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RESEARCH PAPER

Development of a certified reference material for accurate determination of the leaching of Pb and Zn in solid waste

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Abstract

Certified reference materials (CRMs) with high accuracy and traceability play a significant role in the calibration of equipment and validation of analytical methods. However, there is still a lack of suitable solid waste CRMs for quality assurance and quality control. Thus, a CRM (GBW(E)085538) was developed for accurate determination and reliable measurement of the leaching of Pb and Zn in solid waste according to the requirements of ISO 17034 and the recommendations of ISO Guide 35. This study describes the steps performed for the development of the CRM. These steps include material preparation, homogeneity, and stability during transport and storage, assignment of certified values, and their uncertainties. The material was dried, ground, sieved and well-mixed, and the final bulk material was bottled in 1 kg portions. Analytical techniques like inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectrometry (ICP-OES), and flame atomic absorption spectrometry (AAS) have been used for the characterization of property values. Concurrently, an inter-laboratory comparison study involving 9 qualified laboratories was implemented to support the certification study. The certified values of Pb and Zn were (4.66 ± 0.21) mg/L and (2.95 ± 0.14) mg/L with 7-month stability.

Keywords Certified reference material · Solid waste · Inductively coupled plasma-mass spectrometry · Homogeneity and stability assessment

Introduction

With the acceleration of urbanization and industrialization, solid waste generation has increased and has become a global concern [1]. Effective management of solid waste is being strengthened in both developed and developing countries due to its adverse impacts on human health and the environment. For many years, the tailings have been considered as an industrial solid waste, and the leaching of heavy metals from tailings has become an increasingly urgent and necessary issue worldwide. Solid waste contains various hazardous elements, such as easily leachable heavy metals. Without proper management, the leaching of heavy metals is a matter of concern when precipitation or surface water passes through solid waste [2]. Heavy metals are not biodegradable and have long lasting effect in soil, surface water, and groundwater, which pose a serious threat to the environment [3–5]. In addition, it has been proven that the leaching of heavy metals can endanger human health through the food chain [6]. Pb and Zn are common harmful heavy metal elements leached from solid waste. High concentration of Pb in the blood can result in severe damages to multiple systems, such as the nervous, endocrine, and hematopoietic system [7, 8]. It is particularly hazardous to the neurological development of children. Zn plays an important role in the physiological functions of the human body, but excessive ingestion can have adverse effects on human health. Zn if present in excess can cause iron deficiency anemia, damage digestive system, and weaken immune system [9–11].

A series of regulations have been issued due to the adverse health effects of the leaching of Pb and Zn in solid waste. The World Health Organization has established a standard that the maximum levels for Pb and Zn in drinking water are 0.01 mg/L and 3 mg/L, respectively [12]. In light of the extraction toxicity of hazardous wastes, the US EPA has established the identification standard, setting the regulatory limit for Pb at 5.0 mg/L. Therefore, the implementation of precise analysis method is of great significance for reliably enforcing the current laws and regulations. This necessity has prompted the scientific community to develop diverse methods for analyzing the extraction toxicity of solid wastes based on inductively coupled

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plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectrometry (ICP-OES), and flame atomic absorption spectrometry (AAS) [6, 13].

Nevertheless, testing laboratories using above analytical methods should conduct appropriate validation studies of the experimental methods and instruments. These methods cannot eliminate the influence of matrix effects, nor can they guarantee the accuracy and comparability of measurement results. In this instance, certified reference materials (CRMs), serving as the simplest and the most effective tools to achieve this purpose, are continuously needed [14–16]. CRMs are materials with specified properties, characterized for sufficient stability and homogeneity [17–19]. Furthermore, CRMs have become fundamental pillars in the development of new analytical method validation and proficiency testing, as well as in the determination and establishment of metrological traceability within the framework of standards [20–22]. However, there are little CRMs currently available for solid waste, let alone CRMs for the leaching of Pb and Zn in solid waste.

In order to monitor environmental content, ensure human health safety, and provide accurate and reliable test results, our laboratory has launched a project to develop a CRM for accurate determination of the leaching of Pb and Zn in solid waste. In the early stage of candidate material selection, to ensure the applicability of CRM and meet the needs of measurement calibration and quality control in the actual detection process, the concentration of Pb in the candidate CRM should be controlled close to the limit of at 5.0 mg/L, set by the US EPA. Hence, among various tailing ponds that were surveyed for the leaching of Pb and Zn in solid waste, a lead–zinc tailing pond in Zhejiang Province (120°32′41.81″E, 28°11′54.93″N) was selected for the preparation of the candidate CRM.

This article reports the development of a CRM for accurate determination of the leaching of Pb and Zn in solid waste. The complete processes, including material preparation, analytical methods validation, homogeneity and stability assessments, value assignment, as well as evaluation of uncertainties of certified values, were described in detail in accordance with ISO 17034 [23] and ISO Guide 35 [24]. The reference method for the certification of CRM was carried out using ICP-MS, ICP-OES and AAS. Furthermore, an inter-laboratory comparison study involving 9 qualified laboratories was conducted in order to support the assignment of certified values.

Materials and methods

Chemicals and materials

Nitric acid (65.0% ~ 68.0% w/w%) was obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Concentrated sulfuric acid (GR) was purchased from Zhejiang Zhongxing Chemical Reagent Co., Ltd (Jinhua, Zhejiang, China). Hydrochloric acid (36% ~ 38% w/w%) was obtained from Kunshan Jincheng Chemical Reagent Co., Ltd (Kunshan, Jiangsu, China). Standard Solution of Lead (Pb) (GBW08619, (1000 ± 2) μg/mL) and Standard Solution of Zinc (Zn) (GBW08620, (1000 ± 1) μg/mL) were purchased from the National Institute of Metrology, China. Ultrapure water was prepared using a Milli-Q system at 18.2 MΩ.cm (Shanghai, China). Extractive agent was prepared pH at 3.20 ± 0.05 by adding concentrated sulfuric acid and concentrated nitric acid with a mass ratio of 2:1 into 1000 mL of ultrapure water [25].

Calibrated analytical balance and micropipettes were used when mentioned in the validated method. The ICAP RQ ICP-MS (Thermo Fisher Scientific, Waltham, MA, USA), ICAP7600 ICP-OES (Thermo Fisher Scientific, Waltham, MA, USA) and PinAAcle 900F AAS (PerkinElmer, Waltham, MA, USA) were used to measure the concentrations of Pb and Zn in the leachates.

Selection and preparation of the candidate CRM

The selection of a candidate raw material is a critical step in the development of CRMs. Ten tailing ponds in Wenzhou, Zhejiang, China were screened for the leaching of Pb and Zn in solid waste to determine their levels of contamination. Among those tailing ponds, a lead–zinc tailing pond in Yongjia County (120°32′41.81″E, 28°11′54.93″N) showed the concentrations of Pb and Zn at considerable levels. Therefore, it was selected as a candidate raw material, and 1200 kg of this material were immediately mined from the same mining area.

The candidate raw material was subjected to dry in ventilation places indoors and crushed to less than 5 mm with a crusher, followed by drying in a 105℃ oven for 12 h. The material was smashed and grinded by using a ball mill and coarse particles were removed by passing through 20 and 60 mesh sieve rings consecutively. After that, 1000 kg of material were weighed and grinded with the ball mill for 24h, and then sieved through 200 mesh sieve which gave the size of ≤ 0.074 mm. Thereafter, the candidate raw material was sealed in 25 L container with plastic film inside. In total, the packaging unit was divided into 988 bottles, with each bottle containing 1 kg reference material. 988 units were firmly sealed in high density polyethylene bottles and stored at room temperature away from light.

Analytical method

The analytical methods used for the certification and assignment of certified values were ICP-MS, ICP-OES and AAS. Additionally, ICP-MS method was selected as the analytical

method for homogeneity and stability testing of the CRM. A brief description of the sample pretreatment procedure was as follows.

An accurately weighed test portion of 150 g was taken in a 2 L extraction bottle. 1500 g extractive agent was added and oscillated at 23℃ at 30 r/min for 18 h. Then the leachate was filtered using a 0.65 μm membrane and stored at 4℃. It is noteworthy that if gas is produced during the oscillation, the extraction bottle should be opened regularly in a fume hood to release excessive pressure.

Firstly, 25 mL of the leachate was transferred into a 40 mL PTFE tube. Then 5 mL nitric acid was added and mixed well. Next, the sample was digested by Graphite-digestion device at 180℃ until dryness. After cooling, all of the extracts were eluted with 5 mL 1% HNO_3. The eluate was transferred to a 25 mL volumetric flask, dilute with 1% HNO_3 to volume, and mix.

Analytical methods validation

The validation of the analytical method with respect to limit of detection (LOD), limit of quantification (LOQ), precision, and accuracy were experimentally established. For method validation, seven process blanks were analyzed for the LOD and LOQ calculations. Three different amounts of solid waste samples were selected, of which the acid leaching of Pb and Zn formed a concentration gradient. Precision was calculated by analyzing six replicates of solid waste samples. Three different amounts of standard substances were added in the acid extraction of solid waste samples before determination and the obtained data was used for the calculation of recovery and as the evaluation basis of accuracy.

Sample preparation and pretreatment analysis

A series of contrast experiments were conducted to investigate the effects of different sample preparation and pretreatment (the particle size of samples, moisture content of samples and measurement methods of extractive agent) on the concentrations of Pb and Zn in the leachate, as presented in Supplementary Table S1. The samples were analyzed by the ICP-MS method.

Homogeneity testing

For within and between bottle homogeneity testing of the candidate CRM for its concentration of Pb and Zn in the leachate, thirty units were selected from the 988 units (stored at room temperature) according to the random number table. Three subsamples of each unit were analyzed by the ICP-MS method and 150 g was the minimum sampling for analysis. One-way analysis of variance (ANOVA) was used to analyze the data with 95% confidence level. The homogeneity of the values of the candidate CRM was evaluated by F-test for checking the significant difference.

Stability assessment

The short-term stability assessment was executed using the isochronous stability study scheme. The short-term stability of the candidate CRM at different temperatures was assessed: –20◦C and 60◦C (simulation of possible minimum and maximum temperatures during transportation) for 0, 3, 8 and 15 days. At each predetermined time period, two units from the whole batch stored under the regular storage conditions (at room temperature) were moved to the areas of the designated temperatures in advance of the testing point. At the defined end time of this study, the units were collected at each temperature, and two subsamples from each unit were analyzed by the ICP-MS method.

The long-term stability of the concentrations of Pb and Zn in the candidate CRM stored under regular storage conditions (at room temperature) was also assessed at 0, 1, 2, 3, 5 and 7 months using the classical stability study design. At each time period, three units were randomly selected and measured by the ICP-MS method.

Value assignment

The value assignment of the concentrations of Pb and Zn in the candidate CRM was based on an inter-laboratory comparison involving 9 qualified laboratories. Two units of the candidate CRM samples were provided to each participant and three subsamples from each unit were analyzed by the ICP-MS, ICP-OES or AAS method. Nine participating laboratories were instructed to use the same sample pretreatment procedure to obtain the eluate in acid condition. Our laboratory, as the guider, analyzed the sample eluate by ICP-MS, ICP-OES and AAS methods, and the other eight laboratories used the same ICP-MS method as the reference method. Standard Solution of Lead (Pb) (GBW08619, (1000 ± 2) μg/mL) and Standard Solution of Zinc (Zn) (GBW08620, (1000 ± 1) μg/mL) were used as primary reference materials to conduct calibration curves.

Results and discussion

Analytical methods validation

All the concentrations of Pb and Zn in the leachate were measured using ICP-MS, ICP-OES and AAS methods. The LOD was determined by repeated analysis of a blank test

portion (7 replicates were taken). The LOQ of Pb and Zn concentrations in the leachate was calculated by 4 times of the LOD. The LOD and LOQ of Pb and Zn for each method are shown in Table S2.

Precision is an important parameter of analytical method validation. It can be defined as the measurement repeatability, which is an estimate of the dispersion degree of the results obtained with the same sample (or subsamples of the same sample). Precision was measured as relative standard deviation (RSD) obtained by six replicates of three different solid waste samples followed by ICP-MS, ICP-OES and AAS measurements. Precision limits of Pb and Zn for each method are listed in Table S3.

Another key parameter of analytical method validation is accuracy. The accuracy was evaluated by the recovery method. Leachate of solid waste samples was added to correlative standard substances before digestion. Although recovery method is not ideal to establish the accuracy of the analytical method, in the absence of matrix matched CRM this was the best way. The recovery experimental results based on added and recovery are shown in Table S4. The recoveries of ICP-MS, ICP-OES and AAS methods meet current Chinese national standards and environmental protection industry standards [26–28].

These obtained results suggest that the LOD, LOQ, precision, and accuracy are accurate for the analysis of the solid waste samples by ICP-MS, ICP-OES and AAS methods. These methods are suitable for determination of the leaching of Pb and Zn in the candidate CRM.

Sample preparation and pretreatment analysis

The contrast experiment of different particle sizes of samples was conducted using 20 ~ 60 mesh, 140 ~ 200 mesh and above two mixed samples. The concentrations of Pb and Zn in the leachate with different particle sizes of samples are shown in Table S5. It could be observed that the concentrations of Pb and Zn in the leachate would increase slightly with a decrease in the particle size of samples. Therefore, to avoid the effect of the particle size of samples on the concentrations of Pb and Zn in the leachate, 99% of the candidate materials should be ensured that the particle size is less than 200 mesh during the sample preparation process.

The concentrations of Pb and Zn in the leachate at different moisture contents of samples are shown in Table S6. Comparing the samples dried to a constant weight at 105℃ with the non-drying samples, the experimental results verified that the moisture content of samples had a small or even a negligible effect on the concentrations of Pb and Zn in the leachate. Therefore, the candidate CRM can be used directly without any drying pretreatment.

In order to study the effect of the measurement methods of extractive agent on the concentrations of Pb and Zn in the leachate, three kinds of measurement methods (electronic balance, graduated cylinder, volumetric flask) were designed. Ten units of solid waste samples were selected randomly and the extractive agent was measured in three ways respectively. The experimental results are reported in Table S7. In three kinds of measurement methods, the average concentrations of Pb and Zn were close to each other and the RSD was very small (in the range of 1.1 ~ 1.9%). It indicated that three different measurement methods were consistent. Therefore, during the candidate CRM pretreatment process, extractive agent can be measured by electronic balance, graduated cylinder, volumetric flask.

Homogeneity testing

The homogeneity (within-bottle homogeneity and between-bottle homogeneity) of a CRM is an indispensable property for testing CRM. The homogeneity of the candidate CRM was evaluated using the ICP-MS method. The ANOVA (F-test) was performed to check homogeneity in accordance with ISO Guide 35. The results of homogeneity for the candidate CRM are summarized in Table 1. The average concentration of Pb for the 30 bottles (90 subsamples) was found to be 4.72 mg/L, with a RSD of 2.1%, and the average concentration of Zn for the 30 bottles (90 subsamples) was found to be 2.97 mg/L, with a RSD of 3.4%. The $F_{calculated}$ of the candidate CRM for Pb and Zn were all smaller than the critical $F_{0.05(29,60)}$ suggesting that there was no significant difference between the variance within-bottle and between-bottle. Thus it is concluded that the candidate CRM has significant homogeneity.

The uncertainty of between-bottle heterogeneity u_{bb} comes from the inhomogeneity among the bottles (MS_{among}) and the inhomogeneity within the bottles (MS_{within}). The formula of u_{bb} is as follows, where n means the number of repeating tests for each unit.

Table 1 The results of homogeneity for the candidate CRM

Element	Parameters							
	Average (mg/L)	RSD (%)	MS_{within}	MS_{among}	$F = \frac{MS_{within}}{MS_{among}}$	$F_{0.05(29,60)}$	Judgment	u_{bb}
Pb	4.72	2.1	0.0127	0.0079	1.62	1.65	$F < F_{0.05(29,60)}$	0.040
Zn	2.97	3.4	0.0115	0.0092	1.26	1.65	$F < F_{0.05(29,60)}$	0.028

Table 2 The results of short-term stability for the candidate CRM

Temperature	Element	Parameters							
		Average (mg/L)	RSD (%)	MS_{within}	MS_{among}	$F = \frac{MS_{within}}{MS_{among}}$	$F_{0.05(3,4)}$	Judgment	u_{sts}
– 20℃	Pb	4.75	1.2	0.0039	0.0027	1.46	6.59	$F < F_{0.05(3,4)}$	0.020
	Zn	2.96	0.9	0.0011	0.0003	3.19	6.59	$F < F_{0.05(3,4)}$	0.016
60℃	Pb	4.75	1.9	0.0080	0.0076	1.04	6.59	$F < F_{0.05(3,4)}$	0.011
	Zn	2.97	1.6	0.0029	0.0017	1.75	6.59	$F < F_{0.05(3,4)}$	0.020

$$u_{bb} = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (1)$$

Stability assessment

In this study, the stability of Pb and Zn concentrations in candidate CRM under short-term transportation and long-term storage was studied. The short-term stabilities of the CRM at – 20℃ and 60℃ and the long-term stability of the CRM at room temperature were assessed using the scheme that was described in "Value assignment" section.

For short-term stability, one unit for each time–temperature point was selected and two duplicate subsamples from each unit were analyzed by the ICP-MS method. The data are analyzed using ANOVA and the calculated parameters are listed in Table 2. The short-term stability results are also graphically presented in Figs. 1 and 2. As a result, the $F_{calculated}$ of the candidate CRM for Pb and Zn at each temperature were all smaller than the $F_{0.05(3,4)}$. The results obtained under each designated condition for all short-term stability test periods are in good agreement with the initial values within the measurement uncertainty. This confirmed that Pb and Zn concentrations in this CRM are sufficiently stable for up to 15 days at –20℃ and 60℃. Therefore, the CRM can be reliably transported to cold or hot regions for up to 15 days without temperature control measures.

For long-term stability, three units were taken at a predetermined point in time and analyzed using the same ICP-MS method as was used for the short-term stability testing. The data are summarized in Table 3 and the results are shown in Figs. 3 and 4. According to the statistical evaluation, the curves had no tendency to change in one direction and the concentrations of Pb and Zn fluctuated within their expanded uncertainties. Furthermore, the absolute values of slope $|\beta_1|$ were all smaller than the product of $t_{0.95,n-2}$ and the standard deviation of stability slope $s(\beta_1)$, which indicated that the CRM is stable for up to 7 months at room temperature (under the storage condition). In addition, long-term stability monitoring will continue to extend the expiry date of the candidate CRM.

The calculation formula of the uncertainty of short-term stability (u_{sts}) at each temperature was the same as that of the homogeneity testing, as expressed in Eq. (2). Meanwhile,

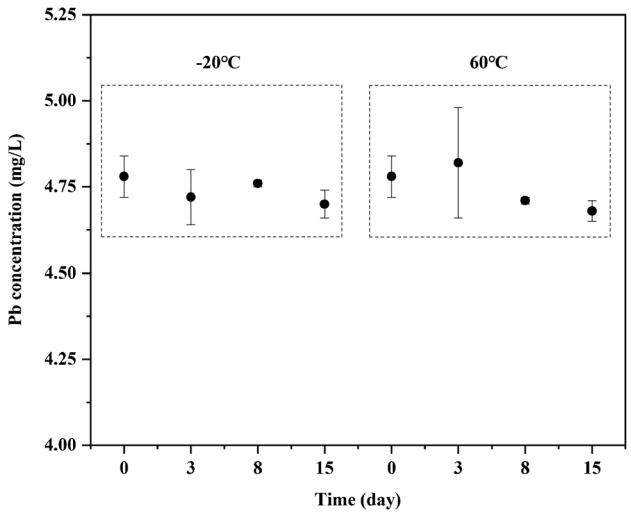

Fig. 1 Results of short-term stability concentration at – 20℃ and 60℃ for Pb in the candidate CRM

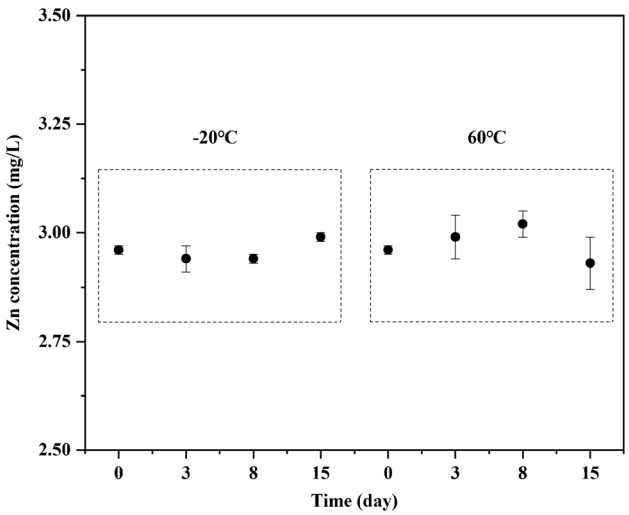

Fig. 2 Results of short-term stability concentration at – 20℃ and 60℃ for Zn in the candidate CRM

Table 3 The results of long-term stability for the candidate CRM

Element	Average (mg/L)	Parameters					
		β_1	$s(\beta_1)$	$t_{0.95(4)}$	$t_{0.95(4)}s(\beta_1)$	Judgment	u_{lts}
Pb	4.72	−0.011	0.0085	2.78	0.024	$\lvert\beta_1\rvert < t_{0.95,n-2} \bullet s(\beta_1)$	0.060
Zn	2.95	−0.0044	0.0043	2.78	0.01196	$\lvert\beta_1\rvert < t_{0.95,n-2} \bullet s(\beta_1)$	0.030

the uncertainty of long-term stability (u_{lts}) is calculated as follows, where n means the number of months.

$$u_{lst} = \mathrm{s}(\beta_1) \times n \tag{2}$$

Assignment of certified values and their uncertainties

Nine laboratories participated in the assignment of certified values and their results are presented in Table 4. Through statistical tests, it is found that the measured values are in accordance with normal distribution. Thus, assignment of certified values was carried out by weighted average method using the averages reported by participating laboratories. The average values of Pb and Zn, 4.66 mg/L and 2.95 mg/L, were assigned as the initial certified values for this CRM.

The certified values for Pb and Zn have good metrological traceability. All measuring instruments, such as electronic balance and volumetric flasks, used in the pretreatment have traceable calibration certificates within expiry date, which can trace to the source of nation standards. Standard Solution of Lead (Pb) (GBW08619, (1000 ± 2) μg/mL) and Standard Solution of Zinc (Zn) (GBW08620, (1000 ± 1) μg/mL) were used as primary reference materials to conduct calibration curves. The measurement results of ICP-MS, ICP-OES, and AAS can trace to international system (SI) of units.

The uncertainty of the definite value was evaluated as per ISO Guide 35 using the following definition. Characterization uncertainty can be categorized into two main groups, u_A and u_B, as described in Table S8. The uncertainty of measurement results from nine laboratories (u_A) is calculated as follows, where s means the standard deviation of all laboratories and n means the number of the groups of data.

$$u_A = \frac{s}{\sqrt{n-1}} \tag{3}$$

All uncertainty components in the measurement u_B includes $u_{B\text{-}1}$, $u_{B\text{-}2}$, $u_{B\text{-}3}$, $u_{B\text{-}4}$, and $u_{B\text{-}5}$, where $u_{B\text{-}1}$ means the uncertainty from the weighing of samples, $u_{B\text{-}2}$ means the uncertainty from the weighing of extractive agent, $u_{B\text{-}3}$ means the uncertainty of leachate density, $u_{B\text{-}4}$ means the uncertainty of volumetric glassware in the solution transfer, $u_{B\text{-}5}$ means the uncertainty from the preparation of standard solution, and $\overline{X}$ means the average of the measurement results. The formula of u_B, is as follows:

$$u_B = \overline{X} \times \sqrt{{u_{B-1}}^2 + {u_{B-2}}^2 + {u_{B-3}}^2 + {u_{B-4}}^2 + {u_{B-5}}^2} \tag{4}$$

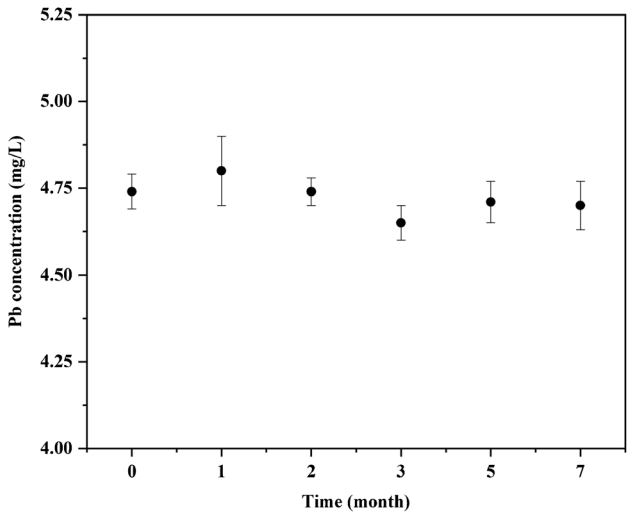

Fig. 3 Results of long-term stability for Pb concentration in the candidate CRM

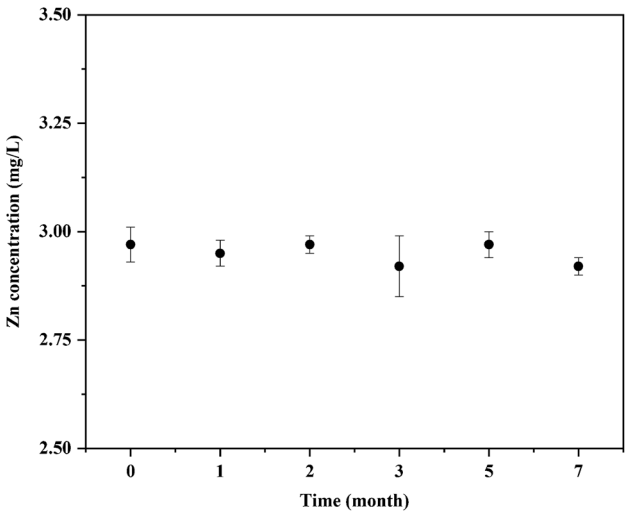

Fig. 4 Results of long-term stability for Zn concentration in the candidate CRM

Table 4 Inter-laboratory comparison results for the assignment of certified values

Lab code	Method	Pb		Zn	
		Average (mg/L)	RSD (%)	Average (mg/L)	RSD (%)
1	ICP-MS	4.72	2.7	2.96	2.0
2	ICP-MS	4.88	2.9	2.99	3.2
3	ICP-MS	4.42	0.5	2.95	0.8
4	ICP-MS	4.80	1.8	2.93	2.3
5	ICP-MS	4.74	1.1	2.93	0.7
6	ICP-MS	4.71	0.4	2.96	0.8
7	ICP-MS	4.87	2.1	3.08	1.7
8	ICP-MS	4.27	2.3	3.10	3.4
9	ICP-MS	4.75	0.4	2.98	2.5
	ICP-OES	4.50	1.0	2.72	2.7
	AAS	4.62	0.6	2.82	3.1

The combined uncertainty of definite value u_{char} is calculated by u_A and u_B as follows:

$$u_{char} = \sqrt{{u_A}^2 + {u_B}^2} \tag{5}$$

The combined uncertainty of short-term stability (u_{sts}) is calculated by $u_{sts(-20℃)}$ and $u_{sts(60℃)}$ as follows:

$$u_{sts} = \sqrt{{u_{sts(-20°C)}}^2 + {u_{sts(60°C)}}^2} \tag{6}$$

The combined uncertainty of stability (u_s) is calculated by u_{sts} and u_{lts} as follows:

$$u_s = \sqrt{{u_{sts}}^2 + {u_{lts}}^2} \tag{7}$$

The combined uncertainty u_{CRM} can be obtained as the combination of uncertainties from the characterization study, homogeneity test, and stability study, which is calculated as the following:

$$u_{CRM} = \sqrt{{u_{char}}^2 + {u_{bb}}^2 + {u_s}^2} \tag{8}$$

The expanded uncertainty U_{CRM} is calculated as follows:

$$U_{CRM} = u_{CRM} \times k \ (\mathrm{k} = 2) \tag{9}$$

To conclude, the uncertainty values of Pb and Zn concentration in the CRM are shown in Table 5.

Conclusions

A certified reference material GBW(E)085538 was developed for accurate determination of the leaching of Pb and Zn in solid waste. Characterization, homogeneity testing, stability assessments, value assignment, and uncertainty evaluation of the CRM were carried out by ICP-MS, ICP-OES, and AAS methods. The standard values and expanded uncertainties of Pb and Zn concentrations in the CRM were 4.66 ± 0.21 and 2.95 ± 0.14 mg/L. The solid waste CRM demonstrated very good homogeneity, as analyzed by ICP-MS method. Long-term stability assessments revealed that Pb and Zn concentrations in the CRM were sufficiently stable for at least 7 months under regular storage conditions (at room temperature), whereas the short-term stability at − 20°C and 60°C was experimentally confirmed for up

Table 5 The certified values and uncertainty of Pb and Zn concentration in the CRM

Components	Parameters (mg/L)	Pb	Zn
Certification uncertainty	u_{char}	0.068	0.046
Homogeneity test	u_{bb}	0.040	0.028
Short-term stability study (− 20℃)	$u_{sts(-20℃)}$	0.020	0.016
Short-term stability study (60℃)	$u_{sts(60℃)}$	0.011	0.020
Combined $u_{sts(-20℃)}$ and $u_{sts(60℃)}$	u_{sts}	0.023	0.026
Long-term stability study	u_{lts}	0.060	0.030
Combined u_{sts} and u_{lts}	u_s	0.064	0.040
Combined uncertainty	u_{CRM}	0.103	0.068
Expanded uncertainty	U_{CRM}	0.207	0.136
Certified values	F	4.66 ± 0.21	2.95 ± 0.14

to 15 days. Moreover, the long-term stability of the CRM will continue to be monitored, and the expiry date will be extended based on the test results. Therefore, the new CRM can be used as an effective tool in testing laboratories for analytical method validation, quality assurance, and quality control. It also plays a significant role in accurate determination of the leaching of Pb and Zn in solid waste.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s00216-023-04912-3.

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Declarations

Competing interest The authors declare no competing interests.

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