

Solid Phase Exteraction of cobalt (II) using Octadecyl Silica Membrane Disks Modified Nano-Fe₃O₄-Encapsulated-Dioctylphthalate and Linked-Diethylenetriamine

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ABSTRACT

A novel and selective method for the fast determination of trace amounts of cobalt (II) in water samples has been developed. The procedure is based on the selective formation of Cd(II) using octadecyl-bonded silica membrane disks modified by nano-Fe₃O₄-encapsulated-dioctylphthalate and linked-diethylenetriamine (magnetic nano- Fe₃O₄ -DOP- DTA) at different pH in the presence of acetate as an enhancement agent of Co(II) signals followed by elution with organic eluents and determination by atomic absorption spectrometry. The maximum capacity of the employed disks was found to be $385 \pm 5 \mu\text{g}$ for Co(II). The detection limit of the proposed method is 6.0 ng.L⁻¹ for Co(II). The effect of potential interfering ions, ligand amount, stripping solvent, and sample flow rate were also investigated. Under the optimal experimental conditions, the break-through volume was found to about 1000mL providing a preconcentration factor of 500. The proposed method was successfully applied for determination of Co(II) in different water samples.

Keywords: Co²⁺; Extraction; Octadecylsilica disks; atomic absorption spectrometry; magnetic nano-Fe₃O₄ -DOP- DTA.

INTRODUCTION

Cobalt at little concentrations acts in the role of micronutrient and a toxicant in pelagic and water systems [1-3]. This metal is needed by herbs at only very small levels and is toxicant at higher levels. At high levels, Co²⁺ ions can bind to the membrane of the cell and hinder the transport procedure through the cell wall. Co²⁺ ions at nearly 38 ng/mL are required for the usual metabolism of many living organisms [4]. Moreover, Co²⁺ ions are an important element in a multitude of industries. Thus, the development of novel methods for selective extraction, concentration, and specification of it in sub-micro levels in various industrial, pharmaceutical, and samples of environmental is of continuing interest. The assessment of Co²⁺ ions is usually carried out by graphite and flame furnace atomic absorption spectrometry^{11, 12}, and other spectrometric methods[5]. Although due to the presence of Co²⁺ ions in pharmaceutical and environmental samples at

little levels, its separation from other metals presents and also the application of a preconcentration step before its determination is usually necessary.

Various methods, especially Liquid- Liquid micro extraction of Co²⁺ ions in the presence of different classical[3]and macrocyclic [6]co-extractant ligands have attracted remarkable attention. However, the use of classical extraction techniques for this goal is usually labor-intensive, time-consuming, and requires a large value of high purity solvents for separation. Meantime, several other methods for the preconcentration and extraction of Co²⁺ ions have been proposed by liquid chromatography [7], flotation[8], aggregate film formation [9], supercritical fluid extraction[10], liquid membrane[11], column adsorption from pyrocatechol violet-cobalt complexes on carbon activate[12], ion-pairing[13], and SPE extraction using disks cartridges and C₁₈[14].

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The ability of the FAAS system to perform multi-element trace metal analysis of environmental samples provided commercial laboratories with the needed incentive to enter into the business of trace metal analysis. Despite the selectivity and sensitivity provided by this technique, there is a crucial need for the preconcentration of ultra trace elements before their analysis due to their frequent low concentrations in numerous samples. Additionally, since FAAS emission is normally subjected to high levels of interferences that usually accompany analytes; a cleanup step is often required. Many methods have been developed for the preconcentration of trace heavy metals from various samples. These include coprecipitation, electrode position, liquid-liquid extraction and solid phase extraction. Solid phase extraction (SPE) has received much attention in recent years for the analysis of trace concentrations in samples. This technique reduces the use of solvents, which reduces disposal costs and extraction times[15-17]. In past years, the octadecyl-bonded silica SPE disks were utilized for the determination and extraction of different organic compounds from biologic matrices[18]. Besides, the SPE disks were modified by suitable ligands species are successfully applied for selective extraction and preconcentration of heavy metal ions[19].

In a recent series of research, [20]we have presented the usage of metal-DNA conjugates with nucleic acid sequence specification with catalytic signal amplification; the evaluation relies on the esterase activity from DNA-linked cobaltcomplex.

For system optimization and structure-activity relationships identification, a sensitive probe would be helpful, which allows straightforward observation of esterase activity of ligated cobalt (II) in small concentrations. This study was the development of a fast, highly sensitive and efficient method for selectively extraction and preconcentration of ultra-trace values of cobalt (II) from aqueous media with octadecyl silica membrane disks was modified by nano-Fe₃O₄-encapsulated-dioctylphthalate and the linked-diethylenetriamine (nano-Fe₃O₄-DOP-DTA magnetic and AAS determination.

EXPERIMENTAL

Reagents

All acids were of the great purity available from Merck and have been used as received.

Methanol and chloroform were obtained of HPLC grade from Merck. Nitrate salts of lithium, sodium, potassium, barium, zinc, magnesium, nickel, calcium, barium, zinc, strontium, cadmium, lead, cobalt (II), and cobalt(II) in analytical grade and were of the highest purity. The highest pure organic solvents were prepared from Merck, Darmstat, Germany. Ultra Purity double-distilled deionized water is used throughout the experiments.

The stock standard solution of cobalt (II) was prepared by dissolving 0.1000g of the Co metal powder in 10mL concentrated of the nitric acid solution and diluted to 1000mL with distilled water in a calibrated flask. The working solutions were obtained by appropriate dilution of the stock solution. All the dilutions have been prepared with the highest pure deionized water. Dioctyl phthalate, sodium hydroxide (NaOH), DOP (purity> 99.55%) and diethylenetriamine (DTA) is purchased from BDH, UK.

Preparation of Magnetic Nano-Fe₃O₄ Adsorbent

The magnetic nano- Fe₃O₄ was first prepared according to a previously reported technique. A 6.1 g of FeCl₃·6H₂O sample and 4.2 g of FeSO₄·7H₂O were dissolved in 100 mL of double-distilled water. AnM-NaOH (25.0 mL) was slowly added to the above solution. The reaction mixture was shaken for 240 min. After the complete addition of NaOH, the black precipitateis formed (nano-Fe₃O₄) and then washed several times with deionized water, collected by the assistance of a magnetic field, and then dried in an oven at 70 °C.

Preparation of Nano-Fe₃O₄-DOP Adsorbent

The Fe₃O₄-encapsulated-DOP nano-adsorbent was synthesized by the addition of DOP (20.0 mL) to nano- Fe₃O₄ sorbent (10.0 g). This mixture is heated under stirring at 90 °C for 180 min and the product nano-Fe₃O₄-DOP adsorbent was filtered, washed with methanol solution, and then collected in a pure form with the magnetic field and then dried in an oven (70 °C) until complete dryness.

Preparation of Magnetic Nano- Fe₃O₄ - DOP-DTA Adsorbent

The nano-Fe₃O₄-DOP (10.0 g) addition to DTA (5 mL) and these two reactants was combined with heavy grinding and was mixed in a mortar for 300 min. The produced nano-Fe₃O₄DOP-TETA adsorbent is heated to dryness in an oven (60 °C).

Apparatus

Determination of cobalt (II) ions contents in working samples were performed by a Varian spectra A.200 model with atomic absorption spectrometer equipped with a high-intensity hollow cathode lamp (HI-HCL). These characteristics are tabulated (Table 1).

SPE extractions were performed by glassy membrane disks, ENVI-18DISK™ 47mm of diameter ×0.6 mm thickness with octadecyl silica bonded phase (30 μm particles, 70 Å pore size) prepared from Supelco in conjunction with filtration apparatus of standard Millipore 47 mm equipped with a vacuum pump. The pH measurement was performed by an ATC pH meter (EDT instruments, GP 353).

Table1. The operational conditions of flame for the determination of Co²⁺ ions

Slit width	0.7 nm
Operation current of HI-HCL	15 mA
Resonance fine	324.8nm
Type of background correction	Deuterium lamp
Type of flame	Air/acetylene
Air flow	7.0 mL.min ⁻¹
Acetylene flow	1.7 mL.min ⁻¹

Sample Extraction

Extraction was carried out with glassy membrane disks of ENVI-18DISK™ 47mm diameter ×0.6 mm thickness with octadecyl silica bonded phase of 30 μm particles and 70 Å pore size from Supelco. The disks were applied in conjunctions by a standard Millipore 47mm filtration apparatus was connected to a water aspirator [21].

Sample Treatment

The aqueous samples were filtered through nylon filters (45μm). Sampling vessels are polyethylene bottles soaked in 1 mol/L of HNO₃ overnight and rinsed twice with distilled water. The analysis should be done within 2 days of the sample collection to limit the venture of inter conversion of Co²⁺. Then, the methanol solution (5mL) was added to a 90mL part of each before analysis. The surface of the disks of ENVI-18 DISK™ is not modified with magnetic nano- Fe₃O₄ -DOP- DTA and therefore cannot retain cobalt (II) ions properly. In lieu, magnetic nano- Fe₃O₄ -DOP- DTA(10 mg) was dissolved in a suitable 5mL an organic solvent miscible with water. The most appropriate solvent under the analysis conditions was acetone. The solution of magnetic nano- Fe₃O₄

-DOP- DTA was added to an aqueous solution of cobalt (II) and the mixture was stirred gently.

Disk Cleaning and Conditioning

A disk was put in the device and with methanol (10mL) to eliminate all contaminants arising from the construction process and the environment. Then, the disk is dried by passing air per it for several minutes. To ensure optimum extraction of the analytes of fondness, the disk was again washed with methanol (10 mL), immediately followed by water (10mL), without letting the disk dry surface. This step pre-soak the disk surface before extraction. Improper performance of this stage causes slow flow – rate and negligible analyte recoveries. It is important to eschew any air contact with the disk surface before the addition of the sample.

Sample Addition

After the complete process of homogenization, 100mL portions of accurate volumes of the sample solutions were transferred to the top reservoir from the disk device. Meanwhile, the solution was drawn via the disk by applying a moderate vacuum. Usage of vacuum was continued up to the disk was completely dry (about 5 min).

Analyte Elution

To elute the analyte selectively, just 5mL of acidified HCl solvent (0.1M) in methanol was passed via the disk and collected in a volumetric flask (5.0 mL) under the extraction funnel. It was achieved that the highest pure alcoholic organic solvents are the best eluting agents. The Co²⁺ concentration in the eluates was then determined by FAAS with an external calibration graph.

RESULTS AND DISCUSSION

Evaluation of the Role of the Ligand

Some preliminary analyses were carried out for investigation of the absence or presence of magnetic nano-Fe₃O₄-DOP-DTAon the quantitative extraction of Co²⁺ ions. It was consummate that the membrane disk itself did not show any tendency for the keeping of Co²⁺ ions, but the introduction of aqueous Co²⁺ ions samples (100mL)withCo²⁺ ions (10μg) and magnetic nano- Fe₃O₄ -DOP- DTA(10mg) leads to satisfactory its retention(Table 2). The next case is most probably ascribed to the existence of a remarkable interaction between Co²⁺ ions and the magnetic nano- Fe₃O₄ -DOP-

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DTA. It must be mentioned that stable complexes formation between Co²⁺ and magnetic nano-Fe₃O₄-DOP-DTA at pH=2 is

maybe due to a mechanism of ion-pair formation. However, at higher pH than 2, the retention recovery of Co²⁺ ions is negligible.

Table 2. The effect of the presence of magnetic nano- Fe₃O₄-DOP- DTA on extraction percent of Co²⁺ ions^a

magnetic nano-Fe ₃ O ₄ -DOP-DTA	pH	Extraction percent of Co ²⁺ ions
Absence	2-6	0.01(3.2) ^b
Presence	2-6	96.4(2.4) to 63(2.0)

a Initial samples contained 10µg of Co²⁺ ions in 100mL of water.

b Values in parentheses are RSDs based on five individual replicate analyses.

Choice of Eluent

To select the most proper eluent for the quantitative stripping for retained Co²⁺ ions on the disks, 5mL of different nonorganic (containing 10% V/V of methanol) and various organic solvents were tested. The data are tabulated in Table 3. As shown, the best eluting solvents are found to

be methanol or ethanol (5mL), resulting in quantitative elution of Co²⁺ ions from the disk. It should be corroborated that the presence of methanol in any type of employed solvents aids to better the contact of eluent with the hydrophobic of the disk surface.

Table 3. Effect of various eluting solvents on percentage recovery of Co²⁺ ions adsorbed on the disk ^a

Stripping solution	% Recovery		
	2ml	5ml	10ml
Methanol	81(2.2) ^b	92.5(2.6)	92.0(2.5)
Acidified methanol ^c	55(2.1)	87.0(2.3)	83.7(1.5)
Ammoniacal methanol ^d	55 (2.3)	87.3(2.3)	83.7(2.3)
Ethanol	87.1(1.5)	98(1.3)	99.2(2.1)
Acetonitril	31(2.1)	41(5.2)	51.5(3.2)
Formic acid(1M)10% V/V mthanol	57(1.3)	61.3(1.6)	71.7(2.3)
Hydrochloric acid(1M)10% V/V mthanol	57(1.5)	93.3(2.1)	93.6(1.5)
Hydrochloric acid(1M)10% V/V mthanol	51(1.1)	81(2.3)	91.3(1.1)
Nitric acid(2M)10% V/V mthanol	51(1.5)	83(2.3)	85.9(2.1)
Nitric acid(1M)10% V/V mthanol	35(1.3)	83(2.0)	89.1(1.2)
Ethanol	81(2.9)	91.5(2.2)	93.5(2.1)

a Initial samples contained 10 µg of each Co²⁺ ions in 100 mL water.

b Values in parentheses are RSDs based on five individual replicate analysis.

c Acidified solvents achieved by addition of 0.1M HCl.

d Ammoniacal solvents achieved by addition of 0.1M NH₃

The Effect of the Ph

The pH of all sample solutions was adjusted to various values between 2-9 by suitable buffer addition (sodium acetate, sodium dihydrogen phosphate, hydrochloric acid, acetic acid, and disodium hydrogen phosphate) and then these samples passed through the disks [22, 23].

Finally, the Co²⁺ ions were stripped by pure ethanol or methanol solutions followed by FAAS determination of the eluted Co²⁺ ions. Then, the percentage of recovery at different pH values was determined (Fig. 1). As results are shown in Fig. 1 up to pH 4.0-4.5, perfect recoveries are obtained. However, at values of higher pH, percentage recovery is decreased. This is since in an acidic solution with protonation of magnetic

nano- Fe₃O₄ -DOP- DTA occurs and there is a feeble tendency for retention between cobalt(II) and magnetic nano-Fe₃O₄-DOP-DTA, whereas, at pH > 5, cobalt(II) reacts with hydroxide ions for produce Co(OH)₂. Hence, the buffer of sodium acetate-acetic acid with pH = 4.5 was applied for the preconcentration step. The other solvents used for the dissolving of magnetic nano-Fe₃O₄-DOP-DTA are methanol and ethanol. The efficacy of these solvents on the recoveries amount as a function of pH is compared and shown in Fig. 1. However, other organic solvents are not tested because of their limited solubility, and two phases formation with aqueous solutions and conflict with a flame for higher pH values (>7) are not tested because of the probability of octadecyl silica hydrolysis in the

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disks. cobalt (II) ions could be retained quantitatively by the membrane disk modified via the pH range from 4.0-4.5. But, at lower pH (< 4.0), atoms of nitrogen of the magnetic nano-Fe₃O₄-DOP-DTA could be protonated and reduced the stability of the complex.

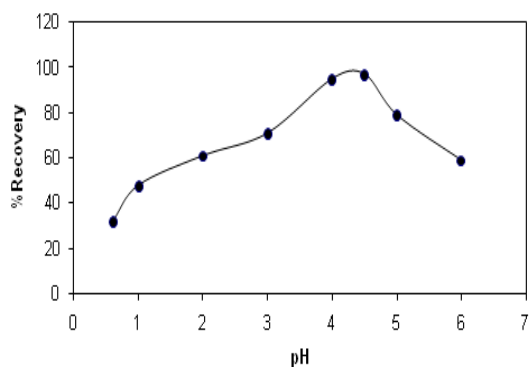


Fig1. Influence of pH of sample and dissolving solvent of magnetic nano- Fe₃O₄ -DOP- DTA on the percentage recovery of cobalt(II).

Effect Amount of Counter Anion

To investigate the effect of counter anion on the recovery of cobalt (II) ions by the modified disks, various opposite anions were tested in Table 4, it is directly obvious that the counter anion nature strongly influences the retention of Co²⁺ ions by the disk. The results ascertained that the magnetic nano-Fe₃O₄-DOP-DTA acts as a neutral ionophore in the pH range, about 4.0 to 4.5, so that the Co²⁺ ions are retained as complexes of ion-pair by the membrane disks. As shown, acetate ion is the maximum efficient counter anion for the cobalt (II) ions extraction. The influence of sodium acetate ion concentration on Co²⁺ ions recovery was investigated, and the results are displayed in Table 4. As shown, the percent recovery of cobalt (II) ions increased with the concentration of acetate until a reagent concentration of 0.1 M is reached, further which the recovery remained quantitative.

Also, acetate ions act as a suitable buffer, while it effectually contributes to the formation of ions- pair. Therefore, in the SPE experiments, there is no necessity for the addition of any solution of the buffer.

Table4. Percent recovery of Co²⁺ ions from the modified membrane disk in the presence of 0.01 M of various counter anions^a

counter anion	%Recovery
Cl ⁻	21.7
Br ⁻	22.9
ClO ₄ ⁻	32.7

SCN ⁻	41.6
Picrate	70.3
Acetate	90.7

The Influence of Flow-Rate

One of the most significant parameters affecting SPE extraction is the rate of the process. Hereupon, the effect of flow-rates on efficiencies of extraction was studied. It was found that in range, about 10-100 mL/min, the retention of cobalt (II) ions was not extremely affected by flow-rates of the sample solutions and leads to reproducible and good results (Fig. 2). So, the flow-rate was held at 89mL/min overall the experiment.

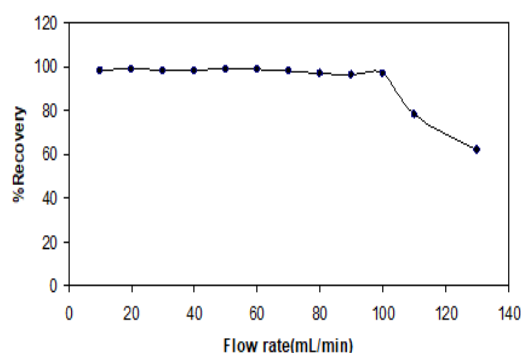


Fig2. The effect of the flow-rate on extraction percent of cobalt(II).

Quantity of the Magnetic Nano-Fe₃O₄-DOP-DTA

The optimized amount of magnetic nano-Fe₃O₄-DOP-DTA for the quantitative extraction of cobalt (II) ions was also evaluated by adding different amounts of it to the solution (2-20 mg). The data are listed in Table 5. The test results demonstrate that the extraction of cobalt (II) ions were quantitative using a solution containing rather than 10 mg magnetic nano-Fe₃O₄-DOP-DTA. Therefore, subsequent extractions were carried out with 15mg value of magnetic nano-Fe₃O₄-DOP- DTA.

Table5. Influence of the magnetic nano- Fe₃O₄ -DOP- DTA value on the recovery of Co²⁺ ions^a

magnetic nano- Fe ₃ O ₄ -DOP- DTA amount (mg)	Recovery(%) of Co ²⁺
2	28(1.9) ^b
5	40(2.8)
8	83(2.2)
10	94.7(2.7)
15	94.8(2.5)
20	94.7(2.2)

^a Initial samples contained 10 μg of each Co²⁺ in 100 mL water.

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^b Values in parentheses are RSDs based on five individual replicate analyses.

ANALYTICAL PERFORMANCE

When solutions of 10 µg Co²⁺ in range 10-3000 mL solutions under optimum experimental conditions have been passed via the disks, the cobalt (II) ions were quantitatively retained in all cases. Afterward, the breakthrough volume for this method must be larger than 2500 mL, providing a value of the concentration factor of >500. The detection limit (LOD) of this method for the determination of cobalt (II) ions was studied under the optimum experimental condition. The LOD based on 3σ of the solution of blank (5 mL form ethanol) was 6 ng per 1000 mL.

The modified disk's capacity (5 mg magnetic nano-Fe₃O₄-DOP-DTA) was characterized by passing 50 mL portions of solutions containing 8 mg of Co²⁺ and 0.1 M of sodium acetate-acetic acid buffers with a pH value of 4.0-4.5, followed by the retained metal ions determination in the eluting solution with AAS. The maximum

capacity of the disk achieved from three replicate measurements is 387 ± 2 µg of cobalt (II) ion on the disk.

To study the selective separation and extraction of cobalt (II) ions from its binary mixture for various heavy metal ions, a fractional of aqueous solution (50 mL) containing 10 µg cobalt (II) ions and mg value of other cations were taken and the recommended process was followed. The results are deposited in Table 6. The results display that the Co²⁺ in the binary mixture is retained almost completely with the modified disk, even in the attendance of up to about 100 mg of different ions. However, retention of other ions metal by the disk is very little and they can be extracted effectively from the cobalt (II) ions. It is interesting to attend that, in other tests, we found that in the attendance of high enough concentration of NH₂OH.HCl as a proper reducing agent (>0.5 M). The no retention of the eventuating single charge cobalt (I) ion can occur with a modified membrane disk.

Table 6. Separation of Co²⁺ from binary mixtures ^a

Sample	Co ²⁺ added (µg)	Co ²⁺ determined (ng/mL)	ICP-AES
Tap water	0.0	ND	ND
	10.0	10.70(2.7) ^a	10.20
Snow water	0.0	2.05(2.9)	ND
	10.0	12.09(2.0)	12.1
Rain water	0.0	1.09(2.5)	ND
	10.0	11.05(2.8)	11.3
Sea Water	0.0	17.06(2.4)	17.12
	10.0	27.29(2.7)	27.3

Initial samples contained 10 µg cobalt and different amounts of various ions in 100 mL water (0.1 M acetate ions).

^b Values in parentheses are RSDs based on five individual replicate analysis

ANALYSIS OF WATER SAMPLES

To evaluate the applicability of the procedure to real samples, it was used for the extraction and determination of Co²⁺ from various water samples. Tap water (Tehran, taken after 15 min action of the tap), The rain water (Tehran, 25 January 2020), The snow water (Tehran, 15 February 2020), and the seawater (taken from

Caspian sea, Mahmoud-Abad shore) samples have been analyzed (Table 7). As can be shown from Table 7 the added Co²⁺ ions could be quantitatively recovered from the waters used. As is shown, the recovered Co²⁺ ion shows that the results are entirely reliable and were in satisfactory agreement with the result obtained by ICP-AES.

Table 7. Recovery of Co²⁺ ions added to 1000 mL of various water samples (containing 0.1 M acetate at pH = 4.0-4.5).

Diverse ion	Amounts taken (mg)	% Found	% Recovery of Co ²⁺ ion
Na ⁺	92.6	1.13(2.6) ^b	94.3(1.85)
K ⁺	92.7	1.22(2.4)	94.2(2.6)
Mg ²⁺	14.6	0.80(1.9)	94.2(1.8)
Ca ²⁺	23.8	2.45(3.3)	94.4(1.5)
Sr ²⁺	2.43	2.24(2.3)	94.0(2.0)
Ba ²⁺	2.44	3.23(2.6)	94.4(2.6)
Mn ²⁺	2.50	1.65(2.7)	93.5(2.0)

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Co ²⁺	2.23	1.70(2.6)	99.5(2.2)
Ni ²⁺	1.37	2.20(2.4)	94.5(2.0)
Zn ²⁺	2.56	1.90(2.2)	97.5(2.2)
Cd ²⁺	2.50	1.95(2.4)	94.5(2.3)
Pb ²⁺	0.43	2.74(1.3)	97.6(2.2)
Hg ²⁺	0.42	2.44(2.2)	97.5(2.5)
Ag ⁺	2.35	3.25(2.5)	94.5(2.0)
Cr ³⁺	1.78	2.76(2.1)	97.6(2.8)
UO ²⁺	2.40	2.45(1.4)	95.4(2.8)

^a Values in parentheses are %RSDs based on five individual replicate analysis

^b Not detected.

CONCLUSION

Results presented in this study indicate well the tremendous possibilities suggested by the solid-phase extraction of little amounts of cobalt (II) ions in water samples with Octadecyl Silica membrane disks modified with magnetic nano-Fe₃O₄-DOP-DTA and its determination with FAAS. The technique developed was easy, reliable, and accurate for determining Co²⁺ in water samples. Besides, the proposed procedure was free of interference comparing to conventional methods to determine Co²⁺ ions.^{51-55,57} This method can be successfully used for the separation and extraction of Co²⁺ ions in binary mixtures.

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