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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YC/T 146-2010

**Leaf tobacco - Threshing and redrying - Technical specification
(English Translation)**

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Foreword

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard was proposed by State Tobacco Monopoly Bureau.

This Standard shall be under the jurisdiction of National Technical Committee on Tobacco of Standardization Administration of China (SAC/TC 144/SC 8).

The drafting organizations of this Standard. China Research Centre for Tobacco, China Tobacco Hunan Industrial Co., Ltd., Shanghai Tobacco (Group) Company, Hongyun Honghe Tobacco (Group) Co., Ltd., and National Tobacco Quality Supervision and Inspection Center.

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Determination of formaldehyde in water-borne

adhesives for cigarette -

High performance liquid chromatographic method

1 Scope

This Standard specifies the determination of formaldehyde in water-borne adhesives for cigarette - High performance liquid chromatographic method.

This Standard is applicable to the determination of formaldehyde in water-borne adhesives for cigarette.

2 Normative references

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

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696.1987, MOD)

3 Principle

Use water to dilute the formaldehyde in the extracted sample. After formaldehyde hydrazine is derived from 2,4-dinitrophenylhydrazine, use HPLC (high performance liquid chromatograph) / diode array detector to determine, and quantify by external-standard method.

4 Reagents

In addition to special requirements, all reagents are analytical pure. The water shall comply with the requirements for Grade-1 in GB/T 6682.

4.1.1 Acetonitrile (chromatographic pure).

If the concentration of sample solution for test exceeds the concentration range of standard working curve, make appropriate adjustment to pretreatment of sample before re-determining it.

6.2 Blank test

Do not add sample. Repeat 6.1 and conduct HPLC analysis.

6.3.6 Gradient elution procedure. shown in Table 2.

Table 2 Gradient elution procedure

Time

min

Mobile phrase A

Mobile phrase B

0.00 70 30

5.00 10 90

15.00 10 90

16.00 70 30

20.00 70 30

6.4 Drawing of standard working curve

Respectively take standard solutions (4.2) to conduct HPLC analysis. The standard working curve of formaldehyde shall be based on the standard solution concentration and formaldehyde response peak area. The linear correlation coefficient of working curve is $R^2 > 0.99$.

Standard curve shall be made for each test. After determination of every 20 samples, it shall add a standard solution of medium solution. If the difference between the determined value and the original value exceeds 3%, it shall re-draw the standard curve.

6.5 Sample determination

Determine the sample (6.1) based on apparatus' conditions. Determine the nature according to retention time. Use external-standard method to quantify. Repeat the determination twice for each sample. Conduct a group of blank test for each batch of sample. Refer to Appendix A for chromatogram of standard

solution and sample derived solution.

7 Results calculation and representation

The content of formaldehyde in the sample shall be calculated according to equation (1).

Where,

X - Content of formaldehyde in the sample, in milligrams per kilogram (mg/kg);

c - Formaldehyde concentration obtained by standard curve, in milligrams per liter (mg/L);

c₀ - Blank value obtained by standard curve, in milligrams per liter (mg/L);

V - Volume of extracted solution, in milliliters (mL);

m - Sample material mass, in grams (g);

S - Dilution factor of sample solution.

Take the average of two parallel determinations as the final result, accurate to

0.1 mg/kg.

Relative mean deviation of two parallel determinations shall be less than 10%.

8 Recovery rate, detection limit and quantitation limit

The recovery rate, detection limit and quantitation limit of this method are shown in Table 3.

Table 3 Recovery rate, detection limit and quantitation limit of this method

Compound Recovery rate %

Detection limit

mg/kg

Quantitation limit

mg/kg

Formaldehyde 91.0~105.7 1.6 5.3