INTERNATIONAL STANDARD

ISO 1304

Fourth 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
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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 1304 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 1304:1999), the method in which has been entirely revised. In particular, a potentiometric titration has been included as an option and the concentration of the sodium thiosulfate solution has been changed.

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

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 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.

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 385:2005, *Laboratory glassware — Burettes*

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

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

ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

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 0,1 mg sensitivity.

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 1042:1998, 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 648:1977 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 385:2005 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.

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 (see 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.

6.1.4 Place the remainder of the KI in a 250 cm^3 beaker with enough water (5.1) to dissolve it.

6.1.5 Weigh, to the nearest 0,005 g, 12,000 g of iodine on the balance (4.1) in a weighing bottle fitted with a ground-glass stopper. Use only a porcelain spoon to transfer the iodine crystals, and close the weighing bottle when making weighings.

6.1.6 Transfer the iodine through a funnel to the potassium iodide solution prepared in 6.1.3.

6.1.7 Wash thoroughly the weighing bottle with portions of the KI solution prepared in 6.1.4 until no colour remains, and transfer the washings through the funnel to the 2 000 cm^3 volumetric flask.

6.1.8 Wash the funnel with the rest of the KI solution prepared in 6.1.4.

6.1.9 Add water (5.1) to almost fill the volumetric flask, cap it with the ground-glass stopper, invert it 2 or 3 times to homogenize and let it stand for about one hour.

6.1.10 Open the flask, make up to the mark with water (5.1), insert a spin bar in the flask, place it on the magnetic stirrer (4.13) and stir for 2 h at least at medium speed.

NOTE At medium speed, the depth of the vortex should be about 5 mm.

6.1.11 Transfer the solution to an amber-glass bottle (4.8) and let it stand overnight prior to any use.

6.2 Sodium thiosulfate solution, 0,05 mol/dm^3 (0,05 N).

NOTE Previous editions of this International Standard required a thiosulfate concentration of 0,039 4 mol/dm^3 (0,039 4 N). Since the concentration of the thiosulfate solution does not have any impact on the iodine adsorption number, this International Standard now includes a more commonly used solution of 0,05 mol/dm^3 (0,05 N). The advantage is that such a solution is readily available commercially. If preferred, it can be prepared from solid sodium thiosulfate as described below.

Use of sodium thiosulfate solution of 0,039 4 mol/dm^3 (0,039 4 N) is still permitted. In this case, the instructions for the preparation of the solution, the equations used in its standardization and the equations used in the calculation of the iodine adsorption number will have to be modified accordingly.

6.2.1 Weigh, to the nearest 0,005 g, 24,817 g of sodium thiosulfate pentahydrate (5.5) into a suitable container.

6.2.2 With the help of a funnel, transfer the weighed sodium thiosulfate to a 2 000 cm^3 volumetric flask (see 4.3).

6.2.3 Add through the funnel about 1 litre of water (5.1). Wash carefully.

6.2.4 Add 10 cm^3 of *n*-amyl alcohol (5.6) to the flask, and shake the solution in the flask vigorously until all crystals are dissolved.

6.2.5 Make up to the mark with water (5.1), insert a spin bar in the flask, place it on the magnetic stirrer and stir for about 2 h at medium speed (see Note to 6.1.10).

6.2.6 Transfer the solution to a glass bottle (4.7).

6.3 Potassium iodate/iodide solution, $c(KIO_3)$ = 0,008 33 mol/dm^3 (0,05 N).

6.3.1 Dry an adequate quantity of potassium iodate (5.4) in the oven (4.2) at 125 °C for 1 h. Allow to cool to ambient temperature in the desiccator (4.12).

6.3.2 In a 1 000 cm^3 volumetric flask (see 4.3), dissolve 57,0 g (weighed to the nearest 0,1 g) of potassium iodide (5.3) in about 200 cm^3 of water (5.1). Allow to stand until the solution attains ambient temperature.

6.3.3 Weigh out, to the nearest 0,1 mg, 1,783 3 g of the freshly dried potassium iodate (5.4) and transfer to the iodide solution in the volumetric flask.

6.3.4 Make up to the mark with water (5.1). Cap the flask and homogenize the solution by inverting the flask 4 to 5 times.

6.3.5 Transfer the solution to an amber-glass bottle (4.8).

NOTE The potassium iodate/iodide solution is a primary standard in this test method, and it is essential that all precautions be taken to ensure its accuracy.

6.4 Sulfuric acid, mass fraction approximately 20 %.

6.4.1 Measure out 175 cm^3 of water (5.1) in a graduated cylinder and transfer to a 250 cm^3 conical flask.

6.4.2 Measure out 25 cm^3 of concentrated sulfuric acid (5.7) in a small graduated cylinder.

6.4.3 Very carefully pour the acid into the flask of water (6.4.1), and swirl gently to mix. Rinse the graduated cylinder with diluted acid from the flask and pour the rinsings back into the flask. Do not use water for rinsing.

6.4.4 Transfer the solution to a 250 cm^3 bottle (4.6), stopper the bottle and allow the solution to cool to ambient temperature before use.

6.5 Starch indicator solution, mass fraction 0,25 % (for method A only).

6.5.1 Place in a 50 cm^3 beaker 2,5 g of powdered soluble starch (5.8), 2 mg of salicylic acid (5.9) and 25 cm^3 of water (5.1). Stir with a glass rod.

6.5.2 Bring 1 000 cm^3 of water (5.1) in a 2 000 cm^3 beaker to the boil on a hotplate.

6.5.3 Pour the starch suspension prepared in 6.5.1 into the boiling water while stirring, and continue to boil for about 10 min.

6.5.4 Allow the solution to cool to ambient temperature and to settle, decant the clear portion into 500 cm^3 glass bottles (4.6) and stopper the bottles.

7 Standardization of the solutions

7.1 General

The potassium iodate/iodide solution is used as a primary standard to standardize the sodium thiosulfate solution. This sodium thiosulfate solution is then used as a secondary standard to standardize the iodine solution.

7.2 Sodium thiosulfate solution

7.2.1 After a resting period of 24 h after preparation, fill a glass (or digital) burette (see 4.5) with the unstandardized sodium thiosulfate solution. Flush 2 cm^3 to 3 cm^3 through the tip and adjust the mark (with a digital burette, flush the inlet and delivery tubes and zero the counter).

7.2.2 With a pipette (see 4.4), transfer exactly 20 cm^3 of potassium iodate/iodide solution (6.3) to a 250 cm^3 conical flask or to a digital-burette titration beaker, respectively.

7.2.3 Add about 3 cm^3 of 20 % sulfuric acid (6.4) to liberate the iodine. Mix thoroughly.

7.2.4 Titration with starch indicator (method A)

7.2.4.1 Add sodium thiosulfate from the burette until a pale-straw colour is observed. Wash the burette tip and the walls of the flask with water (5.1).

7.2.4.2 Add approximately 5 cm^3 of starch indicator (6.5) to the flask.

7.2.4.3 Continue to add sodium thiosulfate solution dropwise until the blue or blue-violet colour almost disappears. Wash the burette tip and the walls of the flask with water (5.1).

7.2.4.4 Slowly continue to add thiosulfate dropwise (or advance the counter of the digital burette by 0,01 cm^3 increments) until the blue colour is totally changed to colourless.