



Integration of hydride generation and photochemical vapor generation for multi-element analysis of traditional Chinese medicine by ICP-OES



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ABSTRACT

A dual-mode chemical vapor generation (DM-CVG) sample introduction system that integrates hydride generation (HG) and photochemical vapor generation (PVG) was coupled to a commercial inductively coupled plasma optical emission spectrometer (ICP-OES) for simultaneous multi-element analysis of traditional Chinese medicine samples for trace hydride-forming elements and non-hydride-forming elements. Three hydride-forming elements (Cd, As, and Ge) and two non-hydride-forming but photochemical vapor-generating elements (Co and Ni) were selected as target elements to validate the proposed method. Optimum experimental conditions for DM-CVG-ICP-OES were carefully investigated, and satisfactory limits of detection (LODs), 0.2–1.4 $\mu\text{g L}^{-1}$, and relative standard deviations (RSDs), 1.3%–4.6%, were obtained for these target elements. A certified reference celery sample (GBW10048) was used to investigate the feasibility of the proposed method for real analysis. Strong suppression on the PVG efficiency occurred either when little nitric acid residue or some unknown coexisting materials remained in the sample solutions. Complete acid-evaporation/catching and moderate dilution of the digests could effectively alleviate this negative effect. Four different acid-evaporation/catching methods were performed and compared. Catching acid by adding an appropriate volume of formic acid gave the best signal response and the results agreed well with the certified values. Satisfactory analytical results were also obtained in real sample analysis of TCM *Curcuma wenyujin* Y.H. Chen et C. Ling.

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1. Introduction

Sample introduction is very important in analytical atomic spectrometry. Pneumatic nebulization (PN) is the most commonly and widely used sampling technique, but often suffers from high matrix interference and low transfer efficiency of analytes. Chemical vapor generation (CVG) has been widely employed as a highly effective sample introduction method for years, and has many outstanding advantages over conventional PN, such as good sensitivity and selectivity, unmatched high analyte-transport efficiency, and effective matrix separation and thus low matrix and spectral interferences. Therefore, CVG provides a powerful sample introduction means that enjoys increasing interest for trace and ultratrace inorganic elemental analysis. It has been combined with almost every kind of atomic spectrometry, and is a popular component of hyphenated techniques used for speciation analysis [1,2].

Hydride generation (HG) is the earliest and the most widely used CVG method, with traditional HG elements which include As, Bi, Ge, Pb, Se, Sb, Sn, Te and Zn, and elemental vapor-forming elements Hg and Cd, via the reaction with tetrahydroborate (THB) for the production of analyte-containing hydride gases phase or elemental vapor [3–6]. Photochemical vapor generation (PVG) is a relatively new and attractive CVG sample introduction technique first introduced in 2003 by Sturgeon et al. [7], and has been demonstrated to be a powerful alternative to conventional CVG for many elements [8–10]. A PVG process generally occurs in the presence of low-molecular-weight (LMW) organic compounds, and different kinds of volatile species, including hydrides, elemental, carbonylated and alkylated analytes, which are generated from their relative non-volatile precursors simply by ultraviolet (UV) irradiation. In comparison with conventional CVG, PVG has many additional inherent advantages, including no inorganic acid and unstable chemicals (e.g., THB), low H_2 generation and little interference from transition metals, and so on [11]. Up to now, PVG has been coupled to almost all major elemental analysis methods including atomic absorption spectrometry (AAS), atomic fluorescence spectrometry (AFS), inductively coupled plasma-optical emission spectrometry and mass spectrometry (ICP-OES/MS) for sensitive determination of a wide range of elements including not only conventional hydride-forming

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elements (As, Bi, Sb, Se, Te), Hg and transition metals (Ni, Co, Fe) but also non-metals (I) [12–22].

The demand for simultaneous and fast multi-element analysis stimulates the exploration and development of integration of different sample introduction techniques. Attention has been paid to the combination of solution and vapor introduction, especially the integration of PN and HG, which are the two most commonly used sample introduction techniques, and made possible real simultaneous determination of hydride-forming and non-hydride-forming elements by coupling with multi-element analysis technique such as plasma-based analytical techniques [5,23–25]. Combination of different types of CVG is rare, although a novel dual-mode chemical vapor generation (DM-CVG) system integrating HG and PVG has recently been designed by our research group, and simultaneous multi-element analysis of hydride-forming (As, Cd) and non-hydride-forming (Fe, Ni) elements by AFS has been well accomplished with high sensitivity and low matrix and spectral interferences [26]. However, AFS is capable of simultaneously determining only a very small number of elements (usually only two elements) in general, and this seriously restricts its application in simultaneous multi-element analysis. ICP-OES is a powerful multi-element analysis tool that is widely used for analysis of various samples. Therefore, the aim of this work is to hyphenate the DM-CVG to ICP-OES and tentatively investigate its application for simultaneous multi-element analysis of complex traditional Chinese medicine (TCM) samples for the abovementioned trace elements.

2. Experimental

2.1. Reagents

All reagents were of analytical-reagent grade unless mentioned otherwise. All solutions were prepared using high-purity deionized water (DIW, 18 M Ω cm) produced from a water purification system (Chengdu Ultrapure Technology Co., China). The standard solutions containing As(III), Cd(II), Ge(IV), Co(II) and Ni(II) were prepared daily by dilution of their 1000 mg L⁻¹ stock solutions from the National Standard Material Research Center of China (Beijing, China). Formic acid (88%), ammonia (25%–28%), sodium borohydride (NaBH₄) and sodium hydroxide (NaOH) were purchased from Kelong Chemical Reagents Co. (Chengdu, China). Ultrapure grade HNO₃ (71%), H₂O₂ (30%), H₂SO₄ (98%) and HClO₄ (72%) were purchased from Suzhou Crystal Clear Chemical Co. (Suzhou, China). High purity Ar (99.99%) was obtained from Qiaoyuan Gas Co. (Chengdu, China).

A certified reference material (CRM) of celery sample (GBW10048) from the National Standard Material Research Center of China (Beijing, China) was used to validate the accuracy of the proposed method. Samples of *Curcuma wenyujin* Y.H. Chen et C. Ling were purchased from a local medical market in Wenzhou, China, and stored at room temperature until analysis.

2.2. Instrumentation

A schematic diagram of the DM-CVG system hyphenated to a commercial inductively coupled plasma optical emission spectrometer (ICP-OES) (ARCOS FHS12, SPECTRO Analytical Instruments GmbH, Germany) is shown in Fig. 1. The laboratory-built DM-CVG system mainly consisted of a UV photochemical reactor and a hydride generator was set up with some modifications from our previous design [26]: (1) a flow injection analysis processor (FIA-3110, Beijing Titan Instruments Co., Ltd., Beijing, China) with two independently controlled three-channel peristaltic pumps was used to separately control the HG and PVG sampling programs; (2) in the photochemical reactor, standard or sample solution, formic acid and ammonia were simultaneously pumped into a four-way Y type polytetrafluoroethylene (PTFE) pipe (A in Fig. 1) at the same rate, mixed and then flowed into the coiled quartz tube for UV irradiation; and (3) in the hydride generator, the

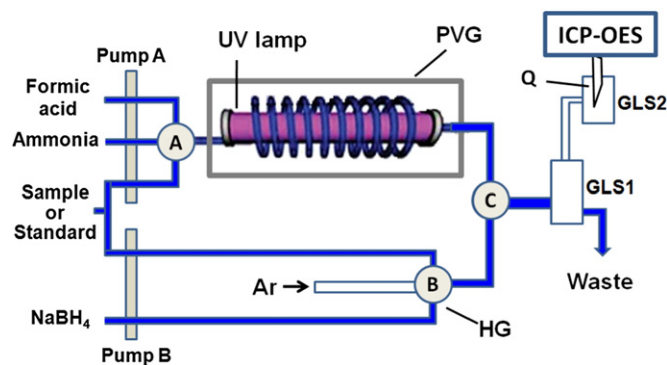


Fig. 1. Schematic diagram of the experimental setup. PVG, photochemical vapor generator; HG, hydride generator; GLS, gas-liquid separator.

nebulizer argon flow of the ICP-OES instrument was directly used here as the carrier gas and joined to one inlet of another four-way Y type PTFE pipe (B in Fig. 1), and carried the HG reactants, which had been simultaneously pumped in through another two inlets of this Y type pipe, to meet with the outflow PVG reactants at a quartz Y type tube (C in Fig. 1). Both the HG reactants and the PVG reactants were flushed into a gas-liquid separating system consisting of two gas-liquid separators (GLS) in tandem for complete gas-liquid separation. Volatile species of analytes were separated from the first GLS wherein the waste was pumped off by a four-channel peristaltic pump (BT100-02, Baoding Qili Precision Pump Co., Baoding, China). Then, the volatile species were transported to the second GLS to ensure that no condensed liquid droplets were transported to the ICP. Finally, the volatile species of target elements were further flushed into the ICP-OES for measurement. This modified setup could be operated more conveniently for either single mode or dual mode, and the procedure could be performed accurately and automatically. All the tube lines were made of PTFE except where otherwise noted. A microwave-assisted digestion system (Master 40, Shanghai Sineo Microwave Chemical Technology Co., China) was used to digest all the test samples and the CRM.

2.3. Procedure

The whole process included five steps. In step 1, the sample or standard solution containing 3% (v/v) formic acid was pumped to mix with 90% (v/v) formic acid and 30% (v/v) ammonia by pump A at a flow rate of 2 mL min⁻¹ for 60 s to fill the coiled quartz tube. In step 2, the photochemical reactor was continuously irradiated with UV for 180 s. In step 3, the sample or standard solution was pumped to merge with 3% (m/v) NaBH₄ solution (in 0.2% NaOH, m/v) at the four-way Y type PTFE pipe (B in Fig. 1) by pump B at a flow rate of about 1.5 mL min⁻¹ for 30 s. UV irradiation onto the photochemical reactor continued. In step 4, pump B worked continuously for 60 s as in step 3; the channel of sample or standard solution used in the PVG was shifted to 3% (v/v) formic acid solution and pump A was switched on for 60 s to push the PVG reactants to flow out of the photochemical reactor to combine with the HG reactants at the quartz Y type tube (C in Fig. 1), during which time the photochemical reactor was washed. The mixture was pushed into GLS1 with Ar carrier introduced in the HG reactor inlet. The volatile species of analytes were separated from the liquid phase and transported to GLS2 for further separation. Finally, the analyte element-containing vapor reached the ICP torch for measurement of atomic emission spectra. In step 5, pump A was switched off; shifting the channel of sample or standard solution of HG to 3% (v/v) formic acid solution and pump B worked continuously for 30 s to wash the hydride generator to get ready for the next measurement. Atomic emission intensity of analytical lines were recorded for quantification, and the optimized instrumental parameters of the ICP-OES were summarized in Table 1.

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