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Differential pulse polorographic determination of cobalt in pharmaceutical preparations and biological samples

N.Y.Sreedhar*, D.Rekha

Department of Chemistry, S.V.University, Tirupati-517502, A.P, (INDIA)

E-mail : sreedhar_ny@reddif mail.com

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ABSTRACT

Highly sensitive, selective, rapid and economical Electroanalytical technique, Differential Pulse Polorography is developed for the determination of cobalt using newly synthesized complexing agent, 4-2-hydroxy phenyl ethaminodiol benzene-1,3-diol (4-2-HPEDB-1,3,D). At pH 5.5 a calibration graph was constructed in the concentration range of 0.1 to 200 μg/mL with correlation coefficient 0.9998. The detailed study of various foreign ions made the method more sensitive and selective. The present method was successfully applied for the determination of cobalt in pharmaceutical preparations and biological samples and the obtained results were compared with reference method which shows the significance of the proposed method.

KEYWORDS

Cobalt;
Differential pulse polorogrphic;
(4-2-HPEDB-1,3,D);
Pharmaceutical preparations;
Biological samples.

INTRODUCTION

Cobalt is an essential trace element in animal nutrition. It is main component in vitamin B_{12} synthesis. Cobalt is used mainly in the industry and poisoning is likely to occupational health hazards. The radio active Cobalt-60 is used as anticancer agent in medicine. Generally the dissolved cobalt may be present in environment is at the concentration range of 0.5-12 μg/lit in sea water and up to 100μg/lit in waste water. Deficiency of cobalt causes retorted growth, loss of appetite and anemia. So many types of pharmaceutical formations are used for the prevention and treatment of vitamin B_{12} deficiency[1-3]. In this view cobalt determination from associated elements is indispensable. Commonly used techniques which is available in the literature for the determination of trace amount of cobalt in various samples are spectrophotometry[4-8], atomic absorption spectrometry[9], inductively coupled plasma atomic emission spectrometry[10], neutron activation analysis[11]. These above reported techniques are very expensive, some techniques requires preliminary separation steps and tedious sampling procedures.

The proposed method is sensitive, selective and economical which is successfully applied for the determination of cobalt in pharmaceutical preparations and biological samples and the results are shows good agreement with reference method.

EXPERIMENTAL

Apparatus

An Elico CL-362 model polarographic system is used for DPP measurements and Elico Li-129 Model glass-calomel combined electrode was employed for measuring pH values. Ag/AgCl (salt KCl) was used as a reference electrode and a platinum wire as an auxiliary electrode.

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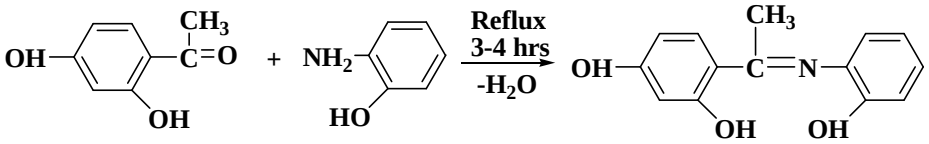
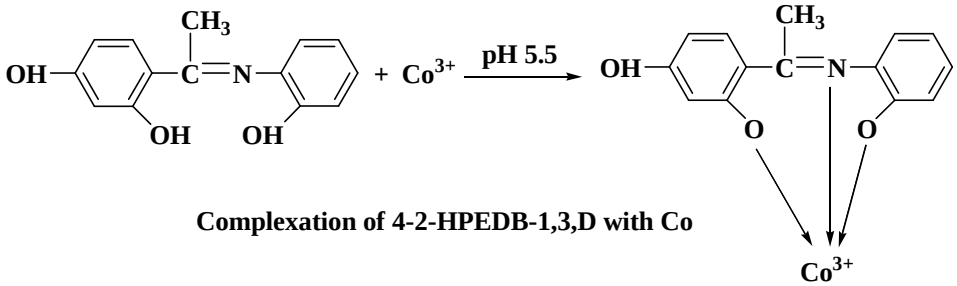


2,4-dihydroxy acetophenone | 2-amino phenol | 4-(2-hydroxy phenyl ethaminodiol), benzene-1.3-diol

Synthesis of 4-(2-hydroxy phenyl ethaminodiol) benzene-1.3-diol (4-2-HPEDB-1,3,D)



Complexation of 4-2-HPEDB-1,3,D with Co

SCHME 1: Synthesis and complexation of 4-2-HPEDB-1, 3,D with Co

Reagents

All reagents used were analytical reagent grade. Double distilled water was used throughout the experiment. A stock solution of cobalt (II) was prepared by dissolving appropriate amount of cobalt nitrate in double distilled water in volumetric flask. Working standard solution was freshly prepared by diluting the stock solution with double distilled water. 0.1 M concentration of 4-2-HPEDB-1,3,D was prepared by dissolving 2.48 g of 4-2-HPEDB-1,3,D in 100 mL of methanol

Synthesis of 4-2-hydroxy phenyl ethaminodiol benzene-1,3-diol (4-2-HPEDB-1,3,D)

Equimolar ratio of 2,4-dihydroxy acetophenone and 2-aminophenol in methanol mixture was refluxed for 3-4 hours and the contents were cooled at room temperature, it gives orange-red colour precipitate. The precipitate was filtered and washed with methanol to pure Schiff base. (M.P-115^{0}C, yield (97%) as shown in SCHEME 1 and I.R spectrum was shown in figure 1.The elementary analysis of the compound is given in TABLE 1.

Recommended analytical procedure for the determination of metal ions

An aliquot of working standard solution containing 1-100 µl of metal ion is taken in to 25 mL volumetric flask. To this 5 mL of acetate buffer solution (pH 5.5), 2ml of reagent solution were added. This mixture was transferred in to polorographic cell and diluted with 9 mL of supporting electrolyte and then deoxygenated with nitrogen gas for ten min. After recoding polorogram small increment (0.2mL) of standard solution is added. In the present study the best precision was obtained at pH 5.5 with a drop time 2 sec, pulse amplitude of 50 mv and an applied potential of -46.0 V. The relative standard deviation and correlation coefficients were found to be 1.24% and 0.9998 respectively for 5 replicants.

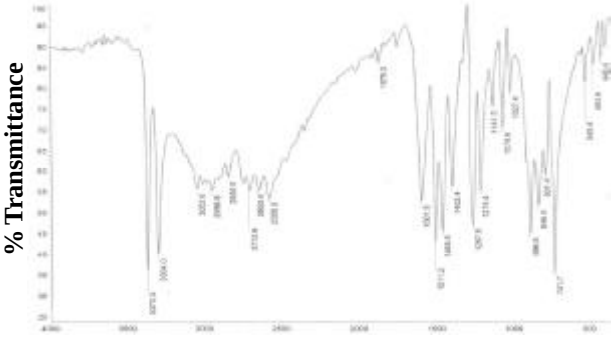


Figure 1 : I.R spectrum for 4-(2-hydroxy phenyl ethamino diol) benzene-1.3-diol (4-2- HPEDB-1,3,D)

TABLE 1 : Microanalysis results for the (4-2-HPEDB-1,3,D)

Results	Elementary analysis			
	O	C	N	H
Theoretical	48	168	14	13
Experimental	47.61	167.5	13.79	12.55

Collection of samples

The samples were collected from different loca-

TABLE 2 : Determination of cobalt in pharmaceutical preparations

Samples[a]	Labeled content	Proposed method (n=5)[c]	AAS method (n=5)[c]
Trivarol	0.125	0.122	0.123
Calcigenol	0.0434	0.0431	0.0433
Pisicoglut[b]	6.5	6.2	6.3
Becoforte	12.00	11.7	11.8
Citoneurim	14.5	14.3	14.4
Mineravit	1.000	0.997	0.998

[a]µ/mL of Cobalt, [b]mg/tablet of Cobalt, [c]Mean values for five determinations

TABLE 3: Determination of cobalt in biological samples

S.no.	Samples	Cobalt concentration			
		Proposed method		Reference method (12) (n=5)[e]	
		Found	RSD(%)	Found	RSD (%)
1	Blood [a]	19.3	0.31	18.5	1.3
2	Urine	8.5	0.82	7.2	1.0
3	Blood [b]	47.1	0.97	44.8	1.8
4	Urine	15.9	0.79	16.5	1.5
5	Blood [c]	561.3	0.66	558.5	1.0
6	Urine	143.1	0.30	140.7	1.6
7	Blood [d]	159.9	0.28	158.6	1.4
8	Urine	49.8	0.70	48.2	1.2

[a]Normal adult (Male),[b]Anemia patient (Male) samples collected from Government Hospital (Ruya hospital) Tirupati, A.P, India, [c]Pulmonary patient (Male), [d]Paralysis patient (Male) samples collected from, SVIMS (Srivenkateswara institute of medical sciences) Tirupati , A.P,India, [e]Mean values for five determinations.

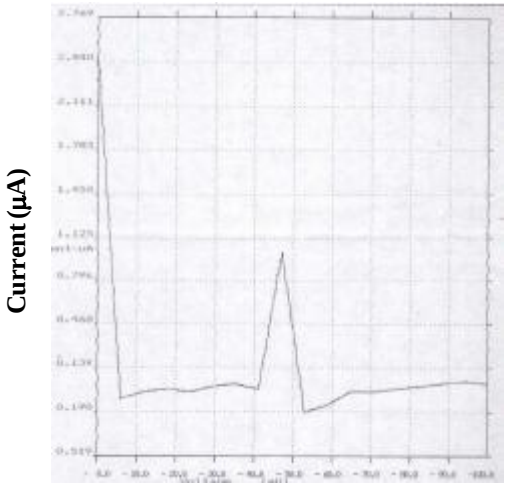


Figure 2 : Differential pulse polarogram of cobalt. Peak at -1.2V

tions of the study areas in and around Tirupati. The necessary and possible precautions were taken at various stages starting from sample containers, sample collection and storage, processing and analyzing the samples.

Analysis of pharmaceutical preparations

A tablet or an appropriate aliquate of each vitamin formulations (Trivarol, Calcigenol, Pisicoglut, Becoforte, Citoneurim, Mineravit) were grounded well and added 2-3 ml of nitric acid for dissolution and transfer in to a 100 ml calibrated flask, diluted to the mark with distilled water and mixed well and analysed for cobalt by the above said general procedure. The results were given in the TABLE 2.

Analysis of biological samples

The serum sample was centrifuged for 30 min by 1000g capacity centrifuger. The serum samples were kept at 253 K for storage. The serum samples were digested with a 5:1 (v/v) mixture of conc. HNO_3 and $HClO_4$ at temperature of 393 K and then diluted to proper volumes. Suitable volumes of these solutions are taken for the determination of cobalt as described in above said procedure and the results were shown in TABLE 3.

RESULT AND DISCUSSION

Differential pulse polorographic studies

Effect of pH

The effect of pH on the peak potential E_p and current intensity i_p, using differential pulse Polorography was examined for [Co-(4-2-HPEDB-1,3,D)]. The pH was varied in the whole pH range 2.5 to 10.5 for [Co-(4-2-HPEDB-1,3,D)] complex. It can be observed from figure 2, -46.0 V that the maximum peak current obtained with pH 5.5. When the pH has been increased from 2.5 to 10.5 the peak potentials have been shifted towards more negative values, indicating proton participation in the reduction process and the results were shown in figure 1.

Effect of pulse amplitude and scan rate

The influence of the pulse amplitude was investigated. The results suggested that DPP peak current reached the maximum value when the pulse amplitude was 50 mV. As for the scan rate; the current response with increasing the scan rate of 40 mVs^{-1} gave the maximum response. Accordingly, the optimum conditions for recording a maximum developed and sharper DPP

peak for 0.5 mM [Co-(4-2-HPEDB-1,3,D)] are scan rate : 40 mVs^{-1} and pulse amplitude : 50 mV.

Other experimental parameters such as temperature and ionic strength were optimized. The stripping peak currents were not modified when the temperature varied between 20-50^{0}C. The value chosen was 25^{0}C.

Study of foreign ions

The effect of interfere ion for the determination of Cobalt is studied. The cations like Be^{2+}, Mg^{2+}, Mo^{2+} and their salt do not interfere even when it is present up to hundred folds excess. Fe^{3+}, Al^{3+}, Cr^{3+} and Zn^{3+} shows positive interference up to fifty fold excess.

CONCLUSION

Differential pulse polorographic method for the assay of the cobalt determination using newly synthesized analytical reagent was developed and the results were compared with reference method with good agreement. The proposed method is successfully applied for the determination of cobalt in pharmaceutical preparations and biological samples. The method has additional advantages over reported methods i.e

1. The synthesis of organic reagent is distancing in terms of selectivity, sensitivity towards metal ions.
2. The reagent was synthesized at ordinary laboratory conditions and more economical.
3. Electrodes used for the proposed method is very sensitive and selective.
4. The risk of contamination is quite low and foreign ions do not interfere in the present method.
5. More accuracy, statistical analysis and avoidance of time taking, lengthy extraction steps makes the methods more sensitive one for the determination of cobalt in pharmaceutical and biological samples.

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