

## Research Article

# Spectrophotometric Determination of Iron(II) after Solid Phase Extraction of Its 2,2' Bipyridine Complex on Silica Gel-Polyethylene Glycol

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A new solid phase extraction procedure was developed for preconcentration of iron(II) using silica gel-polyethylene glycol (silica-PEG) as an adsorbent. The method is based on retention of iron(II) as 2,2' bipyridine complex on silica-PEG. The retained complex is eluted by 1.0 mol L<sup>-1</sup> of sulfuric acid-acetone mixture (1:2) and its absorbance is measured at 518 nm, spectrophotometrically. The effects of different parameters such as pH, concentration of the reagent, eluting reagent, sample volume, amount of adsorbent, and interfering ions were investigated. The calibration graph was linear in the range of 1–60 ng mL<sup>-1</sup> of iron(II). The limit of detection based on 3S<sub>b</sub> was 0.57 ng mL<sup>-1</sup> and relative standard deviations (R.S.D) for ten replicate measurements of 12 and 42 ng mL<sup>-1</sup> of iron(II) were 2.4 and 1.7%, respectively. The method was applied to the determination of of iron(II) in water, multivitamin tablet, and spinach samples.

## 1. Introduction

Nowadays, the extraction and determination of trace metal ions from different matrices have become of great importance and have received more attention. Among heavy metals, iron in small amounts is an essential element for most life on Earth, including humans and animals. It is well known that an iron deficiency is the most common cause of anemia. On the other hand, too much iron can cause several health problems. High levels of iron are associated with an increased risk for cancer, heart, and liver diseases [1]. Therefore iron determination is of great interest and is crucial to develop simple, rapid, and efficient methods for monitoring iron in the environment.

Modern instrumental methods including ICP-MS, ICP-AES, and AAS [2–8] have been used for the determination of traces of metal ions in various media. However, spectrophotometry and flame atomic absorption spectrometry (FAAS) are the most available instrumental techniques in laboratories but their sensitivity and selectivity are usually insufficient for direct determination of elements at a very low concentration levels in real samples. Therefore, a

separation/preconcentration step prior to the analysis is usually necessary for complex matrix environmental samples [7].

Solid phase extraction (SPE) has been used as a great tool for the separation and preconcentration of metal ions in different samples [8]. The important advantages of solid phase extraction such as flexibility, simplicity, being economical, high enrichment factors, low cost because of lower consumption and, being environment friendly have made SPE as an attractive technique for separation/preconcentration of heavy metal ions [9–11]. The choice of appropriate sorbent for solid phase extraction is a critical parameter in order to obtain full recovery and high enrichment factor [12]. For this reason introducing new sorbent is still a challenge for analytical chemists. Various sorption materials, such as functionalized silica or alumina [13, 14], carbon materials [15], naphthalene [16], and molecular-imprinted polymers (MIPs) [17] have been used for this purpose.

We have recently introduced silica gel-polyethylene glycol (silica-PEG) as an adsorbent for preconcentration of cobalt and nickel [18] followed by flame atomic absorption spectrometric determination. In this paper a different

aspect for using this adsorbent is presented and a new solid phase extraction method for preconcentration of trace iron(II) prior to its determination by spectrophotometry is established. Iron(II) as 2,2' bipyridine complex was passed through a column containing silica-PEG. The retained complex was then eluted by sulfuric acid-acetone mixture (1:2) and its absorbance was measured at 518 nm by a spectrophotometer.

## 2. Experimental

**2.1. Instrumentation.** A GBC model Cintra 101, UV-Visible spectrophotometer (Sidney, Australia) was used for recording absorption spectra and absorbance measurements using 1 cm glass cells. A digital pH-Meter model 632, Metrohm (Herisau, Switzerland), with a combined glass electrode, was used for pH adjustments. A Tebazma vacuum pump (Tehran, Iran) was used for pumping the solutions.

**2.2. Reagents.** Analytical reagent-grade chemicals were used. A stock solution of  $1000 \mu\text{g mL}^{-1}$  Fe(II) was prepared by dissolving 0.7020 g of  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (Merck, Darmstadt, Germany) in water and diluting to 100 mL in a volumetric flask.  $6.4 \times 10^{-4} \text{ mol L}^{-1}$  of 2,2' bipyridine (Merck) was prepared by dissolving 0.01 g in water and diluting to 100 mL in a volumetric flask. A phosphate buffer pH 8 was prepared by diluting 4.8 mL phosphoric acid (Merck) to 250 mL in a volumetric flask and then adjusting its pH to 8 by adding 1.0 mol  $\text{L}^{-1}$  NaOH (Merck) and using a pH meter. Silica gel (Aldrich, USA, 70–270 mesh, 60 Å, surface area  $> 500 \text{ m}^2/\text{g}$ ) was activated by treatment with 5 mom  $\text{L}^{-1}$  HCl (Merck) and dried in vacuum at 120°C. PEG was heated at 80°C under vacuum for 30 min before use to remove traces of moisture.

**2.3. Adsorbent Preparation by Immobilization of Polyethylene Glycol on Silica Gel.** An oven dried 250 mL flask equipped with a magnetic stirrer was charged with dried activated silica gel (20 g) under nitrogen atmosphere. Then freshly distilled  $\text{SOCl}_2$  (45 mL) was added slowly to the flask through an addition funnel and the reaction mixture was stirred at room temperature. Evolution of copious amounts of HCl and  $\text{SO}_2$  occurred instantaneously. After stirring for 4 h, the excess unreacted thionyl chloride was distilled off and the resulting grayish silica chloride was flame dried and stored in airtight container before use.

To a well-stirred silica chloride (20 g) in dry  $\text{CH}_2\text{Cl}_2$  (40 mL) PEG (10 g) was added dropwise under nitrogen atmosphere and at room temperature. HCl was instantaneously evaluated. After stirring for 2 h, the obtained silica-gel-supported PEG was removed by filtration. For elimination of any additional PEG, the PTC was washed several times by acetone (3–30 mL). Silica-gel-supported PEG was dried in a vacuum oven overnight [19] and was used as an adsorbent in this work.

**2.4. Recommended Procedure.** 0.1 g of silica-PEG adsorbent was placed in a glass tube (7 cm length and 7 mm i.d.) with

a very fine bore and used as a preconcentration column. 250 mL of the solution containing 1–60 ng  $\text{mL}^{-1}$  of iron(II), 2 mL of  $6.4 \times 10^{-4} \text{ mol L}^{-1}$  2,2' bipyridine, and 5 mL of phosphate buffer pH 8 was passed through the mini column at a flow rate of 10  $\text{mL min}^{-1}$ . The adsorbed iron complex was eluted with 2.0 mL of 1.0 mol  $\text{L}^{-1}$  of sulfuric acid-acetone mixture (1:2). The absorbance of the color complex was measured at 518 nm. A blank solution was also run under the same analytical conditions without adding any Fe(II).

**2.5. Sample Pretreatments.** The tap water sample was collected, acidified, boiled, and filtered. An aliquot of water sample was then treated under recommended procedure.

Fresh spinach sample was purchased from local market Ahvaz, Iran. It was washed thoroughly with water and dried in an oven at 100°C for 7 h. It was then grinded to produce a fine powder. 12 g of this powder was placed in a porcelain crucible and burned on a flame until there was no fume. It was then placed in a muffle furnace at 600°C for 5 h; 2 mL of hydrochloric acid was added and boiled for 30 min. It was then filtered and diluted to 50 mL in a volumetric flask. An aliquot of this solution was taken and treated under the recommended procedure.

The content of a multivitamin tablet was dissolved in water; 5 mL of hydrochloric acid 10% was added, filtered, and diluted to 250 mL in a volumetric flask. An aliquot of this solution was taken and treated under the recommended procedure.

## 3. Results and Discussion

The PEG-grafted silica particles are special type of branched inorganic–organic copolymer which has polyether functional groups in the side chains that have been found to be stable to acid, base, high temperature, and oxygen. Its FT-IR spectrum shows the presence of OH and ethylene oxide groups on the surface of the adsorbent. Our preliminary experiments showed that iron(II) as 2,2' bipyridine complex is retained by silica-PEG. The 2,2' bipyridine complex is probably adsorbed through interaction with OH groups of the PEG. Since the amounts of the complex retained on the adsorbent is very small, the FT-IR spectrum after loading the complex shows very slight shifts.

The absorption spectra of eluted complex from the column by sulfuric acid-acetone mixture (Figure 1) showed that maximum absorption takes place at 518 nm. Therefore absorbance measurements were conducted at this wavelength. In order to achieve the highest performance for the system, the effect of different parameters such as pH, concentration of the reagent, type and concentration of eluent flow, rates and amount of adsorbent on the solid phase extraction were studied.

**3.1. The Effect of pH.** It has been mentioned in the literature that Fe(NN) chelates such as iron(II)-2,2' bipyridine complex are stable in the pH range of 3–10 [20]. Therefore, the influence of the pH of the sample solution on the iron(II)

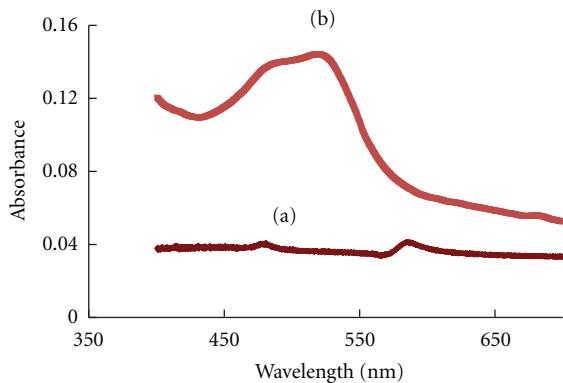


FIGURE 1: Absorption spectra of (a) blank and (b) the eluted iron(II) as 2,2' bipyridine complex from the column.

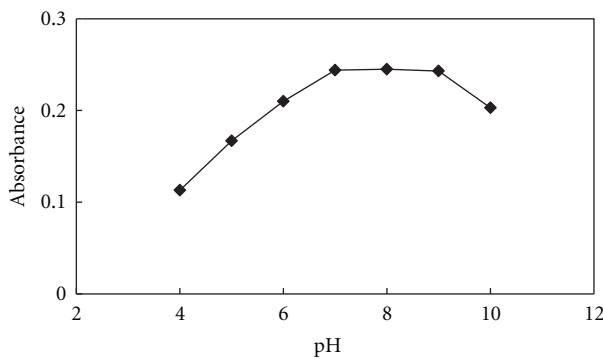


FIGURE 2: Effect of pH on the absorbance of 50 ng mL<sup>-1</sup> iron(II) as 2,2' bipyridine complex after desorption from the column.

ions as 2,2' bipyridine complex formation and hence its adsorption on the silica-PEG adsorbent were studied. For this purpose, the pH value of a set of solutions (100 mL) each containing 50 ng mL<sup>-1</sup> of iron(II) was adjusted in the range of 4.0–10.0 by adding 0.1 mol L<sup>-1</sup> of HCl or NaOH. The obtained solutions were passed through the column, eluted by sulfuric acid-acetone mixture (1 : 2), and the absorbance of the color complex was measured at 518 nm. According to the results shown in Figure 2, the absorbance is constant over the pH range of 7.0–9.0. Therefore, pH 8 was selected as optimum for further work and 5 mL of phosphate buffer pH 8 was added to 100 mL solutions to maintain this pH.

**3.2. Effect of the Ligand Concentration.** The effect of 2,2' bipyridine concentrations on the absorbance of iron(II)-2,2' bipyridine complex eluted from the column was investigated. The results indicated that the absorbance was increased up to a ligand concentration of  $0.96 \times 10^{-5}$  mol L<sup>-1</sup> and became almost constant above that (Figure 3). For this reason, the present study was carried out with a ligand final concentration of  $1.28 \times 10^{-5}$  mol L<sup>-1</sup> as optimum value. Thus 2.0 mL of  $6.40 \times 10^{-4}$  mol L<sup>-1</sup> of 2,2' bipyridine was added to 100 mL solutions to maintain this concentration.

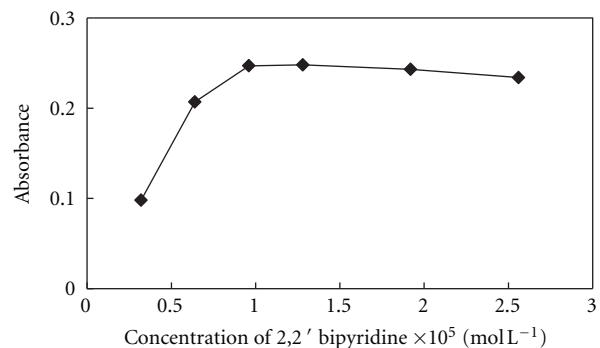


FIGURE 3: Effect of 2,2' bipyridine concentrations on the absorbance of 50 ng mL<sup>-1</sup> iron(II) as 2,2' bipyridine complex after desorption from the column.

TABLE 1: Effect of different eluents on the absorbance of eluted Fe(II)-2,2' bipyridine complex.

Eluent	Absorbance
Acetone	0.05
DMSO	0.04
H <sub>2</sub> SO <sub>4</sub> (1 mol L <sup>-1</sup> )	0.06
HCl (1 mol L <sup>-1</sup> )	0.03
HNO <sub>3</sub> (1 mol L <sup>-1</sup> )	0.04
Acetonitrile-H <sub>2</sub> SO <sub>4</sub> (1 mol L <sup>-1</sup> )	0.27
Acetone-H <sub>2</sub> SO <sub>4</sub> (1 mol L <sup>-1</sup> )	0.78
Acetonitrile-HCl (1 mol L <sup>-1</sup> )	0.18

**3.3. Choice of Eluent.** The other important factors which could affect the solid phase extraction procedure are the type, volume, and concentration of the eluent used for desorption of metal complex from the adsorbent. In order to choose a proper eluent for the retained complex after its extraction, the analyte was eluted with a series of selected eluents such as various inorganic acids, organic solvents, and their mixtures. The results of this investigation presented in Table 1 show that among different eluents used sulfuric acid-acetone mixture provided the highest absorbance for the eluted complex. Therefore, this solution was chosen as an eluent for further investigations. The sulfuric acid concentration and H<sub>2</sub>SO<sub>4</sub>-acetone ratio were also optimized. According to the obtained results H<sub>2</sub>SO<sub>4</sub> concentration of 1.0 mol L<sup>-1</sup> with a ratio of 1 : 2 (H<sub>2</sub>SO<sub>4</sub>-acetone) was most suitable solvent for eluting the complex from the column. The effect of eluent volume was also investigated and 2 mL of 1 : 2 (H<sub>2</sub>SO<sub>4</sub>-acetone) was found to be adequate for the elution of retained iron(II)-2,2' bipyridine complex from the column.

**3.4. The Effect of the Mass of Adsorbent.** In order to test the effect of the mass of adsorbent on quantitative retention of iron(II)-2,2' bipyridine complex different amounts of silica-PEG adsorbent were chosen and the experimental method was applied. The results showed that quantitative adsorption for the complex was obtained in the range of 0.05–0.25 g of adsorbent. Quantitative adsorption was not obtained when

the mass of extractant was smaller than 0.05 g. Thus 0.10 g of silica-PEG was selected for further studies.

**3.5. The Effect of Flow Rate.** In a solid phase extraction procedure, the flow rate of sample solution not only affects the recoveries of the analytes, but also controls the analysis time. Therefore, the effect of sample solution flow rate was examined under the optimum conditions by passing 100 mL of sample solution through the minicolumn at different flow rates. It was found that the retention of iron(II)-2,2' bipyridine complex was independent of flow rate up to 10 mL min<sup>-1</sup> and the absorbance of the solution was decreased slightly when the flow rate was over 10 mL min<sup>-1</sup>. Therefore, a flow rate of 10 mL min<sup>-1</sup> was employed in this work.

**3.6. The Effect of Breakthrough Volume.** In order to achieve a high preconcentration factor, the breakthrough volume of sample solution should be established. The effect of sample volume on the adsorption of iron(II)-2,2' bipyridine complex was studied in the range of 100–500 mL. Each solution contained same amount of Fe<sup>2+</sup> (5 µg) and the adsorption and desorption processes were performed under the optimum conditions. The results showed that the Fe-2,2' bipyridine present in various volumes of solution was completely and quantitatively adsorbed on silica-PEG. The adsorption decreased at higher volumes. Therefore, for determination of trace quantities of Fe<sup>2+</sup> in samples, a sample volume up to 500 mL could be selected in order to increase the preconcentration factor to 250. However, in order to decrease the analysis time we used 250 mL for the preparation of calibration curves for which a preconcentration factor of 125 was employed.

**3.7. Analytical Performance.** The analytical characteristics of the developed method such as the limit of detection, reproducibility, linear range, and correlation coefficient were obtained by processing standard solutions under optimum conditions. A linear calibration graph was obtained for the determination of iron(II) under the proposed experimental conditions. The data yielded a good linearity in the range of 1–60 ng mL<sup>-1</sup> with the equation of  $A = 0.016C + 0.029(r = 0.9989)$  where  $A$  is the absorbance and  $C$  is the concentration of iron(II) in ng mL<sup>-1</sup>. The limit of detection, defined as the concentration of the analyte giving signals equivalent to three times the blank standard deviation, was 0.57 ng mL<sup>-1</sup> and relative standard deviations (R.S.D) for ten replicate measurements of 12 and 42 ng mL<sup>-1</sup> of iron(II) were 2.4 and 1.7%, respectively.

**3.8. Interference Studies.** In order to investigate the selectivity of the solid phase extraction procedure, the influence of different ions which could interfere with the determination of iron(II) in various environmental samples was studied. For this purpose constant amounts of iron(II) were taken with different amounts of diverse ions and the general procedure was followed. Any deviation of ±5% or more from the

TABLE 2: The effect of different ions on the determination of 50 ng mL<sup>-1</sup> of iron(II).

Foreign ions	Tolerance ratio [ion]/[Fe <sup>2+</sup> ]
K <sup>+</sup> , NO <sub>3</sub> <sup>-</sup> , SCN <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , Br <sup>-</sup> , Mg <sup>2+</sup> , Cl <sup>-</sup>	1000
Pb <sup>2+</sup> , I <sup>-</sup> , Ca <sup>2+</sup> , F <sup>-</sup>	500
Cd <sup>2+</sup> , Ba <sup>2+</sup> , Cr <sup>3+</sup> , Th(IV)	100
Ni <sup>2+</sup> , Hg <sup>2+</sup>	10
Al <sup>3+</sup> , Cu <sup>2+</sup> , Co <sup>2+</sup>	5

TABLE 3: Determination of iron (II) in different samples.

Sample	Added (ng mL <sup>-1</sup> )	Found <sup>a</sup> (ng mL <sup>-1</sup> )	Recovery (%)
Tap water	—	ND	—
	4	3.9 ± 0.5	97.5
	8	7.8 ± 1.0	97.5
Multivitamin <sup>b</sup>	—	7.6 ± 1.0	—
	10	17.6 ± 1.0	100.0
Spinach <sup>c</sup>	20	27.5 ± 1.2	99.5
	—	39.0 ± 0.8	—
	10	48.2 ± 1.3	97.9

<sup>a</sup>  $x \pm ts/\sqrt{n}$  at 95 % confidence ( $n = 5$ ).

<sup>b</sup> Amount of iron per tablet was 9.7 mg.

<sup>c</sup> Amount of iron was 40.6 µg g<sup>-1</sup> of spinach.

ND: not detected.

absorbance value of the standard solution was selected as interference. Results given in Table 2 show that the presence of major cations and anions has no significant effect on the determination of iron(II) under selected conditions.

## 4. Applications

The developed method was applied to the determination of iron in tap water, multivitamin tablet, and spinach samples, with satisfactory results (Table 3). The concentration of iron(II) could not be determined in water due to its very low concentration. There was a good agreement between the added and the recovered concentrations of the analyte. The obtained amount of iron per tablet was 9.7 mg and the reported value was 10.0 mg which shows that there is good agreement between the two results.

## 5. Conclusion

Silica-PEG is a promising adsorbent which could be used in different ways and this paper presents one of them. The method presented in this paper is highly sensitive and selective for the determination of trace amounts of Fe(II) by spectrophotometry. The detection limits achieved are better than or comparable to some of the previously reported works. A comparison of the proposed method with some of the methods reported in literature is given in Table 4. Moreover, the adsorbent is ecofriendly and stable for at least six month.

TABLE 4: Comparison of the proposed method with some of the methods reported in literature.

System	PF <sup>a</sup>	DL (ng mL <sup>-1</sup> )	Breakthrough volume (mL)	Ref.
Amberite XAD-2000	50	0.32	250	[1]
Modified activated carbon	10	0.38	100	[2]
Flotation method	93	0.70	750	[3]
BHAPN/SDS-alumina	63	2.60	500	[21]
Naphthalene loaded with tetraoctylammonium bromide	36	12.00	180	[16]
Silica-PEG	250	0.57	500	This work

<sup>a</sup> Preconcentration factor.

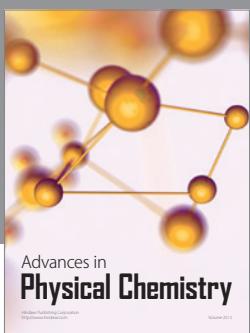
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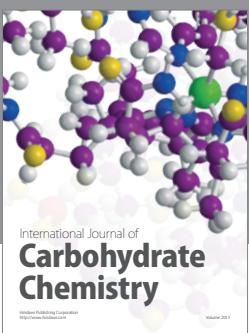
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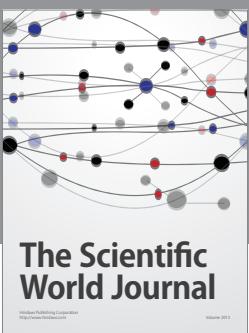
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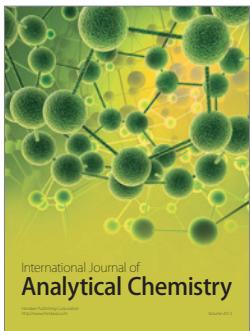
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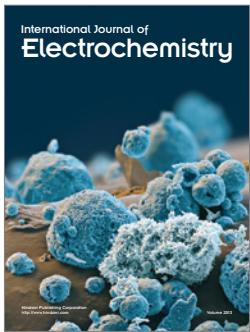
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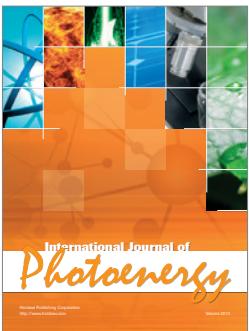
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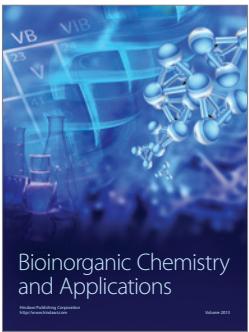
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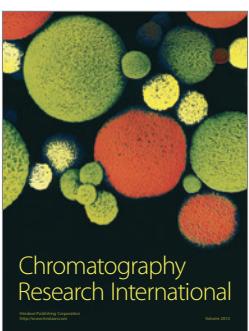
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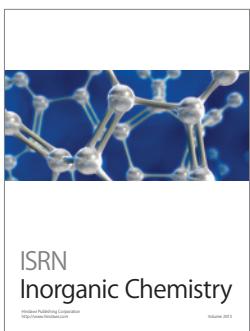
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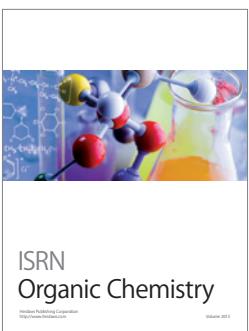
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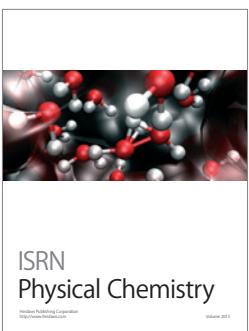
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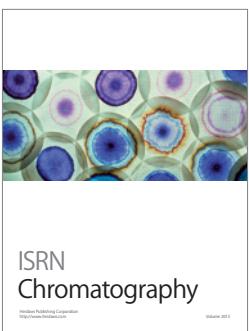
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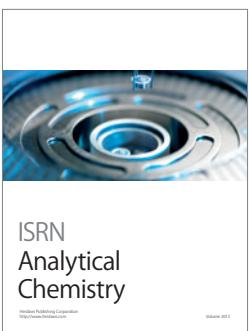
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