

A SPECTROPHOTOMETRIC METHOD FOR VANADIUM DETERMINATION IN SEVERAL GEOLOGICAL MATRICES USING ALIZARIN RED S

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ABSTRACT

A simple versatile developed spectrophotometric method for the determination of vanadium (V) in several geological matrices was suggested and studied in the present work, using alizarin red S as a chromogenic dye. Several factors were studied for optimum determination, namely; effect of pH, buffer solution, dye concentration, sequence of addition, duration time and molar ratio. The complex was stable for two hours. Beer's law was obeyed in the concentration range 0.05-6 $\mu\text{g ml}^{-1}$ at λ_{max} 483 nm with molar absorptivity (ϵ) value $0.72 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, percentage relative standard deviation (%RSD) (4.5%) and % error (1.97%). Tolerance limits of foreign cations and anions were studied. A precipitation method for separation of the concomitant interfering ions was applied to overcome the interference effect of these elements with vanadium, to ease its determination with accurate and precise values. The proposed method was successfully applied to analysis of vanadium in different rock types.

Keywords: Vanadium, determination, Alizarin red S, Geological samples

I. INTRODUCTION

Vanadium is a naturally occurring trace element with several valences; 3, 4, or 5 in nature and, found as oxides or combined with other elements, where it forms more than 40 minerals in the plateau ores. It has great industrial, biological, and pharmacological importance. Vanadium foil is used in cladding titanium to steel ⁽¹⁾, the moderated thermal neutron-capture cross section and the short half life of the isotope produced by neutron capture makes vanadium a suitable material for the inner structure of fusion reactor ⁽²⁾. Vanadium pentoxide is used in several industries; as catalyst in manufacturing sulfuric acid ⁽³⁾ in ceramics ⁽⁴⁾, VO_2 is used in production of glass coating⁽⁵⁾ and the vanadate can be used for protecting steel against rust ⁽⁶⁾. Vanadium is also an essential element for normal growing cell, essential component of some enzymes and vanadium complex can reduce growth of cancer cells but can be toxic when present in higher concentrations where its toxicity depends on its oxidation state ⁽⁷⁾.

In view of crucial role in different fields, it is necessary to develop several sensitive analytical procedures for the determination of trace amount of vanadium. The procedures

include high performance gravimetry, ⁽⁸⁾ inductively coupled plasma optical emission spectroscopy (ICP-OES) ⁽⁹⁾, atomic absorption spectrometry (FAAS),^(10,11) colorimetric ⁽¹²⁾, chromatography ⁽¹³⁾ and X-ray fluorescence spectroscopy (XRF) ⁽¹⁴⁾, however, the relatively high instrumental cost and the need for preconcentration, chromatographic separation, extraction or coprecipitation are common disadvantages.

Although these techniques are sensitive, expensive and time consuming. Many of these procedures are complicated and involve preliminary isolation and preconcentration procedures.

Several spectrophotometric methods were reported for the determination of vanadium in environmental, biological and rock samples using different reagents such as, **variamine blue** ⁽¹⁵⁾, **cryamine R** ⁽¹⁶⁾, 2,6-dithiol-4-propylphenole ⁽¹⁷⁾, 6-chloro-3-hydroxy-7-methyl-2-(2-thienyl)-4H-chromen-4-one⁽¹⁸⁾, 1-(2-quinolyazo)-2,4,5-trihydroxy-benzene,⁽¹⁹⁾ 5-bromo-2-(2-pyridylazo)-5-diethylaminophenol⁽²⁰⁾, phosphorrtungstate⁽²¹⁾, benzyl acetate ⁽²²⁾, pyrogallol ⁽²³⁾ and tannic acid ⁽²⁴⁾.

Alizarin red S was used for spectrophotometric determination of vanadium in

presence of hexadecyl trimethyl ammonium at pH 7.7, the fluorescence was monitored at 607 nm with excitation at 505 nm and measure vanadium in sea water after treatment with oxine.⁽²⁵⁾

Alizarin red S was also used for the determination of vanadium in alloys and water samples in presence of Tween 80 at pH 5.5 and the absorbance measure at 500 nm⁽⁷⁾.

Determination of vanadium in rocks necessitates a separation process or using masking agents before determination to overcome the interfering effect of other elements.

In the present work, a versatile, rapid, simple and selective spectrophotometric method for the determination of vanadium using alizarin red S as a chromogenic dye without prior separation using either solvent extraction, ion exchange, solid phase extraction or using masking agents was studied. The factors affecting optimum complex formation such as pH, concentration of dye, buffer, contact time, detection limit. The effect of interfering ions were also studied and then applied on different types of geological and certified rock samples and was treated to overcome the interfering effect by a simple precipitation method.

II. EXPERIMENTAL

All reagents in the different studied experiments in the present work were analytical grade and chemical pure reagents.

A double beam UV-Visible spectrophotometer model Shimadzu, UV-1601 spectrophotometer equipped with 1 cm cell path length was used for all absorbance measurements.

A pH meter model Jenway 3510, England, was applied satisfactorily and calibrated daily using two successive buffer solutions (pH 4 and 7 or 7 and 10).

Preparation of alizarin red S dye

10^{-3} M of alizarin red S dye was prepared by dissolving 0.0085 g in double distilled water and completed in 25 ml volumetric flask.

Preparation of sodium acetate

0.2 M sodium acetate was prepared by dissolving 2.72 gm sodium acetate trihydrate with double distilled water in 100 ml volumetric flask.

Preparation of buffer pH 4

To 9 ml of 0.2 M sodium acetate, 41 ml of 0.2 M acetic acid were added and then completed in 100 ml volumetric flask with double distilled water.

Preparation of different acid concentrations

Different acids were prepared (10^{-2} M) from HCl, H₂SO₄, HNO₃, and HClO₄ where different aliquots were withdrawn (with different concentrations) to find out their tolerance limits (as anions) on vanadium alizarin red S complex formation.

Preparation of different concomitant concentrations

Stock solutions of the expected concomitant elements (UO₂²⁺, Al³⁺, Zn²⁺, Cr²⁺, Fe²⁺, Ca²⁺, Cd²⁺, Ni²⁺, Mn²⁺, Co²⁺, Cu²⁺, Ba²⁺) were prepared from their salts. Proper dilutions were performed to find out their tolerance limits effect on the complex formation.

Preparation of vanadium (V) solution

A stock solution of 1000 ppm vanadium (V) was prepared by dissolving 2.34g of ammonium metavanadate dissolved in 100ml of slightly acidic nitric acid solution then completed to 1 liter with double distilled water. Proper dilutions were performed for the different factors studied.

Preparation of 1%(w/v) ammonium chloride

Dissolve 1 gm of ammonium chloride in double distilled water and complete the volume in 100 ml volumetric flask.

Recommended procedure

In 10 ml volumetric flask, a standard solution containing not more than 2 µg ml⁻¹ vanadium, 2 ml from 10^{-3} M of alizarin red S dye and 1 ml from buffer pH 4 were added and the mixture was diluted to the mark with double

distilled water where the absorbance of the complex in a 1 cm cuvettes against a proper blank (at 483 nm) was measured.

RESULTS AND DISCUSSION

The factors affecting the formation of complex between alizarin red S and vanadium were optimized. These factors includes pH of dye, formation of complex, concentration of dye, molar ratio, surfactant, duration time, calibration curve and the tolerance limits of various interfering ions.

Effect of pH on the absorption spectrum of alizarin red S (ARS) dye

The absorption spectra of 10^{-3} M alizarin red S dye was studied at different pH values from 1-6 using diluted ammonia and nitric acid. Figure (1) indicated that the maximum wave length was at 421 nm at pH 2.

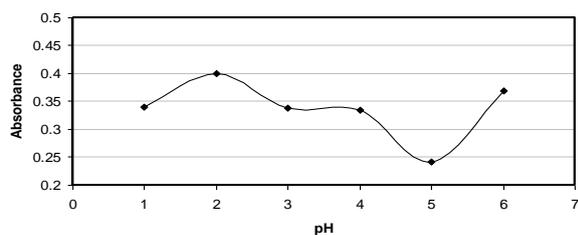


Figure (1): Effect of pH on the absorption spectrum of alizarin red S dye

Effect of pH on the complex formed between vanadium and ARS

The absorption value of V-ARS complex was studied by measuring the pH of complex during addition of acid in the range from 1-6, where 1 ml of vanadium solution (containing not more than $2 \mu\text{g ml}^{-1}$) and 1 ml dye (10^{-3} M) were mixed and completed with distilled water to 10 ml volumetric flask and absorbance was measured. Figure (2) indicated the formation of complex at pH 3-5.5 where maximum absorption occurred at 473.5 nm. Figures (1,2) showed a shift of wavelength from 421 nm to 483 nm which proved the formation of complex.

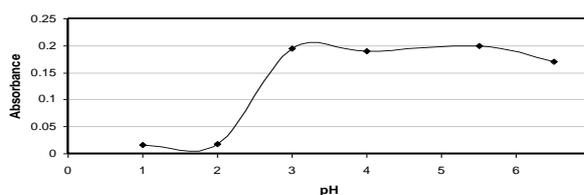


Figure (2): Effect of pH on the complex formed between vanadium and ARS

Effect of optimum of buffer solution

To keep the pH of complex constant a buffer solution was prepared and its suitable volume was determined by the addition of various volumes of buffer solution. The results shown in Table (1), indicated that 1ml from buffer pH 4 was suitable for maintaining the pH of the solution to the proper pH value at λ_{max} 483 nm.

Table (1) Effect of buffer solution on complex formation

Volume (ml)	pH 3.5		pH 4		pH 4.5		pH 5	
	λ (nm)	ABS						
0.2	484.5	0.212	483	0.217	470	0.224	455.5	0.143
0.5	483.5	0.258	483	0.255	483	0.193	476.5	0.174
1	483.5	0.270	483	0.277	483	0.19	477	0.189
1.5	484	0.252	483	0.245	483	0.184	480	0.189
2	485	0.25	483	0.245	483	0.18	480	0.175

Effect of ARS dye concentration

The optimum concentration of alizarin red S dye was studied by recording the maximum absorbance of complex using different volumes from 10^{-3} M dye with 1ml from $20 \mu\text{g ml}^{-1}$ vanadium and 1 ml from buffer pH 4. Figure (3) indicated that the optimum alizarin dye concentration was 2ml from 10^{-3} M stock dye solution.

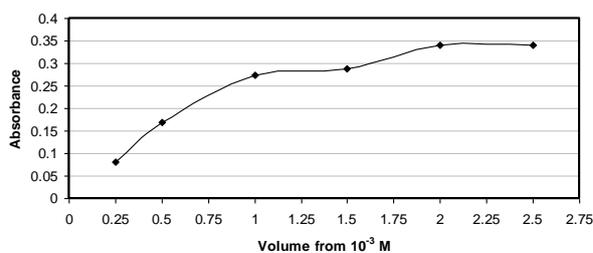


Figure (3): Effect of alizarin red S dye concentration

Effect of surfactant on the absorbance

The effect of adding surfactant was studied where it has the ability to raise the absorbance values of the complex (refer.) hence increasing the molar absorptivity value where in accordance increases the sensitivity of the method for determination. A series of tests using different values from 10^{-4} M cetyl trimethyl ammonium bromide (CTAB) and 10^{-4} of cetylpyridinium chloride (CPC) were used. From Table (2) it indicated that surfactants have negative effect on the absorbance values.

Table (2): Effect of surfactant on absorbance of complex

Volume (ml)	CTAB 10^{-4} M	CPC 10^{-4} M
	Absorbance	
-	0.277	0.277
0.5	0.251	0.234
1	0.231	0.208
1.5	0.201	0.176
2	0.198	0.125

Effect of sequence of addition

It was necessary to optimize the sequence of addition of the complex species that gives the optimum absorbance value; in this manner a series of four different sequences of additions were studied at the optimum previously studied factors for complex formation. It was found from Table (3) that the optimum sequence was the addition of vanadium, dye and buffer or dye, buffer and vanadium which recorded maximum absorption values.

Table (3): Effect of sequence of addition on the absorbance of the complex

Sequence of addition	Absorbance
Dye-vanadium-buffer	0.277
Vanadium-buffer-dye	0.331
Vanadium-dye-buffer	0.382
Dye-buffer-vanadium	0.381

Effect of time on the formation and stability of the complex

The effect of duration time on the formation and stability of the studied vanadium complex was studied by measuring the absorbance of vanadium-alizarin red S complex under the optimum conditions previously studied with time interval. It was found from Table (4) that the complex was formed after 5 minutes and was stable for 2 hours after which the complex began to dissociate.

Table (4): Effect of time on the formation and stability of the complex

Time (min)	Absorbance
2	0.306
5	0.377
10	0.377
20	0.377
30	0.376
45	0.378
60	0.378
80	0.378
100	0.378
120	0.379
140	0.332
160	0.321

Stoichiometric ratio of Vanadium : Alizarin red S complex

In the present work, the composition of complex under the optimized operating conditions was studied by using the continuous variation method. The method was applied to investigate the metal to dye ratio. Figure (4) showed that from the obtained results the optimum complex ratio suggested was 1:1.5 vanadium - alizarin red S.

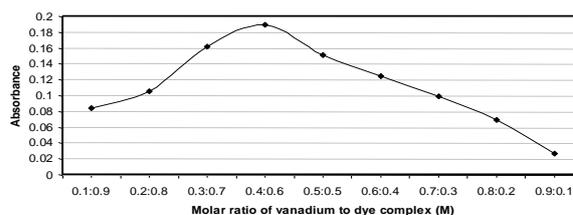


Figure (4): Stoichiometric ratio of vanadium : Alizarin red S complex

Calibration Curve

The adherence to Beer's law was studied by measuring the absorbance value of the series of solution containing different concentration of the metal ion. A linear working calibration line was drawn between absorbance and different metal ion concentrations, where the results obtained in Figure (5) indicated that V(V) was obeyed in range from 0.05-6 $\mu\text{g ml}^{-1}$ with molar absorptivity (ϵ) value $0.723 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$.

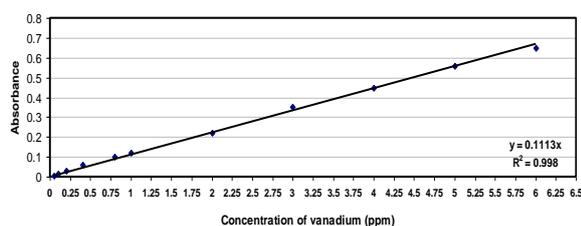


Figure (5): The working calibration curve of vanadium alizarin red S complex

The analytical parameters pertaining to the proposed method were given in Table (5). From data shown, it indicated that the method is precise and accurate, where seven replicate measurements were used to estimate the % RSD and % error.

Table (5): Summing up of all optimized parameters and values

Parameters	Value
Beer's law limit (ppm)	0.05-6
Molar absorptivity ($\text{L mol}^{-1} \text{ cm}^{-1}$)	0.723
Correlation coefficient	0.998
% RSD	4.50
% Error	1.97
pH	4.0
Metal : Ligand	1 : 1.5
Color	Orange
Maximum wavelength	483

Effect of anion concentrations

The effect of different anion concentrations (SO_4^{2-} , NO_3^- , Cl^- , HClO_4^-) on the absorbance of complex was studied under the studied optimized conditions, at 483 nm. The data gathered in Table (6), indicated that the determination of vanadium in presence of these

anions was possible up to $1 \times 10^{-2} \text{ M HClO}_4$, $1 \times 10^{-2} \text{ M HNO}_3$, $2 \times 10^{-2} \text{ M HCl}$ and $0.5 \times 10^{-2} \text{ M H}_2\text{SO}_4$.

Table (6): Effect of interfering anions on the measurements of vanadium

Volume of acids (ml)	HClO4 (10-2M)	HCl(10-2 M)	HNO3(10-2 M)	H2SO4(10-2M)
	ABS			
0.1	0.384	0.38	0.382	0.382
0.25	0.384	0.367	0.355	0.35
0.5	0.353	0.354	0.343	0.35
1	0.332	0.354	0.344	0.120
1.5	0.281	0.344	0.288	0.094
2	0.216	0.344	0.227	0.076

Effect of diverse ions

The effect of various amounts of concomitant ions on the selectivity of the spectrophotometric determination of vanadium-alizarin red S complex was studied using 3 $\mu\text{g ml}^{-1}$ vanadium at the optimum reached conditions; 2 ml of alizarin red S and 1 ml acetate buffer solution pH 4 in 10^{-2} M HCl . Data represented in Table (7) indicated the maximum concentration limits above which interference occurs.

Application of the optimized method on several geological samples

After optimizing several factors for the spectrophotometric method of vanadium determination using alizarin red S dye, it was applied on different rock types bearing vanadium namely; black shale, black chert, standard phosphate and standard basalt. Black shale and black chert, from Quseir area, Eastern Desert, Egypt were analyzed for major and trace elements by inductively coupled plasma-mass spectrometry (ICP-MS) in Achme laboratories of Vancouver, Canada. The results of chemical analysis of the selected rocks were represented in Table (8).

From Table (8) it was indicated that it was difficult to determine vanadium concentration

Table (7): Influence of interfering ions on the determination of vanadium (V)

Sample	UO ₂ ²⁺	Al ³⁺	Zn ²⁺	Cr ²⁺	Fe ²⁺	Ca ²⁺	Cd ²⁺	Ni ²⁺	Mn ²⁺	Co ²⁺	Cu ²⁺	Ba ²⁺
Conc.(ppm)	2	20	7	6	30	50	6	8	8	6	8	8

(ppm) in presence of high concentrations of major oxide values. When using masking agents as ethylene diamine tetra acetic acid (EDTA), cyclohexane diamine tetraacetic acid (CDTA) or NaF the absorbance values were drastically decreased and not all interference from several other ions was solved due to the high values of major oxide elements. To overcome this problem the major oxides must be separated by a selective precipitation method, as follows:

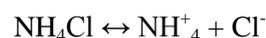
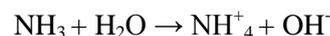
Table (8) Chemical analysis of selected applied samples

Oxides (%)	Black shale	Black chert	Standard Phosphate (2)	Standard Basalt (6)
SiO ₂	16.9	72.71	8.74	51.78
Al ₂ O ₃	4.37	0.19	1.62	15.58
Fe ₂ O ₃	1.61	0.71	1.34	11.81
MgO	0.68	0.07	5.51	4.56
CaO	14.72	13.6	37.99	6.71
Na ₂ O	0.12	0.15	0.63	3.87
K ₂ O	0.41	0.04	0.17	1.49
TiO ₂	0.21	0.01	0.07	1.92
P ₂ O ₅	3.38	5.83	20.5	0.34
MnO	0.02	< 0.01	0.057	0.17
Total C	21.81	1.63	14.74	--
Total S	3.20	0.74	0.40	0.01
Σ Loss on ignition	32.37	5.40	4.50	0.96
V (ppm)	2422	39	195.7	177
U (ppm)	41	35	20	7
Zn (ppm)	2314	74	252	109
Cu (ppm)	163	3.8	30.0	126
Ni (ppm)	272	14.4	24.2	42
Cd (ppm)	232.4	2.3	186.8	----
Mo (ppm)	569.5	5.7	31.5	126

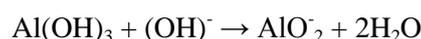
The sample was decomposed by using acidic mixture (0.5 gm of sample + 20 ml hydrofluoric acid + 10 sulfuric acid + 6ml perchloric acid) and heated till terminating all

acidic fumes and the residue was dissolved in 10 ml 5M hydrochloric acid and up to volume in 25 ml volumetric flask with doubled distilled water, to convert all oxidation state of vanadium to pentavalent state.⁽²⁶⁾

- To 10 ml aliquot sample, solid ammonium chloride was added first and then ammonia solution till the pH 9, where a precipitate of several ions was formed. Here a common buffer solution was formed according to the following equations:



- The precipitate was filtered and washed with 1% (w/v) ammonium chloride. Almost the entire matrix of aluminum, iron, calcium, and titanium were removed.⁽²⁷⁾
- NH₄Cl acts as a common equilibrium of NH₃ where it reduces the concentration of OH⁻ ion thus preventing dissolution of Al(OH)₃ because it dissolves in excess of (OH)⁻ forming soluble aluminate ion as follows:



- The precipitate of phosphate sample was dried and analyzed (semi quantitatively) using the Scanning Electron Microscope (SEM) as shown in Figure (6). The filtrate was quantitatively analyzed using wet chemical analysis, where the concentration of major oxides were lowered drastically to few parts per million (ppm), as iron oxide (70ppm), Al₂O₃ (30ppm), P₂O₅ (< 15 ppm).
- The filtrate was evaporated on hot plate till near dryness then dissolved in 10 ml 10⁻²M hydrochloric acid.
- An aliquot of 1ml was taken with 2 ml of ARS dye and 1ml acetate buffer then up to

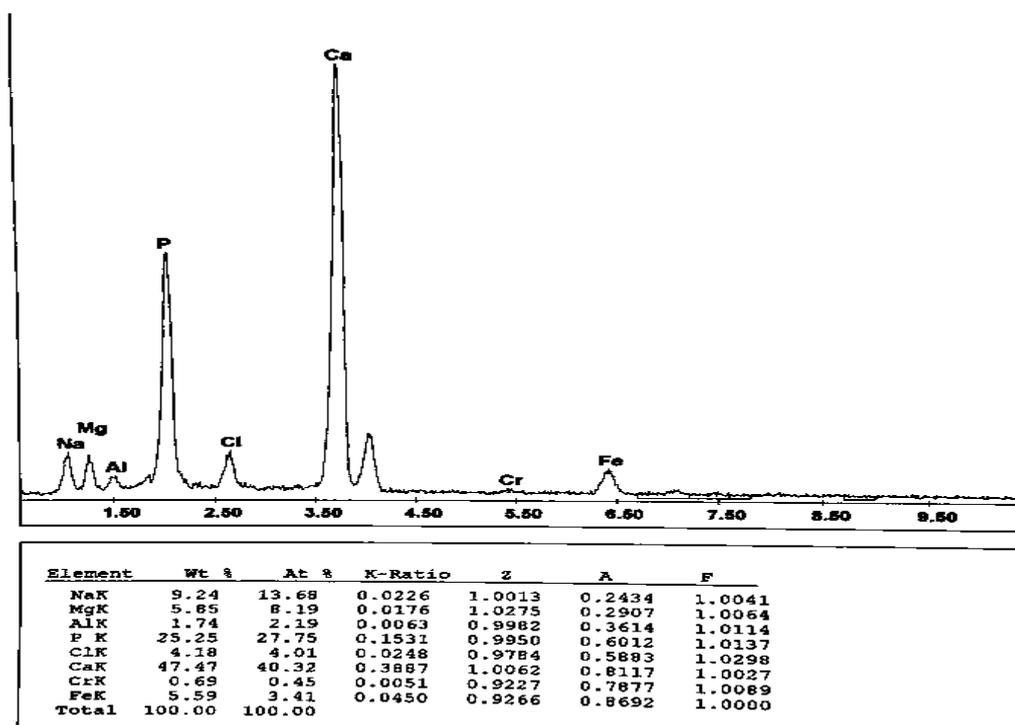


Figure (6): Scanning electron microscope analysis of the phosphate precipitate

volume in 10 ml volumetric flask and measure the absorbance at λ_{\max} 483 nm.

The method was applied successfully on all types of the selected studied samples and the results were gathered and shown in Table (9). It indicated that the developed alizarin red S method was very close to the true values which referred that the precipitation method has overcome the interference effect in different types of rock samples with different matrices.

Table (9) Determination of vanadium in different rock sample

Sample	Concentration of vanadium using present method	Concentration of vanadium using atomic absorption	% RSD	% Error
Black shale ⁽²⁸⁾	2490	2422	2.80	1.20
Black chert ⁽²⁸⁾	37	39	5.0	2.50
Phosphate ⁽²⁸⁾	128	133	4.10	2.40
Standard Phosphate (2) ⁽²⁹⁾	190	195	4.50	2.0
Basalt standard (6) ⁽³⁰⁾	171	177	4.0	1.60

CONCLUSIONS

The developed alizarin red S method used for vanadium determination in different bearing rocks with variable concentrations of vanadium

after separation of interfering ions by precipitation with ammonia and ammonium chloride was proposed in the present work. The developed method was reasonable, simple and selective for determination of vanadium in different rock samples with variable matrices.

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طريقة طيفية لونية لتقدير الفاناديوم في عدة عينات جيولوجية بأوساط مختلفة باستخدام صبغة الأليزارين رد اس

راندا ماجد الركايبى

في هذا العمل تم دراسة تطوير لعمل طريقة مبسطة و سهلة و متعددة الجوانب و الإستعمالات لتقدير الفاناديوم طيفيا في عدة عينات جيولوجية بأوساط مختلفة باستخدام صبغة الأليزارين رد اس اللونية. تم دراسة عدة عوامل للوصول إلى أفضل تكوين للمركب. و قد تبين أن أفضل تكوين للمركب كان عند قيمة أس هيدروجيني من 3 إلى 5,5، تركيز الصبغة 2 مللى من 0,003 عيارى، عند أفضل طول موجى 483 نانومتر. و بعد ضبط هذه العوامل الكافية لثبات المركب وجد أن المركب ثابت لمدة ساعتان دون تغيير و أن قانون بيبير كان متوافقا في النطاق من 0,05 إلى 6 ميكرو جرام لكل مللى و قيمة إمتصاص عيارى (E) $10^4 \times 0,72$ لتر لكل مول لكل سم و نسبة مئوية من الإنحراف المعياري (%RSD) مقدارها 4,5% و نسبة خطأ بقيمة 1,97%. تم دراسة السماحية لتركيز بعض الكاتيونات و الأنبيونات المصاحبة للفاناديوم دون تداخل في تقديره. و قد إستخدام طريقة الترسيب للعناصر المتداخلة المصاحبة للتغلب تأثير هذه العناصر في تقدير الفاناديوم بدقة و حساسية عالية. و قد نجحت هذه الطريقة المطورة لتقدير الفاناديوم في عدة أنواع من الصخور الحاملة للعنصر.