International Standard ISO 6209

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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

Second edition — 1983-09-01

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Descriptors : rubber industry, carbon black, chemical analysis, determination, solvents.

Foreword

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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.

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

The first edition (ISO 6209-1981) had been approved by the member bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Canada	Italy	Sri Lanka
China	Korea, Rep. of	Sweden
Czechoslovakia	Libyan Arab Jamahiriya	Switzerland
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France	New Zealand	United Kingdom
Germany, F. R.	Poland	USA
Greece	Romania	USSR

The member body of the following country had expressed disapproval of the document on technical grounds :

Netherlands

This second edition, which cancels and replaces ISO 6209-1981, incorporates draft Amendment 1, which was circulated to the member bodies in January 1982 and has been approved by the member bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Ireland	Sri Lanka
Bulgaria	Italy	Sweden
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Germany, F. R.	Romania	

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

1 Scope and field of application

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 References

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

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

ISO 1310, *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[1)]

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 controlled at 70 ± 2 °C for drying the extract and at 125 ± 2 °C for drying 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.

1) The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the Twelfth Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes. Apparatus with either type of marking is satisfactory for use with this International Standard.

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 drying.

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

Take duplicate test portions from the dried 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 ± 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

$$\frac{(m_2 - m_1) - m_3}{m_0} \times 100$$

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;

m_3 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 results obtained for each determination;

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

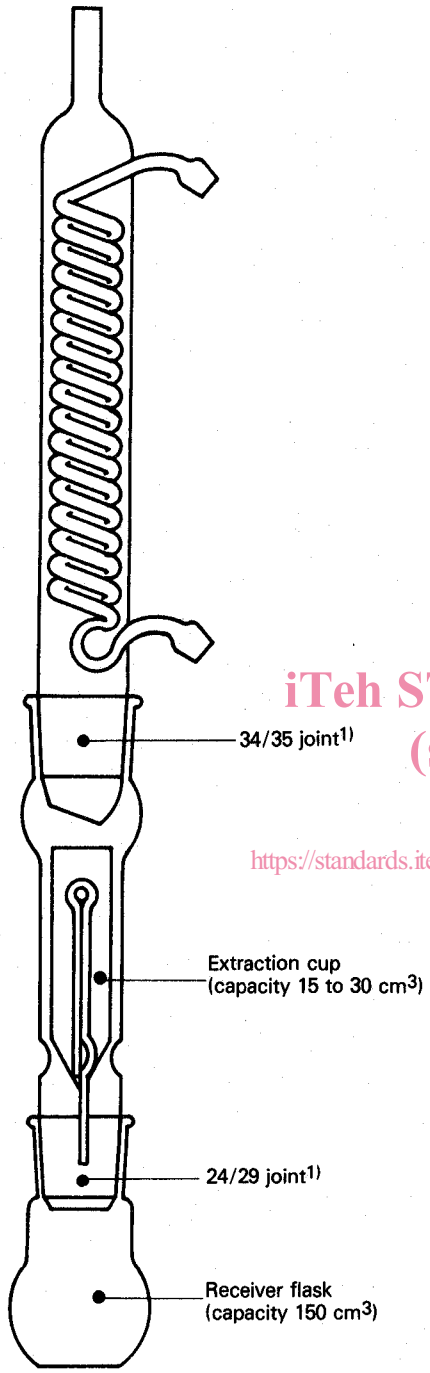

Figure 1 — Type 1 extraction apparatus

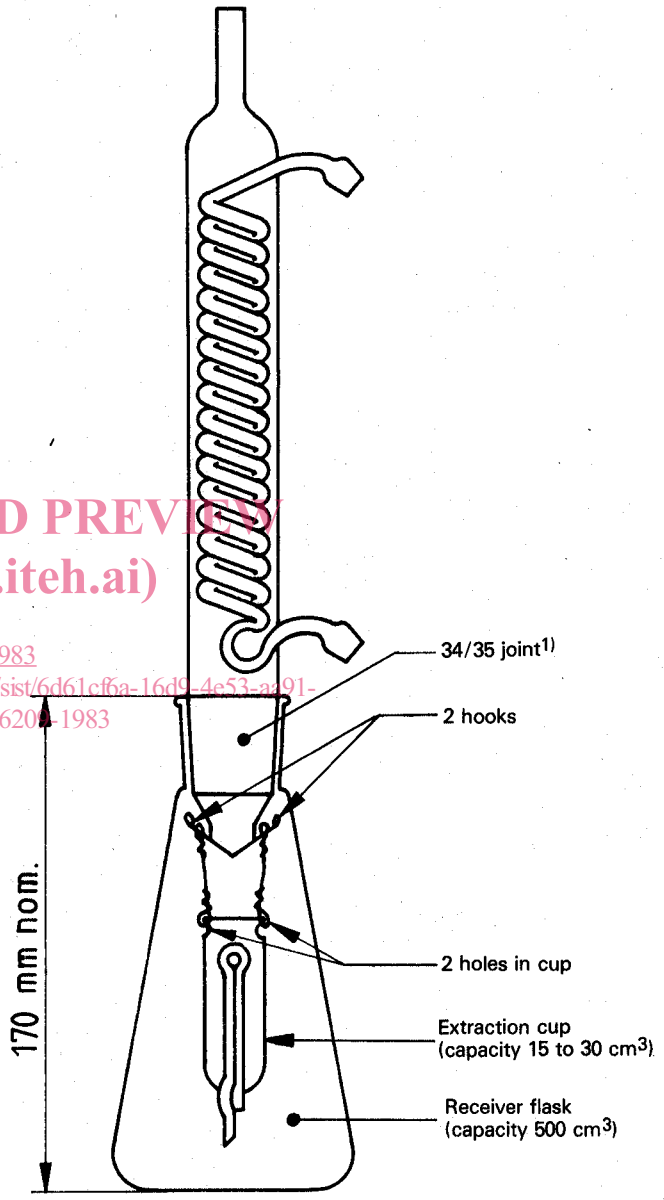

Figure 2 — Type 2 extraction apparatus

1) See ISO 383.