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# Multielemental analysis of botanical samples by ICP-OES and ICP-MS with focused infrared lightwave ashing for sample preparation

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ABSTRACT

A novel dry ashing method based on focused infrared lightwave ashing (FILA) was proposed for preparation of botanical samples enriched organic matter. High performance infrared quartz tubes were used in dry ashing devices as heating elements for the first time. A layer of gold was plated on the surface of the heating tube in order to reflect and focus infrared light and enhance the effect of heating. Meanwhile, the self-designed flow-through quartz ashing tube ensures oxygen to penetrate the entire sample layer, thus increasing the efficiency of sample ashing. In addition, the heating chamber has a very small space and the size of the proposed device is only one-fifth of a traditional muffle furnace's size. The sample can be quickly heated from room temperature to 900 °C just in 1 min. For most kinds of samples, ashing can be completed within half an hour. After the ash was dissolved with diluted nitric acid, multielements were determined in the solution by inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS), respectively. Five certified reference materials of plants were used as examples to demonstrate the performance of the developed device. The agreement to the certified values was between 80 and 114% for Be, Na, Mg, P, K, Ca, Mn, Co, Ni, Cu, Zn, Rb, Sr, Mo, Cs, Ba, Pb, Bi, Th, U and rare earth elements. Moreover, the FILA system was also applied to the analysis of five real botanical samples. The results showed no significant difference from the ones by microwave digestion (MW).



## 1. Introduction

In the past decades, inductively coupled plasma optical emission spectroscopy and inductively coupled plasma mass spectroscopy have become more and more powerful tools in the element analysis for botanical samples including vegetables, leaves, plant food et al. [1–5]. They have been equipped in more and more laboratories as routine instruments. However, either ICP-OES or ICP-MS usually requires a pre-conversion of solid samples into liquid solutions, because both of the instruments are usually equipped with liquid sample introduction systems [6].

Compared with short time of detection by ICP-OES and ICP-MS, time of sample preparation shows a very long process. Sample preparation has become a bottleneck for the whole process of analysis [7]. Classical or traditional methods of sample preparation, by which solid samples will be transferred from solid state to liquid state, are often referred to as wet digestion or dry ashing [8]. Compared to wet digestion, dry ashing has many advantages for preparation of samples enriched organic matter. Especially, the sample matrix is substantially simplified and the resulting ash is completely free of organic matter. Dry ashing is environmentally benign by avoiding the use of large amounts of strong acids in wet digestion. The acidity of the final solution can be controlled efficiently [9–11].

Due to the many advantages listed above, dry ashing as a classical method has been applied to pretreatment of organic-matrix samples for determination of elements by atomic spectrometry [12]. In the past decades, different methods of dry ashing have been developed from electric heating to microwave heating, from open systems to closed systems. These methods use different types of heating ways including electricity, microwave, plasma, ultraviolet light and laser [11], while the instrument most widely used in dry ashing of samples is electrothermal muffle furnace, in which high-temperature alloys and silicon compounds are usually used as heating conductors. The main disadvantages of the conventional electronic heating furnaces are the long duration of the temperature rise and a large size. Recently, microwave muffle furnaces have been applied for sample ashing, calcination, fusion, ash tests, ignition loss, etc. [8,12–15]. There is no direct effect of radiation on many organic-matrix samples due to their non-absorbing microwave. The samples can be indirectly heated by some microwave-

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absorbing materials, such as silicon carbide and zirconia [16]. However, the high cost of instrument is a drawback for the wide use of microwave ashing. Laser as a heat source has also been used to decompose organic matter as early as 20 years ago [17]. At present, it is mainly used in localized and surface analysis as a direct introduction technique coupled with atomic spectrometry. Plasma [18,19] and ultraviolet [20,21] were also used as an ashing way. Under the action of plasma and ultraviolet light, oxygen will generate a reactive monatomic oxygen. As a reactive species, a monatomic oxygen combines with the organic matter to form ash. Because the oxidation reaction of organic molecules either by plasma or by ultraviolet light is very slow, the ashing generally occurs at temperatures below 150 °C. The two methods are mainly used in the ashing of coal and biological samples. But they are limited for routine use [7].

However, conventional dry ashing methods have lower efficiency compared with wet digestion, because they are time-consuming, energy-consuming, high cost, low throughput, or large size of the whole device. In the past few decades, infrared radiation has been successfully applied in wet digestion [22–25], but has not been reported in the application of dry ashing sample preparation. With regard to the drawbacks, in this work, we developed a small-size, simple, rapid and novel ashing system using focused infrared lightwave tubes as heating elements. Tungsten wires were wrapped in a pole-like quartz tube, and one side of inside wall of the quartz tube was covered with a layer of gold. When electric currents flowed through tungsten wires, the infrared light produced. A part of the infrared light with high intensity directly irradiated the sample, and another part was reflected and focused on the sample. Thus the resulted high temperature made the sample ashed rapidly. The size of the proposed device is only one fifth of a traditional muffle furnace. Twelve samples were able to be ashed simultaneously. The self-designed flow-through ashing tube allows auxiliary gas to penetrate through the entire sample layer. In the carbonization stage, argon was used as the auxiliary gas, thus the carbonization of sample could be rapidly carried out at a very high temperature without sample combustion. In the ashing stage, oxygen was used as the oxidizing agent instead of air. Both the outside of the sample and the inside of the sample were fully exposed to oxygen, and accelerated the ashing of sample. Therefore most samples were completely ashed within 30 min.

The performance of the proposed system was evaluated by determining macro, micro and trace elements, including Be, Na, Mg, P, K, Ca, Mn, Co, Ni, Cu, Zn, Rb, Sr, Mo, Cs, Ba, Pb, Bi, Th, U and rare earth elements (REEs), in five botanical certified reference materials by ICP OES and ICP-MS. Moreover, some real samples were also used as the actual analytical application of the proposed method.

## 2. Experimental

### 2.1. Instrumentation

The details of the proposed rapid FILA are shown in Fig. 1. The system (27 cm length, 28 cm width, and 44 cm height) mainly comprises a lightwave heating furnace, a temperature controller, a cooling fan, and a gas module. The lightwave heating furnace (Fig. 2) includes a heating chamber and three infrared quartz tubes (380 mm length, 30 mm width, and 8 mm height). The heating chamber is made of mullite refractory and has 12 orifices (25 mm diameter) on its upper surface to insert the ashing tubes. There are two inlets of cooling gas in the front end of the chamber. Three infrared quartz tubes provide uniform heating with 12 orifices for quartz ashing tubes (18 mm internal diameter, 20 mm outside diameter and 215 mm height). One is transparent, and the others are semi-gold-plated. The temperature is achieved by means of an armoured K-type thermocouple. The equipment is operated via an industrial grade control terminal where the heating program is created by setting up target temperatures and hold times. The system

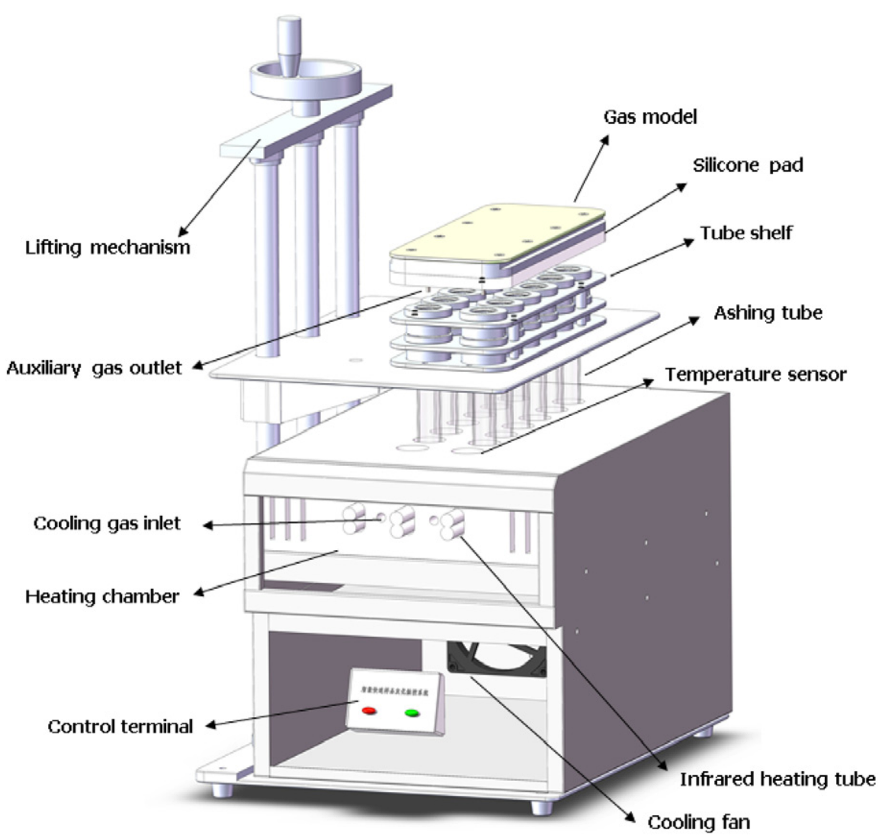


**Fig. 1.** Overview photograph of the FILA system.