



Speciation Studies of Cobalt (II) and Cobalt (III) and Its Application to Sample Analysis

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ABSTRACT

Speciation studies of cobalt species, cobalt (II) and cobalt (III) have been carried out using spectrophotometric determination with the chromophoric reagent bis (5-bromosalicylaldehyde) orthophenilinediamine (BBSOPD). The methodology is direct and non extractive and is carried out in 40% ethanolic medium containing acetate buffer. The colour development is almost instantaneous and remained constant for over 24 h. The maximum absorbance was obtained at 458 nm. The average molar absorption coefficient was found to be $5.3 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. The calibration studies showed a correlation coefficient value of 0.995. The linear dynamic range is 0.2-6 mg L⁻¹. The interference due to cations, anions and complexing agents in the determination was evaluated. The method was applied to the determination of cobalt(II) and cobalt (III) in a synthetic mixture, tap water and sea water samples.

Keywords: Cobalt, non extractive spectrophotometry, Bis (5-bromosalicylaldehyde) orthophenilinediamine, Speciation studies

1. INTRODUCTION

Cobalt has applications both in industries as well as in biological systems. It is present in vitamin B₁₂, which is required in the production of red blood cells and its deficiency causes pernicious anemia, loss of weight, or retarded growth whereas in larger amounts it is toxic and causes pulmonary disorders, and hyperglycemia [1]. A dietary intake of about 50 µg cobalt, of which 40 µg in the form of vitamin B₁₂, maintains cobalt equilibrium in the human. The normal level of cobalt in human urine and blood are about 1.0 and 0.18 µg L⁻¹, respectively [2]. Cobalt present in soils and in amounts less than 50 µg g⁻¹. Dissolved cobalt is present in the environment at concentrations of 0.5 to 12 µg L⁻¹ in sea water and upto 100 µg L⁻¹ in waste water [3].

In expanding analytical fields such as environmental, biological and material monitoring of trace metals, there is an increasing need to develop the simple, sensitive and selective analytical techniques that do not use expensive or complicate test equipment. Rapid and accurate analysis of trace metals in natural waters is essential to the study of earth surface geochemical transfer processes Many sensitive methodologies have been developed for the determination of cobalt [4-8]. High sensitivity procedures for the determination of cobalt generally use graphite furnace atomic absorption spectrometry (GFAAS) followed by a preconcentration step, which usually involves risk of sample contamination and analyte loss [9,10]. Voltammetric techniques though useful

for low levels of cobalt [11,12] suffer from interferences from various electroactive compounds present in real samples. Spectrometric techniques [13-18] have the advantages of being simple and not requiring expensive or complicated equipment. A wide variety of spectrophotometric methods for the determination of cobalt have been developed. Most of the complexing reagents are non-selective, sensitive at pH, involve long extraction/ color development period, [19] or the products become water insoluble and thus separation [20-22] of the species prior to its determination becomes essential. One of the common methodology adopted in the spectrophotometric method is to make use of a colour forming compound. One such compound which has been used by Ahmed et al [23] is Bis (5-bromo salicylaldehyde) ortho pheniline diamine (BBSOPD) as the chromophoric reagent for the determination of cobalt in different types of samples. Though the authors had reported the determination of cobalt in various samples, it was noted that the results were given as total cobalt only. Moreover, the procedure adopted by the authors for the synthesis and also the spectrophotometric determinations were tedious and involved use of strong reagents like sulphuric acid. Also the speciation studies were carried out in synthetic mixtures only and not in different water samples.

Moreover, the procedure adopted by them for speciation studies was to convert Co(III) (present in a synthetic mixture of Co(II) and Co(III)) to Co(II)

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by reduction and the total Cobalt was measured. Then in another aliquot, they masked Co(III) and determined Co(II). The difference of the two measurements gave Co (III).

Hence it was thought of using the same reagent with two motives; firstly to develop a simpler procedure than that was mentioned earlier [23]; secondly was to carry out the speciation studies without any oxidation or reduction procedure.

In the present study, the synthesis procedure has been simplified compared to the earlier procedure reported by Ahmed et al [23]. The spectrophotometric determination has been modified and sulphuric acid has been avoided completely. The main highlight of this present work is that this reagent has been applied for the determination of both cobalt (II) and cobalt (III) present together in the mixture. The determination did not involve any oxidation or reduction. It was based on the use of simple chelating agents. The methodology was applied to the determination of cobalt species (cobalt (II) and cobalt (III)) in various water samples.

2. EXPERIMENTAL

Standard stock solutions of Co(II) and Co(III) of $1000 \mu\text{g mL}^{-1}$ were prepared by dissolving the appropriate amounts of accurately weighed cobalt salts (obtained from Aldrich) and made up using distilled water. Sodium acetate, 8-hydroxy quinoline, tartaric acid, obtained from E.Merck, all were used as received to prepare the solutions of required concentration. 5-bromosalicylaldehyde, ortho phenylenediamine, (Aldrich) was used as such. Bis (5-bromo salicylaldehyde) ortho phenylene diamine (BBSOPD) was synthesized and used as the chromophore. 0.1% Bis (5-bromo salicylaldehyde) ortho phenylene diamine (BBSOPD) chromophoric reagent

The reagent Bis (5-bromo salicylaldehyde) ortho phenylene diamine (BBSOPD) was synthesized as follows. A solution of 2.44 g of salicylaldehyde in 25 mL of ethanol was added dropwise to a solution containing 1.08 g of o-phenylenediamine in 25 mL. The reaction mixture was stirred for 2 h and then warmed to about 40°C . The solution was cooled to room temperature and the precipitate was filtered and washed with distilled water several times, and then with ethanol and finally dried well. The prepared BBSOPD was characterized by IR and elemental analyses.

An aliquot containing not more than $1\text{--}15 \mu\text{g mL}^{-1}$ of cobalt (II) or (III) was transferred into 10 ml calibrated flask, a known volume of BBSOPD reagent solution in ethanol was added and the

solution was maintained at a particular pH by the addition of sodium acetate and the mixture was diluted to volume with ethanol. After 2 minutes, the absorbance was measured at 458 nm with a 1 cm cell against reagent blank solution. The reagent blank was prepared in a similar manner without cobalt. All measurements were carried out at room temperature. The cobalt content in an unknown sample was determined using a concurrently prepared calibration graph. It is to be noted that for both cobalt (II) and cobalt (III), the procedure adopted is same. For the determination of either Co(II) or Co(III) in a mixture containing both cobalt (II) and cobalt (III), the above procedure was adopted with a slight modification. If for example, Cobalt (II) is to be determined in presence of cobalt (III), then in the above procedure, prior to the addition of the chromophoric reagent, an appropriate masking agent for In addition to the reagent and sodium acetate, a suitable masking agent was added to enable the selective determination of the species of interest. For the determination of both cobalt (II) and cobalt (III) in a mixture, one of the species was masked using appropriate masking agent and the same. The water samples were pretreated before analysis. 500 mL of the sample was taken in a beaker and evaporated to near dryness and then the spectrophotometric methodology given above was followed. Since there was no reference material available for this species in environmental water samples, the samples were spiked with varying amounts of cobalt species before pretreatment.

3. RESULTS AND DISCUSSION

The chromophore synthesized was air-stable, non-hygroscopic and orange-yellow colored solid which is insoluble in water and soluble both in ethanol and methanol. The elemental analysis is consistent with the calculated results from the empirical formula of the compound. IR spectral data of BBSOPD shows a strong absorption peak at 3414 cm^{-1} which can be ascribed to the phenolic hydroxyl group. The various other peaks due to different groups are given in Table 1.

The electronic spectra show the presence of bands at 318, 291, and 261 nm which are assigned to the benzene $\pi\text{--}\pi^*$ transitions.

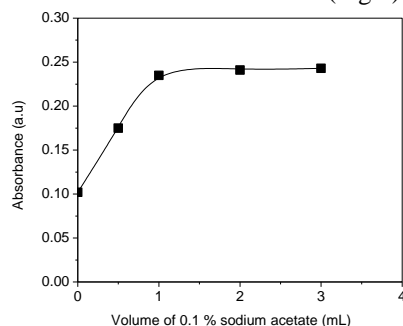
The spectrophotometric method in this work was carried out with an aim of studying the speciation of cobalt. The stoichiometry of the cobalt – BBSOPD complex was ascertained using Job's method (of continuous variation) and the molar ratio method. From these two studies, it was inferred that Cobalt forms a 1:1 complex with BBSOPD. The absorption maximum of the complex is at 458 nm and the average molar

Table 1: Characterization of BBSOPD.

Elemental Analysis		IR spectra		
Calcd	Obtd	Wavenumber (cm ⁻¹)	Due to	
C	75.93	75.78	1610(strong) 2923(very strong), 2854(strong)	C=C C-H
H	5.10	5.12	1581(medium) 1184(strong) 1028(strong) 1410(s), 1109(w), 746(s)	C=N C-N C-O Ring vibrns

absorption coefficient was found to be $5.3 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, whereas the reagent BBSOPD did not show any absorbance at this wavelength. Further spectrophotometric measurements were carried out at 458 nm. It is seen from studies using pure solutions that both Co(II) and Co(III) form complexes with BBSOPD with a maximum absorbance at 458 nm. Various factors that affect the absorbance and hence optimization of these factors becomes important to get maximum sensitivity and also improve the selectivity with respect to other interfering ions. The spectrophotometric studies in the present work were carried out in aqueous ethanolic medium and the use of solvents like dioxane [23] has been avoided.

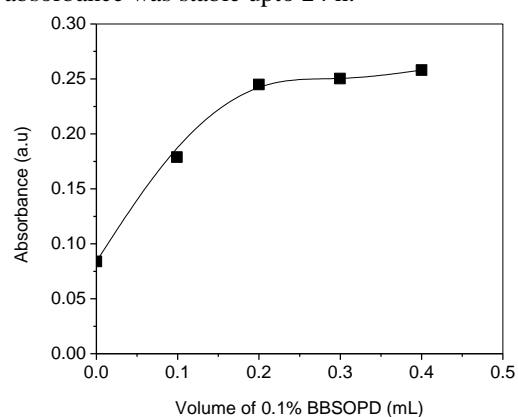
The acidity or the pH of the external solution was expected to have an effect on the absorbance values. Ahmed et al [23] showed that the absorbance was maximum using sulphuric acid of 5mM concentration. However, in the present study, the use of strong acids, was avoided. Thus the pH variations were carried out as follows. To an aliquot containing $1 \mu\text{g mL}^{-1}$ of Co(II), was added 0.2 mL of 0.1% BBSOPD and varying volumes of 1 % sodium acetate solution. The solution was made upto 10 mL and the absorbance of the resulting solution were measured and plotted as a function of the volume added (Fig.1).


Figure 1: Effect of acidity on absorbance.

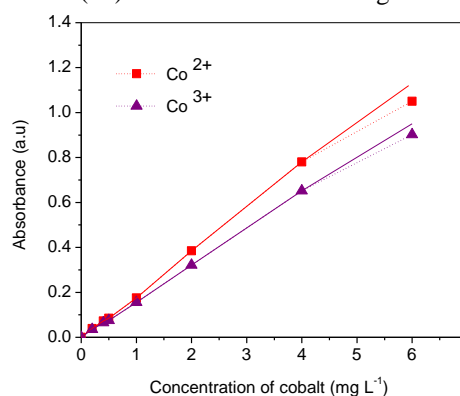
From these results plotted in Fig.1, it is seen that 1mL of 1% sodium acetate was the optimum amount and this was used for further experiments.

Hence it has become very easy to adjust the pH using only sodium acetate.

The effect of variation of reagent concentration was studied by adding different amounts of 0.1% BBSOPD to a 10 mL solution containing $1 \mu\text{g mL}^{-1}$ of Co(II) and 1mL of 1 % sodium acetate solution. The solution was made upto 10 mL and the absorbance of the resulting solution were measured and plotted as a function of the volume added (Fig.2). From these results, it is seen that 0.2 mL of BBSOPD was observed to give maximum absorbance and beyond this volume, the absorbance remained constant. Hence further experiments were carried out using 0.2 mL of BBSOPD. The reaction is instantaneous and the absorbance was stable upto 24 h.


Figure 2: Effect of reagent concentration

The calibration graph was plotted under optimum conditions for pure solutions of cobalt (II) and cobalt (III) ions and is shown in Fig.3.


Figure 3: Calibration for pure Co (II) and (III) ions

It was observed that for the same concentration, the absorbance values were slightly higher for Co (II) as compared to Co (III). The average molar absorption co-efficient was found to be $5.3 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. In the earlier paper, the determination of only cobalt (II) has been carried out with this reagent.

The effect of different ions on the determination of cobalt (II) was studied. The criterion for interference [24] was an absorbance value varying by more than 5% from the expected value for cobalt alone. The results are given in Table 2. It was seen that various ions like Cd^{2+} , Cl^- nitrate and sulphate did not cause interferences even present at high concentrations. However, the system showed tolerance to 100 fold levels of Ca^{2+} , As(VI) and Pb^{2+} while interference levels for Cu, Mg, Ni and Mo was 50 folds. However for ions like U, Fe, Cr, the tolerance was 20 fold. It was observed that Co(III) when present at equal concentration lead to severe interferences. This led to the development of a procedure for the determination of both Cobalt (II) and cobalt (III).

Table 2: Interference studies.

IN	Tolerance (in folds)	IN	Tolerance (in folds)
U	20	As	100
Fe	20	Pb	100
Cr	20	Cd	1000
Cu	50	Mn	1000
Mg	50	Na	>1000
Ni	50	Cl^-	>1000
Mo	50	NO_3^-	>1000
Ca	100	SO_4^{2-}	>1000

The main highlight of the present study was that this method could be used to determine both Cobalt (II) and cobalt (III) ions using suitable masking agents. Usually the procedure adopted in speciation studies is to first determine the total cobalt followed by the determination of any one of the species. The difference in the two concentrations gives the concentration of the other species. However, in this present study, this is not the case. By using the suitable masking agent, Cobalt (III) is masked in the determination of cobalt (II) and for the determination of cobalt (III), cobalt (II) is masked. The choice of masking agent was based on the stability of the complex formed by the cobalt species [25]. In the determination of cobalt (II), cobalt (III) was masked using tartaric acid [24]. Preliminary experiments using tartaric acid along with Co-BBSOPD showed that decolorisation occurred. Hence it was inferred that 0.2mL of BBSOPD was not sufficient. Optimization studies showed that 2mL of BBSOPD when added along with tartaric acid did not show any decolorisation and gave the same value of absorbance as obtained for pure solution of Co(II). For the determination of cobalt (II) in a mixture containing both Co(II) and Co(III) in the ratio of 1:10, added 1mL 1% sodium acetate solution, followed by varying amounts of 1% tartaric acid solution, 2ml of

0.1%BBSOPD solution and made up to the mark using ethanol and measured the absorbance of the solution at 458nm. The results of this variation showed that 2ml of 1% tartaric acid is sufficient to mask Co(III). For the determination of Co(III) in the mixture containing both Co(II) and Co(III) in the ratio of 10:1, added 1mL of 1% sodium acetate solution, varying amounts of 0.1% 8-hydroxy quinoline solution, 0.2ml of 0.1%BBSOPD solution and made up to the mark using ethanol and measured the absorbance of the solution at 458nm. From these studies it was found that 2 ml of 0.1% 8-hydroxy quinoline was sufficient to mask Co(II). It was seen that the addition of 8-hydroxy quinoline, did result in decolorisation of Co-BBSOPD. Figures 4a and 4b are the calibration curves of Co(II) and Co (III) respectively in presence of one another. It is seen that upon the addition of suitable masking agent, the absorbance values of the unmasked specie is quite near to that observed for pure solutions. This shows that the masking is effective and can reduce the interferences of the species of Co on one another.

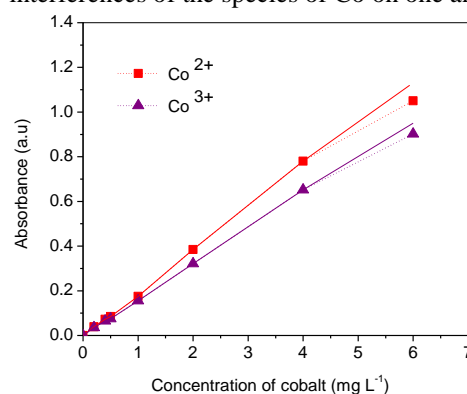


Figure 4: Calibration curves for (a) Co^{2+} in presence of Co^{3+} (masked with Tartaric Acid) (b) Co^{3+} in presence of Co^{2+} (masked with Oxine).

This method was extended to the determination of Co (II) and Co (III) in synthetic binary mixtures varying concentrations of Co (II) and Co (III). The results are shown in Table 3. From this table, it is seen that the both Co (II) and Co (III) do not interfere in the determination of one another even at ratios of 1: 10.

The precision of the method was evaluated by analyzing replicate samples (5). The relative standard deviation ($n = 5$) was 0–2% for 0.2–6 mgL^{-1} of cobalt indicating that this method is highly precise and reproducible. The average molar absorption co-efficient was found to be $5.3 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. Regression analyses of Beer's law plots at 458 nm revealed a good correlation ($R^2 = 0.998$). The method was tested by analyzing several synthetic mixtures containing cobalt and diverse ions (Tables 2 and 3). The reliability of this

procedure was tested by recovery studies. The percentage recoveries of both cobalt (II) and cobalt (III) in synthetic water samples are given in Table 2. It is seen that the recovery is more than 98 %.

The present method was successfully applied to the determination of cobalt(II) and cobalt (III) in actual water samples. The results are given in Table 4.

Table 3: Determination of Co in synthetic binary mixtures of varying concentration (std.dev for 5 determinations).

Amount Added ($\mu\text{g/mL}$)		Amount Found ($\mu\text{g/mL}$)		% Recovery	
Co^{2+}	Co^{3+}	Co^{2+}	Co^{3+}	Co^{2+}	Co^{3+}
1	1	0.99	0.98	99.0 \pm 0.3	98.0 \pm 0.4
1	5	0.98	4.98	98.0 \pm 0.2	99.6 \pm 0.5
1	10	0.99	9.98	99.0 \pm 0.4	99.8 \pm 0.3
5	1	4.99	0.99	99.8 \pm 0.3	99.0 \pm 0.6
10	1	9.98	0.99	99.8 \pm 0.4	99 \pm 0.5

Table 4: Determination of Co (II) and Co (III) in water samples (std.dev for six determinations).

Water sample	Amount added ($\mu\text{g/L}$)		Amount found ($\mu\text{g/L}$)	
	Co (II)	Co (III)	Co (II)	Co(III)
Sea Water	0	0	2.5 \pm 0.5	<1
	50	50	52.4 \pm 0.3	50.3 \pm 0.4
	100	100	102.3 \pm 0.3	100.5 \pm 0.4
Tap Water	0	0	<1	<1
	50	50	50.4 \pm 0.3	50.1 \pm 0.3
	100	100	100.5 \pm 0.3	100 \pm 0.3

The results for the speciation of cobalt(II) and cobalt(III) were highly reproducible. The recovery of Co (II) and Co (III) were 99.8 % and 99.5 % respectively. In the present work, the procedure was applied to only water mixtures unlike the earlier work [23] wherein, they had determined cobalt in different samples like alloy, biological fluids, soil and pharmaceutical tablets etc This is because in all these samples, pretreatment with an oxidizing agent was required for dissolution and hence this pretreatment would convert the entire cobalt into only the +3 oxidation state. However in our present development, we wanted to demonstrate speciation studies are possible under mild experimental conditions without the requirement of converting one valence state into another.

4. CONCLUSIONS

The present study is a non-extractive spectrophotometric method for the determination of both cobalt(II) and cobalt (III) in aqueous solutions. The main highlight of this work is that the

determination of both cobalt (II) and cobalt (III) can be achieved in presence of one another by using simple complexing agents. This proposed method is superior to the already existing methodologies in the sense that in the existing methodologies, either cobalt (II) or cobalt (III) is first determined and then the total cobalt is determined. The difference in the two values gives the concentration of the other species. However, in the present study, masking agents are used to mask one of the species in the determination of the other. The present methodology using BBSOPD is under milder conditions. Moreover, both the species of cobalt (Co(II) and Co(III)) form a strong complex with BBSOPD and the maximum absorbance is obtained at 458 nm. This method is sensitive, selective and reproducible with a detection limit of 0.4 mg L⁻¹ for cobalt. This proposed method has been conveniently used for the determination of trace levels of cobalt in various water samples.

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Bibliographical Sketch



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