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## SPECTROPHOTOMETRIC DETERMINATION OF Co(II) AS A COMPLEX WITH 1, 2 - PROPANEDIONE, 1- PHENYL - 1 - ( 2 - HYDROXYL - 5 - BROMO - BENZILIDINEAZINE)-2-OXIME [PDPHBBAO]

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**ABSTRACT:** The reagent was synthesized and characterization was carried out by FTIR, NMR, elemental analysis as well as Mass spectrometry. This reagent was then applied for the development of the analytical method for the extractive spectrophotometric determination of Cobalt (II). Cobalt metal forms yellow coloured complex, which can be extracted in chloroform at pH 9.0 having absorption maxima at 430 nm. Beer's law is obeyed in the concentration range 1-10  $\mu\text{g}$ . The molar absorptivity and Sandell's Sensitivity of the extracted species are found to be  $5.8470 \times 10^3 \text{ Lit mol}^{-1} \text{ cm}^{-1}$  and  $10.088 \times 10^{-3} \mu\text{g} / \text{cm}^2$  respectively. The developed method is highly sensitive, selective, simple, rapid, accurate, and has been satisfactorily applied for the determination of cobalt in the synthetic mixtures, pharmaceutical samples, and alloys.

**INTRODUCTION:** Cobalt has many applications in chemicals, pharmaceuticals and medical field. Cobalt is used in the preparation of alloys such as Superalloys, for parts in gas turbine aircraft engines, Corrosion- and wear-resistant alloys, High speed steels, Cemented carbides and diamond tools. It is used as drying agent in paints, varnishes, and inks. Catalysts for the petroleum and chemical industries. It is used in radiotherapy, biomedical implant and medical tests. It is used as a radiolabel for vitamin B-12 uptake. It is used in Sterilization of medical supplies, medical waste, radiation treatment of food, radiography and Dentistry. Hence owing to the significance of cobalt, its determination from associated elements by extractive spectrophotometry has been of considerable importance.

A wide variety of chelating agents<sup>1-29</sup> have been reported for the spectrophotometric determination of cobalt. However these methods suffer from limitations such as critical pH<sup>13, 15, 19, 23</sup>, low stability of complex<sup>14</sup>, requirement of surfactants<sup>20</sup> or other agents<sup>24</sup>,

requirement of heating<sup>21</sup>, and interference from some ions<sup>21, 27</sup>, inconvenient extractant<sup>30</sup> etc. A method, far superior in sensitivity and selectivity to these reported in the literature<sup>12, 16-18, 22, 25-29</sup> is developed for the extractive spectrophotometric determination of cobalt with with 1, 2 -propanedione, 1, 2 – propanedione – 1 – phenyl – 1 - (2 – hydroxyl – 5 – bromo - benzilidineaizine)-2-oxime [PDPHBBAO]

A close literature survey indicates that PDPHBBAO has so far not been employed for either co-ordination or analytical studies. The developed method is highly sensitive, selective, simple, rapid, accurate, and has been satisfactorily applied for the determination of cobalt in the synthetic mixtures, pharmaceutical samples, and alloys. The proposed method is free from many limitations.

**EXPERIMENTAL:** The PDPHBBAO was synthesized<sup>30, 31</sup>, characterized<sup>33</sup> and used for extractive spectrophotometric determination of Co(II). A stock solution of PDPHBBAO was prepared by dissolving it in

methanol to give 0.005% reagent solution of PDPHBBAO.

**Cobalt (II) Solution:** A weighed quantity of cobalt sulphate was dissolved in double distilled water containing dilute sulphuric acid and then diluted to the desired volume using double distilled water. The cobalt solution was then standardized by nitroso-R-salt method 34.

**Recommended procedure:** Mix 1cm<sup>3</sup> aqueous solution containing 1-100µg of cobalt and 1.5 cm<sup>3</sup> of 0.005 % methanolic solution of PDPHBBAO in 25 cm<sup>3</sup> beaker. Adjust the pH of the solution to required value with dilute solution of H<sub>2</sub>SO<sub>4</sub> and NaOH. Make the final aqueous volume up to 10 cm<sup>3</sup>. Transfer the solution into 125 cm<sup>3</sup> separating funnel and equilibrate for 1min with 10 cm<sup>3</sup> chloroform. Allow the two phases to separate and measure the absorbance of the organic extract containing the complex at 430 nm against reagent blank.

## RESULTS AND DISCUSSION:

TABLE 1:

Condition	Results
Absorption Maxima	430 nm
Solvent	Chloroform
pH range	8.5 to 9.5.
Equilibration time	1 min
Stability of Iron-PDPHBBAO	48 hs
Beer's range	1 to 10 µg / cm <sup>3</sup>
Molar absorptivity	5.8470 X 10 <sup>3</sup> Lit mol <sup>-1</sup> cm <sup>-1</sup>
Sandell's sensitivity	10.088 X 10 <sup>-4</sup> µg / cm <sup>2</sup>
Mole Ratio of Co : PDPHBBAO	1:1

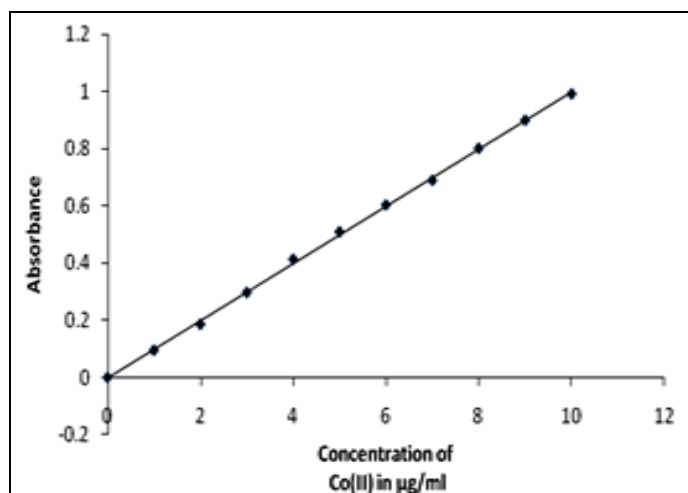


FIG. 1 CALIBRATION PLOT

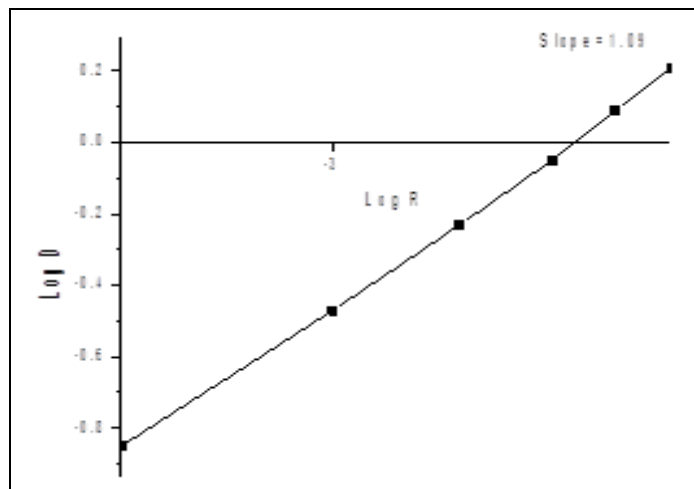


FIG. 2 SLOPE RATIO METHOD

## Effect of foreign ions

The effect of diverse ions on the cobalt(II) determination was studied, in presence of a definite amount of a foreign ion. Various cations and anions were investigated in order to find the tolerance limit of these foreign ions in the extraction of cobalt (II) (Table 2). The tolerance limit of the foreign ion was taken as the amount required to cause an error of not more than +2% in the recovery of cobalt (II).

TABLE 2: EFFECT OF FOREIGN IONS

Anion added	Amount added in mg.	Cation added	Amount added in mg.
Chloride	20	Cd(II)	2 mg
Fluoride	20	Mn(II)	5 mg
Bromide	20	Al(III)	10 mg
Iodide	20	Ca(II)	10 mg
Bromate	5	Zn(II)	10 mg
Iodate	5	Ce(IV)	5 mg
Chlorate	5	Mg(II)	10 mg
Sulphite	20	Li(II)	5 mg
Dichromate	10	As(III)	10µg
Carbonate	10	Pb(II)	5µg
Urea	20	Cr(II)	5 mg
Thiourea	20	Mo(VI)	10 mg
SCN <sup>-</sup>	20	V(VI)	2 mg
Acetate	20	Th(IV)	5 mg
Citrate	20	U(VI)	2 mg
Oxalate	20	Zr(II)	10µg
Nitrate	10	Tl(I)	5 mg
Nitrite	10	Ag(I)	5 mg
Sulphate	10	Rh(II)	1 mg

**Applications:** The present method was applied for determination of amount of Cobalt (II) in various samples as alloys, vegetable oil, synthetic mixtures and

pharmaceutical samples. The results obtained were well in agreement with those of standard methods (Table 3).

TABLE 3: APPLICATIONS

Sr. No.	Sample	Amount of Co (II)	
		Standard method	Present method
<b>I</b>	<b>Cobalt alloys</b>		
1	Cobalamine	70.2%	70.1%
2	Steel	9.86%	9.85%
<b>II</b>	<b>Vegetable Oil</b>	0.0019%	0.0018%
<b>III</b>	<b>Synthetic mixture</b>		
1	Co(10) + Zn (10)	9.99ppm	9.98ppm
2	Co (10) + Mo (10)	9.99ppm	9.98ppm
3	Co(10) +Mg(10)	9.99ppm	9.98ppm
<b>IV</b>	<b>Pharmaceutical Samples</b>		
1	Surbex –T (Abott)	0.33mg	0.32mg
2	Vitamin B12	50.0mg	49.9mg

**CONCLUSION:** The results obtained show that the newly developed method in which the reagent PDPHBAO was used, can be effectively used for quantitative extraction and estimation Co (II) from aqueous media. The proposed method is quick and requires less volume of organic solvent. The results show good agreement with the standard methods. The method is very precise, faster and simpler than other methods. The method is precise, accurate, less time consuming and easily employed anywhere, even in small laboratories as it requires only uv visible spectrophotometer and not much sophisticated and costly measurement devices or instrumentation.

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