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Methods for determination of formaldehyde in air of public places

公共场所空气中甲醛测定方法

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Foreword

This standard is developed to enforce the implementation of "P*ublic places Hygienic Management Regulations"*, GB 9663~9673-1996, and GB 16153-1996 "*Public Places Hygienic Standard";* and to strengthen the supervision and management of public place Hygiene. The methods in this standard are the monitoring and testing methods that match with GB 9663~9673-1996 and GB 16153-1996.

This first method of this standard is arbitration method.

This standard is released for first-time.

Annex A of this standard is normative.

Annex B of this standard is informative.

This standard was proposed by Ministry of Health of People's Republic of China.

Drafting organizations of this standard: Wuhan sanitary and anti-epidemic station, and Liaoning Providence sanitary and anti-epidemic station.

Main drafters of this standard: Zhang Qisheng, Wang Hanping (phenol reagent method), Jiang Shuqiu, Gao Wei, and Gao Guichun (gas chromatographic method).

Methods for determination of formaldehyde in air of public places

1. Scope

This standard specified the methods for determination of formaldehyde concentration in air of public places.

This standard applies to the determination of formaldehyde concentration in air of public places.

Method One Phenol reagent spectrophotometry

2. Principal

Formaldehyde in the air reacts with phenol reagent to generate triazine. Triazine is oxidized by ferric ion in an acidic solution to form blue-green compound. The colorimetric quantification shall be compared according to the color depth.

3. Reagents

The water used in this method is re-distilled water or deionized exchange water; the purity of agent used is usually analytically pure.

1.1 Original solution of absorbing solution: weight 0.10g of phenol reagent [$C_6H_4SN(CH_3)C$: NNH_2•HC1, abbreviated as MBTH]. Add water to dissolve it. Pour into 100 mL measuring cylinder with stopper. Add water to the mark. Store it in the refrigerator, and it can be kept stably for three days.

1.2 Absorbing solution: weight 5 ml of original absorbing solution. Add 95 ml of water. That is the absorbing solution. When sampling, prepare it when using.

1.3 1% ammonium iron sulfate solution: weight 1.0 g of ammonium iron sulfate [$NH_4Fe(SO_4)_2$•$12H_2O$]. Use 0.1 mol/L hydrochloric acid to dissolve. Dilute to 100 ml.

1.4 Iodine solution [$c(½\ I_2)$ = 0.1000 mol/L]: Weight 40 g of potassium iodide. Dissolve it in 25 ml of water. Add 12.7 g of iodine. After it has been dissolved completely, dilute it with water to 100mL. Then move it into a brown bottle; store it in dark place.

1.5 1 mol/L sodium hydroxide solution: weight 40 g of sodium hydroxide. Dissolve it in

A_0 - Absorbance of blank solution;

B_g - The calculation factor obtained in 6.1, the unit is μg/absorbance;

V_0 - The conversed sampling volume under standard condition, the unit is L.

8 Determination range, interference and troubleshooting

8.1 Determination range

Use 5 mL of sample solution; the determination range of this method is 0.1 -1.5 μg; when the volume of sample is 10 L, the determinable concentration range is 0.01 - 0.15 mg/m^3.

8.2 Sensitivity

The sensitivity of this method is 2.8 μg / absorbance.

8.3 Detection lower limit

This method detects 0.056 μg formaldehyde.

8.4 Interference and troubleshooting

20 μg of phenol, 2 μg of aldehyde, and nitrogen trichloride have no interference effect on this method. When they coexist with sulfur dioxide, the determination result will be lower. Therefore, the interference of sulfur dioxide shall not be ignored. It may use manganese sulfate filter-paper filter (see Annex B) to remove it.

8.5 Reproducibility: when the formaldehyde content is 0.5, 0.6 or 1.5μg/5 mL respectively, the re-determined variable coefficient is 5%, 5% or 3% correspondingly.

8.6 Recovery: when the formaldehyde content is 0.4 ~ 1.0 μg / 5 mL, the recovery of the sample and standard is 93%~101%.

Method Two Gas chromatographic method

9 Principles

Formaldehyde in the air is absorbed on the supporter coated with 2,4 dinitrophenyl hydrazine (2,4-DNPH) 6201; then generate stable formaldehyde hydrazone. After being eluted by carbon disulfide and separated by 0V- chromatographic column, use hydrogen flame ionization detector to determine; so as to retain the time qualitation and peak height quantification.

The detection lower limit is 0.2μg/mL (the eluant of injection sample is 5 μL).

L/min. after sampling, use plastic cap to cover it; record the temperature and atmospheric pressure at sampling point.

13 Analysis steps

13.1 Gas chromatography test conditions

When analyzing, it shall formulate the best test conditions to analyze formaldehyde according to the model and performance of gas chromatograph. The test conditions listed below is an example.

Chromatographic column: a glass tube with column length of 2 m and inner diameter of 3 mm; it is filled with 0V-1 + Shimalitew supporter.

Column temperature: 230°C.

Temperature at testing room: 260°C.

Temperature at vaporization chamber: 260°C.

Flow rate of carrier gas (N_2): 70 mL / min.

Oxygen flow rate: 40 mL / min.

Air flow rate: 450 mL / min.

13.2 Draw standard curve and determine correction factor

Draw standard curve and determine correction factor when determining samples.

13.2.1 Draw standard curve: take five sampling tubes; remove glass wool at one end of each tube. Add one drop (about 50 μL) of 20 mol/L hydrochloric acid solution on the surface of adsorbent directly. Add formaldehyde standard solution (1.00 mL contains 1 mg of formaldehyde) accurately by using micro syringe into each tube, so as to prepare such standard tubes that there are five concentration points – the formaldehyde content is within range 0-20 μg - on adsorbent in the tube; fill with glass wool; react for 10 minutes. Then transfer the adsorbent in each standard tube to five 5 mL color-comparison tubes. Add 1.0 mL of carbon disulfide in each tube. Shake it gently and soak it for 30 minutes. These are the elution standard series tubes of solution of formaldehyde. Then, take 5.0 μL of standard eluant with different concentration points; add into chromatographic column to get chromatographic peak and retention time. Repeat three times for each concentration points; determine the average value of peak. Take the concentration (μg/mL) of formaldehyde as abscissa, and take the average peak height (mm) as ordinate; calculate the slope of regression line. Take the slope value as the calculation factor B_s [μg / (mL•mm)] in determining samples.

13.2.2 Determine correction factor: Within the determination range, single-point

V_1 - The total volume of sample elution solution, the unit is mL;

E_s - The average elution efficiency determined experimentally, the unit is mL;

V_0 - The converted sample volume under the standard condition, the unit is L.

14.2 Calculate the concentration of formaldehyde in the air by using the single-point calibration method according to formula (6):

$$c = \frac{(h - h_0) \cdot f}{V_0 \cdot E_s} \cdot V_1 \qquad \cdots\cdots(6)$$

Where,

c - The concentration of formaldehyde in the air;

h - The average value of peak height of sample solution, the unit is mm;

h_0 - The average value of peak height of reagent blank solution, the unit is mm;

f - The calculation factor obtained by using single-point calibration method, the unit is μg/(mL • mm);

V_0 - The converted sample volume under the standard condition, the unit is L;

E_0 - The average elution efficiency determined experimentally, the unit is mL;

V_1 - The total volume of sample elution solution, the unit is mL.

Annex B

(Informative)

Preparation of manganese sulfate filter paper

Take 10 mL of manganese sulfate solution of which the concentration is 100 mg/L. Drop it onto a piece of 250cm^2 glass fiber filter paper. After the paper is air-dried, cut it into pieces. Put the pieces into a U-shaped glass tube with size of 1.5×150mm. Connect the tube in front of formaldehyde absorption tube. The manganese sulfate filer paper made according to this method can absorb sulfur dioxide. However, it is greatly affected by atmospheric humidity. When the relative humidity is greater than 88%, the gas production rate is 1 L/min, and the concentration of sulfur dioxide is 1 mg/m^3, it can eliminate more than 95% of sulfur; and the filter paper can be maintained to be effective for 50 hours. When the relative humidity is 15% ~ 35%, the ability of absorbing sulfur dioxide will be lowered gradually. Therefore, when the relative humidity is very low, it shall change to newly-made manganese sulfate filter paper.

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