INTERNATIONAL STANDARD

ISO 1125

Fourth edition
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Rubber compounding ingredients — Carbon black — Determination of ash

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination du taux de cendres

Reference number
ISO 1125:2015(E)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is 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 edition (ISO 1125:1999), of which it constitutes a minor revision. It also incorporates ISO 1125:1999/Amd.1:2011.

The modifications include updating the normative references (in Clause 2 and throughout the text), moving precision data in an informative annex and adding a Bibliography.

Rubber compounding ingredients — Carbon black — Determination of ash

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 a method for determining the ash of all types of carbon black for use in the rubber industry.

2 Normative references

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

ISO 1124, *Rubber compounding ingredients — Carbon black shipment sampling procedures*

3 Principle

An accurately weighed portion of dried sample is ignited in a crucible until all the carbonaceous material is oxidized. The crucible is cooled in a desiccator, weighed, and the percentage of ash calculated.

4 Apparatus

4.1 Muffle furnace, capable of maintaining a temperature of 550 °C ± 25 °C (or other required temperature).

NOTE The use of a vented furnace would decrease the time of heating to constant mass (see 6.3).

4.2 Porcelain crucible, tall form, diameter 35 mm, height 30 mm, with lid.

The use of the lid on the crucible is optional. If it is not used, this shall be mentioned in the test report.

4.3 Analytical balance, accurate to 0,1 mg.

4.4 Desiccator.

4.5 Oven, preferably of the gravity-convection type, capable of temperature regulation to within ± 1 °C at 125 °C and temperature uniformity to within ± 5 °C.

5 Sampling

Carry out sampling in accordance with ISO 1124.

6 Procedure

6.1 Heat the crucible (4.2), with its lid (if used), in the muffle furnace (4.1) at a temperature of 550 °C ± 25 °C for 1 h. Place the crucible (and lid) in the desiccator (4.4). Cool to ambient temperature and reweigh to the nearest 0,1 mg.

NOTE If, after cleaning, drying and reweighing at the end of the determination (see 6.4), the crucible is stored in a dessicator, step 6.1 is needed only for new crucibles.

6.2 Dry a little more than 2 g of furnace carbon black or a little more than 5 g of thermal or channel carbon black in the oven (4.5) at a temperature of 125 °C for 1 h. Allow to cool to room temperature.

6.3 Weigh, to the nearest 0,1 mg, about 2 g of dried furnace carbon black or 5 g of dried thermal or channel black into the crucible tared in 6.1, place in the furnace at a temperature of 550 °C ± 25 °C and heat uncovered until constant mass is obtained.

Cover with the lid (if used), remove to the desiccator, and allow to cool to ambient temperature. Weigh to the nearest 0,1 mg. To avoid repeated handling of the crucible, an adequate ashing time shall be established at each test site.

IMPORTANT — Take the following precautions:

a) keep the door of the furnace open about 0,5 cm to admit air to support the combustion of organic material;

b) after the test portion has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible due to air currents.

By mutual agreement between the interested parties, it is permissible to use another ashing temperature such as 750 °C ± 25 °C or 825 °C ± 25 °C (4.1, 6.1 and 6.3 would have to be adapted accordingly).

However, this temperature leads to lower ash contents than the ones obtained at 550 °C, and shall not be used for reference purposes.

6.4 Clean the crucible (and lid), dry in the oven (4.5) at 125 °C and reweigh to the nearest 0,1 mg.

7 Expression of results

The ash A is given, as a percentage by mass, by the following equation.

$$A = \frac{m_2 - m_3}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the crucible (and lid) before the determination;

m_1 is the mass, in grams, of the crucible (and lid) plus the test portion;

m_2 is the mass, in grams, of the crucible (and lid) plus the ash;

m_3 is the mass, in grams, of the crucible (and lid) after the determination (should be the same as m_0).

8 Precision

See Annex A.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

c) the results and the units in which they have been expressed;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or any operation regarded as optional such as the ashing temperature if different from the specified one or the omission of the crucible lid;

f) the date of the test.

Annex A
(informative)

Precision data

A.1 The precision of the method was determined in accordance with ISO/TR 9272. Refer to this document for terminology and other statistical details.

A.2 The precision data given below merely give an estimate of the precision. The precision parameters shall not therefore be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific test protocols that include this method.

A.3 A type 1 interlaboratory precision programme was conducted. Both repeatability and reproducibility were measured under short-term testing conditions. Ten laboratories tested five carbon black samples (A, B, C, D and E) twice on two different days.

Difference values were not measured.

A.4 The results of the precision calculations are given in Table A.1, with the materials arranged in ascending order of the mean ash value.

Table A.1 — Precision data

Material	Mean ash	Within laboratory			Between laboratories		
	%	s_r	r	(r)	s_R	R	(R)
Black A	0,17	0,016	0,045	25,497	0,021	0,060	35,188
Black E	0,35	0,020	0,057	16,121	0,037	0,014	29,663
Black B	0,45	0,030	0,085	18,919	0,043	0,122	27,067
Black C	0,61	0,027	0,076	12,483	0,037	0,106	17,445
Black D	0,83	0,016	0,045	5,620	0,023	0,066	7,961
Pooled or averaged values	0,48	0,02	0,06	13,25	0,03	0,09	19,63

$p = 10$, $q = 5$ and $n = 4$

s_r is the within-laboratory standard deviation

r is the repeatability (in measurement units)

(r) is the repeatability (in percent)

s_R is the between-laboratory standard deviation

R is the reproducibility (in measurement units)

(R) is the reproducibility (in percent)

A.5 The precision for the pooled values for the ash may be expressed as follows:

a) **Repeatability**: The repeatability r of the result has been established as 0,06 % ash. Two individual test results (or determinations) that differ from each other by more than 0,06 % ash shall be considered suspect and dictate that some appropriate investigative action be taken.

b) **Reproducibility**: The reproducibility R of the result has been established as 0,09 % ash. Two individual test results (or determinations) produced in separate laboratories that differ by more than 0,09 % ash shall be considered suspect and dictate that some appropriate investigative action be taken.

A.6 Bias: In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.