

Preconcentration and Simultaneous Determination of Lead(II) and Copper(II) by N,N'-Disalicylideneethylenediamine by Electro Thermal Atomic Absorption Spectrometry

ALI MOGHIMI* and MOHAMAD JAVAD POURSHARIFI†
Department of Chemistry, East Tehran (Ghiam Dasht) Campus
Islamic Azad University, Tehran, Iran
E-mail: kamran9537@yahoo.com

A simple, highly sensitive, accurate and selective method for simultaneous determination of trace amounts of Pb(II) and Cu(II) in water samples is presented. The method is based on selective chelation of Pb(II) and Cu(II) on surfactant coated alumina, modified with a Schiff base N,N'-disalicylideneethylenediamine. The retained ions were then eluted with 4 mL of 4 M nitric acid and determined by electro thermal atomic absorption spectrometry at 283 and 324 nm for Pb and Cu, respectively. The influence of flow rates of sample and eluent solutions, pH, breakthrough volume, effect of foreign ions on chelation and recovery were investigated. 1.5 g of surfactant coated alumina adsorbs 40 mg of the Schiff base which in turn can retain 15.0 ± 0.9 mg of each of both metal ions. The limit of detection (3σ) for Pb(II) and Cu(II) were found to be 5.57 and 1.69 ng L⁻¹, respectively. The enrichment factor for both metal ions are 100. The present method was successfully applied on determination of lead and copper in different water samples. The ions were also speciated by means of three columns system.

Key Words: Lead(II), Copper(II), Preconcentration, ETAAS, N,N'-Disalicylideneethylenediamine.

INTRODUCTION

Trace amounts of metals are present in natural biosphere. Presence of some of these metals in very low concentrations and certain oxidation states are necessary. Higher concentrations and other oxidation states might be toxic and dangerous. Unfortunately, the difference between these two levels are very small^{1,2}. Lead occurs in nature mostly as PbS. It is used in batteries, tetraethyl lead, guns, solders and X-ray instruments³. Copper on the other hand occurs as CuS, CuS₂, CuFeS₂, CuSO₄·5H₂O and other forms. More than 75 % of copper production is used in electrical industries. It is also used in pigments, metallic blends and household. Hence determination of lead and copper in industry and environment are both very important. A preconcentration step is advisable in trace analysis. Lead and copper have been so far determined

†Department of Chemistry, Saveh Campus, Islamic Azad University, Saveh, Iran.

by various methods such as spectrophotometry^{5,6}, liquid-liquid extraction⁷⁻⁹, cloud point extraction^{10,11} and electrochemical measurements¹². Some of these methods suffer from poor limit of detection and usage of harmful solvents. On the other hand, effect of foreign ions on the analyte is not negligible in many instances. In such cases, preconcentration of the analyte makes the determination easier and the composition of the sample less complicated. In recent years, solid phase extraction has offered attractive possibilities in trace analysis. It reduced the solvent and time consumption considerably¹³⁻¹⁸. In order to increase the preconcentration or extraction power of solid phase extraction an organic or inorganic ligand is used in conjunction with the sorbent. Some of the ligands used for determination of lead and copper are Amberlit XAD-2 with 3,4-dihydroxybenzoic acid¹⁹, silica gel modified with 3-amino-propyl triethoxysilane²⁰, Levatit with di(2,4,4-trimethylpentyl)phosphinic acid²¹, silica gel functionalized with methyl thiosalicylate²², silicagel modified with zirconium phosphate²³ and C₁₈ disks modified with a sulfur containing Schiff base²⁴.

Comparing these examples with the present method, they have either a lower enrichment factor or a higher limit of detection. On the other hand, the C₁₈ disks can be used only a few times, while the proposed sorbent could be used more than 50 times without loss of efficiency.

Surfactant coated alumina modified with chelating agents has been used for extraction and preconcentration of environmental matrixes and metals^{25,26}. Here, the surfactant molecules have been associated on the alumina surface forming an admicell or hemimicell. Organic molecules attach themselves on the hydrophobe part and low concentration of metallic elements also on the hydrophobe part, which includes the chelating agent²⁶. Schiff bases which are obtained from salicylaldehyde are known as multidentate ligands. These agents can form stable complexes with transition metal ions^{27,28}.

The main aim of the present work is to develop a fast, sensitive and efficient way for simultaneous enrichment and extraction of trace amounts of Pb(II) and Cu(II) from aqueous media by means of a surfactant coated alumina modified with N,N'-disalicylideneethylenediamine (Fig. 1). The chelated ions were desorbed and determined by electro thermal atomic absorption spectrometry (ETAAS).

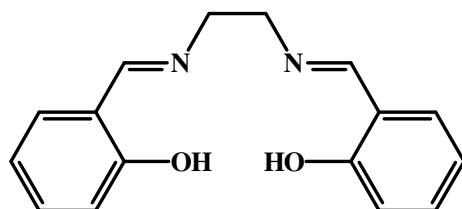


Fig. 1. Structure of N,N'-disalicylideneethylenediamine (NNDED)

The modified solid phase could be used at least 50 times with acceptable reproducibility without any change in the composition of the sorbent, ligand or sodium dodecyl sulfate. On the other hand, in terms of economy it is much cheaper than C₁₈ disks.

EXPERIMENTAL

All solutions were prepared with doubly distilled deionized water. Alumina powder γ -type for chromatography with diameter of about 50 μm obtained from Katayama Chemicals. It was conditioned before use by suspending in 4 M nitric acid for 20 min and then washed two times with water. Sodium dodecyl sulfate (SDS) obtained from Merck and used without any further purification.

Synthesis and spectral characteristics of N,N'-disalicylideneethylenediamine (NNDED)

A mixture of 0.002 mol salicylaldehyde and 0.001 mol of ethylenediamine in 10 to 15 mL of absolute methanol was refluxed in a round bottom flask for 1 h. The solvent was then evaporated and the residue was crystallized from 95 % ethanol as a yellow precipitate. The precipitate was thereafter recrystallized in absolute ethanol and finally dried in vacuum. The specifications of the yellow crystals obtained as product were: m.p. 126-128 °C; IR (KBr, ν_{max} , cm^{-1}): 3050, 2932, 2901, 2870, 1636, 1497, 1283, 1149, 1042, 1021, 857, 749, 741; MS, m/z (%): 268 (M^+ , 23); Anal. calcd. for C₁₆H₁₆N₂O₂ (268.31): C, 71.62; H, 6.01; N, 10.44 Found: C, 71.8; H, 6.05; N, 10.3 %. ¹H NMR (250.1 MHz, CDCl₃): δ 3.9 (s, 4H, CH₂), 6.8 (m, 4H), 7.3 (m, 4H), 8.3 (s, 2H), 13.2 (brs, 2H, Ar-OH).

Lead nitrate, copper sulfate pentahydrated, ammonium dihydrogen phosphate were of analytical reagent grade and RP-C₁₈ silica (40-63 μm) obtained from Merck. The anion exchanger resin Dowex 1X-8 mesh 100-200 obtained from Fluka. The chelating resin, chelex-100, mesh 100-200 obtained from Bio Rad.

Standard stock solution of 1000 $\mu\text{g mL}^{-1}$ of Pb(II) and Cu(II) were prepared by dissolving appropriate quantity of their salts in water. A solution of 0.35 M of ammonium dihydrogen phosphate in water was prepared as the matrix modifier for determination of lead. A solution of SDS-NNDED was prepared as follow: to a solution of 0.2 g of NNDED in 10 mL of ethanol, 5 drops of 1 M NaOH and 0.5 g of SDS were added, dissolved and its volume adjusted to 50 mL with water.

Preparation of admicell column: To 40 mL of water containing 1.5 g of alumina, 10 mL of the above NNDED solution was added. The pH of the suspension was adjusted to 2.0 by addition of 4 M HNO₃ and stirred by mechanical stirrer for 20 min. Then the top liquid was decanted (and discarded) and the remained alumina was washed three times with water, then with 5 mL of 4 M HNO₃ and again three times with water. The prepared sorbent was transferred to a polypropylene tube (i.d 5 mm, length 10 mm).

The measurements were performed by a Perkin-Elmer model 300 Analyst atomic absorption spectrometer, equipped with a graphite furnace of the type HGA-850, furnace auto sampler of the type AS-800 and a deuterium lamp for background correction. Instrumental conditions and furnace parameters for the atomizer are listed in Tables 1 and 2. A metrohm 691 pH meter equipped with a combined glass calomel electrode was used for pH measurements.

TABLE-1
INSTRUMENTAL PARAMETERS FOR DETERMINATION OF LEAD
Wavelength (nm) = 283.3, Slit width (nm) = 0.7, Lamp current (mA) = 10

Furnace conditions		Time (s)		Argon gas flow (mL min ⁻¹)
Step	Temperature (°C)	Ramp	Hold	
Drying	130	20	15	250
Pyrolysis I	350	5	5	250
Pyrolysis II	450	15	5	250
Atomization	2250	0	5	0
Cleaning	2600	1	3	250

Matrix modifier 0.241 mg NH₄H₂PO₄

Absorbance measurements were based on peak heights.

TABLE-2
INSTRUMENTAL PARAMETERS FOR DETERMINATION OF COPPER
Wavelength (nm) = 324.8, Slit width (nm) = 0.7, Lamp current (mA) = 15

Furnace conditions		Time (s)		Argon gas flow (mL min ⁻¹)
Step	Temperature (°C)	Ramp	Hold	
Drying	100	20	5	250
Pyrolysis I	140	15	15	250
Pyrolysis II	1000	20	10	250
Atomization	2300	5	0	0
Cleaning	2600	3	1	250

Absorbance measurements were based on peak heights.

Procedure: The pH of a solution containing 100 ng of each Pb(II) and Cu(II) was adjusted to 2.0. This solution was passed through the admicell column with a flow rate of 5 mL min⁻¹. The column was washed with 10 mL of water and the retained ions were desorbed with 1 mL of 4 M HNO₃ with a flow rate of 2 mL min⁻¹. The desorption procedure was repeated 3 more times. All the acid solutions (4 mL all together) were collected in a 10 mL volumetric flask and diluted to the mark with water. The concentrations of lead and copper in the solution were determined by ETAAS at 283 and 324 nm, respectively.

Determination of lead and copper in water samples: Polyethylene bottles, soaked in 1 M HNO₃ overnight and washed two times with water were used for sampling. The water sample was filtered through a 0.45 µm pores filter. The pH of a 1000 mL portion of each sample was adjusted to 2.0 (4 M HNO₃) and passed through the column under a flow rate of 5 mL min⁻¹. The column was washed with water and the ions were desorbed and determined as the above mentioned procedure.

Speciation of lead and copper in water samples: The method has been evaluated and optimized for speciation and its application on complex mixtures³⁰⁻³³.

The chelating cation exchanger (Chelex-100) and anion exchanger, Dowex 1X-8 resins were washed with 1 M HCl, water, 1 M NaOH and water, respectively. 1.2 g of each resin was transferred to separate polyethylene columns. Each column was washed with 10 mL of 2 M HNO₃ and then 30 mL of water. The C₁₈ bounded silica adsorber in a separate column was conditioned with 5 mL of methanol, then 5 mL of 2 M HNO₃ and at the end with 20 mL of water. 5 mL of methanol was added on top of the adsorber and passed through it until the level of methanol reached just the surface of the adsorber. Then water was added on it and connected to the other two columns. A certain volume of water sample was filtered through a 0.45 µm filter and then passed through the three columns system, Dowex 1X-8, RP-C₁₈ silica adsorber and Chelex-100, respectively. The columns were then separated. The anion and cation exchanger columns were washed with 10 mL of 2 M HNO₃ and the C-18 column with 10 mL of 1 M HCl. The flow rate of eluents was 1 mL min⁻¹. The lead and copper content of each eluted solution were determined by ETAAS.

RESULTS AND DISCUSSION

N,N'-Disalicylideneethylenediamine (NNDED) is a tetradentate ligand. It is insoluble in water, however, soluble in slightly alkaline ethanol. Primary investigations revealed that surfactant coated alumina could not retain Pb(II) and Cu(II) cations, but when modified with NNDED, retains these cations selectively. It was then decided to investigate the capability of NNDED as a ligand for simultaneous preconcentration and determination of lead and copper on admicell.

The alumina surface in acidic media (1 < pH < 6) attracts protons and becomes positively charged. The hydrophilic part of SDS (-SO₃⁻), is attached strongly to these protons. On the other hand, the ligand molecules are attached to hydrophobic part of SDS and retain small quantities of metallic cations²⁶.

Effect of pH: The effect of pH of the aqueous solution on the extraction of 100 ng of each of the cations Pb(II) and Cu(II) was studied in the pH range of 1-10. The pH of the solution was adjusted by means of either 0.01 M HNO₃ or 0.01M NaOH. The results indicate that complete chelation and recovery of Pb(II) occurs in pH range of 2-4 and that of Cu(II) in 2-8 are shown in Fig. 2. It is assumed that at higher pH values, the cations might be hydrolyzed and complete desorption does not occur. Hence, in order to prevent hydrolysis of the cations and also keeping SDS on the alumina, pH = 2.0 was chosen for further studies.

Effect of flow rates of solutions: The effect of flow rate of the solutions of the cations on chelation of them on the substrate was also studied. It was indicated that flow rates of 1-5 mL min⁻¹ would not affect the retention efficiency of the substrate. Higher flow rates cause incomplete chelation of the cations on the sorbent. The similar range of flow rate for chelation of cations on modified alumina with SDS and a ligand has been reported in literature^{25,26}. Flow rate of 1-2 mL min⁻¹ for desorption

of the cations with 4 mL of 4 M HNO₃ has been found suitable. Higher flow rates need larger volume of acid. Hence, flow rates of 5 and 2 mL min⁻¹ were used for sample solution and eluting solvent throughout, respectively.

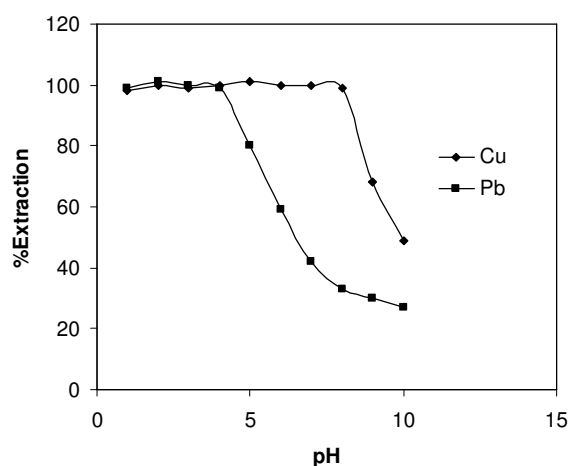


Fig. 2. Extraction percentage of Pb(II) and Cu(II) against pH

Effect of NNDED quantity: To study optimum quantity of NNDED on quantitative extraction of lead and copper, 50 mL portions of solutions containing 100 ng of each cation were passed through different columns the sorbent of which were modified with various amounts, between 10-50 mg of the ligand. The best result was obtained on the sorbent which was modified with 40 mg of the ligand.

Figures of merit: The breakthrough volume is of prime importance for solid phase extractions. Hence, the effect of sample volume on the recovery of the cations was studied. 100 ng of each cation was dissolved in 50, 100, 500 and 1000 mL of water. It was indicated that in all the cases, chelation and desorption of the cations were quantitative. It was then concluded that the breakthrough volume could be even more than 1000 mL. Because the sample volume was 1000 mL and the cations were eluted into 10 mL solution, the enrichment factor for both cations is 100, which is easily achievable.

The maximum capacity of 1.5 g of the substrate was determined as follow: 500 mL of a solution containing 50 mg of each cation was passed through the column. The chelated ions were eluted and determined by ETAAS. The maximum capacity of the sorbent for three individual replicates was found to be 15.0 ± 0.9 μg of each cation.

The limit of detection (3σ) for the cations³⁴ were found to be 5.57 and 1.69 ng L⁻¹ for lead and copper ions, respectively. Reproducibility of the method for extraction and determination of 100 ng of each cation in a 50 mL solution was examined. As the results of seven individual replicate measurements indicated, they were 2.85 and 2.98 % for Pb(II) and Cu(II), respectively.

Effect of foreign ions: Effect of foreign ions were also investigated on the measurements of lead and copper. Here a certain amount of foreign ion was added to 50 mL of sample solution containing 100 ng of each Pb(II) and Cu(II) with a pH of 2.5. The amounts of the foreign ions and the percentages of the recovery of lead and copper are listed in Table-3. As it is seen, it is possible to determine lead and copper without being affected by the foreign ions.

TABLE-3
EFFECT OF FOREIGN IONS ON THE RECOVERY OF 100 ng OF Pb²⁺ OR Cu²⁺

Diversion ion	Amounts taken (mg) added to 50 mL	Found (%)	% Recovery of Cu ²⁺ ion	% Recovery of Pb ²⁺ ion
Na ⁺	92.40	1.15 (2.9)*	99.7 (1.8)	99.3 (2.8)
K ⁺	92.50	1.32 (2.1)	98.5 (2.2)	98.5 (2.2)
Mg ²⁺	14.50	0.70 (1.8)	98.7 (1.7)	99.5 (2.7)
Ca ²⁺	28.30	1.29 (2.0)	99.0 (1.8)	99.0 (2.8)
Sr ²⁺	3.45	2.85 (2.2)	99.8 (2.9)	98.9 (2.6)
Ba ²⁺	2.66	3.16 (2.4)	99.3 (2.5)	98.8 (2.5)
Mn ²⁺	2.66	1.75 (2.3)	98.6 (1.8)	98.6 (3.3)
Co ²⁺	2.17	1.40 (2.3)	99.1 (2.9)	99.9 (2.9)
Ni ²⁺	2.67	2.00 (2.4)	98.5 (2.4)	99.5 (2.4)
Zn ²⁺	2.78	1.97 (2.1)	99.5 (3.2)	98.5 (2.2)
Cd ²⁺	2.58	1.90 (2.0)	98.2 (2.8)	99.2 (2.7)
Bi ³⁺	2.54	2.70 (1.9)	99.5 (2.7)	98.5 (2.4)
Al ³⁺	2.43	2.81 (2.1)	98.7 (2.8)	98.7 (2.8)
Fe ³⁺	2.67	3.45 (2.9)	96.6 (2.8)	96.6 (2.8)
Cr ³⁺	1.76	2.92 (2.3)	97.3 (2.4)	97.3 (2.4)
UO ²⁺	2.86	1.30 (2.9)	100.0 (2.9)	99.6 (2.7)
NO ₃ ⁻	5.00	2.30 (2.3)	99.3 (2.8)	99.9 (3.6)
CH ₃ COO ⁻	5.00	2.20 (2.6)	99.4 (2.2)	99.3 (3.6)
SO ₄ ²⁻	5.00	2.90 (3.1)	98.7 (2.4)	99.4 (3.6)
CO ₃ ²⁻	5.00	1.80 (2.4)	99.3 (2.5)	99.3 (3.6)
PO ₄ ³⁻	2.50	2.10 (2.2)	98.9 (2.4)	100.3 (3.2)

*Values in parenthesis are CVs based on 3 individual replicate measurements.

Analysis of the water samples: The prepared sorbent was used for analysis of real samples. To do this, the amounts of lead and copper were determined in different water samples namely: distilled water, tap water of Tehran (Tehran, taken after 10 min operation of the tap), rain water (Tehran, 26 January, 2006), Snow water (Saveh, 6 February, 2006) and two synthetic samples containing different cations. The results are tabulated in Table-4. The amounts of lead or copper added to the water samples are extracted and determined quantitatively which indicates accuracy and precision of the present method.

Separation and speciation of cations by three columns system. It is possible to preconcentrate and at the same time separate the neutral metal complexes of organic ligands, anionic complexes and free ions from each other by this method³¹. Water samples were passed through the 3 connected columns *i.e.*, anion exchanger,

TABLE-4
RECOVERY OF LEAD AND COPPER CONTENTS OF WATER SAMPLES

		Amount added (μg)	Found (μg)	Recovery (%)
Sample distilled water (100 mL)	Pb	–	–	–
		0.050	0.048(3.45)	96
		0.100	0.098(2.95)	98
	Cu	–	–	–
		0.050	0.049(2.75)	98
		0.100	0.101(2.47)	101
Tap water (100 mL)	Pb	–	0.015(4.50)	–
		0.050	0.067(3.42)	101
	Cu	–	0.054(3.71)	–
		0.050	0.106(2.31)	100
Snow water (50 mL)	Pb	–	0.048(3.25)	–
		0.100	0.151(2.65)	100
	Cu	–	0.060(3.66)	–
		0.050	0.120(2.55)	100
Rain water (100 mL)	Pb	–	0.042(3.25)	–
		0.100	0.148(2.45)	102
	Cu	–	0.044(3.55)	–
		0.100	0.143(2.46)	98
Synthetic sample 1 Na ⁺ , Ca ²⁺ , Fe ³⁺ , Co ²⁺ , Cr ³⁺ , Hg ²⁺ , 1 mg L ⁻¹ of each cation	Pb	–	–	–
		0.100	0.101(3.43)	101
	Cu	–	–	–
		0.100	0.101(3.31)	101
Synthetic sample 2 K ⁺ , Ba ²⁺ , Mn ²⁺ , Cd ²⁺ , Ni ²⁺ , Zn ²⁺ , 1 mg L ⁻¹ of each cation	Pb	–	–	–
		0.100	0.101(2.71)	101
	Cu	–	–	–
		0.100	0.101(3.11)	101

a: Values in parenthesis are CVs based on three individual replicate measurements.

TABLE-5
RESULTS OF SPECIATION OF LEAD AND COPPER IN DIFFERENT
SAMPLES BY THREE COLUMNS SYSTEM

Column	Tap water (1000 mL)		Water sample (1000 mL)†		River water (50 mL)	
	Pb (μg)	Cu (μg)	Pb (μg)	Cu (μg)	Pb (μg)	Cu (μg)
Dowex1X8	–	–	–	–	–	–
Silica C-18	–	–	–	–	–	–
Chelex-100	0.012 (4.7)‡	0.053 (3.8)	0.105 (3.1)	0.104 (3.1)	0.103 (3.1)	0.157 (2.8)

†This was a solution containing 0.1 μg of each cation in 1000 mL of distilled water.

‡Values in parenthesis are CVs based on three replicate analysis. The samples are the same as those mentioned in Table-4.

C₁₈-silica adsorber and chelating cation exchanger. Each species of lead or copper is retained in one of the columns; anionic complexes in the first column, neutral complexes of organic ligands in the second and the free ions in the third. The results

of passing certain volumes of different water samples through the columns are listed in Table-5. According to the results, it is indicated that lead and copper present only as cations. On the other hand the t-test comparing the obtained mean values of the present work with those published indicate no significant difference between them.

Conclusion

A method for simultaneous determination and preconcentration of Pb(II) and Cu(II) in water samples using surfactant coated alumina impregnated with a Sciff base has been proposed. This method offers simple, highly sensitive, accurate and selective method for simultaneous determination of trace amounts of lead(II) and copper(II) in water samples.

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AL-MADINAH AL-MUNAWWARAH, SAUDI ARABIA

Contact:

Dr. Fethi Kooli

Taibah International Chemical Conference (TICC-2009)

Department of Chemistry, Taibah University, P.O.Box 30100

Al-Madinah Al-Munawwarah, Saudi Arabia

Voice: +966.559970918

Fax: +966.48470235

E-mail: fkooli@taibahu.edu.sa; info@taibah.ticc-2009.com

Web: <http://www.taibah.TICC-2009.com>