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Determination of Potassium Picrate (KP) in KP-$KClO_4$ Ignition Composition by Spectrophotometry Analysis

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Abstract: When potassium picrate (KP) and $KClO_4$ are dissolved in water, the solution obtained is bright yellow for the former and colorless for the latter in appearance. With the help of this characteristic, the content of KP in KP-$KClO_4$ ignition composition is determined by spectrophotometry analysis. The determining conditions is discussed, and the relationship between the absorbance and the concentration of KP is also studied. Results show that the uncertainty and the inaccuracy of the experimental data are 0.2% and 1.3%, respectively, which indicate that the method of determining the content of KP in KP-$KClO_4$ ignition composition is convenient and feasible for the system.

Key words: analytical chemistry; spectrophotometry; KP-$KClO_4$ ignition composition; potassium picrate (KP)

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1 Introduction

The admixture of potassium picrate (KP) and $KClO_4$ is an ignition composition, which has safe and low environment effect. It has low sensitivity, low moisture absorption, low environment pollution except high ability of ignition[1-6]. It is found that the proportion of KP and $KClO_4$ can affect the igniting ability seriously, therefore, how to determine this proportion becomes a difficult problem in the course of ignition composition examination.

Spectrophotometer can be used to analyze a substance quantitatively and qualitatively, according to its absorbance to the ray of special or definite wavelength.

KP is a yellowish color compound, which shows bright yellow stably and obviously, while dissolving in water. $KClO_4$ solution is colorless. So, we can determine the content of KP by spectrophotometric analysis exactly.

2 Experimental

2.1 Reagents and instrument

The KP-$KClO_4$ ignition composition and KP used in this work were prepared by ourselves. The absorbance of KP in the ignition composition was obtained by a 722S spectrophotometer. KOH and $KClO_4$ were of A.R. grade. The concentration of HNO_3 used as solvent was 65% ~ 68%. The concentration of KOH and HNO_3 in the deionized water was 0.2 mol·L^{-1} and 0.5 mol·L^{-1} respectively. The conductivity of the deionized water was 15.6 s·cm^{-1}.

2.2 Preparation of the solution

An accurately weighed ignition composition of 200 mg was dissolved in 150 mL water, transferred into 250 mL standard flask and diluted to the mark with water. Five mililitre of sample were placed to another 250 mL standard flask, diluted to the mark with water, either. The absorbance of the solution was determined by a spectrophotometer.

3 Results and discussion

3.1 Confirmation of the method of direct photometric analysis

Potassium picrate standard solution of 16 mg·L^{-1} was prepared. Using water as a reference, the absorbance (A) of the standard solution in the wavelength (λ) range of 340 nm to 380 nm was measured and A vs. λ curve was shown in Fig. 1, which shows that the wavelength that KP can be absorbed the most is 364 nm.

We found that there was no absorbance in visible light when $KClO_4$ solution was determined in the same way. So, the existence of $KClO_4$ can not affect the consequence when we determine KP in this way.

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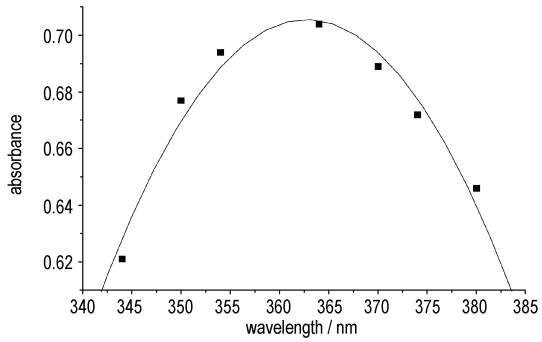

Fig. 1 Curve of A vs. λ for KP standard solution

3.2 Effect of pH value of the solution

A series of 16.4 mg · L^{-1} KP standard solution with different pH value were obtained by using 0.2 mol · L^{-1} KOH solution and 0.5 mol · L^{-1} HNO_3 solution as pH value adjustment. The value of A at 364 nm was measured and curve of A vs. pH value was shown in Fig. 2, indicating that the value of A did not change in the pH value range of 2 to 13. So, we could draw a conclusion that the pH value did not affect the value of A in this range.

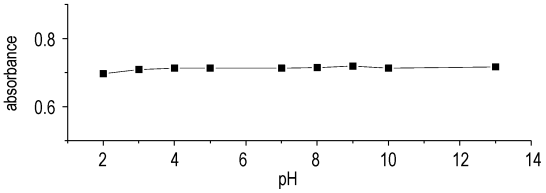

Fig. 2 Value of A as a function of pH value

3.3 Determination of the linearity range for working curve

The standard solutions of ignition composition with different concentration (C) were dispensed. Their absorbance was measured and the A vs. C relation was shown in Fig. 3, which shows a liner relationship between the parameter C and A, in the concentration range of 2 – 20 mg · L^{-1}, $C = -0.44024 + 21.62208A$, with the correlation coefficient of 0.9975.

3.4 Determination of the solution absorbance

Three copies of solutions of ignition composition were prepared, and their absorbance at 364 nm was measured for 6 times separately. The results were abstracted in Table 1 which indicates that the uncertainty by twice the standard deviation of the mean for all the data is within ±0.2%.

Substituting the consequence of absorbance into the regression equation, the content of KP in the concentration range of 2 – 20 mg · L^{-1} is obtained. The content of KP in the ignition composition by calculation is 55.8%, which is in agreement with the content of 55.1% of KP in formulation, showing that the inaccuracy of the measured result is 1.3%.

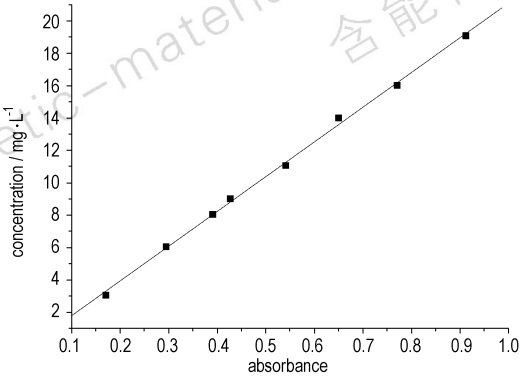

Fig. 3 Curve of A vs. C for the standard solution

Table 1 Absorbance of the solution of ignition composition

No.	1	2	3	4	5	6
1	0.435	0.435	0.434	0.434	0.434	0.433
2	0.434	0.433	0.433	0.435	0.436	0.436
3	0.433	0.436	0.436	0.433	0.433	0.433

4 Summary

The solution of KP shows bright yellow with the most absorption at 364 nm. The pH value of the solution does not affect the absorbance in the range of 2 – 13. The linear calibration graph is obtained in the concentration range of 2 – 20 mg · L^{-1} and the equation of calibration graph is $C = -0.44024 + 21.62208A$. The content of KP in KP-$KClO_4$ ignition composition is obtained by determining the absorbance with the equation of calibration graph. The results indicate that the method is convenient and feasible.

参考文献:

[1] LI Xing-wang, GAO Yi. A new electric igniter composite [J]. *Explosive Materials*, 1999, 28(2): 13 – 15.

[2] CHENG Yi, CHEN Shou-wen. Study on ignition characteristics of ignition composition [J]. *Initiators and Pyrotechnics*, 2001, (4): 21 – 23.

[3] ZHOU Qing-qing. The erosion effect of fuseheads of potassium chlorate-carbon and red lead-silicon systems on iron legwires [J]. *Explosive Materials*, 1994, 23(2): 22 – 25.

[4] SONG Yu-qin, CHU Yong-xian. The properties and application of D · E coprecipitating primary explosive [J]. *Initiators and Pyrotechnics*, 1997, (3): 26 – 31.

[5] XIAO Xiang-jie. Analysis of application of $KClO_3$ and $KClO_4$ [J]. *Technology and Market of Fireworks and Pyrotechnics*, 1995, (2): 15 – 17.

[6] Faber Michael, Herdin Gunther, Zollner, et al. Ignition elements and finely graduatable ignition component [P]. USP 6651563, 2003.

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Study on Charcoal-free Black Powder

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Abstract: Through modifying proportions of potassium nitrate, sulfur, phenolophthalein and potassium hydrate, formula of charcoal-free black powder is optimized by replacing charcoal with reactant of phenolphthalein and potassium hydrate. Properties between ordinary black powder and charcoal-free powder are compared, including mechanical sensitivity, static eletricity sensitivity, thermal decomposition process and output capacity. The results indicate that the mechanical sensitivity, static electricity sensibility and ignition temperature of charcoal-free black powder is lower than that of ordinary black powder, and the safety ability, working capacity and output stability of charcoal-free black powder is higher than that of ordinary black powder.

Key words: military chemistry and pyrotechnics; black powder; ignition powder; phenolphthalein

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Desensitizing Technique of Ammonium Nitrate

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Abstract: Ammonium nitrate is modified by adding anti-explosion agent to reduce its impact sensitivity. According to thermal decomposition of AN and hot-spot theory, six chemical materials such as ZnO, $(NH_2)_2CO$, Na_2HPO_4, camphor, $Mg_2(OH)_2CO_3$, Na_2CO_3, NH_4NO_3 were selected as components of anti-explosion agents. Modified AN were prepared by adding these chemical materials to formulate industrial explosives. The impact sensitivity of these explosives at drop height of 65 cm with 10 kg drop hammer was tested. Results show that comparing with the pure explosion in which has no additive, when 5% anti-explosion agent ZnO/Na_2HPO_4/$Mg_2(OH)_2CO_3$ = 33/34/33 are added in system, the impact sensitivity can be decreased from 88% to 24% and the impact sensitivity also declines with the decrease of hardness and stacked density of modified AN. The same result can be obtained by slow crystallization method.

Key words: applied chemistry; ammonium nitrate (AN); additive; impact sensitivity; anti-explosion agent

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分光光度法测定 KP-$KClO_4$ 点火药中苦味酸钾含量

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摘要: 根据苦味酸钾(KP)水溶液呈亮黄色和 $KClO_4$ 水溶液呈无色这一特点,用分光光度法测定了 KP-$KClO_4$ 点火药中 KP 的含量,讨论了测试条件,研究了苦味酸钾的吸光度与百分含量的关系。结果表明: 测定 KP-$KClO_4$ 点火药中 KP 含量的方法简便,可行,使用该方法得到的实验结果的不确定度和误差分别为 0.2% 和 1.3%。

关键词: 分析化学; 分光光度法; KP-$KClO_4$ 点火药; 苦味酸钾(KP)

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