Talanta 116 (2013) 882–886

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Ionic liquid-linked dual magnetic microextraction of lead(II) from environmental samples prior to its micro-sampling flame atomic absorption spectrometric determination

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ARTICLE INFO

Article history:
Received 22 March 2013
Received in revised form
1 August 2013
Accepted 4 August 2013
Available online 9 August 2013

Keywords:
Ionic liquid-linked dual magnetic microextraction
Ammonium pyrrolidinedithiocarbamate
Fe_3O_4 magnetic nanoparticles
Lead
Ionic liquid

ABSTRACT

A novel and rapid microextraction approach termed as ionic liquid-linked dual magnetic microextraction (IL-DMME), was developed for the atomic absorption spectrometric determination of lead. The developed method based on a combination of dispersive liquid–liquid microextraction (DLLME) and dispersive micro solid-phase extraction (D-μ-SPE). In the first DLLME step, 1-butyl-3-methylimidazolium hexafluorophosphate $[C_4mim][PF_6]$, was selected to extract the lead-pyrrolidine-dithiocarbamate (Pb-PDC) complex from sample solution by the assistance of vortex agitator. After the first step, fifty milligrams of Fe_3O_4 magnetic nanoparticles (MNPs) were added to extraction of the ionic liquid and Pb-PDC complex in aqueous solution. The effective factors in proposed IL-DMME procedure, including volume of 1-butyl-3-methylimidazolium hexafluorophosphate, amount of Fe_3O_4 magnetic nanoparticles, vortex time, amount of ammonium pyrrolidinedithiocarbamate, sample volume and matrix effect were optimized in details. Under the optimal conditions, the method present has low detection limit (0.57 μg L^{-1}), high preconcentration factor (160) and good repeatability ($<7.5\%$, $n=10$). The accuracy of the developed method was evaluated by the analysis of the certified reference materials and addition-recovery tests. The method was successfully applied to the determination of lead in water, plant and hair samples.

1. Introduction

Lead is one of the most dangerous toxic elements and has deleterious effects on multiple organs of the human body [1,2]. Due to adverse effects of lead at trace levels for human life and for environment, spectrometric determination of trace and ultra trace lead level in water, food and biological samples have become of increasing significance for analytical chemists [3–6]. Due to lower level of analyte elements than the detection limit of detection system, enrichment-separation procedures have been used prior to these determinations [7–10]. In these days, the usages of microextraction techniques are very popular for the enrichment-separation of metal ions at trace levels [11–15].

Up to now, many miniaturized preconcentration-separation methods such as headspace liquid phase microextraction (HS-LPME) [16], cold-induced aggregation microextraction (CIAME) [17], in situ solvent formation microextraction (ISFME) [18] and dispersive liquid–liquid microextraction (DLLME) [19] have been developed not only to reduce solvent use but also to benefit from the ability of this approach to economic and convenient determination of metals and organic compounds at trace levels in real samples.

Ionic liquids have some good physical and chemical properties, such as negligible vapor pressure, non-flammability, good solubility for inorganic and organic compounds and good extractability for organic compounds and metal ions [20]. Ionic liquids are very important and green alternative to organic solvents in classical extraction techniques [20–22]. Various works have been presented by the usage of ionic liquids on the preconcentration and separation of organic and inorganic analytes at trace levels [23–26].

The main disadvantages of IL microextraction studies are the necessity of using dispersive solvents like acetonitrile, acetone, 2-propanol, n-butanol and methanol [27], isolation of IL from water phase, loss of IL at the isolation step and low recoveries of the analytes. In order to overcome disadvantages mentioned above, very recently, a new combinated microextraction procedure which is termed as ionic liquid-linked dual magnetic microextraction (IL-DMME) was developed and successfully used for the fast and efficient determination of benzoylurea insecticides in environmental water samples [28]. This method shows a nice combination between ionic liquid dispersive liquid–liquid microextraction (IL-DLLME) and dispersive μ- magnetic nanoparticle solid-phase extraction (D-μ-SPE). Also Li et al. [29] and Mukdasai et al. [30]

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http://dx.doi.org/10.1016/j.talanta.2013.08.002

have been used this combination with some modification for the preconcentration and separation of pyrethroids and carbaryl, respectively.

The objective of the present study is to develop a novel and simple microextraction method, ionic liquid-linked dual magnetic nanoparticle microextraction (IL-DMME), as an alternative new method for the preconcentration and determination of lead in water, plant and hair samples prior to its micro sampling-flame atomic absorption spectrometric determinations.

2. Experimental

2.1. Instrumentation

A Perkin-Elmer Model 3110 model flame atomic absorption spectrometer (FAAS) (Norwalk, CT, USA) including air–acetylene flame and a hollow cathode lamp was used for measurement of lead concentration. The instrumental parameters were adjusted as recommended by the manufacturer. The samples were introduced to the nebulizer of the FAAS by using a micro injection unit [31,32]. A 100-μL amount of the samples was injected to a mini home-made Teflon® funnel with an Eppendorf® pipette that was connected to the nebulizer with capillary tubing.

VWR international model (Germany) vortex mixer was used during the experimental period. A Nel pH-900 (Ankara-Turkey) pH meter with a combined glass electrode was used for pH measurements.

2.2. Reagents and solutions

All chemicals were of the analytical grade. 0.3% (m/V) solution of ammonium pyrrolidinedithiocarbamate (APDC) supplied from Merck, Darmstadt, Germany, was prepared in ethanol. Distilled/deionized water (Millipore Milli-Q system 18 MΩ cm^{-1} resistivity) was used for all experiments. Standard solutions of Pb(II) (1000 mg L^{-1}) were prepared by dissolving in water from nitrate salt (Merck).

1-butyl-3-methylimidazolium hexafluorophosphate [C_4mim][PF_6] (Merck no: 4.90065.0025) was obtained from Merck (Germany). The phosphate buffer solutions (pH 2.0–4.0), acetate buffer solution (pH 5.0–6.0) and phosphate buffer (pH 7.0) were used to adjust of pH of sample solutions.

NCS ZC 81002b human hair (NCS Beijing, China), TMDA-54.4 (National Water Research Institute, Ontario, Canada), TMDA-70 Ontario lake water (National Water Research Institute, Ontario, Canada), TMDA-64.2 Water (National Water Research Institute, Ontario, Canada) and SPS-WW2 Waste water (Spectrapure Standards AS, Oslo, Norway) certified reference materials were used.

Fe_3O_4 nano-particles were synthesized by using the earlier report [33]. Primarily, 5.2 g of $FeCl_3.6H_2O$ and 2.0 g of $FeCl_2.4H_2O$ were dissolved in 50 mL of deionized water. Afterward, argon gas was passed in the solution for 15 min at 80 °C. Then, 10 mL concentrated NH_3 was added slowly to the solution using a dropping funnel. The pure black solid product was formed and separated using a magnet after 15 min and washed three times with 50 mL solution of 0.1 mol L^{-1} NaCl and once with ethanol and dried at 80 °C in oven at 24 h.

2.3. Ionic liquid-linked dual magnetic microextraction procedure

Fifteen milliliters of an aqueous solution containing 33.3 μg L^{-1} of Pb(II), 0.75 mg ammonium pyrrolidinedithiocarbamate and 2.0 mL of 2.0 mol L^{-1} phosphate buffer (pH 3.0) were placed in a test tube. Then, 175.0 μL of the ionic liquid was injected into the sample solution and the mixture was vigorously shaken using a vortex agitator for 1 min at 2800 rpm to accelerate the formation of the fine droplets of the IL phase and to enhance the extract of the Pb(II). After this process, Fifty milligrams of Fe_3O_4 magnetic nanoparticles were added into the tube. The mixture was again vigorously shaken by the same vortex for 1.5 min. at 2800 rpm. The IL phase and the remaining Pb-PDC complex in aqueous solution were successfully extracted to MNP phase after the high-speed vortex process.

A magnet was subsequently held around the test tube to collect the nanoparticles at the bottom of the test tube. The upper aqueous phase was removed and completed to 250 μL with concentrated HNO_3. Then the solution was vortexed for 30 s to desorb the IL as well as Pb(II) from the MNPs. Finally, the nanoparticles were isolated from solution with a magnet, and 40 μL of sample was aspirated into the FAAS nebulizer using a microinjection system and the peak height in continuous aspiration mode was measured.

2.4. Application to real samples

The plant samples (squash, peppermint, potato, eggplant and broccoli) and herbal tea (dried flowers of linden tree for linden tea) was obtained from local markets of Kayseri. The scalp hair samples were taken from a child live in Kayseri and a male live in Kayseri who is a worker in battery factory, Turkey. The plant and hair samples were dried at temperature 80 °C for 24 h and plant samples were homogenized with the help of an agate mortar and pestle. Edible parts of each plant samples were digested and analyzed.

A wet ashing procedure was used to digestion of all plant and hair samples. Two hundred fifty mg of each sample were directly weighed into beakers and were treated with 15 mL of concentrated nitric acid (65% w/w) on hot plate until dryness at about 100 °C. After cooling, the residue was again treated with mixtures of concentrated nitric acid (15 mL) and hydrogen peroxide (5 mL) on hot plate until dryness. Digested samples were dissolved with 5 mL distilled water. Then the proposed IL-DMME procedure given Section 2.3 was applied to the samples.

The water samples including waste water from Yahyali village-Kayseri, water from a well which is an excavation in the ground at Incesu village- Kayseri, water from Sarmisakli Dam Lake- Kayseri and water from Yamula Dam Lake, Kayseri were collected at autumn of 2012. Before use, each water samples were filtered through a cellulose membrane filter of 0.45 μm (Millipore). The proposed IL-DMME procedure was applied to the samples mentioned above.

3. Results and discussion

3.1. Effects of pH

The pH of the working media in the separation-preconcentration studies is an important parameter for the complex formation and quantitative recoveries of the analytes [34–36]. Lead forms stable complexes with APDC at acidic pHs [12,35–37]. The influences of pH of the studying media on the recovery values of lead for the IL-DMME procedure were investigated in the pH range of 2.0–7.0. The results for pH were depicted in Fig. 1. The quantitative recovery values (> 95%) were obtained in the pH range of 2.0–5.0. This range is agreement with separation-preconcentration works for Pb-PDC [12,35–37]. So for all further works, pH 3.0 is selected as optimal working pH.

3.2. Effect of the volume of ionic liquid

The volume of IL used in preconcentration procedure based on microextraction techniques was a crucial parameter for obtaining high performance and a high volume ratio of phases [38,39].

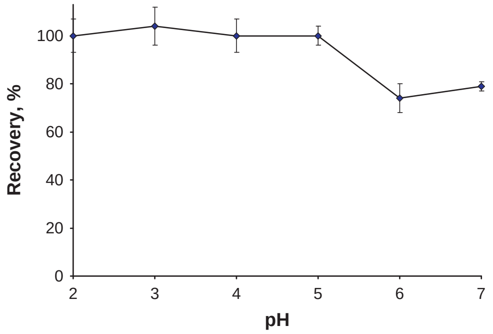

Fig. 1. Effect of the pH on the recoveries of lead(II) (IL volume = 150 µL, amount of Fe_3O_4 = 50 mg, and amount of APDC = 0.75 mg, N = 3).

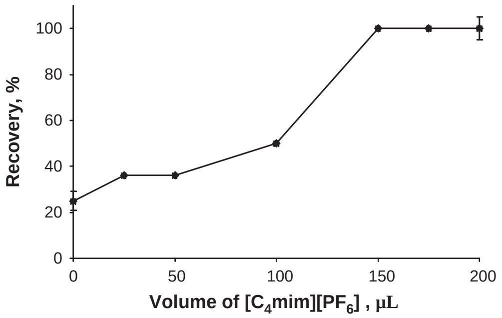

Fig. 2. Effect of the volume of $[C_4mim][PF_6]$ on the extraction efficiency of Pb(II) (N = 3, pH = 3, amount of Fe_3O_4 = 50 mg, and amount of APDC = 0.75 mg).

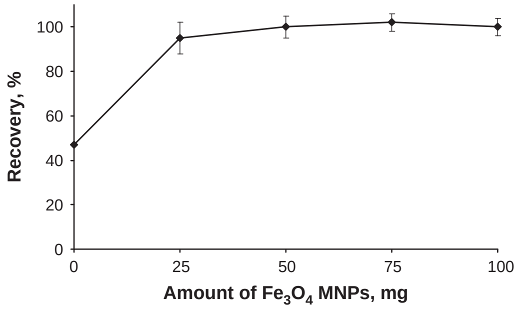

Fig. 3. Effect of the amount of Fe_3O_4 MNPs on the extraction efficiency of Pb(II) (N = 3, pH = 3, amount of APDC = 0.75 mg, and volume of IL = 175 µL).

Hence, it should be optimized precisely. The effect of the IL volume on the recovery of Pb(II) was studied in the range of 0.0–200 µL keeping other parameters constant. As shown in Fig. 2, the recoveries of lead(II) without IL was about 25%. The recoveries increased with the increasing amounts of IL and the quantitative recoveries were obtained between 150 µL and 200 µL of IL. These show that, the usage of IL in the presented work is the necessity for the quantitative recoveries (> 95%) of lead ions. All further works were performed by using 175 µL of IL.

3.3. *Effect of the amount of MNP*

Compared to conventional nano-microsized adsorbents, MNPs offer high extraction capacity, rapid extraction dynamics and high extraction efficiencies [40]. The amount of MNP has a direct effect on the extraction of Pb(II). The amount of the Fe_3O_4 MNPs was optimized in the range of 0.0–100 mg. The results are depicted in Fig. 3. Without Fe_3O_4 MNPs with optimal amounts of IL, the recovery value for lead(II) was about 45%. After addition Fe_3O_4 MNPs, the recoveries are increased and to reach quantitative value at 25 mg of Fe_3O_4 MNPs. These increases in the recoveries of lead are related with the adsorption of Pb-PDC complex on the surface of MNPs. Fifty mg of MNPs was selected for subsequent experiments.

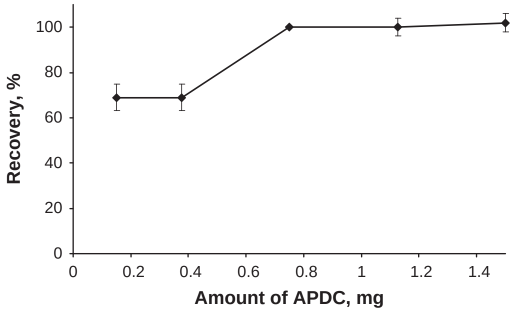

Fig. 4. Effect of the amount of APDC on the extraction efficiency of Pb(II) (N = 3, pH = 3, amount of Fe_3O_4 MNPs = 50 mg, and volume of IL = 175 µL).

3.4. *Effect of the amount of APDC*

The influence of amount of APDC on the extraction efficiency of Pb(II) ions was studied in the amount range of 0.15–1.5 mg (50–500 µL of 0.3% (m/V) solution). The results were given in Fig. 4. The amount of APDC was increased until the quantitative extraction of Pb(II) was obtained. In the range of 0.15–0.75 mg of APDC, due to insufficient amounts of ADPC, the recoveries were not quantitative. The extraction efficiency was quantitative in the range of 0.75–1.5 mg of APDC. Therefore, the amount of APDC was selected as 0.75 mg (250 µL of 0.3% (m/V) solution) for the preconcentration of Pb(II).

3.5. *Effect of the vortex time*

It is well-known that vortex process is an effective way to enhance the mass transfer from aqueous phase to extraction phase for all microextraction techniques [41,42]. Therefore, vortex time and speed for DLLME and D-µ-SPE steps were optimized under the optimum conditions. The obtained results show that 1.0 min. and 1.5 min. vortex times at 2800 rpm vortex speed were adequate for DLLME and D-µ-SPE steps, respectively.

3.6. *Effect of the sample volume*

The sample volume is a key factor due to the low concentration of lead in real samples. The lead in these samples should be extracted into smaller volume to obtain high preconcentration factor [43–45]. Hence, as large a volume of sample solutions as possible was optimized. The effects of sample volume on the recovery of lead were investigated range of 15.0–50.0 mL for 0.5 µg L^{-1} of Pb(II) solution keeping other conditions constant. The recovery values of the Pb(II) were not affected by sample volume below 40.0 mL. Above 40.0 mL the recovery of Pb(II) was not quantitative. This point shows that we could use the presented procedure in the sample volume range of 15–40 mL.

3.7. *Effects of potential interfering species*

The alkali, alkaline earth and transition metal at high concentrations are affected atomic absorption spectrometric determination of metals at trace levels [46–49]. The effect of some potentially interfering ions in real samples on extraction of Pb(II) was evaluated by adding various concentration of possible interfering ions to the model solutions including 0.5 µg mL^{-1} Pb(II). The maximum tolerance limits of the investigated ions are given in Table 1. The high concentration of common anions, alkali and alkaline earth metal ions do not interfere with determination of Pb(II). While Co(II), Ni(II), Mn(II), Cd(II), Fe(III), Hg(II) and Zn(II) ions showed interference effect on determination of Pb(II) due to complex formation of these metals with PDC.

3.8. Analytical Figures

The limit of detection and limit of quantification for lead under the optimum extraction conditions (LOD=3 s, three times of the standard deviation of the blank signals, LOQ=10 s, ten times of the standard deviation of the blank signals), were 0.57 μg L^{-1} and 1.9 μg L^{-1}, respectively. The calibration curve was [$A=-0.619\times 10^{-3}$+0.681C], where A is the absorbance and C is Pb(II) concentration in mg L^{-1} linear with a correlation coefficient of 0.991.

The relative standard deviation (RSD) of the developed method, determined by analyzing the standard solution at 7.5 μg L^{-1} of Pb(II) (n=10), was < 7.5%. The enhancement factor that, calculated from analytical curves with and without the preconcentration step, was 139. A preconcentration factor of 160 can be achieved when using 40.0 mL of the sample and 0.25 mL of final volume.

Table 1
Effect of some matrix ions on the extraction efficiency of Pb(II) (N=3, pH=3, amount of Fe_3O_4 MNPs=50 mg, Volume of IL=175 μL, and amount of APDC=0.75 mg).

Matrix ion	Added as	Tolerable concentration (mg L^{-1})
Na(I)	$NaNO_3$	2500
K(I)	KCl	5000
Mg(II)	$Mg(NO_3)_2\ 6H_2O$	2500
Ca(II)	$Ca(NO_3)_2\ 4H_2O$	2500
Co(II)	$Co(NO_3)_2\ 6H_2O$	25
Ni(II)	$Ni(NO_3)_2\ 6H_2O$	25
Mn(II)	$Mn(NO_3)_2\ 4H_2O$	2.5
Zn(II)	$Zn(NO_3)_2\ 6H_2O$	25
Cd(II)	$Cd(NO_3)_2\ 4H_2O$	5
Hg(II)	$Hg(NO_3)_2\ H_2O$	40
Fe(III)	$Fe(NO_3)_3\ 9H_2O$	10
Cu(II)	$Cu(NO_3)_2\ 3H_2O$	40
Al(III)	$Al(NO_3)_3\ 9H_2O$	40
Cl^-	KCl	5000
SO_4^{2-}	Na_2SO_4	1000

Table 2
Determination of lead in certified reference materials after application of IL-DMME procedure (N=3, pH=3, amount of Fe_3O_4 MNPs=50 mg, volume of IL=175 μL, and amount of APDC=0.75 mg).

Certified reference material	Found[a]	Certified value	Recovery (%)
TMDA 54.4 Fortified Lake Water	527 ± 28 μg L^{-1}	514 ± 5 μg L^{-1}	103
TMDA-70 Ontario Lake Water	451 ± 25 μg L^{-1}	444 ± 7 μg L^{-1}	102
TMDA-64.2 Water	291 ± 23 μg L^{-1}	286 ± 12 μg L^{-1}	102
SPS-WW2 Waste Water	492 ± 40 μg L^{-1}	500 ± 3 μg L^{-1}	98
NCS ZC 81002b Human Hair	3.93 ± 0.08 μg g^{-1}	3.83 ± 0.18 μg g^{-1}	103

[a] Mean of three determinations ± standard deviation.

3.9. Applications to real samples

The accuracy of the developed method was evaluated by the analysis of the certified reference materials. Under the optimum conditions, the recommended procedure was applied for determination of lead in the certified reference materials. As shown in Table 2, the determined values were in good agreement with the certified values.

Also to verify of the presented method, addition-recovery experiments in the presence of digested eggplant, digested broccoli and well water samples were carried out. 0.5 and/or 1.0 μg of lead were spiked to these samples. The sample volume was 15 mL at the addition-recovery experiments (Table 3). The recoveries of analyte for the spiked samples were in the acceptable range of 97–101%. The results obtained addition-recovery experiments show that the developed method was reliable, suitable and free from interferences for analysis of wide range of samples.

We also used the proposed method to determination of lead concentration in various water, plant and hair samples collected from Kayseri, Turkey. The results are given in Table 4.

3.10. Comparison with other preconcentration techniques

The presented IL-DMME procedure was compared with the other preconcentration methods used for the determination of lead in real samples (Table 5). Our method shows high preconcentration factor and low detection limit with reasonable operation time (2.5 min). All these results indicate that IL-DMME is a simple and low cost technique. Any toxic dispersive solvent used conventional DLLME like ethanol, acetone, acetonitrile was not used. The important steps of the conventional microextraction techniques like centrifugation, cooling of sample solution after extraction and the use specific

Table 4
Concentration of Pb(II) in water, plant and hair samples after application of IL-DMME procedure (N=3, pH=3, amount of Fe_3O_4 MNPs=50 mg, volume of IL=175 μL, and amount of APDC=0.75 mg).

Sample	Concentration[a]
Water from Yamula Dam Lake, Kayseri	3.2 ± 0.3 μg L^{-1}
Water from Sarmisakli Dam Lake, Kayseri	1.3 ± 0.0 μg L^{-1}
Waste water from Yahyali village, Kayseri	3.1 ± 0.0 μg L^{-1}
Squash	0.49 ± 0.02 μg g^{-1}
Peppermint	0.61 ± 0.03 μg g^{-1}
Potato	0.60 ± 0.05 μg g^{-1}
Dried flowers of linden tree for linden tea	0.68 ± 0.03 μg g^{-1}
Hair from a child	25.5 ± 2.8 μg g^{-1}
Hair from a worker in battery factory	326 ± 6 μg g^{-1}

[a] Mean of three determinations ± standard deviation.

Table 3
Addition and recovery of Pb(II) from water and plant samples after application of IL-DMME procedure (N=3, pH=3, amount of Fe_3O_4 MNPs=50 mg, volume of IL=175 μL, and amount of APDC=0.75 mg).

Added (μg)	Water from a well at Incesu village, Kayseri		Eggplant		Broccoli	
	Found[a] (μg)	Recovery (%)	Found (μg)	Recovery (%)	Found (μg)	Recovery (%)
0.00	0.25 ± 0.03	–	0.44 ± 0.02	–	0.46 ± 0.04	–
0.50	0.73 ± 0.04	97	0.95 ± 0.08	101	0.94 ± 0.12	98
1.00	1.24 ± 0.12	99	1.39 ± 0.04	97	1.43 ± 0.09	98

[a] Mean of three determinations ± standard deviation.

Table 5
Comparison of the IL-DMME with other methods for the determination of lead.

Method	LOD ($\mu g\ L^{-1}$)	EF	Samples	Reference
CPE-FAAS	4.5	19.6	Water	[50]
SPE-FAAS	0.61	125	Water, food	[51]
DLLME-FAAS	0.6	273	Table salt, water	[52]
CPC-FAAS	2.5	25	Water, food	[53]
SFODME-GFAAS	0.0009	500	water	[54]
IL-DMME-FAAS	0.57	160	Water, plant, hair	Present work

LOD: Limit of detection, *EF:* Enrichment factor, *CPE:* Cloud point extraction, *SPE:* Solid phase extraction, *IL-DLLME:* Ionic liquid-dispersive liquid–liquid microextraction, *CPC:* Coprecipitation method, *SFODME:* Solidified floating organic drop microextraction, *FAAS:* Flame atomic absorption spectrometry, *GFAAS:* Graphite furnace atomic absorption spectrometry.

equipment like conical-bottom centrifuge tubes were eliminated by using the presented work.

4. Conclusions

A novel and simple ionic liquid-linked dual magnetic microextraction (IL-DMME) was developed for fast and effective determination of lead combined with FAAS. In our report, [C_4mim][PF_6] was used to extract complex of Pb-PDC in DLLME step without organic solvents. In the D-μ-SPE step of microextraction method, Fe_3O_4 magnetic nanoparticles were used to extraction of the ionic liquid and Pb-PDC complex. Because of using MNPs and IL in the IL-DMME, the procedure can be described as environmentally friendly. No specific equipment, time-consuming and complex processes used other conventional preconcentration methods are required. The main advantage of the method is that can be applied to water, plant and hair samples without matrix interferences.

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