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The Analysis of UV on No Traces Combustion-supporting in Fire Residue LI Ying-yu^{a,b}, SHEN Hao^{a,b}, LIANG Dong^{a,b,*}

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Abstract

In this paper, the ethyl nitrite which ethanol and sodium nitrite's reaction product has the UV absorption peaks in 300 nm \sim 400nm to qualitative identification for ethanol. The methanol has the similar absorption peaks in the same range, but these peaks have blue shift or red shift as the steric effects. Quantitative analysis of residual ethanol by the establishment of standard curves and recovery tests and other data analysis. Thus complete the qualitative and quantitative identification of ethanol.

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Key Words: UV -visible spectrophotometry, ethanol, no trace combustion-supporting, absorption peaks.

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1. Introduce

Fire investigation which is given to the public security fire control institutions by fire law is an important duty. The main tasks is to investigate and identify the cause of the fire, approve the fire losses, identify the fire responsibility[1]. Not only the gasoline, kerosene, diesel oil but also ethanol, acetone, ethyl which have no trace after burning have been become the common in the current cases of arson. That increase the degree of difficulty in fire investigation [2]. Therefore, the study about the no trace combustion-supporting has become the hot research. Hu ye and other scholars have been make the preliminary study on ethanol, acetone, etc.

2. Experimental section

2.1. Experimental principle

Ethanol itself does not produce the absorption peaks, but the ethyl nitrite which is the reaction product of ethanol and the sodium nitrite under the condition of hydrochloric acid has a group UV absorption peaks in 300nm \sim 400nm. These peaks are called "Five fingers peaks"[3]. The main reactions as following:

HL+NaNO2=NaCL+HNO2

CH3CH2OH+HNO2=CH3CH2NO2+H2O

The main by-products have nitric acid and acetaldehyde. The ethyl nitrite has a group of absorption peaks which have a distance of 1100cm-1 around 360nm. At 323nm, 333nm,344nm, 357nm, 371nm which are called "Five fingers peaks". Experimental results show that the methanol has the similar absorption peaks in the same

range, but these peaks have blue shift or red shift due to the steric effects. Respectively blue shift 3nm , 4nm , 4nm , 5nm , 5nm. The positions of absorption peaks at 320nm, 329nm, 340nm, 352nm, 366nm . The ether, acetone and other no trace combustion-supporting have no absorption peaks in this range. Therefore the ethanol can be identified accurately.

2.2. Reagents and Instruments

2.2.1 Reagents

Ethanol, $w\% \ge 99.7\%$; methanol, $w\% \ge 99.5\%$; ether, $w\% \ge 99.5\%$; acetone, $w\% \ge 99.5\%$; hydrochloric acid, $w\% \ge 99.7\%$; sodium nitrite, $w\% \ge 99.7\%$.

2.2.2Instruments

Agilent 8453 UV visible spectrophotometer

2.3. Experimental procedure

2.3.1 Preparation of reagents

Prepared sodium nitrite, 25%; Hydrochloric acid solution, $18 \sim 19\%$; The mixture of hexane and ethanol, 10g/l. 2.3.2 Preparation of Samples

[I] Preparation of standard samples: Take the mixture of hexane and ethanol of 10g/l about 20ml and sodium nitrite solution of 1ml and hydrochloric acid solution to a pear-shaped funnel. Reacting for 15min . In order to reaction adequately need to fully shake the funnel until it completely. The washing 4 times , until the solution colorless. Constant volume the product to 100ml, then taking 1ml,2ml,4ml,6ml,8ml. Set the volume at 25ml,numbered 1 # , 2 # , 3 # , 4 # , 5 # and are placed in the refrigerator at 10°C to make the standard curve.

[II] Preparation of interference sample: Separately take the methanol, acetone, ether of 1g reaction with the mixture of hexane and ethanol of 10g/l about 20ml under the condition of sodium nitrite of 1ml and hydrochloric acid solution of 1ml.the reaction process should be the same with the process of preparation of standard samples. Finally constant volume the product to 25ml,then placed in the refrigerator at 10° C.

[III]Preparation of the residual material of fire: Weight 3copies filter paper (a), cotton (b), wood (c), fabric (d) of 1g, numbered1a, 2a, 3a,;1b, 2b, 3b; 1c, 2c, 3; 1d, 2d, 3d;No.1,complete combustion under the condition of no combustion-supporting.No.2, complete combustion under the condition of adding ethanol of 2.5ml.Record the completely burning time, as following: 110s, 220s, 180s, 150s. No. 3,combustion under the condition of adding ethanol of 2.5ml, but control the burning time as 55s, 110s, 90s, 75s. Then extract the combustion products twice with distilled water of 20ml, which react with the mixture of hexane and ethanol of 10g/l about 20ml and sodium nitrite solution of 1ml and hydrochloric acid solution of 1ml. Reaction for 15min . In order to reaction adequately need to fully shake the funnel until it completely. The washing 4 times , until the solution colorless. Constant volume the product to 25ml, then placed in the refrigerator at 10°C.

3. Results and discussion

3.1. The establishment of standard curve

Make the UV determination for the standard samples that have been prepared. As following: 1 # , 2 # , 3 # , 4 # , 5 #. To the blank as reference, determine the A(absorbance) at the 357nm.

Sample No	1	2	3	4	5
ethanol content(mg)	0.08	0.32	0.64	0.96	1.12
Absorbance	-0.08834	0.09921	0.3756	0.6434	0.7857

Table 1 . Absorbance of standard sample is equivalent to the content of ethanol

By analyzing the experimental data can obtain the equation between ethanol concentration and absorbance: Y=0.84251x-0.16261

The correlation coefficient r=0.99987 that's better for ethanol. Measured linear range of $0.08 \sim 3.2$ mg/ml, so this method can be used in determination of ethanol.

3.2. Qualitative identification of ethanol

With UV visible spectrophotometer scanning the standard samples in the range of $300 \sim 400$ nm to determine the position of absorption peaks. Experiment's results show that the positions of absorption peaks at 323nm, 333nm, 344nm, 357nm, 371nm, the same with theoretical positions. To do the interference experiments with methanol, acetone, ether and find that methanol has the similar absorption peaks but these peaks have blue shift or red shift due to the steric effects. Respectively blue shift 3nm , 4nm , 5nm , 5nm. The positions of absorption peaks at

320nm, 329nm, 340nm, 352nm, 366nm. The ether, acetone and other no trace combustion-supporting have no absorption peaks in this range as shown in the Figure 1. Therefore the ethanol can be identified accurately. According to the size of absorption peaks the quantity can be calculated.

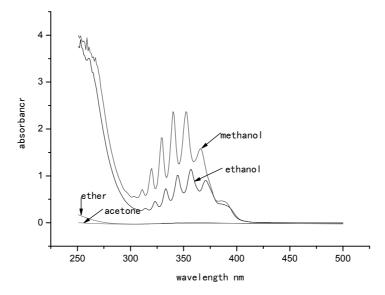


Figure 1 . Qualitative and interference test of ethanol

3.3. Quantitative identification of ethanol

The following is the results of UV on the combustion residue sample:

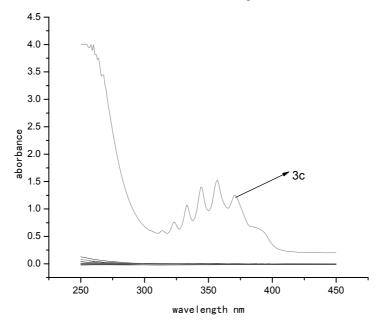


Figure2. The ethanol content of different burning residues

The chart shows that there didn't detect ethanol in the combustion products of the paper, cotton, cloth except the wood which didn't fully burning. According to the test results that it's very important for fire investigation to pick up the wood which didn't fully burning from the fire scene .The absorbance of not fully burning wood at 357nm is 1.52433. According to the standard curve equation:

Y=0.84251-0.16261

Calculated ethanol content is 2.0035mg. Thus complete the quantitative identification of ethanol.

In addition, the collection of fire evidence is the first step of fire investigation and is the most important step. It is the premise and guaranty for accurately identify the reason of fire. The law enforcement offers must targeted to collect the evidence of fire because fire scene is very messy. The chart 2 shows that there didn't detect ethanol in the combustion products of the paper, cotton, cloth except the wood which didn't fully burning. According to the test results that it's very important for fire investigation to pick up the wood which didn't fully burning from the fire scene .

3.4. Recovery test

Take three copies ethanol hexane solution of 0.2 ml at 25° C. Then dripping on the three copies wood of 1g. After 5min extract twice with distilled water of 10ml, then set the volume to 25ml. Then react with sodium nitrite solution of 1ml and hydrochloric acid solution to a pear-shaped funnel. Reacting for 15min . In order to reaction adequately need to fully shake the funnel until it completely. The washing 4 times , until the solution colorless. Then analysis the absorbance at 357 nm . Measured the recovery rate is 130%.

4. The future of UV-visible spectrophotometry in the fire investigation

Molecular absorption is the base of the UV-visible spectrophotometry with simple equipment, easy operation, wide application, high accuracy and precision. It has faster development in the analysis of organic. At present, it was used in the industry of medicine. With the development of discipline of fire investigation and reference from others disciplines, it will be wildly used in fire investigation as well as others disciplines.

In addition, it can combine with the Chemometrics to identify the reason of fire and calculate the content of combustion-supporting .What's more, the fire evidence identification technology is constantly improving, more and more advanced pre-treatment technology are used in fire investigation, such as derivatization reaction. This will enable the application of UV-visible spectrophotometry become more extensive in the fire investigation.

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