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Research and Development of ME–101 Multifunction Micro extraction Instrument

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Summary

Multifunctional liquid phase micro extraction apparatus adopts gas purge micro–syringe extraction technique, high–temperature heating and semiconductor refrigeration techniques. It is a gas chromatography (GC) sample pretreatment apparatus that integrates extraction, purification, concentration and predissociation. The GC–MS total ion chromatograms(TICs)of volatile oil extracted from the rhizome of atractylodes japonica shows that volatile components is extracted by the micro extraction apparatus outnumber the ones that extracted by the steam distillation. Furthermore, the content of major constituents extracted by the apparatus is higher. The apparatus has characteristics of less reagent dosage, short extraction time, high enrichment efficiency, simple structure, convenient operating and low operating cost. Therefore it can be applied to the GC sample pretreatment of solid or liquid sample in the areas of medicine, agriculture, food, quality control and so on.

*Keywords***:** liquid phase micro extraction; gas chromatography; sample pretreatment

1. Introduction

With the development of modern analytical apparatus, the requirement of automation and intelligence for sample pretreatment method is put forward. The development of analytical chemistry is limited by a fact that modern sample pretreatment methods and analytical apparatus can't be a good match. Therefore, to develop a sample pretreatment method which can match with modern analytical apparatus is the trend of analytical chemistry. Liquid phase micro extraction (LPME) technology device is very simple 0. A variety of solvents can be applied to it, and only an ordinary micro–syringe or porous hollow fiber is needed in the device. However, LPME has some problems, such as the low recovery of high–boiling target, poor quantification accuracy, and inconvenient operation. Gas purge micro–syringe extraction (GP–MSE) technique is equal to that the inert gas is continuously fed to the apparatus of conventional HS–LPME to make enrichment system become an open system. It can realize higher enrichment efficiency than traditional HS–LPME device by using high temperature heating and refrigeration device [3]. A set of gas phase chromatography sample pretreatment apparatus which integrates functions of extraction, purification and concentration, is researched and developed in this paper. It is based on the GP–MSE, high–temperature heating and semiconductor refrigeration techniques[4]. The apparatus has the characteristics of fast extracting speed, high efficiency, simple operation and high automation.

2. Structure of Multifunctional Liquid Phase micro extraction Apparatus

Multifunction liquid phase micro extraction apparatus (referred to as micro extraction apparatus) is mainly composed of semiconductor condenser, mini heater, flow controller and automatic control circuit. Fig. 1 illustrates the schematic diagram of micro extraction apparatus. The right part of Fig. 1 is semiconductor condenser. The cold end of semiconductor condenser is close to an aluminum box which is used to speed up the heat transfer on both ends of the condenser when the condenser works. The hot end of the refrigeration piece is connected to a heat sink and fan. The heat transmitted from the cold end to the hot end is dissipated through it. The micro–syringe is placed in 8mm diameter C–shaped slot inside the aluminium box. When the condenser works, the cold end of refrigeration piece plays cooling effect for extraction solvent in the micro–syringe by absorbing heat. The adjustment range of condensing temperature is from room temperature to –10°C.

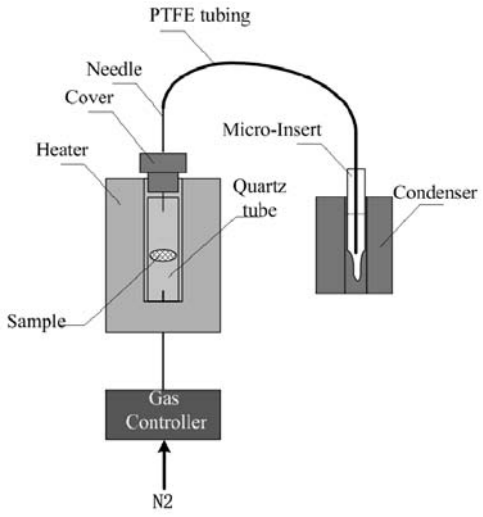

Figure 1. Schematic of the semiconductor condenser and heater in micro extraction Apparatus.

The left part of Fig. 1 is micro–heater and the heating part of it is alumina ceramics heater band. A quartz sample tube is placed in a cylindrical sample pool inside the micro–heater.

Samples are placed above an asbestos layer of sample tube, which is heated during the extraction process. The heater is sealed by the PTFE cap, besides; the gas pipe is connected into the micro–heater from the bottom of the heater. The inert gas can be passed into the micro–heater by the gas pipe. The range of sample heating temperature is from 50°C to 350°C, and the adjustment range of gas flow is from 0 SCCM to 5 SCCM.

The sample tube in the semiconductor and micro–heater are connected by injection needle and polytetrafluoroethylene hose.

The hose is connected to the micro peristaltic pump through the syringe connector.

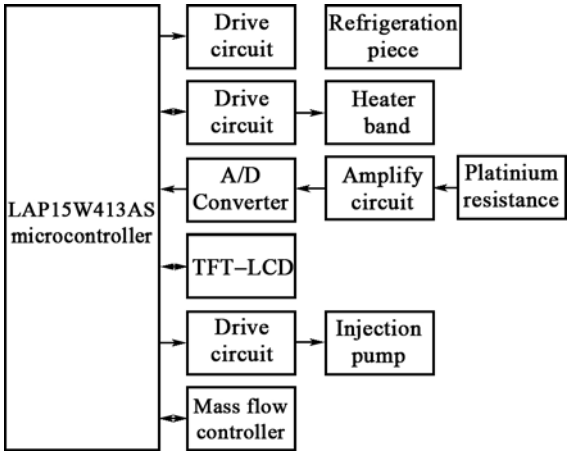

Figure 2. Electrical schematic diagram of micro extraction Apparatus

3. Constitution and Working Principle of micro extraction Apparatus

3.1 Constitution of micro extraction Apparatus

Control system of micro extraction Apparatus is mainly composed of the microcontroller, semiconductor condenser, heater, flow controller, switch power supply, A/D converter, temperature sensor, and TFT–LCD. Switch power supply provides +5V, ±15V, +40V DC voltage. Fig. 2 illustrates the electrical schematic diagram of the total system.

3.2 Working Principle of micro extraction Apparatus

The refrigeration temperature, heating temperature, flow rate of inert gas and timing time can be set by a LCD touch keyboard. After starting the microextraction apparatus, the DS18B20 temperature sensor starts to measure the current temperature of semiconductor condenser, and it converts the temperature into a digital electric signal with 11–bit reading each. After that, the digital signal is transferred over the 1–wire interface of DS18B20 to microcontroller by issuing Read Scratchpad [BEh] commands. Then, the IAP15W413AS[5] microcontroller calculates error amounts by comparing the temperature measured by the DS18B20 sensor with the one set by touch keyboard, and PID algorithm is used to Fig. out controlled variables. At the same time, the platinum resistance unbalanced bridge measures real–time heating temperature of micro–heater and it outputs a bridge voltage corresponding with the current temperature .The output is connected to LM124 operational amplifier to be amplified, so that it can be reached the required input voltage range of A/D converter. The analog temperature signal is converted to the digital signal by A/D converter, and then the digital signal is fed to the microcontroller to be processed. In the system, the analog–to–digital conversion chip ADS1286 [6] with 12–bit resolution is used to convert the analog heating temperature signals to the digital signals.

The S49–32B/MT mass flow controller[7] (MFC) is adopted in GP–HS–LPME system to measure and control the gas flow of inert gas which is introduced into the sample tube through the gas pipe. The gas flow analog signal detected by MFC is converted to 8–bit digital signal by the A/D conversion function of the microcontroller, besides the microcontroller controls solenoid valve through the drive circuit to make inert gas reach the set gas flow rate.

In conclusion, under the control of the microcontroller, the whole system can automatically achieve the requirements for refrigerating temperature, heating temperature and inert gas flow.

4. Experiment

4.1 Method of Sample Processing by microextraction Apparatus and Steam distillation

5mg crushed sample of atractylodes japonica (rhizome) is accurately weighed, then it is placed into the sample slot. After the injection pad is plugged, 250uL gastight syringe is pieced into the injection pad through the condenser of the micro extraction apparatus, and the pinpoint may just pass through the end of the injection pad. After the pinpoint is pulled out, 20μL extraction solvent n–hexane is added and nitrogen is purged. After extraction, constant volume is reached to 100μL with n–hexane, then the 2μL solvent is taken for GC–MS analysis. The extraction conditions are as follows. The gas flow rate of nitrogen is 2mL/min, the extraction temperature is 250°C, the condensing temperature is –4°C, the extraction time is 2min and the volume of the extraction solvent is 20μL. 5 mg crushed plant samples are weighed, and it is placed into the 1 000mL water vapor generator. Half and one third of the volume of distilled water is respectively added into the distillation device and water vapor generator. Then, open the condensed water and start heating. Timing starts when the first drop of distillation dropped into the extraction solvent in the receiving tube, and the extraction time is 6 hours. After the extracted sample is dried by the anhydrous Na_2SO_4, its volume is reached to 1mL. At last, 2μL solvent is taken for GC–MS analysis.

4.2 Analysis of Experimental Results

Fig. 3 illustrates the GC–MS TICs of the volatile oil that extracted from the rhizome of atractylodes japonica by steam distillation method and microextraction apparatus respectively. The Fig. shows that volatile components is extracted by the microextraction apparatus outnumber the ones that extracted by the steam distillation. For example, 2–Furaldehyde, 2–Furanmethano, 2–Methyl–5–formylfuran, 5–Hydroxymethyl–2–furaldehydecompounds and some other compound can only be detected in the extraction solvent of the microextraction apparatus. After comparing the components and their contents, the major components of the volatile oil extracted by the two methods is the same (Caryophyllene, Elixene, .beta.–Eudesmene) , but the content of major constituents extracted by the microextraction apparatus is more than the ones extracted by the steam distillation.

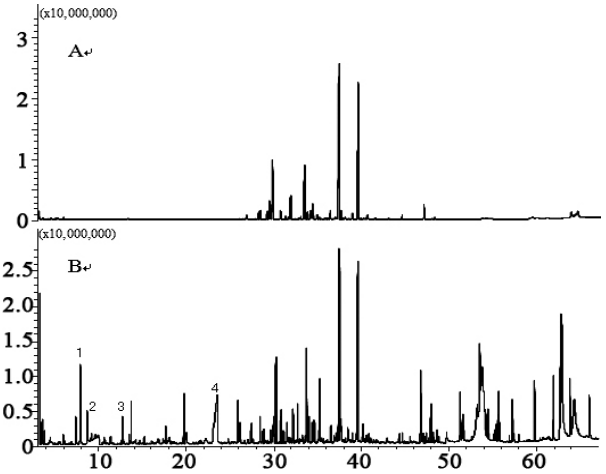

Figure 3. GC–MS TICs of the volatile oil that extracted from the rhizome of atractylodes japonica

5. Conclusion

Sample pretreatment plays an important role in the analytical chemistry process and it's development has a significant impact on the development of analytical chemistry. Micro extraction apparatus researched in this paper is a quick, easy and automated sample pretreatment. It can not only save time and effort, but also reduce the error caused by the different personnel operating and multiple transfers of sample. The micro extraction apparatus has already passed the new product identification of Jilin Province Industry and Information Technology Department Organization. It is widely used in medicine, food, tobacco, petroleum, environment and other industries, especially in the field of quality control, authenticity identification of plant raw materials and determination of trace.

Acknowledgment

This study was supported by a Grant from the National Natural Science Foundation of China (c21065014 and no. 21027009).

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