BRITISH STANDARD

BS ISO 6209:2009

# Rubber compounding ingredients — Carbon black — Determination of solvent-extractable material

ICS 83.040.20



BSI
British Standards

# National foreword

This British Standard is the UK implementation of ISO 6209:2009. It supersedes BS 5293-16:1990 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber - Raw, natural and synthetic, including latex and carbon black.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Compliance with a British Standard cannot confer immunity from legal obligations.**

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BS ISO 6209:2009

INTERNATIONAL STANDARD

**ISO 6209**

Fourth edition
2009-07-15

# Rubber compounding ingredients — Carbon black — Determination of solvent-extractable material

*Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination des matières extractibles par les solvants*

Reference number
ISO 6209:2009(E)

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# Contents

Page

# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 6209 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 edition (ISO 6209:1988), which has been technically revised.

The main changes are as follows:

— the extraction time has been reduced to 8 h (instead of 16 h);

— precision data have been included (see Annex A).

**INTERNATIONAL STANDARD** **ISO 6209:2009(E)**

# Rubber compounding ingredients — Carbon black — Determination of solvent-extractable material

**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.**

**Carbon blacks may contain polycyclic aromatic hydrocarbons, some of which are known carcinogens. These compounds, when present, are so strongly bound to the carbon black that they are not biologically active. They can, however, be removed by the procedure specified in this International Standard. Care should therefore be taken to avoid skin contact with solvent extracts from such carbon blacks.**

## 1 Scope

This International Standard specifies a method for the quantitative determination of the solvent-extractable material in carbon black for use in the rubber industry. The method is applicable to all types of carbon black.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. 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*

ISO 1126:2006, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*

## 3 Principle

A test portion is extracted for 8 h. The solvent is then eliminated by evaporation and the extract obtained is weighed.

NOTE If the carbon black contains extractable materials which are volatile at the temperature required to eliminate the solvent, or materials which are removed by the preliminary drying, such materials will not be detected by the procedure specified.

This test provides a uniform and precise method for the gravimetric determination of organic-solvent-extractable materials in carbon black. Accuracy and precision are acceptable for most specification or regulatory purposes, or both. However, carbon black with a very low extract (less than 0,02 %) may require a more rigorous extraction procedure.

## 4 Extraction solvent

The solvent used shall be of recognized analytical grade. The use of toluene is recommended as it is the solvent of choice in most food-contact-related legislation.

# 5 Apparatus and material

## 5.1 Extraction apparatus

Use one of the following types of apparatus:

**5.1.1 Type 1 extraction apparatus**, comprising a 150 $cm^3$ receiver flask, a jacketed Soxhlet extractor and a condenser as shown in Figure 1. The extraction cup has a capacity of 15 $cm^3$ to 30 $cm^3$.

**5.1.2 Type 2 extraction apparatus**, comprising a 500 $cm^3$ receiver flask, a condenser and an extraction cup suspended from two hooks on the condenser by clean wire as shown in Figure 2. The extraction cup has a capacity of 15 $cm^3$ to 30 $cm^3$.

## 5.2 Other apparatus and material

**5.2.1 Extraction thimbles**, of 15 $cm^3$ to 30 $cm^3$ capacity, of sufficiently fine porosity to retain carbon black. They may be made of greaseless paper, cellulose or alundum and shall be of the appropriate size to fit the extraction cup. Thimbles shall be extracted with solvent and dried before use.

**5.2.2 Distillation head and condenser** or **rotary evaporator**.

**5.2.3 Gravity-convection oven**, capable of maintaining temperatures of 70 °C ± 5 °C for drying the extract and 125 °C ± 5 °C for drying the carbon black prior to extraction.

**5.2.4 Cotton wool**, greaseless, or **glass wool**, solvent-washed and dried.

**5.2.5 Analytical balance**, accurate to 0,1 mg.

**5.2.6 Heating device**, suitable for the extraction apparatus (5.1).

**5.2.7 Desiccator**.

# 6 Sampling

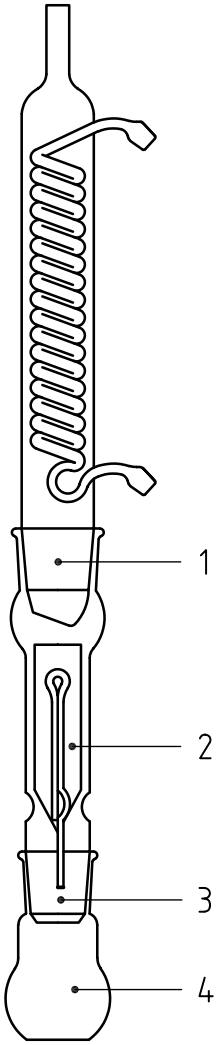
Carry out sampling in accordance with ISO 1124.

Crush all carbon blacks to destroy the pellet configuration before drying.

Dry approximately 20 g of the carbon black sample for 1 h at a temperature of 125 °C ± 5 °C in the oven (5.2.3) as specified in ISO 1126:2006, method 1. Allow to cool to room temperature in a desiccator (5.2.7). Keep the dried sample in the desiccator until ready for testing.

Carbon black shall not be dried at a temperature higher than that specified, nor dried using infra-red lamps, as some of the extractable matter may be driven off, thus affecting the results.

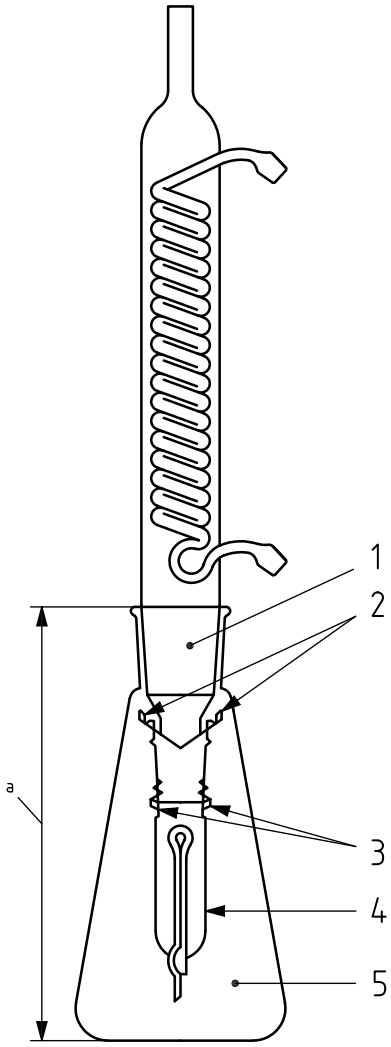
Take duplicate test portions from this dried sample.




**Key**

1 34/35 joint
2 extraction cup (15 $cm^3$ to 30 $cm^3$ capacity)
3 24/29 joint
4 receiver flask (150 $cm^3$ capacity)

**Figure 1 — Type 1 extraction apparatus**




**Key**

1 34/35 joint
2 two hooks
3 two holes in extraction cup
4 extraction cup (15 $cm^3$ to 30 $cm^3$ capacity)
5 receiver flask (500 $cm^3$ capacity)

[a] 170 mm nominal.

**Figure 2 — Type 2 extraction apparatus**

## 7 Procedure

**7.1** Take a test portion of about 10 g from the prepared sample, place it in a weighed thimble (5.2.1) and reweigh to the nearest 0,1 mg to obtain the mass of carbon black. Close the opening with a plug of cotton or glass wool (5.2.4).

For carbon blacks with a low extractable-material content (less than 0,02 %, for example), the test portion can be increased to 20 g.

**7.2** Weigh the clean, dry receiver flask (see 5.1) to the nearest 0,1 mg and pour in 100 $cm^3$ of solvent (Clause 4).

**7.3** Place the extraction thimble containing the test portion in the extraction cup, assemble the apparatus (5.1) and adjust the rate of heating of the heating device (5.2.6) so that the distilled solvent fills the extraction cup about 10 times per hour.

Allow the extraction to proceed for 8 h to 8,5 h.

**7.4** Turn off the heating device, allow the apparatus to cool, then remove the extraction cup and discard the thimble.

**7.5** Remove the receiver flask, fit the distillation head and condenser (5.2.2) and distill off the bulk of the solvent into a suitable vessel, retaining no more than 5 $cm^3$ in the receiver flask. A rotary evaporator can also be used to remove the solvent.

Discard the distilled solvent.

**7.6** Allow the apparatus to cool and then disconnect the receiver flask, which now contains the concentrated extract. Remove most of the remaining solvent by passing a gentle stream of clean, dry air into the flask.

**7.7** Dry the flask and its contents for 2 h at 70 °C ± 5 °C in the oven (5.2.3), cool to ambient temperature in the desiccator (5.2.7) and weigh to the nearest 0,1 mg.

**7.8** Carry out a blank test, using the same quantity of solvent and the same type of extraction apparatus as used for the determination, but omitting the test portion.

**7.9** Carry out two determinations.

## 8 Expression of results

The solvent-extractable material, expressed as a percentage by mass, is given by the formula:

$$\frac{(m_2 - m_1) - \Delta m}{m_0} \times 100$$

where

$m_0$ is the mass, in grams, of the test portion (see 7.1);

$m_1$ is the mass, in grams, of the empty receiver flask (see 7.2);

$m_2$ is the mass, in grams, of the receiver flask plus the extract after drying (see 7.7);

$\Delta m$ is the increase in mass, in grams, of the receiver flask during the blank test (see 7.8).

## 9 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

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

c) the type of extraction apparatus used;

d) the solvent used;

e) the results obtained for each determination and their arithmetic mean;

f) the date(s) of the determinations.

# Annex A
(informative)

# Precision of the test method

An interlaboratory test programme (ITP) was carried out with three laboratories. The results are given in Table A.1.

**Table A.1 — Results of the ITP after an 8 h extraction**

| Material | Number of participating laboratories | Number of replicates | Average value | $s_r$ | $r$ | $(r)$ | $s_R$ | $R$ | $(R)$ |
|---|---|---|---|---|---|---|---|---|---|
| N326 | 3 | 6 | 0,074 | 0,021 | 0,059 | 80 | 0,062 | 0,175 | 237 |
| N375 | 3 | 6 | 0,267 | 0,050 | 0,142 | 53 | 0,084 | 0,238 | 89 |

$r$ is the repeatability, in measurement units;

$(r)$ is the repeatability, in percent (relative);

$s_r$ is the repeatability standard deviation;

$R$ is the reproducibility, in measurement units;

$(R)$ is the reproducibility, in percent (relative);

$s_R$ is the reproducibility standard deviation.

# Bibliography

[1] ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

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