INTERNATIONAL STANDARD

ISO 1126

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

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

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1126 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*.

This fourth edition cancels and replaces the third (ISO 1126:1992). Two additional methods have been included: a moisture balance method and an infrared irradiation method.

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

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 specifies methods for determining the 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 is used:

— method 1: gravity-convection oven method;

— method 2: moisture balance method;

— method 3: infrared irradiation method (rapid method).

Note that method 1 is considered as the reference method. Apparatus equivalent to that specified may be used provided the same results are obtained.

2 Method 1: Gravity-convection 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, the temperature of which can be regulated to within ± 1 °C at 125 °C and the temperature uniformity of which is ± 5 °C or better.

2.2.2 Weighing bottle, squat-form, 30 mm in height and 60 mm 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 To prevent loss of carbon black due to air currents, keep the weighing bottle stoppered when transferring to and from the desiccator.

2.3.2 Determination

2.3.2.1 Dry the weighing bottle (2.2.2) and the stopper, with the stopper removed, for 30 min in the oven (2.2.1) set at 125 °C. Then place the bottle and the stopper in the desiccator (2.2.4) and allow to cool to room 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 (2.2.1) for 1 h at 125 °C, with the stopper removed.

2.3.2.4 Insert the stopper and transfer the bottle and contents to the desiccator. Remove the stopper and allow to cool to ambient temperature. Re-insert the stopper in the weighing bottle and reweigh to the nearest 0,1 mg.

2.4 Expression of results

Calculate the loss on heating, expressed as a percentage by mass, to the nearest 0,1 % using the equation

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

where

H is the loss on heating, in %;

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.

3 Method 2: Moisture balance method

3.1 Principle

A test portion of carbon black is heated at a temperature of not more than 125 °C and the decrease in mass measured using a moisture balance.

3.2 Apparatus

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

3.3 Procedure

3.3.1 Set up the moisture balance in accordance with the manufacturer's instructions. The temperature shall 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 shall be recorded to the nearest 0,1 %.

3.4 Calculation

If the test result is not displayed automatically by the instrument, calculate the percent loss on heating as follows:

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

where

H is the loss on heating, in %;

A is the mass, in grams, of the test portion before heating;

B is the mass, in grams, of the test portion after heating.

4 Method 3: Infrared irradiation method (rapid method)

4.1 Principle

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

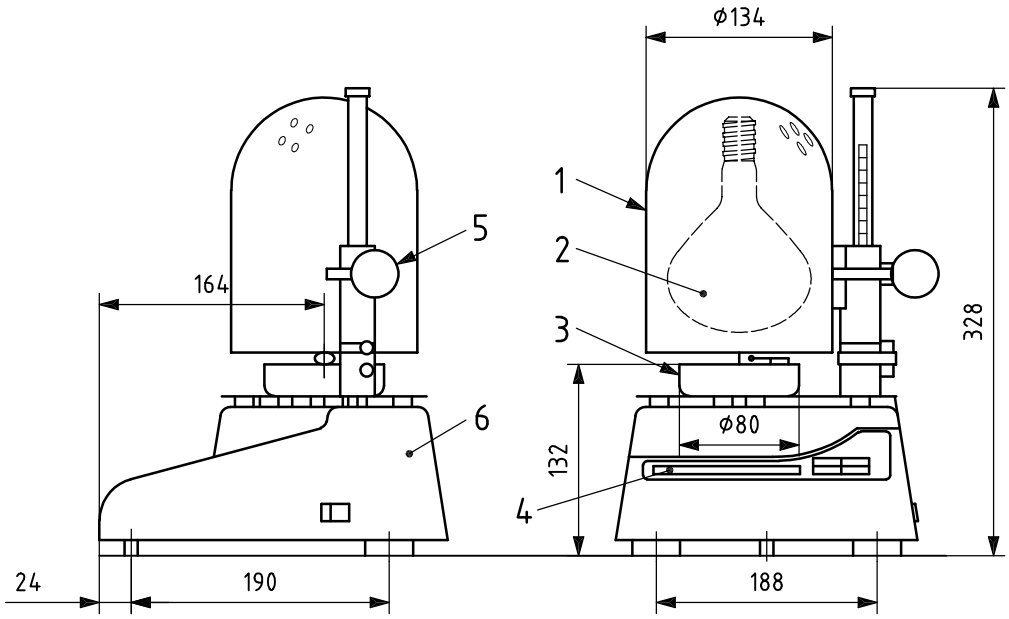
4.2 Apparatus

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

The moisture meter shall be either a distance-adjusting-type meter using a 185 W infrared lamp or a voltage-adjusting-type meter using a 250 W infrared lamp. In either case, the meter shall be equipped with an analytical balance. Figure 1 shows an example of an infrared moisture meter.

In principle, the meter used shall have a capacity of 5 g, regardless of whether it is a distance- or voltage-adjusting-type meter. A meter of different capacity may be used provided that it offers the same accuracy as a 5 g capacity meter. When using a newly purchased meter, the calibrated scale of the meter shall be checked, either after a specific period of time or as deemed necessary.

Dimensions in millimetres



Key

1 infrared lamp hood
2 infrared lamp (∅ 100 mm × 137 mm height)
3 sample dish
4 digital display
5 grip
6 moisture meter

Figure 1 — An example of an infrared moisture meter

4.3 Procedure

4.3.1 Set up the moisture meter in accordance with the manufacturer's instructions.

4.3.2 Weigh 5 g of the sample to the nearest ± 0,01 g and spread it evenly over the sample dish of the infrared moisture meter.

4.3.3 Start the machine.

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

4.3.5 When the change in the meter reading falls to 0,05 % or below per minute of irradiation, record the value given after a further 2 min have elapsed. This value is taken as the loss on heating. It is expressed as a percentage.

5 Differences between method 2 and method 3

Table 1 — Comparison of methods 2 and 3

	Method 2	Method 3
Apparatus	Moisture balance	Infrared moisture meter
Sensitivity	0,1 mg	1,0 mg
Measurement method	Indirect heating source	Direct heating source Distance-adjusting-type Voltage-adjusting-type
Measurement temperature	Not to exceed 125 °C	—
Mass of test portion	About 2 g	About 5 g
Point at which reading is taken	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 is recorded to the nearest 0,1 %.	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 min have elapsed is recorded.
Calculation of result	$H = \frac{A - B}{A} \times 100$	Automatic readout

6 Precision

6.1 General

Method 1 and method 3 were compared in various laboratories using several different samples. The precision data are reported in Table 2 and Table 3.

6.2 Precision data for method 1 using the gravity-convection oven method (drying for 1 h at 125 °C)

Table 2 — Precision data — Loss on heating (%) by method 1

Sample	Mean level	Within lab			Between labs		
		s_r	r	(r)	s_R	R	(R)
A	0,39	0,034	0,097	25,10	0,037	0,105	27,24
B	0,78	0,052	0,146	18,73	0,052	0,148	19,02
C	1,36	0,065	0,183	13,47	0,065	0,183	13,47
D	2,40	0,091	0,258	10,76	0,105	0,296	12,33
E	4,49	0,142	0,402	8,96	0,143	0,403	8,99
Pooled	1,88	0,085	0,242	12,85	0,087	0,252	13,37

s_r = within-laboratory standard deviation;
r = repeatability (in measurement units);
(r) = repeatability (in percent);
s_R = between-laboratory standard deviation;
R = reproducibility (in measurement units);
(R) = reproducibility (in percent).