

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Stationary source emission. Determination of acrylonitrile. Gas chromatography

固定污染源排气中丙烯腈的测定 气相色谱法

(English Translation)

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1.1

This standard is applicable to the determination of acrylonitrile for both intentional and fugitive emissions from stationary pollution sources.

1.2

When the sampling volume is 30L, the detection limit of the method is 0.2 mg/m³.

The quantitative measurement concentration range of the method is 0.26~33.0mg/m³.

2 Method Principle

Acrylonitrile (CH₂=CHCH₂CN) is adsorbed and enriched by activated charcoal at room temperature; and then desorbed by carbon disulfide at room temperature. The components in the desorbed liquid are separated by the chromatographic column and then enter the hydrogen flame ionization detector (FID). From the measured acrylonitrile chromatogram peak height (or area), quantify the concentration of acrylonitrile in the desorption solution; and finally calculate the concentration of acrylonitrile in the gas sample from the volume, concentration, and sampling volume of the desorption solution.

3 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard.

GB 16297-1996 Comprehensive Emission Standard of Air Pollutants

GB 16157-1996 The Determination of Particulates and Sampling Methods of Gaseous Pollutants Emitted from Exhaust Gas of Stationary Source

4.1 Acrylonitrile

Chromatographically pure (or analytically pure, but there must be no chromatographic interference peaks for acrylonitrile).

and dilute to the mark with carbon disulfide.

5.1 Gas chromatograph

Equipped with hydrogen flame ionization detector

5.2.1 Column material

Glass or PTFE.

5.2.2 Column length

3m.

5.2.3 Column inner diameter

3mm.

5.2.4 Column type

packed column.

5.2.5 In-column packing

GDX-502, 60~80mesh.

5.2.6

Preparation and aging of chromatographic column (see Appendix B).

5.3 Micro syringe

1.0μL

5.4 Sampling instrument

Refer to 9.3 in GB 16157-1996 to configure the sampling instrument.

5.4.1.1 Sampling tube

It is made of stainless steel, hard glass or PTFE, with appropriate dimensions, and is equipped with an insulation jacket that can be heated to above 120°C.

5.4.1.2 Sample collecting device

Activated charcoal adsorption tube (4.7).

5.4.1.3 Flowrate measuring device

See 9.3.6 of GB 16157-1996.

5.4.1.4 Suction pump

See 9.3.7 of GB 16157-1996.

5.4.1.5 Connecting pipe

PTFE hose or silicone rubber tube lined with PTFE film.

1-flue; 2-heating sampler; 3-bypass adsorption tube; 4-thermometer; 5-pressure gauge; 6-adsorption tube; 7-three-way valve; 8-dryer; 9-flow meter; 10- suction pump.

Figure 2 - Flue Gas Sampling System

6.1.3 Sample collection

Plug an appropriate amount of glass wool into the port of the sampling tube; then extend it to the sampling point in the exhaust cylinder; start the suction pump; and first make the gas in the exhaust cylinder circulate flow through the bypass adsorption tube to fully wash the sampling pipeline. Then let the exhaust gas pass through the adsorption tube; and record the sampling time, temperature, and flow rate. After sampling, the adsorption tube shall be removed immediately; and its two ends shall be tightly covered by plastic caps; and then take back to the laboratory for analysis.

6.1.4 Heating temperature control of sampling tube

The heating temperature of the sampling tube shall be based on the principle that water vapor and the sample do not condense on the wall of the sampling tube; but the maximum heating temperature shall not exceed 160°C. If the exhaust gas temperature is close to room temperature, the sampling tube is exempted from being heated.

6.1.5 Sampling flowrate

The sampling flowrate shall generally be controlled between 0.3L/min and 1.0L/min.

When the temperature is higher than 30°C, the sampling flow rate shall be reduced to no greater than 0.5L/min; so that ensure that the adsorption capacity of the activated charcoal in the Section-B is less than 2% of the total adsorption capacity.

6.1.6 Sampling amount

For each activated charcoal adsorption tube, the sampling amount shall be controlled to the concentration of acrylonitrile in the carbon disulfide desorption solution at

10~400µg/mL. The maximum sampling amount of each activated charcoal adsorption tube shall generally not exceed 1.6mg; and the acrylonitrile adsorbed by the activated charcoal in Section-B shall not exceed 2% of the total adsorbed amount of acrylonitrile.

6.2.1 Sampling location and sampling point

Determine the location of the fugitive emission monitoring point according to the provisions of Appendix C in GB 16297-1996; or determine the location of the sampling point according to other specific requirements.

6.2.2 Connection of sampling device

The solutions of each concentration are injected for three times; take the average value; and draw the calibration curve by the concentration c_i (µg/ml) and the corresponding peak height h_i (or area A_i); and calculate the linear regression equation of the calibration curve.

7.3.1 Desorption of the sample

Open the caps at both ends of the activated charcoal adsorption tube after collecting the sample; use a hooked iron wire to take out the V-shaped spring hook pressed on the activated charcoal particles in Section-A; and respectively transfer the activated charcoal in Section-A (including the upper end of glass wool) and the activated charcoal in Section-B (including sponge) to two dry test tubes with grinding mouths;

and immediately used a pipette to pipette 2.00mL and 1.00mL of carbon disulfide; and