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QUANTITATIVE SPECTROPHOTOMETRIC DETERMINATION OF Au(III) IONS IN AQUEOUS SOLUTION WITH BASIC VIOLET 10 DYE

BY

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Abstract. Spectrophotometric determination of Au(III) ions with Basic Violet 10 dye is a rapid, sensitive and accurate method that can be used in quantitative analysis. The colour reaction occurs immediately in acidic aqueous solution (pH = 0, HCl solution), and this represents an important advantage from a practical point of view. The qualitative parameters of coloured Au(III)-complex (maximum wavelength (500 nm), width of the spectral band (120 nm) and molar absorption coefficient ($2.55 \cdot 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$)) suggest the possibility of using this reaction in quantitative analysis. Lambert-Beer's law was obeyed in the Au(III) concentration range of 0.4 – 2.0 mg·L⁻¹, while the detection limit and limit of quantification were found to be 0.067 mg·L⁻¹ and 0.22 mg·L⁻¹. This colour reaction is quite selective for Au(III) ions in the presence of some interfering ions (Cu(II), Zn(II), Na(I)), while other metal ions (such as Ca(II), Mg(II), Co(II)) interfere to a great extend. The measurements performed using tap water as a background, showed that there are significant differences between the concentration of Au(III) ions, added and calculated from the calibration curve, which limits the applicability of this method in the analysis of real samples.

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Keywords: Spectrophotometric method; Au(III) ions; Basic Violet 10; quantitative analysis.

1. Introduction

Due to their high chemical and thermal stability, very good electrical conductivity and biocompatibility with biological active organic molecules (Zhang *et al.*, 2020; Roy *et al.*, 2022), the use of Au(III) ions in industrial and research activities is still of great interest. But in addition to these various applications, it is equally important to find an analysis method, which allow the quantitative determination of Au(III) ions, and which must be cheap and easy to use (Cui and Zhange, 2008).

According to studies in the literature, for the quantitative determination of Au(III) ions, especially in the aqueous solution, the use of flame atomic absorption spectrometry (AAS) and inductively coupled plasma mass spectrometry (ICP-MS) is recommended (Christian, 1994; Emre *et al.*, 2016; Tao *et al.*, 2017). These two methods are characterized by high sensitivity and selectivity, and allow the determination of very low concentration of Au(III) ions (below 1 ppm) from samples that have complex compositions (soil, water, biological samples, etc.) (Medved *et al.*, 2004; Pyrzynska, 2005). Unfortunately, both methods require a laborious sample preparation methodology, expensive equipments and specialized personnel for their use, conditions that are not always met in research laboratories where students work.

Potentiometric method is another example of analytical method that can be used for the determination of Au(III) ions in aqueous solutions (Zdrachek and Bakker, 2019). Due to the spontaneous reduction tendency of Au(III) ions, a gold wire electrode can be easily constructed for use in potentiometric measurements. Although the potentiometric method can be easily used in laboratory practice (due to inexpensive equipment and high selectivity), the sensitivity of the method is moderate. In general, the linear dynamic range of potentiometric methods is quite wide (2-3 orders of magnitude) (Dean, 1995), which makes it difficult to determine close concentration of Au(III) ions in different samples as is the case in laboratory experiments.

Spectrophotometric methods are much more suitable for such situations. The simplicity of the experimental methodology, the ease of use of the laboratory equipments, the short working time, the appropriate linear dynamic range, etc., are just some of the most important advantages of using spectrophotometric methods for such determinations (Zuoto and McCreeedy, 1999; Fazli *et al.*, 2009; Nagaraja *et al.*, 2017). The main aspect that must be taken into account in the case of spectrophotometric methods is finding a suitable colour reagent, which will react with the species to be analyzed (under well-defined experimental conditions) and allow the formation of a colored complex. If such a colour reagent selectively reacts with the species to be

analyzed, the spectrophotometric method exhibits high selectivity and, therefore, its area of applicability is wider (Tournebize *et al.*, 2011).

In the case of Au(III) ions, a number of organic reagents (such as rhodanine, aminoquinoline, ammonium peroxy-disulfate, etc.) (El-Zawawy *et al.*, 1995; Zuoto and McCreed, 1999; Rancic *et al.*, 2005) can be used to obtain colored complexes that can be analyzed spectrophotometrically. The spectrophotometric performances of these colour reagents are quite close, therefore the selection of a specific reagent for the determination of Au(III) ions depends mostly on its availability in the laboratory.

Based on these considerations, in this study, Basic Violet 10 dye was used for the quantitative determination of Au(III) ions using a direct spectrophotometric method. The experimental studies aim, on the one hand, to establish the optimal conditions (pH, type of mineral acid, reaction time) to ensure a maximum sensibility of the colour reaction, and on the other hand, qualitative and quantitative characterization of the proposed method. Also, the determination of Au(III) ions was tested using tap water samples, to evaluate the applicability of the proposed method.

2. Experimental

A $100 \text{ mg}\cdot\text{L}^{-1}$ HAuCl_4 solution (purchased from Sigma-Aldrich) was used to prepare the standard solutions needed in the experimental studies. The acid media was obtained by adding volumes of 2N mineral acid solution (HCl, HNO_3 or H_2SO_4) (from Chemical Company, Iași). Basic Violet 10 dye solution (0.05%) was obtained by dissolving a given amount of solid dye (purchased from Sigma-Aldrich) in ethanol (96%).

The experimental studies aimed, on the one hand, to establish the optimal conditions for obtained the colored Au(III)-dye complex, and on the other hand, to determined the spectrophotometric characteristics of this complex, in order to develop an analysis method. Thus, to establish the optimal reaction conditions, different volumes (0.25 – 2.0 mL) of 2N mineral acids solutions (HCl, HNO_3 and H_2SO_4) and different volumes (0.25 – 2.0 mL) of dye solution (0.05%), were added to a constant volume of 0.5 mL Au(III) solution ($100 \text{ mg}\cdot\text{L}^{-1}$). All samples were diluted with distilled water to a final volume of 25 mL and homogenized. For each sample, VIS spectra were recorded (Digital Spectrophotometer S104 D, 1 cm glass cell, against distilled water), in the spectral domain between 340 and 700 nm, and the spectral parameters (wavelength corresponding to absorption maximum (λ_{max} , nm), maximum absorbance (A_{max}), width of spectral band ($\Delta\lambda_{1/2}$, nm) and molar absorption coefficient (ϵ , $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) were determined. The optimal conditions of the colour reaction were considered those for which the values of A_{max} and ϵ are maximum.

The optimal reaction conditions were then used to obtain the calibration curve. Thus, over 1 mL of 2N HCl solution and 0.5 mL of 0.05% dye solution, different volumes (0.1 – 1.5 mL) of Au(III) solutions ($100 \text{ mg}\cdot\text{L}^{-1}$) were added. For each sample, the absorbance was determined at 500 nm against a blank solution (distilled water). These experimental results were then used to evaluate the applicability of the method in quantitative analysis. The selectivity of the proposed method was evaluated using the selectivity coefficients determined for a series of common interfering ions (Na(I), Ca(II), Mg(II), Cu(II), Zn(II) and Co(II)). Also, to validate the proposed method, different volumes (5.0, 10 and 15 mL) of tap water (taken from our laboratory) were used as background. The concentration of Au(III) ions was adjusted to $1.2 \text{ mg}\cdot\text{L}^{-1}$ using the stock solution, and Au(III) content of these samples was determined from the calibration curve based on the experimentally measured absorbance values.

3. Results and discussion

Basic Violet 10 (Fig. 1) is an organic dye used in analytical chemistry to identify metal ions, including Au(III) ions (Ripan *et al.*, 1963). The colour reaction (Fig. 1) between this dye and Au(III) ions occurs only in string acidic media, when at least one of the N atoms is positively charged and can interact with the negative AuCl_4^- species. As the reaction proceeds, the colour of the solution changes from light yellow to red-orange.

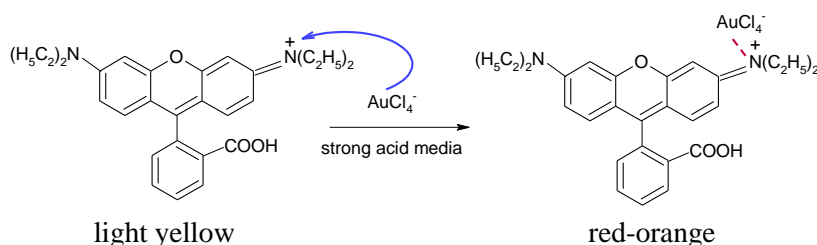


Fig. 1 – Colour reaction between Basic Violet 10 and Au(III) ions.

Therefore, to optimize the reaction conditions, the selection of the type and concentration of the mineral acid is mandatory to test the applicability of this reaction in spectrophotometric determinations.

3.1. Establishing optimal conditions for obtaining acid media

Three mineral acids were tested to obtain the strong acid media required for the colour reaction, namely: HNO_3 , H_2SO_4 and HCl . This, 1 mL of each mineral acid solution (2N) was added to each sample (25 mL), and the experimentally recorded VIS spectra are shown in Fig. 2a.

As can be seen from Fig. 2a, the highest absorption band intensity is obtained in the case of HCl, when the experimentally measured absorbance value at 500 nm (the highest value) is 6.6% higher than in the case of H₂SO₄ and 24.04% higher than in the case of HNO₃. Therefore, HCl can be considered more suitable for achieving the colour reaction between Au(III) ions and Basic Violet 10 dye. This behaviour is easy to understand. The neutralization of the positive charge on the N atom (Fig. 1) by a Cl⁻ ion facilitates the interaction of the AuCl₄⁻ species with the dye molecules, due to the common ion effect. Consequently, the colour reaction occurs easily, and the measured absorbance values are high.

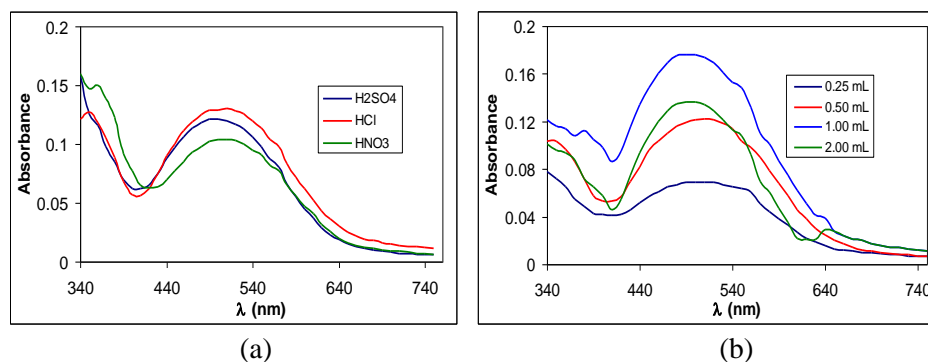


Fig. 2 – VIS spectra of coloured complex between Au(III) ions and Basic Violet 10 dye, at different types of mineral acids (a) and at different concentration of HCl (b).
 (Experimental conditions: $c_{\text{Au(III)}} = 1.2 \text{ mg}\cdot\text{L}^{-1}$; $c_{\text{dye}} = 0.002\%$).

In order to establish the optimal concentration of HCl solution necessary for the development the colour reaction, four samples were prepared. In these samples, the volume of HCl solution was varied between 0.25 and 2.0 mL. The VIS spectra recorded in this case are illustrated in Fig. 2b.

As can be seen from Fig. 2b, increasing the volume of the HCl solution causes an increase in the absorbance values measured in the range 0.25 – 1.0 mL. Analysing the VIS spectra recorded in this case, it can be seen that with the increase in the volume of added HCl, in addition to the increase in the absorbance values, the width of the spectral bands ($\Delta\lambda_{1/2}$) also decreases (from 138 to 121 nm), while the maximum wavelength remains at the same value (500 nm). This behaviour shows that for the protonation of the N atoms in the dye molecule, the pH of the solution must be close to 1.0 (1.09 for 1.0 mL of HCl, 2N). At volumes greater than 1.0 mL, the experimentally measured absorbance values decrease (see Fig. 2b). This decrease in absorbance values recorded at large volume of HCl (2.0 mL) may be due to the much too strongly acid media (pH less than 1.0) that can destroy the organic dye molecules.

Under these conditions, a volume of 1.0 mL of 2N HCl solution was considered optimal to obtain the acid media required for the colour reaction between Au(III) ions and Basic Violet 10 dye, and this value was used in all other experimental studies.

3.2. Optimizing Basic Violet 10 dye concentration

The optimal dye concentration required to achieve the colour reaction with Au(III) ions was determined using VIS spectra (Fig. 3a) recorded under the following experimental conditions: 1 mL 2N HCl solution, $c_{\text{Au(III)}} = 1.2 \text{ mg}\cdot\text{L}^{-1}$.

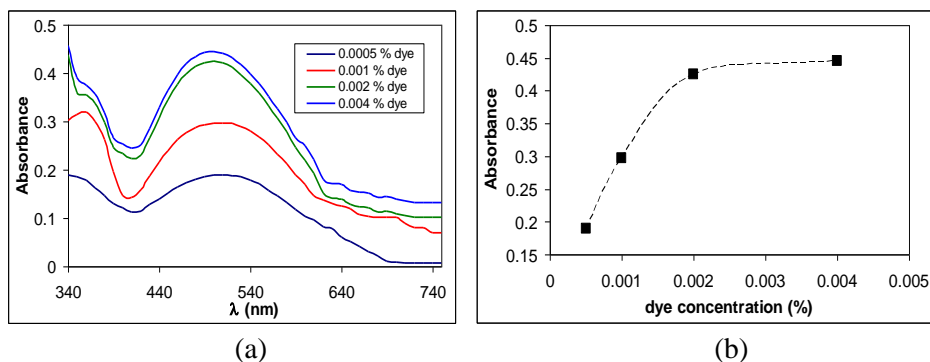


Fig. 3 – (a) VIS spectra of coloured complex between Au(III) ions and different concentrations of Basic Violet 10 dye; (b) Variation of absorbance as a function of dye concentration at 500 nm. (Experimental conditions: $c_{\text{Au(III)}} = 1.2 \text{ mg}\cdot\text{L}^{-1}$; 1 mL 2N HCl solution).

The analysis of the VIS spectra (Fig. 3a) highlights two aspects, namely: (i) regardless the dye concentration, the absorption maximum is obtained at 500 nm, which indicates that the composition of Au(III)-Basic Violet 10 dye complex does not change, and (ii) the maximum absorbance measured at 500 nm increases with the increasing dye concentration up to 0.002%, after which it remains almost constant (Fig. 3b). Increasing the dye concentration from 0.002% to 0.004% causes only a 4.7% increase in absorbance (from 0.425 to 0.445) (Fig. 3b), which is most likely due to the excess of dye (which is coloured in light yellow). Therefore, a concentration of 0.002% of Basic Violet 10 dye was selected as optimal, and was used in all other experimental studies.

3.3. Spectrophotometric characterization of colour reaction

For the spectrophotometric characterization of the colour reaction between Au(III) ions and Basic Violet 10 dye, the VIS spectra were recorded at three different concentrations of Au(III) ions, under the experimental conditions

determined to be optimal (1 mL 2N HCl solution, dye concentration of 0.002%). For each recorded VIS spectrum (Fig. 4), the spectral parameters (maximum wavelength (λ_{\max} , nm), maximum absorbance (A_{\max}), width of spectral band ($\Delta\lambda_{1/2}$, nm) and molar absorption coefficient (ϵ , $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) were determined, and the obtained values are summarized in Table 1. All these parameters are important for the qualitative characterization of the coloured complex, and their determination is the starting point in the design of a quantitative analysis method.

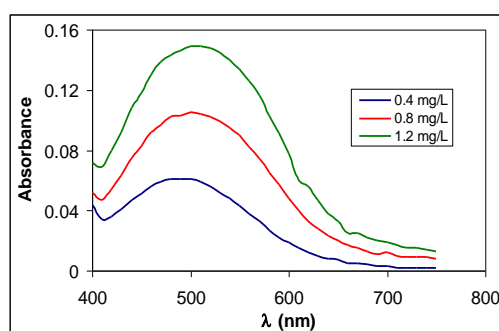


Fig. 4 – VIS spectra of coloured complex obtained at different concentrations of Au(III) ions and constant Basic Violet 10 dye concentration. (Experimental conditions: 1 mL 2N HCl solution, $c_{\text{dye}} = 0.002\%$).

Table 1

The values of spectral parameters obtained for Au(III)-Basic Violet 10 dye complex

$c_{\text{Au(III)}}[\text{mg}\cdot\text{L}^{-1}]$	$\lambda_{\max} [\text{nm}]$	A_{\max}	$\Delta\lambda_{1/2} [\text{nm}]$	$\epsilon [\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}]$
0.4	500	0.061	124	$2.6249 \cdot 10^4$
0.8	500	0.105	118	$2.5863 \cdot 10^4$
1.2	500	0.149	120	$2.4466 \cdot 10^4$

As can be seen from Fig. 4, for all values of the Au(III) ions concentration, the absorption band of the coloured complex shows a maximum at 500 nm, which suggest that the composition of the complex remain de same regardless of the initial concentration of metal ions. This is also supported by the close values of molar absorption coefficient (ϵ , $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) and spectral band widths ($\Delta\lambda_{1/2}$, nm) (Table 1). Based on these results, it was determined that for the Au(III)-Basic Violet 10 dye complex, the maximum absorption is obtained at 500 nm, and this wavelength value provides the highest sensitivity of the colour reaction. Furthermore, due to the close values of the molar absorption coefficient and the increasing dependence of the absorbance on the Au(III) ions concentration (Table 1), this colour reaction can be used in quantitative determinations.

3.4. Calibration curve and quantitative parameters

To use this colour reaction in quantitative analysis, it is necessary to obtain a calibration curve, in which the dependence between the Au(III) ions concentration in the solution and the experimentally measured absorbance is linear, at least for a certain concentration range (according with Lambert-Beer law). In this study, the dependence between Au(III) ions concentration and absorbance was examined for eight standard solutions (0.4 – 6.0 mg·L⁻¹), and the obtained results are illustrated in Fig. 5.

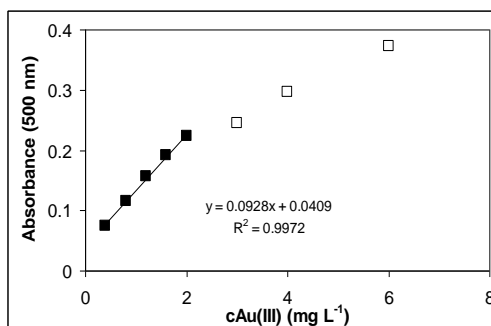


Fig. 5 – Dependence of the experimental absorbance on the concentration of Au(III) ions in the solution. (Experimental conditions: 1 mL 2N HCl solution, $c_{\text{dye}} = 0.002\%$).

Table 2

Analytical characterization of the proposed spectrophotometric method

<i>Analytical parameter</i>	<i>At 500 nm</i>
<i>Blank solution</i>	Distilled water
<i>Calibration sensitivity</i>	0.0928 L·mg ⁻¹
<i>Correlation coefficient</i>	0.9972
<i>Linear dynamic range</i>	0.4 – 2.0 mg·L ⁻¹
<i>Limit of detection (3σ)</i>	0.067 mg·L ⁻¹
<i>Limit of quantification (10σ)</i>	0.2234 mg·L ⁻¹
<i>Precision</i>	2.35%

The experimental data presented in Fig. 5 shows that Lambert-Beer law is obeyed in the concentration range between 0.4 and 2.0 mg·L⁻¹. At higher values of the Au(III) ions concentration, deviations from this law appear, and the dependence between absorbance and concentration is no longer linear. Therefore, this colour reaction can be used in the spectrophotometric determination of Au(III) ions only if the concentration is included in this range (which represent the linear dynamic range) and if the absorbance is measured against distilled water (blank solution) at 500 nm.

The most important quantitative parameters required for the analytical characterization of the proposed method, calculated from the statistical analysis, are presented in Table 2. The values of these parameters: the rather large linear dynamic range, high detection limit and precision, high sensitivity of calibration, etc. (Table 2), highlight the possibility of using this colour reaction in the quantitative determination of Au(III) ions in aqueous solutions.

3.5. Selectivity of spectrophotometric method

The selectivity of the colour reaction for Au(III) ions was tested in the presence of Na(I), Ca(II), Mg(II), Cu(II), Co(II) and Zn(II) ions, considering interfering ions. These ions frequently occur in wastewater from the metal plating industry, and therefore their interference in the determination of Au(III) ions by the proposed spectrophotometric method is important from application point of view.

The selectivity coefficients ($\alpha_{i, \text{Au(III)}}$) were used to evaluate the selectivity of the proposed method. By definition (Faber *et al.*, 2003; Soomro *et al.*, 2008), these selectivity coefficients represent the ratio between the interfering ions concentration (c_i , $\text{mg}\cdot\text{L}^{-1}$) and the Au(III) ions concentration (c_{Au} , $\text{mg}\cdot\text{L}^{-1}$), that causes a 5% change in the experimentally measured absorbance in a reference solution. In this case the concentration of Au(III) ions in the reference solution was $1.2 \text{ mg}\cdot\text{L}^{-1}$, and the concentration of interfering ions was varied between 12 and 38 $\text{mg}\cdot\text{L}^{-1}$. The values of absorbance measured for each interfering ion are illustrated in Fig. 6, while the values of selectivity coefficients are summarized in Table 3.

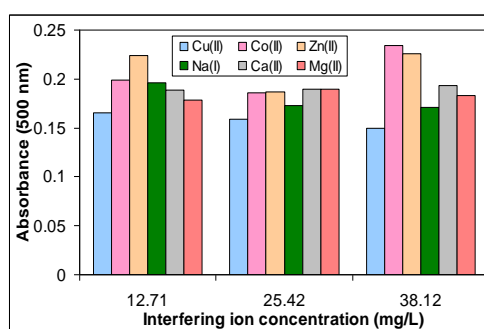


Fig. 6 – Experimental absorbances at different concentration of interfering ions. (Experimental conditions: 1 mL 2N HCl solution, $c_{\text{Au(III)}} = 1.2 \text{ mg}\cdot\text{L}^{-1}$, $c_{\text{dye}} = 0.002\%$).

The values of the selectivity coefficients show that the colour reaction has a fairly high degree of selectivity for Au(III) ions compared to the interfering ions. Thus, according to the values in Table 3, Cu(II), Zn(II) and

Na(I) ions can be present in concentration up to 10^2 higher than the concentration of Au(III) ions to lead to a change in absorbance of 0.05 units, while for Co(II), Ca(II) and Mg(II) ions, the concentration limit is between 30 and 60 times. Therefore, the spectrophotometric determination of Au(III) ions can be done in complex samples, such as real samples. However, the presence of Ca(II) and Mg(II) ions in concentrations higher than $50 \text{ mg}\cdot\text{L}^{-1}$ and $70 \text{ mg}\cdot\text{L}^{-1}$ respectively, as in the case of tap water samples, may interfere with the determination of Au(III) ions by the proposed method.

Table 3
Values of the selectivity coefficients

<i>Interfering ion</i>	$c_j [\text{mg}\cdot\text{L}^{-1}]$	$c_{\text{Au(III)}} [\text{mg}\cdot\text{L}^{-1}]$	$\alpha_{i, \text{Au(III)}}$
Cu(II)	214.20	1.20	178.50
Co(II)	40.23	1.20	33.53
Zn(II)	138.30	1.20	115.25
Na(I)	214.40	1.20	201.67
Ca(II)	59.37	1.20	49.47
Mg(II)	82.29	1.20	68.57

To test this last hypothesis, different volumes (5, 10 and 15 mL) of tap water (from our laboratory) were used as a background for the preparation of the solutions. In each case the added concentration of Au(III) ions was adjusted at $1.2 \text{ mg}\cdot\text{L}^{-1}$ (using standard solution). After measuring the absorbance (at 500 nm, against distilled water), the found concentration of Au(III) in each sample was determined by linear interpolation from the calibration curve. The results obtained for the validation of the proposed method are presented in Table 4.

Table 4
The validation of proposed method.

<i>Sample</i>	$v_{\text{water}} [\text{mL}]$	$c_{\text{Au(III), add}} [\text{mg}\cdot\text{L}^{-1}]$	$c_{\text{Au(III), found}} [\text{mg}\cdot\text{L}^{-1}]$	$\Delta c_{\text{Au(III)}} [\text{mg}\cdot\text{L}^{-1}]$
1	5.0	1.20	1.83	0.63
2	10.0	1.20	2.08	0.88
3	15.0	1.20	2.32	1.12

The results summarized in Table 4 show that as the volume of tap water increase, so does the difference between the added and found Au(III) ions concentration, calculated from the calibration curve. This means that the ions present in tap water significantly influence the spectrophotometric determination of Au(III) ions by proposed method. Therefore, this method can only be used for laboratory solutions.

4. Conclusions

In this study, the colour reaction between Au(III) ions and Basic Violet 10 dye was spectrophotometrically analyzed, both qualitatively and quantitatively. The experimental results showed that in strong acid media (1 mL HCl solution, 2N) a red-orange complex is formed, which has a maximum absorption at 500 nm. Furthermore, the close values of the molar absorption coefficients and spectral band widths, obtained for different concentrations of Au(III) ions indicate that this colour reaction can be used in quantitative analysis. Although the spectrophotometric method developed from this colour reaction has a fairly wide linear dynamic range (0.4 – 2.0 mg·L⁻¹), a low detection limit (0.067 mg·L⁻¹) and good selectivity, it can only be used for Au(III) ions determination from laboratory solutions. The measurements performed using tap water as a background showed that there are significant differences between the concentration of Au(III) ions, added and calculated from the calibration curve, which significantly limits the use of the proposed method in the analysis of real water samples.

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DETERMINAREA SPECTROFOTOMETRICĂ CANTITATIVĂ A IONILOR DE Au(III) ÎN SOLUȚII APOASE UTILIZÂND COLORANTUL BASIC VIOLET 10

(Rezumat)

Determinarea spectrofotometrică a ionilor Au(III) cu colorant Basic Violet 10 este o metodă rapidă, sensibilă și precisă care poate fi utilizată în analiza cantitativă. Reacția de culoare are loc imediat în soluție apoasă acidă (pH = 0, soluție de HCl), iar acest lucru reprezintă un avantaj important din punct de vedere practic. Parametrii calitativi ai complexului colorat (lungimea de undă maximă (500 nm), lățimea benzii spectrale (120 nm) și coeficientul de absorbție molar ($2,55 \cdot 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$)) sugerează posibilitatea de utilizare a acestei reacții în analiza cantitativă. Legea lui Lambert-Beer a fost respectată în intervalul de concentrație Au(III) de 0,4 – 2,0 mg·L⁻¹, în timp ce limita de detecție (LOD) și limita de cuantificare (LOQ) sunt de 0,067 mg·L⁻¹ și 0,22 mg·L⁻¹. Această reacție de culoare este destul de selectivă pentru ionii Au(III) în prezența unor ioni interferenți (Cu(II), Zn(II), Na(I)), în timp ce alți ioni metalici (cum ar fi Ca(II), Mg(II), Co(II)) afectează într-o măsură mai mare precizia determinărilor experimentale. Măsurătorile efectuate folosind apă de la robinet, au arătat că există diferențe semnificative între concentrația ionilor Au(III), adăugată și calculată din curba de calibrare, ceea ce limitează aplicabilitatea acestei metode în analiza probelor reale.