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Rubber compounding ingredients — Carbon black — Determination of loss on heating

[Revision of third edition (ISO 1126:1992)]

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la perte à la chaleur

ICS 83.040.20

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Foreword

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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.

International Standard ISO 1126 was prepared by the Technical Committee ISO/TC 45, Rubber and rubber products, Sub-committee SC3, *Raw materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 1126:1992).

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Rubber compounding ingredients — Carbon black — Determination of loss on heating

Warning – Persons using this 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 specifies methods for determining loss on heating of carbon black for use in the rubber industry. This loss on heating is due primarily to loss of moisture, but traces of other volatile materials may also be lost.

These methods are not applicable to treated carbon blacks which contain added volatile materials.

One of the following three methods shall be adopted.

(1) Method 1 - Convection –Gravity Oven Method

(2) Method 2 - Moisture Balance Method

(3) Method 3 – Infrared Irradiation Method (Rapid Method)

2 Method 1: Convection - Gravity Oven Method

2.1 Principle

A test portion of carbon black is heated for 1 h at a temperature 125 °C in a weighing bottle. The weighing bottle plus contents is allowed to cool in a desiccator to room temperature and weighed, and the percentage loss on heating calculated.

2.2 Apparatus

2.2.1 Oven, gravity-convection type, capable of maintaining a temperature of 125 °C ± 2 °C.

2.2.2 Weighing bottle, squat-form 30mm in height and 60mm in diameter, fitted with a ground-glass stopper.

When larger samples are required for other tests, use an open vessel of dimensions such that the depth of the black is not greater than 10 mm during conditioning.

2.2.3 Analytical balance, accurate to ±0,1 mg.

2.2.4 Desiccator

2.3 Procedure

2.3.1 Precautions

2.3.1.1 Take the sample of carbon black in a tightly stoppered glass bottle or friction-top can. Allow the closed container to reach ambient temperature before starting the test.

2.3.1.2 Keep the weighing bottle stoppered when transferring to and from the desiccator, to prevent loss of carbon black due to air currents.

2.3.2 Determination

2.3.2.1 Dry the weighing bottle (2.2.2) and the stopper, with the stopper removed, in the oven (2.2.1) at a temperature of $125\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 30 min. Place the bottle and the stopper in the desiccator (2.2.4) and allow to cool to ambient temperature. Weigh the bottle with stopper to the nearest 0,1 mg.

2.3.2.2 Weigh about 2 g of carbon black to the nearest 0,1 mg into the weighing bottle.

2.3.2.3 Place the weighing bottle, test portion and stopper in the oven for 1 h at a temperature of $125\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, with the stopper removed.

2.3.2.4 Replace the stopper and transfer the bottle and contents to the desiccator. Remove the stopper and allow to cool to ambient temperature. Replace the stopper on the weighing bottle and reweigh to the nearest 0,1 mg.

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2.4 Expression of results

Calculate the loss on heating, expressed as a percentage by mass, using the formula

$$H = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

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where:

- H is the heating loss, %
- m_0 is the mass, in grams, of the weighing bottle and stopper;
- m_1 is the mass, in grams, of the weighing bottle, stopper and test portion before heating;
- m_2 is the mass, in grams, of the weighing bottle, stopper and test portion after heating;

2.5 Test report

The test report shall include the following particulars:

- a) a reference to this International Standard;
- b) method adopted for test (record that the method convection-gravity oven was adopted);
- c) all details necessary to identify the sample;
- d) the results, and the units in which they have been expressed;
- e) any unusual features noted during the determination;
- f) any operation not included in this International Standard or regarded as optional.

3 Method 2: Moisture Balance Method

3.1 Principle

A carbon black sample is heated at a temperature which should not exceed 125 °C, and its quantitative decrease in mass is measured using a moisture balance.

3.2 Apparatus

3.2.1 Moisture balance, having a sensitivity of 0,1 mg and an indirect heating source.

3.3 Procedure

3.3.1 Set up the moisture balance according to the manufacturer's instructions. The temperature should not exceed 125 °C.

3.3.2 Place approximately 2 g of carbon black into the moisture balance and determine its mass to the nearest 0,1 mg.

3.3.3 Close the lid and start the machine.

3.3.4 Once the mass loss, under these drying conditions, is less than 1 mg over 30 s the test portion is considered dry and the percent mass loss should be recorded to the nearest 0,1%.

3.4 Calculation

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Calculate the percent heating loss to the nearest 0,1 % as follows.

$$H = \frac{A - B}{A} \times 100$$

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where:

- H is the heating loss, %;
- A is the mass of test portion before heating, g;
- B is the mass of test portion after heating, g.

3.5 Test report

The test report shall include the following particulars:

- a) a reference to this International Standard;
- b) method adopted for the test (record that the moisture balance method was adopted);
- c) all details necessary to identify the sample;
- d) the results, and the units in which they have been expressed;
- e) any unusual features noted during the determination;
- f) any operation not included in this International Standard or regarded as optional.

4 Method 3: Infrared Irradiation Method (Rapid Method)

4.1 Principle

A carbon black sample is heated by infrared irradiation using an infrared lamp and the loss on heating is measured as the subsequent decrease in mass using an infrared moisture meter.

4.2 Apparatus

4.2.1 Infrared moisture meter, having a sensitivity of 1 mg.

NOTE 1 The moisture meter shall be either a distance adjusting-type meter that runs on 100 V ~ 240 V and uses a 185 W infrared lamp, or a voltage adjusting-type meter that runs on 100 V ~ 240 V and uses a 250 W infrared lamp. Either meter should be equipped with an analytical balance. Figure 1 shows an example of an infrared moisture meter.

NOTE 2 In principle, the meter used should have a capacity of 5 g, regardless of whether it is a distance or a voltage adjusting-type meter. A meter of different capacity may be used provided that it offers the same accuracy as the meter of 5g capacity. The calibrated scale of the meter should be checked when purchasing a new meter, after a specific period of time or as deemed necessary.

4.3 Procedure

4.3.1 Set up the moisture meter according to the manufacturer's instructions.

4.3.2 Weigh out, with precision, 5,00 g of the sample and spread it evenly on the infrared moisture meter's sample dish.

4.3.3 Start the machine.

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NOTE 3 The upper rim of the sample dish is set at a distance of 75 mm $\pm$ 2 mm from the central surface of the infrared lamp of the moisture meter. Infrared irradiation is then delivered to the sample.

4.3.4 When the change in the meter reading (loss on heating) falls to 0,05% or below per minute of irradiation, then the value given after a further 2 minutes have elapsed, is recorded. This value indicates the loss on heating.

4.4 Calculation

The loss on heating is the reading given by the meter and it is expressed as a percentage.

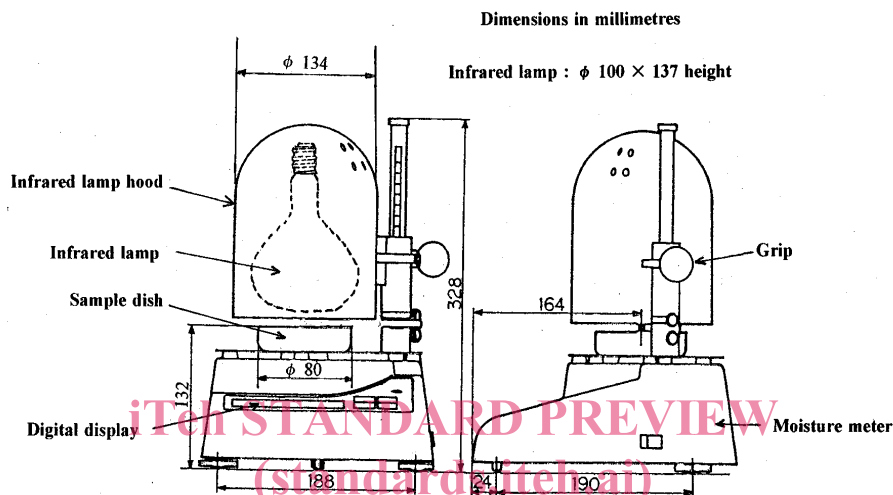
4.5 Rounding off of test results

Round off the test results and express them to one decimal place.

4.6 Test report

The test report shall include the following particulars:

- a) a reference to this International Standard;
- b) method adopted for test (record that the infrared irradiation method was adopted);
- c) all details necessary to identify the sample;
- d) the results, and the units in which they have been expressed;
- e) any unusual features noted during the determination;
- f) any operation not included in this International Standard or regarded as optional.



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Figure 1 — An example of an infrared moisture meter
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5. Differences between Method 2 and Method 3

	Method 2	Method 3
Apparatus	Moisture balance	Infrared moisture meter
Sensitivity	0,1 mg	1,0 mg
Measuring method	Indirect heating source	Direct heating source Distance adjusting-type Voltage adjusting-type
Measuring temperature	Should not exceed 125 °C	---
Sample volume	About 2 g	About 5 g
Condition before reading	Once the mass loss, under these drying conditions, is less than 1 mg over 30 s the test portion is considered dry and the percent mass loss should be recorded to the nearest 0,1 %.	When the change in the meter reading (loss on heating) falls to 0,005 % or below per minute of irradiation, then the value given after further 2 minutes has elapsed, is recorded.
Determination	A- B $H = \frac{\text{-----}}{A} \times 100$	By reading

6. Precision statement

Method 1 and Method 3 were compared in various laboratories on different samples. The test data are reported in Table 1 and Table 2.

6.1 Test data , Method 1 using the Convection-Gravity Oven Method

Drying for 1 h in a thermostatic dryer at $125\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$

Table 1 Test data - Loss on heating (%)

Sample		A	B	C	D	E
Laboratory	Sample No.					
I	1	0,4	0,8	1,4	2,2	4,5
	2	0,4	0,8	1,4	2,4	4,5
	3	0,4	0,8	1,3	2,4	4,2
	4	0,4	0,9	1,4	2,3	4,3
II	1	0,4	0,8	1,4	2,4	4,6
	2	0,3	0,8	1,4	2,5	4,6
	3	0,4	0,7	1,4	2,3	4,3
	4	0,4	0,8	1,3	2,4	4,4
III	1	0,4	0,8	1,4	2,4	4,7
	2	0,4	0,8	1,4	2,5	4,5
	3	0,4	0,8	1,3	2,3	4,4
	4	0,4	0,7	1,3	2,5	4,7
IV	1	0,4	0,8	1,4	2,4	4,6
	2	0,4	0,8	1,4	2,2	4,6
	3	0,4	0,7	1,2	2,4	4,4
	4	0,4	0,7	1,3	2,4	4,4
V	1	0,4	0,8	1,4	2,5	4,7
	2	0,3	0,8	1,4	2,6	4,4
	3	0,4	0,8	1,3	2,5	4,4
	4	0,3	0,7	1,3	2,4	4,5

Sample	Mean Level	Within Labs		
		Sr	r	(r)
A	0,39	0,034	0,096	24,62
B	0,78	0,052	0,147	18,85
C	1,36	0,065	0,184	13,53
D	2,40	0,091	0,258	10,75
E	4,49	0,142	0,402	8,95
Pooled	1,88	0,085	0,242	12,87