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Trace Detection of Pb(II) Using Square Wave Anodic Stripping Voltammetry with Stainless Steel Electrodes

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ABSTRACT. Lead (Pb(II)) is well known as a dangerous environmental contaminant that harms public health worldwide. Early Pb(II) detection before release into the water system is important. This work describes an inexpensive Pb(II) determination using 5 mm diameter stainless steel rod type 304 as a working electrode. Using a batch system, the research employed 10 mL of 0.1 M acetate buffer at pH 4.5. The best operation was at a deposition potential of -1.2V for 300 s. It provides a linear range in the concentration range of 0.075 – 1 µg/mL Pb(II) (r = 0.994). The limit of detection (LoD) and limit of quantification (LoQ) of Pb(II) were at 0.057 µg/mL and 0.189 µg/mL, respectively. Repeatability and reproducibility were expressed in the relative standard deviation range of 1.26 – 3.71% in the testing a Pb(II) concentration range of 0.2-1.0 µg/mL and 5.32% in testing a Pb(II) concentration of 0.4 µg/mL. A very low-cost stainless-steel electrode proposed a high operational stability up to 10 measurements with a Relative Standard Deviation (%RSD) of 4.39%.

INTRODUCTION

Lead is a harmful heavy metal present in the Earth's crust. Its extensive applications have led to environmental pollution, adversely affecting public health globally (Boskabady *et al*., 2018; Zheng *et al*., 2023). Primary sources of lead exposure come from mining, smelting, manufacturing, recycling activity, and use in various things such as fuel, vehicle exhaust, paint, water pipes containing lead, waste disposal, ceramic mixture, dust, and even from the air and soil (Ericson *et al*., 2019; Forsyth *et al*., 2019; Obeng-Gyasi, 2019). Lead poisoning will cause toxic effects in several organs, such as the central nervous system and peripheral system (difficulty concentrating, anemia, and anxiety), cardiovascular system (can cause hypertension), hemopoietic system (anemia), kidneys, absorption, reproductive system, and carcinogenic effects (Bjørklund *et al*., 2023; Lee *et al*., 2019; Mallongi *et al*., 2023; Satarug *et al*., 2020).

Lead exposure can be prevented by early detection. Identifying lead presence early on helps mitigate risks (WHO, 2019). Some electrochemical methods give an alternative choice to analyze heavy metals with high sensitivity, cheapness, ease, and capability to simultaneously analyze several heavy metal ions (Zamhari *et al*., 2017). Of the several techniques available, voltammetry and stripping are the most frequently used to analyze heavy metals because these methods have the highest selectivity and sensitivity (Mirceski *et al*., 2013; Thangavelu *et al*., 2016). This method includes three main stages i.e., separation, preconcentration, and determination in one process (Oztekin *et al*., 2011). The quality of results from this technique is highly affected by the chemical and electrochemical properties of the working electrode (Abollino *et al*., 2019).

Conventional electrodes, such as hanging mercury drop electrodes (HMDE) and bismuth-based electrodes, have been abandoned due to their toxicity (Rodrigues *et al*., 2011; Švancara *et al*., 2010). Eco-friendly electrodes have been becoming the primary concern in this matter lately. Several novelty electrodes, such as screen-printed

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gold electrodes (Wan *et al*., 2015), Fe_3O_4-chitosan nanoparticles (Zhou *et al*., 2016), and glassy carbon electrodes modified with biochar, nanodiamonds, and chitosan have addressed this issue (Wong *et al*., 2020). However, these novelties still need a sufficient cost for their fabrication. Stainless steel is a promising material for use as a working electrode due to its excellent corrosion resistance, high electrical conductivity, and strong mechanical properties, making it a low-cost and friendly environmental electrode. Chromium alloys present in stainless steel contribute to its good corrosion resistance by forming a passive chromium oxide coating that prevents corrosion (Chen *et al*., 2011; Hermas *and* Morad, 2008; Suroso, 2017). The presence of Cr-depleted areas near grain boundaries and reduced carbon content in the austenite crystal lattice contribute to its high electrical conductivity (Radojković *et al*., 2023). Additionally, stainless steel possesses superior mechanical strength, with SS 304 having a tensile strength of 646 MPa, yield strength of 270 MPa, elongation of 50%, and a hardness of 82 HRB (Gardner, 2019; Giao *et al*., 2019; Sumarji, 2011).

This study employed the square wave anodic stripping voltammetry technique (SWASV), applying 5 mm diameter stainless steel (SS) type 304 as the working electrode to determine lead levels. The SS electrode was chosen not only because of its characteristics to meet the required specifications but also because of its safety for the environment, cheapness, and commercial availability. Experimental parameters such as deposition potential and deposition time that affect the electroanalytical signal were optimized. Other parameters such as repeatability and reproducibility, linear range, limit of detection (LoD), and limit of quantification (LoQ) have successfully verified the success of the SS as an alternative working electrode with well-defined and sharp stripping peaks, high sensitivity, and precision. This study provides the use the commercial use of low-cost stainless steel as a working electrode achieved a limit of quantification (LoQ) of 0.189 µg/mL that has never been achieved before.

RESEARCH METHODS

Stainless steel 304 (5 mm diameter) rods were purchased from Rajawali 3D Online Store, Indonesia. Lead (II) nitrate ($Pb(NO_3)_2$) and potassium chloride (KCl) have been purchased from Merck Germany. Glacial acetic acid (CH_3COOH), sodium acetate (CH_3COONa), and nitric acid (HNO_3) were purchased from Merck, Germany. Distilled water was purchased from Alpha Kimia, Indonesia. All chemicals used are analytical grade. Alumina powder (0.1 µm) was purchased from the Kimyong Online Store in Indonesia. Acetate buffer solutions 0.1M (pH 4.5) were used as supporting electrolytes for stripping analysis throughout the experiments.

Electrochemical measurements were performed using Potentiostat (IO Rodeo, USA), which was controlled by a computer. A batch system was implemented with three electrodes. A platinum wire and Ag/AgCl (saturated KCl) were used as counter electrodes and reference electrodes. The stainless steel electrode as a working electrode has been prepared according to the procedure described below. All the experiments were performed at room temperature (26 °C ± 1°C).

Electrode Preparation and Optimization of Experimental Parameters

The type 304 stainless steel rod has been cut to a 4.0 cm length. The side of the stainless steel rod is coated with HDPE from melted plastic bottles to prevent its contact with the buffer system. Before use, the rod was smoothed with emery papers (0.5 and 1.0 grit) and polished alumina slurry (0.1 µm). The polished surface was rinsed with distilled water, and then the electrode was dipped in 0.1 M HNO_3 for 5 minutes. Subsequently, the electrode underwent electrochemical cleaning via amperometry, applying an anodic potential of +0.5 V for 6 minutes. The prepared electrode was optimized by following the experimental parameters of the effect of deposition potential and deposition time in 0.1 M acetate buffer solution (pH 4.5) containing 0.4 µg/mL Pb(II).

Procedure and Analytical Performance

Pb (II) determination was performed by square wave anodic stripping voltammetry (SWASV) in the batch system containing 10 mL of 0.1 M acetate buffer (pH 4.5). The test was carried out by immersing three electrodes into a batch system, where the working electrode use had been optimized. Deposition or accumulation potential of -1.2 V was applied for 300 seconds under stirring throughout the measuring process. Deposition potential helps the metal ion accumulate on the surface of the electrode. However, a potential that is too negative will produce hydrogen bubbles and affect the result. The stirring was stopped for 30 seconds to ensure equilibrium before the measuring process was finished. The SWASV was recorded at -1.2 to 0.5 V. Electrode was cleaned by applying anodic potential +0.5 V for 5 minutes before the next Pb(II) determination. The validation parameters assessed included linearity (linear range), repeatability, reproducibility, stability, and the determination of the limits of detection (LoD) and quantification (LoQ).

RESULTS AND DISCUSSION

Electrode Preparation and Optimization of Experimental Parameters

The polishing process of the working electrode is required to gain the best result for the next experiment. An electrochemical cleaning process is necessary for zero interference before Pb(II) determination. The flat results indicate no peak produced after cleaning, as shown in Figure 1, which means the absence of impurities on the electrode surface. The electrodes received had shown promising results, and this preparation was carried out before Pb(II) determination. The entire electrode preparation was ended by rinsing distilled water on the electrodes to ensure no impurities were left behind.

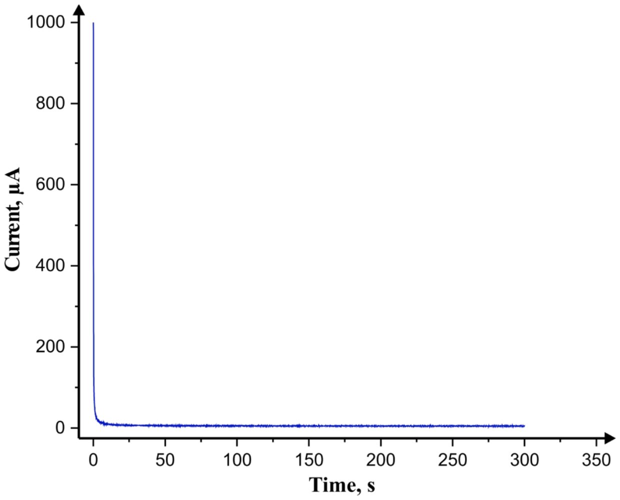

Figure 1. Chronoamperometry cleaning of the stainless steel electrode in 0.1 M HNO_3 solution (0.5 V; 300 s).

A cyclic voltammetry test was conducted to ensure that stainless steel electrodes could give a good signal. This step was investigated in a solution of 1 mM $K_3[Fe(CN)_6)]$ in 0.10 M KCl with stainless steel as working electrode, Ag/AgCl as a reference electrode, and platinum wire as a counter electrode by applying a voltage range of -0.6 V to 0.7 V with a sample rate of 100 Hz and a scan rate of 0.5 V/s. Figure 2 shows that high and wide $[Fe(CN)_6]^{-3/-4}$ peaks at anode and cathode peaks. It is confirmed that the stainless steel electrode has a good performance and is ready for use. To achieve simultaneous determination of Pb(II) using SWASVs at type 304 stainless steel electrodes, the experimental parameters were optimized accordingly.

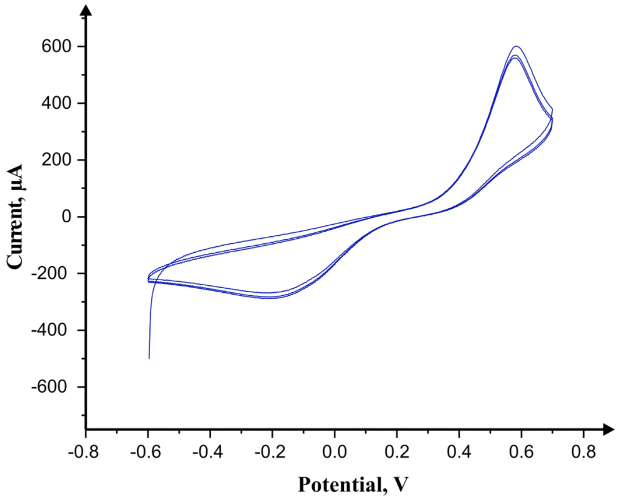

Figure 2. Cyclic Voltammogram of stainless steel electrode in a solution of 1 mM $K_3[Fe(CN)_6)]$ in 0.10 M KCl by cyclic voltammetry method with a voltage range of -0.6 to 0.7 V with a sample rate of 100 Hz and a scan rate of 0.5 V/s.

The effect of deposition time

The duration of preconcentration can enhance the reduction of metal ions on the electrode surface. This means that preconcentration time can increase the sensitivity of the working electrode. The dependence of peak current for Pb(II) was analyzed at a concentration of 0.4 µg/mL Pb(II) within 10 mL of 0.1 M acetic buffer at pH 4.5. Figure 3(a) shows changes in peak current with increasing deposition time. Peak striping current increases with increasing deposition time in 120 to 360 seconds. However, the increasing deposition time above 300 seconds causes an unstable Pb(II) determination peak current might be because the electrode surface was covered by hydrogen gasses produced from the solution, causing inconsistent determination (Wang *et al*., 2019). It can be seen from the high error bar (Figure 3a). Therefore, a deposition time of 300 seconds was selected as the optimum time for further determination.

The effect of deposition potential

The deposition process helps the electrode accumulate the targeted metal onto its surface. The effect of deposition potential was studied from -1.0 V to 1.4 V by varying the applied potential. As shown in Figure 3(b), the Pb(II) signal increased along with the potential shift from -1.0 to -1.3 V. It might be because the more negative deposition potential will affect the deposition of metal ion onto the surface of the working electrode (Wang *et al*., 2019). However, a more negative deposition potential than -1.2 V can affect unstable peaks of Pb(II) determination, as shown in the test results on deposition potential -1.3 V. It might be because a lot of hydrogen gas was formed and covered the electrode surface when the test was carried out at a deposition potential of less than -1.2 V. Therefore, a deposition potential of -1.2 V was selected for further determination.

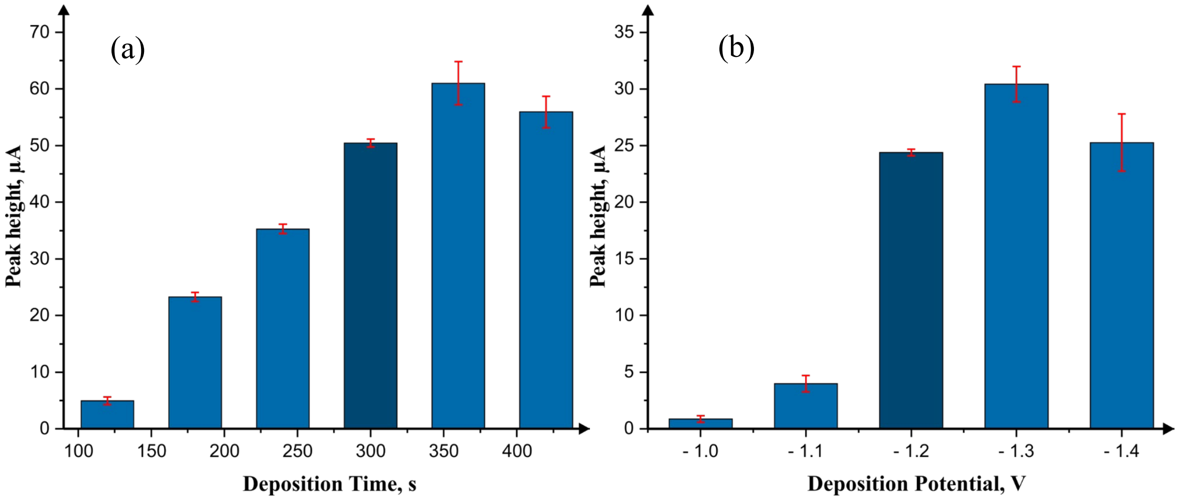

Figure 3. Effect of (a) deposition time, (b) deposition potential containing 0.4 µg/mL Pb(II); SWASV setting: frequency 20Hz, potential step 4 mV, and amplitude 25mV.

Analytical Performance

Linier Range, Limit of Detection (LoD), and Limit of Quantification (LoQ)

A linear range or calibration curve is tested to determine the range of Pb(II) concentration that can be tested by the developed method. This linear range was carried out with a Pb(II) concentration of 0.000 µg/mL to 1000 µg/L. Figure 4(a) shows the voltammogram of the Pb(II) test using a stainless steel electrode containing 0.1 M acetate buffer (pH 4.5). The method used was Square Wave Anodic Stripping Voltammetry with a sample rate of 20 Hz, accumulation or deposition potential (Ea) of -1.2 V for 300 seconds, and voltammetry ranges from -1.2 V to 0.5 V. The voltammogram shows a peak of Pb(II) at -0.46 V. This indicates that Pb(II) is released from the electrode surface and returns to the solution at a potential of -0.46 V. Figure 4(b) shows that the developed method has linear ranges in the range of 0.075 – 1.000 µg/mL (R^2 = 0.994). The limit of detection (LoD) and limit of quantification (LoQ) of Pb(II) were 0.057 µg/mL and 0.189 µg/mL, respectively. The LoD value covers the maximum Pb(II) level in the drinking water, 2.000 µg/mL. Table 1 shows that the inexpensive electrode of 5 mm diameter stainless steel successfully detected the Pb(II) with acceptable LoD and wide linear ranges. With the comparable result, this work offers a low-cost and eco-friendly working electrode. The stainless steel rod can be achieved with a price lower than USD 1.

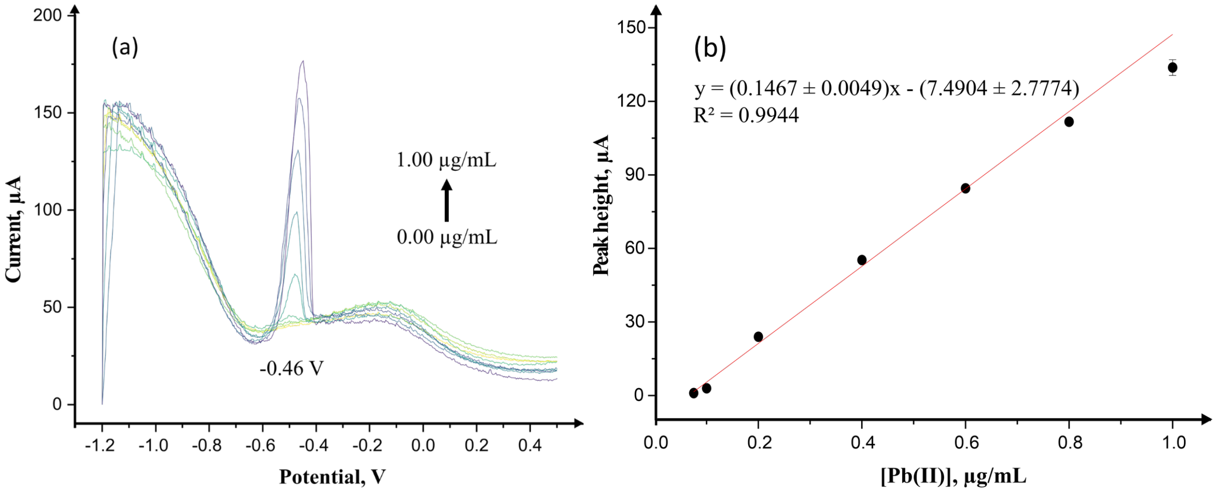

Figure 4. (a) voltammogram, (b) calibration curve of Pb(II) determination in 10 mL of 0.1 M acetate buffer pH 4.5 with a concentration range of 0.00 – 1.00 µg/mL.

Table 1. A comparison of the performance of the stainless steel electrode with the other electrodes.

Electrode	Method	LoD (µg/mL)	LoQ (µg/mL)	Linearity (µg/mL)	References
Stainless steel	SWASV	0.0570	0.1890	0.075 – 1.000	This work
SnO$_2$ modified electrode	SWASV	0.0021	0.0053	0.006 – 0.020	(Lameche *et al.*, 2023)
Fe$_3$O$_4$-chitosan modified GCE	SWASV	0.0087	0,0291	0.021 – 0.103	(Zhou *et al.*, 2016)
Glassy carbon electrode modified with biochar	SWASV	0.0116	0.0386	0.050 – 1.240	(Wong *et al.*, 2020)
Cork-graphite	SWASV	0.0620	0.2070	0.200 – 5.200	(Wang *et al.*, 2019)
Zn-Fe LDH/PANI	DPV	0.0350	0.1160	0.200 – 0.800	(Kamel *et al.*, 2023)
GCE modified with GQDs and NF	SWASV	0,0084	0,0283	0.020 – 0.200	(Pizarro *et al.*, 2020)
In situ Bi/carboxyphenyl-modified GCE	SWASV	0.0100	0.0330	0.025 – 0.500	(Phal *et al.*, 2021)
boron-doped diamond film electrodes	ASV	0.0012	0.0037	0.002 – 0.040	(Ferreira *et al.*, 2021)

Repeatability and Reproducibly

Precision tests are carried out using repeatability and reproducibility. Repeated investigations were carried out by testing Pb(II) in solution six times in a concentration range of 0.2 – 0.8 µg/mL. The repetition results reveal that %RSD is in the range of 1.26 – 3.71% Figure 5(a), which is still below the 11% set by AOAC in that concentration range (AOAC, 2012). Reproducibility was investigated for the Pb(II) assay using six electrodes on different days. The test was carried out in a 0.2 – 0.8 µg/mL Pb(II) concentration. The research results show good reproducibility with an %RSD of 5.32% Figure 5(b), which is still below the 16% set by AOAC. They revealed that the repeats for detecting Pb(II) provide good precision.

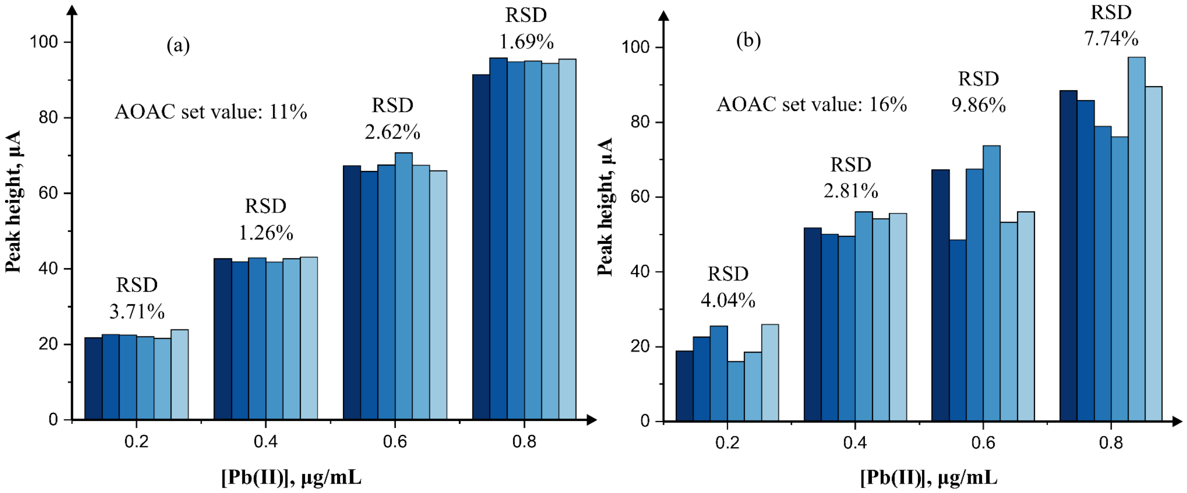

Figure 5. (a) Repeatability, (b) reproducibility study on Pb(II) in a concentration range of 0.2 – 0.8 µg/mL using stainless steel electrodes.

Operational Stability

This research was studied to determine the performance of the method developed by testing the analytes repeatedly. Stability tests were conducted at a Pb(II) concentration of 0.4 µg/mL. This investigation shows that 5 mm stainless steel electrodes can be used simultaneously for 10 Pb(II) tests (Figure 6). As relative response, the first result was set as 100% and the rest was compared to the first response. Prior to the change of 10%, both higher and lower, were set as the stability of the method. The eleventh test resulted in a response change of 11.53%. The average percentage obtained was 97.89%, with an %RSD of 4.39%. It reveals a method of performing acceptable results with low random error in determining Pb(II).

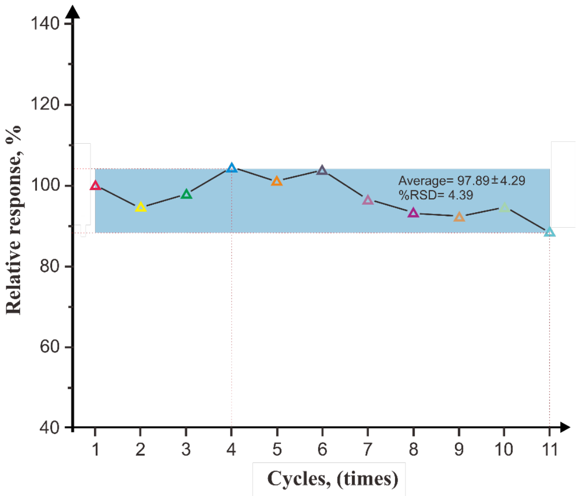

Figure 6. Stability investigation of a 5 mm-diameter stainless steel electrode to detect Pb(II) at a concentration of 0.4 µg/mL.

CONCLUSION

Stainless steel rod type 304 as a working electrode has promising results with suitable electrodes and inexpensive Pb(II) determination methods. The validation results show that stainless steel electrode has a linear range in the concentration range of 0.075 – 1.000 µg/mL Pb(II), good repeatability and reproducibility, accepted by AOAC, and high operational stability up to 10 measurements.

CONFLICT OF INTEREST

The authors state that there are no conflicts of interest related to the publication of this article. They have no ties or involvement with any organizations or entities that have financial or non-financial stakes in the content or materials presented in the article.

AUTHOR CONTRIBUTION

SH: Conducted research, drafted original manuscript, validated data, developed methodology, performed formal analysis, and conceptualized the study. MZ: Edited and reviewed the manuscript, drafted original manuscript, validated data, supervised research, developed methodology, secured funding, performed formal analysis, and conceptualized the study. AFA: Conducted research, validated data, developed methodology, and performed formal analysis. SNF: Conducted research, validated data, developed methodology, and performed formal analysis. TK: validate data and methodology.

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