INTRODUCTION

Heavy metal pollution made the metals exceeding of Chinese traditional herbs in China are more and more. China has not yet established national standards in the detection of the amounts of heavy metals in Chinese traditional herbs. In this paper a hydride-atomic fluorescence spectrometry method was developed for the determination of trace arsenic in Panax notoginseng by atomic fluorescence spectrometry. The total arsenic content in Panax notoginseng grown in Yunnan were determined.

EXPERIMENTAL

AFS3100 double channel atomic fluorescence spectrometer (KCHG), Encoding arsenic hollow cathode lamp (Beijing Nonferrous Metal Research Institute), OPTIPLEX 380 computer system (Dell), DHG-9140A Blast Oven (Shanghai Yi Heng scientific instrument), CP224C electronic balance (Ohaus Instrument, USA), High-pressure digestion tank (100 mL). Operating conditions of AFS3100 is as given in Table-1.

Arsenic standard stock solution: 100 mg L⁻¹ (National Research Center of standard materials); nitric acid: 68 %; sulfuric acid: 98 %; hydrochloric acid: 99 %. The standard stock solution of arsenic was diluted step with step by 5 % hydrochloric acid solution to 1 mg L⁻¹ as the working solution, arsenic standard stock solution and working solution were placed in the refrigerator to save; pre-reducing agent: thiourea, ascorbic acid; reducing agent: 10 g L⁻¹ KBH₄-5 g L⁻¹ NaOH solution; carrier solution: 5 % hydrochloric acid. All above reagents were guaranteed reagent. Water were deionized water.

To determine the total arsenic in Panax notoginseng a hydride generation atomic fluorescence spectrometry method was developed by atomic fluorescence spectrometry (AFS-3100). The samples were pretreated by high pressure digest. The experimental conditions were studied and optimized. There was a good linear relationship between the fluorescence intensity and arsenic concentration in the range of 0-80 µg L⁻¹ with a correlation coefficient of 0.9995, while the detection limit was 0.036 µg L⁻¹, a relative standard deviation of 1.7 % arsenic in the samples was 0.391 µg g⁻¹ and a recovery range of 90.7-103.5 % was obtained.

Key Words: Arsenic, Panax notoginseng, Atomic fluorescence spectrometry.
Sample digesting conditions: In the experiment, different amount of reagents and digestion, digestion time and the influence of temperature on the digestion and determination were tested. The results showed that the fluorescent intensity of blank sample were very high with adding perchloric acid, sulfuric acid, it is not in favour of determination; mixed solution of nitric acid and hydrogen peroxide system is most suitable, when added 0.5 g sample, with the digesting time of 4-5 h, temperature of 150 °C; Digestion reagent nitric acid 9 mL, hydrogen peroxide 3 mL; get the most complete digestion, it is a colourless or slightly yellow solution.

Carry gas and Shielding gas flow rate: The carrier gas flow rate increased, would reduce the concentration of arsenic atoms, the intensity of fluorescence decreased; however, if the carrier gas flow rate were too small, it would affect the stability of argon flame. Experiment tested the fluorescent intensity of flow of 300-600 mL/min, when the carrier gas flow rate were 500 mL/min, the results were best. Shielding gas is to protect the internal reaction gas, too low shielding gas were difficult to stop the fluorescence quenching affected by the external gas, too much will dilute atomic vapor, reducing sensitivity. In this paper, the influence of the shielding gas to the intensity of fluorescence were tested at 800-1100 mL/min, the results show that it is best when the shielding gas at 1000 mL/min.

Selection of -HV of PMT and lamp current: When the negative high voltage of the photomultiplier tube were between 200-300 V, the standard series solution of arsenic were measured; the results showed that with the negative pressure increases, the instrument sensitivity increases, while the fluorescence values and standard curve linearity increased.

The acidity of medium: In this paper, the influence of the acidity of medium was selected at 5 %.

Effect of the acidity of the liquid carrier: The acidity of liquid carrier would affect the fluorescent intensity of blank samples and the efficiency of hydrogenation. Experiment the effect of the liquid carrier acidity with concentration of 1-10 % to the fluorescent intensity of sample with 0.4 µg L⁻¹ arsenic was tested, the results show that when the concentration of liquid carrier acidity were in the area of 3-6 %, the fluorescent intensity of arsenic were stable and higher, However, the greater the liquid carrier acidity grown, the bigger the value of the fluorescence of blank grown, the argon hydrogen flame grown also which made the stability of the fluorescence decreased; and the liquid carrier acidity should not too low so as not to affect the efficiency of hydride reaction. So the liquid carrier acidity was selected at 5 %.

Effect of the pre-reducing agent and time: The As(V) must be pre-reduced to As(III), to facilitate the hydrogenation reaction. The thiourea, thiourea-ascorbic acid = 1:1, ascorbic acid these three kinds of systems as pre-reduction agent were tested. The result shown the effect of thiourea with content of 10 g L⁻¹ was best. And the effect of pre-reducing time with 5-120 min to the fluorescent intensity of sample with 2 µg L⁻¹ arsenic was tested, the results show that when the pre-reducing time was in the area of 40-60 min, the effect was best.

Effect of the reducing agent: Reducing agent (KBH₄) mainly affects the efficiency of hydride generation and argon hydrogen flame. The higher concentration of reducing agent is conducive to the efficiency of hydrogenation, But the higher concentration of KBH₄ generated too much hydrogen, which made the argon-hydrogen flame increased and the fluorescence weaken. In the experiment the effect of the KBH₄ with concentration of 5-30 g L⁻¹ to the fluorescent intensity of sample with 2 µg L⁻¹ arsenic was tested, the results show that when the concentration of KBH₄ were 20 g L⁻¹, the fluorescent intensity of arsenic were stable and higher, So the concentration of KBH₄ was selected at 10 g L⁻¹.

Effects and elimination of interfering ions: The effects of 2000 times of K⁺, Na⁺, Ca²⁺, Mg²⁺; 2000 times of Fe³⁺, Zn²⁺, Cu²⁺, Co²⁺, Ni²⁺; 200 times of Pb²⁺; 100 times of Bi³⁺, 20 times of Sb⁵⁺, Hg²⁺ to the determination of sample with 4 µg L⁻¹ arsenic were tested. The result shown that with the 10 g L⁻¹ thiourea, all above the existence of these multiple metal elements did not affect the determination of arsenic.

Linear range, detection limit and precision: The results showed that the working curve of arsenic had good linear coefficient in the range of 0-80 µg L⁻¹, with a correlation coefficient of 0.9995. Continuous determination of the standard blank...
solution 11 times, according to the instrument detection limit DL = 3\(\sigma/K\) (\(\sigma\): the standard deviation of fluorescent intensity of blank sample solution, K: the slope of standard curve), while the detection limit was 0.036 \(\mu\)g L\(^{-1}\). The standard arsenic solution with a concentration of 0.6 \(\mu\)g L\(^{-1}\) was continuously measured for 11 times to calculate the relative standard deviation was 1.7%.

**Determination of samples and recovery:** The results shown in Table-2.

| Samples          | Total arsenic (\(\mu\)g g\(^{-1}\)) | RSD (%) | Added (\(\mu\)g g\(^{-1}\)) | Recovery (%) |
|------------------|-------------------------------------|---------|-----------------------------|--------------|
| Panax notoginseng| 0.391                               | 3.7     | 0.2/0.3/0.4/0.5             | 90.7-103.5   |

**Conclusion**

This determination of total arsenic in the samples were higher than the standard of some foods\(^5\), the total arsenic in notoginseng was 0.391 \(\mu\)g g\(^{-1}\), which may be related to heavy metal pollution in the growing areas.

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