



SELECTIVE PRECONCENTRATION AND DETERMINATION OF COBALT(II) USING *N,N'*-BIS(2-HYDROXY-5-BROMO-BENZYL)-1,2-DIAMINOPROPANE

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ATOMIC SPECTROMETRY

SELECTIVE PRECONCENTRATION AND DETERMINATION OF COBALT(II) USING *N,N'*-BIS(2-HYDROXY-5-BROMO-BENZYL)-1,2-DIAMINOPROPANE

Derya Kara,^{1,*} Mahir Alkan,¹ and Şeref Güçer²

¹Department of Chemistry, Art and Science Faculty,
Balıkesir University, 10100 Balıkesir, Turkey

²Department of Chemistry, Art and Science Faculty,
Uludağ University, 16100 Bursa, Turkey

ABSTRACT

The present work describes a selective, rapid, and economical method for the determination of cobalt using *N,N'*-bis(2-hydroxy-5-bromo-benzyl)-1,2-diaminopropane (HBDAP). The investigation included a study of the characteristics that are essential for solvent extraction and preconcentration of cobalt for determination by FAAS (Flame atomic absorption spectrometry) and also the organic phase loaded with Co(II) from aqueous phase was stripped in one stage by using different mineral acid solutions. It was found that stripping efficiency was quantitative in case of HNO₃. Furthermore, a highly sensitive, selective, and rapid spectrophotometric method is described for the determination of trace amounts

*Corresponding author. Fax: +090 266 2456366; E-mail: dkara@balikesir.edu.tr



of Co(II) by HBDAP. This compound reacts with cobalt(II) in the range of pH 6–10 to produce a greenish yellow complex (2:3 molar ratio of Co(II)/HBDAP) soluble in chloroform. The complex obeys Beer's law from 0.7 to 7.25 $\mu\text{g mL}^{-1}$ with an optimum range. The relative standard deviations were 0.05% (13 samples, each containing 4.2 $\mu\text{g mL}^{-1}$ Co(II)). The precision was determined from 25 results obtained for 1×10^{-5} M Co(II); the mean value of a Co(II) was 1.03×10^{-5} M with a standard deviation of 2.35×10^{-7} M Co(II).

Key Words: Cobalt(II) determination; Preconcentration; FAAS; Spectrophotometry; Solvent extraction; *N,N'*-bis(2-hydroxy-5-bromo-benzyl)-1,2-diaminopropane

INTRODUCTION

Preconcentration of trace elements holds an important place among the techniques used in modern analytical chemistry. Preconcentration has increased the possibilities of many analytical determinations by eliminating the matrix effect which often significantly worsens the detection limits and other metrological parameters of the procedure and which can sometimes prevent the determination of one or trace elements. Another advantage of preconcentration is that it enables large representative samples to be treated and thereby reduces the sampling error. Solvent extraction is probably the most extensively used method of preconcentration. Extraction methods are suitable for absolute and relative preconcentration. They may be used for (selective or group) separation of trace elements in the extract or matrix separation in analysis of diverse industrial and natural materials. The important advantage of extraction methods is that they are universal with respect to the type of elements to be isolated and to their concentration. Usually, solvent extraction ensures high efficiency of preconcentration and can be combined with different methods of subsequent determinations.^[1] The large distribution ratios possible in solvent extraction systems allow the analytical determination of substances present in otherwise nondetectable concentrations. In other words, a large increase in sensitivity is obtained in the analytical method, even when the analyte is analytically detectable in the original sample: its preconcentration by means of solvent extraction permits use of smaller samples, simplification of the procedure, and increased accuracy of the analysis.^[2]

There is concern about the presence of cobalt in natural and environmental samples. Although it is an essential element, being required for the

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coenzyme vitamin B12, it is also believed to be toxic. There have been reports of soluble compound being weakly mutagenic^[3] and evidence for inhibition of DNA repair^[4] and cardiotoxicology.^[5] Airborne cobalt is known to cause bronchial sensitisation and lung damage.^[6] The determination of trace amounts of cobalt is therefore of importance.

Atomic absorption spectrometry (AAS) provides accurate and rapid determination of many heavy metals in natural waters. Nevertheless, very frequently for the extremely low concentration of cobalt in waters, a direct determination cannot be applied without their previous preconcentration and separation.^[7] In analytical practice very modern expensive separation instrumentation is used, but many labs around the world which cannot provide them, can apply some of the classical preconcentration methods such as solvent extraction, ion exchange, evaporation, flotation, and coprecipitation.^[8,9]

The spectrophotometry of micro amounts of cobalt has recently attracted much attention owing to environmental concerns. Various spectrophotometric methods for the determination of cobalt using dithizone,^[10] 4,4'-diazobenzenediazoaminobenzene,^[11] 2-(2-benzothiazolylazo)-2-*p*-cresol,^[12] nitrosochromotropic acid,^[13] dithizone,^[14] etc., have already been reported.

Synthesis of *N,N'*-bis(2-hydroxy-5-bromo-benzyl)-1,2-diaminopropane as a new extractant was introduced by Kara and Alkan for the spectrophotometric determination and speciation of ferric iron.^[15] Then the reagent was used for the preconcentration and determination of copper at ppb levels in different water samples.^[16] In the present work, extraction and preconcentration of cobalt(II) from wastewater samples and determination by atomic absorption spectrometry have been reported. Very high preconcentration factors could be achieved using this ligand by solvent extraction method for the determination of cobalt in water samples by FAAS. Furthermore solvent extraction spectrophotometric method including solvent extraction as a first step for determining Co(II) have been reported. The spectrophotometric method has been applied to the determination of cobalt in vitamin B12.

EXPERIMENTAL**Reagents**

The synthesis procedure of HBDAP was given in our previous study.^[15] The structure of HBDAP is shown in Fig. 1. All reagents and solvents were of analytical reagent grade. The aqueous solutions were prepared by deionized redistilled water. The stock solution of cobalt was prepared from CoCl₂·6 H₂O. The subsequent standard solutions of this analyte were prepared by diluting the stock solution. Adjustment of pH of the

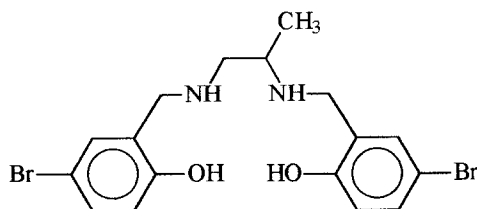


Figure 1. Structure of *N,N'*-bis(2-hydroxy-5-bromo-benzyl)-1,2-diaminopropane (HBDAP).

aqueous phase was carried out with buffers (acetic, phosphoric and boric acids and their potassium salts). Potassium chloride was added to give a constant ionic strength of 0.1 M.

Apparatus

AA 929 Unicam Spectrometer was used for FAAS measurements with an air-acetylene flame. Absorbance measurements were made using a Cary 1-E UV-Vis Spectrophotometer with 1.0 cm quartz cells. A pH meter (Metrohm 691 pH Meter) was also used. All extractions were performed by using a mechanical flask agitator in 50 mL glass flasks.

Procedures

Extraction Procedure

Aqueous solutions containing 1.0×10^{-5} – 1.0×10^{-4} M cobalt(II) chloride in appropriate buffer were equilibrated with equal volumes of the chloroform solution of the ligand (1.0×10^{-4} – 1.0×10^{-3} M) by shaking in a mechanical shaker at 25°C. In most cases distribution equilibrium was attained in less than 15 min and a shaking time of 30 min was therefore chosen as an optimum equilibration time to obtain reproducible results. The ionic strength of the aqueous solution was 0.1 M KCl in all experiments except those in which the effect of ionic strength was studied. After agitation, the solutions were allowed to stand for 10 min. The Co(II) concentration of the aqueous phase was determined by FAAS, and that of the organic phase from the difference by considering the mass balance. The pH of the aqueous phase was recorded as equilibrium pH. The absorption of the



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organic phase after extraction had been established was measured spectrophotometrically at 370 nm.

Preconcentration Procedure

Extraction of the aqueous phase (100 mL) containing 18.86 μg Co(II), a 30 mL portion of the organic phase (10^{-3} M HBDAP in chloroform) was stripped with 15 mL of aqueous acid solutions, including HCl, H_2SO_4 , and HNO_3 . The amount of cobalt in aqueous phase after stripping the organic phase was determined by FAAS, and then the recovery percentage (R%) was calculated.

Determination of Trace Amounts of Cobalt in Wastewater Samples Using Preconcentration Procedure by FAAS

Hundred milliliters sample of wastewater from different plants before and after the addition of 18.86 μg Co(II) to the sample was digested with 2 mL of concentrated HCl and 5 mL of concentrated HNO_3 . Digested water sample was extracted into 20 mL of 1 mM HBDAP in chloroform and then cobalt-loaded organic phase was stripped into 10 mL of 10% HNO_3 . Co(II) amounts in stripped solutions were determined using a standard addition method by FAAS.

Determination of Cobalt in Vitamin B12 Samples by Spectrophotometric Method

Vitamin B12 was digested with 2 mL of concentrated HNO_3 and 2 mL of 30% H_2O_2 and the digest was evaporated to dryness. The residuum was dissolved and diluted to 50 mL with water. The concentrations of cobalt were determined spectrophotometrically at 370 nm.

RESULTS AND DISCUSSION

The Effect of pH on the Extraction of Co(II)

Figure 2 shows the effect of pH on the extraction of Co(II) into chloroform with HBDAP. As shown in Fig. 2, the cobalt extraction is quantitative within the pH range 8.5–9.5.

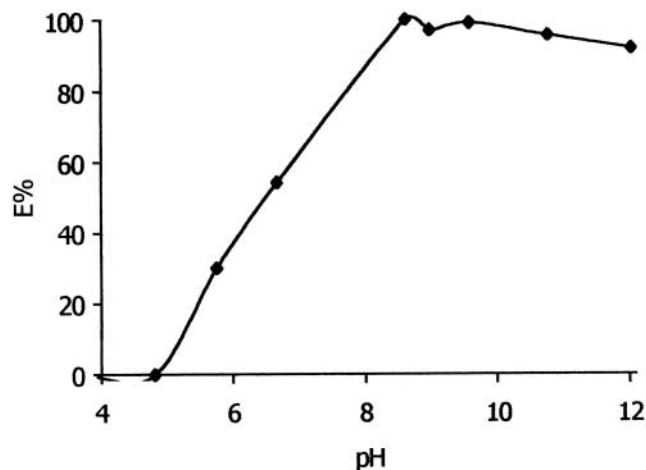


Figure 2. Effect of pH on the extraction of Co(II).

Co(II)–HBDAP Colour System for Spectrophotometric Determination of Cobalt

Absorption Spectra of the HBDAP and Co(II)–HBDAP Complex and the Effect of pH on the Absorbance of the Complex

The absorption spectra of the ligand (HBDAP) and cobalt–HBDAP complex are shown in Fig. 3. As seen, the spectra of cobalt–HBDAP complex have two maxima, one which overlaps with the maximum of the ligand at 285 nm. The other maximum appears at 370 nm, where the ligand has no absorbance. So, 370 nm has been used in all subsequent measurements. The absorbance of Co(II)–HBDAP complex increased greatly from pH 6.7 up to 8.5, remaining constant between 8.5 and 9.5 and then gradually decreased. So it was concluded that quantitative extraction of cobalt(II) with HBDAP was attained in the pH range 8.5–9.5 (Fig. 4).

Composition of the Extracted Species

In order to confirm the stoichiometry of the Co(II)–HBDAP complex, spectrophotometric measurements were performed by using Job's and slope-ratio methods.^[18] In Job's method the concentration of cobalt in the aqueous phase and HBDAP in the organic phase are varied so that their sum is equal



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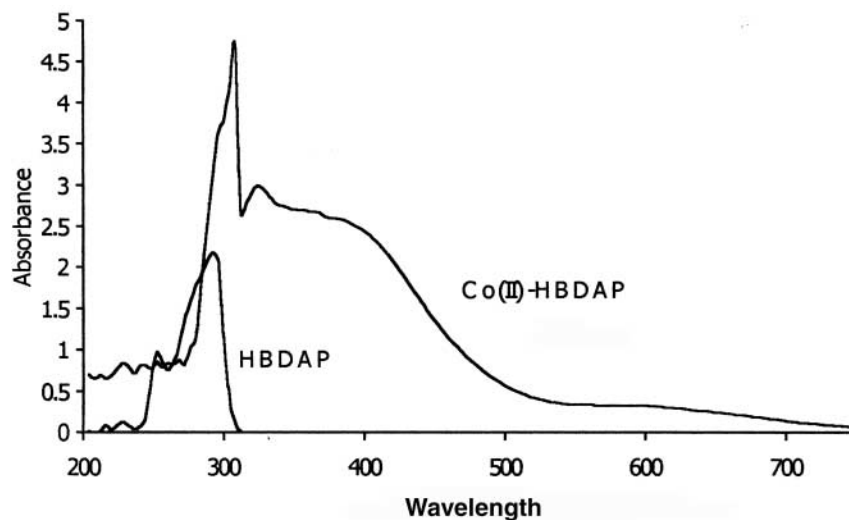


Figure 3. The absorption spectra of the ligand (HBDAP) and cobalt-HBDAP complex.

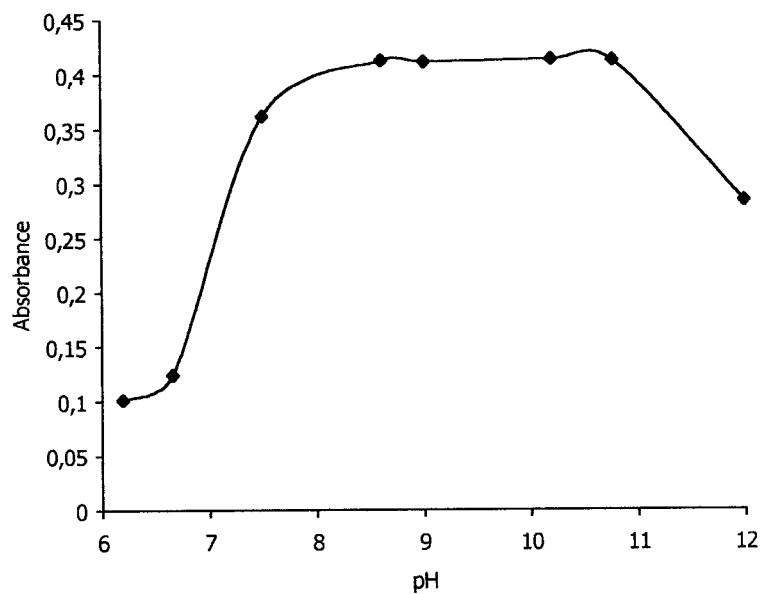


Figure 4. Effect of pH on absorbance of Co(II)-HBDAP complex.

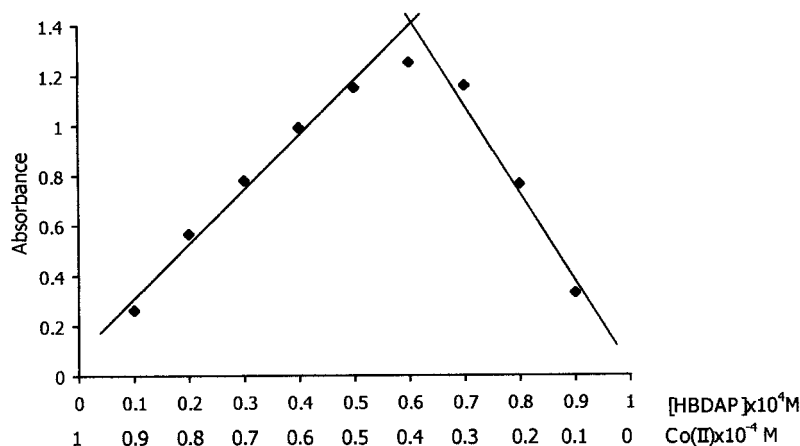
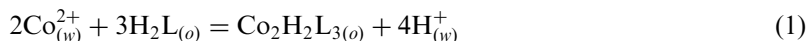


Figure 5. Cobalt complex stoichiometry determination in the extraction with HBDAP by spectrophotometric measurements at 370 nm (Job's method).

to 0.1 mM. The pH was kept at 8.5 by a borate buffer and the ionic strength was kept constant at 0.1 M by adding a solution of KCl. The absorbance of the cobalt complex in the organic phase which develops a greenish yellow color was measured at $\lambda = 370$ nm (Fig. 5). The maximum absorbance value corresponds to a molar ratio $[\text{Co}^{2+}]_w/[\text{H}_2\text{L}]_o$ of 2/3 which confirms the assumption of a $\text{Co}_2\text{H}_2\text{L}_3$ stoichiometry. The slope ratio method leads to similar results. It consists of two series of experiments: in the first series $[\text{H}_2\text{L}]_o$ is kept constant at 1 mM while $[\text{Co}^{2+}]_w$ is varied from 4×10^{-5} to 24×10^{-5} M keeping the pH constant and the ionic strength as in Job's method; in the second series $[\text{Co}^{2+}]_w$ is kept at 1 mM while $[\text{H}_2\text{L}]_o$ is varied from 4×10^{-5} to 24×10^{-5} M. Two absorbance straight lines were obtained as shown in Fig. 6. The slope ratio of both lines is equal to 1.5 which suggests again that the complex stoichiometry can be taken as $\text{Co}_2\text{H}_2\text{L}_3$. According to the discussions above, the extraction process may be represented by the equation,



where H_2L represents the extractant reagent and subscripts (w) and (o) denote the aqueous and organic phases, respectively. The extraction constant of the species $\text{Co}_2\text{H}_2\text{L}_3$ is then given by

$$K_{\text{ext}} = \frac{[\text{Co}_2\text{H}_2\text{L}_3]_o [\text{H}^+]_w^4}{[\text{Co}^{2+}]_w^2 [\text{H}_2\text{L}]_o^3} \quad (2)$$



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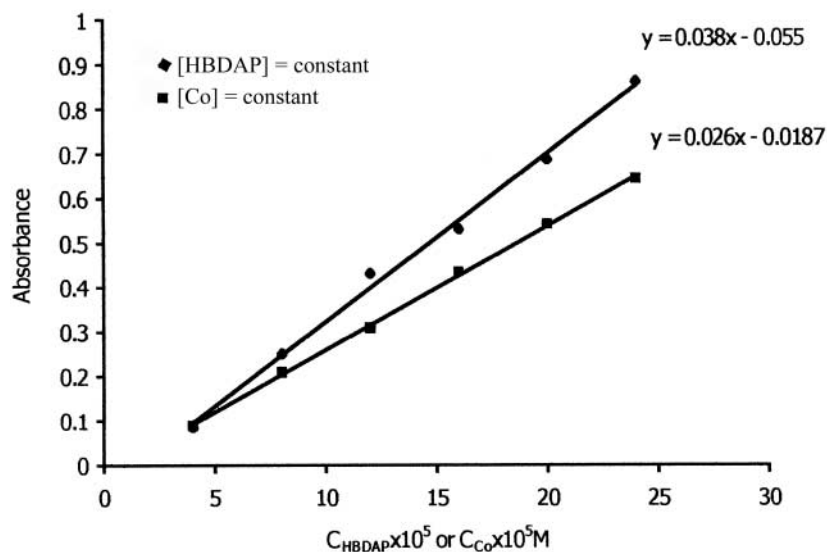


Figure 6. Cobalt complex stoichiometry determination in the extraction with HBDAP by spectrophotometric measurements at 370 nm (Slope ratio method).

The metal distribution ratio (D) and the extraction constant are related by

$$\log D = \log K_{\text{ext}} + 4\text{pH} + \log[\text{Co}^{2+}] + \log 2 + 3 \log[\text{H}_2\text{L}]_o \quad (3)$$

According to Eq. (3), a plot of $\log D - \log[\text{Co}^{2+}]$ against $\log[\text{H}_2\text{L}]_o$ at constant pH, will give a straight line of slope = 3 and intercept = $\log(K_{\text{ext}}) + 4\text{pH} + \log(2)$; hence, from the graph (Fig. 7) the extraction constant ($-\log(K_{\text{ext}})$) has been calculated as 12.6. Additionally, $-\log(K_{\text{ext}})$ values were calculated by using 9 other different extraction data obtained at different pHs between 6 and 7. The results obtained confirmed the value of $-\log K_{\text{ext}}$ given above.

Determination of Co(II)

The applicability of the HBDAP as a spectrophotometrically determination method for Co(II) was studied in the range of 5×10^{-6} – 24×10^{-5} M buffered Co(II) solutions. The concentration of HBDAP in chloroform was 1 mM. The effective molar absorption was calculated from the data obtained by the measurements of organic phase absorbance at the con-



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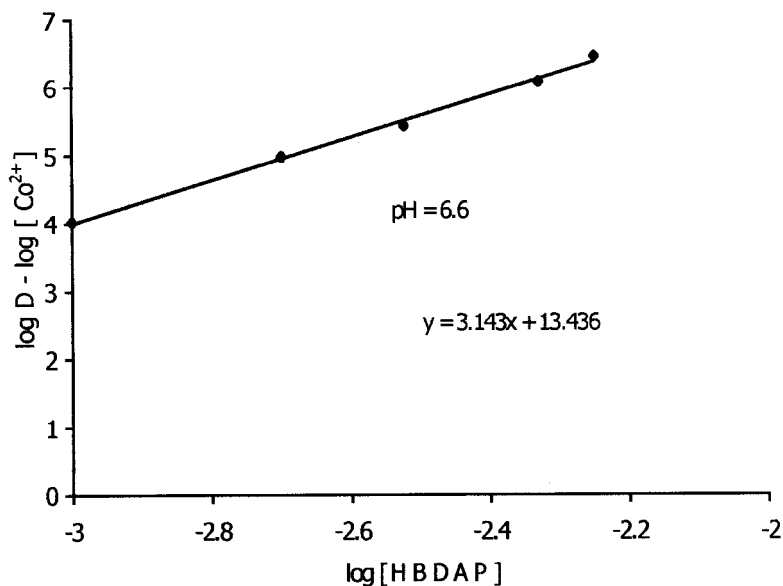


Figure 7. Graphical calculation of the extraction constant. The equation of the straight line is $[\log D - 2\text{pH}] = 0.9966 \log[\text{H}_2\text{L}] - 4.05$.

ditions extraction was completed. The calibration graph obtained was a straight line passing through the origin over the range of mentioned above. The effective molar absorption coefficient at 370 nm was $7.26 \times 10^3 \text{ mol}^{-1} \text{ cm}^{-1}$. The complex was found to obey Beer's law from 0.7 to $7.25 \mu\text{g mL}^{-1}$. The RSD were calculated as 0.05% ($n=13$, $4.2 \mu\text{g mL}^{-1}$). The precision was determined from 25 results obtained for $10 \mu\text{M Co(II)}$; the mean value of a Co(II) was $10 \mu\text{M}$ with a standard deviation of $2.35 \times 10^{-7} \text{ M Co(II)}$. There were no measurable changes in the absorbance of the extracts even after standing for 5 days in a glass-stoppered tube at room temperature. The molar absorption coefficient found for Co(II)-HBDAP complex seems to be lower than for some reagents and higher than some cobalt chelating reagents as seen in Table 1. The developed procedure is highly sensitive and easy to apply for determining the concentration of Co(II) in aqueous solutions.

Effect of Foreign Ions

A 15 mL solution containing $7.07 \text{ mg L}^{-1} \text{ Co(II)}$ and various amounts of foreign ions was treated as described in the procedure. The results are



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Table 1. The Other Reagents for Co(II) Determination as Spectrophotometrically

Reagent	λ_{\max}	$\varepsilon \times 10^{-3}$ ($\text{mol}^{-1} \text{ l cm}^{-1}$)	Interfering Ions	Solvent
2-Chloroquinoline-3-carboxyaldehyde thiosemicarbazone ^[17]	415	1.75		DMF
Dithizone ^[17]	550	4.6	Sn ²⁺ , Fe ²⁺	CCl ₄
3-Bromo-4-mercaptoacetamidotoluene ^[17]	490	8.575		CHCl ₃
<i>N</i> -Hydroxy- <i>N,N</i> -diphenylbenzamide ^[17]	405	7	Fe ³⁺ , Cu	Toluene
<i>p</i> -Methylisonitroisoacetophenone ^[17]	380	23	Cu ²⁺ , Mn ²⁺ , Zn ²⁺ , U, Zr, Th, Be, Ce, Cd, Ag, Cr, Pd, Ru, citrate, oxalate,	CHCl ₃
Ephedrine ^[17]	620	4.3	Cr ₂ O ₇ ²⁻ , Cu, Fe	IBMK
3-Hydroxyl-3- <i>p</i> -chlorophenyl-1- <i>p</i> -carboxyphenyltriazene ^[17]	418	38		Dithizonate
HBDAP	370	7.26		Chloroform

given in Table 2. One milligram per liter concentration of Al(III), Cr(III) and Mn(II) interfered to the absorbance of the complexes as seen in the Table 2. The interference from CO₃²⁻ was observed after 3 mg L⁻¹ and the interference from Cu(II) and Fe(III) was observed after 5 mg L⁻¹. The interference from Ni(II) were observed after 20 mg L⁻¹, the interference from citrate ion was observed after 75 mg L⁻¹ and the interference from Pb(II) and Cd(II) was observed after 200 mg/L. The other ions listed in Table 2 didn't interfere in amounts up to 1 g L⁻¹ with an error below 5%.

Analysis of Vitamin B12 Samples

The spectrophotometric method was applied to the determination of Co(II) in pretreated Vitamin B12 solutions. The results are given in Table 3. The results from proposed method was compared with the results from

**Table 2.** Effect of Foreign Ions on the Determination 7.07 mg L^{-1} of Co(II)

Ion	Amount Added (mg/L)	Co ²⁺ Found (mg/L)	Error (%)
None	—	7.07	0
F ⁻	1000	7.19	1.66
C ₄ H ₄ O ₆ ²⁻	1000	7.0	-0.99
Zn ²⁺	1000	7.19	1.66
Na ⁺	1000	7.06	-0.14
NH ₄ ⁺	1000	7.19	1.66
I ⁻	1000	7.07	0
SCN ⁻	1000	7.01	-0.85
K ⁺	1000	7.07	0
NO ₃ ⁻	1000	7.07	0
Cl ⁻	1000	7.07	0
Ba ²⁺	750	7.07	0
Mg ²⁺	750	7.06	-0.14
Br ⁻	750	7.08	0.17
Ca ²⁺	750	7.19	1.66
SO ₄ ²⁻	750	7.25	2.55
Cd ²⁺	200	6.96	-1.56
Pb ²⁺	200	6.95	-1.7
Citrate ion	75	7.18	1.56
Ni ²⁺	20	7.09	0.28
Cu ²⁺	5	7.36	4.1
Fe ³⁺	5	7.3	3.2
CO ₃ ²⁻	3	6.95	-1.7
EDTA	3	7.01	-0.85
Mn ²⁺	1	7.66	8.3
Al ³⁺	1	5.77	-18.4
Cr ³⁺	1	5.09	-58

Table 3. The Analysis of Vitamin B12 Samples

Sample	Proposed Spectrophotometric Method	FAAS	Certified Value
Nuritrex B12 (mg Co)	$0.0449 \pm 3.087 \times 10^{-3}$	$0.0442 \pm 2.76 \times 10^{-3}$	0.0438
Dovex (mg Co)	$0.0445 \pm 1.74 \times 10^{-4}$	$0.0446 \pm 1.41 \times 10^{-3}$	0.0438
Tribexol (mg Co)	$0.0439 \pm 2.01 \times 10^{-3}$	$0.0441 \pm 4.08 \times 10^{-4}$	0.0438

**PRECONCENTRATION AND DETERMINATION OF Co(II)****2589****Table 4.** The Effect of Acids on Stripping

	HCl			H ₂ SO ₄			HNO ₃		
	3%	5%	10%	5%	10%	20%	5%	10%	20%
Acid (%)	3%	5%	10%	5%	10%	20%	5%	10%	20%
Found (µg)	18.86	18.52	14.91	10.36	9.8	10.96	17.94	19.05	18.77
R (%)	99.85	98.2	79.06	54.93	51.96	58.11	95.1	101	99.5

Table 5. The Application of Preconcentration Procedure at Wastewater Samples

Wastewater Samples	Added (µg/mL)	Found (µg/mL)	Recovery	Recovery (%)	RSD (%)
Dede Carpet Plant	—	0.064	—	—	1.23
	0.1886	0.243	0.179	94.9	2.41
Sulfuric Acid Plant	—	1.75	—	—	2.84
	0.1886	1.939	0.189	100.2	3.2
Leather Plant I	—	0.031	—	—	0.89
	0.1886	0.209	0.178	94.4	1.23
Leather Plant II	—	0.068	—	—	0.45
	0.1886	0.24	0.172	91.1	2.48
Boric acid Plant	—	0.052	—	—	1.77
	0.1886	0.245	0.193	102	1.95

FAAS measurements and certified values in cyanocobalamine samples. The results indicate that the proposed procedures provide a very good accuracy and precision.

Preconcentration of Co(II) and Determination with FAAS

The effect of various acids on the stripping of the aqueous solution containing 18.86 µg Co(II)/100 mL has been given in Table 4 for the preconcentration purpose. The highest recovery values have almost quantitatively been obtained with 3% HCl and 10% HNO₃. The proposed procedure has also been applied to the wastewater samples obtained from different plants before and after the addition of 18.86 µg Co(II) into a sample of 100 mL. It has been concluded that the amount of Co(II) can be determined by using FAAS after the proposed preconcentration procedure (Table 5). By following the proposed procedure, the effect of various ions on the preconcentration of Co(II) and on the determination by FAAS was investigated. For the determination by FAAS of 5.9 ppm of Co(II) in 100 mL, no interference was caused by



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1 g/L of Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Cl^- , NO_3^- , SO_4^{2-} , Ba^{2+} , CO_3^{2-} , I^- , F^- , NH_4^+ , tartaric anion, Zn^{2+} , Cu^{2+} , Ni^{2+} , Cd^{2+} , Co^{2+} , Pb^{2+} and SCN^- . The interferences were observed for 100 ppm Al^{3+} , 100 ppm Cr^{3+} and 250 ppm of Fe^{3+} .

CONCLUSIONS

The results indicate that HBDAP in organic phase extracts efficiently Co(II) in aqueous phase containing 0.1 M KCl in the pH range 8.5–9.5 at 25°C. The extraction mechanism corresponds to a cation exchange, in which a complex of stoichiometric formula ($\text{Co}_2\text{H}_2\text{L}_3$) is formed in the organic phase liberating at the same time 4 mol H^+ ions in the aqueous phase. Extraction constant has been calculated as $\log(K_{\text{ex}}) = -12.6 \pm 0.03$. The proposed method may suffer interferences from only a few ions (Fe^{3+} , Al^{3+} , Mn^{2+} and Cr^{3+}). When the developed procedure was applied to vitamin B12 samples, the results obtained were satisfactory. A preconcentration procedure has been proposed for the determination of Co(II) in wastewater samples which contain only trace concentrations of Co(II) that can not be measured directly by FAAS. Extraction of aqueous phase containing Co(II) with organic phase containing HBDAP and then stripping the organic phase with 10% HNO_3 gave a solution Co(II) that could be directly analyzed by FAAS. The proposed method was validated by spectrophotometry (Table 3).

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