



Combination of knotted reactor with portable tungsten coil electrothermal atomic absorption spectrometer for on-line determination of trace cadmium



Xiaodong Wen*, Shengchun Yang, Haizhu Zhang, Qingwen Deng

College of Pharmacy and Chemistry, Dali University, Dali, Yunnan 671000, China

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ABSTRACT

In this work, flow injection (FI) on-line precipitation–dissolution in a knotted reactor (KR) was established and combined with a portable tungsten coil electrothermal atomic absorption spectrometer (W-coil ET-AAS) firstly for preconcentration and determination of ultra-trace cadmium. As a new instrument, the application of the portable W-coil spectrometer was expanded to carry out on-line preconcentration and detection through the coupling with a KR system. A self-assembled FI system was employed to hyphenate the KR system with W-coil ET-AAS. The instrumental conditions and influencing factors relevant to KR efficiency, such as concentration of ammonia, pH, conditions of sampling and elution were studied systematically. The coupling improved the analytical performance of the portable spectrometer considerably. Under the optimal conditions, the limit of detection (LOD) for cadmium was 0.006 µg/L, with sensitivity enhancement factor (EF) of 41. The established method could be expanded and used for on-line preconcentration and detection of some other trace metal ions.

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1. Introduction

Accurate analysis of ultra trace metal elements has been an academic topic for the application of analytical chemistry. Atomic absorption spectrometry (AAS) is an important and effective means of metal element analysis. Among the various instrumental techniques, electrothermal atomic absorption spectrometry (ET-AAS) is relatively cost-effective with high sensitivity, and graphite furnace atomic absorption spectrometry (GF-AAS) occupies an important position. As another method of ET-AAS, tungsten coil electrothermal atomic absorption spectrometry (W-coil ET-AAS) has gained attention for the potential to provide portable instrument and field analysis [1].

The portable tungsten coil electrothermal atomic absorption spectrometer is a new developed electrothermal atomic absorption spectrometer based on a tungsten coil atomizer, charge coupled device (CCD) [2,3] and years of academic accumulation in the filed of W-coil ET-AAS [4–8]. This instrument is designed as portable spectrometer, which can be applied in field analysis and move traditional AAS analysis out of lab.

When portable W-coil spectrometer is applied to field analysis, some rapid and simple preconcentration and separation methods are still necessary before instrumental analysis due to low concentrations

and matrix effects of environmental and biological samples. In our previous works, many preconcentration methods have been coupled with this instrument to improve its analytical performance and expanded its applications [1,6–13]. But almost all the methods were coupled with W-coil ET-AAS through the off-line mode. In this work, a KR preconcentration method was firstly combined with the portable instrument by FI system for on-line preconcentration and determination.

Knotted reactor (KR) was made from polytetrafluoroethylene (PTFE) tubing for the flow injection on-line separation/preconcentration of trace metals, which combined with different atomic spectrometric techniques [7]. The use of KR for the online sorption preconcentration combined with FAAS determination was first reported by Fang et al. [14]. Thereafter, the use of KR has been greatly extended to its applications through the combination with FAAS [7], atomic fluorescence spectrometry (AFS) [15], ET-AAS [16,17], inductively coupled plasma mass spectrometry (ICP-MS) [18], etc. On-line coprecipitation involving collection of precipitates on a PTFE or Microline KR was proposed by Fang et al. as an effective preconcentration method for ET-AAS [19,20]. This approach opens another important application area for KR tubing as an excellent filterless collector for inorganic precipitations [7].

To the best of our knowledge, the KR technique was first hyphenated with the portable W-coil ET-AAS instrument for on-line preconcentration and determination in this work. The developed combination expanded the applications of KR technique and the new portable AAS instrument, while improved the analytical performance of this instrument considerably. As one of the applications of the method established

* Corresponding author.

E-mail address: wenxdong129@163.com (X. Wen).

by the present work, trace cadmium was determined as a prevalent toxic element. Peng Wu et al. reviewed some recent works corresponding to the detection of cadmium in biological samples [21,22]. The analytical method established in this work has certain advantages in terms of sensitivity. The characteristics and performance parameters of the established on-line method KR–W-coil ET–AAS were described below.

2. Experimental

2.1. Apparatus

Portable tungsten coil electrothermal atomic absorption spectrometer model WFX-910 (Beijing Rayleigh Analytical Instrument Co., Ltd, Beijing, China) was introduced for determination and investigation as a new commercial instrument, which consisted of three main parts arranged horizontally, including a hollow cathode lamp (HCL), a W-coil atomizer enclosed in a quartz cell and a spectrometer-charge coupled device (CCD). This instrument is developed to accomplish field analysis and the portable design embodies in the following aspects. Overall dimension (length × width × height): 610 mm × 230 mm × 335 mm, weight: 18 kg. A customized box is equipped to be carried out conveniently. A rechargeable lithium battery is equipped in the portable instrument to provide enough power for field analysis without commercial power supply. A specially customized and portable cylinder of argon containing 20% H₂ is equipped with this spectrometer for field analysis.

A lab-assembled flow injection system was used to accomplish on-line KR pre-concentration and sample elution/introduction, which was consisted of two peristaltic pumps (Model: HL-2D, Shanghai Huxi Instrument Factory, Shanghai, China) and a standard rotary injection valve (eight ports on the rotor and eight ports on the stator). The knotted reactor was a 0.30 mm i.d. PTFE tubing (ca. 150 cm) by tying interlaced 30–35 knots.

The pH values were measured by a pH-meter Model PHS-25 (Shanghai Hongyi Instrument Co., Ltd, Shanghai, China).

A laboratory pure water system Model DZG-303A (Chengdu Tangshi Kangning Science and Technology Development Co., Ltd, Chengdu, China) was used to prepare ultra pure water.

A model ELAN DRC-e ICP-MS instrument (PerkinElmerSCIEX, USA) was used to detect the real water samples to compare with the developed method. The major instrumental parameters include: ICP RF power, 1150 W; plasma gas flow, 15 L/min; auxiliary gas flow, 1.20 L/min; nebulizer gas flow, 0.93 L/min; lens voltage, 6.50 V; dual detector mode, pulse counting; and isotope monitored, ¹¹¹Cd.

2.2. Reagents

Cadmium standard solution (1000 mg/L) was purchased from National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials (NCATN, Beijing, China). Working standard solution was obtained daily by stepwise dilution from standard stock solution in ultra pure water. Ammonia and nitric acid (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) were diluted to optimal concentrations (0.8 mol/L and 1 mol/L) by ultra pure water daily. Other chemical reagents were all of analytical grade.

2.3. Knotted reactor (KR) on-line pre-concentration procedure

The experimental procedure of flow injection on-line precipitation pre-concentration included two steps for adsorption and elution respectively, which was illustrated in Fig. 1. In the first step, both the pumps were activated and the injection valve was set in the fill position. The sample or standard solution and ammonia solution were mixed in a three way valve just before entering the KR. The resultant precipitate of cadmium hydroxide was adsorbed by the KR. In the next step, the collected precipitate should be eluted and transferred to instrumental analysis. Pump 2 was paused and pump 1 was still activated at the flow rate of 60 rpm, whereas the valve was turned to the inject position to pump the elution of 1 mol/L HNO₃ through the KR. The precipitate adsorbed on the inner wall of the KR was eluted and then introduced into W-coil ET-AAS at the flow rate of 10 rpm for 4 s to ensure the volume of sample was 20 μL. After determination, the KR should be rinsed with some dilute nitric acid and ultra pure water to make ready for next measurement.

2.4. Operation procedure of KR–W-coil ET–AAS

The KR pre-concentration system was firstly combined with W-coil ET-AAS in this work. Via the flow injection analysis system, the precipitation pre-concentration process was accomplished and then elution and sampling were respectively accomplished as mentioned above and illustrated in Fig. 1. The adsorbed cadmium hydroxide was efficiently eluted by pump 1 at a flow rate of 60 rpm. When the eluted solution approached the W-coil atomizer through the sampling tube, the flow rate of pump 1 was set at 10 rpm to introduce the resultant sample onto the atomizer. A total sampling time was 4 s, which was counted from the sample solution dripping out from the end of the sampling tube. Thus the sampling volume was controlled at 20 μL. After determination, the KR was rinsed to make ready for next measurement.

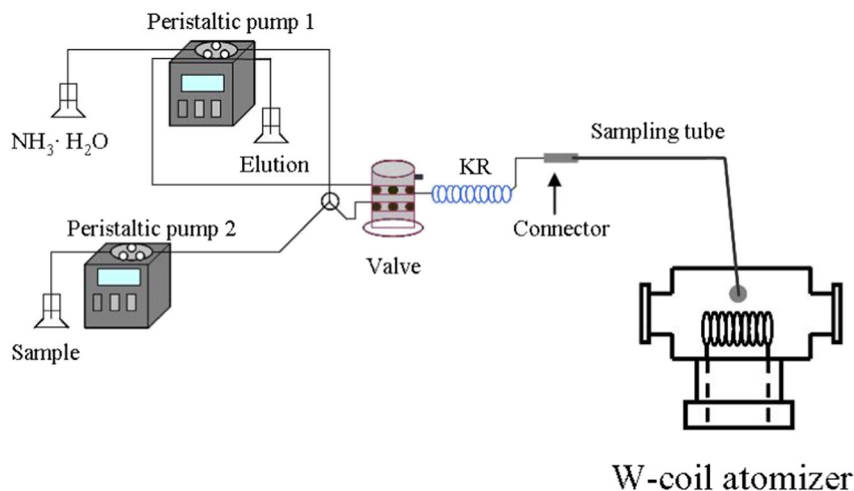


Fig. 1. Instrumental arrangement of the KR–W-coil ET–AAS system.

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