

# Square wave anodic stripping voltammetric determination of $\text{Pb}^{2+}$ using acetylene black paste electrode based on the inducing adsorption ability of $\text{I}^-$

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## Abstract

Herein, a sensitive and simplified electrochemical method was proposed for the determination of trace levels of  $\text{Pb}^{2+}$  by anodic stripping voltammetry (ASV) based on the inducing adsorption ability of  $\text{I}^-$  toward  $\text{Pb}^{2+}$ . In the presence of low concentration of  $\text{I}^-$ ,  $\text{Pb}^{2+}$  was induced to accumulate onto the acetylene black (AB) paste electrode surface, and then reduced at  $-0.90$  V. During the following square wave sweep from  $-0.90$  to  $-0.30$  V, the reduced Pb was oxidized, resulting in a sensitive and well-shaped stripping peak at  $-0.56$  V. Further studies indicate that low concentration of  $\text{I}^-$  significantly enhances the sensitivity of determination of  $\text{Pb}^{2+}$ . After all the experimental parameters were optimized, a novel and sensitive method was developed for the electrochemical determination of  $\text{Pb}^{2+}$ . The linear range is found to be from  $2.0 \times 10^{-8}$  to  $4.0 \times 10^{-6}$   $\text{mol L}^{-1}$ , and the lowest detectable concentration is estimated to be  $6.0 \times 10^{-9}$   $\text{mol L}^{-1}$ . This newly proposed method was finally demonstrated with water samples. Otherwise, the anodic stripping responses of  $\text{Pb}^{2+}$  on AB paste electrode and graphite paste electrode were compared.

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## 1. Introduction

The pollution caused by heavy metal ions is becoming more and more severe all over the world, promoting extensive attention into developing sensitive, reliable and rapid analytical method. Lead is toxic and linked to various adverse health effects [1,2].  $\text{Pb}^{2+}$  easily accumulates in the environment and produces toxic effects on plants and animals, even at low concentrations [3–5] since  $\text{Pb}^{2+}$  is not biodegradable. Therefore, it is very valuable and welcome to develop rapid and sensitive method for the determination of  $\text{Pb}^{2+}$ .

The commonly used method for  $\text{Pb}^{2+}$  analysis is atomic spectrometry, including atomic absorption spectrometry (AAS) and atomic emission spectrometry (AES). These methods have high sensitivity and excellent selectivity, however, they have the intrinsic drawbacks: requirement of complicated and expensive instruments, high cost, not for in situ measurement, et al. Dislike

this, electrochemical method is attractive for in situ determining  $\text{Pb}^{2+}$  since it exhibits high sensitivity, good selectivity, rapid response, easy data read-out and low cost. What is more, the instruments employed in electrochemical method are relatively simple, and conveniently miniaturized for in situ and automated detection.

Anodic stripping voltammetry (ASV) is an ideal electrochemical technique for the determination of trace levels of metal ions because it possesses very high sensitivity. In the past years, mercury electrodes, consisting of dropping mercury electrode (DME), hanging drop mercury electrode (HDME) and thin mercury film electrode (MFE), were dominantly used in ASV. This is due to the predominant properties of mercury electrode: easy formation of amalgam with reduced metal, excellent reproducibility, easy surface renewal. However, mercury itself is also toxic, so numerous efforts have been made to develop various mercury-free solid electrodes. Among these mercury-free solid electrodes, paste electrode (also called carbon paste electrode) has obtained increasing attention and extensively used in electroanalysis or electrochemistry since it possesses following advantages: easy preparation, porous surface, wide potential

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range (from  $-1.40$  to  $+1.30$  V), low residual current, low cost and convenient surface renewal. To date, all kinds of mercury-free solid electrodes or chemically modified solid electrodes (CMEs) have been reported for the determination of  $\text{Pb}^{2+}$  [6–11]. However, to the best of our knowledge, determination of trace levels of  $\text{Pb}^{2+}$  using acetylene carbon black (AB) paste electrode based on the inducing adsorption ability of  $\text{I}^-$  is firstly reported here.

The main objective of the current work is to develop a sensitive and convenient electrochemical method for the determination of  $\text{Pb}^{2+}$  utilizing the excellent properties of AB as well as the inducing adsorption ability of  $\text{I}^-$  toward  $\text{Pb}^{2+}$ .

AB, a special type of carbon black, is made by the controlled combustion of acetylene in air under pressure. Due to its excellent electric conductivity, large specific surface area and strong adsorptive ability, AB has been widely used in electrochemistry and electroanalytical chemistry. Otherwise, previous reports [12,13] prove that  $\text{I}^-$  can induce some metal ions to adsorb at electrode surface, and proper mechanism has been given. To achieve the goal, low concentration of KI was added into supporting electrolyte to induce  $\text{Pb}^{2+}$  to accumulate onto AB paste electrode, significantly enhancing the surface amount of  $\text{Pb}^{2+}$  as well as its stripping peak current. Therefore, the sensitivity of  $\text{Pb}^{2+}$  analysis was remarkably improved in the presence of  $\text{I}^-$ . This new sensing and determining system possesses following advantages: extreme simplicity, low detection limit, free of mercury and low cost.

## 2. Experimental section

### 2.1. Reagents

Stock solution of  $1.00 \times 10^{-2} \text{ mol L}^{-1} \text{ Pb}^{2+}$  was prepared by dissolving  $\text{Pb}(\text{NO}_3)_2$  (Shanghai Reagent Corporation, China) into redistilled water, and then diluted to working solution at desired concentration with redistilled water. Other chemicals used are analytical reagents and water used is re-distilled.

Acetylene carbon black (purity >99.99%, particle size = 150–200 nm) was purchased from STREM Chemicals (USA). Graphite powder (spectral reagent) and paraffin oil were purchased from Shanghai Reagent Corporation, China.

### 2.2. Instruments

Electrochemical experiments were carried out using a CH Instruments 650B electrochemical workstation (CH Instrument, Austin, TX). A conventional three-electrode system, consisting of an AB paste working electrode, a saturated calomel reference electrode (SCE) and a platinum wire auxiliary electrode, was employed. The body of working electrode was a polytetrafluoroethylene (PTFE) cylinder that was tightly packed with AB paste. A copper wire inserted into the paste providing electrical contact.

Atomic absorption spectrometric measurements were conducted with AA 6300 Atomic Absorption spectrophotometer (SHIMADZU, Japan).

### 2.3. Preparation of AB paste electrode

The AB paste electrode was prepared by mixing 100.0 mg AB and 100.0  $\mu\text{L}$  paraffin oil in a small mortar to form a homogeneous AB paste. Then the paste was pressed into the end cavity (3-mm in diameter, 1-mm in depth) of working electrode body. The electrode surface was smoothed against weighing paper. It is important to note that the amount of paraffin oil must be carefully controlled because excessive paraffin oil will lower the conductivity of AB paste, while insufficient paraffin oil is not beneficial to obtain uniform AB paste. The graphite paste electrode was also prepared as above-mentioned procedure but the ratio of graphite powder and paraffin oil is 100.0 mg–40.0  $\mu\text{L}$ .

### 2.4. Analytical procedure

Unless otherwise stated,  $0.1 \text{ mol L}^{-1} \text{ HClO}_4$  solution containing  $7.00 \times 10^{-3} \text{ mol L}^{-1} \text{ KI}$  was used as supporting electrolyte for  $\text{Pb}^{2+}$  determination. The accumulation step was proceeded at  $-0.90$  V for a desired time with stirring solution, then the square wave stripping voltammograms were recorded from  $-0.90$  to  $-0.30$  V after 15 s quiescence. The peak current was measured at  $-0.56$  V for  $\text{Pb}^{2+}$ . After each measurement, the used paste was carefully removed from the end cavity and a new AB paste was pressed into. That is to say, each AB paste electrode was used one time to achieve better reproducibility.

## 3. Results and discussion

### 3.1. Electrochemical response of $\text{Pb}^{2+}$ at AB paste electrode

Fig. 1 shows the anodic stripping voltammetric responses of  $\text{Pb}^{2+}$  in different conditions. After 4-min accumulation at  $-0.90$  V in  $0.1 \text{ mol L}^{-1} \text{ HClO}_4$  solution, an ill-shaped

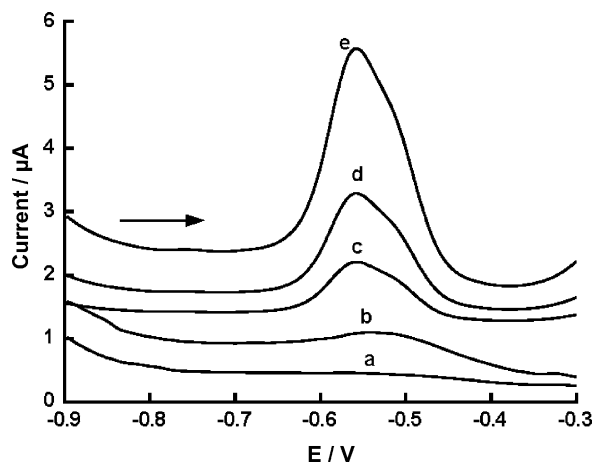


Fig. 1. Square wave anodic stripping voltammograms of  $\text{Pb}^{2+}$  in  $0.1 \text{ mol L}^{-1} \text{ HClO}_4$  at AB paste electrode: (a) blank voltammograms; (b)  $1.0 \times 10^{-7} \text{ mol L}^{-1} \text{ Pb}^{2+}$ ; (c–e)  $5.0 \times 10^{-8}$ ,  $1.0 \times 10^{-7}$ ,  $2.0 \times 10^{-7} \text{ mol L}^{-1} \text{ Pb}^{2+}$  in presence of  $7.0 \times 10^{-3} \text{ mol L}^{-1} \text{ KI}$ . Accumulation potential:  $-0.90$  V, accumulation time: 4 min, pulse amplitude = 25 mV, frequency = 25 Hz, potential increment = 4 mV. Arrow means potential sweep direction.

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