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SPECTROPHOTOMETRIC DETERMINATION OF Cu(II), Co(II) AND Ni(II) IONS IN MONO AND MULTI-COMPONENT SYSTEMS

BY

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Abstract. The selective determination of Cu(II), Co(II) and Ni(II) ions from aqueous solution was spectrophotmetrically investigated using rubeanic acid as color reagent. The reaction between Cu(II), Co(II) and Ni(II) ions with rubeanic acid occurs rapidly at pH 3.5 in case of Cu(II) ions and at pH 9.0 in case of Co(II) and Ni(II) ions (buffered solutions). The metal complexes have absorption maximums at different wavelength (380 nm for Cu(II), 480 nm for Co(II) and 590 nm for Ni(II), respectively) and this characteristic underlies the possibility of their determination in multi-components systems. This method allows the determination of these metal ions in a relatively wide concentration range, with acceptable detection limits. The interferences caused by each metal ion in the determination of the other were evaluated on the basis of the selectivity coefficients. The validation of Cu(II), Co(II) and Ni(II) determination through this method was done by recovery tests of metal ions in tap water. The obtained results shows that this spectrophotometric method can be successfully used for the analysis of Cu(II), Co(II) and Ni(II) ions in water samples, whether are in mono- or multi-components systems.

Keywords: metal ions; rubeanic acid; aqueous solution; spectrophotometric method.

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1. Introduction

Metal ions, such as Cu(II), Co(II) and Ni(II), are still used in many technological processes (Qu *et al*., 2013; Silva *et al*., 2015), and for this reason the aqueous effluents often contains important concentrations of these metal ions. The discharge of such effluents in environment has serious negative consequences on ecosystems quality, mainly because the metal ions are considered persistent pollutants which cannot be destroyed or degraded, and have an accumulation tendency (Khan *et al*., 2007; Ahmaruzzaman, 2011). Various and serious diseases (such as neurological, cardiac, renal, digestive, etc.) (Salem *et al*., 2000; Mishra *et al*., 2010) are caused by the increased level of such metal ions in human body, due to the environment pollution. In consequence, the detection and analysis of such metal ions is still considered important, since the permissible limits in aqueous effluent becomes progressively low.

Many modern and sophistical methods (such as flame atomic absorption spectrometry, inductively coupled plasma-mass spectrometry, inductively coupled plasma emission spectrometry, neutron activation analysis, anodic stripping voltammetry, etc.) have been developed for the rapid detection and analysis of Cu(II), Co(II) and Ni(II) ions from aqueous media, both in mono- or multi-component systems (Ichinoki *et al*., 1987; Sancho *et al*., 2000; Vinas *et al*., 2000; Citak *et al*., 2009; Zhu *et al*., 2011), from various kind of samples. Unfortunately, most of these methods are unsuitable for the determination of metal ions in the analysis laboratories from industry, because of high cost of maintenance, expensive equipments, multi-step and complicated sample preparation, time consuming procedures and well-controlled experimental conditions. From this point of view, the development of rapid, simple and inexpensive method that can be used in industrial field for usual analysis is still one area of interest.

UV-VIS molecular absorption spectrometry (or spectrophotometry) is an adequate method for the development of simple, rapid and inexpensive analytical procedures for determination of various analytes (Flaschka and Barnard, 1972; Dean, 1995; Abbasi-Tarighat and Afkhami, 2012; Moghadam *et al*., 2016). This method is based on the property of colored solutions, which contain the analyte, to absorb radiation with specific wavelength (Bulgariu, 2011). The main characteristics (such as simplicity, short work time, adaptability, feasibility of wide-range of concentration, accuracy, inexpensive equipments, low cost, etc.) (Christian, 1994) make this method suitable for analysis of various analytes (including metal ions) in industrial laboratories.

Unfortunately, in case of determination of metal ions by a spectrophotometric method, the absence of chromophore groups makes that their solutions to be colorless or weak-colored, and in consequence it is necessary the use of color reagent, which must form a colored complex with analyzed metal ion. Various color reagents have been used in literature for the spectrophotometric determination of Cu(II), Co(II) and Ni(II) ions in aqueous media, in single-component systems. Some examples of such color reagents used for this purpose are presented in Table 1.

Table 1

Some Example of Color Reagents Used for Spectrophotometric Determination of Considered Metal Ions

Color Reagent	Analyzed Metal Ion	Reference
bis-thiosemicarbazone	Cu(II), Co(II), Ni(II)	Moghadam *et al.*, 2016
Schiff base	Cu(II), Co(II), Ni(II)	Afkhami *et al.*, 2009
thiosemicarbazone	Cu(II), Co(II), Ni(II)	Chandra and Kumar, 2007
zincon	Cu(II), Co(II), Ni(II)	Ghasemi *et al.*, 2003
1-(2-thiazolylazo)-2-naphthol	Cu(II), Co(II), Ni(II)	Niazi and Yazdanipour, 2008
nitroso-R-salt	Cu(II), Co(II), Ni(II)	Ghasemi *et al.*, 2004

Difficulties appear when it is necessary the determination of metal ions in multi-components systems, as is the case of most real samples. This is because the color reagents can complexed with many metal ions from analyzed samples, and the formed colored complexes causes interferences in spectrophotometric measurements.

In this study, the selective determination of Cu(II), Co(II) and Ni(II) ions from aqueous solution was spectrophotometrically examined, using rubeanic acid as color reagent. The analytical characteristics of spectrophotometric determination of each metal ion were established on the basis of experimental results. The interferences caused by each metal ion in the determination of the other were also evaluated experimental. The validation of Cu(II), Co(II) and Ni(II) determination through this method was done by recovery test of metal ions in tap water samples.

2. Experimental

2.1. Materials

Stock solutions of 650 mg M(II)·L^{-1} (M(II) – Cu(II), Co(II) and Ni(II) respectively), were prepared by dissolving metal sulfate salts in distilled water. Working solutions of metal ions were prepared by dilution from the stock solution with distilled water. The rubeanic acid solution was obtained by dissolving 0.05 g of solid reagent in 100 mL of 96% ethanol. The pH values required for metal ions complexation was obtained by using buffer solution (citrate buffer for pH 3.5, and borate buffer for pH 9.0), prepared according with the standard procedure (Seracu, 1989). All the chemical reagents used in the experiments were of analytical reagents degree and were used without supplementary purifications. Spectrophotometric measurements were performed with Digital Spectrophotometer S 104 D, in a 1.0 cm glass cells, at room temperature.

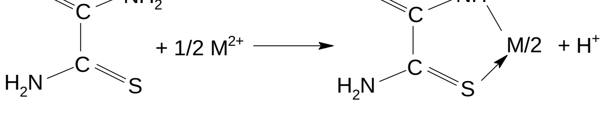
2.2. Methods

Volumes between 0.5 and 2.0 mL of Cu(II) (32.96 mg·L^{-1}), Co(II) (29.87 mg·L^{-1}), and Ni(II) (30.63 mg·L^{-1}) respectively, were transferred into 25 mL volumetric flasks. 5.0 mL of buffer solution (with pH of 3.5 (citrate buffer) in case of Cu(II) ions and with pH of 9.0 (borate buffer) in case of Co(II) and Ni(II) ions) and 1.0 mL of rubeanic acid ethanolic solution (0.05%) were added. The solutions were diluted to the mark with distilled water and mix. The absorbance of each solution was measured at 380 nm in case of Cu(II), at 470 nm in case of Co(II) and at 590 nm in case of Ni(II) respectively, against blank solutions which have the same composition but without metal ions. These values of wavelength corresponding to the maximum of absorption were established experimental, from absorption spectra. For the recording of absorption spectra, 2.0 mL of each metal ion solution was processed as mentioned above, and the absorbance measurements were performed in wavelength domain between 340-790 nm, both against blank solutions and distilled water. The selectivity coefficients were calculated for each case, as ratio between the analyzed metal ion concentration and interfering ion concentration which gives a 5% change of absorbance in a reference solution (Christian, 1994). The recovery test was done using tap water from laboratories of our Faculty. The content of Cu(II), Co(II) and Ni(II) respectively, were determined using a prepared calibration graph.

3. Results and Discussions

3.1. Qualitative Characterization of Spectrophotometric Method

It is well known that rubeanic acid is a versatile ligand which form colored complexes with many metal ions (Soylak and Erdogan, 2006), including Cu(II), Co(II) and Ni(II), according with the reaction:



(1)

In order to determine the qualitative characteristics of absorbing species, the VIS absorption spectra of each complex was recorded in 340-790 nm range, against distilled water. The obtained spectra are illustrated in Fig. 1. Under these conditions, the rubeanic acid has a maximum absorbance at 340 nm, but in the same spectrum region are obtained and the highest values of absorbance for the Cu(II), Co(II) and Ni(II) complexes. Because the absorption maximum both for reagent solution (rubeanic acid) and metal ions complexes are very close, the absorbance measurements must be performed against blank solutions, which have the same composition but without metal ions. The absorption spectra of metal ions complexes recorded against blank solutions are presented in Fig. 2.

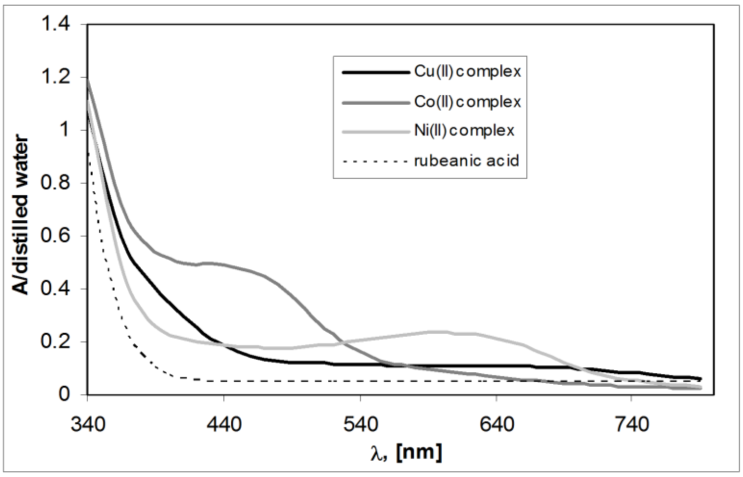


Fig. 1 − Absorption spectra recorded for rubeanic acid and Cu(II), Co(II) and Ni(II) complexes with rubeanic acid, against distilled water.

It can be observed from Fig. 2 that in case of Ni(II) the highest value of absorbance is obtained at 590 nm, and this value was considered as λ_{max} and was used for quantitative determinations, while in case of Cu(II) and Co(II), both complexes have a maximum of absorption in 370-410 nm region. The values of λ_{max} in case of Cu(II) and Co(II) complexes were established considering the shape of obtained absorption spectra. Thus for Cu(II), the narrow absorption band has a maximum at 380 nm, and this value was considered as λ_{max}. In case of Co(II), the ratio between absorbances measured for Co(II) and Cu(II) is highest at 470 nm, and this value was considered the maximum of absorption for Co(II) complex (λ_{max}).

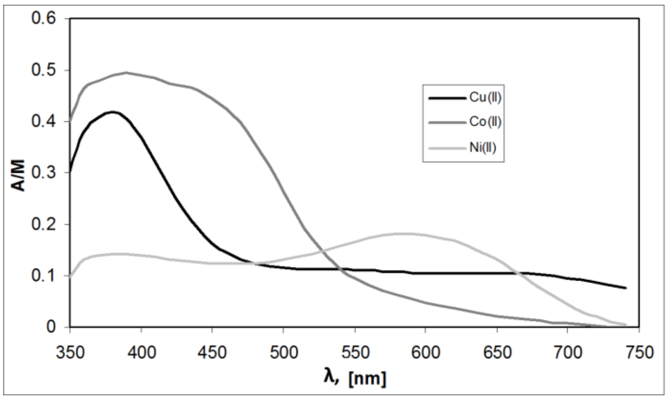


Fig. 2 – Absorption spectra of Cu(II), Co(II) and Ni(II) complexes with rubeanic acid recorded against blank solutions (M).

On the basis of absorption spectra presented in Fig. 2, the main qualitative absorption characteristics of Cu(II), Co(II) and Ni(II) complexes with rubeanic acid were calculated, and the obtained values are summarized in Table 2.

Table 2

Qualitative Absorption Characteristics of Studied Metal Ions Complexes

Characteristic	Cu(II)	Co(II)	Ni(II)
λ_{max}, [nm]	380	470	590
$\Delta\lambda_{1/2}$, [nm]	98	241	218
ε, [L mol^{-1} cm^{-1}]	1.0072 10^{4}	7.6940 10^{3}	4.3601 10^{3}

It can be observed from Table 2 that the difference between λ_{max} values corresponding to the three complexes is quite high (around 100 nm), and this suggest the possibility to use this method for the quantitative determination these metal ions in multi-component systems.

3.2. Quantitative Analysis in Mono and Multi-Component Systems

In order to test the applicability of this method for the quantitative analysis of Cu(II), Co(II) and Ni(II) ions from aqueous solutions, the calibration curves were constructed in mono and multi-components systems.

In mono-component systems, the calibration curves (Fig. 3) were obtained for each metal ion as is described in the experimental section, using four standard solutions with different concentrations, and measuring the absorbance against blank solutions (M) at wavelength of λ_{max}.

The linear regression equations (inside of Fig. 3) was obtained by use the mean values of seven replicate measurements, where y represents the absorbance, x is the concentration of each metal ion and R^2 is the regression coefficient.

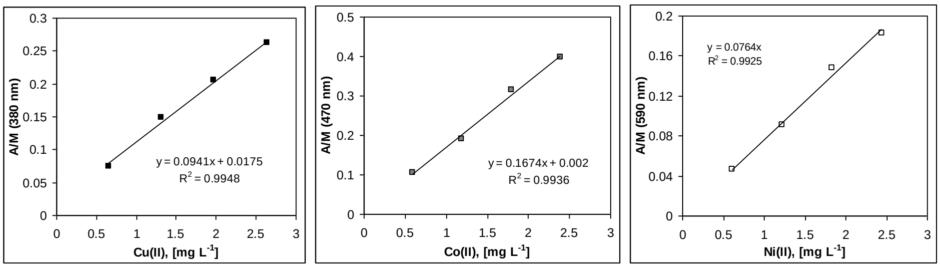


Fig. 3 – Calibration curves obtained in case of spectrophotometric determination of Cu(II), Co(II) and Ni(II) with rubeanic acid.

The main quantitative characteristics of Cu(II), Co(II) and Ni(II) spectrophotometric determination with rubeanic acid in mono-component systems, such as linear dynamic range, detection limit (calculated as three times of standard deviation of seven replicate measurements (Christian, 1994)), the calibration sensitivity (which is slope of calibration curve), precision (RDS, %), etc., are summarized in Table 3.

Table 3

Quantitative Characteristics of Spectrophotometric Method

Characteristic	Cu(II)	Co(II)	Ni(II)
Color reagent	Rubeanic acid ethanolic solution (0.05 %)		
λ_{max}, [nm]	380	470	590
Linear range, [mg L^{-1}]	0.65 – 2.65	0.50 – 2.40	0.60 – 2.45
Regression coefficient	0.9948	0.9936	0.9925
Calibration sensitivity, [L mg^{-1}]	0.0941	0.1674	0.0764
Limit of detection, [ppm]	2.31	2.58	2.65
RDS, [%]	1.16	2.08	1.97

The quantitative characteristics presented in Table 3 shows that the spectrophotometric method which use rubeanic acid as color reagent, is rapid, sensitive, accurate, has a reasonable linear concentration range and can be successfully used for the analysis of Cu(II), Co(II) and Ni(II) ions from aqueous solution, in mono-component systems, including in industrial laboratories, mainly due to its low cost.

Unfortunately, the mono-component systems have a limited applicability in practice. In most of real samples, beside the analyzed ion there are other chemical species, which can influence the accuracy of its determination.

In this study, the influence of Cu(II), Co(II) and Ni(II) ions have each in the determination of the other was assessed using selectivity coefficients, while the influence of other chemical species on the determination of studied ions was evaluated using the recovery tests.

The selectivity coefficients ($k_{i,j}$) were calculated for each case as the ratio between analyzed ion concentration (c_i, [mg L^{-1}]) and interfering ion concentration (c_j, [mg L^{-1}]) which gives a 5% absorbance change in a reference solution. As reference solutions were used solutions with constant concentration of each metal ion (1.3184 mg Cu(II) L^{-1}, 1.1948 mg Co(II) L^{-1} and 1.2252 mg Ni(II) L^{-1}, respectively), constant concentration of rubeanic acid ethanolic solution (1 mL, 0.05 %) and adequate pH (3.5 for Cu(II) and 9.0 for Co(II) and Ni(II)). The calculated values of the selectivity coefficients are summarized in Table 4.

Table 4

The Values of Selectivity Coefficients

Analyzed Ion / Interfering Ion	LOG $K_{I,J}$		
	Cu(II)	Co(II)	Ni(II)
Cu(II)	–	–0.6002	–1.1157
Co(II)	0.5531	–	–1.2874
Ni(II)	–1.0774	–0.5524	–

The values obtained for the selectivity coefficients indicate that any metal ion interfere in the determination of the others only when its concentration is at least 4 times higher than the concentration of the analyzed one. The more accurate determination is obtained in case of Ni(II) ions, where is necessary a concentration of Cu(II) and Co(II) at least 15 times higher than the concentration of Ni(II) ions to produce a change of absorbance by 0.05 units.

On the basis of the values presented in Table 4 it can be said that the spectrophotometric determination of Cu(II), Co(II) and Ni(II) ions with rubeanic acid as color reagent can be done in multi-component systems, with the condition that their concentrations to have the same order of magnitude. This condition is fully satisfied by the many wastewater samples resulted from galvanization processes, and in such cases this spectrophotometric method can be used for the determination of these metal ions, even in industrial laboratories.

In order to check if the other components present in real water samples interfere in the determination of Cu(II), Co(II) and Ni(II) ions with rubeanic acid, the recovery tests were done, using tap water from the laboratories of our faculty. In order to examined the recovery of Cu(II), Co(II) and Ni(II) by this method, 10 mL of tap water was transferred to each of three series 25 mL volumetric flasks, and 1.0, 1.5 and 2.0 mL of metal ions standard solution was added to each flask. All the samples were analyzed according with the procedure described in experimental sections. The metal ions content in each sample was determined using a calibration graph at 380 nm in case of Cu(II), at 470 nm in case of Co(II) and at 590 nm in case of Ni(II), respectively. The average values of recovery percents obtained for the addition of metal ions solutions to tap water samples are presented in Table 5.

Table 5

The Recovery Test

Cu(II)		Co(II)		Ni(II)	
Added [mg L^{-1}]	Recovered [%]	Added [mg L^{-1}]	Recovered [%]	Added [mg L^{-1}]	Recovered [%]
1.3184	99.65	1.1948	99.48	1.2252	95.16
1.9776	97.57	1.7922	105.57	1.8378	103.73
2.6368	101.80	2.3896	92.63	2.4504	101.76

As can be observed from Table 5, the good recovery of all studied metal ions (Cu(II), Co(II) and Ni(II)) from tap water sample was obtained, indicating that the constituents of water samples do not significantly interfere in their spectrophotometric determination with rubeanic acid. Therefore, this method can be a good alternative for the determination of Cu(II), Co(II) and Ni(II) from aqueous solution, both in mono and multi-components systems, and can have large applicability in the industrial laboratories.

4. Conclusions

In this study, the selective determination of Cu(II), Co(II) and Ni(II) ions from aqueous solution by a spectrophotometric method using rubeanic acid as color reagent is proposed. The color reaction between studied ions and rubeanic acid occurs rapidly, in buffered solution (pH of 3.5 for Cu(II), and pH of 9.0 for Co(II) and Ni(II)), and the formed metal complexes have a maximum of adsorption at 380 nm in case of Cu(II), 480 nm in case of Co(II) and 590 nm in case of Ni(II), respectively. The high differences between maximum absorption wavelengths suggest the possibility of determination of these ions in mono and multi-components systems. In mono-component systems, the determination of studied metal ions can be done in a relatively wide concentration range, with acceptable detection limits. The interferences caused by each metal ion in the determination of the other were evaluated on the basis of selectivity coefficients. The obtained values have indicate that it is possible the determination of these metal ions and in multi-component systems, with the conditions that their concentration in the mixture, to have the same order of magnitude. The validation of Cu(II), Co(II) and Ni(II) determination through this method was done by recovery test of metal ions in tap water. The obtained results shows that the spectrophotometric method which use rubeanic acid as color reagent can be successfully used for the analysis of Cu(II), Co(II) and Ni(II) ions in water samples, whether they are in mono- or multi-components systems.

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DETERMINAREA SPECTROFOTOMETRICĂ A IONILOR DE Cu(II), Co(II) ŞI Ni(II) ÎN SISTEME MONO- ŞI MULTI-COMPONENT

(Rezumat)

Determinarea selectivă a ionilor de Cu(II), Co(II) şi Ni(II) din soluţii apoase a fost investigată spectrofotometric utilizând acidul rubeanic ca şi reactiv de culoare. Reacţia dintre ionii de Cu(II), Co(II) şi Ni(II) cu acidul rubeanic are loc rapid, la pH 3,5 în cazul ionilor de Cu(II), şi la pH 9,0 în cazul ionilor de Co(II) şi Ni(II) (soluţii tamponate). Complecşii metalici formaţi au maximele de absorbţie la lungimi de undă diferite (380 nm pentru Cu(II), 480 nm pentru Co(II) şi respectiv 590 nm pentru Ni(II)), iar această caracteristică evidenţiază posibilitatea determinării lor în sisteme multi-component. Această metodă permite determinarea acestor ioni metalici într-un domeniu de concentraţie relativ larg, cu limite de detecţie acceptabile. Interferenţele cauzate de fiecare ion metalic în determinarea celuilalt au fost evaluate pe baza determinărilor experimentale. Validarea determinării Cu(II), Co(II) şi Ni(II) prin această metodă s-a realizat prin teste de recuperare a ionilor metaici din apa de la robinet. Rezultatele obţinute arată că această metodă spectrofotometrică poate fi utilizată cu succes la analiza ionilor de Cu(II), Co(II) şi Ni(II) din probe de apă, indiferent dacă aceştia sunt în sisteme mono- sau multi-component.