

## Potassium Analysis by Atomic Absorption

### ■ Introduction

K exists primarily as silicate in the earth's crust. Moreover, it is present at about 400ppm in sea water, which is about 1/30 of the concentration of Na. This difference is due to the fact that while  $\text{Na}^+$  passes as it is from the weathering of rocks into the oceans,  $\text{K}^+$  adheres to colloidal substances from the earth's crust to be absorbed by plants, so that only a small amount flows into the oceans. K obtained by electrolysis of melted KOH or KCl is a silver white soft metal which oxidizes quickly in the air, and since it reacts explosively with water to ignite, it is stored in petroleum. Since K forms the superoxide  $\text{KO}_2$  and is abundantly reactive, it is different from Na, another of the alkali metals. Compounds of K include  $\text{KNO}_3$ , which is used as an oxidant in matches and fireworks, etc., KOH, a soap,  $\text{K}_2\text{CO}_3$ , a raw material in glass, and KCl, used in fertilizer formulations.

K is involved in various physiological functions, such as protein synthesis, intra- and extra-cellular water transport and signal transmission, and is an essential

element for living organisms. K is present in the human body at about 2g/kg, with concentrations in muscle at about 16,000ppm and blood at about 2100ppm. Food-contained K is absorbed in the small intestine, and approximately 90% of excess K is excreted from the kidney, and the remainder via fecal matter and perspiration. It is thought that K deficiency almost never occurs due to the action of homeostasis, however, K deficiency brought about by extreme diarrhea or vomiting can cause symptoms such as arrhythmia.

On the other hand, when uremia or urinary tract blockage occurs, hyperkalemia can result, causing arrhythmia or other abnormalities on electrocardiograms. KCl is used as a supplement for K, in Ringer's solution (1L containing 8.6g NaCl, 0.3g KCl, 0.33g  $\text{CaCl}_2$ ), and in various medicinal drugs, such as in diuretics, etc.

We introduce here the differences and utilization of the peak height method and peak area method for signal processing in furnace analysis of K as an example.

### ■ Basic Data of K

Atomic Weight	39.098
Melting Point	63.7°C(KCl 776°C, $\text{KNO}_3$ 339°C, $\text{K}_2\text{SO}_4$ 1069°C)
Boiling Point	774°C(KCl 1500°C)
Oxidation Number,	-1 in liquid ammonia +1 $\text{K}_2\text{O}$ , KOH, KCl
Solubility	$\text{KNO}_3$ 31.6g/100g Water(20°C) $\text{K}_2\text{SO}_4$ 11.1g/100g Water(20°C)

Reference Physics and Chemistry Dictionary, etc.

### ■ Wavelength of K

	Sensitivity Ratio
766.5nm	1.0
769.9nm	0.25
404.4nm	0.003

### ■ Furnace Analysis of K

The types of peak signal processing used in furnace analysis consist of the peak height method and peak area method. In order to use the peak height method, the peak shapes of the standard solution and those of the samples must be similar at least. Even if they are similar, the accuracy of the analytical results cannot be guaranteed, but prior to selecting the peak signal processing, comparison of the standard solution's and sample's peak shapes is required. Dissimilarity of the peak shapes can be attributed to interfering substances in the sample, inappropriate heating conditions, etc. The presence of an atomization-inhibiting substance in the sample may cause peak-widening. Conversely, sharpening of peaks could be caused by the presence of substances that promote atomization. When there is a difference in peak shape, some measurement accuracy improvement can be expected by using the peak area method. However, since this cannot be said with complete certainty, the decision as to whether to use the calibration curve method or the standard addition method should be made based on the results of recovery rate tests conducted beforehand.

The validity of the standard addition method's analytical conditions, comprising the standard solution addition concentration and the heating conditions, can be confirmed when compared with the results of the peak height method and those of peak area method. Using the standard addition method, the analytical values obtained from the peak height and peak area, respectively, should match. However, if they are different, we'd figure that the calibration curve concentration range was over than the range of linearity which is expected with the standard addition method, so it will be necessary to take another look at the sample dilution factor or standard solution addition concentration, with a view to changing the conditions.

↘ Shown below are comparison examples of K analysis using the peak height method and peak area method. A high density tube was used for the graphite cuvette, and the sample injection volume was 20 $\mu$ L. The heating conditions shown in Table 1 were used for generating the calibration curves. Fig.1 shows the calibration curve obtained using peak height and Ar gas (0.1L/min.) at the time of atomization. Fig.3 also uses peak height and Ar gas, however, the gas flow is stopped at the time of atomization. Fig.2 and 4 show the calibration curves generated using peak area.

Since the readings using peak area are normally smaller compared to those using peak height, the calibration curve linearity ranges are different even if the concentrations are the same. Moreover, the peak shapes at atomization with the Ar gas flowing show a tendency to be sharper than when the gas flow is stopped for sensitivity adjustment.

Table 1 HEAT CONDITION

Temp(°C)	Time(sec)	Heat Mode	Flow Rate(L/min)
150	20	RAMP	0.1
250	10	RAMP	0.1
800	10	RAMP	1.0
800	10	STEP	1.0
2000	2	STEP	0.1/0
2500	2	STEP	1.0

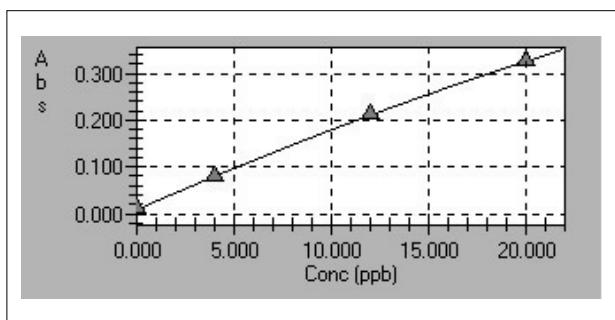


Fig.1 Calibration Curve (Peak Height, Gas0.1)

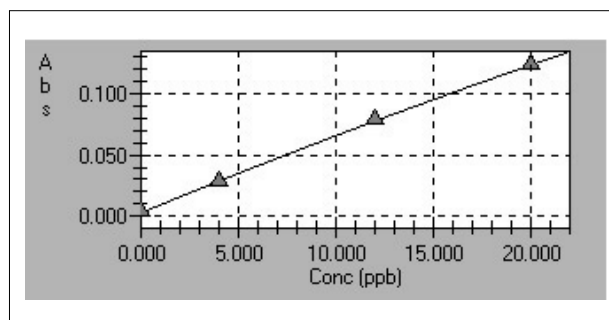


Fig.2 Calibration Curve (Peak Area, Gas0.1)

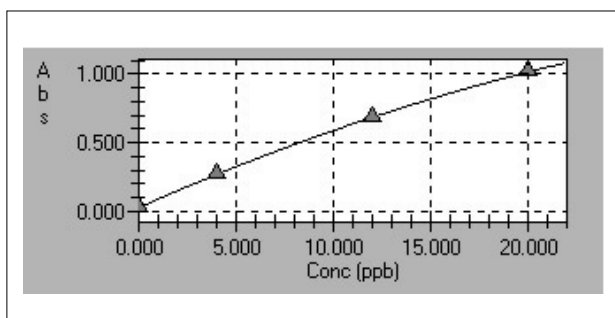


Fig.3 Calibration Curve (Peak Height, Gas0)

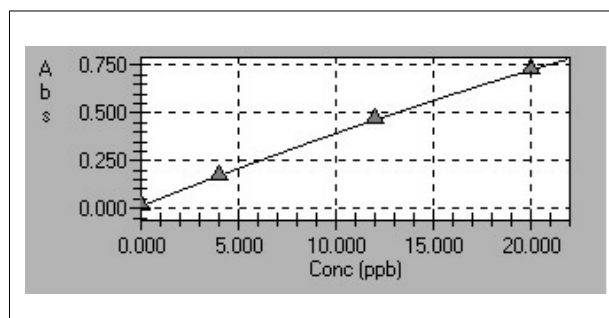


Fig.4 Calibration Curve (Peak Area, Gas0)

## ■ Conclusion

K is present in the earth's crust at a rate of about 2.4% and in the soil at about 1.4%, and like Na and Zn, is an element that is easily affected by pollution from the environment. Therefore, in trace analysis of K, it is necessary to consider pollution from equipment and reagents, not to mention the environment in which the analysis is conducted. Moreover, in cases where K is an impurity in the graphite tube, if the atomization temperature is too high, the K in the tube itself is atomized, causing phenomena such as high blank

values, peak splitting, etc.

Even when the same phenomena are seen with other elements, verify that the atomization temperature is not set higher than necessary. Halogenated compounds of K like KCl and KI become background components in the analysis of other elements. Also, the addition of nitric acid is effective to change these to forms that are easily volatilized during ashing. This utilizes the relatively low melting point of the nitrate salt of K.



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710  
Cable Add.:SHIMADZU TOKYO