

## STUDIES ON THE BINARY AND TERNARY COMPLEXES OF ALUMINIUM(III) WITH GALLIC ACID AND ADRENALINE

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**Abstract**

The reaction of aluminium(III) ions with gallic acid (3,4,5-trihydroxybenzoic acid) (GAL) and adrenaline (epinephrine) (1-(3,4-dihydroxyphenyl)-2-methylaminoethanol) (ADR) was investigated by potentiometric and optical means. Stability constants of the binary and ternary complexes at 25°C and in 0.1 M NaNO<sub>3</sub> ionic strength in aqueous solution have been determined. The complex formation equilibria involving adrenaline were characterized

The constant due to  $\Delta \log k_{Al} = \log k_{Al(GAL)(ADR)}^{Al(GAL)} - \log k_{Al(ADR)}^{Al}$  is 0.75. The results indicate that the overall ratio of the ternary complex Al(GAL)(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.

**Keywords:** Mixed-ligand Complexes, Aluminium, Gallic Acid, L-adrenaline, Complexation equilibria, Stability Constants, Spectral, Potentiometry.

**Introduction**

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 [1]. Adrenaline (C<sub>9</sub>H<sub>13</sub>NO<sub>3</sub>) (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 [2]. 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 [3,4], respiration [5] and pituitary hormone secretion [6]. 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. An study suggest a correlation between the natural anti-oxidants found in green tea and overall good health [7]. Gallic acid and tea polyphenols are found in tea and considered as antioxidants [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 disease. Tea inhibits the activity of several enzymes related to tumour promotion and cell proliferation including ornithine decarboxylase, protein kinase c, cyclooxygenase and lipoxgenase. 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 o-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]. I have been reported and discussed the dissociation constants of gallic acid and its ternary copper(II) [11], mercury(II) [12] and thorium(IV) [13,14] complexes. The microscopic constants for side-chain ammonium and phenolic hydroxy groups of adrenaline have been determined from spectrophotometric and potentiometric data [15,16]. The dissociation constants of adrenaline were determined from potentiometry [17,18] and spectrophotometry titrations [19]. The last literature gives a comparison on the different stability constants values of adrenaline of various studies. Several studies on some metal-ions complexes formed with adrenaline were published [15-17, 20-22]. The chemistry of aluminium is of great importance their presence in every house, in many alloys and their biological activities in human body. Low amounts of aluminium can cause human acute toxicity. There is a correlation between aluminium concentration in drinking water and the incidence of Alzheimer's disease [23]. The kinetics of the reactions of aluminium(III) with gallic acid or adrenaline have been reported [24]. The potentiometric results reported by Lars-olof

Öhman et al [25,26] determined the percent of the complexed species formed from the reaction of gallic acid with aluminium(III). The reaction of L-adrenaline with aluminium(III) 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 aluminium(III)-adrenaline or gallic and aluminium(III)-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 studied. The working conditions were established in order to obtain fundamental information about the stability of the complexed biligand system and the possible equilibria that exist in solution. The basic characteristics of the mixed-ligand complex of aluminium(III) with ADR and gallic acid (GAL) in a 1 : 1 : 1 molar ratio were investigated potentiometrically. The Irving and Rossotti pH-titration technique [27] and its related modification [28,29] were employed. Fundamental, the spectrophotometric study of the binary and ternary complexes have also been characterized. There is no example of this behaviour in the literature.

## **Experimental**

### **Reagents**

All chemicals were of analytical grade. L-adrenaline (L-epinephrine) and gallic acid were purchased from Fluka and were used without further purification. Aluminium nitrate, nitric acid, sodium nitrate, sodium hydroxide and potassium hydrogen phthalate and 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 aluminium nitrate ( $5 \times 10^{-2}$  M) was prepared in deionized water and standardized complexometrically with Oxine (BDH) [30]. 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 ( $\text{NaNO}_3$ ).

### **Equipment**

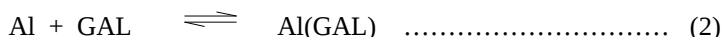
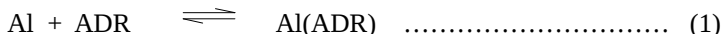
pH measurements were carried out using a Corning 215 pH meter with a combined glass electrode. The glass electrode was calibrated before each titration with two Merck standard buffer solutions, first with the pH 7.0 followed by a pH 4.0 at 25°C by coupling the titration cell with a thermostatic bath set at this temperature. The electronic spectra of solutions of the legend GAL or ADR and its different metal complexes were recorded on a Perkin-Elmer (Lambda 35) computerized spectrophotometer equipped with 1 cm matched quartz cells.

### 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 [31]. The program has been used to calculate acidity constants in systems previously studied [12,32]. The absorbance vs. pH graphs were analyzed graphically as described previously [11]. Timberlake [33] have been shown that adrenaline has four weakly acidic function groups, the first ionisation is relatively strong and is attributed to imino group, the second and third to one and the other 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  $2.5 \times 10^{-3}$  M  $\text{HNO}_3$  and  $\text{NaNO}_3$  ( $I = 0.1$  M) in the presence and absence of the ligand ( $1 \times 10^{-4}$  M) with standard carbonate free NaOH solution ( $1.08 \times 10^{-2}$  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,  $\text{pK}_1$  was found to be  $8.52 \pm 0.20$ . The dissociation constants for hydroxyl catechol groups were found, as reported by Grgas-Kuznar et al [17]. 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 and 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  $L/\text{Al}^{\text{III}}$  ratios where  $L = \text{ADR}$  or  $\text{GAL}$ . The ligand/ metal ratio was varied from 4 : 1 to 1 : 1. The stability constant for the binary  $\text{Al-ADR}$  and  $\text{Al-GAL}$  complexes were calculated from titration curves in which the metal to ligand ratio was 1 : 2 and the concentration of  $\text{Al(III)}$  was  $8 \times 10^{-5}$  M. The final

results given for the overall stability constant (see Table 1) are always the averages of at least five independent pairs of titrations.

For the following equilibria in binary systems containing ADR or GAL:



The equilibrium constants for the ternary systems were calculated from titration curves obtained for a 1 : 1 :1 molar ratio of Al–ADR–GAL and multi-titrations (usually six) were carried out with Al(III)–ADR–GAL 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

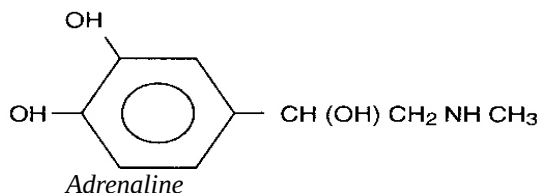


were calculated considering the relevant data or the acid dissociation constants and the cumulative binary and ternary constants.

**Results and discussion**

**Acid-base properties of the reagents**

The values of the acid dissociation constants of gallic acid were determined potentiometrically in our previous work [11]. In this work under our experimental conditions the acid – base properties of GAL in the pH range 2-11.5 indicated that the ligand GAL exists in four different forms neutral (GAL-H<sub>4</sub>), monoanionic (GAL-H<sub>3</sub>)<sup>-</sup> species is predominant up to pH < 6.8, while in the more alkaline solution the dianionic (GAL-H<sub>2</sub>)<sup>2-</sup> and trianionic (GAL-H)<sup>3-</sup> forms predominate. The potentiometric titration graph for ADR in the neutral (H<sub>4</sub>–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  $H_4\text{-ADR}$ , the constants corresponding to the following equilibria were also determined under our experimental condition:

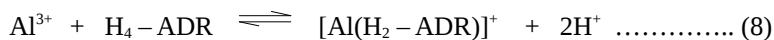


The proton-ligand formation numbers were calculated by Irving and Rossotti's expression [27].

#### STABILITY CONSTANT OF AL (III) binary complexes

The stoichiometry of the complexes formed during the interaction of Al(III) with ADR ( $H_4\text{-ADR}$ ) 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 Al(III). The titration graph for a system containing a 1 : 2 molar ratio of Al(III) and ADR exhibits an inflection at  $m = 2$  ( $m =$  moles of base added per mole of metal ion), indicating the formation of mono binary complex. This proves the formation of 1:1 Al-ADR complex.

The corresponding equilibria may be represented as follows :



The stability constants determined in this study are listed in Table 1. The titration graph for a system containing 1:1 or 1:2 molar ratio of  $Al^{3+}$  and monoanionic  $[GAL-H_3]^-$  exhibits an inflection at  $m = 2$  ( $m =$  moles of base added per mole of metal ion), indicating the formation of mono binary complex.

The corresponding equilibria may be represented as follows :



**Table 1. Negative Logarithms of the acidity constants of the ligands and Logarithms of its stability constants of the Al(III) binary complexes.**

Ligand (L)	H pK H <sub>3</sub> L	H pK H <sub>2</sub> L	H pK HL	Al log $\beta$ Al L
ADR	8.52	10.04	11.95	8.48
GAL	8.51	10.70	-	7.55

**Stability of mixed ligand complexes Al(GAL)(ADR)**

For the ternary complexes composed of L-adrenaline (ADR) , Al(III) and gallic acid (GAL) the expermental data show that the formation of 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 Al(III), ADR and GAL in a 1 : 1 : 1 molar ratio exhibit a single steep inflection at m = 4. The composite curve draen by adding the horizontal distance of the Al-ADR titration curve to the GAL curve is not superimposable with the mixed ligand titration curve, there by confirming the formation of the Al–ADR–GAL complex. The stability constants for the ternary systems were computed from the titrations in which the concentrations of Al(III) : GAL: ADR were kept in the ratio 1: 1: 1, listed in Table 2. According to the results, the complex equilibria of Al–GAL–ADR can be represented by the following :

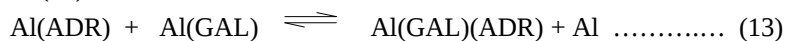


In order to compare the stabilities of the ternary complex species with those of the parent binary complexes the value  $\Delta \log K$ , the difference between the stabilities of the binary and the ternary complexes , were determined. The parameter  $\Delta \log K$  is determined by Equations 9 to 13 [34] :



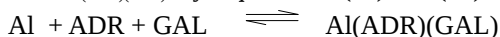
$$\Delta \log k_{Al} = \log k_{Al(ADR)(GAL)}^{Al(ADR)} - \log k_{Al(GAL)}^{Al} \dots\dots\dots (12)$$

The value of  $\Delta \log K_{Al}$  is the logarithm of the equilibrim constant due to equation (13):

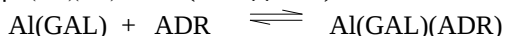


The overall constant,  $\beta_{Al(GAL)(ADR)}^{Al}$ , which was determined experimentally (Table 2) is connected with

$K_{Al(ADR)(GAL)}^{Al}$  and  $K_{Al(ADR)(GAL)}^{Al(ADR)}$  by equations (11) and (12) respectively.



$$\beta_{Al(ADR)(GAL)}^{Al} = [Al(ADR)(GAL)] / [Al][ADR][GAL] \dots\dots\dots (13)$$



$Al(GAL)$



$$K_{Al(GAL)(ADR)} = [Al(GAL)(ADR)] / [Al(GAL)][ADR] \dots\dots\dots (14)$$



$$K_{Al(ADR)(GAL)} = [Al(ADR)(GAL)] / [Al(ADR)][GAL] \dots\dots\dots (15)$$

$$\log K_{Al(ADR)(GAL)} = \log \beta_{Al(ADR)(GAL)}^{Al} - \log K_{Al(ADR)}^{Al} \dots\dots\dots (16)$$

$$\log K_{Al(GAL)(ADR)} = \log \beta_{Al(GAL)(ADR)}^{Al} - \log K_{Al(GAL)}^{Al} \dots\dots\dots (17)$$

Comparing the curves resulting from the titration of Al(III) 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 Al-ADR-GAL ternary complex are given in Table 2. By using the data given in Table 2, the values of  $\Delta \log K_{Al}$  calculated.

**Table 2. Logarithms of the stability constants of the ternary Al-GAL-ADR complexes and some related data [I=0.1, water media, 25°C ]**

$\log \beta_{Al(GAL)(ADR)}^{Al}$	$\log K_{Al(ADR)(GAL)}^{Al(ADR)}$	$\log K_{Al(GAL)(ADR)}^{Al(GAL)}$	$\Delta \log K_{Al(GAL)(ADR)}^{Al(ADR)}$
16.44	8.89	7.96	0.45

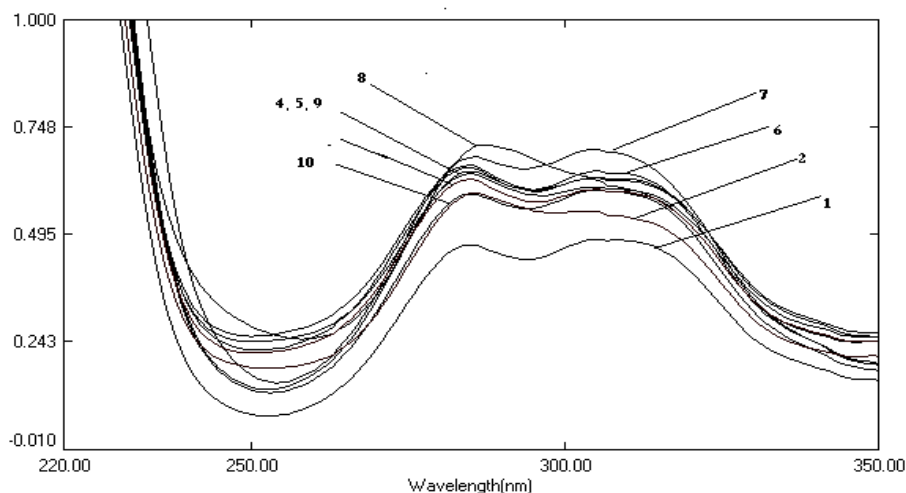
The results proved that ADR is the primary ligand and GAL the secondary ligand and ternary system is more stable corresponding binary one.

**Spectrophotometric study of binary and ternary complex of Al(III) with GAL and ADR**

**Absorption spectra and optimum pH**

I have been reported the absorption spectra [12] of the acid – base properties of L-adrenaline. The absorption spectra of L-adrenaline solution ( $1 \times 10^{-4}$  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 bathchromic shift in nature up to a 296 nm at pH 11.2. In the pH range 3 - 8, the absorption spectrum of the Al(III)–ADR 1 : 1 complexes (Fig. 1) was characterized by an absorption band at  $\lambda = 308$  nm. 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 a bathochromic shift to shorter wavelengths on adding a Al(III) solution. The spectra of the Al(III)-GAL 1 : 1 complex with reagent blank as reference are characterized by two states of the equilibria as showing in Fig.

2, the two absorption bands at  $\lambda = 305$  and  $285$  nm. The solution containing equimolar concentration of GAL and ADR undergoes a change in colour to pale yellow when mixed with the Al(III) solution. The spectrum of the reaction mixture against a blank solution containing the same concentration of the two ligands shows band at  $\lambda = 280$  nm. The latter band is unambiguously due to the formation of a mixed-ligand complex of Al(III) with the maximum colour development attained in the in the pH range 7 – 8. The absorbance vs. pH graphs were analyzed graphically using the relations derived earlier by Sommer et al [35,36]. Fig. 4. shows the variation of the absorbance values with pH of Al(III)-ADR, Al(III)-GAL and Al-GAL-ADR systems. The results showed that the ligand ADR is the primary and GAL the secondary in the ternary system Al-GAL-ADR, this means that the reaction of Al(III) with ADR is faster than its reaction with GAL [37]. Job's method of continuous variation [38,39] was applied to establish the composition of binary systems and the ternary Al-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 Al-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 [40].



**Fig. 1. Absorption spectrum of 1:1 Al(III)-ADR complex at different pH values,  $C_L = C_M = 2.5 \times 10^{-4}$  M; pH (1) 3, (2) 4, (3) 4.5, (4) 5, (5) 5.5, (6) 6, (7) 6.5, (8) 7, (9) 7.5, (10) 8.**

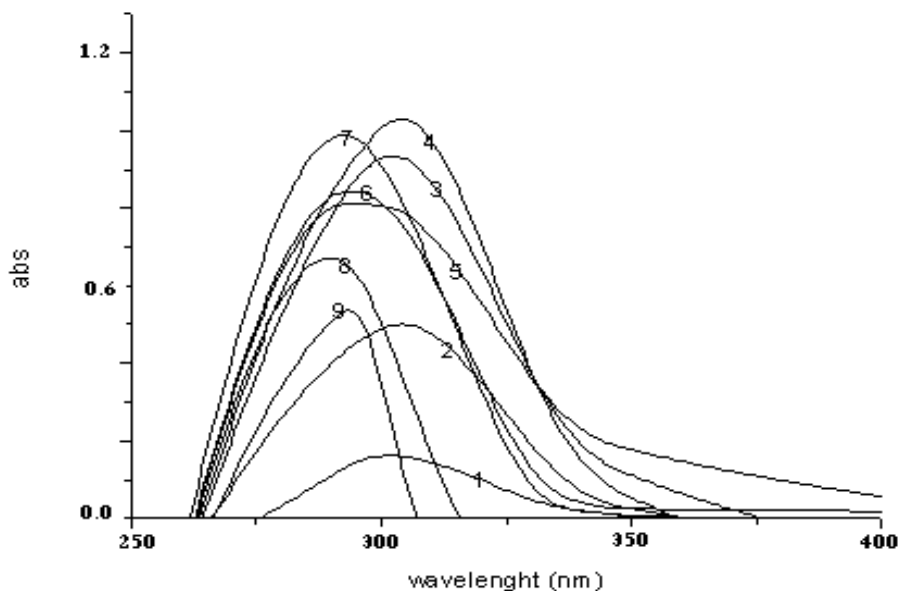


Fig. 2. Absorption spectrum of 1:1 Al(III)-GAL complex at different pH values,  $C_L = C_M = 2.5 \times 10^{-4} \text{ M}$ ; pH (1) 3, (2) 3.5, (3) 4, (4) 4.5, (5) 5, (6) 6, (7) 6.5, (8) 7.5, (9) 8.

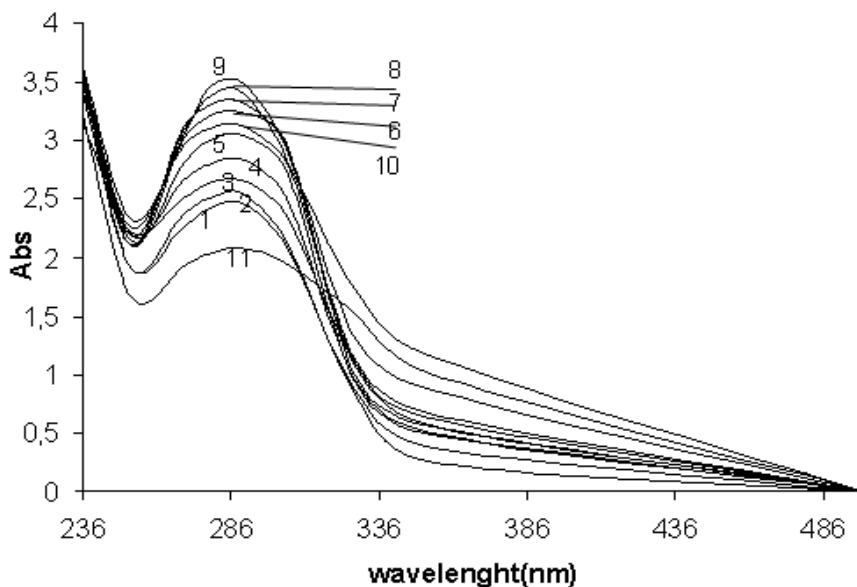
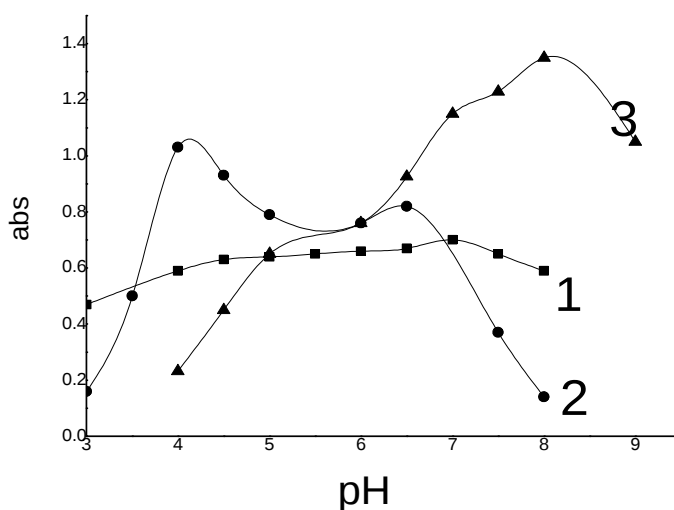


Fig. 3. Absorption spectrum of 1:1:1 Al(III)-GAL-ADR complex at different pH values,  $C_L = C_M = 2.5 \times 10^{-4} \text{ M}$ ; pH (1) 2, (2) 2.5, (3) 3, (4) 4, (5) 4.5, (6) 5, (7) 6, (8) 7, (9) 8, (10) 8.5, (10) 9.



**Fig. 4. Absorbance vs. pH graphs for 1) [■] Al(III)-ADR, 2) [●] Al(III)-GAL and 3) [▲] Al-GAL-ADR at pH 6.5,  $C_L=C_M=2.5 \times 10^{-4}$  M and  $I = 0.1$  M**

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## ملخص البحث

تم فى هذا البحث دراسة إترانات تفاعلات التراكب بين أيون الألمونيوم الثلاثى 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 إترانات التراكب الموجودة وظروف تكوين المتراكبات فى مدى الرقم الهيدروجينى المناسب. وكذلك أمكن تحديد النسب التكوينية للمتراكبات الناتجة من تفاعلات متراكب أيون الألمونيوم وهرمون الأدرينالين مع حمض الجاليك. وعين ثابت التكوين للمتراكب مختلط اللجائن ذات النسبة التكوينية 1:1:1 وأمكن تقييم ثباته بالمقارنة مع ثبات المتراكبات الثنائية. وتم أيضا تحديد الأطوال الموجية لهذه المتركبات الثنائية والمتراكبات مختلطة اللجائن بالأطياف المرئية والفوق بنفسجية. ووضع تصور عن الإرتباط بين مركب الأدرينالين وحمض الجاليك مع أيون الألمونيوم وثبتت من الدراسة ترابط الأدرينالين مع أيون الألمونيوم أولا ودخول حمض الجاليك كمترابط ثانوى. وحسبت ثوابت التآين لمركب الأدرينالين فى الظروف التجريبية المذكورة.