu_{\text{s(NH)}}$ and $\delta_{\text{(NH)}}$. A comparison of the spectra of Hg-ADR binary complex with that of the respective ligand bring out interesting feature. The spectra of the binary system shows the characteristic band at 3448 cm^{-1} due to alcoholic $\nu_{\text{(OH)}}$ of the side chain ethanolamine. This indicate that two OH phenolic groups are coordinated to mercury ion through deprotonation. There is no FT-IR spectrum for the ternary Hg-GAL-ADR complex because the third phenolic group of gallic acid has not ionized and the band of $\nu_{\text{(OH)}}$ will appear in the spectra.

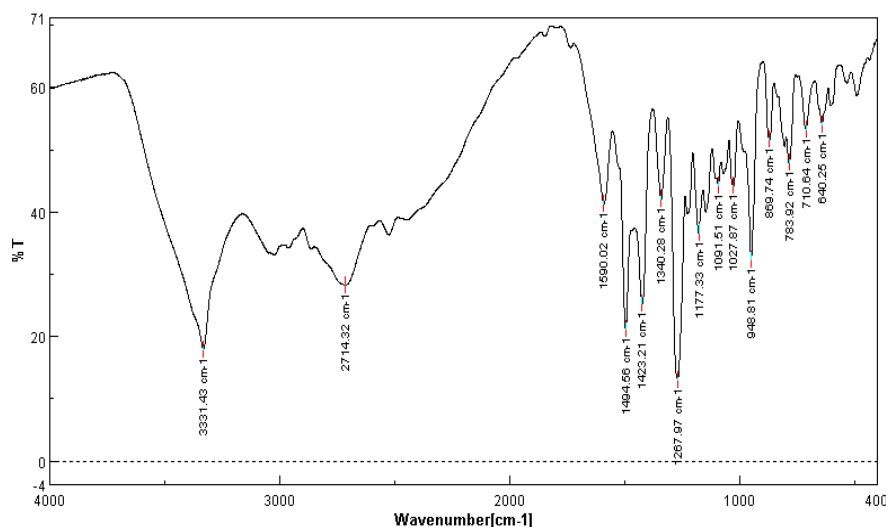


Fig. 4: FT-IR spectra of ADR

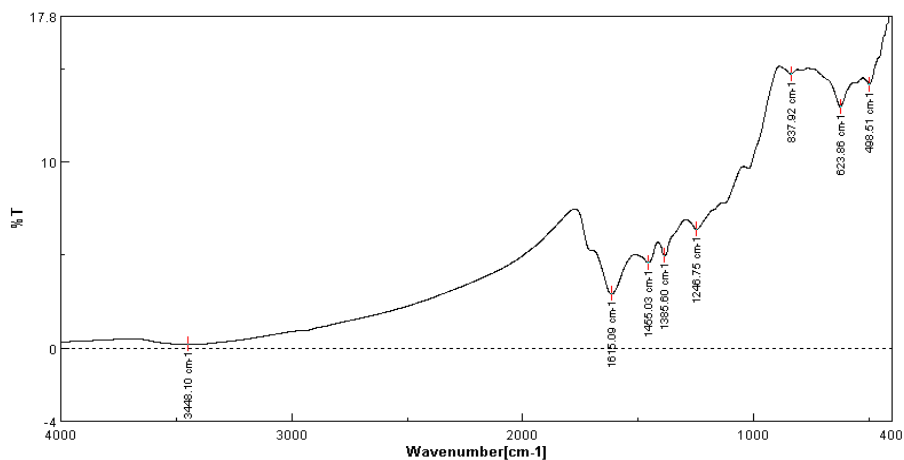


Fig. 5: FT-IR spectra of 1:1 Hg(II)-ADR binary complex

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دراسات على المتراكبات الثنائية الثلاثية لأيون الزئبقيك مع حمض الجاليك وهرمون الأدرينالين

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تم فى هذا البحث دراسة إتزانات تفاعلات التراكب بين أيون الزئبقيك وهرمون الأدرينالين وحمض الجاليك فى محاليل مائية بطرق المعايرة الجهدية والطيفية بتتبع تغيير الرقم الهيدروجينى للمحلول ذو القوة الأيونية 100 ميللى مول من نترات الصوديوم. وتم التعرف على إتزانات التراكب الموجودة وظروف تكوين المتراكبات فى مدى الرقم الهيدروجينى المناسب. وكذلك أمكن تحديد النسب التكوينية للمتراكبات الناتجة من تفاعلات متراكب أيون الزئبقيك وهرمون الأدرينالين مع حمض الجاليك. وعين ثابت التكوين للمتراكب مختلط اللجان ذات النسبة التكوينية 1:1:1، وأمكن تقييم ثباته بالمقارنة مع ثبات المتراكبات الثنائية. وتم أيضاً تحديد الأطوال الموجية لهذه المتركبات الثنائية والمتراكبات مختلطة اللجان بالأطياف المرئية، وال فوق بنفسجية. ووضع تصور عن الارتباط بين مركب الأدرينالين مع أيون الزئبقيك باستخدام الأشعة تحت الحمراء، وثبت من الدراسة ترابط الأدرينالين مع أيون الزئبقيك بمجموعتى الفينوليك. وحسبت ثوابت التآين لمركب الأدرينالين فى الظروف التجريبية المذكورة.