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Rapid determination of lead extracted by acetic acid from glazed ceramic surfaces by flow injection on-line preconcentration and spectrophotometric detection

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Abstract

A rapid method has been developed for the determination of lead extracted by acetic acid from glazed ceramic surfaces by flow injection analysis without any pretreatment. An aliquot of 4% acetic acid solution, which has been kept in a teacup for 24 h in the dark, is injected into a carrier solution (1 M nitric acid) and passed through a Pb-Spec resin column. After washing the column with an ammonium nitrate solution, the lead adsorbed on the column is eluted with an ammonium oxalate solution and then merged with a 4-(2-pyridylazo)resorcinol (PAR) solution, followed by measurement of the absorbance of the lead–PAR complex at 530 nm. The detection limit, concentration giving a signal equal to three times the standard deviation of the blank signal, is 8 ng ml^{-1}. The relative standard deviation of measurements at the 0.8 μg ml^{-1} level is 0.35% ($n = 5$). The sample throughput is 12 per hour.

Keywords: Lead determination; Flow injection; Glazed ceramic

1. Introduction

Lead is one of the most toxic metals. It adversely affects the central and peripheral nervous systems and the kidney [1]. However, its unique properties make itself to be used as industrial material in diverse fields. Glaze is a typical example of materials containing lead. It is a thin layer of liquid, which is put on a piece of pottery and becomes hard and shiny when the pottery is heated in a hot oven. The possibility of elution of the lead from the surface is, therefore, present in some of glazed ceramic (dinnerware). Hence, the Japan food sanitation law limits the amount of lead released by 4% acetic acid from glazed ceramic (dinnerware) surfaces: the limits for lead specified in the law are 17 μg cm^{-2} for flatware of the internal depth <25 mm, 5 mg l^{-1} for hollow ware of the capacity <1.1 l and 2.5 mg l^{-1} for hollow ware of the capacity >1.1 l.

The Japanese official method for the determination of the lead released from the surface of dinnerware is tedious and time-consuming because the 4% acetic acid solution, which is kept in dinnerware to be tested for 24 h in the dark, must be evaporated up and treated with hydrochloric acid to improve the precision of the determination of lead by FAAS [2]. The aim of the present work is to develop a rapid method for determining the lead extracted by acetic acid from glazed ceramic surfaces.

The various flow injection methods have so far been proposed for the determination of trace lead in diverse samples. Most of the reported methods employed the on-line preconcentration coupled with the appropriate detection. The following examples have been reported for these several years.

Lead in drinking water was preconcentrated as 2-(5-bromo-2-pyridylazo)-5-diethyl aminophenol complexes on a mini-column packed with Amberlite XAD-16 prior to its determination by ICP-AES using pneumatic nebulization [3]. A flow injection method using a mini-column loaded with 8-hydroxyquinoline immobilized on controlled pore glass was also described for the determination of trace lead along with copper, cadmium, zinc, nickel and iron by ion chromatography [4]. On-line preconcentration and simultaneous determination of heavy metal ions in different water samples

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by ICP-AES was carried out using retention of diethyldithiocarbamate chelates on an octadecyl silica mini-column [5]. Trace lead in a variety of water samples was collected as lead hydroxide precipitates onto the inner walls of a knotted reactor and dissolved by dilute nitric acid to be delivered to FAAS [6]. Ion-exchange for preconcentration and elimination of interferences was also used for the spectrophotometric determination of lead in water samples [7]. Methylthiosalicylated silica gel and chitosan were used for preconcentration of lead for ICP-AES determination [8] and spectrophotometric detection of lead–dithizone complex in aqueous medium [9], respectively. Lead in seawater was retained on a mini-column containing Amberlite XAD-4 impregnated with 1-(2-pyridylazo)-2-naphthol [10] or was complexed with 8-hydroxyquinoline-5-sulfonic acid (8-HQS) and then collected on a mini-column filled with florisil [11]. Total lead and lead isotope ratios in natural waters were determined using sorption of lead complexes with 1-phenyl-3-methyl-4-benzoylpyrazol-5-one on the inner walls of PTFE knotted reactor in advance of the on-line ICP-TOFMS detection [12].

Lead in water and sediment collected as 1-nitroso-2-naphthol complexes on a Diaion HP-20 column for FAAS determination [13]. The simultaneous determination of lead and cadmium in river water and soil was accomplished utilizing in-line cation exchange separation and spectrophotometric detection [14]. Lead in wine and water was preconcentrated on a mini-column filled with polyurethane form modified with 2-(2-benzothiazolylazo)-*p*-cresol [15] or Pb-Spec resin [16] for the FAAS determination of lead. Cadmium, copper and lead in wine were determined by FAAS after solid phase extraction of diethyldithiophosphate complexes on a mini-column filled with C18 bonded silica gel or powdered polyethylene as sorbent [17]. For the determination of lead in environmental samples, the on-line formed lead–pyrrolidinedithiocarbamate complex was sorbed on the polyurethane form, subsequently eluted by 2-methyl-4-pentanone, and determined by FAAS [18]. The determination of lead in biological samples was carried out using a flow injection on-line micelle-mediated preconcentration and separation without phase separation coupled with electrothermal atomic absorption spectrometry (ETAAS) [19]. A chelating resin, Muromac A-1 [20], and a new packing material, acrylic acid grafted PTFE fibers [21], were used for on-line preconcentration of lead in urine, and environmental and biological samples, respectively.

The aforementioned techniques are accurate, but they are time-consuming. The present paper will describe the rapid method for the determination of lead in 4% acetic acid, which had been used to extract lead from the surface of glazed ceramic, by coupling the on-line preconcentration of lead with a Pb-Spec resin (EIChrom Industries, Darien, IL) column with the spectrophotometric detection. In the previous papers, we have already described the flow injection determination of lead in iron and steel [22], river water [23] and seawater [23,24] using Pb-Spec resin for on-line preconcentration of lead and atomic spectroscopic detection. Pb-Spec resin is prepared by impregnating Amberchrom CG-71md resin (Supelco) with an isodecanol solution of bis-4,4′(5′)-[*tert*-butylcyclohexano]-18-crown-6 and exhibits the high selectivity for lead [25].

2. Experimental

2.1. Instrumentation

The block diagram of FIA system used for the spectrophotometric determination of lead is shown in Fig. 1. PTFE tubing (0.5 mm i.d.) was used to construct the manifold. The carrier and washing solutions, eluent and reagent solution were delivered by a pulseless pump PUD-16 (GL Sciences Inc., Tokyo), a mini-chemical pump NP-KX-120U (Nihon Seimitsu, Tokyo) and a double plunger type pump DMX-2000 (Sanuki Kogyo, Tokyo), respectively, and a six-way auto switching valve MPV-6A (GL Sciences Inc., Tokyo) used for sample injection was filled with sample solutions by a peristaltic mini pump SJ-1211 (Atto, Tokyo). The absorbance of the colored lead complex was monitored by a visible detector S-3250 (Soma Optics, Tokyo) and recorded by a chart recorder Shimadzu U-135 (Shimadzu, Tokyo).

An SII 1700 ICP spectrophotometer (Seiko Instruments, Tokyo) was used under the experimental conditions summarized in Table 1 for the measurement of the distribu-

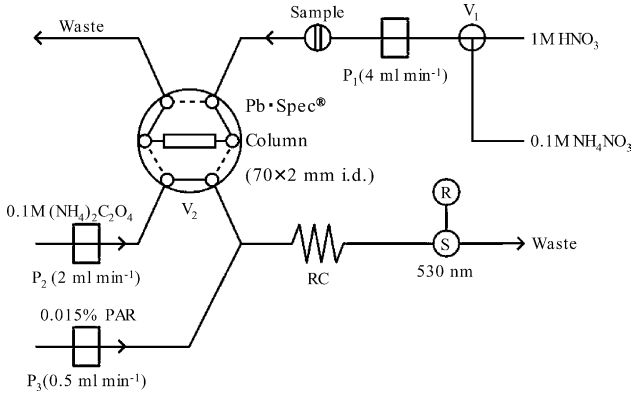

Fig. 1. Flow injection system for the determination of lead. P: pump; V: valve; RC: reaction coil; S: spectrophotometric detector; R: recorder.

Table 1
Operating conditions of ICP-AES[a]

Plasma conditions	
Incident RF power (kW)	1.3
Plasma gas (l min^{-1})	18.0
Auxiliary gas (l min^{-1})	0.5
Carrier gas (l min^{-1})	0.45
Sampling conditions	
Observation height (mm)	12
Sample flow rate (ml min^{-1})	1.0
Integration time	4 s × 3 times
Wavelength (nm)	220.422

[a] Seiko Instruments Plasma Spectrometer SPS 1700HVR.