

SPECTROPHOTOMETRIC DETERMINATION OF Fe(III), Cu(II) AND UO₂(II) IONS BY A NEW ANALYTICAL REAGENT DERIVED FROM CONDENSATION OF MONOETHANOLAMINE AND ACETYL ACETONE

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A highly sensitive and selective spectrophotometric method is proposed for direct trace determination of Fe(III), Cu(II) and UO₂(II) in aqueous solutions. The method is based on the reaction of those cations with a new analytical reagent 2-ethanolimino-2-pentylidino-4-one (B3). Under the optimum reaction conditions and other important analytic parameters, B3 reacts with the investigated cations and forms colored complexes. The optimum pH for complex formation has been adjusted. 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 $\lambda_{\max} = 440$ nm and pH = 3.5, the Cu(II) complex is detected at $\lambda_{\max} = 340$ nm and pH = 6.0, while that of UO₂(II) is detected at $\lambda_{\max} = 370$ nm and pH = 4.0. Beer-Lambert's law is obeyed in the concentration range = $0.5 - 3.0 \cdot 10^{-4}$ M ($2 - 17$ $\mu\text{g/ml}$ for Fe(III), $3 - 9$ $\mu\text{g/ml}$ for Cu(II) and $13 - 81$ $\mu\text{g/ml}$ for UO₂(II) complexes. The stoichiometries of the formed complexes are determined using different spectrophotometric methods. The conditions for the complexation were determined. The rate of the reaction between Fe(III) ion and the ligand has been evaluated under pseudo first order condition. The ability of the present ligand to determine micrograms of Fe(III), Cu(II) and UO₂(II) ions is tested and the resulted data are analyzed using statistical parameter to obtain the minimum error. The effect of various substances on the determination of the investigated cations is also investigated in detail. The results indicate that most of the studied co-existing substances could be tolerated in considerable amounts. The proposed method offers the advantages of sensitivity, rapidity, selectivity and simplicity without any prior separation or extraction. It has been applied to the analytic samples with satisfactory results.

Keywords: ethanolamine; Schiff base; spectrophotometry; acetyl acetone; condensation

INTRODUCTION

The separation and determination of heavy metal ions in the environmental and biochemical research have been one of the most important topics of analytical chemistry. As compared with the other techniques, spectrophotometry is very simple, rapid and less expensive for determination of elements in a variety of samples. Developing highly functional chelating agents such as Schiff bases has been a great concern of many analytical chemists. Many investigations have been centered on the structure and bonding in Schiff bases but few have been directly concerned with analytical applications [1–6]. Copper is essential for life but

is highly toxic above certain limits to organisms like certain algae, fungi and many bacteria or viruses [7, 8]. In addition, the accumulation of copper in the human liver is a characteristic of Wilson's disease, which produces neurological and psychiatric defects [9]. There are conventional methods for copper(II) determination [10–12]. However, the colorimetric methods are often preferred due to the fact that they involve less expensive instruments and show rapid results. Iron is the most important nutrient in the human diet as it is complexed with hemoglobin and plays a major role in respiratory enzymes such as cytochromes

[13]. Several methods for the analysis of iron in pharmaceuticals and environmental samples have been reported [14–20]. Considerable interest has developed in the determination of trace uranium in environmental sites as well as in facilities of the nuclear industry. Electroanalytical techniques have frequently been used for this purpose. In particular, adsorptive stripping voltammetry is becoming a widely accepted tool for ultra-trace measurement of uranium [21–25]. This work has been aimed to develop a highly sensitive and efficient spectrophotometric method for iron, copper and uranyl cations determination, based on the formation of colored complexes which were formed by the reaction of those cations with 2-ethanolimino-2-pentylidino-4-one (B3) (Fig. 1). Various factors influence

the sensitivity of the proposed method such as wavelength, pH, effect of foreign ions, and ranges of applicability of the Beer's law in the determination of the investigated cations are also included. The method has been applied to some pharmaceutical and environmental water samples.

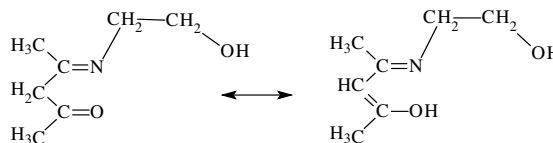


Fig. 1. The structural formulae of the tridentate ligand 2-ethanolimino-2-pentylidino-4-one.

EXPERIMENTAL

Reagents

All chemicals were of analytical-grade quality and freshly doubly distilled deionized water was used throughout the course of the investigation.

Synthesis of the ligand, B3

The 2-ethanolimino-2-pentylidino-4-one (B3) as shown in Fig. 1 was prepared as reported before [26]. The purity of the ligand was checked by the elemental analysis and physicochemical methods.

Apparatus

The pH measurements were carried out using the Fischer Scientific Accumet pH-meter model 825 MP, fitted with the Fischer combined electrode and calibrated by a standard buffer solution at the desired temperature. All uv-visible spectra of the investigated compounds were obtained at room temperature by the uv-visible spectrophotometer model Perkin-Elmer 550 S using 1 cm quartz cells.

Analytical method

A solution containing less than 30 μg of each of the iron(III), copper(II) and uranyl(II) cations was transferred into a 25 ml calibrated flask, 5.0 ml of either 0.1 M KOH or HNO₃ solution to reach the optimum value of pH (3.5 for Fe(III), 6.0 for Cu(II) and 4.0 for UO₂(II)) and 6.0 ml of 0.3 % 2-ethanolimino-2-pentylidino-4-one solution were added successively, the solution was diluted to the mark with water and mixed well. It was waited 90 min and the absorbance at the required wavelength (440 for Fe(III), 340 for Cu(II) and 370 nm for UO₂(II)) in a 1 cm quartz cell against the reagent blank was measured. All absorbance measurements were carried out with a model Perkin-Elmer 550 S.

Reference method

The measurements were carried out with standard methods. Iron(III) was determined spectrophotometrically [20, 27, 28]. The copper(II) determination was determined by the conventional spectrophotometry method [10]. The recommended procedure for the detection of uranyl(II) determination was carried out by the conventional method [29, 30].

RESULTS AND DISCUSSION

Optimal conditions for formation of the complexes

This work was carried out on the complexes of Fe(III), Cu(II) and UO₂(II) with the entitled

ligand due to the great tendency of these ions to form chelate compounds with characteristic colors. The effect of pH on the absorption spectra of Fe(III), Cu(II) and UO₂(II)-ligand mixtures were

studied by mixing $1 \cdot 10^{-5}$ M of the metal ions with $3 \cdot 10^{-5}$ M ligand under controlled pH values. The pH's were adjusted to the required values using portions of 0.1 M HNO₃ and 0.1 M KOH and the absorbance values of the solutions were measured in the range of $\lambda = 300 - 500$ nm. It is evident from the results, that the absorbance gave maxi-

imum value at pH = 6.0 for Cu(II) – ligand mixture at $\lambda = 340$ nm, at pH 4.0 for UO₂(II)-ligand mixture at $\lambda = 370$ nm and at pH 3.5 for Fe(III)-ligand mixture at $\lambda = 440$ nm. The validity of the Beer's law checked under the optimum condition gave a good straight line and the molar absorptivity was calculated for each mixture at different λ (Fig. 2).

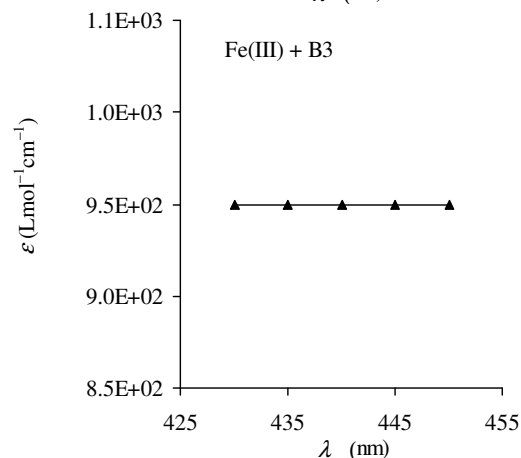
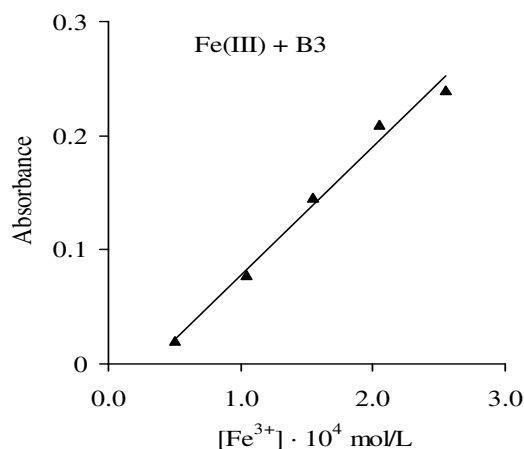
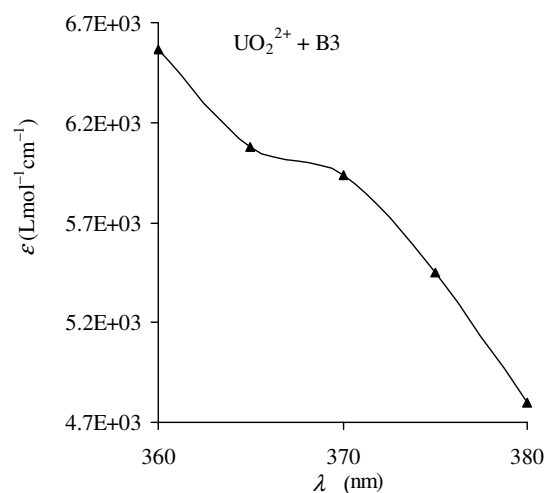
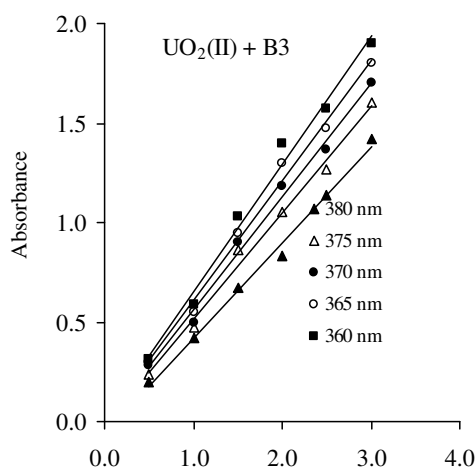
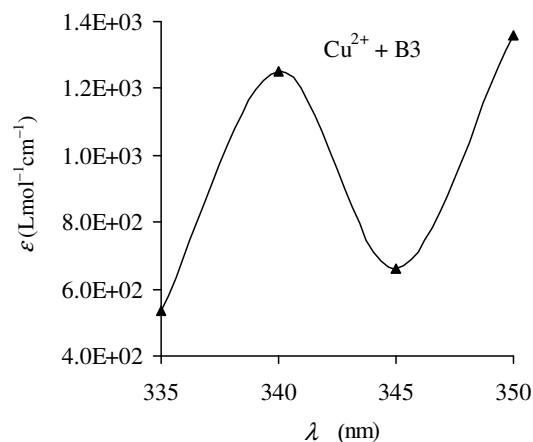
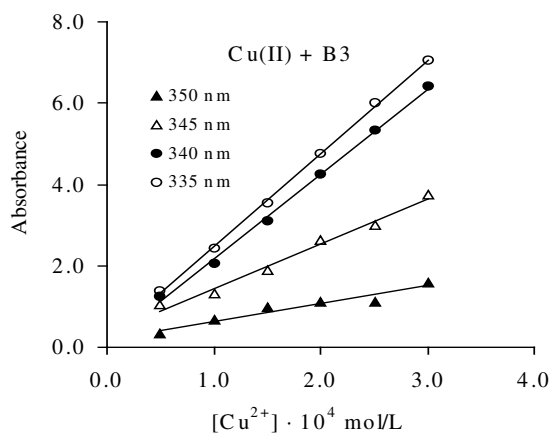


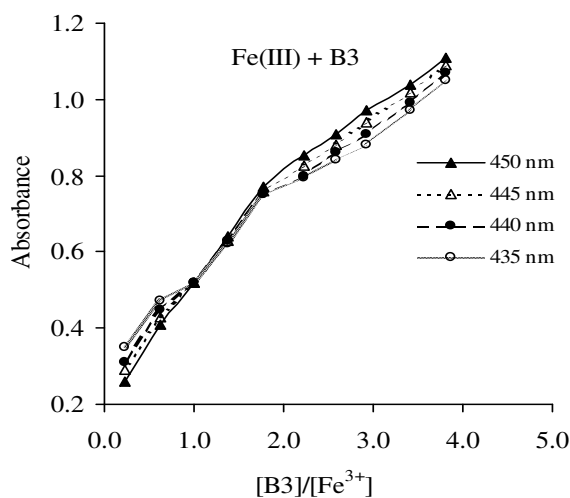
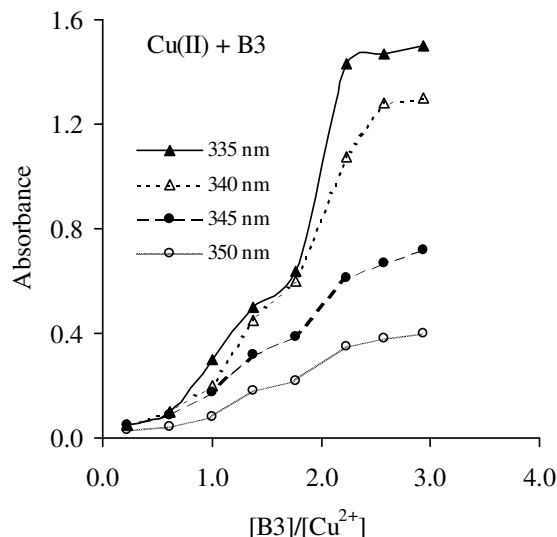
Fig. 2. Confirmation of the Beer's law for the investigated complexes
Absorption spectra

The entitled ligand B3 reacts with trivalent iron to form a yellow complex which has an absorption maximum at 440 nm at optimum pH = 3.5. The Beer's law was valid over a range of concentrations from 5 – 60 ppm. Determination of Fe(III) was also carried out using the standard method [28]. The iron in clay and limestone was determined using the present ligand where it gave nearly a similar result to that obtained with EDTA [31–33]. The Fe(III) % obtained by the new reagent had an error in the range of 0.01 %. The formed green Cu-ligand complex had an absorption maximum at 340 nm at pH = 6.0. The Beer's law was valid in the range of concentrations from 6 – 70 ppm [34–39]. The yellow orange UO₂(II)-ligand complex obeyed the Beer's law in a wide range of concentration (27–270 ppm) at optimum pH = 4.0 and $\lambda = 370$ nm [40–42].

Characteristics of the complexes

The colored ligand-metal complexes could be formed rapidly at 10 – 40 °C and their absorbances remained stable for at least a week at 25 °C. The compositions of these complexes were determined by different methods such as the molar ratio method [43], the method of continuous variation (the Job's method) [44, 45], the Haymann's method [46] and the straight-line method [47]. The molar ratio method carried out on a series of 50 ml solution which was previously prepared by mixing $1 \cdot 10^{-5}$ M metal ions with ligand of concentration range $2 \cdot 10^{-6}$ – $3 \cdot 0 \cdot 10^{-5}$ M at the optimum pH for each complex and the graphs plotted for the molar ratio method were shown in Fig. 3. The data indicated that the composition of the formed complexes was as 1:1 and 1:2 (metal ions : ligand). The continuous variation method proceeded on a series of constant concentrations ($6 \cdot 10^{-5}$ M) at the optimum pH (Fig. 4). The results confirmed the composition of the formed complexes as 1:1 and 1:2. The Haymann's method was discussed using a series of 20 ml solution which was prepared by mixing the metal ions and ligand keeping the ratio of metal ions : ligand 1:2 and the concentration of metal ions regularly increased from $2.5 \cdot 10^{-21}$ M to $3.0 \cdot 10^{-3}$ at desired pH. The plotting $C_M \cdot C_L / A_\lambda$ Vs. $(C_L + C_M)$, where C_L and C_M were the concentration of the ligand and the metal ions respectively, A_λ was the absorp-

tion at certain λ , gave a good straight line which confirmed 1:2 composition (Fig. 5).



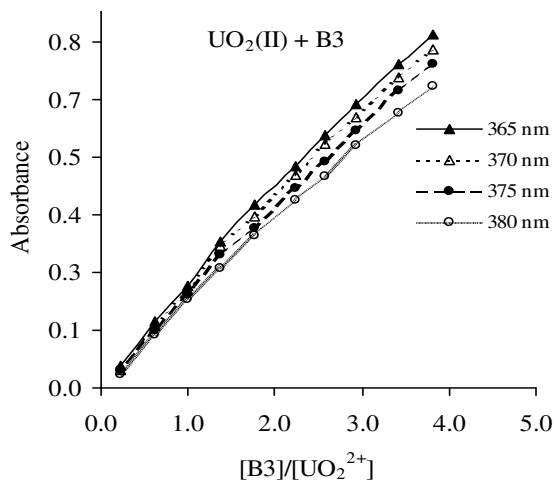


Fig. 3. Determination of the formed M-B3 complexes by the molar ratio method

The straight-line method was carried out using constant metal ions concentration at $1 \cdot 10^{-3}$ M while the ligand concentration varied from $0.6 \cdot 10^{-3}$ to $3 \cdot 10^{-3}$ M and the pH of solution was adjusted at the optimum value for each complex. The plot $1/V^n$, (where V = volume of ligand added and $n = 1, 2, 3$), vs. $1/A_\lambda$ gave a linear relationship, when $n = 1, 2, 3$, indicating 1:1 and 1:2 complexes and also plotting $\log V_L$ vs. $\log A_\lambda$ gave straight lines which confirmed the 1:1 and 1:2 complexes.

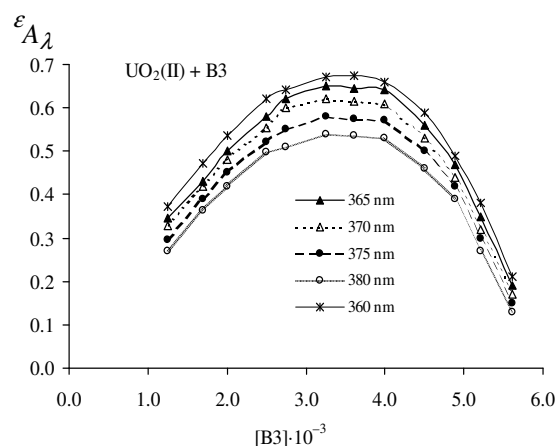
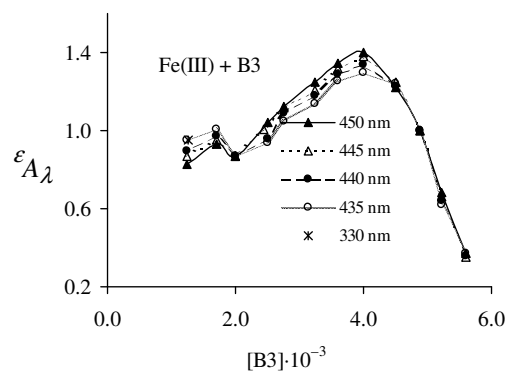


Fig. 4. Determination of the stoichiometries of the metal complexes by the Job's method

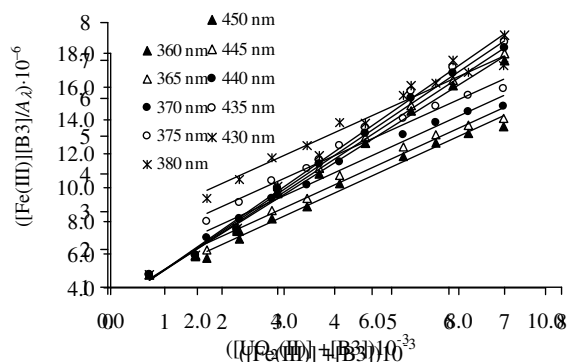
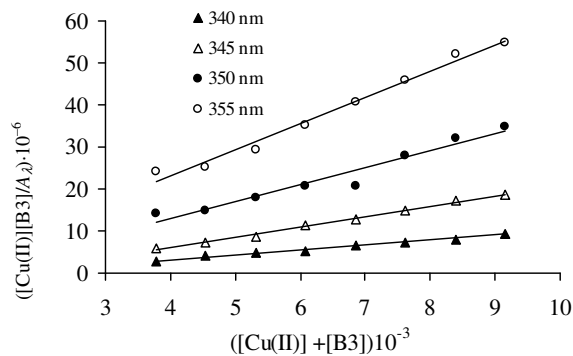
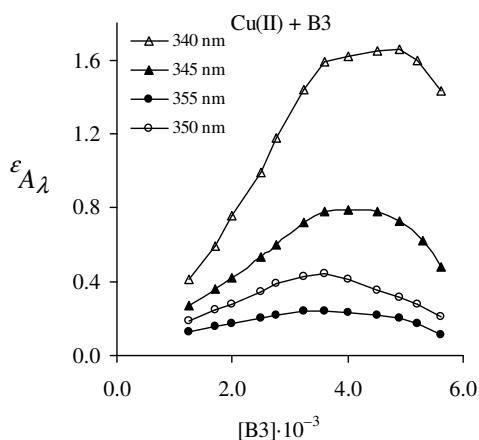
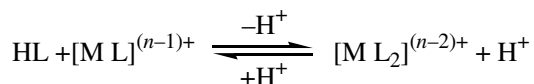
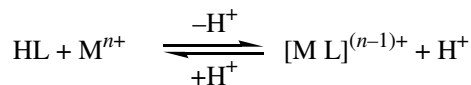


Fig. 5. Determination of the stoichiometries of the metal complexes by the Haymann's method

From this result we could postulate the interaction of metal ions with ligand as:



where M = Fe(III), Cu(II) or UO₂(II).

Complexation kinetics

The kinetics of the complexation of Fe(III) with the ligand was investigated by studying the factors that affect the rate of reaction (Table 1).

Table 1

The calculated $t_{1/2}$ for the reaction between Fe(III) ion and the ligand

Series (1)	[Ligand] = 4.0·10 ⁻³ M, pH = 3.5 and λ = 440 nm		
[Fe(III)]	0.001	0.002	0.003
<i>k</i> (s ⁻¹)	7.40·10 ⁻³	4.76·10 ⁻³	6.45·10 ⁻³
<i>t</i> _{1/2} (s)	93.65	145.59	107.44
Series (2)	[Fe(III)] = 2.0·10 ⁻³ M, pH = 3.5 and λ = 440 nm		
[Ligand]	0.004	0.005	0.006
<i>k</i> (s ⁻¹)	4.55·10 ⁻³	4.65·10 ⁻³	4.18·10 ⁻³
<i>t</i> _{1/2} (s)	152.31	149.03	165.79
Series (3)	[Ligand] = 4.0·10 ⁻³ M, [Fe(III)] = 2.0·10 ⁻³ M and λ = 440 nm		

Table 2

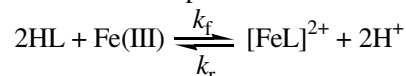
Interference of foreign ions on the determination of 10 mg/ml of the investigated cations under optimum conditions

Foreign ion	Tolerance limit:		
	weight of species/weight Fe(III)	weight of species/weight Cu(II)	weight of species/weight UO ₂ (II)
Cr ⁶⁺ , Ni ²⁺ , Cr ³⁺ , Mo ⁶⁺ , Mn ²⁺ , Te ⁴⁺	100	89	33
Se ⁴⁺ , Hg ²⁺ , Mg ²⁺ , Ca ²⁺	91	140	55
Na ⁺ , NH ₄ ⁺ , Ag ⁺ , K ⁺ , Pb ²⁺ , Cl ⁻ , Br ⁻ , I ⁻ ,	86	111	67
C ₂ O ₄ ²⁻ , NO ₃ ⁻ , CH ₃ COO ⁻ ,	78	66	160
S ₂ O ₃ ²⁻	70	90	140
Al ³⁺ , Zn ²⁺	50	100	94
Co ²⁺	34	40	36
NO ₂ ⁻ , F ⁻	157	160	180

Statistical comparison

pH	3.0	3.5	4.0
<i>k</i> (s ⁻¹)	5.56·10 ⁻³	8.33·10 ⁻³	4.55·10 ⁻³
<i>t</i> _{1/2} (s)	124.64	83.19	152.31

The rate has been found nearly unaffected with the variation of ligand concentration, but affected by Fe(III) ions concentration. Thus, the obtained data fitted nicely a pseudo first order and the reaction could be represented as follows:



A plot of ln ([A_o] - [A_e])/([A_t] - [A_e]) versus time gave a straight line of slope representing the rate of reaction [48, 49]. The *t*_{1/2} was calculated using the following equation: *t*_{1/2} = 0.693/*k* (Table 1). From the result we found the rate of reaction was maximum at pH = 3.5 [Fe(III)] = 0.001 M and [ligand] = 0.003 M and was minimum at pH = 4.0 [Fe(III)] = 0.002 M and [ligand] = 0.003 M.

Interference study

The effect of coexisting ionic species on the determination of each of the investigated cations was investigated. More than 25 ions were examined for their possible interferences in the determination of 10 mg/ml of each of iron(III), copper(II) and uranyl(II) under the optimum conditions. The tolerance limit was defined as the concentration, which gave an error of 3.0 %. The results are presented in Table 2. From the results, it is concluded that the method is free from interferences of many foreign ions.

A comparison was made between the proposed method and the Reference method and the results are given in Table 3. This comparison was made to establish whether the proposed system gives reliable results and is accepted for the determination of iron^{III}, copper^{II} and uranyl^{II}. We conducted the null hypothesis testing and a *t*-test with a multiple sample mean (paired by difference) was applied to examine whether the two methods differ significantly at a 95 % confidence level.

Table 3

Comparison of the results (as mg/ml) obtained for the investigated cations.

All values are averages from four determinations

Samples	Proposed method	Reference method	Claimed values
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Iron(III)	7.34 ±0.033	7.30 ±0.012	7.5
Copper(II)	12.74 ±0.032	12.63 ±0.431	13
Uranyl(II)	17.77 ±0.070	17.89 ±0.357	18

The *t* calculated values for samples 1, 2, 3 and 4 are 1.87, 0.74, 0.63 and 0.44, respectively, for the manual UV-visible spectrophotometric method. The tabulated critical value of *t* at 95 % confidence level and six degrees of freedom is 2.45 [50]. Since, the calculated *t*-values are much less than the tabulated critical value, the null hypothesis (H₀) cannot be rejected and indicates that there is no significant difference between the proposed and manual methods for the determination of iron(III), copper(II) and uranyl(II).

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Резиме

**СПЕКТРОФОТОМЕТРИСКО ОПРЕДЕЛУВАЊЕ НА ЈОНИТЕ НА Fe(III), Cu(II) И UO₂(II)
СО НОВ АНАЛИТИЧКИ РЕАГЕНС ДОБИЕН СО КОНДЕНЗАЦИЈА
НА МОНОЕТАНОЛАМИН И АЦЕТИЛАЦЕТОН**

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Клучни зборови: етаноламин; Schiff-ови бази; спектрофотометрија; ацетилацетон; кондензација

Предложен е високочувствителен и селективен спектрофотометриски метод за директно определување на траги од Fe(III), Cu(II) и UO₂(II) во водни раствори. Методот е базиран на реакцијата на овие катјони со нов аналитички реагенс 2-етанолимино-2-пентилидино-4-он (В3). При оптимални услови на реакцијата и при оптимални други аналитички параметри, В3 реагира со испитуваните катјони и формира обоени комплекси. Утврден е и оптималниот рН за формирање на комплексите. Обоената реакција е брза и апсорбанцата е стабилна најмалку една недела при собна температура. Комплексот на Fe(III) се детектира на $\lambda_{\max} = 440$ nm и рН = 3,5, комплексот на Cu(II) на $\lambda_{\max} = 340$ nm и рН = 6,0, додека комплексот на UO₂(II) на $\lambda_{\max} = 370$ nm и рН = 4,0. Ламбер-Беровиот закон важи во концентрационото подрачје од 0,5 до $3,0 \cdot 10^{-4}$ mol/l, односно од 2 до 17 $\mu\text{g/ml}$ за комплексот на Fe(III), од 3 до 9 $\mu\text{g/ml}$ за комплексот на Cu(II) и од 13 до 81 $\mu\text{g/ml}$ за комплексот на

UO₂(II). Определена е и стехиометријата на формираните комплекси со примена на различни спектрофотометриски методи. Утврдени се и условите за формирањето на комплексите. Пресметана е и брзината на реакцијата помеѓу јонот на Fe(III) и лигандот, претпоставувајќи кинетика од псевдо-прв ред. Испитана е можноста на предложениот лиганд за определување на ниски концентрации од јоните на Fe(III), Cu(II) и UO₂(II) и добиените резултати се анализирани со статистички параметри за да се добие минимална грешка. Во детали е испитувано влијанието на различни супстанции на определувањето на испитуваните катјони. Резултатите покажуваат дека може повеќето од испитуваните супстанции да се толерираат до соодветни количества. Предложениот метод е чувствителен, брз, селективен и едноставен без претходна сепарација или екстракција. Применет е на реални примероци и добиени се задоволителни резултати.