
**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*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 1304:2016](https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-2d2e570cd67c/iso-1304-2016)

[https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-
2d2e570cd67c/iso-1304-2016](https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-2d2e570cd67c/iso-1304-2016)



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 1304:2016

<https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-2d2e570cd67c/iso-1304-2016>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Normative references.....	1
3 Principle.....	1
4 Apparatus.....	2
5 Reagents.....	3
6 Preparation of solutions.....	3
7 Standardization of the solutions.....	6
7.1 General.....	6
7.2 Sodium thiosulfate solution.....	6
7.3 Iodine solution.....	7
8 Procedure.....	8
8.1 Conditions of test.....	8
8.2 Sample preparation.....	8
8.3 Iodine number determination.....	8
9 Expression of results.....	9
10 Verification using standard reference blacks.....	10
11 Test report.....	10
Annex A (informative) Precision.....	11
Annex B (informative) CAS numbers of reagents.....	13
Bibliography.....	14

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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. (standards.iteh.ai)

In case of dispute, the preferred method is method B (potentiometric titration).
ISO 1304:2016

2 Normative references

<https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-2d2e570cd67c/iso-1304-2016>

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 cm³, with a tolerance of $\pm 0,60$ cm³;
- b) 1 000 cm³, with a tolerance of $\pm 0,40$ cm³.

4.4 Repetitive dispenser, 25 cm³ capacity, calibrated to within $\pm 0,03$ cm³ accuracy, or **one-mark pipettes**, high precision, of capacities:

- a) 20 cm³, with a tolerance of $\pm 0,03$ cm³;
- b) 25 cm³, with a tolerance of $\pm 0,03$ cm³.

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 cm³ with distilled water, a temperature correction being made if necessary to show the true delivery at any volume used to within 0,01 cm³. 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 cm³ and 25 cm³ pipettes have calibration corrections of the same magnitude and in the same sense.

4.5 Digital burettes, with 0,01 cm³ increment counter and zero-reset control, calibrated to within $\pm 0,05$ cm³ accuracy, or **burettes** (for method A only), high precision, side-arm filling, graduated in 0,05 cm³ and with automatic zero, of capacities:

- a) 25 cm³, with a tolerance of $\pm 0,05$ cm³;
- b) 50 cm³, with a tolerance of $\pm 0,05$ cm³.

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 cm³ with distilled water, a temperature correction being made if necessary to show the true delivery at any volume used to within 0,01 cm³. 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 cm³ and 500 cm³.

4.7 Glass bottle, with ground-glass stopper, of capacity 2 000 cm³.

4.8 Amber-glass bottles, with ground-glass stoppers, of capacities 1 000 cm³ and 2 000 cm³.

4.9 Centrifuge tubes, of capacity 50 cm³, 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 (I₂).

iTeh STANDARD PREVIEW

5.3 Potassium iodide (KI). (standards.iteh.ai)

5.4 Potassium iodate (KIO₃).

ISO 1304:2016

<https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4->

5.5 Sodium thiosulfate pentahydrate (Na₂S₂O₃·5H₂O).

5.6 *n*-Amyl alcohol (C₅H₁₁OH).

5.7 Sulfuric acid (H₂SO₄), mass fraction 98 %, ρ = 1,84 Mg/m³.

5.8 Soluble starch (for method A only).

5.9 Salicylic acid (C₇H₆O₃) (for method A only).

6 Preparation of solutions

6.1 Iodine solution, 0,023 64 mol/dm³ (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 cm³ beaker.

6.1.2 Place about three-quarters of the KI in a clean 2 000 cm³ 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 cm³ beaker with enough water (5.1) to dissolve it.

6.1.5 Weigh, to the nearest 0,01 g, 12,00 g of iodine on the balance [4.1 a)] 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 cm³ 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.

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/dm³ (0,05 N).

NOTE Previous editions of this International Standard required a thiosulfate concentration of 0,039 4 mol/dm³ (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/dm³ (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/dm³ (0,039 4 N) is still permitted. In this case, the instructions for the preparation of the solution, the formulae used in its standardization and the formulae 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 cm³ volumetric flask (4.3).

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

6.2.4 Add 10 cm³ 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 6.1.10).

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

6.3 Potassium iodate/iodide solution, $c(\text{KIO}_3) = 0,008\ 33\ \text{mol/dm}^3$ (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 cm³ volumetric flask (see 4.3), dissolve 57,0 g (weighed to the nearest 0,1 g) of potassium iodide (5.3) in about 200 cm³ 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 cm³ of water (5.1) in a graduated cylinder and transfer to a 250 cm³ conical flask.

6.4.2 Measure out 25 cm³ 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 cm³ 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 cm³ beaker 2,5 g of powdered soluble starch (5.8), 2 mg of salicylic acid (5.9) and 25 cm³ of water (5.1). Stir with a glass rod.

6.5.2 Bring 1 000 cm³ of water (5.1) in a 2 000 cm³ 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 cm³ 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 (4.5) with the unstandardized sodium thiosulfate solution (6.2). Flush 2 cm³ to 3 cm³ through the tip and adjust the mark (with a digital burette, flush the inlet and delivery tubes and reset the counter to zero).

7.2.2 With a pipette (4.4), transfer exactly 20 cm³ of potassium iodate/iodide solution (6.3) to a 250 cm³ conical flask or to a digital-burette titration beaker, respectively.

7.2.3 Add about 3 cm³ 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 cm³ 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 cm³ increments) until the blue colour is totally changed to colourless.

7.2.4.5 Record the titration volume as V_1 to the nearest 0,025 cm³ (or 0,01 cm³).

To achieve maximum performance with a glass burette, it is recommended that a small magnifier be used to read the burette to the nearest 0,025 cm³.

7.2.4.6 Repeat the sequence 7.2.2 to 7.2.4.5 to give a duplicate determination.

7.2.4.7 Proceed to 7.2.6.

7.2.5 Potentiometric titration (method B) — Preferred method

7.2.5.1 Place the titration beaker in the automatic titrator, immerse the electrode in the solution and start the titration with thiosulfate solution in accordance with the manufacturer's instructions.

7.2.5.2 When the titration has finished, read the titration volume V_1 as displayed by the titrator to the nearest 0,01 cm³.

7.2.5.3 Repeat the sequence 7.2.2, 7.2.3, 7.2.5.1 and 7.2.5.2 to give a duplicate determination.

7.2.6 Calculate the concentration, in mol/dm³, of the sodium thiosulfate solution c_1 as follows:

$$c_1 = \frac{20 \times 6 \times 0,008\ 333}{V_1} \quad (1)$$

where

20 is the volume of iodate/iodide solution (6.3) titrated, expressed in cm³;

6 is a stoichiometric factor;

0,008 333 is the concentration of the iodate/iodide solution (6.3), expressed in mol/dm³;

V_1 is the average titration volume, in cm³, of the duplicate determinations.

7.3 Iodine solution

7.3.1 Fill a glass (or digital) burette with the standardized sodium thiosulfate solution as indicated in 7.2.1.

7.3.2 With a pipette (4.4), transfer exactly 20 cm³ of the unstandardized iodine solution (6.1) into a 250 cm³ conical flask or a digital-burette titration beaker, respectively.

7.3.3 Titrate the contents of the flask with the standardized sodium thiosulfate solution, following the procedure described in 7.2.4 or 7.2.5.

7.3.4 Calculate the concentration, in mol/dm³, of the iodine solution c_2 as follows:

$$c_2 = \frac{V_2 \times c_1}{2 \times 20} \quad (2)$$

ISO 1304:2016
<https://standards.iteh.ai/catalog/standards/sist/167158c7-e324-451f-b1f4-2d2e570cd67c/iso-1304-2016>

where

V_2 is the average titration volume, in cm³, of the duplicate determinations;

c_1 is the concentration of the standardized sodium thiosulfate solution, expressed in mol/dm³, as calculated in 7.2.6;

2 is a stoichiometric factor;

20 is the volume of the unstandardized iodine solution titrated, expressed in cm³.

7.3.5 To be acceptable, the concentration c_2 of the iodine solution shall be 0,023 64 mol/dm³ ± 0,000 05 mol/dm³.

If the concentration is outside this range, the solution can be adjusted as follows:

- if the solution is too strong, add water (5.1) (4,2 cm³ of water per 1 000 cm³ of solution for each 0,000 1 mol/dm³ over 0,023 64 mol/dm³);
- if the solution is too weak, add iodine (0,025 4 g of iodine per 1 000 cm³ of solution for each 0,000 1 mol/dm³ under 0,023 64 mol/dm³).

NOTE The iodine may be more conveniently dispensed from a concentrated solution.

In either case, it is essential to homogenize the adjusted solution thoroughly and to restart the entire standardization procedure (7.3).