YC

TOBACCO INDUSTRY STANDARD OF

THE PEOPLE'S REPUBLIC OF CHINA

ICS 65.160

X 85

Record number: 30430-2011

YC/T 373-2010

Determination of benzene, toluene, ethylbenzene and xylene in polypropylene fiber tow and polypropylene fiber tow filter rod - Gas chromatography-mass spectrometry method

丙纤丝束及丙纤滤棒 苯、甲苯、乙苯和二甲苯残留量的测定 气相色谱-质谱联用法

Issued on: December 01, 2010 **Implemented on: January 01, 2011**

Issued by: State Tobacco Monopoly Administration

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1 Scope

This Standard specifies the determination method of benzene, toluene, ethylbenzene and xylene in polypropylene fiber tow and polypropylene fiber tow filter rod -- Gas chromatography-mass spectrometry method.

This Standard applies to the determination of benzene, toluene, ethylbenzene and xylene in polypropylene fiber tow and polypropylene fiber tow filter rod.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 15270-2001, Tobacco and tobacco products - Polypropylene fiber tow filter rod

YC/T 27-2002, Polypropylene fiber tow for cigarette

3 Principle

Use the n-pentane solution that contains internal standard substance to extract benzene, toluene, ethylbenzene and xylene in the sample; use the gas chromatography-mass spectrometry method to determine the content of benzene, toluene, ethylbenzene and xylene in the extract; then, calculate the residual amount of benzene, toluene, ethylbenzene, and xylene in the sample.

4 Reagents

Except for special requirements, analytical reagents shall be used.

4.1 Reagents

4.1.1 N-hexane (C_6H_{14}), Super Pure grade or pesticide residue grade.

4.1.2 N-pentane (C_5H_{12}), chromatographic grade.

4.1.3 Benzene (C_6H_6), purity ≥99%.

4.1.4 Toluene (C_7H_8), purity ≥99%.

4.1.5 Ethylbenzene (C_8H_{10}), purity ≥99%.

4.1.6 O-xylene (o-C_8H_{10}), purity ≥99%.

4.1.7 M-xylene (m-C_8H_{10}), purity ≥99%.

4.1.8 P-xylene (p-C_8H_{10}), purity ≥99%.

4.1.9 2-hexanone (internal standard), if other substances are used as internal standard, the applicability shall be verified.

4.2 Preparation of internal standard solution

4.2.1 Internal standard stock solution: weigh 0.1 g of 2-hexanone (4.1.9) (accurate to 0.1 mg) in a 50 mL volumetric flask; use n-hexane (4.1.1) to dilute to the mark; mix well, to obtain an internal standard stock solution whose concentration is 2 mg/mL. Seal and store the internal standard stock solution in a refrigerator at 0 °C ~ 4 °C; the validity period is 3 months.

4.2.2 Internal standard solution: accurately pipette 2.5 mL of internal standard stock solution (4.2.1) into a 50 mL volumetric flask; use n-hexane (4.1.1) to dilute to the mark; mix well to obtain an internal standard solution whose concentration is 100 μg/mL. Seal and store the internal standard solution in a refrigerator at 0 °C ~ 4 °C; the validity period is 1 month.

4.3 Preparation of standard solution

4.3.1 Mixed standard stock solution: respectively weigh 0.1 g of benzene (4.1.3), toluene (4.1.4), ethylbenzene (4.1.5), o-xylene (4.1.6), m-xylene (4.1.7), p-xylene (4.1.8) (accurate to 0.1 mg) in the 100 mL volumetric flasks; use n-hexane (4.1.1) to dissolve them and dilute to the mark; mix well, to obtain the mixed standard stock solution, of which the concentration of each component is 1 mg/mL, respectively. Seal and store the mixed standard stock solution in a refrigerator at 0 °C ~ 4 °C; the validity period is 3 months.

4.3.2 Mixed standard solution: use n-hexane (4.1.1) to gradually dilute the mixed standard stock solution (4.3.1) to obtain a mixed standard solution whose concentration is 5 μg/mL. Formulate this solution when needed.

Perform selective ion scanning on the standard working solution; calculate the peak area ratio of benzene, toluene, ethylbenzene, and xylene in each standard solution to the internal standard 2-hexanone; draw the standard working curve of concentration-to-peak area ratio of benzene, toluene, ethylbenzene and xylene. The standard working curve shall be straight line relationship; the correlation coefficient shall be R^2>0.99.

A standard working curve shall be made for each test. After every 20 samples, add a medium-concentration standard working solution. If the measured value differs from the original value by more than 10%, the standard working curve shall be made again.

6.5 Sample determination

Measure the sample solution (6.2) according to the instrument test conditions; repeat the measurement twice for each sample. At the same time, make a set of blanks for each batch of samples.

7 Calculation and expression of results

Calculate the residual amount (X) of benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene in the sample according to Formula (1):

$$X = \frac{50(c - c_0)}{m} \qquad \cdots\cdots(1)$$

Where:

X -- the residual amount of benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene in the sample, in milligrams per kilogram (mg/kg);

c -- the concentration of benzene, toluene, ethylbenzene, p-xylene, meta-xylene and o-xylene in the sample solution that is read from the standard working curve, in micrograms per milliliter (μg/mL);

c_0 -- the concentration of benzene, toluene, ethylbenzene, p-xylene, m-xylene and o-xylene in the blank test that is read from the standard working curve, in micrograms per milliliter (μg/mL);

m -- sample mass, in grams (g).

The final residual amount of xylene in the sample is the sum of the residual amounts of p-xylene, m-xylene and o-xylene.

Take the average of two parallel determinations as the final determination result, accurate to 0.01 mg/kg.

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