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Direct Determination of Arsenic in Potassium Citrate Tablet Using Graphite Furnace Atomic Absorption Spectrometry

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A B S T R A C T

In this study, a rapid and simple method based on graphite furnace atomic absorption spectrometry (GFAAS) is described for direct determination of arsenic in potassium citrate tablet samples. Pyrolytic graphite tubes were used as atomizers. Nickel nitrate was used as chemical modifier. The ashing and atomization temperature and the concentration of modifier were also optimized. The detection limit of the method was 0/8 ng/ml As in potassium citrate tablet samples.The relative standard deviation for ten determination of a spiked sample with concentration of 30 ng/ml As ranged was 2.2 %. The accuracy of the method was confirmed by the analysis of spiked samples. The linear rang of calibration is in the range of 0.8-80 ng/ml of arsenic.

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Introduction

Potassium citrate is a potassium salt of citric acid. Potassium citrate is used to regulate acidity. Medicinally, it may be used to control kidney stones derived from either uric acid or cystine. Potassium citrate is rapidly absorbed when given by mouth and is excreted in the urine as the carbonate. It is, therefore, effective in reducing the pain and frequency of urination when these are caused by highly acidic urine. It is used for this purpose in dogs and cats, but is chiefly employed as a non-irritating diuretic. Potassium citrate is an effective way to treat/manage gout and arrhythmia, if the patient is hypokalemic. In common with other substances that render the urine alkaline, it may be used to reduce the danger of crystalluria during sulfonamide therapy. It is widely used to treat urinary calculi (kidney stones), and is often used by patients with cystinuria. A study of 500 patients with recurrent stones found that it reduced the frequency of stones from 2 per year to a half per year. It is also used in many soft drinks as a buffering agent [1]. Arsenic is a toxic trace element. The malignant symptoms may appear even when trace arsenic is ingested. The toxicity mechanism of arsenic has been shown that it binds to enzymes, which are inhibited for functioning [2]. The toxicity of arsenide depends on the forms of chemical compound. It can be relatively retained in tissue but slows excretion. The organic arsenic complexes, which are commonly found in food, are readily absorbed in the body and excreted through urination. Arsenic in body shows high affinity to cuticle. Therefore, arsenic can be more readily accumulated into hair and nail than other tissues [3]. Some analytical methods such as: Colorimetry [4, 5], Atomic Absorption Spectrometry [5,6], hydride generation system combined with Atomic Absorption Spectrm-etry [6,7], and Atomic Fluorescence Spectrometry [8] have been used for trace determination of arsenic. Using above methods, sample prepartion is required prior to instrumental analysis. Because of low concentration of arsenic in potassium citrate and the low boiling point of arsenic compounds digestion is difficult for this sample and direct determination without digestion is a simple, low cost and time consuming way for determination of arsenic in potassium citrate tablet. The aim of this work was to investigate the possibility of the direct measurement of arsenic in potassium citrate tablet using graphite furnace Atomic Absorption Spectrmetry without any dry or wet sample digestion.

Materials and Methods

Instruments and apparatus

All of the analysis were performed using an AA-6650 atomic absorption spectrophotometer (Shimadzu Co. Ltd., Japan) equipped with a deuterium lamp background cor-rection system, a GFA-EX7 graphite furnace and an ASC-6100 autosampler was employed for the determination of arsenic. Hallow cathode lamps (HCL) were used as the light source. All the measurements were based on inte-grated absorbance. The wavelengths used were 248.3 nm (slit 0.5nm) for arsenic. Pyrocoated graphite tubes, with integrated platforms or L'vov platforms, were used for the atomization of arsenic. Nitric acid 69% (Merck) were also used for diluting the samples and sample mineralization. All of potassium citrate tablet samples (urocit-k) were ob-tained from pharmacy. Argon N50, with 99.999% purity, was used as sheath gas for the atomizer and to purge internally. All glassware were kept in 10% nitric acid for at least 48 h and rinsed with ultrapure water before use.

ETAAS detection

The samples were analyzed by ETAAS under optimum conditions (Table 1, 2). Nickel was used as a chemical modifier for Arsenic determination. 2.0 g of powdered potassium citrate tablet was dissolved in the water and the mixture volume up to 10 ml then10 µL of solution of the sample and 10 µL modifiers were simultaneously injected into the pyrocoated graphite tube of the furnace.

Results and Discussion

Optimization of ashing and atomization temperature

The optimum ashing temperature was found to be 1100 ^{0}C over the temperatures from 600 to 1300 ^{0}C tested when the atomizing temperature at 2100 ^{0}C was used.

Optimization of nickel concentration

Nickel on the absorbance signal of As in the dissolved potassium citrate was also studied and the obtained results are shown in Figure 2. It is obvious that with 4 mg/ml of Ni as modifier the matrix interferences are practically eliminated and the

absorbance signal for as is in the best sensitivity. The effect of atomization temperature on absorbance signal investigated by increasing the atomization temperature from 1800 ^{0}C to 2500 ^{0}C and the best sensitivity was obtained in 2100 ^{0}C (Figure 1). Thus this temperature was used for further studies as atomization temperature.

Modifier the matrix interferences are prac-tically eliminated and the absorbance signal for As is in the best sensitivity.

Table 1. mesurment condition for determination of arsenic using AA-6650 Shimadzu atomic absorption spectrophot-ometer.

Parameter	Setting
Wavelength	193.7 nm
Band pass	0.5 nm
Signal Measurement	AA-BG (peak height)
Type of Tube	Pyrolytic coated
Type of Inert Gas	Helium
Type of Lamp	Hallow Cathode Lamp
Lamp Current	10 mA

Table 2. Temperature program for determination of arsenic in potassium citrate samples.

Step	Temp (c)	Time (s)	Heat mode	Flow rate
1	150	20	Ramp	0.1
2	250	10	Ramp	0.1
3	1100	10	Ramp	1
4	1100	10	Step	1
5	1100	3	Step	0
6	2100	2	Step	0
7	2500	2	Step	1

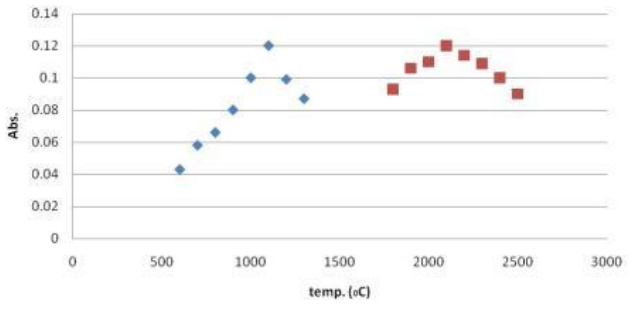

Fig. 1. The effect of ashing and atomization temperature on absorbance signal of arsenic (10 ng/ml) in dissolved potassium citrate sample.

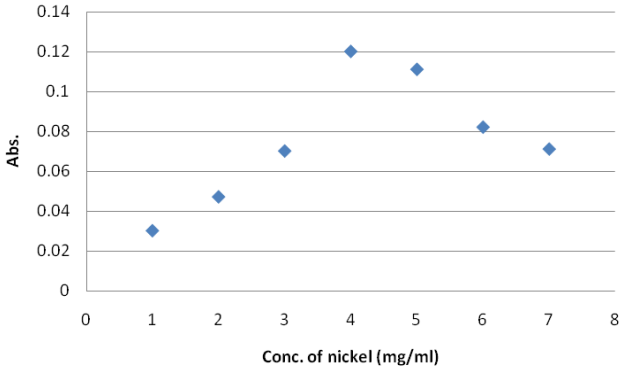

Fig. 2. The effect of the different masses of nickel, used as a modifier, on the absorbance signal of As (10 ng/ml) in the dissolved potassium citrate (0.02 g/l)

Comparison of the slopes of the standard curves of spiked sample and standard solution

The comparison of the slope of linear calibration curve of standard solutions and standard addition curve of spiked sample were performed for evaluation of matrix elimination of sample. using common statistical tests (ANOVA, Student's t test, 95% confidence level) the comparison between the slope of linear calibration curve (0.004 ± 0.005)and standard addition curve of spiked sample (0.004 ± 0.015) shows good agreement and demonstrate that there is no significant differences between standard addition method and linear calibration method and indicates the analysis is not interfered by sample matrix and conforming the suitability of the method for direct determination of arsenic in potassium citrate samples.

Accuracy, detection limit and precision of the method

In order to checking of the accuracy, recovery test was performed by spiking several concentrations of arsenic standard to sample which were then analyzed using the established method. Recoveries from sample at 10, 20, and 60 ng/ml of arsenic were found to be 102, 98 %, and 103 %, respectively (Table 3). All recoveries were greater than 90% . Above results indicate the established method is capable of yielding a satisfactory recovery. The precision of the method was evaluated using the relative standard deviation, *RSD %* , of repeated determination of the analytes at 30 ng/ml concentration level, The precision obtained,

was 2.2 %. The limit of detection was calculated to be 0.8 ng/mL, which was determined by analyzing potassium citrate sample, ten times consecutively. Using the above optimum temperatures to analyze an arsenic standard solution, the equation of calibration curve plotted by peak height versus concentration was regressed to be Y=0.004X + 0.042 with a relative coefficient of 0.999. The linear rang of calibration is in the range of 0.8-80 ng/ml of arsenic.

Table 3. Recovery of arsenic from potassium citrate samples using proposed method.

Added arsenic (ng/ml)	Founded arsenic (ng/ml)	Recovery (%)
-	N.D	-
10	10.2 (± 0.07)	102
20	19.6 (± 0.09)	98
40	41.2 (± 0.02)	103

Conclusions

The proposed ET-AAS method for direct determination of arsenic in potassium citrate tablet samples is efficient, simple and fast. Potassium citrate tablet could be prepared by simple dilution in water. As a consequence, analytical costs and reagents consumption are reduced. All parameters studied (the ashing temperature, atomization temperature and the amount of modifier) influence the determination efficiency. The main goals achieved with the proposed method include, high speed and lower analytical costs, when compared with decomposition procedures prior to analysis.

Conflict of interest

Authors certify that no actual or potential conflict of interest in relation to this article exists.

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