HJ

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Ambient Air – Determination of Benzene and Its Analogies by Activated Charcoal Adsorption Carbon Disulfide Desorption and Gas Chromatography

环境空气 苯系物的测定 活性炭吸附/二硫化碳解吸—气相色谱法

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1 Scope of Application

This Standard specifies using the activated charcoal adsorption/carbon disulfide desorption-gas chromatography method for the determination of benzene and its analogies in the air.

This Standard is applicable to the determination of benzene, toluene, ethylbenzene, o-xylene, m-xylene, p-xylene, cumene and styrene in ambient air and indoor air. This standard is also applicable to the determination of benzene and its analogies in low-humidity exhaust gas at room temperature.

When the sampling volume is 10L, the method detection limits of benzene, toluene, ethylbenzene, o-xylene, m-xylene, p-xylene, cumene and styrene are all 1.5×10^{-3} mg/m^3, and the lower limit of determination is 6.0×10^{-3} mg/m^3.

2 Method Principle

Use the activated charcoal sampling tube to enrich the benzene and its analogies in ambient air and indoor air; carbon disulfide (CS_2) is desorbed; and use the gas chromatograph with hydrogen flame ionization detector (FID) for determination and analysis.

3 Interference and Cancellation

The main interference comes from the impurities of carbon disulfide. Carbon disulfide shall be identified by gas chromatograph for the presence of interference peaks before use. If there are interference peaks, carbon disulfide shall be purified. See Appendix A for the purification method.

6.3 Storage of samples

After collecting the sample, immediately seal the two ends of the activated charcoal sampling tube with a Teflon cap; and store it in a tightly sealed manner in the dark; and measure it within 8h at room temperature. Otherwise, put it in an airtight container and store in a refrigerator at −20°C; the shelf life is 1d.

6.4 Desorption of the sample

Take out Section-A and Section-B of the activated charcoal sampling tube and put them into the ground test tube with stopper. Add 1.00mL of carbon disulfide (4.1) to each test tube and seal it; shake it gently; and desorb at room temperature for 1h before testing.

7 Analysis Procedures

7.1 Recommended analysis conditions

7.1.1 Reference conditions of packed column gas chromatography

Carrier gas flow rate: 50mL/min; inlet temperature: 150°C; detector temperature: 150°C; column temperature: 65°C; hydrogen flow rate: 40mL/min; air flow rate: 400 mL/min.

7.1.2 Reference conditions for capillary column gas chromatography

Column and oven temperature: keep at 65°C for 10min; heating at a rate of 5°C/min to 90°C, and keep it for 2min. Column flow rate: 2.6mL/min; inlet temperature: 150°C; detector temperature: 250°C; makeup gas flow rate: 30mL/min; hydrogen flow rate: 40 mL/min; air flow rate: 400mL/min.

7.2 Calibration

7.2.1 Drawing of calibration curve

Take an appropriate amount of standard stock solution (4.2) and dilute it into 1.00mL of carbon disulfide (4.1) to prepare calibration series with mass concentrations of 0.5, 1.0, 10, 20, and 50μg/mL. Take 1.0μL of the standard series solution and inject it into the injection port of gas chromatograph. Draw a calibration curve based on the mass and response value of each target component.

7.2.2 Standard chromatogram

7.2.2.1 Refer to the chromatogram for the capillary column, see Figure 2.

Calculate the target component content according to the calibration curve.

7.4 Blank test

The on-site blank activated charcoal tube is measured in the same batch as the sample tube that has been sampled, and the analysis procedure is the same as the measurement (7.3).

8 Calculation and Representation of Results

8.1 The concentration of the target compound in the gas is calculated according to Formula (1).

$$\rho = \frac{(W - W_0) \times V}{V_{\mathrm{nd}}} \qquad (1)$$

Where:

ρ – mass concentration of tested components in the gas, in mg/m^3;

W - mass concentration of the sample desorption solution calculated from the calibration curve, in μg/mL;

W_0 - mass concentration of the blank desorption solution calculated from the calibration curve, in μg/mL;

V – volume of desorption solution, in mL;

V_{nd} – sampling volume in the standard state (101.325kPa, 273.15K), in L.

8.2 Representation of results

When the measurement result is less than 0.1mg/m^3, it is retained to four digits after the decimal point; when it is greater than or equal to 0.1mg/m^3, three significant digits shall be retained.

9 Precision and Accuracy

9.1 Precision

Capillary column gas chromatography: Five laboratories respectively measure uniform samples with a content of 0.5μg and 50.0μg. The relative standard deviations in the laboratory range from 1.1% to 2.6% and 1.1% to 2.5%, respectively. The relative standard deviations among the laboratories range from 0.2% to 1.0%, and 0.1% to

1.0%; the repeatability limits range from 0.01 to 0.03μg, and 1.95 to 3.27μg; and the reproducibility limits range from 0.02 to 0.04μg, and 1.95 to 3.32μg. See Appendix C for detailed parameters.

Packed column gas chromatography: Five laboratories respectively measured uniform samples with a content of 0.5μg and 50.0μg. The relative standard deviations in the laboratory range from 1.1% to 2.6% and 1.1% to 3.7%, respectively. The relative standard deviations among the laboratories range from 0.1% to 0.7%, and 0.3% to 1.0%; the repeatability limits range from 0.02 to 0.03μg, and 2.10 to 3.06μg; and the reproducibility limits range from 0.02 to 0.03μg, and 2.12 to 3.06μg. See Appendix C for detailed parameters.

9.2 Accuracy

Five laboratories measure two concentrations of standard samples. The spiked amount of each component is 100μg. The final relative error value of capillary column gas chromatography ranges from −2.6% to 11.6%. The final spiked recovery rate value ranges from 92.2% to 105%; the final relative error value of the packed column gas chromatography ranges from −1.2% to 6.0%; and the final spiked recovery rate value ranges from 92.9% to 104%. See Appendix C for detailed parameters.

10 Quality Assurance and Quality Control

10.1 When the water vapor or water mist in the air is too large to condense in the activated charcoal tube, it shall influence the penetration volume and sampling efficiency of the activated charcoal tube, and the air humidity shall be less than 90%.

10.2 The relative deviation of the flow rate before and after sampling shall be within 10%.

10.3 The adsorption efficiency of the activated charcoal sampling tube shall be above 80%, that is, the components collected by the activated charcoal in the Section-B shall be less than 25% of the Section-A; otherwise, the flow rate or sampling time shall be adjusted, and the sampling shall be repeated. Calculate the adsorption efficiency (%) of activated charcoal tube according to Formula (2):

$$K=\frac{M_1}{M_1+M_2}\times 100 \qquad (2)$$

Where:

K – absorption efficiency of sampling, in %;

M_1 – the sampling amount in Section-A, in ng;

------ The End ------