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## Determination and preconcentration of nickel in some geological, environmental and biological samples by FAAS

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## ABSTRACT

In this study, new, sensitive, simple and validated solid-phase extraction method is developed for the determination of nickel. Nickel was preconcentrated as 4-(2-Pyridylazo) resorcinol chelates (Ni-PAR) from sample solutions using a column containing Duolite XAD-761 and determined by flame atomic absorption spectrometry (FAAS). The optimal experimental parameters for the nickel assay have been investigated. The optimum pH value for quantitative sorption of Ni-PAR was found between 3.5 and 4.5. Elution process was performed by 5 mL of 2 mol L<sup>-1</sup> HCl. The sorption capacity of resin was determined to be 1.8 mg g<sup>-1</sup> for Ni. The preconcentration factor was 120. In optimized conditions, the detection limit for nickel was found to be 0.64 µg L<sup>-1</sup>. The method was applied to different waters, food and guinea pig tissue samples. Calculated recoveries for waters, bean and guinea pig lung samples were as 98.6-102.1, 98.3 and 98.9 % respectively. The method was extended for determination of nickel in standard reference materials (NIST 1573 a-tomato leaves) and to investigate the accuracy of the proposed procedure. Influences of some interfering species were also © 2010 Trade Science Inc. - INDIA investigated.

### **INTRODUCTION**

Nickel is a toxic trace element of widespread distribution in the environment. It, usually, enters waters from waste disposals of different industrial processes such as electroplating, batteries, pigments for paints and ceramics, surgical and dental prostheses, magnetic tapes and computer components, catalysts and also it is emitted to the atmosphere from volcanoes and windblown dusts<sup>[1-3]</sup>. It is well known that this metal is quickly absorbed by plant roots and is highly mobile in plants<sup>[2,3]</sup>. The high nickel accumulation in organisms causes vari-

## KEYWORDS

Solid phase extraction; Duolite XAD-761; PAR; Nickel; Preconcentration; FAAS.

ous cancer kinds (lung, nose, larynx and prostate cancer) lung embolism, respiratory failure, birth defects, asthma and chronic bronchitis, allergic reactions such as skin rashes and heart disorders<sup>[4-8]</sup>. Therefore, the development of sensitive new methods for determining nickel in the environmental, biological and food samples is necessary and important<sup>[9]</sup>.

The determination of nickel in environmentally and biologically has been carried out by instrumental techniques such as neutron activation analysis (NAA)<sup>[10]</sup>, inductively coupled plasma optic emission spectrometry (ICP-OES)<sup>[11,12]</sup>, inductively coupled plasma mass

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spectrometry (ICP-MS)<sup>[13]</sup>, X-ray fluorescence spectroscopy<sup>[14]</sup> and chromatography<sup>[15,16]</sup>. Recently, atomic absorption spectrometry has been used as the most common method for trace metal determination in various samples. The levels of metal ions in natural samples are usually lower than the detection limit of most instruments, and metals usually exist in very complex matrix environments. Direct analysis of metals without using a sample preparation technique is impossible due to interferences<sup>[17,18]</sup>. Therefore, preconcentration step is usually required for determination of metal ions with concentrations lower than the detection limit of technique. There are many methods of preconcentration such as, coprecipitation, solvent extraction, electrochemical deposition, membrane extraction, and solid phase extraction<sup>[19]</sup>. Solid-phase extraction (SPE) has become a preferred method at enrichment of many metals ions prior to their analysis by FAAS and other techniques. SPE is more useful method from the point of flexibility, higher enrichment factors, absence of emulsion, low cost, high speed and simplicity of procedures, safety with respect to hazardous samples and more importantly environmental friendly according to other methods<sup>[20]</sup>.

In the present work, a new method was developed for the determination of trace amount of nickel by using FAAS in different samples. Nickel-PAR chelates can be adsorbed on XAD-761 polymer resin in column and optimum analytical conditions on nickel recovery were investigated. Analytical parameters such as precision and accuracy of the method have also been studied. The developed method has been successfully employed for the determination of the analytes in various samples.

## EXPERIMENTAL

### Apparatus

A Perkin Elmer Aanalyst model 700 (Shelton, CT, USA). Flame Atomic Absorption Spectrometer (FAAS) with a hallow cathode lamp and a deuterium background corrector, at respective resonance line using an air–acetylene flame was used. The instrumental parameters for nickel determination were found as follows: wavelength 232.0 nm, lamp current 7.5 mA, bandpass 0.2 nm. Schott Lab-Star pH meter was used to measure the pH of solutions.

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### **Reagents and solutions**

All reagents used were of analytical grade and all solutions were prepared by using triple distilled and deionized water. Duolite XAD-761 is an ester acrylic polymer and it was purchased from Sigma–Aldrich. The size of resin particles was 20–60 mesh. The PAR monosodium salt hydrate was purchased from Aldrich and standard PAR solution of 0.024% (w/v) was prepared by dissolving 0.012 g of PAR in 50 mLmixture of methanol and 0.2 mol L<sup>-1</sup> NaOH solution (55:45 % v/v). Metal solutions were prepared by atomic absorption standard solutions ( $1000 \pm 2 \text{ mg L}^{-1}$ ). Nitric acid (65% w), hydrochloric acid (37% w), methanol, ethanol, acetonitrile (ACN) and other chemical reagents were from Merck (Darmstadt, Germany). All chemicals were used without further purification.

Adsorption column were prepared according to literature<sup>[21]</sup>. The glass column was 12 cm length and 0.8 cm internal diameter. A small amount of glass wool was placed at the bottom of the column in order to held resin. 0.45 g dried resin was placed and another small glass wool plug was inserted onto the tap of the resin. The bed height of the resin in the column was approximately 2.0 cm. It was washed successively with water, ethanol, 2 mol L<sup>-1</sup> HCl and HNO<sub>3</sub> solutions, respectively.

### **Preconcentration procedure**

The standard test solutions were prepared as follows: 5 mL of 0.5 mg L<sup>-1</sup> of Ni (II) standard solution added to 1 mL of standard PAR solution and 2 mL buffer solutions in a volumetric flask The pH of the solution was adjusted to optimum pH by the addition of diluted NaOH or HCl solutions. Afterwards, final volume was diluted to 50 mL with distilled water. This solution was permitted to flow through the column under gravity at flow rate of 4 mL min<sup>-1</sup>. The adsorbed nickel ions on the column were eluted with 5 mL of 2 mol L-1 HCl solution that has a flow rate of 4 mL min<sup>-1</sup>. Nickel was analyzed with method of direct calibration curve by FAAS. Device setting is controlled every five readings. A blank solution was also run under the same conditions without adding any nickel. In this study each measurement was repeated three times by FAAS.

### Analysis of water samples

The water samples (from city line and Kizilirmak

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river in Kirsehir-Turkey) were filtered Whatman filter paper (No. 40) and 600 mL of water sample was transferred to a beaker. Then 1 mL of standard PAR solution and 2 mL of sodium acetate-acetic acid buffer solutions were added. The pH of the solution was adjusted to 4.5 and passed through the Duolite XAD-761 column. After elution process, analyses of samples were performed according to recommended preconcentration procedure.

### Analysis of standard reference material

To validate the developed method, standard reference material (SRM) was used. 0.2 g of SRM (NIST 1573 a-tomato leaves) was digested with 8 mL of HNO<sub>2</sub> and 2 mL HClO<sub>4</sub> in a microwave digestion system according to our previous studies<sup>[22]</sup>. Digestion conditions in polytetrafluorethylene (PTFE) vessels were applied as 650W, 5 min; 800W, 10 min; 650 W, 5 min. Vessels were left to cool for an hour and were carefully opened. Colorless solution was transferred into a beaker and solvent was evaporated to dryness by using hot plate. Afterwards, 25 mL of distilled water, 2 mL of sodium acetate-acetic acid buffer solution and 2 mL of 0.1 mol L<sup>-1</sup> sodium citrate solutions (to eliminate interference effect of Fe<sup>3+</sup> ions on nickel recovery) were added and filtered through a filter paper. Then, 1 mL of standard PAR solution was added and pH of this solution was adjusted to 4.5 and diluted to 50 mL with distilled water. Afterwards, the preconcentration procedure given above was applied. The experiments for blank solutions were carried out in the same way.

## Analysis of food and guinea pig lung

0.5 g dried bean and 0.2 g guinea pig lung samples were weighed into PTFE bombs. For the acid digestion of samples 8 mL HNO<sub>3</sub> (65% w) and 2 mL HClO<sub>4</sub> (60% w) were added. Preparations and analysis of samples were performed according to analysis of standard reference material procedure.

## **RESULTS AND DISCUSSION**

In order to obtain quantitative recoveries of nickel on the XAD-761 polimer resin, the separation/ preconcentration procedure was optimized for various analytical parameters, such as pH of sample solution, amount of resin, volume and type of elution solution, amount of ligand, flow rate of sample solution and volume of sample solution. The interfering effects of other ions were also investigated. The recovery of analyte ion separated and preconcentrated on the column was calculated from the amounts of metal ion in the starting sample and the amount of metal ion in the eluent.

## Effect of pH of the aqueous solution on the retention of Ni(II)

The adsorption of Ni-PAR chelates on Duolite XAD-761 resin at different pH range was investigated with the use of different buffers (sodium citrate-HCl, sodium acetate-acetic acid, sodium mono hydrogen phosphate-potassium di hydrogen phosphate and ammonium chloride-ammonia). Effect of the pH on the recovery values were summarized in Figure 1. As shown in the figure, the optimum recoveries were found between the pH value of 3.5 and 4.5. Therefore, pH 4.5 was chosen as an optimum pH in sodium acetate-acetic acid buffer for analytical determination of nickel in further studies.



Figure 1 : The effect of pH on the recovery of nickel (sample volume: 50 mL, amount of nickel:  $2.5 \mu g$ , eluent: 5 mL of  $2 mol L^{-1}$  HCl solution, flow rate of sample:  $4 mL min^{-1}$ , sorbent: 450 mg, N=3).

## Influence of amount of 4-(2-Pyridylazo) resorcinol

Different amounts 4-(2-Pyridylazo) resorcinol were added to test solutions containing  $2.5\mu$ g of Ni (II) and these test solutions passed then through Duolite XAD761 column. The results were given in Figure 2. Quantitative recovery was obtained for Ni (II) ions in the 0.1–0.5 mg range of PAR. At amount of PAR less than 0.1 mg and higher than 0.5 mg, the recovery was below 95%, due to competition on the adsorption be-

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tween PAR chelates and excess PAR in the solution. In further works, 0.24 mg of PAR was added to the solutions.



Figure 2 : The effect of PAR volume on the recovery of nickel (sample volume: 50 mL, amount of nickel: 2.5  $\mu$ g, eluent: 5 mL of 2 mol L<sup>-1</sup> HCl solution, flow rate of sample: 4 mL min<sup>-1</sup>, pH: 4.5, sorbent: 450 mg, N=3).

#### Effect of sample volume

One of the important parameters in this kind of studies is the selection of the sample volume, so the influence of the sample volume on nickel recovery was examined on XAD-761 column at a 4.0 mL min<sup>-1</sup> flow rate. For this purpose, 50, 200, 400, 600, 800 and 1000 mL of the test solutions containing 2.5  $\mu$ g of nickel were passed through the column at the optimum conditions. The results are shown in Figure 3. The recovery of nickel was quantitative (>95%) for the sample volume range between 50 mL and 600 mL. For the samples



Figure 3 : The effect of sample volume on the recovery of nickel (amount of nickel: 2.5  $\mu$ g, eluent: 5 mL of 2 mol L<sup>-1</sup> HCl solution, flow rate of sample: 4 mL min<sup>-1</sup>, pH: 4.5, sorbent: 450 mg, N=3).

Analytical CHEMISTRY An Indian Journal have the volumes higher than 600 mL, the percentage sorption of nickel ions was under 95%. By analyzing 5 mL of the final solution after the preconcentration of 600 mL of sample solution, an enrichment factor of 120 was found.

### Effect of flow rate

After the optimization of sample volume, the effects of flow rate on the adsorption of nickel were investigated. To obtain maximum recoveries for nickel, different flow rates were tested on the retention of the Ni-PAR chelates in optimum conditions. The nickel was desorbed from the resin by using 5 mL of 2 mol L<sup>-1</sup> HCl solutions. As shown in Figure 4, it was found that the suitable value for the flow rate of the solution was in the range of 1-4 mL min <sup>-1</sup>. Therefore, an optimum flow rate of 4 mL min <sup>-1</sup> was selected as the working solution flow rate.



Figure 4 : The effect of solution flow rate on the recovery of nickel (sample volume: 50 mL, amount of nickel: 2.5  $\mu$ g, eluent: 5 mL of 2 mol L<sup>-1</sup> HCl solution, pH: 4.5, sorbent: 450 mg, N=3).

### Choice of eluent agents

In order to obtain maximum quantitative recoveries of nickel, various eluent and volume of reagent was studied. For this purpose ethanol, acetonitrile, HCl and HNO<sub>3</sub> solutions were used. The obtained results show that the maximum recovery of nickel was observed by using 5 mL of 2 mol L<sup>-1</sup>HCl solutions. The effects of various eluent on the recoveries of nickel are summarized in TABLE 1.

#### Effect of resin amount

The influence of the amount of XAD-761 resin on

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TABLE 1 : The effect of eluent types on the recovery of nickel (sample volume: 50 mL, amount of nickel:  $2.5 \mu$ g, flow rate of sample: 4 mL min<sup>-1</sup>, pH: 4.5, sorbent: 450 mg, N=3)

Eluent	Recovery (%)
$2 \text{ mol } L^{-1} \text{ HCl}, 4 \text{ mL}$	92±1
$2 \text{ mol } L^{-1} \text{ HCl}, 5 \text{ mL}$	98±3
2 mol L <sup>-1</sup> HNO <sub>3</sub> , 4 mL	72±2
5 mL of ethanol-2 mol $L^{-1}$ HCl mixtures (1:3 v/v)	85±2
4 mL ethanol	12±1
4 mL acetonitrile	18±2

recoveries of nickel studied at different amounts of sorbent. To this aim, the XAD-761 resin was added in the range between 200-800 mg into the adsorption column. The test solution, whose volume is 50 mL and includes  $2.5 \ \mu g$  of nickel, was passed through the column at optimum conditions. The results showed that the optimum amount of sorbent was found in the range of 450 to 800 mg for maximum extraction of Ni (II) (Figure 5). From these results, optimum amount of resin was selected as 450 mg of resin has been used in all further experiments.



Figure 5 : The effect of XAD-761 amount on the recovery of nickel (sample volume: 50 mL, amount of nickel:  $2.5 \mu g$ , eluent: 5 mL of 2 mol L<sup>-1</sup> HCl solution, flow rate of sample: 4 mL min<sup>-1</sup>, pH: 4.5, N=3).

## Influence of interfering species

The interference studies were performed using various possible interfering ions on the retentions of the investigated analyte. These ions were examined in the test solutions. For this purpose, the influences of some cationic and anionic species were investigated. In these experiments, 50 mL of solutions containing 2.5  $\mu$ g of nickel and various amounts of possible interfering ions were treated according to the preconcentration procedure. The degree of interfer-

ence effects were shown as ratio of the recovery in the presence of interfering ions to that in their absence. The results are given in TABLE 2, and the percentages showed that, in excess of 1000 mg L<sup>-1</sup> of K<sup>+</sup>, Na<sup>+</sup>, Ca <sup>2+</sup>, Mg <sup>2+</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, SO<sub>4</sub> <sup>2-</sup> and 20 mg L-1 of Ag<sup>+</sup>, Cu<sup>2+</sup>, Co<sup>2+</sup>, Zn<sup>2+</sup>, Mn<sup>2+</sup>, Pb<sup>2+</sup>, Al<sup>3+</sup>, Fe <sup>3+</sup> and Cr<sup>3+</sup> ions have no significant interferences in the extraction and determination of nickel in test solutions. The tolerance limit is defined as the ion concentration causing a relative error smaller than  $\pm 5\%$ (except Fe<sup>3+)</sup> related to the preconcentration and determination of nickel. Interfering effect was observed when Fe<sup>3+</sup> concentration of more than 20 mg L<sup>-1</sup>. To eliminate the interference effect of Fe<sup>3+</sup> ions, 2 mL of 0.1 mol L<sup>-1</sup> sodium citrate solutions is added as masking agent to 300 mg L<sup>-1</sup> containing Fe<sup>3+</sup> in synthetic solution.

TABLE 2 : The effect of some ions on the recovery of nickel

Ion	Concentration (mg L <sup>-1</sup> )	Recovery (%)	Ion	Concentration (mg L <sup>-1</sup> )	Recovery (%)
$\mathbf{K}^+$	1000	99±3	Co <sup>2+</sup>	20	96±2
$Na^+$	1000	100±2	$Zn^{2+}$	20	98±2
$\mathrm{Ca}^{2+}$	1000	97±2	$\mathrm{Mn}^{2+}$	20	101±2
$Mg^{2+}$	1000	101±2	$\mathrm{Pb}^{2+}$	20	95±2
$\mathrm{Cl}^-$	1000	95±2	$Al^{3+}$	20	96±2
$\mathrm{Br}^-$	1000	96±2	$\mathrm{Fe}^{\mathrm{3+}}$	20	98±3
$\mathrm{SO_4}^{2-}$	1000	98±2	$\mathrm{Fe}^{\mathrm{3}+}$	50	52±2
$Ag^+$	20	95±2	$Fe^{3+a} \\$	300	95±2
$\mathrm{Cu}^{2+}$	20	103±3	$Cr^{3+}$	20	97±1

Results are mean ± standart deviation of three replicate analyses. <sup>a</sup>In Sodium citrate added as masking agent

### Capacity of the resin

To determine the adsorption capacity of of Dualite XAD-761 batch method was used by using Ni-PAR chelates<sup>[23]</sup>. For this process, 450 mg of the resin and Ni-PAR solution of 50 mL volume, 200 mg L<sup>-1</sup> Ni ions content and  $10^{-3}$  mol L<sup>-1</sup> PAR content were shaken at 200 rpm between 1 to 3 hours at optimum conditions. While solution was shaken the nickel ions were stripped off the resin with 5 mL of 2 mol L<sup>-1</sup> HCl solutions and determined by FAAS. It was investigated that the adsorption did not change significantly with contact time after 90 minutes. By using these parameters capacity of the XAD-761 resin has been calculated as 1.8 mg g<sup>-1</sup>. Different resins with sorption capacities and enrichment factors were given in TABLE 3.

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TABLE 3 : Comparison of adsorption capacities and enrichment factors for nickel

Solid phase	Chelating agent	LOD (µg L <sup>-1</sup> )	Adsorption Capacity (mg g <sup>-1</sup> )	Enrichmet factor	References
Ambersorb 563	1-(2-pyridylazo) 2- naphtol	0.23	-	125	[24]
Cibacron Blue F3-GA Immobilized poly(HEMA)	-	28.73	26.12	63	[25]
(DVB)-ethylene glycol dimethacrylate	quinoline-8-ol	2	-	200	[26]
Amberlite XAD-2	o-Amino phenol	7.5	3.24	65	[27]
Activated carbon	4,6-dihydroxy-2- mercaptopyrimidine	3.5	0.54	260	[19]
Amberlyst 36	-	0.86	143	100	[28]
Dualite XAD-761	PAR	0.64	1.8	120	Present work

### **Analytical performance**

For present study, the optimized experimental parameters and analytical performance of methods were given in TABLE 4. Under these experimental conditions the analytical features of the proposed method such as, linear range of calibration curve and limit of detection (LOD) were also examined. LOD for nickel was obtained from calibration curve as 0.64 µg L<sup>-1</sup>. Calculation of LOD was based on three times the standard deviation of the blank signals of ten measurements. After preconcentration of 50 mL of standard solutions buffered at pH 4.5 in different concentrations, calibration graph was obtained For this purpose, standard solutions containing Ni(II) ion in the range of 2-1000  $\mu$ g L<sup>-1</sup> were examined by the proposed procedure and it was observed that calibration curve were linear in this range. The regression equation and correlation coefficient were A  $(10^{-3}) = 7.703$ C- 0.9172, R<sup>2</sup> = 0.9983.

#### TABLE 4 : Analytical performance and optimum condition of the proposed method for determination of nickel

pH (in sodium acetate-acetic acid buffers)	4.5
Amount of PAR 0.024 % (w/v)(mL)	1
Amount of resin (mg)	450
Eluent volume (2 mol L <sup>-1</sup> HCl) (mL)	5
Temperature	Ambient
Elue flow rate (mL min <sup>-1</sup> )	4
Sample flow rate (mL min <sup>-1</sup> )	4
Maximum sample volume (mL)	600
Enrichment factor	120
Linear range (µg L <sup>-1</sup> )	2-1000
Regression equation ( $\mu g L^{-1}$ )	$A(10^{-3}) =$ 7.703C- 0.9172
Correlation coefficient (R <sup>2</sup> )	0.9983
Detection limit ( $\mu g L^{-1}$ )	0.64
Precision (R.S.D, N=7) (%)	2.8

In this equation  $A(10^{-3})$  is absorbance corresponding to concentration C (µg L<sup>-1</sup>) of Ni (II). The precision from seven repeated measurements of  $5 \,\mu g \, L^{-1}$  of nickel is excellent with RSD values of 2.8 %. These values show that, the developed method is suitable for determination of nickel. To study the validation of the development method, standard reference materials (NIST 1573 a-tomato leaves) was used for the determination of nickel. Each experiment repeated three times and the results are given in TABLE 5.

TABLE 5 : Results for certified reference material (NIST 1573 a-tomato leaves)

Element	Found <sup>a</sup>	Certified	Recovery
	(µg g <sup>-1</sup> )	(µg g <sup>-1</sup> )	(%)
Ni	$1.62\pm0.12$	$1.59{\pm}0.07$	101.8

<sup>a</sup>Mean and Standart deviation from three determinations

## **Determination of nickel in real samples**

The validity of the proposed method was further proven by analyzing spiked nickel samples. For this aim, 2.0-100.0 µg kg<sup>-1</sup> nickel solutions were spiked to sample prepared by mixing of 600 mL of water, 0.5 g dried food samples and 0.2 g guinea pig lung samples. After homogenizing the samples and applying the procedure, nickel was determined by using proposed method. TABLE 6 shows the experimental results of spiked nickel samples. The relative standard deviations were less than 10%. Calculated recoveries for waters, bean and guinea pig lung samples were found as 98.6-102.1, 98.3 and 98.9 % respectively. The levels of nickel were found as  $2.8\pm0.4$  µg TABLE 6 : The determination of nickel in different samples (Initial volume for water samples: 600 mL, bean (0.5 g) and guinea pig lung samples (0.2 g): 50 mL)

Sample	Added	Found <sup>*</sup>	Recovery %		
City line	-	$2.8\pm0.4~\mu g~L^{-1}$	-		
	$2.0 \ \mu g \ L^{-1}$	$4.9\pm0.5~\mu g~L^{-1}$	102.1		
River	-	$8.2\pm0.6 \ \mu g \ L^{-1}$	-		
	$4.0 \ \mu g \ L^{-1}$	$12.4\pm0.8 \ \mu g \ L^{-1}$	101.6		
Bean	-	186±14 μg kg <sup>-1</sup>	-		
	50 μg kg <sup>-1</sup>	232±18 μg kg <sup>-1</sup>	98.3		
Guinea Pig lung	-	954±62 μg kg <sup>-1</sup>	-		
	$200~\mu g~kg^{1}$	$1142\pm78~\mu g~kg^{-1}$	98.9		
Geothermal water	-	10.8±0.8 µg L <sup>-1</sup>			
	4.0 μg L <sup>-1</sup>	14.6±1.2 μg L <sup>-1</sup>	98.6		
*Mean $\pm$ ts/vN with 95% confidence level					

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L<sup>-1</sup>,  $8.2\pm0.6 \ \mu g \ L^{-1}$ ,  $10.8\pm0.8 \ \mu g \ L^{-1}$ ,  $186\pm14 \ \mu g \ kg^{-1}$ and  $1142\pm78 \ \mu g \ kg^{-1}$  at 95 % confidence level for city line water, river water, geothermal water, bean and guinea pig lung samples, respectively. The resin on the column can be used at least 250 times. The sufficiently good recoveries and low relative standard deviations reflect the high accuracy and precision of the proposed solid-phase extraction.

### CONCLUSION

A novel solid-phase extraction method involving PAR was proposed to determine nickel content in different samples. The proposed method has distinct advantages such as simplicity, low cost, short time of analysis, high precision and accuracy. Furthermore sophisticated instrumentation is not needed. The method developed has been successfully employed for the determination of the analytes in geological, environmental and biological Samples. The method presented is most promising for nickel ions as the preconcentration factor is 120. This method can be safely used an alternative method to determine the nickel content of different samples.

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