



On-line coupling of sequential injection lab-on-valve to differential pulse anodic stripping voltammetry for determination of Pb in water samples

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ABSTRACT

Sequential injection lab-on-valve (LOV) was first proposed for analyzing ultra-trace amounts of Pb using differential pulse anodic stripping voltammetry (DPASV) with a miniaturized electrochemical flow cell fabricated in the LOV unit. Deposition and stripping processes took place between the renewable mercury film carbon paste electrode and sample solution, the peak current was employed as the basis of quantification. The mercury film displayed a long-term stability and reproducibility for at least 50 cycles before next renewal, the properties of integrated miniature LOV unit not only enhanced the automation of the analysis procedure but also declined sample/reagent consumption. Potential factors that affect the present procedure were investigated in detail, i.e., deposition potential, deposition time, electrode renewable procedure and the volume of sample solution. The practical applicability of the present procedure was demonstrated by determination of Pb in environmental water samples.

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1. Introduction

Differential pulse anodic stripping voltammetry (DPASV) has proved a powerful method for the characterization and quantitative detection of trace metals, analyte preconcentration onto a working electrode surface and metal deposition/stripping detection steps were involved during one electroanalytical circulation. Compared with many other analytical techniques, this procedure was recognized as the sensitive technique by features such as simplicity of use, great sensitivity and low cost of instrument [1,2]. However, routine DPASV procedure was laborious and large sample/reagent consumption when using batch mode operations, from this respect, which was not suitable for expensive samples analysis.

Sequential injection lab-on-valve (SI-LOV) introduced by Ruzicka provided an excellent alternative for sample pretreatment [3–8], which integrated all necessary laboratory facilities for a variety of sample manipulation operations, precise liquid manipulation, microliter levels sample and reagent consumption. So far, the sequential injection LOV system has exhibited many practical applications as the sample pretreatment front end [9–14]. In valve renewable beads were utilized as adsorption materials to separate and preconcentrate trace analytes coupled to electrother-

mal atomic absorption spectrometry (ETAAS) [15–18], inductively coupled plasma mass spectrometry (ICPMS) [19], atomic fluorescence spectrometry (AFS) [20] and liquid chromatography (LC) [21], these schemes eliminated the disadvantages of the conventional solid-phase extraction approaches. The lab-on-valve has also proved to be a new platform for miniaturization of instrumentation, a miniature atomic fluorescence spectrometric in the lab-on-valve system was designed for mercury and arsenic analysis, respectively [22–24]. In addition, by inserting optical fiber into the channels of multi-purpose flow cell in the lab-on-valve, fluorescence detection mode can be obtained and the applications of the above-mentioned procedure were demonstrated by DNA detection and purification [25,26].

The purpose of this study is to develop a simple, low sample/reagent consumption and automatic on-line anodic stripping voltammetry detection procedure coupled with SI-LOV system. The electrochemical flow cell (EFC) was designed as a part of the LOV unit, three electrodes were fabricated into the channels of LOV EFC, by on-line plating mercury solution onto the surface of carbon paste working electrode, a rapid separation/enrichment and determination procedure can be achieved when the sample solution passing through the EFC, the produced current during the stripping step can be associated with the amount of the analyte in the solution.

The feasibility of the established system was demonstrated by lead detection in environmental water samples. It has been widely recognized that exposure to the excessive lead can result in high

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blood pressure and hypertension, damage to the brain and nervous system with high levels of lead accumulation in their bodies [27,28]. In this respect, particular attention should be paid to screen the concentration of lead in the environmental samples because of their irreversible deleterious effects on the natural environment and the health of human [29,30]. Thus, lead was chosen as a model analysis material. Compared with batch mode operations, not only sample and reagent consumption were reduced, but also sampling frequency was enhanced, this characteristic is especially beneficial for minimizing electrochemical sample/reagent consumption and automatic analysis.

2. Experimental

2.1. Instrument

The sequential injection lab-on-valve system used in the present work consists of three independent components, as described in Fig. 1. A FIALab-3000 sequential injection system (FIALab Instruments, Bellevue, WA, USA) equipped with a 2.5 mL syringe pump (Cavro, Sunnyvale, CA, USA) was employed for sample and reagent delivery. The central part of the system, i.e., a homemade LOV unit, which was made of transparent plexiglass, incorporated an electrochemical flow cell with a volume of ca. 200 μL . The sampling port was set to port #3 while the waste was destined for port #8, the central channel was connected to a holding coil and other remaining ports were individually communicated with the central channel for fluidic arranged. The electrochemical signals measurements were performed with CHI660A electrochemical workstation (Chenhua Instrument, Shanghai, China).

A detailed electrochemical flow cell configuration is illustrated in Fig. 1, a three-electrode system consists of a 3 mm i.d. homemade carbon paste working electrode (CPE), a Ag/AgCl (3 mol L⁻¹ KCl) reference electrode (RE) and a platinum wire auxiliary electrode. These electrodes were encapsulated into the LOV unit in line and the CPE was placed in the middle of the flow cell. In addition, the air bubbles in the solutions may adsorb on the electrode surface, which could cause negative effect in the deposition/stripping procedure. To minimize such effect, the port #7 of flow cell was

designed to connect port #6 in the valve via a connecting tube, thus, the air bubbles adsorption could effectively minimize through this interface design.

All externally used tubes were made of 0.8 mm i.d. PTFE tubing (Upchurch Scientific, Oak Harbor, WA, USA) and the capacity of holding coil was 2.5 mL. The operations of the sequential injection lab-on-valve and data acquisition systems were synchronously controlled by FIALab software and CHI660A Electrochemical Workstation for Windows, respectively.

2.2. Chemicals

All reagents used were at least of analytical reagent grade, and de-ionized water (18 M Ω cm⁻¹) was used throughout.

A 1000 mg L⁻¹ Pb stock solution were prepared by dissolving 0.1598 g Pb(NO₃)₂ (Jinshan Chemicals, Shanghai, China) in 100 mL 1% (v/v) nitric acid. A 500 mg L⁻¹ Hg (II) stock solution were prepared by dissolving 0.06778 g HgCl₂ (Tongyin Chemicals, Guizhou, China) in 100 mL 1% (v/v) hydrochloric acid. Working standard solutions were obtained by step-wise dilution. 0.1 mol L⁻¹ acetate buffer (pH 4.6) was prepared by mixing the appropriate amounts of sodium acetate (Shanghai Chemicals Co., Shanghai, China) and glacial acetic acid (99.5%, Shanghai Chemicals Co., Shanghai, China). De-ionized water was employed as carrier stream and nitrogen gas (99.999%) was used for purging oxygen in a solution to provide an inert atmosphere inside the flow cell.

2.3. Preparation and activation of the carbon paste electrode

The working electrode was pretreated as follows: 0.5 g spectroscopic grade carbon powder and 0.3 g silicone oil were blended in an agate mortar and homogenized by a pestle, then the mixture were firmly packed into a 3 mm i.d. PTFE tube, into which a copper wire was plugged to establish electrical contact with the exterior equipment. Prior to measurement, the electrode surface was polished manually to obtain a fresh surface, then, the CPE was immersed in the 0.5 mol L⁻¹ H₂SO₄ supporting electrolyte and cycled from -1.0 to 1.0 V at the scan rate of 100 mV s⁻¹ until the reproducible background was obtained.

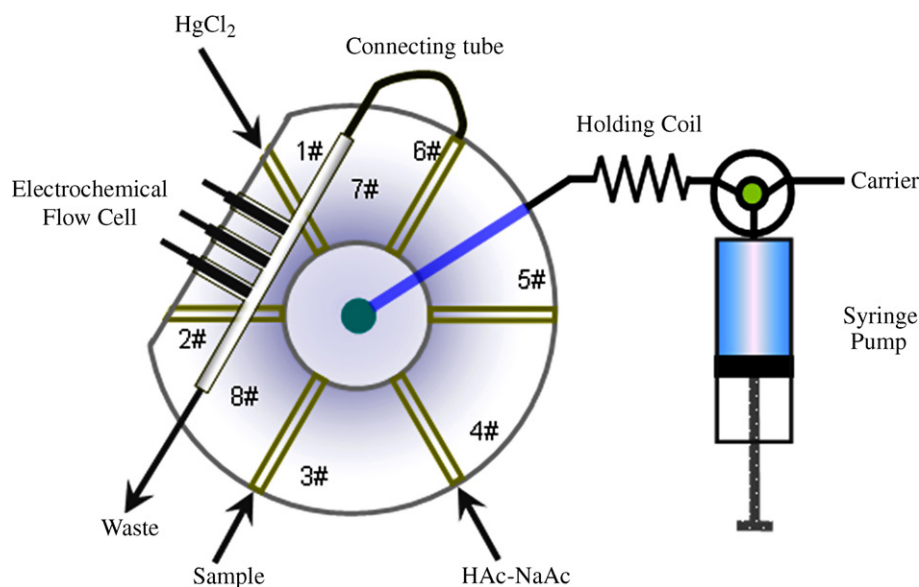


Fig. 1. Flow manifold of the sequential injection lab-on-valve (LOV) system with an integrated electrochemical flow cell for Pb determination with differential pulse anodic stripping voltammetry.

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