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Application of portable tungsten coil electrothermal atomic absorption spectrometer for the determination of trace cobalt after ultrasound-assisted rapidly synergistic cloud point extraction

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ABSTRACT

In this work, ultrasound-assisted rapidly synergistic cloud point extraction (UARS-CPE) was firstly established and combined with a portable tungsten coil electrothermal atomic absorption spectrometer (W-coil ET-AAS) for preconcentration and determination of trace cobalt. As a new instrument, the application of the portable W-coil spectrometer was expanded through the coupling with UARS-CPE, which was accomplished at room temperature rapidly by the effect of ultrasound and revulsant/synergic reagent. The instrumental conditions and influencing factors relevant to UARS-CPE efficiency, such as concentrations of surfactant and chelating agent, volume of revulsant/synergic reagent, pH, and ultrasound extraction time were studied systematically. The coupling improved the analytical performance of the portable spectrometer considerably. Under the optimal conditions, the limit of detection (LOD) for cobalt was 0.30 μg/L, with sensitivity enhancement factor (EF) of 52. In order to validate its application in field analysis, some environmental water samples were field-analyzed with satisfactory results. The established method could be expanded to pre-concentrate and detect many other trace metal ions.

1. Introduction

As an important instrumental method of electrothermal atomic absorption spectrometry (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 [1,2] and experienced a longer period of development and accumulation, including the applications of modifiers [3,4,5], combination with some separation and preconcentration techniques [6,7,8,9], electrothermal vaporization (ETV) as a sample preconcentration and/or introduction technique for atomic fluorescence spectrometry (AFS) [10,11,12,13], inductively coupled plasma atomic emission spectrometry (ICP-AES) [14,15] and inductively coupled plasma mass spectrometry (ICP-MS) [16], etc. After years of academic accumulation of W-coil ET-AAS, a portable tungsten coil electrothermal atomic absorption spectrometer was developed and firstly realized the commercialization of such instrument under the cooperation of Hou's group of Sichuan University and Beijing Rayleigh Analytical Instrument Co., Ltd [17]. The portable W-coil spectrometer has the potential to provide field analysis and has considerable application prospect. In recent years, Wen et al. has carried out some research work on the application of W-coil ET-AAS and the portable spectrometer, including combination with some simple and rapid separation and preconcentration techniques [9,18,19], field analysis of some metal ions such as nickel [20], bismuth [21] and chromium [2]. In order to further improve the analytical performance of the instrument and extend its application, there are many research work worth continuing to carry out.

In this work, ultrasound-assisted rapidly synergistic cloud point extraction (UARS-CPE) was firstly established and combined with a portable W-coil spectrometer for preconcentration and determination of trace cobalt, including the field analysis of environmental water samples. UARS-CPE was a new type of fast CPE technology, which was based on the rapidly synergistic cloud point extraction (RS-CPE) technology reported in the previous research work of Wen's group [22,23,20]. Through the application of ultrasound to the extraction process, the extraction efficiency was further improved and the reproducibility and stability of extraction process could be better controlled compared to the completely manual shaking extraction process in RS-CPE. UARS-CPE followed the application of revulsant/synergic reagent in RS-CPE to realize the rapid cloud point extraction at room temperature without any heating treatment [22]. This was its biggest advantage over the ultrasound-assisted cloud point extraction (UA-CPE) technology, which was more time-consuming and tedious for the dependence on water bath heating [24,25]. This was the first report of UARS-CPE technology and its application to the portable atomic absorption spectrometer for the enrichment and determination of cobalt.

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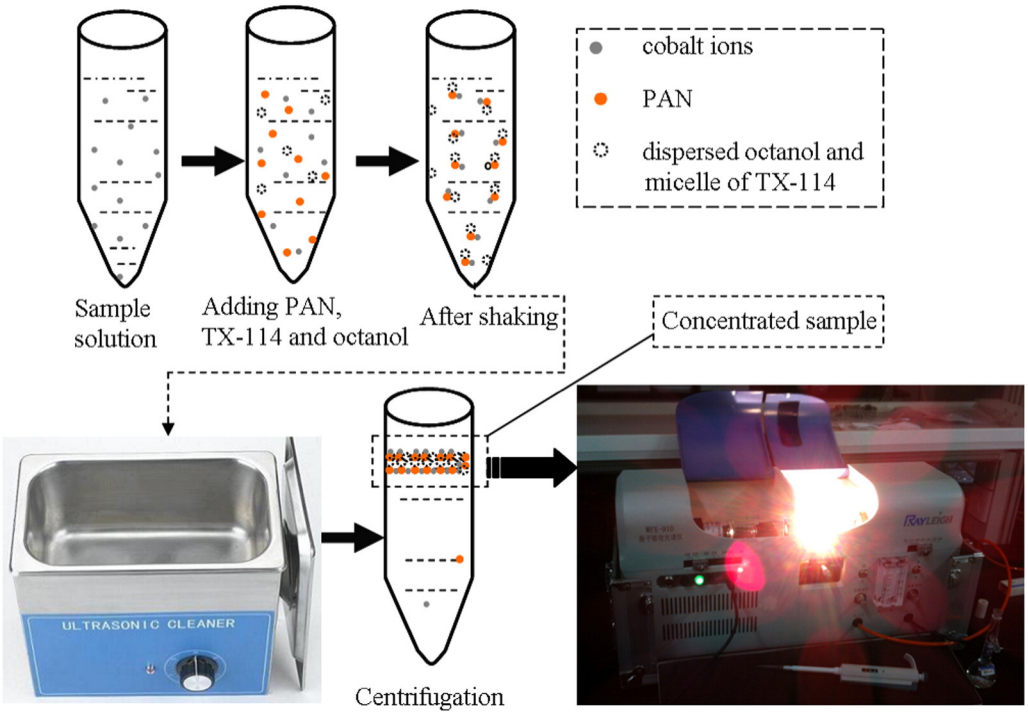

Fig. 1. Schematic diagram of the UARS-CPE-W-coil ET-AAS procedure.

Cobalt is known to be essential at trace levels to man, animals and plants for metabolic processes. Many instrumental methods have been reported for the determination of cobalt including inductively coupled plasma-optical emission spectrometry (ICP-OES) [26], atomic fluorescence spectrometry (AFS) [27], capillary electrophoresis (CE) [28], etc. In the previous research work of W-coil ET-AAS, the investigation of cobalt analysis was relatively seldom carried out for the poor sensitivity of direct determination by this method. Therefore, the combination of UARS-CPE enrichment with this portable spectrometer could significantly improve its analytical performance of cobalt analysis, which had a good research value and practicality. This was the reason why the cobalt element was chosen as the target analyte in this work. The developed combination of UARS-CPE with portable W-coil ET-AAS has the advantages of simplicity, rapidity and high sensitivity, which could move traditional AAS out of lab to realize field analysis. The characteristics and performance parameters of the established combination 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 equipped inside the portable instrument can provide enough power for at least 200 times of determination in the field without commercial power supply. A specially customized and portable cylinder of argon containing 20% H_2 is equipped with this spectrometer for field analysis.

An ultrasonic cleaner with temperature control Model SB5200DT (Ningbo Xinzhi Biotechnology Co., Ltd, Ningbo, China) was used for UARS-CPE.

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 ultrapure water.

An ICP-AES instrument model Optima 8000 (PerkinElmer) was used to the determination of real water samples to validate the accuracy of the established method.

2.2. Reagents

Cobalt 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 ultrapure water. Non-ionic surfactant Triton X-114 (TX-114) (Sigma-Aldrich) and octanol (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) were used as extractant and cloud point revulsant/synergic reagent, respectively. 1-(2-Pyridylazo)-2-naphthol (PAN, Sinopharm Chemical

Table 1
Heating program of W-coil for cobalt determination (sampling amount: 10 μL).

Steps		Desolvation	Pyrolysis	Cooling	Atomization	Cleaning
Current (A)-Temperature (°C) /Duration (s)	Without UARS-CPE	2.9–600/30	2.9–600/15	0/4	9.0–2300/4	9.0–2300/2
	After UARS-CPE	2.9–600/20	2.9–600/10	0/4	9.0–2300/4	9.0–2300/2