



Beryllium Determination in Some Geological Samples by Derivative Spectrophotometry Using Rhodamine B Dye

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Abstract

A rapid, simple and accurate procedure for direct derivative spectrophotometric determination of beryllium in some geological samples is described. Derivatization of spectra is a straightforward technique for enhancing selectivity. This process helps to overcome spectral interference and thus results in an increase in test selectivity. The process of derivatization is a widely recognized technique for separating usable signals from noisy data. Through the application of first, second, third, and fourth derivatives, it has been determined that the selectivity is enhanced by the employment of the first derivative. The method is based on the reaction of beryllium II with rhodamine B in the presence of sodium hydroxide to form a stable purple complex, which has an intense absorption at 524 nm in zero order and 504 nm in 1st order derivative spectrophotometry. The complex was examined by FTIR spectroscopy and ESEM-EDX. The average molar absorptivity (ϵ) and Sandell's sensitivity of the complex were found to be $0.22 \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$ and 4.09×10^{-2} respectively. Beer's law was obeyed over the range of 0.5 – 10 ppm. Removing interference from beryllium measurements caused by common cations is as simple as precipitating by ammonia at pH 9 and utilising the 1st order derivative. Successful determination of beryllium II in various geological materials has been achieved using this approach.

Keywords: Beryllium, Derivative spectrophotometry, determination

Introduction

Alkaline earth metals (like beryllium) are brittle, robust, lightweight, and have a steel-gray color, according to IPCS [1]. Due to its structural properties, beryllium is primarily used as an alloy hardening agent; it has a low coefficient of thermal expansion, a high melting point, and remarkable high-temperature stability [2].

Moreover, beryllium outperforms zirconium and titanium when it comes to high-temperature corrosion resistance. It has several uses, some of which involve nuclear power and others that do not. For aeronautical components, its low density makes it a promising candidate, particularly when other nuclear properties are sought after. Its combination of qualities makes it an ideal material for neutron reflector applications. The neutron moderator property of beryllium makes it a desirable material; this property allows it to lower the energy of neutrons, making it an ideal neutron reflector.

Gravimetric, volumetric, infrared, x-ray fluorescence (XRF), inductively coupled plasma (ICP), and spectrophotometric techniques for beryllium determination were among the many classical and modern approaches detailed. In order to regularly monitor the process (in quality control / quality assurance programs), the aforementioned methods have a number of drawbacks, such as expensive prices, specialized installation, and complicated maintenance.

Factors contributing to spectrophotometry's widespread use include its low cost, ease of use for analysts, sensitivity to a wide range of modern applications, and precision that can rival or even surpass that of competing methods [3, 4]

Prior research on beryllium spectrophotometric determination often involved isolating the element from any potentially interference-causing substances in order to improve the method's selectivity, accuracy, and sensitivity. Some methods for separation include ion exchange separation, solvent extraction, volatilization, precipitation and co-precipitation with collectors, and solvent extraction [5, 6, and 7].

Reagents that are appropriate for beryllium analyses limit the scope of the following survey; These include; beryllon II [8], carminic acid [9], Eriochrome Cyanine R [10], Titan yellow [11], xylenol orange [12], 1,8- dihydroxyanthron [13], and Chrome Azurol S [14]. To make matters worse, the majority of these approach are time-consuming and laborious, necessitating careful regulation of pH, temperature, and reagent while also preemptively separating Be from other potentially interfering elements, including Al (III), Fe (III), and Cu (II).

One of the most sophisticated and current spectrophotometric method is derivative spectrophotometry. It is predicated on "derivative spectra." [15] which are generated from parent zero-order ones. The most straightforward method for improving selectivity is derivatization of spectra, which removes interferences from the spectrum and makes the test more yield specific. Because spectrophotometer-integrated computers and their supporting software may produce derivative spectra of varying orders, digital data derivatization has become a popular technique for removing valuable signals from noisy data [16].

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Receive Date: 15 August 2024, Revise Date: 29 September 2024, Accept Date: 01 October 2024

DOI: 10.21608/ejchem.2024.312384.10204

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Reviewing recent uses of derivative spectrophotometry is presented by many authors. As stated by Abd El-Rehim et al. [16], the determination of the Samarium, Europium, and Gadolinium complex with chrome azurol S dye was done using the fourth derivative spectrum. The fourth-derivative spectroscopy was used to determine the amount of Pr (III) that was separated from the REEs concentrate using a cationic Dowex 50W-X8 ion exchange resin [17]. The use of fluorescein dye for simultaneous determination of lutetium, terbium and thallium without extraction in phosphate rock was done using 1st order derivative [18]. Using Cyanex 921 as an extractant and Chrome azurol S dye, the fourier transform spectra were employed to ascertain Sm (III), Pr (III), and Nd (III) from a concentration of REEs monazite [19]. Researchers looked at first-derivative spectra and EDTA to remove interference and give thorium and uranium selective estimation with 3-aminomethylalizarin-N-N diacetic acid dye [3]. On a final note, Mohammed [20] details how the first derivative spectrophotometric approach demonstrated excellent sensitivity and accuracy when it came to determining yttrium in rare earth element (REE) concentrates.

Because of its overall positive charge, the amphoteric dye rhodamine B (Rh B) is typically classified as basic. According to Lide and Milne [21], it is a Xanthene red dye that is very compatible with water.

No previous work has documented the use of Rhodamine B dye as a ligand for the spectrophotometric measurement of beryllium; this is the first study. Thus, the primary goal of this research is to create a reliable, fast, cheap, and easy way to measure the concentration of beryllium (Be) in beryl that forms a stable complex with Rhodamine B at a pH of 9.

Experimental

Apparatus

Shimadzu developed a double beam UV-visible spectrophotometer model (UV-11601) was used for measuring the absorbance of beryllium-dye complex. The wavelength capability of the spectrophotometer is ± 0.5 nm, and its resolution is 2 nm. Its operating range is from 190 to 1100 nm.

In all measurements, two glass cells with a path length of 10 mm were used for the sample and blank. The blank was adjusted to give zero readings during measurements, which were carried out at room temperature (about 25 C°).

A pH meter model, Jenway, was used for measuring the pH of different solutions. Daily calibration of the pH meter was performed using two successive buffer solutions 4, 7 and 10.

Reagents

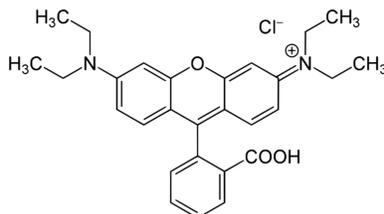
High-purity water with a resistivity > 18 M Ω was used in all analyses.

All used acids were prepared from highly pure concentrated acids (Burdick & Jackson, Germany).

The standards which were used in the calibration and interference study were prepared from standard solutions of 1000 ppm spikes on 4% nitric acid manufactured in Canada.

Rhodamine B dye (C₂₈H₃₁ClN₂O₃), purchased from BDH Co. (0.01%) stock solution was prepared by dissolving 0.01 g in ultra - pure water and diluted to 100 ml.

Sodium hydroxide (NaOH) (Riedel-de Haan,) solution of 0.1 M was prepared by dissolving 0.4 gm of NaOH with ultra-pure water and the volume was completed to 100 ml.



Scheme 1: The chemical structure of Rhodamine B dye.

Dissolution of Beryl & Preparation of leach solution

The determination of beryllium in geological sample requires initials dissolution followed by measurement using the chosen analytical method.

After being ground to a size of 0.074 mm and weighed at 0.05 g, the samples were subjected to alkali fusion with sodium hydroxide for digestion [22]. Boiling the mixture until it became transparent and adding 20 ml of 6 M hydrochloric acid (HCl) brought the total volume to 100 ml.

The digested sample was treated with solid ammonium chloride (NH₄Cl) and ammonium hydroxide (NH₄OH) to produce a gelatinous precipitate with a pH of 9. After filtering, the precipitate was washed with 1% ammonium chloride. Beryllium leach solution was obtained by dissolving the gelatinous precipitate in 3 ml of 0.1 M sodium hydroxide (in a total volume of 10 ml) and filtering the mixture.

To lower the ammonia solution's ionization due to the common ion (NH₄⁺) effect and to avoid the precipitation of Group IV cations as hydroxides with Group III, (NH₄Cl) is added to the precipitating agent (NH₄OH).

Unlike other hydroxides, amphoteric Be(OH)₂ and Al(OH)₃ dissolve in excess alkali. The elements can be separated from the mixed precipitated hydroxides [23, 24, and 25]. Be (II) was then ready for the determination by derivative spectrophotometry using rhodamine B dye.

Results and Discussion

Several factors have been studied to optimize the formation of Be-dye complex to obtain the most accurate determination for beryllium. These factors include the appropriate derivative order for spectrometry, the stability of the complex, the concentration of the dye and the concentration of the media.

1) Spectrum of Rhodamine B dye and complex

Fig. [1] shows the color spectrum of 10^{-3} M dissolved Rhodamine B dye in water. At λ_{\max} 570 nm, the maximum wavelength at which Rhodamine B dye (Rh.B) may be absorbed in an aqueous phase is observed. To create Be- Rh.B complex, mix 1 mL of 10^{-3} M Rh.B dye solution with 1 mL of 10^{-3} M Be (II) ions in the presence of sodium hydroxide (1 ml of 10^{-3} M). Dilute the mixture using a 10-ml measuring flask filled with distilled water, and then scan in the visible range of 400–650 nm, as shown in Fig. [1].

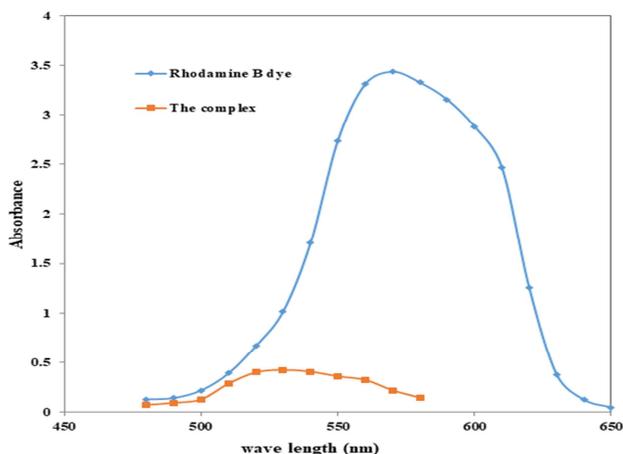


Figure 1: Spectrum of ligand [Rh.B] and its Be(II) complex.

2) The selection of the derivative order

Derivatisation of spectra is a simple method to increase the selectivity and overcome spectral interference. The absorption spectra of Be- Rh.B complex was measured in the range between 400 – 650 nm to give maximum absorption at 524 nm (Zero order, Fig.1). After testing all derivative orders, the first order derivative was found to be the most suitable for Be complex, yielding a result at 504 nm (Figure. 2).

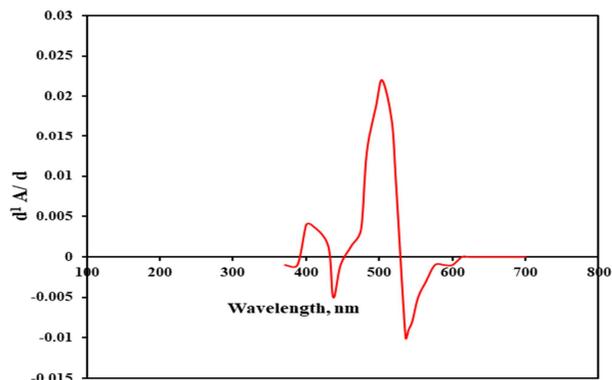


Figure 2: First order spectrum of [Be-Rh.B] complex.

3) Effect of sodium hydroxide concentration

It was necessary to study the effect of various concentration of sodium hydroxide (NaOH) on the absorption of Be complex because it is the final media of the sample. A modestly charged cation group of the dye can interact with an anionic complex formed when hydroxy anion, acting as a ligand, binds with be in the aqueous phase. Therefore, the volume of Rh.B ($1 \text{ ml of } 10^{-3} \text{ M}$) was maintained constant in order to find the optimal sodium hydroxide concentration. Be concentration was 10 ppm as well, with working sodium hydroxide solutions ranging from 0.5 to 7 ml and a concentration of 10^{-3} M . All of these solutions were diluted to a final volume of 10 ml. Next, we used spectrophotometry to determine the absorbance of each solution at 504 nm, taking the first derivative. Based on Table 1, it was determined that the beryllium ionic associate complex had the highest absorbance value when subjected to the conditions mentioned earlier, specifically a concentration of 1.5×10^{-4}

M sodium hydroxide (pH 12), which is necessary for the formation and spectrophotometric determination of the beryllium triple complex using Rh.B basic dye.

Table 1: The 1st order absorption of Be complex using different NaOH concentrations.

Vol. of 10 ⁻³ M NaOH (ml)	1 st . order	
	λ (nm.)	Absorption
0.5	504	0.008
1.5	504	0.015
2.5	504	0.011
5	502	0.006
7	502	0.002

4) Effect of dye concentration

Rhodamine B dye concentration should be optimized to obtain the most accurate determination. For this purpose, different volumes of 10⁻³ M Rhodamine B dye were added to a series of volumetric flasks of 10 ml volume containing 10 ppm Be and 1.5 mL of 10⁻³ M NaOH and the volume was completed to 10 ml, the absorbance of each solution was then measured against its blank using 1st.Order at λ_{max} = 504 nm. According to the results displayed in Fig. 3, the Be triple complex reaches its peak absorbance under the specified experimental circumstances when the Rh.B dye concentration is 0.5×10⁻⁴ M (0.5 ml from 10⁻³ M). Consequently, 0.5×10⁻⁴ M of the reagent is kept for all upcoming results.

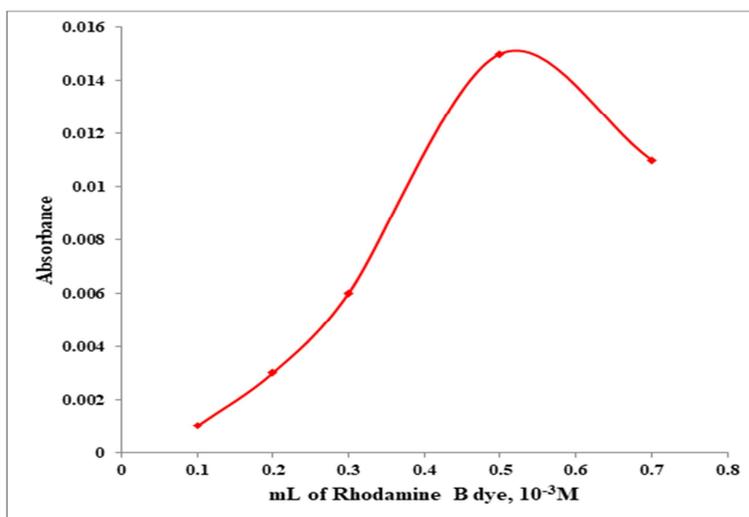


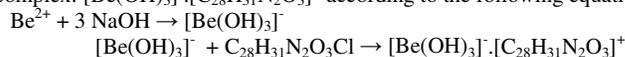
Figure 3: Impact of Rh.B dye on Rh.B-Be complex formation absorbance (using 1st Order at λ_{max} = 504).

5) Stoichiometry of the “metal - dye” complex

Following is the procedure for estimating the molar ratio of Be to the dye using the continuous variation approach, often known as Job's method [3, 4].

The total moles of the dye and metal remain constant during the operation, which is used to continuously alter their molar ratio. Working solutions for Be and Rh.B dye were made (1×10⁻³ M) and 1×10⁻³ M, respectively, for the purpose of determining the molar ratio. Each 1 mL solution contains 1.5 ×10⁻³ M of sodium hydroxide concentration, ensuring that, the total number of moles of metal and dye remains constant throughout the experiment. Varying volumes of Be and Dye solutions to ensure a constant end volume by adding the appropriate volumes. Next, 10 ml of each solution was taken and measured using 1st Order at λ_{max} = 504. Figure 4 shows that, the mix of 1 mole of beryllium and 1 mole of Rhodamine B dye produced the complex with the highest absorbance. A molar ratio of 1:1 between Be and Rh.B dye is so obviously present.

An acido complex, [Be(OH)₃]⁻, is formed by first complexing beryllium ions (Be²⁺) with three moles of working ligand (sodium hydroxide), according to the molar ratio found for beryllium, sodium hydroxide, and Rhodamine B dye. The latter triple-complex is formed by reacting with one mole of Rhodamine B dye, which contains a single-charged cation. Here is the likely formula for this complex: [Be(OH)₃]⁻. [C₂₈H₃₁N₂O₃]⁺ according to the following equations:



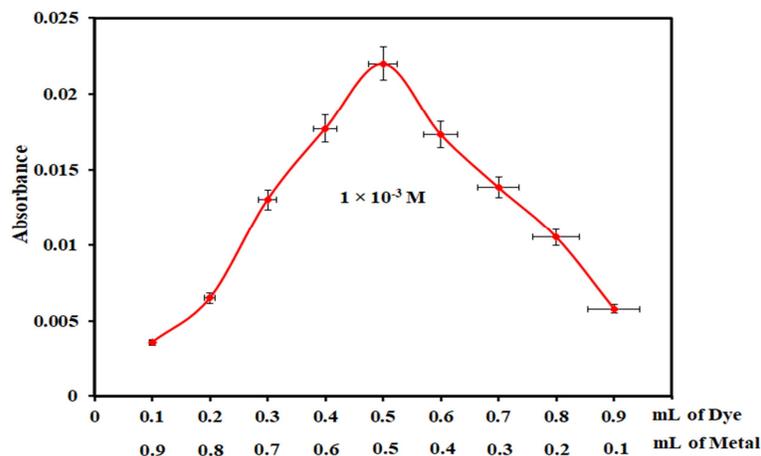


Figure 4: Continuous variation method for determination of the molar ratio between Be and Rh.B dye.

ESEM-EDX analysis:

To evaluate the chemical composition of the Rh.B dye after complexation with Be, ESEM-EDX analysis (Fig. 5) is taken. The results show that the presence of distinct peak of Be, indicating that Be has been complexed with the Rh.B dye.

FTIR analysis:

In order to better understand the reaction mechanism of Rh.B and Be^{2+} , FTIR was performed to assess their interaction (Fig. 6). The reactive functional groups of Rh.B dye before and after complexing with Be were assessed using FTIR, and the results are represented in Fig. 6. As shown in Fig. 6, the blank Rh.B showed well-reprehensive peaks at 2923 and 2853 cm^{-1} related to the asymmetric stretching vibration of C-H bonds of CH_3 and CH_2 present in Rh.B dye [26, 27, 28, and 29]. O-H stretching vibration at 3445 cm^{-1} [28, 30 and 31]. The band at 1346 cm^{-1} corresponds to the C-N stretching groups [27, 32]. The strong peak found at 1642 cm^{-1} is ascribed to the C=N stretching bond [29, 31]. The band at 1016 cm^{-1} is assigned to C-O stretching mode [28, 31]. The frequency at 1434 cm^{-1} is assigned to C=C stretching mode, while the peak at 950.7 cm^{-1} is attributed to C-C stretching vibrations [32]. After Be complexing with the Rh.B dye, the peaks of C-N stretching were disappear. The band of C=N was reduced and shifted to 1642 cm^{-1} .

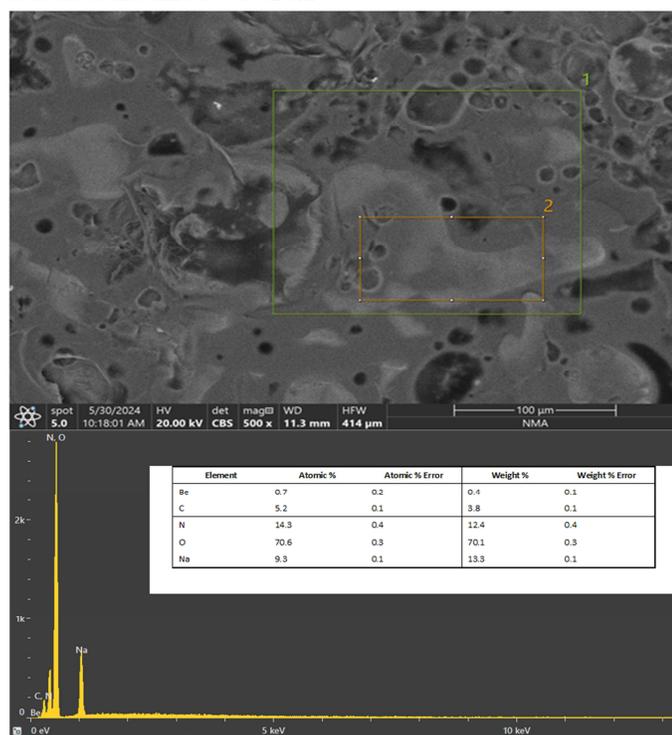


Figure 5: ESEM-EDX of the Be - Rh.B dye complex.

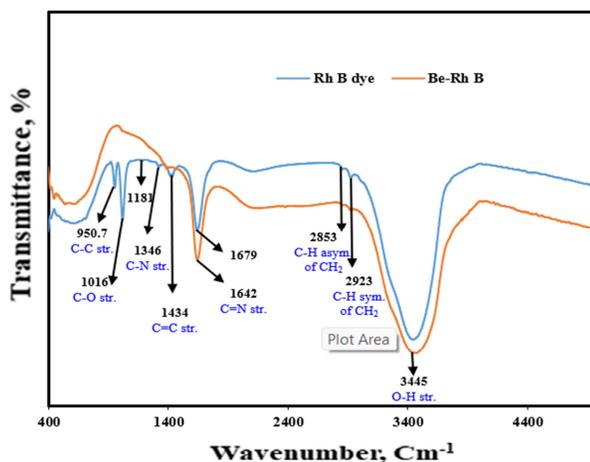


Figure 6: FTIR spectroscopy of Rh.B dye before and after complexing with Be.

6) Effect of duration time on the stability of the complex

To verify the stability of the complex, the absorbance of the complex was measured periodically each 10 minutes for the first half hour, then left to half hour and measure again and then measured after another half an hour (Total time is 90 minutes). Table (2) indicates that, the complex is stable for at least one hour.

Table 2: The effect of duration time on the complex's stability

Time (minutes)	1 st order absorbance
Zero	0.022
10	0.022
20	0.022
30	0.022
60	0.022
90	0.013

7) Construction of calibration curve

After detailed study of all factors affecting the spectrophotometric determination of beryllium by Rhodamine B dye using first derivative, it was necessary to study the effect of changing the concentration of beryllium on the absorbance and construct the calibration curve which show that, the concentration range of beryllium which obey Beer's law. To do this, a series of beryllium concentrations were created while fixing the other parameters (1.5 ml from 10^{-3} M NaOH, and 0.5 ml from 10^{-3} M Rhodamine B dye). At the specified λ_{\max} of 504 nm, each complex's absorbance was measured in relation to an appropriate blank solution. It is abundantly clear from the acquired data Fig.7 that the maximum concentration limit for Be(II) reached is $10 \mu\text{g mL}^{-1}$. The beryllium complex's average molar absorptivity (ϵ) was determined to be $0.22 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ based on this straight line. The computed values for the molar absorptivity (ϵ), specific absorptivity (a), Sandell index (S), the limit of detection, and Beer's law obedience range for the binary complex of Be(II) are summarized in Table 3.

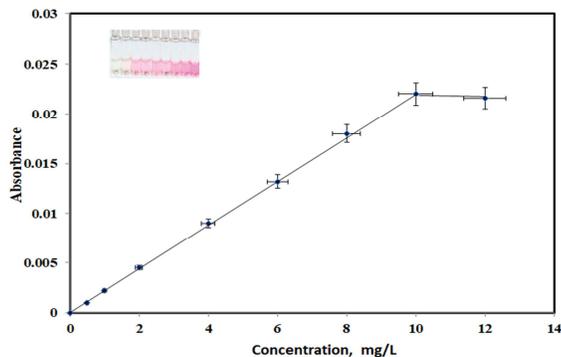


Figure 7: The calibration curve of Be – dye complex using 1st Order at $\lambda_{\max} = 504$.

Table 3: Summarizes the calculated data of Be – Rh.B complex.

Parameters	Value
Molar absorptivity (ϵ)	0.22×10^3
specific absorptivity (a)	2.44×10^{-2}
Sandell index (S)	4.01×10^{-2}
Range obeyed	$0.5 - 10 \mu\text{g mL}^{-1}$
Detection limit	$0.5 \mu\text{g mL}^{-1}$
Relative standard deviation	0.23 %

A comparison of the reagent possibility (Rhodamine B) under consideration for the spectrophotometric determination of Beryllium with the other methods (Table 4) shows that the proposed reagent (Rhodamine B) is one of the most active reagents for the determination of Beryllium.

Table 4: Comparison of spectrophotometric methods for determination of Be (II) with various chromogenic reagents

Reagent	λ_{max} (nm)	Molar absorptivity ($\text{L mol}^{-1}\text{cm}^{-1}$)	References
Carminic acid	580	2.25×10^3	[9]
1,8-dihydroxyanthrone	545	0.47×10^4	[13]
Chrome Azurol S	568	0.22×10^4	[33]
Chrome azurol S and cetylpyridinium chloride	620	1.02×10^4	[34]
Rhodamine B	504	0.22×10^3	This work

8) Effect of interfering elements

The interference effect on the determination of 10 ppm Be were studied under the optimized experimental conditions. The obtained results are summarized in Table 5.

Table 5: The interference effect

Element	Added conc.	Zero order		1 st . order derivative	
		Δ	Abs.	Δ	Abs.
Al^{3+}	50 ppm	524	0.616	504 - 508	0.020 - 0.016
Fe^{3+}	50 ppm	524	0.120	504	0.009
Ca^{2+}	50 ppm	526	0.111	505	0.007

The derivative spectrophotometric determination seemed to be moderately selective for (Al^{3+}) not for (Fe^{3+}) and (Ca^{2+}) which interfere seriously, perhaps these ions are masking for beryllium and avoid it to reacts with reagent or there is spectral interference between them. In case of the presence of Al^{3+} , beryllium possesses many properties similar to aluminium [35]. In order to try to overcome this interference on its precise and accurate quantitative determination of Be, $\text{NH}_4\text{Cl}/\text{NH}_4\text{OH}$ method (described in the experimental section) must be applied before the first order derivative determination.

9) Application of the method

The proposed method was applied to determine beryllium in different geological samples. One sample is Egyptian beryl minerals (analyzed by ICP technique), the 2nd sample is an international certified rock sample (MA-N) (granite rock sample) (Table 6).

Table 6: Determination of beryllium in different geological samples

Sample	Actual conc. (ppm) by ICP	Obtained conc.(ppm) by present developed method
Beryll 1	6400	6393
MA-N	300	297.3

Statistical evaluation of beryllium results

Due to the limited samples numbers, a comprehensive statistical analysis could not be conducted on the data. However, an evaluation using relevant statistical parameters was undertaken to provide some insight into the importance of the results (Table 7).

Table 7: Statistical calculations of beryllium determination in some geological samples

Sample	Be conc. (ppm)	Mean (X)	Standard deviation (SD)	Relative Standard deviation (RSD)	Standard error (SE)	Accuracy (Δ)	% Error
Beryll 1	6391	6393	1.400	0.022	0.816	2.00	0.013
	6395						
	6393						
MA-N	297.2	297.3	0.173	0.058	0.099	0.173	0.033
	297.2						
	297.5						

Conclusion

Various geological samples can now be determined using a spectrophotometric approach that is easy to use, quick, cheap and selective for beryllium. Utilizing first derivative, the optimized Be-Hydroxy-Rh.B triple complex was synthesized and evaluated. With a maximum absorption at 524 nm (zero order) and 504 nm in the 1st order derivative, the complex is stable. The molar absorbance at 504 nm is $0.22 \times 10^3 \text{ L.M}^{-1} \text{ cm}^{-1}$. There has been adherence to Beer's law in the range of 0.5 to 10 $\mu\text{g} / \text{mL}$ Be (II). Coloured complexes are stable for up to 60 minutes, according to absorbance measurements plotted against time. Successful determination of beryllium in various geological samples has been achieved using the proposed method.

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