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

SN/T 5271-2019

**Rubber and rubber products Fluorine rubber
Thermogravimetric analysis for the determination of vulcanized
and unvulcanized rubber**

橡胶和橡胶制品 氟橡胶 热重分析法测定硫化和未硫化胶的成分

(English Translation)

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Foreword

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

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Ningbo Customs of the People's Republic of China.

1 Scope

This standard specifies a method for the determination of total rubber hydrocarbon content in fluororubber using thermogravimetric analysis.

This standard applies to the determination of total rubber hydrocarbon content in fluororubber.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6379.2 Measurement method and accuracy of results (accuracy and precision) GB/T 8170 Numerical rounding rules and the expression and determination of limit values ISO 1407 Determination of rubber solvent extractables

3 Method summary

A weighed sample is measured according to a preset temperature program. In a nitrogen atmosphere, the sample is first subjected to high temperature decomposition.

It is then oxidized in an oxygen atmosphere. The total rubber hydrocarbon content in fluorine rubber can be calculated by the mass change.

4 Reagents and materials

4.1

Nitrogen. purity $\geq 99.999\%$.

4.2

Oxygen. purity $\geq 99.999\%$.

5 Apparatus and equipment

5.1

Thermogravimetric analyzer. with platinum crucible, in which the balance is 0.001 mg.

5.2

Analytical balance. Sensitivity 0.1 mg.

6 Analysis steps

6.1

Turn on the thermogravimetric analyzer (5.1) and set the initial temperature of the heating furnace to 35 °C.

6.2

Cut the sample to approximately 1 mm x 1 mm.

6.3

Weigh a sample of 5 mg~8 mg, place it in a platinum crucible, and place it on the sample holder of the thermogravimetric analyzer (5.1), and read the quality of the sample.

Quantity (accurate to 0.001 mg).

Note: For some types of instruments, you can set the recorder to 100% after placing the sample. At this time, it is not necessary to pre-weigh accurately.

6.4

Close the heating furnace and purify the system with nitrogen gas (4.1) at a constant rate to ensure that the air in the furnace body is purged clean.

Note: The flow rate and purging time of nitrogen (4.1) are related to the model of the instrument, and can be set by referring to the instrument manual.

6.5

Under a nitrogen (4.1) atmosphere, raise the furnace temperature to 800 °C at a rate of 20 °C/min, and keep the temperature constant for 5 min.

6.6

Under nitrogen (4.1) atmosphere, reduce the furnace temperature from 800 °C to 400 °C; then switch the atmosphere from nitrogen (4.1) to oxygen (4.2), and keep constant temperature for 2 min.

Note: The flow rate of oxygen (4.2) should be consistent with the flow rate of nitrogen (4.2) in 6.4 to ensure that the total gas flow rate remains unchanged after the atmosphere is switched.

6.7

Under an oxygen (4.2) atmosphere, raise the furnace temperature to 800 °C at a rate of 20 °C/min, and keep the temperature constant for 10 min.

6.8

After the heating furnace has cooled to room temperature, open the furnace body, carefully take out the platinum crucible and clean it, and then end the test.

7.1 Record

Record the graphs of the two situations (see Figure 1) to perform the necessary calculations. These graphs are used to obtain the composition of various compounds. content.

Note: The differential curve provided by the thermogravimetric analyzer varies from manufacturer to manufacturer. You can also use the mass loss percentage vs. temperature curve and Differential graph of mass loss percentage versus temperature to obtain the content of various components.

Description.

Solid line-the graph of mass loss percentage and time;

Dotted line-differential graph of mass loss percentage versus time.

Figure 1 Schematic diagram of thermal weight loss

7.2 Total organic content

The first peak on the differential curve (point A) corresponds to the maximum pyrolysis rate in nitrogen (4.1), the mass loss of this part (w_1) it is the thermal decomposition of the sample before 800 °C under nitrogen (4.1) atmosphere, that is, the total organic content.

7.3 Carbon residue content

Fluorine rubber will not be completely pyrolyzed in nitrogen (4.1), leaving a small amount of carbonaceous residue. In order to burn off the carbonaceous residues, heat the furnace is cooled to 400 °C and the atmosphere is switched from nitrogen (4.1) to oxygen (4.2), then the furnace is heated to 800 °C and maintained until the quality is reached no more changes. The second peak (point B) on the differential curve corresponds to the combustion of carbonaceous residues in oxygen (4.2). The quality of this part the quantity loss (w_2) is the carbonaceous residue content.

7.4 Carbon black content

After all the carbonaceous residues are volatilized, the mass loss percentage when heated to 800 °C in oxygen (4.2) is the carbon black in the sample content. The third peak on the differential curve (point C) corresponds to the combustion of carbon black in oxygen (4.2), the mass loss of this part (w_3) that is the content of carbon black.

7.5 Ash

When the temperature is maintained at 800 °C and the mass is no longer changed, the residual mass (w_4) at this time is the ash content. Ash includes Inorganic fillers and ash content of various inorganic and organic components that are pyrolyzed at lower temperatures.

7.6 Total rubber hydrocarbon content

Assuming that all non-rubber organic components can be extracted, the mass fraction of solvent extracts in rubber measured according to ISO 1407 is w_5 , Then the total rubber hydrocarbon content is calculated according to formula (1).