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Determination of Formaldehyde in Carbon-Coated Air Purifying Material by Ultraviolet Spectrophotometry

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Abstract. In this paper, a method for the determination of formaldehyde in carbon-coated air purifying material by ultraviolet spectrophotometry was established. The test determined the detection limit, lower limit of determination, precision and accuracy of the method. When the sampling quality is 2.0g, the detection limit of the method is 0.0412 μ g, the recovery is 98.4%~105.8%, and the precision RSD is 2.646% ~ 3.894%. The results show that this method is suitable for the determination of formaldehyde in carbon-coated air purifying material.

1. Introduction

Air purifier is a small guard of the domestic environment air. As a key material of the purifier, its own environmental protection performance and purification performance have attracted more and more attention from users. Formaldehyde and its derivatives, as common chemical raw materials, play an important role in the production of products. In this paper, the formaldehyde in carbon-coated air purification materials was tested by ultraviolet spectrophotometry, which provided a reference for the control and detection of formaldehyde in carbon-coated air purification materials.

2. Experiment

2.1. Materials and Instruments

2.1.1. Materials

Ammonium acetate, AR, Xilong Scientific Co., Ltd.; glacial acetic acid, AR, Xilong Scientific Co., Ltd.; formaldehyde standard solution (10.4mg/ml), Shanghai Anpu Experimental Technology Co., Ltd.; Acetylacetone, AR, Xilong Scientific Co., Ltd.; water, 18 M Ω .cm, laboratory-made.

2.1.2. Instruments

Water filter membrane (0.45 μ m), Tianjin jinteng experimental equipment Co., Ltd.; electronic balance ME203E/02, METTLER TOLEDO instrument (Shanghai) Co., Ltd.; UV-visible spectrophotometer UV-1780, shimadzu Japan; constant temperature water bath oscillator, Laizhou Yuanmao Instrument Co., Ltd.

2.2. Preparation of Acetylacetone Solution

Add 25g of ammonium acetate to a 100 ml volumetric flask, add a small amount of deionized water to dissolve, then add 3 ml of glacial acetic acid and 0.2 ml of acetylacetone, dilute to the mark with deionized water, store in a dark place with a brown bottle, and used after 12 hours, which can be stable for one month at 2~5 °C.



2.3. Experimental Method

Cut the sample into small pieces. Weigh the sample 2.0g, and put it into a 250ml tripod. Add 80 ml of deionized water into the tripod, and then oscillate it in 40°C water bath for 90 mins. Cool the filtrate temperature reaches room temperature. Transfer 10ml filtrate into a colorimetric tube of 25ml. Add 2ml Cool acetone, heat it in a boiling water bath for 3 min, cool it in dark condition, and make blank sample at the same time. The absorbance of each sample was measured at 412nm, and the formaldehyde concentration C was calculated. The content of free formaldehyde is calculated as follows:

$$\text{Formaldehyde extraction}(\mu\text{g/g}) = \frac{C \times V}{10 \times M} \quad (1)$$

C , formaldehyde concentration value calculated according to the standard curve of formaldehyde, μg ;

V , adding the volume of the extract, ml;

10 , conversion factor of solution volume of extraction liquid volume removed from the extraction solution, color reaction, ml;

M , the mass of the sample, g.

3. Results and Discussion

3.1. Standard Working Curve

Dilute 10.4mg/ml formaldehyde standard solution into a working solution of 1.04 mg/L. Transfer the working solution of 0, 2.0, 4.0, 6.0, 8.0, 10.0 ml to 25 ml colorimetric tube respectively. Add 2 ml acetylacetone solution to each bottle, shake well after constant volume, and cool it in boiling water bath for 3 minutes. The absorbance was measured at 412 nm wavelength. Standard curve was drawn with absorbance y as ordinate and formaldehyde concentration x (μg) as ordinate.

The standard curve equation of standard solution concentration and absorbance obtained by linear regression: $y=0.0200x-1.3810$, $R^2=0.9991$. Y is the absorbance; x is the formaldehyde concentration, as shown in figure 1.

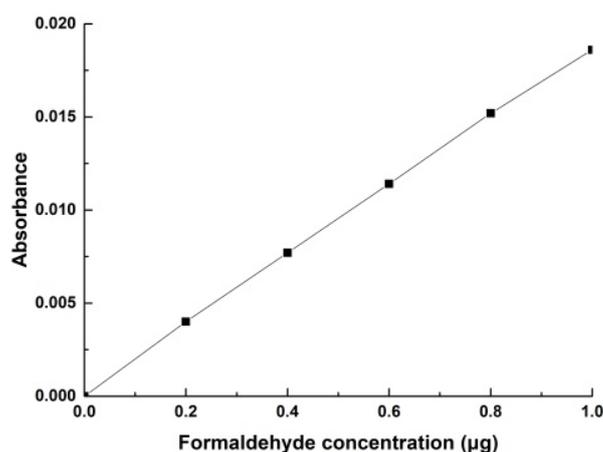


Figure 1. Standard working curve

3.2. Detection Limit

3.2.1 Instrument detection limit

The physical meaning of the instrument detection limit is the amount of the substance to be tested corresponding to the minimum detection signal that can be distinguished from the instrument noise

within a certain confidence range. By measuring 12 parts of a certain concentration of dilute solution, it can be calculated by the following formula:

$$D_L = kS \frac{C}{\bar{X}} \quad (2)$$

$$S = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N - 1}} \quad (3)$$

C , formaldehyde concentration value calculated according to the standard curve of formaldehyde, μg ;

D_L , instrument detection limit;

K , confidence factor, generally takes 3;

S , standard deviation of sample measurement readings;

\bar{X} , sample measurement reading average

The test was repeated 12 times with a standard working solution of $0.0400 \mu\text{g}$. The measurement results are shown in Table 1. The instrument detection limit was calculated according to the formula (2) and (3) to be $0.0164 \mu\text{g}$.

Table 1. Instrument detection limit

| | | | | | | |
|--|--------|--------|--------|--------|--------|--------|
| No | 1 | 2 | 3 | 4 | 5 | 6 |
| Absorbance(A , $\times 10^{-3}$) | 0.8 | 0.8 | 0.8 | 0.9 | 0.8 | 0.8 |
| Formaldehyde concentration (C , μg) | 0.0357 | 0.0333 | 0.0341 | 0.0283 | 0.0357 | 0.0391 |
| No | 7 | 8 | 9 | 10 | 11 | 12 |
| Absorbance(A , $\times 10^{-3}$) | 1.0 | 0.9 | 0.8 | 1.0 | 0.9 | 0.9 |
| Formaldehyde concentration (C , μg) | 0.0389 | 0.0309 | 0.0405 | 0.0341 | 0.0392 | 0.0405 |
| Average value(\bar{X} , μg) | 0.0359 | | | | | |

3.2.2 Method detection limit

According to all the steps of sample analysis, repeat the n (≥ 7) blank test, convert each measurement result into the concentration or content in the sample, calculate the standard deviation of the parallel measurement, and calculate the detection limit according to formula (4).

$$MDL = t_{(n-1,0.99)} \times S \quad (4)$$

MDL , method detection limit

n , parallel measurement times of samples

S , standard deviation of n parallel determinations

t , the t-distribution with a degree of freedom of $n - 1$ at the confidence of 99%, when $n=7$, the degree of freedom is 6, and the value of t can be taken 3.143.

The test was repeated 7 times with a standard working solution of $0.2000 \mu\text{g}$. The measurement results are shown in Table 2. According to the formula (4), when the sample amount is 2.0 g , the method detection limit is $0.0412 \mu\text{g}$.

Table 2. Method detection limit

| | | | | | | | |
|--|--------|--------|--------|--------|--------|--------|--------|
| No | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
| Absorbance(A , $\times 10^{-3}$) | 3.7 | 3.9 | 3.7 | 4.2 | 3.5 | 4.1 | 3.9 |
| Formaldehyde concentration (C , μg) | 0.1882 | 0.1989 | 0.1882 | 0.2151 | 0.1774 | 0.2097 | 0.1989 |
| Average value(\bar{X} , μg) | 0.1989 | | | | | | |

3.2.3 Minimum quantitative detection limit

The minimum quantitative detection limit means that the minimum concentration or content of the test substance can be accurately and quantitatively determined by a specific method under the premise that the limit error can satisfy the predetermined requirement, and the detection limit of 4 times is generally used as the minimum quantitative detection limit. Therefore, when the sample amount is 2.0 g, the lower limit of measurement is 0.1649 μg .

3.3. Accuracy and Precision

According to the conditions of the measurement method, three standard solutions of low, medium and high levels were added to the carbon-coated air purifying material samples, and six parallel determinations were performed at each level. The recovery and precision of the calculation method were as shown in Table 2. It can be seen that the recovery of formaldehyde is 98.4%~105.8%, and the precision RSD is 2.646%~3.894%. Therefore the accuracy and precision of the method are good.

Table 3. Accuracy and precision

| Levels | Formaldehyde extraction ($\mu\text{g/g}$) | Average value (\bar{X} , $\mu\text{g/g}$) | The amount of standard solution added ($\mu\text{g/g}$) | Recovery (%) | Standard deviation (SD_i $\mu\text{g/g}$) | Relative standard deviation (RSD_i , %) |
|--------|---|---|---|--------------|---|--|
| Sample | 0.954 | 0.956 | — | — | 0.007 | 0.706 |
| | 0.950 | | | | | |
| | 0.962 | | | | | |
| | 0.966 | | | | | |
| | 0.952 | | | | | |
| | 0.950 | | | | | |
| Low | 1.453 | 1.448 | 0.5 | 98.4 | 0.041 | 2.803 |
| | 1.446 | | | | | |
| | 1.388 | | | | | |
| | 1.420 | | | | | |
| | 1.503 | | | | | |
| | 1.478 | | | | | |
| Medium | 1.988 | 1.960 | 1.0 | 100.4 | 0.076 | 3.894 |
| | 2.002 | | | | | |
| | 1.838 | | | | | |
| | 2.035 | | | | | |
| | 1.894 | | | | | |
| | 2.002 | | | | | |
| High | 3.000 | 3.071 | 2.0 | 105.8 | 0.081 | 2.646 |
| | 2.986 | | | | | |
| | 3.183 | | | | | |
| | 3.050 | | | | | |
| | 3.051 | | | | | |
| | 3.158 | | | | | |

3.4. Actual Sample Testing

Three sets of carbon-coated air purifying material of different manufacturers were tested on the test method randomly selected in the market. A set of parallel tests was tested for each set of samples under this test condition.

Table 4. Actual sample detection value

| No | Formaldehyde extraction($\mu\text{g/g}$) | Average value ($\mu\text{g/g}$) | Measurement results($\mu\text{g/g}$) |
|------------|--|-----------------------------------|--|
| Sample 1-1 | 2.587 | 2.504 | 2.504 |
| Sample 1-2 | 2.421 | | |
| Sample 2-1 | 1.409 | 1.472 | 1.472 |
| Sample 2-2 | 1.534 | | |
| Sample 3-1 | 0.79 | 0.829 | 0.829 |
| Sample 3-2 | 0.867 | | |

4. Conclusion

In this paper, a method for the determination of formaldehyde in carbon-coated air purifying material by ultraviolet spectrophotometry was established. The detection limit, minimum quantitative detection limit, accuracy and precision were determined at the extraction solid-liquid ratio 1:40, extraction time 90 min, extraction temperature 40°C. The characteristics of the test method were studied, and the actual samples with different quality levels were determined. The test results show that the method has the advantages of fast pretreatment, simple operation and low test cost. It can be applied to the rapid screening of formaldehyde content in carbon-coated air purifying material, which is beneficial to the production of carbon-coated air purifying material.

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