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Solid phase extraction method for selective determination of Pb(II) in water samples using 4-(4-methoxybenzylidenimine) thiophenole

M. Ghaedi [a], M. Montazerozohori [a], M. Soylak [b,*]

[a] *Chemistry Department, University of Yasouj, Yasouj 75914-353, Iran*
[b] *Chemistry Department, University of Erciyes, 38039 Kayseri, Turkey*

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Abstract

A sensitive and selective extractive preconcentration procedure for the determination of traces of lead in water samples has been developed. An alumina-sodium dodecyl sulfate (SDS) coated modified with 4-(4-methoxybenzylidenimine) thiophenole (MBITP) was used for preconcentration and determination of Pb(II) by flame atomic absorption spectrometry. Lead was adsorbed quantitatively on modified column due to its complexation with MBITP and quantitatively eluted using 5 mL 1 mol L^{-1} nitric acid in acetone. The effects of parameters such as pH, amount of solid phase, amount of MBITP, flow rate, type and concentration of eluting agent were examined. The effect of interfering ions on the determination of Pb(II) was also investigated. The response of proposed method is linear in the concentration range 0.05–1.2 μg mL^{-1} of Pb(II). The limit of detections ($3S.D._b/m$, $n = 4$) and relative standard deviations ($n = 11$) are 1.6 ng mL^{-1} and 0.9%, respectively. The presented procedure was successfully applied for determination of lead content in real samples such as river, spring, waste and drinking water.

Keywords: 4-(4-Methoxybenzylidenimine) thiophenole; Lead; Surfactant coated alumina; Atomic absorption spectrometry; Solid phase extraction

1. Introduction

Lead is a serious cumulative body poison [1] and enters our body system through air, water, and food. Inorganic lead binds itself with the SH group in enzymes or proteins and acts as an enzyme inhibitor [2]. Acute lead poisoning in humans causes severe damage in the kidneys, liver, brain, reproductive system and central nervous system, and sometimes causes death. Mild lead poisoning causes anemia, headache and sore muscles and the victim may feel fatigued and irritable. Chronic exposure to lead causes nephritis, scaring and the shrinking of kidney tissues [3]. It is emitted into the biosphere in considerable amounts, owing to its increased industrial use and its application as a fuel additive [4,5]. In recent years concern has increased over the concentration of lead in drinking and natural waters [6]. Several analytical techniques such as inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are available for the determination of trace metals with sufficient sensitivity for most of applications but the required instruments are expensive, day-to-day maintenance cost is high and various types of inherent interferences appear. The determination of metal ions at the ng mL^{-1} level generally requires prior separation and/or preconcentration steps in order to improve sensitivity [7–10].

There are many methods for Pb(II) pre-concentration, separation and determination such as liquid–liquid extraction (LLE), preconcentration with flow injection analysis, sorption on the various adsorbents along them the extractive spectrophotometric methods [11–16] has been developed for Pb(II) determination. Liquid–liquid extraction and transport as conventional methods for separation and determination of metal ion content because of some problems such as exposure to harmful organic solvent, disposal costs and large extraction time is less efficient from economic and health view.

In the present work a simple, selective and sensitive solid phase extraction method on SDS coated alumina for preconcentration and determination of lead in natural water samples was developed.

* Corresponding author. Tel.: +90 352 4374933; fax: +90 352 4374933.
E-mail address: soylak@erciyes.edu.tr (M. Soylak).

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2. Experimental

2.1. Reagents and solutions

Doubly distilled deionized water was used throughout. Analytical grade nitrate salts of lead, cadmium, mercury, cobalt, nickel, copper, zinc, magnesium, calcium, strontium, barium, silver, sodium and potassium (all from Merck) were of the highest purity available and used without any further purification. pH of the working solution was obtained by adding an appropriate amount of dilute nitric acid or sodium hydroxide to phosphate buffer. The γ-Al_2O_3 mesh 10–50 was purchased from Merck Company and used as received.

2.2. Apparatus

A Shimadzu UV-Vis 160 was used to measure the absorbance of un-adsorbed ligand. A Metrohm 691 pH/Ion meter with a combined glass and calomel electrode has been applied for adjustment of the pH. A pyrex glass column containing 1 g of SDS coated alumina in water suspension was 40 cm long and 1 cm in internal diameter. The bed height in the column was approximately 2 cm.

The lead determinations were carried out on a Perkin-Elmer 603 atomic absorption spectrometer (AAS) with a hollow cathode lamp and a deuterium background corrector, at a wavelength of 217.0 nm using an air-acetylene flame. The AAS determinations were performed under the recommended conditions for each metal. The IR of MBITP was recorded on 680 FT-IR JASCO model and ^{1}H NMR was recorded on 250 MHz Brucker.

2.3. Synthesis of 4-(4-methoxybenzylidenimine) thiophenole

To 0.2 mmol (0.2501 g) 4-amino thiophenole in 10 ml methanol, 0.2 mmol (0.2724 g) of 4-methoxybenzaldehyde was added and reaction mixture was stirred at room temperature. The progress of reaction was monitored by TLC. After 4 h a yellowish precipitate was formed. The reaction mixture was filtered and residue was washed twice with methanol, and finally dried under vacuum. A 0.340 g of product was obtained (yield ~70%). The compound was characterized by IR and ^{1}H NMR. The spectral data for MBITP are as follows:

IR (cm-1, in KBr): 2996 (w), 2962 (w), 2928 (w), 2831 (w), 2563 (w), 1617 (vs), 1600 (vs), 1577 (vs), 1514 (s), 1486 (s), 1457 (s), 1400 (m), 1309 (s), 1246 (vs), 1195 (s), 1161 (vs), 1104 (m), 1035 (s), 967 (m), 933 (w), 881 (m), 842 (vs), 819 (vs), 807 (vs), 867 (m), 728 (m), 539 (m), 568 (m).

^{1}H NMR ($CDCl_3$): (8.37 ppm, s, 1H), (7.52 ppm, d, 2H), (7–7.2 ppm, dd, 4H), (6.79 ppm, d, 2H), (3.71 ppm, s, 3H), (2.9 ppm, s, 1H).

2.4. General extraction procedure for ascernating complex structure

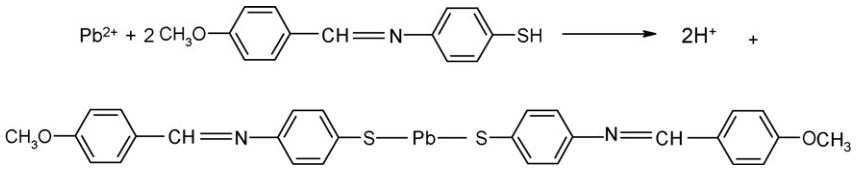
An aliquot of solution containing 1–500 μg mL^{-1} of Pb(II) (100 mL for low concentration and 20 mL for large concentration) was taken in separator funnel. To this 2 mL of phosphate buffer solution of pH 5.0 was added and the total volume was made up to mark with distilled water. This solution was then equilibrated with 10 mL of chloroform containing various amount of 0.01 mol L^{-1} MBITP and stirred for 2 min. After allowing adequate time for phase separation, the absorbance of organic phase containing the complex was measured at 370 nm against a reagent blank. Schematic diagram of complexation between MBITP and lead ion has been presented in Scheme 1.

2.5. Preparation of MBITP coated alumina

A volume of 10 mL SDS-MBITP solution was added to 40 mL of water solution containing 1 g alumina particles. The pH was adjusted to 2 with 2 mol L^{-1} hydrochloric acid to form MBITP-impregnated ad-micelles on alumina particles while shaking the suspension with a stirrer. After mixing for 15 min, the supernatant solution was discarded and the remaining was packed into a column. The column was washed by passing 5 mL of 2 mol L^{-1} HNO_3, and then the column was neutralized with 0.01 mol L^{-1} aqueous ammonia. When kept in a refrigerator the sorbent is stable at least for 1 week. The concentration of SDS was fixed below the critical micellization concentration (CMC) (8×10^{-3} M) of SDS.

2.6. Measurement of amount of ligand loaded on the SDS coated alumina

A 10 mL of 0.005 mol L^{-1} NaOH solution containing 25 mg of MBITP was added to one gram of γ-alumina and 100 mg of SDS in a 25 mL vial and shaken. After 1–2 days, a portion of the supernatant liquid was diluted to the appropriate volume and the absorbance of the solution was measured at maximum wavelength. Comparisons of the absorbance of supernatant solution with absorbance of ligand solution before addition indicate the amount of MBITP adsorbed on SDS coated alumina.



Scheme 1. Schematic diagram of complexation between MBITP and lead ion.

2.7. Preconcentration procedure

The pH of the solution (500–1000 mL) was adjusted to ~5.5 with hydrochloric acid and passed through the MBITP coated alumina column at a flow rate of 4 mL min^{-1} with the aid of a suction pump. The analyte was then eluted with 5 mL of 1 mol L^{-1} nitric acid in acetone. The lead content of the eluent was measured by flame-AAS.

3. Results and discussion

Based on the well known hard-soft acid–base theory, the presence of sulfur donor atom in the flexible structure of MBITB is expected to increase both the stability and selectivity of its Pb(II) complex over other metal ions including alkali, alkaline earth and many transition metal ions. To ascertain the nature and structure of the desired complex between lead ion and MBITP, a fixed amount of lead ion was extracted in to chloroform according to experimental section with various amounts of MBITP. The spectrophotometric studies in chloroform solution revealed that MBITP as a chelating agent can form a fairly stable complex with a 2:1 stochiometry, indicating the association of two MBITP molecules with the extracted lead species into organic phase, i.e. the composition of extracted species is $Pb(MBITP)_2$. The slope of log *D* versus log [MBITP] curve was also support the stoichiometric relation of MBITP to Pb(II) (*D* is distribution ratio: concentration of Pb^{2+} in organic phase/Pb^{2+} concentration in aqueous phase). Due to the strict hindered and linear structure of ligand, coordination numbers more than two and linkage via nitrogen atoms of ligand seem to be impossible, respectively.

3.1. Optimization of variables for preconcentration of lead

The effective parameter such as pH of sample, amount of MBITP and solid phase, type of eluting agent and its concentration and flow rate must be optimized for obtaining maximum accurate and precise signal for evaluation of lead content.

3.1.1. Effect of pH on recovery

In the solid phase extraction studies, pH is an important point for the quantitative recoveries of analytes [29–31]. The effect of pH on the recovery of lead(II) on SDS loaded with MBITP was studied by using 250 mL of model solution containing 50 μg of Pb(II) in the pH range of 2.2–8.0. The corresponding data is shown in Fig. 1. At the pH range of 3.3–6.8, quantitative recoveries were obtained for lead(II). The decrease in signal at pH > 6.8 is probably due to the precipitation of lead ions in the form of hydroxide, and at pH < 3.3 may be due to competition from hydronium ion toward lead ions for complexation with MBITP, which led to the decrease in recovery. Therefore, the pH 5.5 ± 0.5 was selected for the all subsequent works. pH 5.5 was obtained by adding an appropriate amount of dilute sodium hydroxide to phosphate buffer.

3.1.2. Effect of MBITP amount of solid phase on recovery

The amount of ligand has a large effect on the absorbance of lead as desired complex with MBITP incorporated in the interior core of ad-micelle. Therefore, the influences of the amount of MBITP on the recoveries of lead ions keeping other parameters constant were investigated. The recovery of lead increased rapidly with increasing MBITB concentration up to 25 mg. After 25 mg of MBITP the recoveries were quantitative. All further works 25 mg of MBITP was used.

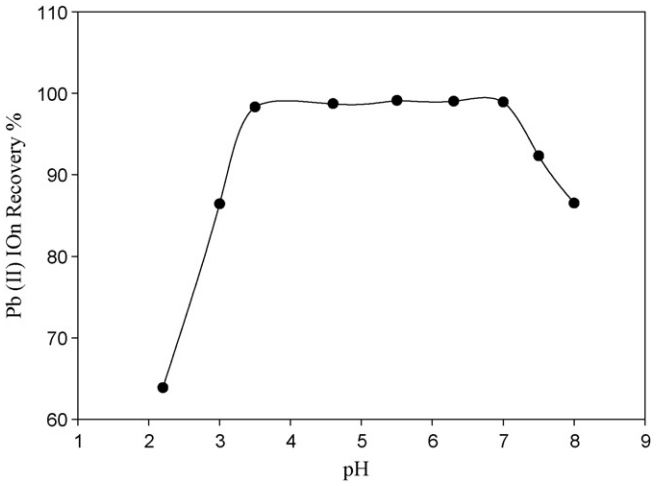


Fig. 1. Effect of pH on Pb(II) recovery (sample volume: 250 mL; concentration of lead: 0.2 μg mL^{-1}; flow rate: 4 mL min^{-1}; eluting solution: 5 mL of 1 mol L^{-1} HNO_3 in acetone).

3.1.3. Effect of eluting solution on recovery

The nature and concentration of eluting agents were found to have a significant effect on the desorption process of the adsorbed ions from the column [32–36]. For the investigation of effects of eluent, the elution was performed with 0.2–1.5 mol L^{-1} HNO_3, H_2SO_4 or HCl. The results are given in Table 1. Quantitative elution was achieved for 5 mL of 1 mol L^{-1} HNO_3 in acetone. It is obvious that 5 mL of 1.0 mol L^{-1} nitric acid in acetone were sufficient for quantitative recovery of absorbed lead.

3.1.4. Investigation of method performances

By passing a 250 mL solution of 0.02–2.00 μg mL^{-1} of Pb(II) at the optimum conditions, the calibration curves were

Table 1
Effect of type and concentration on the recoveries of lead (pH: 5.5, flow rate: 5 mL min^{-1})

Condition of eluting agent	Recovery (%)
1 HCl in acetone	92.3 ± 1.2[a]
1 mol L^{-1} HNO_3 in acetone	99.1 ± 0.6
1 mol L^{-1} H_2SO_4 in acetone	90.1 ± 0.8
0.2 mol L^{-1} EDTA	73.1 ± 1.1
0.2 mol L^{-1} $P_4O_7^{2-}$	74.2 ± 1.0
0.2 mol L^{-1} HNO_3 in acetone	59.6 ± 1.2
0.6 mol L^{-1} HNO_3 in acetone	76.5 ± 1.1
0.8 mol L^{-1} HNO_3 in acetone	89.9 ± 1.0
1.5 mol L^{-1} HNO_3 in acetone	99.2 ± 0.6

[a] Mean ± standard deviation.

Table 2
Evaluating the tolerance limit of common interfering ion on the recoveries of lead (II)

Ion	[*M*]/[Pb(II)]
Co^{2+}	1000
Ni^{2+}	1000
Cu^{2+}	300
Ag^{+}	300
Hg^{2+}	200
Zn^{2+}	1000
Cd^{2+}	1000
Mg^{2+}	1000
K^{+}	1000
Ba^{2+}	1000
Fe^{2+}	700
Fe^{3+}	500
Cr^{3+}	1000
Al^{3+}	1000

obtained. The effluent was sent to AAS for ions content evaluation. The calibration graph exhibits linearity over the range of 0.05–1.20 μg mL^{-1} with a correlation of 0.9998 ($Y = 1.89 \times 10^{-3}C + 1.8 \times 10^{-4}$). The relative standard deviation ($n = 11$) at 200 ng mL^{-1} (sample volume 250 mL) was 0.9% with the detection limit of 1.6 ng mL^{-1} ($3S.D._b/m$, $n = 5$).

In order to investigate reproducibility and repeatability of method at optimum condition eight experiments were repeatedly carried out on the same column and different columns for preconcentration of lead ions. The results show that efficiency for Pb(II) recovery based on SDS coated alumina treated with MBITP is 98.4 ± 0.9 and 99.0 ± 0.7 that indicate repeatable and reproducible results. The results demonstrate that the column can be utilized many times, without reloading the ligand.

3.1.5. Effect of foreign ions

Preconcentration/separation procedures for trace elements in the high salt content samples can be strongly affected by the matrix constituents of the sample. The influences of some alkaline and alkaline-earth ions and transition metal ions on the Pb(II) recovery of the analyte ion were investigated. The results are given in Table 2. The tolerance limit is defined as the ion concentration causing a relative error smaller than ±5% related to the preconcentration and determination of analyte. Pb(II) was quantitatively recovered in the presence of large amounts of alkaline and alkaline-earth ions and some transition metal ions. The matrix ion contents in the eluent solutions were found to be significantly lower and suitable for atomic absorption spectrometric determinations.

Table 4
Lead content of various water samples ($N = 3$; sample volume: 400 mL; eluent volume: 5 mL)

	Concentration (μg L^{-1})
Waste water of Gachsaran city	68.9 ± 1.1[a]
Waste water from oil petroluim Gachsaran	76.9 ± 1.0
Waste water from flour factory in Gachsaran city	59.4 ± 1.2

[a] Mean expressed as 95% tolerance limit.

3.2. Investigation of enrichment factor and loading capacity

For investigation of enrichment factor and break through volume, various volumes of 0.2 μg mL^{-1} of Pb(II) was passed through column. The analyte was eluted by 5 mL 1.0 mol L^{-1} nitric acid in acetone. Lead content of effluent was measured by AAS. The results indicate that lead adsorbed on solid phase and quantitatively was recovered from 1350 to 5 mL. The enrichment factor was 240.

The capacity of immobilized MBITP on surfactant coated alumina on absorption of lead was examined and found to be 0.27 mg/g of solid phase. This indicates that the column is capable of absorbing large amounts of lead.

3.3. Accuracy and applications

We have explored the feasibility of the methodology using preconcentration with MBITP immobilized on surfactant coated alumina for the determination of lead ion in different matrices. The procedure was applied to the determination of lead(II) in different samples, including tap water, spring water and river water by standard addition method. Reliability was checked by spiking experiments and independent analysis. The results for this study are presented in Table 3. The recovery of spiked sam-

Table 3
Recovery studies of trace Pb(II) determination in various real samples

Sample	Added (μg mL^{-1})	Found (μg mL^{-1})	R.S.D. (%)	AAS found (μg mL^{-1})	Recovery (%)
Drinking water	0	0.068	1.1	–	–
	0.5	0.562	0.8	0.57	98.8
River water	0	0.49	0.8	0.55	–
	0.5	1.05	0.5	1.54	106.0
Waste water	0	0.46	0.9	0.44	–
	0.5	0.97	0.4	1.01	102.0
Spring water	0	0.116	1.2	–	–
	0.8	0.930	0.6	1.08	101.8

Table 5
Characteristic performance of some reported SPE of lead ion

E.F.	Capacity	Eluent	pH[a]	D.L.	Sorbent	Reagent	Reference
50	10 μg	0.5 mol L^{-1} HNO_3	6	1	Active Carbon	Pyrogallol Red	[14]
300	0.476 mg	1 mol L^{-1} acetic acid	2–7	0.05	ODSD	BHAQMS	[15]
–	8.6 mg	DMF	8.4–11.5	0.3	Naphthalene	PAN	[16]
–	–	0.03 mol L^{-1} HCl	5	0.86	Sulfohydryl cotton	–	[17]
–	Low	1 mol L^{-1} HCl	6	–	Sepiolite	–	[18]
–	–	HNO_3	–	0.17	Cellulose	PYV	[19]
63	–	Ethanol	–	3	Active carbon	ADEDTP	[20]
–	255 μg	–	–	5	ODSD	DHBPAQ	[21]
–	1.16 mg	Ethanol	9	2	Chromosorb 102	DEDTC	[22]
–	700 μg	–	–	16.7	ODSD	NNBTMEDA	[23]
23	–	0.15 mol L^{-1} HNO_3	10.5	0.002	Silica gel	Macrocycle	[24]
–	–	1 mol L^{-1} HCl	8	0.75	Active carbon	CHDO	[25]
75	–	2 mo L^{-1} HNO_3	9	2.6		SBSEDA	[26]
200	404 μg	4 mol L^{-1} HNO_3	3–7	0.5	SDS coated alumina	Dithizone	[27]
16	41.81 mg	0.05 mol L^{-1} NH_3	10	0.25	Polyurethane foam	TAC	[28]
270	0.27 mg	1.0 mol L^{-1} nitric acid in acetone	3.3–6.8	1.2	Alumina-sodium dodecyl sulfate	MBITP	Present work

E.F.: enrichment factor; D.L.: detection limit (ng ml^{-1}); BHAQMS: bis[1-hydroxy-9,10-anthraquinone-2-methyl] sulfide; PAN: 1-(2-pyridylazo)-2-naphtol; SPE: solid phase extraction; SLE: solid liquid extraction; DMF: dimethylformamide; DB18C6: dibenzo-18-crown-6; ODSD: octadecyl silica membrane disk; PYV: pyrocatechol violet; ADEDTP: ammonium OO-diethyldithiophosphate; DHBPAQ: 1,8-dihydroxy-2,7-bis(prop-1′-enyl)-9,10-anthraquinone; DEDTC: diethyldithiocarbamate; NNBTMEDA: *N*,*N*′-bis(2-thienylmethylene)ethanediamine; CHDO: 1,2-cyclohexanediondioxime; SBSEDA: salen I (*N*,*N*′-bis (salicylidene) ethylenediamine; TAC: thiazolylazo-*p*-cresol; MBITP: 4-(4-methoxybenzylidenimine) thiophenole.
[a] Applicable pH range.

ples is satisfactory reasonable and was confirmed using addition method, which indicate the capability of the system in the determination of lead in natural water samples.

Lead content of the water samples from various sources in Gachsaran (Iran) were also determined by flame atomic absorption spectometry after application of the presented procedure. The results were given in Table 4.

4. Conclusion

The presented procedure is a sensitive and accurate for the determination of lead at low concentrations in natural water samples. The adsorbed lead on the column was simply eluted with 5 mL of 1 mol L^{-1} HNO_3 in acetone. MBITP loaded on SDS coated alumina can be used for at least 10 successive preconcentration of lead without a considerable change. The method due to advantages such as high reliability, reproducibility, sensitivity, and high tolerance limit of common ions and low detection limit is a powerful tool for rapid and sensitive determination of lead ion in various media. The low R.S.D. of real sample analysis is an indication of methods versatility for real sample. Comparative data from some recent studies on solid phase extraction studies are given in Table 5. The proposed method is superior to those reported Pb(II) separation-preconcentration methods in term of selectivity, linear range, detection limit, applicable pH range, capacity, no need to consumption of organic solvents and enrichment factor.

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References

[1] A.K. De, Environmental Chemistry, 3rd ed., New Age International (P) Limited, New Delhi, 1996, p. 263.
[2] R.A. Goyer, C.D. Klaassen, M.O. Amdur, J. Doull (Eds.), Casarett and Doull's Toxicology: The Basic Science of Poisons, 3rd ed., MacMillan Publishing Company, New York, 1986, p. p. 598.
[3] H.W. Nurnberg, Pollutants and their Ecotoxicological Significance, Wiley, Chichester, 1985.
[4] D.R. Lynarn, L.G. Plantanido, J.F. Cole, Environmental Lead, Academic Press, New York, 1975.
[5] J.O. Nriagu, The Biochemistry of Lead in the Environmental, Elsevier, Amsterdam, 1978.
[6] B.P. Lanphear, D.A. Burgoon, S.W. Rust, S. Eberly, W. Galke, Environmental exposures to lead and urban children's blood lead levels, Environ. Res. 76 (1998) 120–127.
[7] M. Ghaedi, F. Ahmadi, H. Karimi, S. Gharaghani, Preconcentration and extraction of copper on activated carbon using 4-amino-2, 3-dimethyl-1-phenyl-3-pyrazoline or 4-(4-methoxybenzylidenimin) thiophenole, J. Korean Chem. Soc. 50 (2006) 23–31.
[8] M. Ghaedi, M.R. Fathi, A. Shokrollahi, F. Shajarat, Highly selective and sensitive preconcentration of mercury ion by cold vapor atomic absorption spectroscopy, Anal. Lett. 39 (2006) 1171–1186.
[9] M. Ghaedi, E. Asadpour, A. Vafaie, Simultaneous preconcentration and determination of copper, nickel, cobalt, lead and iron content using a surfactant coated alumina, Bull. Chem. Soc. Jpn. 79 (2006) 432–436.
[10] M. Ghaedi, A. Shokrollahi, Chromosorb as an alternative suitable support for trace copper enrichment using 2-mercaptobenzoxazole as modifier, Fresen. Environ. Bull., in press.
[11] K. Hiratani, T. Takahashi, H. Sugihara, K. Kasuga, K. Fujiwara, T. Hayashita, R.A. Bartsch, Selective liquid membrane transport of lead(II) by an acyclic polyether dicarboxylic acid ionophore, Anal. Chem. 69 (1997) 3002–3007.
[12] K.Z. Hossain, T. Honjo, Preconcentration and determination of trace amounts of lead (III) as thenoyltrifluoroacetone complex with dibenzo-18-crown-6 by synergistic extraction and atomic absorption spectrometry, Fresen. J. Anal. Chem. 361 (1998) 451–456.
[13] A.L.D. Comitre, B.F. Reis, Automatic flow procedure based on multicommutation exploiting liquid–liquid extraction for spectrophotomet-

ric lead determination in plant material, Talanta 65 (2005) 846–852.
[14] A.A. Ensafi, T. Khayamian, M.H. Karbasi, On-line preconcentration system for lead(II) determination in waste water by atomic absorption spectrometry using active carbon loaded with pyrogallol red, Anal. Sci. 19 (2003) 953–956.
[15] M. Shamsipur, F. Raoufi, H. Sharghi, Solid phase extraction and determination of lead in soil and water samples using octadecyl silica membrane disks modified by bis[1-hydroxy-9,10-anthraquinone-2-methyl]sulfide and flame atomic absorption spectrometry, Talanta 52 (2000) 637–643.
[16] M.A. Taher, Flame atomic absorption spectrometric determination of trace lead after solid–liquid extraction and preconcentration using 1-(2-pyridylazo)-2-naphthol, Croat. Chim. Acta 76 (2003) 273–277.
[17] S. Saracoglu, M. Soylak, D.S.K. Peker, L. Elci, W.N.L. dos Santos, V.A. Lemos, S.L.C. Ferreira, A pre-concentration procedure using coprecipitation for determination of lead and iron in several samples using flame atomic absorption spectrometry, Anal. Chim. Acta 575 (2006) 133–137.
[18] A.R. Turker, H. Bag, B. Erdogan, Determination of iron and lead by flame atomic absorption spectrometry after preconcentration with sepiolite, Fresen. J. Anal. Chem. 357 (1997) 351.
[19] A.M. Naghmush, K. Pryzynska, M. Trojanowicz, Flame AAS determination of lead in water with flow-injection preconcentration and speciation using functionalized cellulose sorbent, Talanta 42 (1995) 851–860.
[20] J.B.B. da-Silva, S.P. Quinaia, M.C.E. Rollemberg, On-line preconcentration with different solid adsorbents for lead determination, Fresen. J. Anal. Chem. 369 (2001) 657–662.
[21] F. Raoufi, Y. Yamini, H. Sharghi, M. Shamsipur, Solid-phase extraction and determination of trace amounts of lead(II) using octadecyl silica membrane disks modified with a recently synthesized anthraquinone derivative and atomic absorption spectrometry, Microchem. J. 63 (1999) 311–316.
[22] L. Elci, Z. Arslan, J.F. Tyson, Flow injection solid phase extraction with Chromosorb 102: determination of lead in soil and waters by flame atomic absorption spectrometry, Spectrochim. Acta Part B 55 (2000) 1107–1114.
[23] O.R. Hashemi, M.R. Kargar, F. Raoufi, A. Moghimi, H. Aghabozorg, M.R. Ganjali, Separation and preconcentration of trace amounts of lead on octadecyl silica membrane disks modified with a new S-containing Schiff's base and its determination by flame atomic absorption spectrometry, Microchem. J. 69 (2001) 1–6.
[24] X.P. Yan, M. Sperling, B. Welz, Determination of (ultra) trace amounts of lead in biological materials by on-line coupling flow injection microcolumn separation and preconcentration to electrothermal atomic absorption spectrometry using a macrocycle immobilized silica gel sorbent, J. Anal. At. Spectrom 14 (1999) 1625–1630.
[25] A. Uzawa, T. Narukawa, T. Okutani, Determination of trace amounts of copper and lead by tungsten metal furnace atomic absorption spectrometry after preconcentration with activated carbon impregnated with 1,2-cyclohexanediondioxime, Anal. Sci. 14 (1998) 395–398.
[26] S. Dadfarnia, A.M.H. Shabani, F. Tamaddon, M. Rezaei, Immobilized salen (*N*,*N*′-bis (salicylidene) ethylenediamine) as a complexing agent for on-line sorbent extraction/preconcentration and flow injection-flame atomic absorption spectrometry, Anal. Chim. Acta 539 (2005) 69–75.
[27] S. Dadfarnia, A.M.H. Shabani, H.D. Shirie, Determination of lead in different samples by atomic absorption spectrometry after preconcentration with dithizone immobilized on surfactant-coated alumina, Bull. Korean Chem. Soc. 23 (2002) 545–548.
[28] O.D.S. Ana, L.S. Jesuino, R.J. Cassella, M.S. Carvalho, R.E. Santelli, Determination of lead by electrothermal atomic absorption spectrometry employing a novel sampling strategy of polyurethane foam impregnated with thiazolylazo-*p*-cresol (TAC), J. Br. Chem. Soc. 15 (2004) 96–102.
[29] M. Hiraide, J. Iwasawa, H. Kawaguchi, Collection of trace heavy metals complexed with ammonium pyrrolidinedithiocarbamate on surfactant-coated alumina sorbents, Talanta 44 (1997) 23–237.
[30] J.L. Manzoori, M.H. Sorouradin, A.M.H. Shabani, Atomic absorption determination of cobalt after preconcentration by 1-(2-pyridylazo)-2-naphthol immobilized on surfactant-coated alumina, Microchem. J. 63 (1999) 295–301.
[31] J.L. Manzoori, M.H. Sorouradin, F. Shemirani, Chromium speciation by a surfactant-coated alumina microcolumn using electrothermal atomic-absorption spectrometry, Talanta 42 (1995) 1151–1155.
[32] M. Soylak, Determination of trace amounts of copper in metallic aluminium samples after separation and preconcentration on an activated carbon column, Fresen. Environ. Bull. 7 (1998) 383–387.
[33] S. Baytak, A.R. Turker, Atomic absorption spectrometric determination of Fe(III) and Cr(III) in various samples after preconcentration by solid-phase extraction with pyridine-2-carbaldehyde thiosemicarbazone, J. Anal. Chem. 61 (2006) 483–489.
[34] V.A. Lemos, L.N. Santos, A.P.O. Alves, G.T. David, Chromotropic acid-functionalized polyurethane foam: A new sorbent for on-line preconcentration and determination of cobalt and nickel in lettuce samples, J. Sep. Sci. 29 (2006) 1197–1204.
[35] J.M. Choi, H.M. Park, S.D. Choi, Application of synergistic solvent extraction by formation of ternary complex for determination of trace Zn(II) in water samples, Bull. Korean Chem. Soc. 27 (2006) 563–567.
[36] M. Soylak, L. Elci, Y. Akkaya, M. Dogan, On-line preconcentration system for lead determination in water and sediment samples by flow injection-flame atomic absorption spectrometry, Anal. Lett. 35 (2002) 487–499.