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Rubber compounding ingredients — Carbon black — Determination of high-temperature loss on heating by thermogravimetry

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la perte à la chaleur à haute température par thermogravimétrie

Reference number
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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 21870 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

Rubber compounding ingredients — Carbon black — Determination of high-temperature loss on heating by thermogravimetry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard describes a method for quantitative determination of the loss on heating (pyrolysis) of carbon black due to the elimination of volatile materials other than those liberated when heating to 125 °C (ISO 1126). The method is applicable to carbon black for the rubber industry.

It serves to assess the decomposition of functional groups and adsorbed substances (e.g. hydrocarbons) present on the surface of the carbon black.

2 Principle

A known mass of carbon black is placed in the cradle of a thermobalance and heated according to a predetermined temperature programme in a controlled, oxygen-free atmosphere. The mass variation recorded provides a thermogram which can be used for quantitative evaluation.

3 Apparatus

There are several thermogravimetric measurement instruments available commercially.

The basic components are:

3.1 Thermobalance, with a sensitivity of one microgram and equipped with an appropriate cradle made of a non-oxidizing material.

3.2 Furnace, allowing temperature regulation from the ambient temperature up to approximately 1 000 °C.

3.3 Temperature programmer, allowing a heating rate of up to 50 °C/min.

3.4 Nitrogen gas distributor, associated with flow-control equipment allowing measurements in a range from 10 cm^3/min to 250 cm^3/min.

3.5 Data acquisition and processing system.

4 Materials

4.1 Cylinder of compressed nitrogen containing less than 10 ppm of oxygen.

5 Procedure

5.1 Power up the various elements of the analyser and preheat the furnace to 40 °C in accordance with the manufacturer's instructions.

5.2 Close the furnace of the thermobalance, purge with a constant, predefined flow of nitrogen (usually 100 cm^3/min) in accordance with the manufacturers' instructions and maintain a constant flow for the duration of the test.

5.3 Wait for the thermobalance to stabilize.

5.4 Place the test sample in the cradle and weigh it to the nearest 0,01 mg. The sample mass should not be less than 5 mg.

5.5 Start the test.

5.6 Heat the furnace to 125 °C at a rate of 20 °C/min.

5.7 Maintain the temperature at 125 °C for 15 min and record the mass.

5.8 Heat the furnace from 125 °C to 950 °C at a rate of 20 °C/min. Due to instrumental limitations, the user may be obliged to run the test at a lower maximum temperature, e.g. at 800 °C. These conditions can lead to slightly lower test results for the loss on pyrolysis and shall therefore be mentioned in the test report.

5.9 Maintain the furnace at a temperature of 950 °C until the mass remains constant. A mass loss lower than 0,05 %, relative to the initial mass, during 15 min is considered as "constant mass".

6 Expression of results

Express the results as percentages of the initial mass:

$$V = \frac{m_{40} - m_{125}}{m_{40}} \times 100$$

$$P = \frac{m_{125} - m_{950}}{m_{40}} \times 100$$

where

V is the volatile matter at 125 °C (%) in nitrogen;

P is the loss on pyrolysis at 950 °C (%) in nitrogen;

m_{40} is the initial mass at 40 °C (g);

m_{125} is the mass at 125 °C (g);

m_{950} is the mass at 950 °C (g).

7 Test report

The test report shall contain the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this International Standard (ISO 21870:2005);

c) the type of apparatus used;

d) the loss on heating to 125 °C (volatile matter), to the nearest 0,1 %;

e) the loss on further pyrolysis (125 °C to 950 °C), to the nearest 0,1 %;

f) any deviations from the procedure specified, e.g. a maximum temperature lower than 950 °C;

g) any unusual features (anomalies) observed during the test;

h) the date of the test.

Bibliography

[1] ISO 1126, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*