INTERNATIONAL STANDARD

ISO 1304

Fifth edition
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Rubber compounding ingredients — Carbon black — Determination of iodine adsorption number

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de l'indice d'adsorption d'iode

Reference number
ISO 1304:2016(E)

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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.

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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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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 fifth edition cancels and replaces the fourth edition (ISO 1304:2006), which has been technically revised with the following changes:

— Clause 2 "Normative references" has been updated;

— the preferred method is stated in the scope and in 7.2.5;

— 4.1 (analytical balance) and 4.12 (desiccator) have been updated;

— the tolerance of the weighting in 6.1.5 has been modified to 0,01 g;

— the precision data have been moved to an informative annex.

Rubber compounding ingredients — Carbon black — Determination of iodine adsorption number

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 the determination of iodine adsorption number of carbon blacks for use in the rubber industry. Two titration methods are described:

— method A: titration using a burette and starch as indicator;

— method B: potentiometric titration with an automatic titrator.

The iodine adsorption number is related to the surface area of a carbon black and is generally in agreement with the nitrogen surface area. However, it is significantly depressed in the presence of a high content of volatile or solvent-extractable materials; the iodine adsorption number therefore does not always provide a measure of the specific surface area of a carbon black. Ageing of carbon black can also influence the iodine number.

In case of dispute, the preferred method is method B (potentiometric titration).

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

3 Principle

A test portion of carbon black is dried, weighed and mixed vigorously with a measured volume of standard iodine solution. The mixture is then centrifuged. A measured volume of the clear iodine solution is titrated with a standard solution of sodium thiosulfate. From this titration value and the mass of the test portion, the iodine adsorption number of the carbon black is calculated.

4 Apparatus

Ordinary laboratory equipment (beakers, funnels, porcelain spoon, weighing bottles, etc.), plus the following:

4.1 Analytical balance, with sensitivities:

a) 0,01 g (for 6.1.5 and 7.3.5);

b) 0,1 mg (for other paragraphs).

4.2 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.

4.3 Stoppered one-mark volumetric flasks, preferably class A in accordance with ISO 1042, of capacities:

a) 2 000 cm^3, with a tolerance of ±0,60 cm^3;

b) 1 000 cm^3, with a tolerance of ±0,40 cm^3.

4.4 Repetitive dispenser, 25 cm^3 capacity, calibrated to within ±0,03 cm^3 accuracy, or **one-mark pipettes**, high precision, of capacities:

a) 20 cm^3, with a tolerance of ±0,03 cm^3;

b) 25 cm^3, with a tolerance of ±0,03 cm^3.

If class A pipettes in accordance with ISO 648 are used, no calibration is necessary. In other cases, pipettes shall be calibrated to the nearest 0,01 cm^3 with distilled water, a temperature correction being made if necessary to show the true delivery at any volume used to within 0,01 cm^3. The true delivered volume is the read volume plus (or minus) the calibration correction at that volume. For high-precision volume determination (see 7.2.2, 7.3.2, 8.3.3, 8.3.6 and 8.3.8), it is recommended that the 20 cm^3 and 25 cm^3 pipettes have calibration corrections of the same magnitude and in the same sense.

4.5 Digital burettes, with 0,01 cm^3 increment counter and zero-reset control, calibrated to within ±0,05 cm^3 accuracy, or **burettes** (for method A only), high precision, side-arm filling, graduated in 0,05 cm^3 and with automatic zero, of capacities:

a) 25 cm^3, with a tolerance of ±0,05 cm^3;

b) 50 cm^3, with a tolerance of ±0,05 cm^3.

If class A burettes in accordance with ISO 385 are used, no calibration is necessary. In other cases, burettes shall be calibrated to the nearest 0,01 cm^3 with distilled water, a temperature correction being made if necessary to show the true delivery at any volume used to within 0,01 cm^3. The true delivered volume is the read volume plus (or minus) the calibration correction at that volume.

4.6 Stoppered bottles, with ground-glass stoppers, of capacities 250 cm^3 and 500 cm^3.

4.7 Glass bottle, with ground-glass stopper, of capacity 2 000 cm^3.

4.8 Amber-glass bottles, with ground-glass stoppers, of capacities 1 000 cm^3 and 2 000 cm^3.

4.9 Centrifuge tubes, of capacity 50 cm^3, with screw cap and polyethylene liner.

Cork, rubber or metal stoppers shall not be used.

4.10 Mechanical shaker, capable of 240 strokes/min, with 25 mm stroke length.

4.11 Centrifuge, minimum speed 105 rad/s (1 000 r/min).

4.12 Desiccator, with silica gel as desiccant.

4.13 Magnetic stirrers and **spin bars**.

4.14 Automatic titrator (for method B only), equipped with a combined electrode for potentiometric titration.

5 Reagents

Unless otherwise stated, all chemicals shall be of reagent grade.

5.1 Water, deionized or distilled.

5.2 Iodine (I_2).

5.3 Potassium iodide (KI).

5.4 Potassium iodate (KIO_3).

5.5 Sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$).

5.6 *n*-Amyl alcohol ($C_5H_{11}OH$).

5.7 Sulfuric acid (H_2SO_4), mass fraction 98 %, ρ = 1,84 Mg/m^3.

5.8 Soluble starch (for method A only).

5.9 Salicylic acid ($C_7H_6O_3$) (for method A only).

6 Preparation of solutions

6.1 Iodine solution, 0,023 64 mol/dm^3 (0,047 28 N), containing 9,5 parts of potassium iodide to 1 part of iodine.

NOTE Since the test result depends on the concentration of both iodine and potassium iodide in the solution, the instructions for the preparation and the standardization of the solution (7.3) have to be followed precisely.

6.1.1 Weigh, to the nearest 0,01 g, 114,00 g of potassium iodide (5.3) into a 100 cm^3 beaker.

6.1.2 Place about three-quarters of the KI in a clean 2 000 cm^3 volumetric flask (4.3) through a large-diameter funnel.

6.1.3 Add enough water (5.1) to cover the KI. Swirl to dissolve, and allow to stand until the solution attains ambient temperature.