



## Detection of rubidium in mica using atomic absorption spectrometry

Xiaozhou Li<sup>a</sup>, Zhiyi Xu<sup>b</sup>, Tianyue Yang<sup>a</sup>, Deli Wang<sup>a</sup> and Su Zhang<sup>c\*</sup>

<sup>a</sup>School of Science, Shenyang Ligong University, Shenyang, China

<sup>b</sup>Applied Technical School, Liaoning Institute of Science and Technology, Benxi, China

<sup>c</sup>Section of Physiology and Biochemistry, Shenyang Sport University, Shenyang, China

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### ABSTRACT

*In this paper, we established a method to detect the rubidium in mica using atomic absorption spectroscopy. In our experiment, the effects of the amount of the deionization agent of potassium ion solution, the concentration of hydrochloric acid and the coexistence ions on the determination of rubidium were investigated. Under the selected measuring condition, the detection limit of atomic absorption spectrometry was 0.0365 µg/mL, the relative standard deviation was 0.08%, and the recovery was 101%.*

**Keywords:** rubidium detection, mica mine, atomic absorption spectroscopy

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### INTRODUCTION

Rubidium belongs to dispersion elements, and has been widely used in the field of national defense, aerospace, biotechnology, medicine, energy and environmental sciences. Detection of rubidium can be used as a main reference for geological prospecting, mineral beneficiation, metallurgy and materials processing. At present, the major methods for the detection of rubidium are atomic absorption spectroscopy[1], atomic emission spectroscopy[2] and fluorescence spectroscopy[3]. Analytic targets are mainly water samples and biological samples, but few on the content analysis of rubidium in ore sample[4]. Considering the disturbance of ionization, coexistence ions and the selection of experimental condition, our research put forward an atomic absorption spectroscopy method for the detection of rubidium in mica ore, on the purpose of fulfilling the requirement of ore treatment plant and suspension experimental analysis.

### EXPERIMENTAL SECTION

#### 1 Agents

10% potassium solution was made by diluting 189.74 g KCl with pure water to 1000 mL.

1 mg/mL Rb<sub>2</sub>O standard stock solution was made by diluting 1.2937 g RbCl with pure water to 1000 mL. Dilute this stock solution to Rb<sub>2</sub>O working solution of 100.

#### 2 Equipment

Spectrophotometer was WYS-402C atomic absorption spectrophotometer. After the determination of the amount of potassium used in our experiment, optimum conditions for rubidium determination was made in accordance to the figure of experimental results: the wavelength was 780 nm, the slit width was 0.7 mm, the lamp current was 4 mA, the air flow was 0.25 m<sup>3</sup>/h, the flow rate of acetylene was 0.07 m<sup>3</sup>/h, the gas height was 8 mm, and the integration time was 3 s.

#### 3 Samples

According to sample content, weighted 0.5000–1.0000 g sample to platinum dishes accurately. Then wetted the

samples with water, added 10 mL hydrofluoric acid and 1 mL nitric acid, and put it in a low-temperature electronic furnace to let it decompose. Added 10 mL hydrofluoric acid after drying and heat it again in the low-temperature electronic furnace. After nearly drying by distillation, added 2 mL perchloric acid, then steamed it again on high temperature furnace to the dryness degree of smoking. Then add 2 mL perchloric acid and evaporated it to near dryness. After that, add 1:1 hydrochloric acid 2 mL or 4 mL, rinse the dish wall using hot water and heated it to dissolve the residue. Moved it in to a 50 mL volumetric flask after cooling.

## RESULTS AND DISCUSSION

### 1 The impacts of potassium solution on the determination of rubidium

In 4  $\mu\text{g/mL}$  rubidium solution, added potassium solution with different concentrations, and we got the relationship between the absorption value of rubidium and the concentration of potassium under the optimized working conditions as shown in Figure 2:

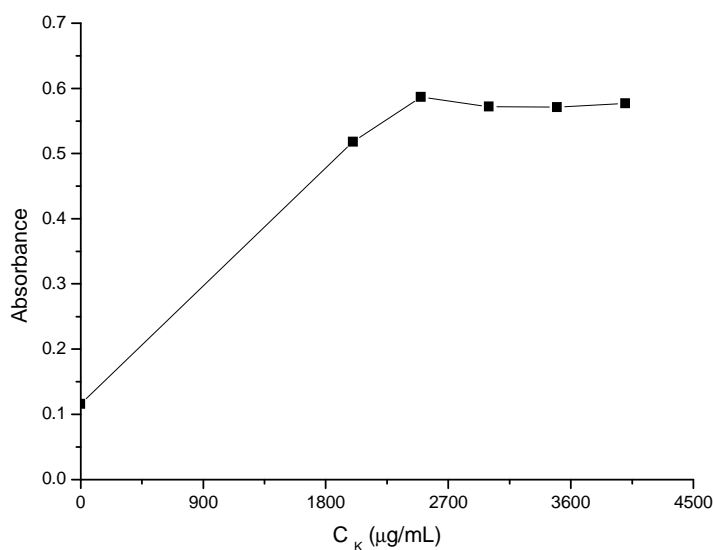


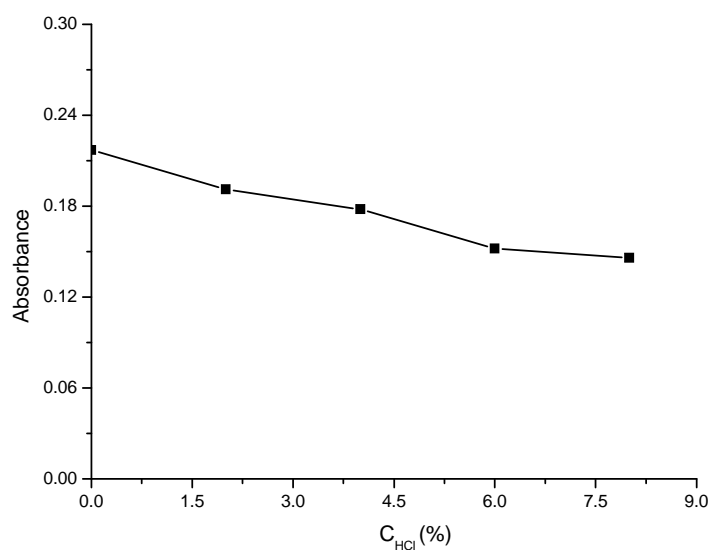
Figure 1 Effects of the concentration of potassium to the absorption value of rubidium

Figure 1 shows the hypersensitization situation between potassium and rubidium. Ionization interference existed when rubidium was in the air-acetylene flame. Hypersensitization widely existed after adding large amount of salt solution, and sensitivity will increase significantly most of the time. This is because rubidium in flames has partially been ionized.

The degree of ionization of rubidium solution with a concentration of  $10^{-3}$  M in air-acetylene flame is 75% [5], resulting in reduced base-state atoms that can be absorbed, this further reduced absorption value of Rb. When there existed potassium which are more easily to be ionized, because of the large amount of ions provided by potassium, the ionization of Rb will decrease, thus the amount of base-state atoms being absorbed by flame increased, then the absorption increases. Seen from Figure 1, when the amount of potassium was added to 2000 - 5000  $\mu\text{g/mL}$ , the ionization of rubidium tends to zero, this means the element ionization does not increase with the increase of strong sensitivity substance, so the sensitization of potassium reached to a constant. When the concentration of potassium is large enough to eliminate the ionization of detection substances, we should use the concentration as low as possible to evade the clogging of nozzles and burner. Therefore, every time before the detection of atomic absorption, the optimal concentration of potassium should be 3000  $\mu\text{g/mL}$ .

### 2 The impact of hydrochloric acid medium

Rubidium standard working solution of 4  $\mu\text{g/mL}$  and potassium solution of 3000  $\mu\text{g/mL}$  were added in hydrochloric acid with different concentrations, under the experimental condition stated above, the results were shown in Figure 2.



**Figure 2** The impact of the concentration of hydrochloric acid medium on the absorption value of rubidium

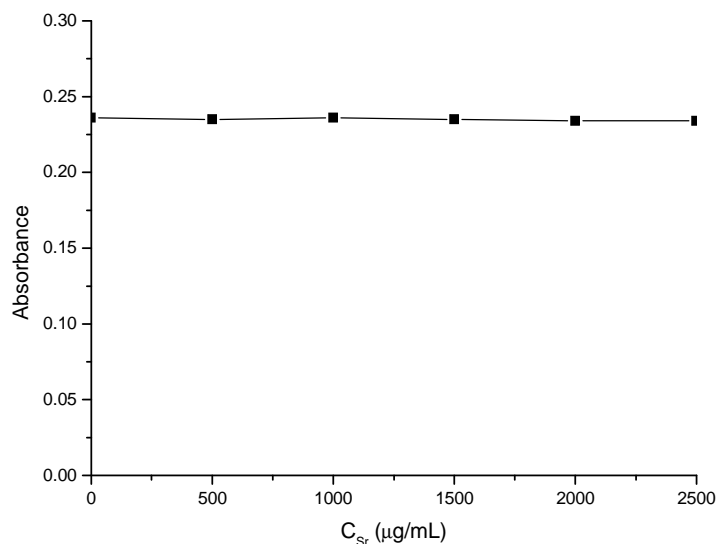
Figure 2 shows that, the absorption value of rubidium decrease with the increase of the concentration of hydrochloric acid, therefore, the concentration of hydrochloric acid medium was maintained at 2%.

### 3 The effects of coexisting ions

There are a wide range of geological samples, and coexisting elements are complex. Our research mainly investigated 5 relatively high content elements in mica mine.

#### 3.1 The impact of strontium on the detection of rubidium

Under the above stated working conditions, test the absorption value of 4  $\mu\text{g/mL}$  rubidium at different concentrations of strontium, and the results were shown in Figure 3.



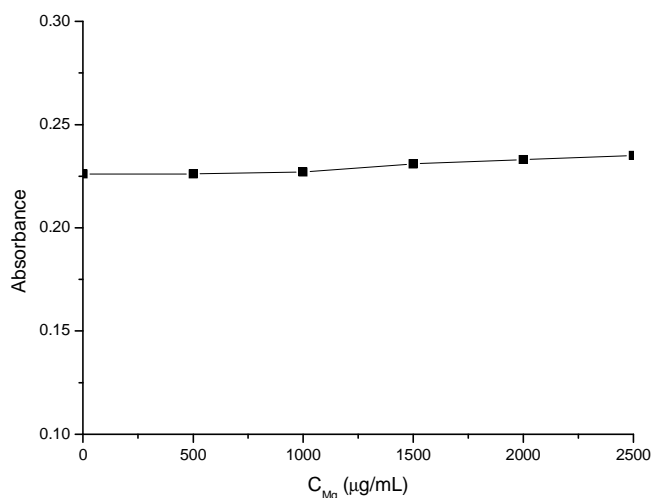
**Figure 3** The impact of strontium on the absorption value of rubidium

From Figure 3 we can see that strontium has no apparent effect on the absorption value of rubidium, therefore, the presence of strontium can be neglected in the detection of the absorption value of rubidium.

#### 3.2 The effect of magnesium on the detection of rubidium

Under the above stated working conditions, test the absorption value of 4  $\mu\text{g/mL}$  rubidium at different

concentrations of magnesium, and the results were shown in Figure 4.

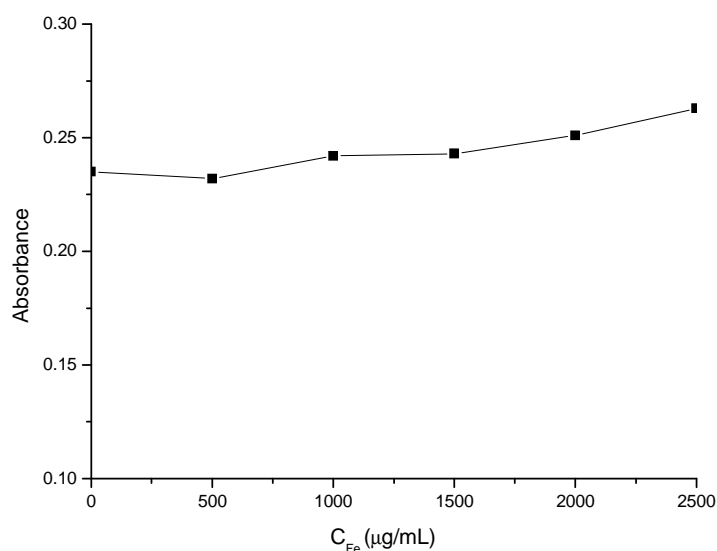


**Figure 4** The impact of magnesium on the absorption value of rubidium

From Figure 4 we can see that magnesium has no apparent effect on the absorption value of rubidium, therefore, the presence of magnesium can be neglected in the detection of the absorption value of rubidium.

### 3.3 The effect of iron on the detection of rubidium

Under the above stated working conditions, test the absorption value of 4 µg/mL rubidium at different concentrations of ions, and the results were shown in Figure 5.

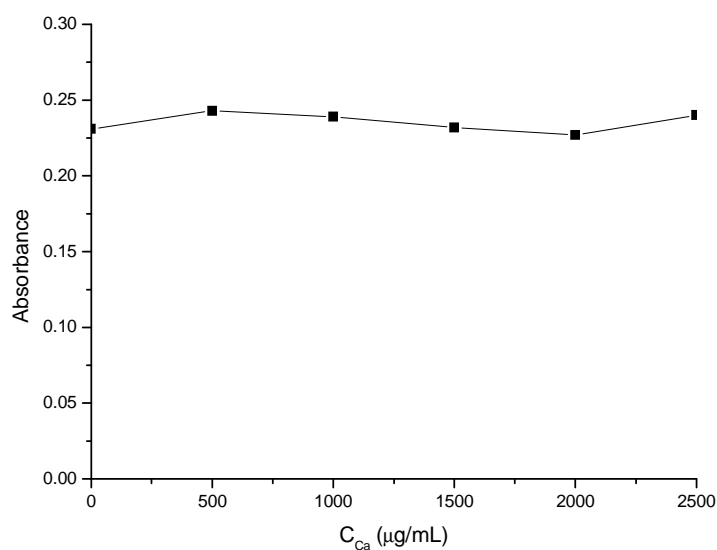


**Figure 5** The impact of iron on the absorption value of rubidium

From Figure 5, we can see that when the concentration of iron was under 1000 µg/mL, it has almost no effects on the absorption value of rubidium in mica ore, but has some effects when the concentration of iron was more than 1000 µg/mL. So the measurement of the absorption value was taken place with the concentration of iron controlled under 1000 µg/mL.

### 3.4 The effect of calcium on the detection of rubidium

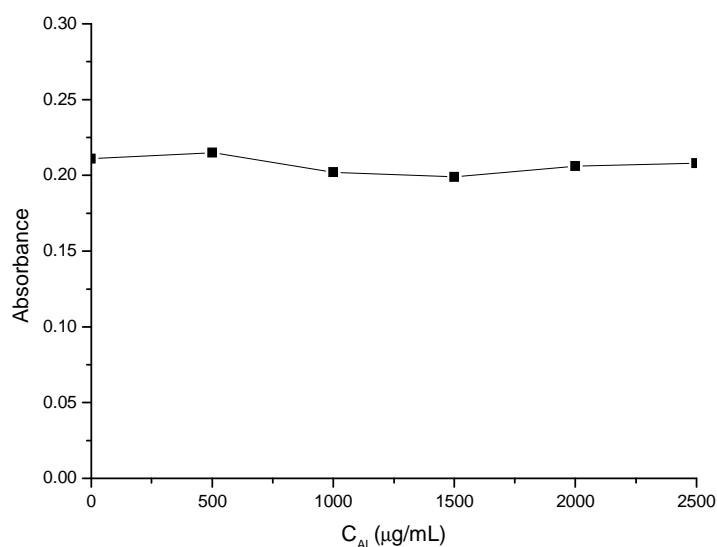
Under the above stated working conditions, test the absorption value of 4 µg/mL rubidium at different concentrations of calcium, and the results were shown in Figure 6.



**Figure 6** The impact of calcium on the absorption value of rubidium

From Figure 6 we can see that calcium has no apparent effect on the absorption value of rubidium, therefore, the presence of calcium can be neglected in the detection of the absorption value of rubidium.

### 3.5 The effect of aluminum on the detection of rubidium

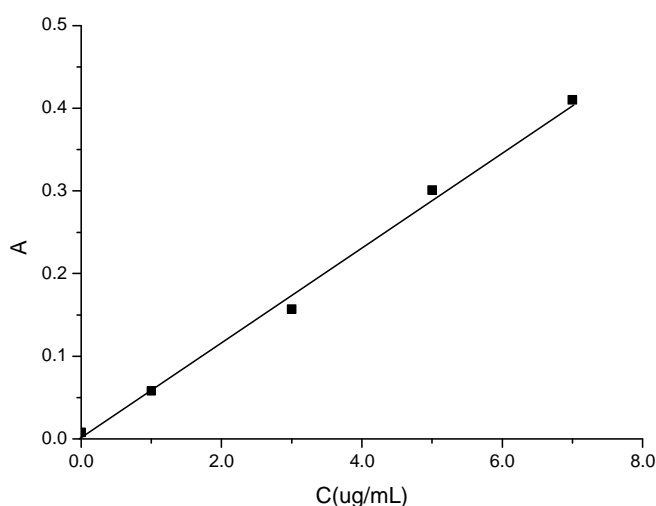


**Figure 7** The impact of aluminum on the absorption value of rubidium

From Figure 7 we can see that aluminum has no apparent effect on the absorption value of rubidium, therefore, the presence of aluminum can be neglected in the detection of the absorption value of rubidium.

### 4 Working curve

Preparing a series of standard solution of rubidium under the optimum experimental conditions. Then measure the absorption value of each solution and plotted the results in Figure 8. The corresponding linear fitting equation is:  $A = -0.0006 + 0.058 \times C$ , the correlation coefficient  $R^2 = 0.998$ . This result showed that rubidium has a good linear relationship within the range of 0 - 7.0 µg/mL.



**Figure 8 Working curve of rubidium**

### 5 Recovery test

Weighted 4 ore samples accurately, added in rubidium with the weight of 0, 50, 100 and 200  $\mu\text{g}$  separately. After the samples being treated by digestion method, the measurement was performed under the pre-setted equipment condition separately. The results are listed in Table 1.

**Table 1 Recovery test of standard addition of ore samples**

No.	Added ( $\mu\text{g/mL}$ )	Recovered ( $\mu\text{g/mL}$ )	Recovery (%)
1	0	-	
2	1	1.01	101
3	2	1.94	97
4	3	2.88	96

### 6 Detection limit

Detection limit was tested under the selected experimental condition. Blank solution was tested for 11 times. In accordance to the regulation of IUPAC[6], the detection limit of rubidium was calculated to be 0.028  $\mu\text{g/mL}$  ( $S/N=2$ ).

### 7 Analysis of mica ore samples

Weighted 5 samples of mica ore accurately, then made them to sample solutions after the treatment method of experiment. Did the absorbance measurement according to the acquired condition of our equipment. The mean value is calculated to be 2.84  $\mu\text{g/mL}$ , and the relative standard deviation is 8%.

## CONCLUSION

Using atomic absorption spectrophotometry to detect the rubidium in mica ore is convenient, time-economic. It need less acids, and has high sensitivity. Our experiment proved that this method is simple and reliable, has high selectivity and good precision.

The value of the ionization degree of rubidium is related to both the lifting capacity and the concentration of analysis solution[7]. When the lifting capacity increase or the solution concentration increase, the amount of solution and the concentration in flames will increase, thus will consume more thermal energy. Meanwhile, the vapor pressure in the flame will increase, and consequently will decrease the ionization. So in addition to add large amount of potassium solution to eliminate the disturbance of ionization, it can also increase the lifting capacity a little. Furthermore, we should note that, because high amount of potassium is likely to have salt accumulation effect, we should always rinse the spray chamber.

Because rubidium is a metal with a low melting point, high lamp current should not be used when measured. The warming-up time of lamp should not be too long before the measurement, and the lamp should not be removed rightly after the detection to ensure the service life of lamp.

**Acknowledgments**

We give our sincere thanks to the support provided by Prof. Xingjia Guo in Liaoning University.

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