Volume 97, issue 2, March 2011
ISSN 0026-265X

MICROCHEMICAL JOURNAL

Devoted to the Application
of Microtechniques
in All Branches of Chemistry

Editor
Joseph Sneddon

Determination of lead by stripping voltammetry with a disposable cartridge for quality control of municipal solid waste molten slag

Hideyuki Mongi [a,*], Akiko Kida [b], Natsuyo Uchida [b,1], Koichiro Iwasa [c], Shigenori Nakano [d]

[a] Recycling Research Team, Tottori Pref. Inst. of Public Health and Environmental Science, 526-1 Minamidani, Yurihama-cho, Tottori, 6820704 Japan
[b] Research Center for Material Cycle and Waste Management, National Institute for Environmental Studies, 16-2 Onogawa, Tsukuba, Ibaraki 305-8506 Japan
[c] Kyoto Research Laboratories Sekisui Chemical Co., Ltd., 2-2 Kamichoshi-cho, Kamitoba, Minami-ku, Kyoto, 601-8105, Japan
[d] Department of Regional Environment, Faculty of Regional Sciences, Tottori University, Koyama-cho, Tottori 680-8551, Japan

ABSTRACT

A novel system of mercury-free anodic stripping voltammetry with a disposable cartridge has been developed for on-site lead measurements to evaluate the environmental safety of secondary materials such as municipal solid waste (MSW) molten slag. The cartridge consists of two key components: a cation-exchange membrane for preconcentration–separation of lead, and carbon and Ag/AgCl electrodes for measurement. The dynamic range of the proposed method was 0.5–10 $\mu g\,L^{-1}$ with a detection limit (S/N = 3) of 0.15 $\mu g\,L^{-1}$. The reproducibility of the method was satisfactory with a relative standard deviation of 3.6% ($n = 7$) for 5 $\mu g\,L^{-1}$ lead (II). Interference from Cu(II) was suppressed by adding 5, 10, 15, 20-tetrakis (N-methylpyridium-4-yl)-21H, 23H-porphine to the sample solutions. The present method was successfully applied to the determination of lead in water-leaching and 1 mol L^{-1} HCl-extracted solutions from MSW molten slag. This method is useful for quality control of MSW molten slag in a manufacturing setting.

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1. Introduction

Municipal solid waste (MSW) and/or incinerated residues from MSW including toxic heavy metals have recently increased in Japan. They were often melted to reduce the final volume of these residues and to decompose dioxins. In 2006, the amount of molten slag generated from the MSW reached 0.74 million tons in Japan [1]. These have been utilized as concrete aggregates or road construction materials [2]. To ensure the safety of slag materials for their use in the environment, acceptable values of heavy metals in slag materials were established in Japanese Industrial Standards (JIS) at 2006, in which manufactures should perform the water-leaching and 1 mol L^{-1} HCl extraction tests for evaluating the risk of groundwater contamination and oral intake [3,4].

Among toxic heavy metals, Pb is frequently detected in leaching solutions from MSW molten slag [5–7]; its acceptable values in JIS are 10 $\mu g\,L^{-1}$ and 4.5 mg L^{-1} in water-leaching and 1 mol L^{-1} HCl-extracted solutions, respectively [3,4]. The Pb content in these solutions should be determined for appropriate control of molten slag quality and melting condition of the facility.

For the determination of Pb in the leaching and extracted solutions, atomic absorption spectrometry, inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) have been recommended in JIS K 0058 [6–9]. Although these techniques have high sensitivity, they are unsuitable for quality control in a manufacturing setting because they require expensive instrumentation and peripheral equipment. Therefore simple and highly sensitive methods are desired for quality control of MSW molten slag and environmental safety.

Electrochemical stripping methods have been recognized as powerful tools for determining trace amounts of Pb owing to their high sensitivity, selectivity, simplicity and quickness [10]. Mercury has been used as an electrode material for stripping techniques, despite its toxic character. Two types of mercury electrodes, the hanging mercury-drop and the mercury film, have been accepted for the development of adsorptive stripping voltammetry. Mercury film electrodes have been prepared by electroplating a thin film of mercury on a suitable substrate such as glassy carbon [11], carbon fiber [12,13], iridium [14–16] or gold [17]. The sensitivity of mercury film electrodes is more satisfactory than that of the hanging-drop electrode, but their disadvantage is a smaller dynamic range. Recently, the mercury-free electrode materials such as carbon paste electrode [18], carbon nanofiber electrode [19], bismuth coated carbon electrode [20,21] and antimony film electrode [22] have been reported. Although the bismuth coated carbon electrode and antimony film electrode are relatively high sensitivity among them, these electrodes are unsuitable for routine analysis because the preparation of bismuth- or antimony-coated electrodes is laborious

* Corresponding author. Tel.: +81 858 35 5416; fax: +81 858 35 5413.
E-mail address: mongih@pref.tottori.jp (H. Mongi).
[1] Present affiliation: Environmental Research Center Co., Ltd., 3-1 Hanare, Tsukuba, Ibaraki, 305-0857 Japan.

and time-consuming, and the quality of the resulting surface layer of the electrodes is dependent on the experimental conditions used during deposition. In addition, this coated material can become deactivated as a result of repeated analyses; the substrate must be cleaned and the coating process repeated. Baś, et al. [23] proposed a renovated silver ring electrode, which was free from coating material. However, the electrode should be refreshed by electrochemical procedure before each measurement and polished when the electrode deteriorated. Recently, Noh and Tothill [24] reported the use of disposable gold electrodes for Pb determination, but a serious interference from Cd(II) was observed in this method. These methods are unsuitable for on-site analysis in a manufacturing setting.

The present paper describes a square-wave anodic stripping voltammetry using a disposable cartridge cell (C-ASV) for the field assay of Pb. The present cartridge consists of two key components: a cation-exchange membrane for preconcentration–separation of Pb, and mercury-free carbon and Ag/AgCl electrodes for detection. Since the cartridge is disposable, it is not necessary to clean and polish the electrode surface after acquiring a measurement. Furthermore, these disposable cartridges are not subject to contamination from previous samples because of no reuse. The proposed method can determine nanograms per milliter levels of Pb in water-leaching and $1 \, mol \, L^{-1}$ HCl-extracted solutions from molten slag.

2. Experimental

2.1. Chemicals

All chemicals used here were of analytical grade. All solutions were prepared with deionized water obtained from a Millipore Ultrapure Water Purification System.

Working standard solutions were prepared by diluting a certified commercial standard solution of Pb(II) ($1000 \, mg \, L^{-1}$; Kanto Chemical Co., Japan) with $10^{-3} \, mol \, L^{-1}$ HNO$_3$ before use.

A $1 \times 10^{-2} \, mol \, L^{-1}$ solution of 5, 10, 15, 20-tetrakis(N-methyl-pyridium-4-yl)-21H, 23H-porphine, tetrakis(p-toluenesulfonate) (TMPyP, Dojindo Laboratories, Japan) was prepared. A mixed solution of NaCl ($1.6 \, mol \, L^{-1}$) and citric acid ($1 \times 10^{-2} \, mol \, L^{-1}$) was used for both the eluent and the supporting electrolyte in the voltammetric cell. The cation-exchange membrane (i.d., 6.6 mm; ion-exchange capacity, 5.5×10^{-3} meq cartridge^{-1}; surface area, $350 \, m^2 \, g^{-1}$; pore size, 8 nm) was obtained from Sekisui Chemical, Co., Japan.

2.2. Instrumentation

Fig. 1 shows the C-ASV system for the determination of Pb. It was composed of a syringe pump, a cartridge holder, an electrical signal processing device equipped with a current booster module (Sekisui Chemical, Co., Japan) and a personal computer. The disposable cartridge consisted of a cation-exchange membrane (i.d., 6.6 mm), a working electrode, a counter electrode and a reference electrode (Fig. 1). The working and counter electrodes were made of carbon and the volume of the voltammetric cell was 12 μL. An Ag/AgCl electrode (SSE) was used as the reference electrode. These parts were sealed by a disposable plastic plate. Electroanalytical measurements were performed with software written with LabVIEW (National instruments, USA). An HM-30G pH meter (DKK-TOA, Japan) was used for pH measurements, and a model 7500 ICP-MS (Agilent Technologies, Japan) was also used.

2.3. Sample preparation

After being air-dried and crushed by hammer, molten slag was sieved through a 2-mm stainless steel screen. For the water-leaching test, 50 g of the particle slag was added to 500 mL of water (solid-to-liquid ratio = $0.1 \, kg \, L^{-1}$) in a plastic bottle [8]. After the bottle was
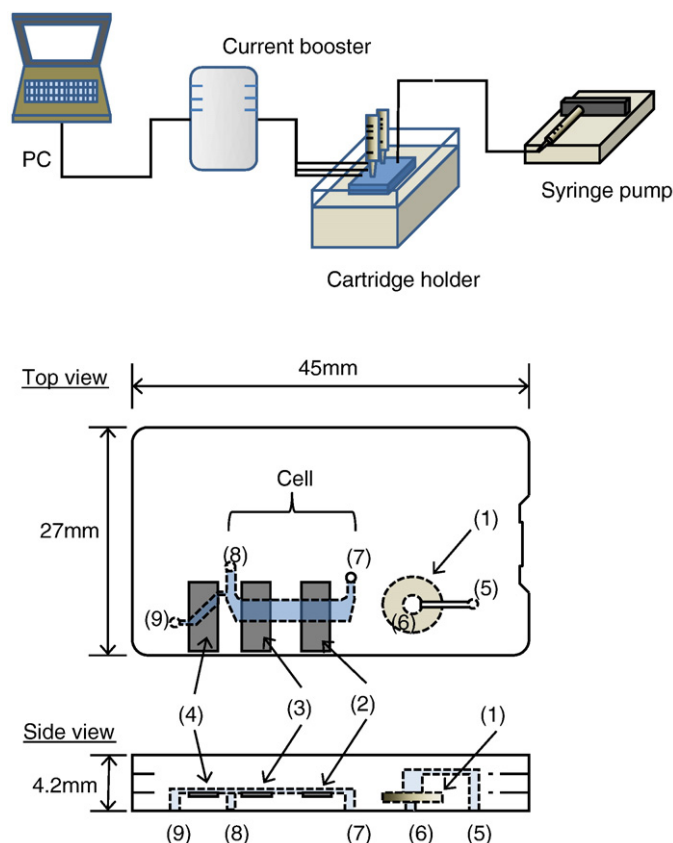

Fig. 1. Schematic diagram of the C-ASV system (a) and disposable cartridge (b). The numbers in (b) correspond to the following components: (1) cation-exchange membrane, (2) working electrode, (3) counter electrode, (4) reference electrode, (5) Inlet 1, (6) Drain 1, (7) Inlet 2, (8) Drain 2 and (9) Inlet 3.

horizontally shaken for 6 h, the leaching solution was separated centrifugally (3000 rpm, 10 min) and filtered through a 0.45-μm membrane filter. To prevent the adsorption and/or precipitation of Pb from the leaching solution, its pH was adjusted to about 2 by adding concentrated HNO$_3$. Prior to voltammetric determination, an appropriate amount of leaching solution was adjusted to pH about 3 by adding $1 \, mol \, L^{-1}$ NaOH solution, and then 340 μL of $1 \times 10^{-3} \, mol \, L^{-1}$ TMPyP and 430 μL of 1% (w/v) L-ascorbic acid solutions were added. The mixed solution was transferred to a 10-mL volumetric flask and diluted to the mark with water.

For $1 \, mol \, L^{-1}$ HCl extraction test, 15 g of the particle slag was added to 500 mL of the acid (solid-to-liquid ratio = $0.03 \, kg \, L^{-1}$) in a plastic bottle [9]. After being horizontally shaken for 2 h, the extracted solution was separated centrifugally (3000 rpm, 10 min) and filtered through a 0.45-μm membrane filter. An aliquot of the extracted solution (100 μL) was transferred to a 100-mL volumetric flask and diluted to the mark with water. To an appropriate amount of this solution, 340 μL of $1 \times 10^{-3} \, mol \, L^{-1}$ TMPyP and 430 μL of 1% (w/v) L-ascorbic acid solutions were added. The mixed solution was transferred to a 10-mL volumetric flask and diluted to the mark with water.

2.4. Procedure

Table 1 shows the optimal parameters of C-ASV for Pb determination. The experimental parameters adopted for square-wave voltammetric scan were frequency 50 Hz, amplitude 25 mV and step potential 2.5 mV. The procedure, as shown in Fig. 2, was as follows: A cartridge was set on the sample injection holder, where Inlet 1 and Drain 1 were opened. From Inlet 1, 5 mL of sample solution (pH 3) was injected by a syringe to retain Pb on the cation-exchange membrane

Table 1
Optimal parameters and their ranges examined for Pb determination.

	Optimal parameter	Range examined
Preconcentration–separation		
Sample volume (mL)	5	1–50
Sample pH	3	0.59–4.0
Eluent (electrolyte, mol L^{-1})	NaCl, 1.6	0–2
	Citric acid, 1.0×10^{-2}	1.0×10^{-3}–5.0×10^{-2}
Flow rate (μL min^{-1})	50	20–1000
Flow volume (μL)	200	50–500
Detection		
Deposition potential (V*)	−1.0	−1.5 to −0.8
Deposition time (s)	240	30–1500
Initial potential (V)	−1.0	−1.2 to −0.8
Final potential (V)	−4.5	0–0.6

* All potentials are vs. Ag/AgCl.

(Step 1, about 1 min). Next, the cartridge was set on the analytical holder, and then it was filled with an eluting solution which consisted of 1.6 mol L^{-1} NaCl and 1.0×10^{-2} mol L^{-1} citric acid (Step 2, 0.5 min). Drain 1 was connected to Inlet 2 in the voltammetric cell
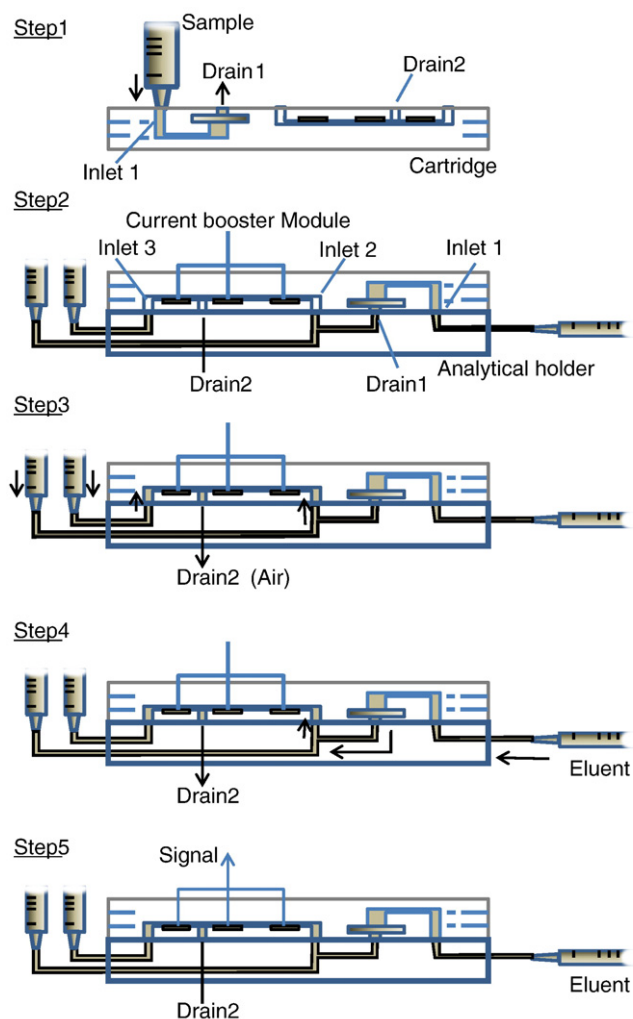

Fig. 2. Procedure for C-ASV analysis of Pb. Step 1: Preconcentration–separation of Pb(II) on cation-exchange membrane. Step 2: Placement of cartridge on analytical holder and connection to terminal. Step 3: Injection of eluting solution into electrochemical cell. Step 4: Injection of eluting solution and reduced Pb(II) onto working electrode. Step 5: ASV for reoxidation and measurement of Pb.

and Drain 2 was opened. After that, the eluting solution was introduced from Inlets 2 and 3 into the cell by two syringes to eliminate air (Step 3, 1 min). When 200 μL of the eluting solution was introduced into Inlet 1 by the syringe pump at a flow rate of 50 μL min^{-1}, the Pb on the membrane was transferred to the cell and deposited on the working electrode (Step 4, 4 min). The electrode potential was scanned positive from −1.0 to +0.6 V, causing the Pb deposited on the electrode surface to be reoxidized and released into the solution (Step 5, 0.5 min), and the resulting voltammogram was recorded. The total time required for preconcentration–separation and analysis was about 7 min per one sample. Although a cartridge could be refreshed for up to 5 additional uses by washing with 5 mL of the electrolyte solution and 5 mL water, it was used only one time in the present study.

3. Results and discussion

3.1. Optimization of analytical conditions

To perform the preconcentration–separation and determination of Pb at microgram per liter, variables were optimized by measuring the peak area of 10 μg L^{-1} Pb(II). Table 1 shows the preconcentration–separation conditions for the cation-exchange membrane and the electrochemical parameters for C-ASV.

The effect of NaCl and KCl concentrations in the eluting solution for Pb(II) was examined in the presence of citric acid. There were no peaks up to 0.5 mol L^{-1} NaCl. After this, an increase in concentration of NaCl increased the peak area for Pb(II), and the peak area remained constant at concentrations higher than 1.2 mol L^{-1} NaCl. The effect of KCl concentration on the peak area for Pb(II) were almost the same as that of NaCl. In the present system, 1.6 mol L^{-1} NaCl was used as an eluting agent.

To prevent hydrolysis and precipitate of Pb(II) in the eluting solution, complexing agents such as citric acid, acetic acid and tartaric acid in the eluting solution were examined in the presence of NaCl. Citric acid was used as a complexing agent because the peak areas for citric acid were a little larger than those for the others. The effect of citric acid concentration was examined up to 5.0×10^{-2} mol L^{-1}. As citric acid concentration increased, the peak area increased. Then it kept almost constant in the range 1.0×10^{-3}–5.0×10^{-2} mol L^{-1}. The concentration of citric acid was fixed at 1.0×10^{-2} mol L^{-1}.

An increase in the sample volume increased the sensitivity. In the present procedure, 5 mL of sample was used because the limit of determinable concentration of Pb(II) was about 10 μg L^{-1} under the conditions as shown in Table 1 without the use of cation-exchange membrane. A slower flow rate of eluting solution provided higher sensitivity but required a longer analysis time. To maintain a reasonable rate of sample throughput, the flow rate of eluting solution was fixed at 50 μL min^{-1}. The peak areas remained constant at eluting solution volumes above 100 μL; an eluting solution volume of 200 μL was chosen for the procedure. Considering the flow rate and eluting solution volume, the deposition potential at −1.0 V and the deposition time at 240 s were set in the procedure.

The effect of sample solution pH on the peak area for Pb(II) was examined in the pH range 0.6–4.0 by adding solutions of 1 or 10 mol L^{-1} HNO$_3$. The peak area remained constant over the pH range 1–4, but Pb was not collected by the cation-exchange membrane at pH lower than 1. The pH of the sample solution was adjusted to about 3 for the procedure because of complexation of TMPyP with Cu, as described below.

3.2. Calibration curve

Under the optimal conditions shown in Table 1, calibration curves were constructed by using the present system. The relationship between peak area (A) and Pb(II) concentration (C$_{Pb}$, in μg L^{-1}) was

linear in the range 0.5–10 μg L^{-1} and was expressed by the following equation:

$$A = 158C_{Pb} + 69.3 (R^2 = 0.997)$$

Calibration curves were also prepared without cation-exchange membrane to estimate the enrichment factor which is the ratio of the slopes of the calibration curves with and without preconcentration. This regression line was $A = 5.32 C_{Pb} + 22.1$ ($R^2 = 0.997$). From these results, the enrichment factor was calculated to be 30.

The detection limit (S/N = 3) with preconcentration was 0.15 μg L^{-1}, which was lower than the acceptable value of Pb for the leaching tests. The reproducibility was satisfactory with a relative standard deviation of 3.6% ($n = 7$) for 5 μg L^{-1} Pb(II). The sample throughput was about 8 samples per hour.

3.3. Effect of foreign ions

The effect of cations on the preconcentration–separation and determination was examined using a 10 μg L^{-1} Pb(II) solution. A large amount of cations showed negative interferences because the enrichment efficiency of the cation-exchange membrane was affected by these ions. No interferences were not found up to at least the following concentration: 5000 mg L^{-1} Na(I), 500 mg L^{-1} of Al (III), 400 mg L^{-1} Ca(II), 100 mg L^{-1} Zn(II), 50 mg L^{-1} of Mg(II), 10 mg L^{-1} of Fe(III).

On the other hand, Tl(I) and Cd(II) at the amount of 5 mg L^{-1} exhibited positive interferences because the peaks for Tl(I) and Cd(I) were observed near the peak for Pb in the voltammograms. The tolerance limits of Tl(I) and Cd(II) were 1 mg L^{-1} of and 0.01 mg L^{-1} respectively.

Fig. 3 shows the effect of Cu(II) concentration in range 0–100 μg L^{-1}. The peaks for Cu(II) shifted positive, and the peak areas increased with increasing Cu(II) concentration. At Cu(II) concentration of 100 μg L^{-1}, the peak for Pb(II) was not observed probably due to formation of a solid solution or intermetallic compound between Cu(II) and Pb(II) [25].
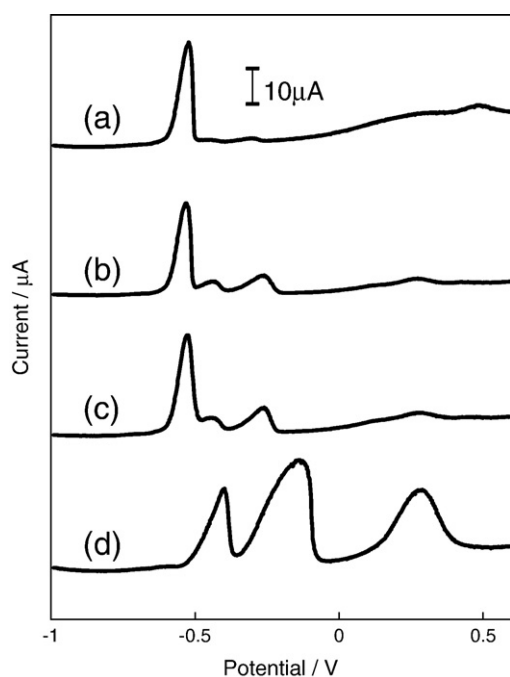
The effect of anions such as SO$_4^{2-}$, NO$_3^-$, HCO$_3^-$ and S^{2-} was also examined. Of these, S^{2-} caused a negative interference because S^{2-} reacted with Pb(II) to form precipitate. Its tolerance limit was 10 mg L^{-1}. Other ions did not interfere with Pb(II) determination up to at least the following concentration: 2000 mg L^{-1} of SO$_4^{2-}$, 6000 mg L^{-1} of NO$_3^-$ and HCO$_3^-$.

3.4. Elimination of interference from Cu(II)

The above observations indicated that Cu(II) severely interfered with Pb determination. Therefore, TMPyP was selected as a masking agent for Cu(II) owing to its complexation. Since the formation rate of the Cu–porphyrin complex was too slow in the absence of a reducing agent, L-ascorbic acid was added to accelerate complex formation [26,27]. Fig. 4 shows the effect of molar ratio of Cu(II) to TMPyP on Pb determination. For 1000 μg L^{-1} Cu(II), the addition of TMPyP at a ratio greater than 1.5 times the molar quantity of Cu(II) suppressed the interference. Therefore, the concentration of TMPyP was fixed at 3.4×10^{-5} mol L^{-1}, which was 2 times the molarity of 1000 μg L^{-1} Cu(II).

3.5. Determination of Pb in the water-leaching and 1 mol L^{-1} HCl-extracted solutions from MSW molten slag

To evaluate the proposed C-ASV method, the proposed method was applied to the determination of Pb in leaching water and 1 mol L^{-1} HCl-extracted solutions from MSW molten slag. To adjust the sample pH and to avoid interference from Ca, the extracted solution in 1 mol L^{-1} HCl was diluted 1000-fold with water. The contents of Pb, Na, Ca and Cu in the same samples were also determined by ICP-MS.

The analytical results obtained by proposed method and ICP-MS are shown in Tables 2 and 3. For the water-leaching solution, the values obtained by C-ASV agreed well with those obtained by ICP-MS. Foreign ions such as Na, Ca and Cu did not interfere with the Pb determination (Table 2). For the 1 mol L^{-1} HCl-extracted solution, the values obtained by C-ASV method were consistent with those

Fig. 3. C-ASVs of Pb(II) in the absence and presence of Cu(II). (a) 10 μg L^{-1} Pb(II); (b) 10 μg L^{-1} Pb(II) + 1 μg L^{-1} Cu(II); (c) 10 μg L^{-1} Pb(II) + 5 μg L^{-1} Cu(II); (d) 10 μg L^{-1} Pb(II) + 100 μg L^{-1} Cu(II). Other conditions as in Table 1.
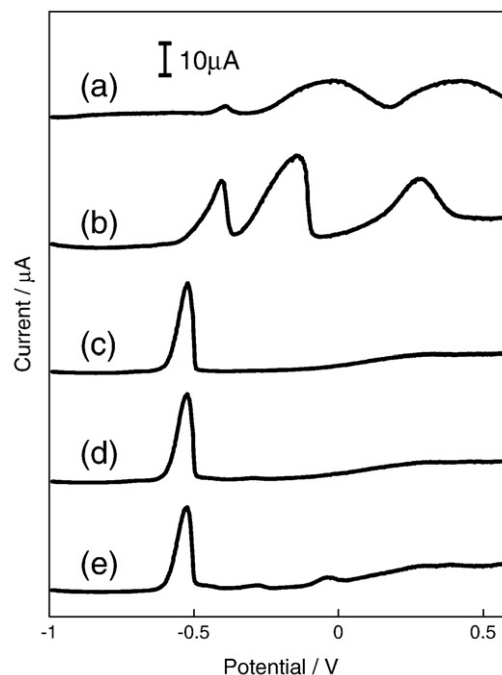
Fig. 4. C-ASVs of Pb(II) and Cu(II) in the absence and presence of TMPyP. C$_{Pb(II)}$, 10 μg L^{-1}; C$_{Cu(II)}$, 1000 μg L^{-1} TMPyP: Cu(II) (molar ratio) = (a) 0:1; (b) 1:1; (c) 1.5:1; (d) 2:1; (e) 5:1. 430 μL of 1%(w/v) L-ascorbic acid solutions were added to sample solution. Other conditions as in Table 1.

Table 2
Determination of Pb(II) in the water-leaching solution from MSW molten slag.

Sample [a]	Pb (μg L^{-1})[b]		Na (mg L^{-1})[c]	Ca (mg L^{-1})[d]	Cu (mg L^{-1})[e]
	C-ASV	ICP-MS			
1	9.9 ± 0.3	9.6 ± 0.2	1.2	2.4	0.054
2	1.8 ± 0.1	1.7 ± 0.1	1.3	3.6	0.036
3	1.1 ± 0.1	0.97 ± 0.03	1.9	3.5	0.012
4	<0.5	0.56 ± 0.03	2.3	3.6	0.040
5	8.4 ± 0.1	7.7 ± 0.3	1.9	4.2	0.044
6	11 ± 1[b]	10 ± 1	1.4	4.4	0.039
7	0.51 ± 0.1	0.75 ± 0.03	3.1	11	0.0068
8	<0.5	<0.5	2.7	10	0.0061
9	<0.5	0.50 ± 0.01	2.3	7.9	0.0063
10	2.8 ± 0.4	2.9 ± 0.1	2.4	12	0.016
11	4.8 ± 0.2	4.5 ± 0.1	2.0	6.3	0.033
12	37 ± 2[b]	32 ± 1	2.0	6.5	0.12
13	4.2 ± 0.2	3.8 ± 0.2	1.7	5.4	0.027
14	15 ± 1[b]	13 ± 1	2.3	5.8	0.099

[a] 50 g of the slag was leached in 500 mL of water.
[b] Diluted 10-fold with water.
[c] Average and standard deviation (n = 3).
[d] Obtained by ICP-AES.
[e] Obtained by ICP-MS.

obtained by ICP-MS; Na, Ca and Cu did not interfere in this case either (Table 3). These results indicate that the proposed C-ASV method is useful for on-site quality control analysis.

4. Conclusions

This paper demonstrates a mercury-free anodic stripping voltammetry with a disposable cartridge for the preconcentration–separation of Pb(II). Under the optimal conditions, the proposed method allowed for the determination of Pb(II) in the range 0.5–10 μg L^{-1}. The interference from Cu(II) was suppressed by adding TMPyP. Satisfactory results were obtained for the determination of Pb(II) in both water-leaching and 1 mol L^{-1} HCl-extracted solutions from MSW molten slag. The proposed system offers many advantages in terms of analysis time, repeatability and compact, and permits quality control of MSW molten slag in a manufacturing setting. The operation of this system is simple, thus allowing routine analysis by operators without extensive training.

Table 3
Determination of Pb(II) in the 1 mol L^{-1} HCl-extracted solution from MSW molten slag.

Sample[a]	Pb (μg L^{-1})[b]		Na (μg L^{-1})[c]	Ca (μg L^{-1})[c]	Cu (μg L^{-1})[d]
	C-ASV	ICP-MS			
1	4.3 ± 0.2	3.9 ± 0.1	110	500	9.0
2	5.5 ± 0.9	4.9 ± 0.1	180	760	2.7
3	2.0 ± 0.5	2.0 ± 0.2	140	660	2.7
4	4.5 ± 0.2	3.9 ± 0.1	100	440	12
5	8.4 ± 0.2	8.2 ± 0.1	190	1000	22
6	6.9 ± 0.2	6.0 ± 0.1	200	1000	14
7	7.4 ± 0.1	6.4 ± 0.1	150	860	19
8	4.7 ± 0.3	4.3 ± 0.1	190	1000	2.0
9	2.6 ± 0.2	2.7 ± 0.1	200	1000	0.79
10	2.5 ± 0.2	2.1 ± 0.1	120	640	3.3
11	3.6 ± 0.2	3.1 ± 0.1	170	800	1.1
12	7.4 ± 0.1	6.3 ± 0.1	170	800	2.6
13	5.0 ± 0.1	3.8 ± 0.1	160	730	1.6
14	4.7 ± 0.2	3.9 ± 0.1	120	540	7.9

[a] 15 g of the slag was leached in 500 mL of the acid. The resulting solutions were diluted 1000-fold with water.
[b] Average and standard deviation (n = 3).
[c] Obtained by ICP-AES.
[d] Obtained by ICP-MS.

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