Ion chromatography of heavy and transition metals by on- and post-column derivatizations

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Abstract

A new method for the simultaneous separation and determination of mercury(II) with other heavy and transition metals by ion chromatography (IC) was developed. This method involves on-column derivatization with 2,6-pyridinedicarboxylic acid (PDCA) and anion-exchange separation, followed by a post-column reaction with 2-[(5-bromo-2-pyridyl)-azo]-5-diethylaminophenol (5-Br-PADAP) to form metal–5-Br-PADAP chelates, which can be sensitively monitored by spectrophotometric detection at 565 nm. PDCA was first combined with Na₂C₂O₄ to separate the metals within 16 min. The optimum separation and derivatization conditions were studied in detail. The detection limits of Pb²⁺, Cu²⁺, Ni²⁺, Zn²⁺, Co²⁺, Cd²⁺, Mn²⁺ and Hg²⁺ were 0.2 mg l⁻¹ (115 µl loop), 1.5, 5, 0.5, 5, 5, 3 and 20 µg l⁻¹ (676 µl loop), respectively. This method was applied to the analysis of standard rice sample, waste cellpacking and municipal solid waste fly ash with satisfying results. ©2000 Elsevier Science B.V. All rights reserved.

Keywords: Ion chromatography; Mercury; Transition metals; Post-column derivatization

1. Introduction

The analysis of heavy and transition metals especially mercury in environmental and biological samples has received increasing attention in recent years [1]. As, from the ecotoxicological point of view, Hg²⁺, Pb²⁺ and Cd²⁺ etc. are the most toxic metals, they can accumulate in various organisms. So it is an important task to establish a rapid and convenient method for the simultaneous determination of these metals.

Numerous analytical techniques have been employed in the determination of heavy and transition metals. But ion chromatography (IC) seems to be one of the most effective and simple techniques to determine both anionic and cationic species owing to its high sensitivity, rapidity, selectivity and simplicity coupled with the possible advantage of simultaneous determination [2]. The widely used cationic IC methods involve a cation-exchange (or a mixed cation- and anion-exchange) separation followed by post-column complexation with 4-(2-pyridylazo)resorcinol (PAR), to form chelates which can be sensitively monitored by spectrophotometric detection [3]. The anion-exchange approach did offer some advantages with respect to selectivity and for the analysis of

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complex samples [4]. At present, the bifunctional ion-exchange column is the most effective analytical column for separating heavy and transition metals [1]. The commonly used eluent is oxalic acid (Ox) or pyridine 2,6-dicarboxylic acid (PDCA). When Ox was used, cadmium and manganese coeluted. Furthermore, iron and aluminum could not be eluted from the analytical column. So PDCA was found to be of particular interest because it is such a strong complexing agent that heavy and transition metals could be well separated as their anionic chelates $M(\text{PDCA})_2^{2-}$ on the mixed-bed resin of the analytical column [5]. Ruth and Shaw [6] could separate Pb^{2+} , Fe^{3+} , Cu^{2+} , Ni^{2+} , Zn^{2+} , Co^{2+} , Cd^{2+} and Mn^{2+} , using 3 mmol l^{-1} PDCA/ 4.3 mmol l^{-1} LiOH/ 2 mmol l^{-1} Na_2SO_4 / 25 mmol l^{-1} NaCl as the eluent and 1 mmol l^{-1} PAR/ 3 mol l^{-1} NH_4OH (adjusted to pH 11 with acetic acid) as the post-column derivatization reagent. However, not many papers describe the simultaneous determination of mercury with other heavy and transition metals using IC. Yan et al. [7] have used the IC method to analyze Zn^{2+} , Cd^{2+} and Hg^{2+} simultaneously by post-column derivatization with a water-soluble porphyrin; the detection limits for Zn^{2+} , Cd^{2+} and Hg^{2+} were 1, 0.05 and $5 \mu\text{g l}^{-1}$, respectively.

This paper is based on the previous work in this laboratory [8] and presents a simple IC method for the determination of Pb^{2+} , Cu^{2+} , Ni^{2+} , Zn^{2+} , Co^{2+} , Cd^{2+} , Mn^{2+} and Hg^{2+} in one injection. 2-[(5-Bromo-2-pyridyl) azo]-5-diethylaminophenol (5-Br-PADAP) was used as the derivatizing agent instead of the commonly used PAR because its metal complexes showed high molar absorptivities ($>10^5$) in the range 550–570 nm and a relatively low background absorbance. Linearity, reproducibility, detection limits and interference due to alkaline earth metals (noticeably Mg^{2+} and Ca^{2+}) were evaluated. The influence of injection volume (up to 1 ml) on the chromatogram was studied.

The method was applied to determine the content of mercury with other heavy and transition metals in standard rice sample and the results were satisfactory. As a further application of this method, waste cell-packing and municipal solid waste fly ash that seriously polluted the environment were analyzed without any pre-treatment after being digested and the results were satisfactory.

2. Experimental

2.1. Instrumentation

Chromatographic analyses were performed on a metal-free Dionex-4000i ion chromatography (Dionex, Sunnyvale, CA, USA), which included one gradient pump GPM, a pneumatic controller for post-column reagent addition, and a VDM-2 variable wavelength absorbance detector at 565 nm.

A Dionex IonPac CG5A guard column (50 mm $\times$ 4 mm ID) and an IonPac CS5A analytical column (250 mm $\times$ 4 mm ID, 9 μm bead diameter, ethylvinyl benzene-functionalized with both quaternary ammonium and sulfonate functional groups) were used to separate the heavy and transition metals. All measurements were made at room temperature and all samples were filtered through a 0.45 μm filter prior to injection.

Data collection and the operation of all components in the system were controlled by Dionex AI-450 chromatographic software interfaced via an ACI-2 advanced computer interface to a 80486 based computer.

The spectrophotometric experiments were carried out by using a Shimadzu SP-UV-3000 spectrophotometer (Kyoto, Japan) with 1 cm glass cells.

2.2. Reagents and standards

All reagents were analytical grade unless specified otherwise. All solutions were prepared with deionized water throughout. Lithium hydroxide monohydrate, sodium oxalate, sodium sulfate anhydrous, sodium hydroxide, amino acetic acid, 2-Br-PADAP, ethyl alcohol (Peking Chemical Works, Peking, China), hydrogen peroxide 30% solution (Tianjin Dongfang Chemical Works, Tianjin, China), sodium chloride (Peking Shunghuan Reagent Factory, Peking, China), TritonX-100 (Carl Roth KG Chemische Fabrik Karlsruhe). Nitric acid (Peking Chemical Reagent Institute, Peking, China) and PDCA (Aldrich, USA) were of chromatographic grade.

Working standard solutions were prepared daily by serial dilution of stock standard solutions of each metal containing 1000 mg l^{-1} (National Research Center for Certified Reference Materials, Peking, China).

All standards and samples were stored in polyethylene bottles that had been cleaned and conditioned

following the recommended procedures for trace metal determination [9,10].

2.3. Sample preparation

A 2.0 g standard rice powder sample (National Research Center for Certified Reference Materials, Peking, China, GBW08508) was weighed and placed in a 50 ml ground-glass flask followed by a few drops of deionized water to moisten the sample. 8 ml concentrated nitric acid (65%) was added. The flask was capped and placed in darkness overnight. A funnel was put above the flask, a few drops of 30% H_2O_2 were added to the flask and then carefully heated on an electrothermic plate. Thirty percent H_2O_2 was added continuously until the brown fume disappeared completely which indicated that the organic compound had been digested completely. Then, the solution was evaporated to almost dryness and diluted with water to 25 ml. A 1.9346 g waste 7[#] Nanfu cellpacking sample (Fujian Nanping Battery Limited Company, Fujian, China) was weighed out in a 50 ml beaker, a few drops of concentrated HNO_3 and 30% H_2O_2 were carefully added to dissolve the sample, and then the sample was diluted to 100 ml. 2.0 g municipal solid waste fly ash sample was digested according to the standard method mentioned in [11] and diluted to 50 ml. All sample solutions were directly injected after being filtered through a 0.45 μm filter.

3. Results and discussion

3.1. Chromatographic separation

Since the IonPac CS5A has both anion- and cation-exchange capacity, metals can be separated by cation- and anion-exchange. Thus, major selectivity changes can be made by simply changing the eluent. When the metals were separated with oxalate as the eluent, the separation comprised both anion- and cation-exchange components. Since Pb^{2+} and Cd^{2+} formed relatively weak complexes with oxalate, they were separated by cation-exchange, the other metals formed stable complexes and were separated by anion-exchange [1]. Using Ox, the detection sensitivity of Pb^{2+} is relatively higher compared to PDCA because it has lower ability than PDCA to chelate

with Pb^{2+} . However, the disadvantage of Ox eluent is as follows: (i) Mn^{2+} and Cd^{2+} coeluted; (ii) Fe^{3+} did not elute because the $\text{Fe}(\text{Ox})_3^{3-}$ complexes were so stable that they were strongly adsorbed by the column; (iii) Ca^{2+} and Mg^{2+} in large amounts may precipitate in the IC system.

When PDCA is used as the eluent, it forms strong anionic complexes with cumulative stability constant $\beta_2 > 10^8$ in case of heavy and transition metals. Thus, the separation of metals with PDCA is based on pure anion exchange. The separation was superior to the oxalic separation and it was possible to separate and determine Fe^{3+} , Cu^{2+} , Ni^{2+} , Zn^{2+} , Co^{2+} , Cd^{2+} , Mn^{2+} and Fe^{2+} in one injection [4]. Unfortunately, Pb^{2+} could not be detected by the post-column reagent PAR because not only was it strongly complexed by PDCA but it also exhibited slow complexation/decomposition kinetics [12].

Bonn and Reiffenstuhl [13] have found that a concentration of 0.03 mmol l^{-1} PDCA, added to the 10 mmol l^{-1} citric acid eluent, halved the retention times of transition metals, and under these conditions, elution was governed mainly by ion-exchange mechanisms. If high levels of PDCA were incorporated into the eluent, the complexation processes were favored. In our study, 3 mmol l^{-1} PDCA together with 3 mmol l^{-1} $\text{Na}_2\text{C}_2\text{O}_4$ was first used as the eluent. It was found that heavy and transition metals in the mixed eluent had a better separation and shorter elution time. In general, the stability constants of metal-PDCA complexes were far higher than those of metal-Ox complexes. Metal-PDCA complexes were formed when metals were injected. So the separation of metals with the mixed eluent was mainly anion-exchange. The optimized conditions are shown in Table 1. A typical chromatogram of a synthetic standard solution is shown in Fig. 1, in which all individual metal peaks are well separated. PDCA is not only a strong complexing agent but also a weak acid. So the pH value of the eluent must also be carefully controlled to optimize the separation. Increasing the pH value increases the complex-forming ability of PDCA and the effective constant of complex formation, resulting in decreased retention times. On the other hand, increasing the concentration of PDCA also decreased retention times. Taking the selectivity of separation into account, 3 mmol l^{-1} PDCA and pH 5.1 were employed.

Table 1
Ion chromatographic conditions

	Pb, Cu, Ni, Zn, Co, Cd, Mn, Hg
Column	IonPac CG5A + CS5A
Eluent	3 mmol l ⁻¹ PDCA 4.2 mmol l ⁻¹ LiOH 3 mmol l ⁻¹ Na ₂ C ₂ O ₄ 2 mmol l ⁻¹ Na ₂ SO ₄ (pH 5.1)
Eluent flow rate	1.0 ml min ⁻¹
Injection volume	41.4 µl (standard); 676 µl (sample)
Detection	visible absorbance (565 nm)
Post-column reagent	0.3 mmol l ⁻¹ 5-Br-PADAP 0.95% Triton X-100 (w/v) 0.066 mol l ⁻¹ amino acetic acid 0.067 mol l ⁻¹ NaOH 0.12 mol l ⁻¹ NaCl (pH 12)
PCR flow-rate	0.45 ml min ⁻¹

Na₂C₂O₄ not only helped to elute the metals, especially the strongly retained Hg²⁺, but it was also used as an inorganic modifier because it could improve the separation of Co²⁺ and Cd²⁺; Mn²⁺ and Hg²⁺ as illustrated in Fig. 1. Besides Na₂C₂O₄, Na₂SO₄ was also used as an inorganic modifier at the same time. It was also found that Co²⁺ and Cd²⁺; Mn²⁺ and Hg²⁺ could not be well separated when Na₂C₂O₄ and Na₂SO₄ were used separately as modifier. So 3 mmol l⁻¹ Na₂C₂O₄ combined with 2 mmol l⁻¹ Na₂SO₄ was used as the eluent.

When 25 mmol l⁻¹ NaCl was employed as the inorganic modifier, unfortunately, the large amount of Cl⁻ in the eluent resulted in the serious Hg²⁺ peak

Fig. 1. Chromatogram of metals in a synthetic standard solution. Chromatographic conditions are shown in Table 1. Peaks: 1, Pb (4 mg l⁻¹); 2, Cu (0.3 mg l⁻¹); 3, Ni (1 mg l⁻¹); 4, Zn (0.2 mg l⁻¹); 5, Co (0.5 mg l⁻¹); 6, Cd (0.5 mg l⁻¹); 7, Mn (0.3 mg l⁻¹); 8, Hg (2 mg l⁻¹), respectively.

tailing (the tail had the slope of a plateau). When the concentration of Hg²⁺ was low enough, the tail started to cover the Hg²⁺ peak, which apparently influenced the quantitative analysis of Hg²⁺. This phenomenon may be related to the adsorption of [HgCl₄]²⁻ on the bifunctional resin. Although the stability constant of [HgCl₄]²⁻ (10^{15.1}) is far lower than that of [Hg(PDCA)₂]²⁻ (10^{20.28}), the Cl⁻ concentration in the eluent was much higher than the PDCA²⁻ concentration, which could compete with PDCA²⁻ to complex with Hg²⁺, thus resulting in the by-peak of Hg²⁺. When Na₂SO₄ was used instead of NaCl, the peak tail of Hg²⁺ disappeared. So the analyzed samples cannot contain Cl⁻.

3.2. Selection of post-column color-development conditions

3.2.1. Selection of detection wavelength

Generally, the absorbance detection is the first choice in IC analysis of heavy and transition metals owing to its simplicity and reliability. The maximum absorption wavelength ($\lambda_{\max}$) of metal-5-Br-PADAP complexes has been reported [1,14,15]. In this study, the experimental results obtained from a spectrophotometer showed that the $\lambda_{\max}$ of the eight metal-5-Br-PADAP complexes were in the range 550–570 nm and 565 nm was a compromise.

3.2.2. Effects of Triton X-100 concentration on peak heights of metals

Since the surface active agent adds the functions of solubilization, sensitization, dispersion and stabilization to the color-development system, it can be used to enhance the solubility and the sensitivity of spectrophotometric detection for metals in an aqueous system. Triton X-100 was the best surface active agent as tested in [1,14,15] and its concentration effectively influenced the peak heights of Pb²⁺, Cd²⁺, Co²⁺, Mn²⁺ and Ni²⁺ as shown in Fig. 2. Fig. 2 also showed that Triton X-100 slightly influenced the peak heights of Hg²⁺, Cu²⁺ and Zn²⁺; however, it could increase the solubility and stability of the complexes. 0.95% Triton X-100 was a compromise because the peak heights of most metals were the highest.

Fig. 2. Effects of Triton X-100 concentration on peak heights of metals. The other conditions are shown in Table 1.

3.2.3. Effects of 5-Br-PADAP concentration on peak heights of metals

5-Br-PADAP has the same chelating system as PAR but is twice as sensitive as PAR for the determination of most metals, having molar absorptivities in the region of $1 \times 10^5 \text{ l mol}^{-1} \text{ cm}^{-1}$ for the transition metal complexes [16]. However, the Pb^{2+} -PDCA complex was so strong that Pb^{2+} could not be detected simultaneously with other heavy and transition metals [4], but this could be improved by using 5-Br-PADAP instead of PAR as the post-column chromogenic reagent. 5-Br-PADAP could form stable complexes with metals. Its concentration had a great influence

on the peak heights of the metals as Fig. 3 showed. At first, increasing 5-Br-PADAP concentration increased the peak heights of the metals. However, when the 5-Br-PADAP concentration reached a certain value, the peak heights of Co^{2+} , Ni^{2+} , Pb^{2+} and Mn^{2+} decreased and the background absorbances increased. This resulted in increased signal-to-noise ratios. Thus, 0.3 mmol l^{-1} 5-Br-PADAP was found to be appropriate.

3.2.4. Effects of pH on peak heights of metals

The sensitivity of determining metals greatly depends on the pH of detection because the pH can

Fig. 3. Effects of 5-Br-PADAP concentration on peak heights of metals. The other conditions are shown in Table 1.

Fig. 4. Effects of pH value on peak heights of metals. The other conditions are shown in Table 1.

influence the 5-Br-PADAP complexation process. A high pH increases the ionization of 5-Br-PADAP, thus increasing its complexing capabilities. However, the background absorbance would also be high. Moreover, hydrolysis of metals could be observed along with the simultaneous disappearance of their signals, whereas low pH values could decrease the detection sensitivities of metals. Therefore, the effect of pH on the peak heights of metals was investigated as shown in Fig. 4. It can be seen from Fig. 4 that the higher the pH was, the higher the peak heights for Pb^{2+} and Ni^{2+} were. So, for the determination of Pb^{2+} and Ni^{2+} , a higher pH value was chosen, but for the determination of the eight metals in one run, pH 11 was a compromise.

3.2.5. Choice of NaCl concentration

Some ligands such as F^- , Cl^- , Br^- , I^- , tartaric acid and sulfosalicylic acid etc. that are smaller than the color-producing reagent can be used as a second ligand (L_2) to form metal–5-Br-PADAP– L_2 complex [15,16]. They can make the metals chelate more easily with 5-Br-PADAP than that with PDCA and thus enhance the detection sensitivity of metals. In this study, Cl^- was employed as the second ligand. It was found that increasing the NaCl concentration could increase the peak heights of Pb^{2+} , Ni^{2+} , Co^{2+} , Cd^{2+} , Mn^{2+} and Hg^{2+} . However, the peak heights of Cu^{2+} and Zn^{2+} were not influenced. Thus, 0.12 mol l^{-1} NaCl was the best.

Table 2

Linear ranges, correlation coefficients, RSD values and detection limits for heavy and transition metals

Metal	Concentration range (mg l^{-1})	Correlation coefficient (r)	RSD (%)	Detection limit ($\mu\text{g l}^{-1}$)
Pb^{2+}	0.2–10	0.9993	1.1	200
Cu^{2+}	0.003–0.225	0.9999	1.7	1.5
Ni^{2+}	0.01–0.75	0.9998	5.2	5
Zn^{2+}	0.002–0.15	0.9999	3.3	0.5
Co^{2+}	0.005–0.375	0.9999	2.2	5
Cd^{2+}	0.005–0.375	0.9999	3.2	5
Mn^{2+}	0.003–0.225	0.9999	1.8	3
Hg^{2+}	0.02–1.5	0.9992	4.4	20

Fig. 5. Chromatogram of the standard rice powder. Sample solution was HNO_3 (pH 2) which was then diluted 1:100 (v/v) prior to injection (chromatographic conditions are shown in Table 1). Peaks: 1, Cu ($4.1 \mu\text{g g}^{-1}$); 2, Zn ($18.1 \mu\text{g g}^{-1}$); 3, Mn ($28.1 \mu\text{g g}^{-1}$).

Fig. 6. Chromatogram of the waste 7# Nanfu cellpacking. Sample solution was HNO_3 (pH 2) which was then diluted 1:1000 (v/v) prior to injection (chromatographic conditions are shown in Table 1). Peaks: 1, Cu (0.16 mg g^{-1}); 2, Ni (12.5 mg g^{-1}); 3, Zn (0.19 mg g^{-1}); 4, Mn (1.5 mg g^{-1}); 5, Hg (4.6 mg g^{-1}).

3.3. Interferences

The selectivity of IonPac CS5A was evaluated in order to avoid the potential interference of the main cationic components in samples, i.e., alkali and alkaline earth metals. Under the specified experimental conditions, both alkali and alkaline earth metals did not interfere with the determination because Mg^{2+} and Ca^{2+} could not react with 5-Br-PADAP. However, on

one hand, Mg^{2+} and Ca^{2+} in many samples may precipitate on the column as insoluble oxalate complexes when present at relatively high levels; on the other hand, they may overload the anion-exchange portion of the column due to the formation of $\text{Mg}[\text{PDCA}]_2^{2-}$ and $\text{Ca}[\text{PDCA}]_2^{2-}$. Thus, the efficiency of the column and the retention times of heavy and transition metals would be adversely affected. Therefore, the method established in this study was not suitable to analyze samples containing Ca^{2+} and Mg^{2+} at high levels. It was tested that, when the Ca^{2+} and Mg^{2+} concentrations were higher than 10 mg l^{-1} , the column pressure increased and the retention times reduced. It was also found that Fe^{3+} and Al^{3+} did not interfere with the determination of metals due to the presence of $\text{Na}_2\text{C}_2\text{O}_4$ in the eluent. However, Fe^{2+} could interfere with the determination of Mn^{2+} and this interference could be eliminated by adding 30% H_2O_2 to the analyzed samples.

3.4. Linearity and detection limits

Under the optimized experimental conditions, all metals showed good linearity between the concentrations and peak height responses. The detection limits, defined as the signals corresponding to three times the noise levels, were also calculated. All the results are listed in Table 2. The use of a large loop lowered the detection limits and did not have any detrimental effect on peak symmetry. However, when the loop exceeded $200 \mu\text{l}$, only the Pb^{2+} peak fell into the large negative peak of water, thus influencing the quantitative analysis of Pb^{2+} . Therefore, we used a $115 \mu\text{l}$ loop to determine Pb^{2+} and a $41.4 \mu\text{l}$ loop to evaluate the precisions by performing seven replicate analyses of a standard solution in which 4 mg l^{-1} Pb^{2+} , 0.3 mg l^{-1} Cu^{2+} , 1 mg l^{-1} Ni^{2+} , 0.2 mg l^{-1} Zn^{2+} , 0.3 mg l^{-1} Mn^{2+} ,

Table 3
Analysis of real samples

Sample		Content					
		Cu	Ni	Zn	Cd	Mn	Hg
Rice	determined ($\mu\text{g g}^{-1}$)	4.1		18.1		28.1	ND ^a
	certified ($\mu\text{g g}^{-1}$)	3.6 ± 0.4		18.1 ± 1.0		28.4 ± 3.8	0.038 ± 0.005
7# Nan-fu waste cellpacking (mg g^{-1})		0.16	12.5	0.19	ND	1.5	4.6
Municipal solid waste fly ash (mg g^{-1})		0.47	0.69	2.2	0.28	1.1	0.26

^a Not detected.

$0.5 \text{ mg l}^{-1} \text{ Co}^{2+}$, $0.5 \text{ mg l}^{-1} \text{ Cd}^{2+}$ and $2 \text{ mg l}^{-1} \text{ Hg}^{2+}$ were contained. The data confirmed that the precision of this method was good.

3.5. Samples

Samples of standard rice powder (A), waste cell-packing (B) and municipal solid waste fly ash (C), which contained little Ca^{2+} and Mg^{2+} , were analyzed. All the samples were digested and acidified with 1 + 1 HNO_3 to pH 2 then diluted with deionized water and directly injected into the IC system through a $0.45 \mu\text{m}$ filter. The chromatograms of Samples A and B are shown in Figs. 5 and 6, respectively. The data for these samples are summarized in Table 3. The concentration of Hg^{2+} in sample A was below the detection limit, and therefore, could not be detected. $43.2 \mu\text{g g}^{-1}$ ferrous in Sample A did not interfere with the determination of metals due to the presence of $\text{Na}_2\text{C}_2\text{O}_4$ in the eluent. Sample B contained a relatively high concentration of Hg^{2+} that could be detected. Many references [2,5,13,17] have determined heavy and transition metals by spiking and have got good results. So in this study, Hg^{2+} was determined by using Sample A spiked with 0.5 mg Hg^{2+} and Sample B spiked with $0.4 \text{ mg l}^{-1} \text{ Hg}^{2+}$. The mean recoveries ($n = 3$) of Hg^{2+} were (83.3 ± 4.6) and $(97.7 \pm 2.1)\%$, respectively.

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