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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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 : 1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6209 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 6209 : 1983), of which it constitutes a minor revision (two preconditioning temperatures, between which the user may choose, are specified).

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

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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383 : 1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 1124 : 1983, *Rubber compounding ingredients — Carbon black — Sampling shipments in bulk or in bins.*

ISO 1310 : 1974, *Carbon black for use in the rubber industry — Sampling packaged shipments.*

3 Principle

Extraction of a test portion with solvent for 16 h. Elimination of the solvent by evaporation and weighing of the extract obtained.

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

4 Reagents

WARNING — All recognized health and safety precautions shall be taken when using the procedure specified in this International Standard.

Carbon blacks may contain polynuclear aromatic compounds, some of which are known carcinogens. These compounds, when present, are so strongly bound to the carbon black that they are biologically inactive, but they may be removed by the procedure specified in this International Standard. Care should be taken to avoid skin contact with solvent extracts from such carbon blacks.

During the analysis, use only reagents of recognized analytical grade.

4.1 Acetone.

4.2 Toluene.

5 Apparatus and material

5.1 Extraction apparatus.

Two types of extraction apparatus are suitable:

5.1.1 Type 1 comprises 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 to 30 cm^3.

5.1.2 Type 2 comprises 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 to 30 cm^3.

5.2 Extraction thimbles, of capacity 15 to 30 cm^3, of sufficiently fine porosity to retain carbon black. They may be made of greaseless paper, cellulose or alundum and shall be of the correct size to fit the extraction cup.

Thimbles shall be extracted with solvent and dried before use.

5.3 Distillation head and condenser.

5.4 Ventilated oven, capable of being set at 70 ± 2 °C for drying the extract and at 105 °C ± 2 °C or 125 °C ± 2 °C for preconditioning the carbon black prior to extraction.

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

5.6 Analytical balance, accurate to 0,1 mg.

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

5.8 Desiccator.

6 Sampling

Carry out sampling in accordance with ISO 1124 or ISO 1310, as appropriate.

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

Precondition approximately 20 g of carbon black for 1 h at 105 °C ± 2 °C or 125 °C ± 2 °C. Allow to cool to ambient temperature in a desiccator before proceeding with the determination. Store the preconditioned sample in a desiccator.

Take duplicate test portions from the preconditioned sample.

7 Procedure

7.1 Take a test portion of about 5 g of the prepared sample, place it in a weighed thimble (5.2) and reweigh to the nearest 0,1 mg to obtain the mass of the carbon black. Close the opening with a plug of cotton or glass wool (5.5).

7.2 Weigh the clean, dry receiver flask (see 5.1) to the nearest 0,1 mg and pour in 100 cm^3 of the solvent (4.1 or 4.2).

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 (5.7) so that the distilled solvent will fill the extraction cup about 10 times each hour.

Carry out the extraction for 16 to 16,5 h. (For convenience, the extraction is usually carried out overnight.)

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 (5.3) and distil off the bulk of the solvent into a suitable vessel, retaining no more than 5 cm^3 in the receiver flask.

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 warm, clean, dry air into the flask.

7.7 Dry the flask and contents for 2 h at 70 °C ± 2 °C in the oven (5.4), cool to ambient temperature in the desiccator (5.8) 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;

m_1 is the mass, in grams, of the empty receiver flask;

m_2 is the mass, in grams, of the receiver flask plus extract after drying;

Δm is the increase in mass, in grams, of the receiver flask during the blank test.

9 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) identification of the sample;

c) the type of extraction apparatus used;

d) the solvent used;

e) the preconditioning temperature used (105 °C or 125 °C);

f) the results obtained for each determination;

g) the arithmetic mean of the results of the two determinations (7.9).

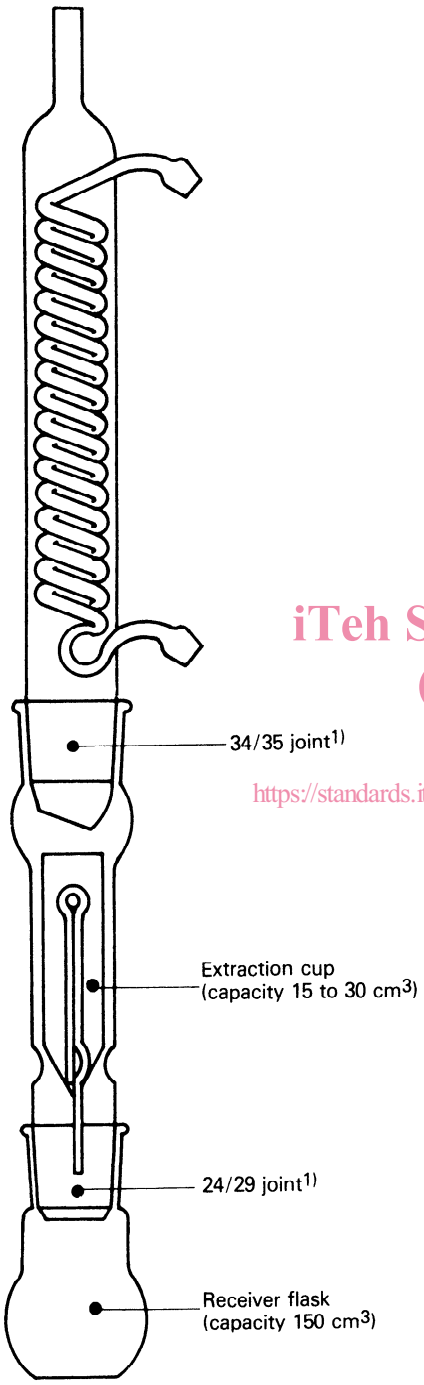

Figure 1 — Type 1 extraction apparatus

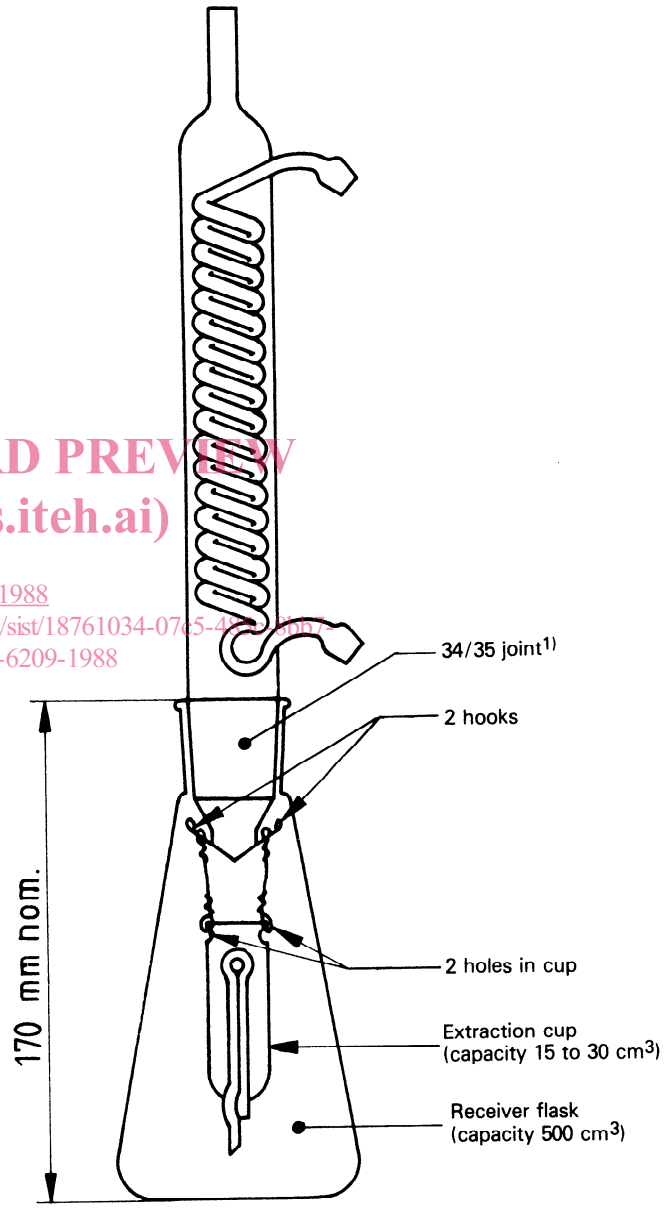

Figure 2 — Type 2 extraction apparatus

1) See ISO 383.

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