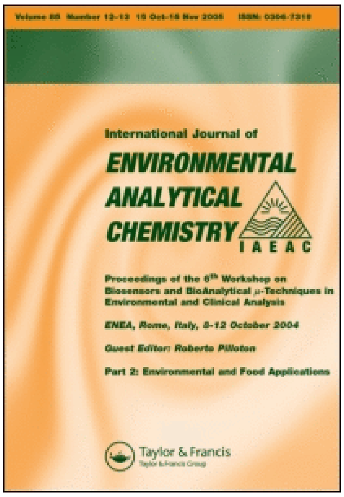

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Flame Atomic Absorption Spectrometric Determination of Cu(II), Co(II), Cd(II), Fe(III) and Mn(II) in Ammonium Salts and Industrial Fertilizers after PrEconcentration/Separation on Diaion HP-20

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FLAME ATOMIC ABSORPTION SPECTROMETRIC DETERMINATION OF Cu(II), Co(II), Cd(II), Fe(III) AND Mn(II) IN AMMONIUM SALTS AND INDUSTRIAL FERTILIZERS AFTER PRECONCENTRATION/SEPARATION ON DIAION HP-20

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A sensitive and simple separation-enrichment technique for the determination of trace amounts of Cu(II), Co(II), Cd(II), Fe(III) and Mn(II) was described. Metal ions were complexed with 1-nitroso-2-naphthol at pH 9. Following solid-phase extraction on Diaion HP-20 resin, metals were determined by flame atomic absorption spectrometry. The effect of the matrix ions were investigated. The recoveries of metal ions were greater than 95%. The detection limits of the analyte ions ($k = 3$, $N = 21$) were varying 0.18 µg/l for Cd(II) to 0.44 µg/l for Fe(III). The method was applied to a stream sediment standard reference material (GBW7309), some ammonium salts and industrial fertilizer samples for the determination of copper, cobalt, cadmium, iron and manganese. The relative standard deviations (RSD) of the determinations for analyte ions at µg/g levels varied from 1 to 10%.

Keywords: Diaion HP-20; FAAS; Preconcentration; Ammonium salts; Industrial fertilizers; Heavy metals; Solid phase extraction

INTRODUCTION

The demand for ultra pure chemicals such as alkaline, earth alkaline and ammonium salts by various high technology industries has increased over the past years. Sensitive and reliable techniques for the determination of metal impurities in the chemicals and other materials are required. In order to detect very low levels of analytes, the determination of metal ions requires powerful analytical techniques including flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS), inductively coupled plasma mass spectroscopy (ICP-MS), inductively coupled plasma atomic

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emission spectrometry (ICP-AES). The effects of the matrix is an important problem for the determination of metal ions by these techniques [1,2]. In order to solve this problem, a lot of enrichment-separation techniques including liquid–liquid extraction [3,4], coprecipitation [5–7], electroanalytical techniques [8], membrane filtration [9], ion exchange [10], cloud point extraction [11–13], solid-phase extraction [14–16] have been proposed for the determination of metals in various materials such as natural waters, geological samples, salts and industrial materials, etc.

Solid-phase extraction (SPE) techniques for the preconcentration of the traces of heavy metal ions have become increasingly popular, compared with the traditional solvent extraction methods. Solid-phase extraction has some advantages over the solvent extraction such as a simple and fast extraction system, easy adaptation for preconcentration and determination of trace metal ions by flow injection analysis technique. It has a relatively high concentration factor and the ability of treating large volume samples free from contamination [1,2].

Efficient sorbents include activated carbon [17–19], microcrystalline naphthalene [20], Amberlite XAD resins [21–25] and other sorbents [26,27]. Main properties of the solid-phases used in the SPE techniques are high surface area, their high purity and good sorption properties including porosity, durability and uniform pore distribution. Diaion HP-20 is a polystyrene divinylbenzene copolymer and has also these properties.

1-Nitroso-2-naphtol has been used for the determination of metal ions of UV–Vis spectrophotometry. The metal-1-nitroso-2-naphthol complexes have also been used in the solvent extracted procedures for the preconcentration of metal ions [28–33].

In the present work, a simple and economic separation/preconcentration procedure has been established for the determination of Cu(II), Co(II), Cd(II), Fe(III) and Mn(II) ions in some ammonium salts, some industrial fertilizers and a reference stream sediment material (GBW07309).

EXPERIMENTAL

Apparatus

The instrumental detection system used was a Perkin–Elmer Model 3110 atomic absorption spectrometer. Atomic absorption measurements for model working were carried out in the air/acetylene flame. The operating parameters and other conditions for working elements which were given in Table I, were set as recommended by the manufacturer.

TABLE I Conditions for flame atomic absorption spectrometric determinations

Element	*Wavelength* (nm)	*Slit* (nm)	*Lamp current* (mA)	*Flow rate of flame gases*	
				Air (l/min)	*Acetylene* (l/min)
Cu	324.8	0.7	25	9.5	2.3
Fe	248.3	0.2	30	9.5	2.3
Mn	279.5	0.2	20	9.5	2.3
Co	240.7	0.2	30	9.5	2.2
Cd	228.8	0.7	12	9.5	2.2

A pH meter, Nel pH-900 Model glass-electrode was employed for measuring pH values in the aqueous phase. The glass column containing 700 mg of Diaion HP-20 resin has 10 cm length and 1 cm inner diameter.

Reagents and Solutions

All chemicals were reagent-grade and all solutions were prepared in distilled–deionised water. Stock solutions were prepared from appropriate amounts of respective nitrates as 1000 mg/l, solutions in 0.01 M HNO_3 and further diluted daily for obtaining reference and working solutions prior to use. A 1.0×10^{-2} M of 1-nitroso-2-naphthol (NN) (Merck no: 6803) solution was prepared by dissolving the requisite amounts of 1-nitroso-2-naphthol in a water/methanol (75/25, v/v) mixture immediately before use.

Phosphate buffer solution was prepared by mixing of appropriate volumes of 1 M phosphoric acid (Merck, Darmstadt) and 1 M sodium dihydrogen–phosphate solutions (Merck, Darmstadt) for pH 2. Acetate buffer solutions were prepared by mixing of appropriate volumes of 1 M acetic acid (Merck, Darmstadt) and 1 M sodium acetate solutions (Merck, Darmstadt) for pH 4 and 6. For pH 7, a 250 ml of borate buffer was prepared by use of 2.5 ml of 1 M HCl and 0.445 g $NaBO_2$ (Merck, Darmstadt). Ammonium chloride buffer solutions were prepared by mixing of appropriate amounts of 0.1 M ammonia (Merck, Darmstadt) and 0.1 M ammonium chloride solutions for pH 8–10. pH 11–13 were obtained with 1 M NaOH.

Diaion HP-20 Resin was purchased from Supelco, Bellefonte, U.S. It was (40–60 mesh fraction, surface area: 600 m^2/g) purified by washing with methanol, water 1 M nitric acid in acetone, water, 1 M NaOH, water and acetone sequentially, in order to eliminate trace metal ions and other inorganic and organic contaminants in the resin.

Preconcentration Procedure

The column method was tested with model solution. Forty to fifty millilitre of solution containing 5–20 µg of the metal ions was added 10 ml of buffer solution (desired pH between 2 and 10) and 1-nitroso-2-naphthol solution. The Diaion HP-20 column was preconditioned by passing buffer solution. The metal/1-nitroso-2-naphthol solution was passed the column at a flow rate of 5 ml/min. The sample solution was permitted to flow through the column under gravity. After passing of this solution completely, the column was rinsed with twice 10 ml of water. The absorbed metal chelates on the column were eluted with 8–10 ml portion of 1 M HNO_3 in acetone. The eluent was evaporated over a hot plate to near dryness. The residue was diluted to 2–5 ml with 1.0 M HNO_3. The eluent was analysed for the determinations of metal concentrations by flame atomic absorption spectrometer.

Pretreatment of Stream Sediment SRM Sample

A 50–60 mg amounts of a stream sediment standard reference material (GBW7309) sample was decomposed with 15 ml of aqua regia and the solution was evaporated to dryness. This process was repeated twice. Ten millilitre of distilled water was added to the residue. The suspension was filtered through a blue band filter paper, and the insoluble part was washed with distilled water. Then, the preconcentration procedure given above was applied to the final solutions. The same procedure was applied to the blank solution.

Application to Real Samples

For the determination of trace metals in ammonium salts and fertilizers. Four grams of salt or fertilizer sample was dissolved in 3 ml of 0.1% HNO_3 and diluted to 25.0 ml with distilled water. The preconcentration and analytical procedures given above were applied to these solutions.

RESULTS AND DISCUSSION

Parameters such as pH of the buffer solution, eluent solution concentration, eluent solution volume, sample flow rate were investigated. This was done to achieve best efficiency concerning sorption and elution, which should result in increased sensibility, as well as high analytical throughput. The amounts of each metal in the model solution was 5–20 μg.

Effect of pH on Sorption

The pH value is one of the most important factors influencing the adsorption behaviour of the heavy metal ions on the Diaion HP-20 resin. The sorption of metal ions on Diaion HP-20 adsorption resin was studied at different pH. The results are given in Fig. 1. The quantitative recoveries (>95%) for copper, manganese, iron, cadmium and cobalt were found at the pH ranges 8–13, 9–10, 6–13, 9–12 and 8–13, respectively. All subsequent studies were carried out at pH 9.

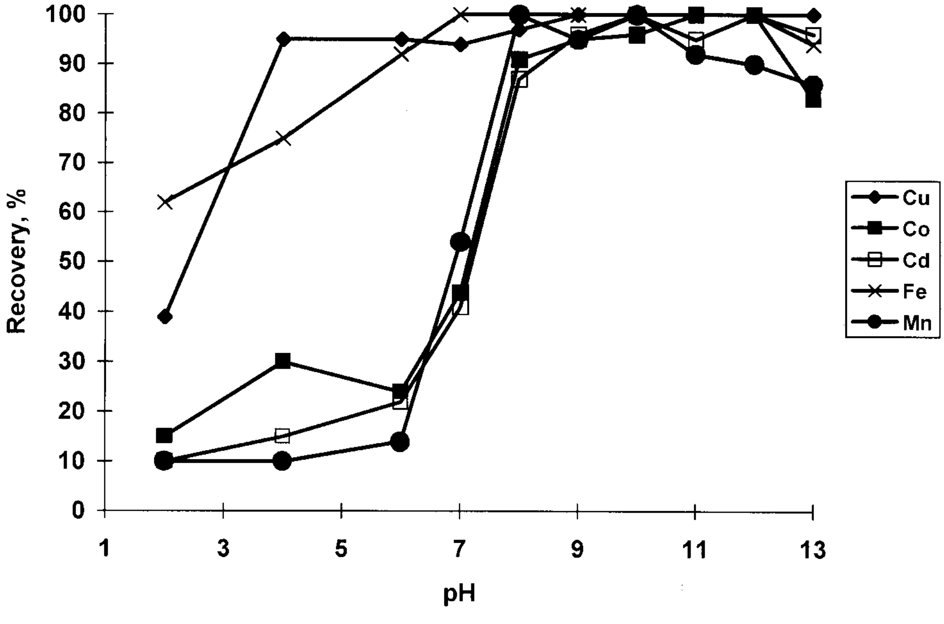

FIGURE 1 Influences of pH on the retention of metal ions on Diaion HP-20 ($N=3$, Ligand: NN, Eluent: 1 M HNO_3 in acetone).

Effect of Amount of 1-Nitroso-2-naphthol

The effects of NN amounts on the sorption of metal ions were investigated. The results are given in Fig. 2. Quantitative recovery values ($> 95\%$) of the investigated metals were obtained by the addition of 1.0–2.0 ml of a 1.0×10^{-2} M of 1-nitroso-2-naphthol solution. The addition of 2.0 ml of 1-nitroso-2-naphthol solution is recommended.

Effect of Sample Volume

The influence of the sample volume on the recovery of the metal ions were also investigated. The metal amounts were 5–20 µg and in the model solution, the metal amounts held constant while increasing the sample volume. The effect of sample volume on the sorption of metal ions were also investigated by passing 25–1000 ml volumes through the Diaion HP-20 column. The results are depicted in Fig. 3. Except cadmium, the adsorption of the metal ions with 700 mg resin was not affected by sample volume till 1000 ml. The quantitative recoveries for cadmium were obtained in the range of 25–250 ml of sample volume.

Influences of the Resin Amount

The effect of amount of resin on the retention of the analyte ions on the HP-20 column were investigated in the range of 300–900 mg. The quantitative recovery values were obtained in the range of 400–750 mg of Diaion HP-20. After 750 mg of resin, the recoveries were not quantitative. All further studies 700 mg resin was used.

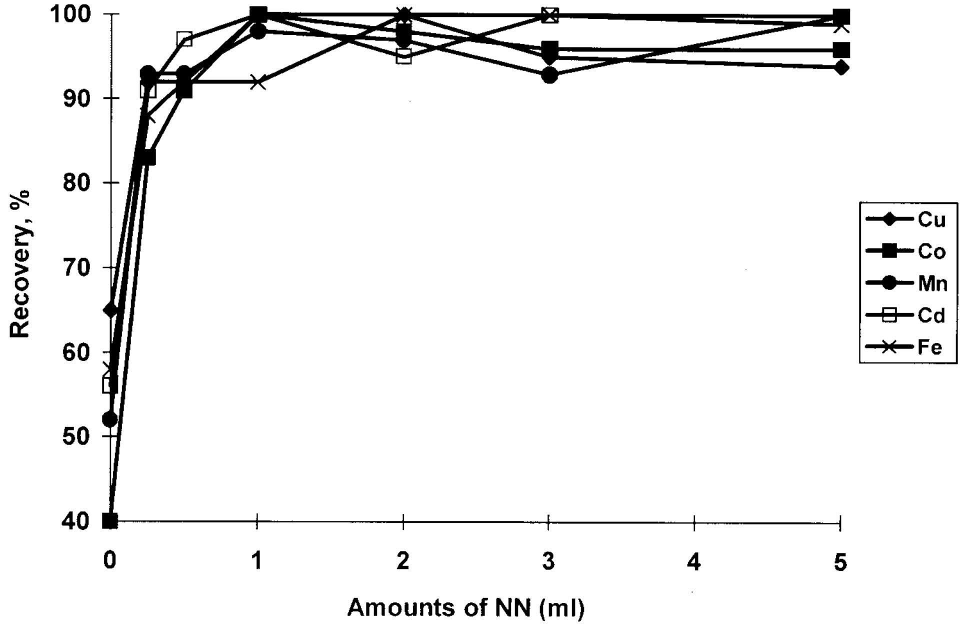

FIGURE 2 Effects of the amount of 1-nitroso-2-naphthol on the recovery of analyte ions (N: 4, Eluent: 1 M HNO_3 in acetone, eluent volume: 10 ml).

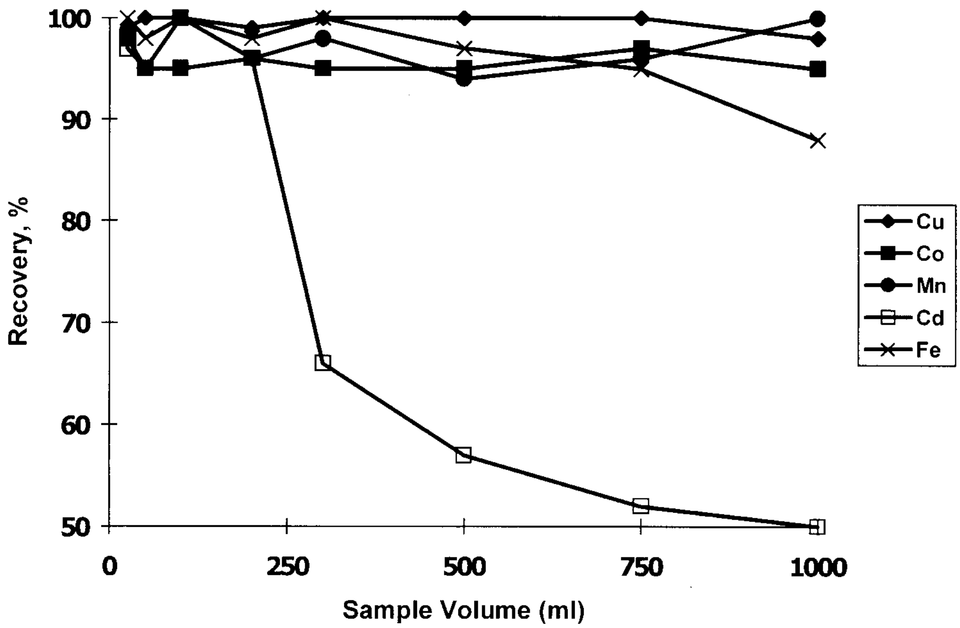

FIGURE 3 Effects of sample volume on the recovery of Cu, Co, Mn, Cd and Fe (*N*: 3, Eluent: 1 M HNO_3 in acetone, eluent volume: 10 ml).

A bead size of resin 40–60 mesh was selected for the preconcentration procedure in Diaion HP-20 column. Smaller resin particles could have improved retention capacity, but the flow rates of sample solution and eluent solution ought to have been reduced, with subsequent increase in preconcentration time.

Influence of Flow Rate on the Adsorption

The influence of flow rate on the adsorption of trace metals ions was studied. Flow rate variations in the range of 1–25 ml/min had no significant effect on the recoveries. All subsequent experiments were performed at 5 ml/min flow rate.

Desorption of the Metal Ions

The possibility of the quantitative recoveries of the analyte ions from the HP-20 column by various eluent were also investigated. The results are given in Table II. Quantitative recoveries were obtained for all investigated metal ions by using 1 M HNO_3 in acetone, 2 M HNO_3 in acetone and 1 M HCl in acetone as eluent solution. These results found present work agree with the data given in our papers [34,35]. As can be seen in Table II, also selective elution of metal ions were possible with some eluents. For example with 2 M HNO_3, only iron was recovered quantitatively (Table III).

The effects of the eluent volume were also studied by using 1 M HNO_3 as eluent. As can be seen in Table II, quantitative recovery values were obtained for all analyte ions in the range of 5–25 ml of eluent volume. All further studies 10 ml of 1 M HNO_3 was used as eluent.

TABLE II Effect of the various eluents on the recoveries of Cu, Co, Mn, Cd and Fe (Eluent Volume: 10 ml, $N=4$)

Eluent	*Recovery, %*				
	Cu	*Co*	*Mn*	*Cd*	*Fe*
1 M NH_3	10	11	14	11	< 5
1 M NaOH	27	35	28	46	28
2 M NH_3	6	< 5	28	6	< 5
1 M HNO_3	82	15	94	83	100
2 M HNO_3	70	35	78	71	96
1 M HCl	42	< 5	19	10	21
2 M HCl	58	< 5	16	10	25
Acetone	100	23	91	92	52
1 M HNO_3 in acetone	95	97	98	98	100
2 M HNO_3 in acetone	92	94	98	94	100
1 M HCl in acetone	100	50	100	100	98
2 M HCl in acetone	73	54	92	75	100

TABLE III Influences of the eluent volume on the recoveries ($N=4$, Eluent: 1 M HNO_3 in acetone)

Volume of eluent (ml)	*Recovery, %*				
	Cu	*Co*	*Cd*	*Fe*	*Mn*
5	96	96	100	96	100
10	97	100	98	100	98
25	100	100	96	100	93

Matrix Effects

The influence of the some alkaline, earth alkaline and ammonium ions on the retentions of analyte ions were also investigated. For this purpose, solutions of these compounds were prepared, then 5–10 µg of each analyte ions added to these solutions, then preconcentration procedure given above was applied. The results are shown in Table IV. The recoveries of metal ions from the column were not influenced from these compounds, because alkaline and earth alkaline ions and ammonia are not complexed with 1-nitroso-2-naphthol. Thus alkaline and earth alkaline ions and ammonia are not retained on the column prior to elution of investigated ions and can be easily washed out through the column.

Accuracy of the Method

The correctness of results was verified by analyzing the concentration after addition of known amounts of analytes into analytical reagent grade of ammonium nitrate from Merck–Darmstadt. As it is seen from Table V, good agreement was obtained between the added and found analyte content using the recommended procedure. The recovery values calculated for the standard additions were always higher than 95%, thus confirming the accuracy of the procedure and its independence from matrix effects.

The detection limits for analyte ions were calculated as the amounts of the analytes required to yield a met peak equal to three times the standard deviation of the background signal. The detection limit values for Cu(II), Co(II), Cd(II), Mn(II) and Fe(III) obtained for the preconcentration of 200 ml of sample solution were 0.29,

TABLE IV Effects of the matrix ions on the recovery of the investigated metal ions (concentration of Cu, Co, Fe and Mn: 0.2 µg/ml, Cd concentration: 0.1 µg/ml in model solutions $N=4$, Ligand: NN)

Ion	mg/l	*Added as*	*Recovery, %*				
			Cu	*Co*	*Cd*	*Fe*	*Mn*
NH_4^+	10,000	$(NH_4)_2SO_4$	100	96	97	100	100
	25,000		97	100	98	98	99
	50,000		98	100	96	98	100
Na^+	5000	NaCl	100	96	94	100	100
	10,000		100	94	99	100	100
	20,000		100	100	64	100	100
Mg^{2+}	500	$MgCl_2$	100	96	99	100	100
	1000		100	94	92	100	100
	2000		100	88	41	98	99
K^+	500	KCl	96	98	96	100	97
	1000		100	97	100	100	100
	2000		100	96	84	96	98
Ca^{2+}	500	$CaCl_2$	100	97	100	100	100
	1000		95	96	94	96	100
	2000		100	99	91	100	98
Cl^-	2500	NaCl	97	98	100	96	96
	5000		99	98	99	100	100
	10,000		99	97	97	100	100
$(SO_4)^{2-}$	10,000	$(NH_4)_2SO_4$	100	100	98	100	100
	25,000		97	100	100	100	99
	50,000		98	89	96	96	100

TABLE V Recovery of analyte ions in analytical reagent grade of ammonium nitrate from Merck–Darmstadt ($N=4$, volume: 50 ml)

Element	*Added,* µg	*Found,* µg	*Recovery, %*
Cu	0	N.D.	–
	5.0	4.9 ± 0.1	98 ± 2
	10.0	10.1 ± 0.4	104 ± 4
Co	0	N.D.	–
	5.0	4.8 ± 0.1	96 ± 2
	10.0	9.9 ± 0.2	99 ± 2
Mn	0	N.D.	–
	5.0	5.1 ± 0.1	102 ± 2
	10.0	10.0 ± 0.2	100 ± 2
Cd	0	N.D.	–
	2.5	2.45 ± 0.05	98 ± 2
	5.0	4.95 ± 0.05	99 ± 1
Fe	0	N.D.	–
	5.0	4.9 ± 0.1	98 ± 2
	10.0	10.0 ± 0.3	100 ± 3

0.36, 0.18, 0.26 and 0.44 µg/l ($N=21$), respectively. Better detection limits are to be expected with larger sample volumes.

Analysis of Standard Reference Material (SRM)

The developed method was applied to a stream sediment standard reference material (GBW7309) for the determination of Cu(II), Co(II) and Cd(II). The results, based on

TABLE VI Results of the analysis a stream sediment standard reference material (GBW7309)

Element	*Certified value* (μg/g)	*Our value* (μg/g)	(s/x)
Cu	32.1	31.3 ± 0.7	0.022
Co	14.0	14.7 ± 0.2	0.013
Cd	0.26	0.31 ± 0.02	0.064

TABLE VII Concentration of investigated metal ions in the samples ($N = 5$, final volume: 2 ml)

Sample	*Concentration* (μg/g)*				
	Cu	*Co*	*Cd*	*Fe*	*Mn*
Ammonium chloride (Technical Grade)	3.41 ± 0.23	BDL	BDL	4.54 ± 0.10	BDL
Ammonium nitrate (Technical Grade)	0.42 ± 0.12	BDL	0.49 ± 0.02	3.30 ± 0.17	0.38 ± 0.03
Fertilizer 1 (Sodasan)	0.99 ± 0.10	2.65 ± 0.10	0.45 ± 0.02	3.36 ± 0.11	0.44 ± 0.02
Fertilizer 2 (DAP1)	3.32 ± 0.15	2.84 ± 0.25	BDL	5.35 ± 0.31	0.66 ± 0.04
Fertilizer 3 (Etisod)	0.63 ± 0.05	1.45 ± 0.02	0.32 ± 0.01	5.00 ± 0.10	0.89 ± 0.04

*$x \pm t.s./\sqrt{N}$, p: 0.95, BDL: below detection limit.

the average of four replicates, are shown in Table VI, where the results obtained are in good agreement with the certified values.

Analysis of Real Samples

The recommended procedure as described in the Experimental Section was applied to determine Cu, Co, Mn, Cd and Fe in technical grade ammonium salts and industrial fertilizers produced in Turkey. The obtained results are given in Table VII. The relative standard deviations of the separation/preconcentration procedure for analyte ions at μg/g levels for flame atomic absorption spectrometric measurements are less than 0.100.

CONCLUSION

The proposed methodology is very simple, efficient, precise and accurate since results obtained with the analysis of the certified samples presented agreement with the certified results. Diaion HP-20 resin presents as advantages low cost, no swelling problems and ability to form quelates with the analyte. The method could be applied for the preconcentration of analyte ions from other matrices.

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