



Analytical Methods

Preconcentration of some metal ions with lanthanum-8-hydroxyquinoline co-precipitation system



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ABSTRACT

A method of separation and preconcentration of cadmium, copper, nickel, lead and zinc at trace level using 8-hydroxyquinoline as a chelating agent and lanthanum(III) as a carrier element is proposed. The heavy metals were determined after preconcentration by inductively coupled plasma optical emission spectrometry (ICP-OES). The results were compared with those obtained using flame atomic absorption spectrometry (F-AAS). The influence of several parameters such as pH, amount of lanthanum(III) as a carrier element, amount of 8-hydroxyquinoline, duration of co-precipitation was examined. Moreover, effects of inorganic matrix on recovery of the determined elements were studied. The detection limits (DL) for ICP-OES were 0.31, 2.9, 1.4, 3.2 and 1.2 $\mu\text{g L}^{-1}$ for Cd, Cu, Ni, Pb and Zn, respectively, whereas for F-AAS DL were 0.63, 1.1, 3.2, 2.7 and 0.74 $\mu\text{g L}^{-1}$. The recovery of the method for the determined elements was better than 94% with relative standard deviation between 0.63% and 2.9%. The preconcentration factor was 60. The proposed method was successfully applied for determination of Cd, Cu, Ni, Pb, and Zn in plant materials. Accuracy of the proposed method was verified using certified reference material (NCS ZC85006 Tomato).

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1. Introduction

Heavy metals, in environment are one of the major sources of pollution. Small amounts of these elements are common in our environment and actually necessary for good health but large amounts of them may cause acute and chronic toxicity. Because of these reasons the determination of metal ion concentrations in environmental samples is one of the most important fields of the analytical chemistry.

The separation and preconcentration procedures are frequently required because of the low concentration of trace metal ions and possible matrix interferences. Up to now, ion exchange (Heiden, Gore, & Stark, 2010; Mahmoud, Kenawy, Hafez, & Lashein, 2010), liquid–liquid microextraction (Mirzaei, Behzadi, Abadi, & Beizaei, 2011; Mohammadia, Afzali, & Baghelan, 2009), co-precipitation (Efendioglu, Yagan, & Bati, 2007; Tokalioglu & Dasdelen, 2011), cloud point extraction (Ghaedi, Shokrollahi, Niknam, Niknam, & Soyak, 2009; Lemos, Silva da Franca, & Moreira, 2007), solid phase extraction (Roldan, Alcantara, Padilha, & Padilha, 2005; Tu et al., 2009), electrolytic deposition (Zawisza & Sitko, 2007), physical preconcentration by the evaporation of the sample (Zawisza & Sitko, 2006) and chemofiltration (Sitko, Zawisza, & Mzyk, 2006) have

widely been used as preconcentration and separation methods of trace metals in various samples.

The co-precipitation method is widely used for the concentration of trace metal ions prior to their flame atomic absorption spectrometric (F-AAS) and inductively coupled plasma optical emission spectrometry (ICP-OES). The main requirement for this technique is that the collector should easily be separated from the matrix solution. This can be done by filtering or centrifuging and washing of the precipitate. In addition, it is desirable that the collector should be a pure and readily available substance. The advantages of this technique are its simplicity and the ability that various analyte ions can be preconcentrated and separated simultaneously from the matrix. Co-precipitation has also an important place in the preconcentration and separation methods due to its some advantages including simplicity, high preconcentration factors, low consumption of organic solvent and short analysis time period for the procedures. In the co-precipitation procedure, a precipitate was occurred by the combination of a carrier element and a suitable inorganic or organic ligand. Among co-precipitation precipitates, metal chelates are the most attractive gathering precipitates because of their excellent multielements trace recovery and sufficient separation factors especially for alkali and alkaline earth elements.

In the present work, simple a co-precipitation system for the preconcentration of cadmium, copper, nickel, lead and zinc ions by using lanthanum-8-hydroxyquinoline has been presented.

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According to our literature survey, this combination is not used for the co-precipitation of multi-element ions, until now. We have investigated a new co-precipitation procedure for trace elements for ICP-OES and FAAS determination and found that lanthanum-8-hydroxyquinoline co-precipitation system has a good collecting ability for this purpose. Also, lanthanum-8-hydroxyquinoline is a convenient co-precipitant to handle, because it can be dissolved in about 5 mL 2 mol L⁻¹ solution of nitric acid without heating. The experimental conditions of Cd, Cu, Ni, Pb and Zn ions with lanthanum-8-hydroxyquinoline co-precipitant including pH, the amounts of carrier elements and reagent, effect of standing time and temperature, sample volume and matrix effects were examined.

The comparison of the presented procedure and some works on preconcentration and separation of heavy metal ions in literature is given in Table 1.

2. Experimental

2.1. Apparatus

A spectroflame atomic emission spectrometer, ICP Model M (Spectro Analytical Instruments, Germany) was used for determination of Cd, Cu, Ni, Pb and Zn. The sequential spectrometer was used with following parameters: frequency, 27.12 MHz; power, 1.1 kW; demountable quartz torch, Ar/Ar/Ar; coolant gas Ar, 14.0 L min⁻¹; auxiliary gas Ar, 0.5 L min⁻¹; nebulizer gas Ar, 1.0 L min⁻¹; nebulizer pressure, 2.4 bar; glass spray chamber according to Scott, sample flow rate, 1.0 mL min⁻¹; observation height 11 mm; holographic grating, 2400 grooves mm⁻¹; dispersion of grating in the first reciprocal order, 0.55 nm mm⁻¹; wavelength range of monochromator 165–460 nm. The wavelengths (integration time) were as follows: for Cd: 228.80 nm (3 s), Cu: 324.75 nm (3 s), Ni: 221.65 nm (5 s), Pb: 220.35 nm (5 s), Zn: 213.87 (4 s).

A flame atomic absorption spectrometer (Solaar M6 TJA Solutions) with deuterium background correction, equipped with a hollow cathode lamp was used for determination of Cd, Cu, Ni, Pb, and Zn. An air-acetylene burner was used. The wavelengths (spectral band pass) were as follows: for Cd: 228.8 nm (0.5 nm), Cu:

324.8 nm (0.5 nm), Ni: 232.0 nm (0.1 nm), Pb: 217.0 nm (0.5 nm), Zn: 213.9 nm (0.5 nm). The nebulizer flow rate was 5 mL min⁻¹.

2.2. Reagents and solutions

All chemicals were of analytical reagent grade. All reagents were dissolved and diluted with purity water obtained from Milli-Q system. The following reagent were used in the experiment: 8-hydroxyquinoline (POCh, Gliwice, Poland), lanthanum(III) nitrate(V) (Loba Feinchemie, Fischamend, Austria), disodium tetra borate, acetic acid, nitric acid, hydrochloric acid, sodium hydroxide, hydrogen peroxide, nitrate(V) salts of magnesium, calcium, strontium, barium, aluminum, iron(III), manganese, chromium (all from POCh, Gliwice, Poland), stock standard solutions of cadmium, copper, nickel, lead and zinc at a concentration of 1000 mg L⁻¹ (Merck, Darmstadt, Germany). 8-Hydroxyquinoline solution (5% w/v) was prepared in acetic acid. The working solutions of the metals were obtained by diluting these stock solutions prior to use. Sodium borate buffer solution was prepared by adding an appropriate amount of hydrochloric acid to disodium tetra borate solution until pH 8.0 was obtained. The accuracy of the method was assessed by analyzing the certified reference material (CRM): NCS ZC85006 Tomato (China National Analysis Center for Iron & Steel 2000).

2.3. Procedures

2.3.1. General procedure of co-precipitation

2 mg of lanthanum(III) and 2 mL of 5% solutions of 8-hydroxyquinoline were added to 100 mL of a solution containing 2 µg of Cd 10 µg of Pb and Ni, and 20 µg of Cu and Zn. The pH value of the obtained solution was adjusted to 8 using the tetra borate buffer. Then, the solution was heated in a water bath for 20 min in the temperature of 50 °C. After the formation of the precipitate, the solution was centrifuged at 3500 rpm for 10 min. The supernatant was removed. Precipitate was digested in 5 mL of 2 mol L⁻¹ solution of hydrochloric acid. The analytes in the final solution were determined by ICP-OES and FAAS.

The calibration samples were prepared using 100 mL of standard solution containing analytes in the ranges: 0.2–4 µg of Cd

Table 1
Comparative data from studies on co-precipitation of heavy metal ions.

Analytes	Co-precipitation system	Dissolving media	PF	DL (µg L ⁻¹)	pH	Detection	References
Cr, Fe, Pb, Zn	3-Phenyl-4-o-hydroxybenzylidenamino-4,5-dihydro-1,2,4-triazole-5-one (POHBAT)	1.0 mL conc. HNO ₃	50 (Pb), 150 (Fe, Cr, Zn)	0.5, 0.3, 2.0, 0.7	7	FAAS	Duran et al. (2009)
Pb, Fe, Co, Cr, Zn	Cu-N-benzoyl-N-phenyl-hydroxylamine (Cu-BPHA)	0.5 mL conc. HNO ₃	150	2.3, 0.7, 0.7, 0.3, 0.4	9	FAAS	Şaçmac and Kartal (2010)
Pb, Co, Cu, Cd, Fe, Ni	Zirconium(IV) hydroxide	1.0 mL conc. HNO ₃	25	2.5, 1.42, 1.55, 0.27, 1.53, 1.05	8	FAAS	Citak, Tuzen, and Soylak (2009)
Cu, Cd, Co, Ni, Fe, Pb	Thulium hydroxide	0.5 mL conc. HNO ₃	120	0.51, 0.10, 0.50, 1.41, 0.50, 1.60	11	FAAS	Soylak and Aydin (2011)
Cd, Cu	MEFMAT	1.0 mL conc. HNO ₃	50	1.49, 0.45	6.8	FAAS	Duran et al. (2011)
Co, Mn	Copper-8-hydroxyquinoline	0.5 mL conc. HNO ₃	25	0.86, 0.98	8	FAAS	Soylak, Kaya, and Tuzen, (2007)
Ni, Cd, Pb	Copper hydroxide	0.5 mL conc. HNO ₃	100	3.0, 2.0, 7.0	9	FAAS	Soylak, Kars, and Narin, (2008)
Pb, Cd, Cr, Ni, Mn	Dibenzylthiocarbamate	0.5 mL conc. HNO ₃	50	0.87, 0.34, 0.75, 0.06, 0.45	9	FAAS	Tuzen and Soylak, (2009)
Fe, Pb, Cu	Salicylaldoxime	5 mL 3 mol L ⁻¹ HNO ₃	15 (Pb), 20 (Fe, Cu)	1.58, 3.56, 1.32	9	FAAS	Tokahoglu and Yildiz (2009)
Cd, Cu, Ni, Pb, Zn	Lanthan-8-hydroxyquinoline	5 mL 2 mol L ⁻¹ HNO ₃	60	0.31, 2.9, 1.4, 3.2, 1.2 0.63, 1.1, 3.2, 2.7, 0.74	8	FAAS ICP-OES	This paper

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