BS ISO 1304:2016

BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of ISO 1304:2016. It supersedes BS ISO 1304:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Raw materials (including latex) for use in the rubber industry.

A list of organizations represented on this committee can be obtained on request to its secretary.

Published by BSI Standards Limited 2016

ISBN 978 0 580 89099 4

ICS 83.040.20

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 October 2016.

Amendments/corrigenda issued since publication

Date	Text affected

INTERNATIONAL STANDARD

ISO 1304

Fifth edition
2016-10-15

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)

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

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.

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.

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 0,01 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 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.

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 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 cm^3 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 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 6.1.10).

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

6.3 Potassium iodate/iodide solution, $c(\mathrm{KIO_3})$ = 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 (4.5) with the unstandardized sodium thiosulfate solution (6.2). 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 reset the counter to zero).

7.2.2 With a pipette (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.

7.2.4.5 Record the titration volume as V_1 to the nearest 0,025 cm^3 (or 0,01 cm^3).

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 cm^3.

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 cm^3.

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/dm^3, of the sodium thiosulfate solution c_1 as follows:

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

where

20	is the volume of iodate/iodide solution (6.3) titrated, expressed in cm^3;
6	is a stoichiometric factor;
0,008 333	is the concentration of the iodate/iodide solution (6.3), expressed in mol/dm^3;
V_1	is the average titration volume, in cm^3, 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 cm^3 of the unstandardized iodine solution (6.1) into a 250 cm^3 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/dm^3, of the iodine solution c_2 as follows:

$$c_2 = \frac{V_2 \times c_1}{2 \times 20} \tag{2}$$

where

V_2	is the average titration volume, in cm^3, of the duplicate determinations;
c_1	is the concentration of the standardized sodium thiosulfate solution, expressed in mol/dm^3, as calculated in 7.2.6;
2	is a stoichiometric factor;
20	is the volume of the unstandardized iodine solution titrated, expressed in cm^3.

7.3.5 To be acceptable, the concentration c_2 of the iodine solution shall be 0,023 64 mol/dm^3 ± 0,000 05 mol/ dm^3.

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 cm^3 of water per 1 000 cm^3 of solution for each 0,000 1 mol/dm^3 over 0,023 64 mol/dm^3);

— if the solution is too weak, add iodine (0,025 4 g of iodine per 1 000 cm^3 of solution for each 0,000 1 mol/dm^3 under 0,023 64 mol/dm^3).

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).

8 Procedure

8.1 Conditions of test

It is preferred that the test be carried out in a room having ambient conditions of either 23 °C ± 2 °C at (50 ± 5) % relative humidity or 27 °C ± 2 °C at (65 ± 5) % relative humidity.

It is recommended that the reagents and the apparatus be maintained at temperature equilibrium in the same room at least for a few hours before being used.

The test room shall be free from fumes or vapours which could contaminate the reagents and apparatus used, and therefore alter the results.

8.2 Sample preparation

Dry an adequate amount of the carbon black for 1 h at a temperature of 125 °C as specified in ISO 1126. Allow to cool to ambient temperature in a desiccator. Keep the dried carbon black in the desiccator until ready for testing. Standard reference blacks shall be dried to constant mass.

NOTE Pellets of carbon black need not be crushed. Unagitated, unpelletized carbon black may be densified, if desired, before drying.

8.3 Iodine number determination

8.3.1 Weigh, to the nearest 0,1 mg, a test portion of the dried carbon black info a centrifuge tube (4.9), taking the amount specified in Table 1.

Use the test portion mass corresponding to the expected iodine number. If the result falls either above or below the range shown for that mass, retest using the mass corresponding to the range in which the result fell.

8.3.2 The test portion masses given in Table 1 are valid only when 25 cm^3 of iodine solution is used as specified in 8.3.3. Different volumes of iodine solution and different test portion masses are permissible only if the iodine solution to test portion ratio is the same as that given in Table 1.

The test portion mass shall be limited to 1,000 g maximum when using a 50 cm^3 centrifuge tube. Should the test portion mass and corresponding volume of iodine solution be increased, then a tube of suitable capacity shall be used in order to ensure efficient shaking.

Table 1 — Test portion

Expected or nominal iodine adsorption number (IAN) g/kg	**Mass of test portion** m mg	**Ratio of volume of iodine solution to mass of test portion** cm^3/g
0 to 130,9	500	50:1
131,0 to 280,9	250	100:1
281,0 to 520,9	125	200:1
521,0 and above	62,5	400:1

8.3.3 With a pipette (or repetitive dispenser) (4.4), introduce 25 cm^3 of the 0,023 64 mol/dm^3 iodine solution into the centrifuge tube containing the test portion and cap immediately.

8.3.4 Place the tube in the mechanical shaker (4.10), with the longitudinal axis of the tube parallel to the direction of shaking, and shake for 1 min at 240 strokes/min.

8.3.5 Immediately after shaking, centrifuge at a speed above 105 rad/s (1 000 r/min) for 1 min for pelletized blacks or 3 min for unpelletized blacks, measured from the moment the centrifuge speed reaches 105 rad/s.

8.3.6 Immediately after centrifuging, decant the iodine solution completely, in one smooth motion, into a 50 cm^3 beaker, leaving the carbon black test portion at the bottom of the centrifuge tube. Immediately after decanting, use a pipette (4.4) to transfer exactly 20 cm^3 of the solution into a 250 cm^3 conical flask.

Alternatively, 20 cm^3 of the iodine solution may be pipetted directly from the centrifuge tube without disturbing the carbon black.

If the titration is not carried out immediately, decant the solution into a small vial and cap it immediately.

8.3.7 Titrate the 20 cm^3 of decanted iodine solution with the standardized 0,05 mol/dm^3 sodium thiosulfate solution following the procedure described in 7.2.4 (titration with starch indicator) or 7.2.5 (automatic titrator).

8.3.8 Repeating the sequence 8.3.3 to 8.3.7, carry out a blank determination on the iodine solution and record the titration volume V_B to the nearest 0,025 cm^3 (or 0,01 cm^3) (see 7.2.4.5).

8.3.9 Carry out a duplicate blank determination, and use the average of the two results in the calculations.

The duplicate blank determination needs to be run only once each day, unless new solutions are introduced during the day. For shift laboratories, it is recommended that duplicate blank determinations be made at the beginning of each shift.

8.3.10 If both the sodium thiosulfate and the iodine solutions are within acceptable limits, the blank average will be 18,91 cm^3 ± 0,05 cm^3. If not, the concentrations of one or both solutions shall be rechecked.

9 Expression of results

Calculate the iodine adsorption number (IAN), expressed in grams of iodine per kilogram of carbon black, to the nearest 0,1 g/kg, using Formula (3):

$$\mathrm{IAN} = \left(V_B - V_S\right) \times \frac{25}{V_B} \times c_2 \times 253{,}82 \times \frac{1}{m} \tag{3}$$

where

V_B	is the volume, in cm^3, of sodium thiosulfate solution required for the blank;
V_S	is the volume, in cm^3, of sodium thiosulfate solution required for the test portion;
c_2	is the concentration of the iodine solution, expressed in mol/dm^3;
m	is the mass, in g, of the test portion;
25	is the volume, in cm^3, of iodine solution in contact with the carbon black (from calibrated pipette or dispenser);
253,82	is the molecular mass of iodine (I_2).

10 Verification using standard reference blacks

It is recommended that the proper execution of the procedure be checked by using standard reference blacks (SRBs) (see ASTM D4821 for standard values and the range for each of them).

If the values for the SRBs do not fall within the accepted ranges, then new solutions shall be prepared and tested, including the primary standard solution prepared in 6.3.

11 Test report

The test report shall include the following particulars:

a) a reference to this International Standard, i.e. ISO 1304:2016;

b) all details necessary for complete identification of the sample;

c) the test conditions used;

d) the result obtained, expressed as specified in Clause 9;

e) details of any deviations from the method specified;

f) the date of the test.

Annex A
(informative)

Precision

A.1 General

A.1.1 The precision of this test method was determined in accordance with ISO/TR 9272:2005, level 2, method. Refer to ISO/TR 9272 for terminology and other statistical details.

A.1.2 The precision results give an estimate of the precision to be expected. The precision parameters shall not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

A.2 Precision details

A.2.1 A type 1 precision interlaboratory-trials programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions. For method A (titration with starch indicator), seven laboratories tested three carbon blacks twice on each of two different days; therefore, $P = 7$, $q = 3$ and $n = 4$. For method B (potentiometric titration), 23 laboratories tested three carbon black samples twice on each of two different days; therefore, $P = 23$, $q = 3$ and $n = 4$. A test result is the value obtained from a single determination. Acceptable difference values were not measured.

A.2.2 The results of the precision calculations are given in Table A.1 (method A) and Table A.2 (method B), with the materials arranged in descending order of mean iodine adsorption number. Outliers have been removed. The number of laboratories remaining after outlier deletion is marked in the tables.

A.3 Precision results

A.3.1 General

The symbols used in Tables A.1 and A.2 are defined as follows.

— s_r is the within-laboratory standard deviation.

— r is the repeatability (in measurement units).

— (r) is the repeatability (in percent).

— s_R is the between-laboratory standard deviation.

— R is the reproducibility (in measurement units).

— (R) is the reproducibility (in percent).

Table A.1 — Precision data — Method A (titration with starch indicator)

Material	Number of laboratories	Mean IAN	Within laboratory			Between laboratories		
		g/kg	s_r	r	(r)	s_R	R	(R)
A (N115)	4	158,21	0,268	0,758	0,479	1,302	3,760	2,376
B (N772)	5	32,67	0,170	0,482	1,475	0,607	1,784	5,460
C (N330)	5	80,77	0,432	1,221	1,512	0,678	2,274	2,816
Average	5	90,55	—	—	—	—	—	—
Pooled values	—	—	0,309	0,875	1,250	0,917	2,738	3,803

Table A.2 — Precision data — Method B (potentiometric titration)

Material	Number of laboratories	Mean IAN	Within laboratory			Between laboratories		
		g/kg	s_r	r	(r)	s_R	R	(R)
A (N115)	19	160,55	0,570	1,612	1,005	2,519	7,306	4,551
B (N772)	20	33,38	0,400	1,131	3,387	0,911	2,814	8,430
C (N330)	19	82,47	0,344	0,972	1,178	1,360	3,969	4,812
Average	19	92,13	—	—	—	—	—	—
Pooled values	—	—	0,448	1,268	2,150	1,734	5,068	6,190

NOTE Statistical analysis has shown that there is a significant difference between the mean level obtained with method A and method B. Method A (starch indicator) gives roughly 1 % lower values than method B (potentiometric titration). The reason for this phenomenon is not yet clear. When comparing the precision data for the two methods, it should be borne in mind that the number of participating laboratories, i.e. the statistical basis, was different: 7 laboratories used method A, whereas 23 laboratories used method B.

The precision for the pooled values of the iodine adsorption number may be expressed as given in A.3.2 and A.3.3.

A.3.2 Repeatability

The repeatability in percent (*r*) for the iodine adsorption number has been established as 1,25 % for method A and 2,15 % for method B. Two single test results (or determinations) that differ by more than 1,25 % (method A) or 2,15 % (method B) shall be considered suspect and dictate that some appropriate investigative action be taken.

A.3.3 Reproducibility

The reproducibility in percent (*R*) for the iodine adsorption number has been established as 3,80 % for method A and 6,19 % for method B. Two single test results (or determinations), produced in separate laboratories, that differ by more than 3,80 % (method A) or 6,19 % (method B) shall be considered suspect and dictate that some appropriate investigative action be taken.

A.4 Bias

In test method terminology, bias is the difference between an average test value and a reference (true) test property value. Reference values do not exist for this test method since the value of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

Annex B
(informative)

CAS numbers of reagents

Subclause	Chemical name	Formula	CAS number [a]
5.2	Iodine	I_2	7553–56–2
5.3	Potassium iodide	KI	7681–11–0
5.4	Potassium iodate	KIO_3	7758–05–6
5.5	Sodium thiosulfate	$Na_2S_2O_3\cdot 5H_2O$	10102–17–7
5.6	*n*-Amyl alcohol	$C_5H_{11}OH$	71–41–0
5.7	Sulfuric acid	H_2SO_4	7664–93–9
5.8	Soluble starch	$(C_6H_{10}O_5)_n$	9005–84–9
5.9	Salicylic acid	$C_7H_6O_3$	00069–72–7

[a] CAS = Chemical Abstracts Service.

Bibliography

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

[2] ASTM D4821, *Standard Guide for Carbon Black — Validation of Test Method Precision and Bias*

ICS 83.040.20
Price based on 14 pages

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.

Standards purchased in hard copy format:

- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services
Tel: +44 345 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 345 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK