INTERNATIONAL STANDARD

ISO 1304

Third edition
1999-03-15

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

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de l’indice d’adsorption d’iode — Méthode titrimétrique

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Reference number
ISO 1304:1999(E)

Foreword

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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.

International Standard 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 third edition cancels and replaces the second edition (ISO 1304:1985), which has been technically revised.

Annex A of this International Standard is for information only.

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

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.

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1 Scope

This International Standard specifies a titrimetric method for the determination of the iodine adsorption number of carbon blacks for use in the rubber industry.

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.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

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

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

ISO 1126:1992, *Rubber compounding ingredients — Carbon black — Determination of loss on heating.*

ISO TR 6809:1996, *Rubber compounding ingredients — Carbon black — Standard reference blacks.*

ISO/TR 9272:1986, *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, 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 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,

or **repetitive dispenser,** 25 cm^3 capacity, calibrated to within ± 0,03 cm^3 accuracy.

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 Burettes, 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,

or **digital burettes,** with 0,01 cm^3 increment counter and zero-reset control, calibrated to within ± 0,05 cm^3 accuracy.

If class A burettes in accordance with ISO 385-1 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 Amber-glass stoppered bottles, with ground-glass stoppers, of capacities 1 000 cm^3 and 2 000 cm^3.

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

Cork, rubber or metal stoppers shall not be used.

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

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

4.11 Desiccator.

4.12 Magnetic stirrers and **spin bars.**

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5 Reagents

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

5.1 Water, deionized, having a pH-value within the range 6,5 to 7,2 (preferably close to 6,9).

NOTE It is recommended that freshly reboiled water be used, obtained from water distilled then polished with a mixed bed of ion-exchange materials and a 0,2 µm to 4 µm membrane filter.

Deionized water shall be protected from atmospheric contamination and from dissolution of container and tubing materials. Extreme care shall be exercised in handling the water. Containers and tubing shall be made of polytetrafluoroethylene, solid tin, quartz, 18-8 stainless steel, polyethylene or some other material proven to be sufficiently resistant to chemical attack.

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), 98 % (m/m), ρ = 1,84 Mg/m^3.

5.8 Soluble starch.

5.9 Salicylic acid ($C_7H_6O_3$).

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6 Preparation of solutions[1]

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.

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 (see 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 exactly[2] 12,000 0 g of iodine on the balance (4.1) in a weighing bottle fitted with a ground-glass stopper. Record the mass to the nearest 0,1 mg. 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.

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[1] It is easier to weigh approximate masses to the nearest 0,1 mg and calculate the results rather than attempt to weigh test portion masses exactly.

[2] In this case, it is not sufficient to calculate a correction factor, since the test result depends on the concentration of the iodine solution (see 7.3.5).

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 (4.12) 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 (4.7) and let it stand overnight prior to any use.

6.2 Sodium thiosulfate solution, 0,039 4 mol/dm^3 (0,039 4 N).

6.2.1 Weigh, to the nearest 0,005 g, 19,560 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 an amber-glass bottle (4.7).

6.3 Potassium iodate/iodide solution, $c(KIO_3)$ = 0,006 567 mol/dm^3 (0,039 40 N).

6.3.1 Dry an adequate quantity of potassium iodate in the oven (4.2) at 125 °C for 1 h. Allow to cool to ambient temperature in the desiccator (4.11).

6.3.2 In a 1 000 cm^3 volumetric flask (see 4.3), dissolve 45,0 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, 1,405 4 g of the freshly dried potassium iodate (6.3.1) 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 (4.7).

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, 20 % (V/V) solution.

6.4.1 Measure out 180 cm^3 of water (5.1) in a graduated cylinder and transfer to a 250 cm^3 conical flask.

6.4.2 Measure out 28 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.

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

6.5 Starch indicator, 0,25 % solution.

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 cap 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 25 cm^3 of potassium iodate/iodide solution (6.3) to a 250 cm^3 conical flask.

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

7.2.4 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).

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

7.2.6 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).

7.2.7 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.

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

NOTE 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.

7.2.9 Repeat the sequence 7.2.2 to 7.2.8 to give a duplicate determination.

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7.2.10 Calculate the concentration, in mol/dm^3, of the sodium thiosulfate solution c_1 (or normality n_1) as follows:

$$c_1 = \frac{25 \times 6 \times 0{,}006\,567}{V_1} \quad \left(\text{or } n_1 = \frac{25 \times 0{,}039\,40}{V_1} \right)$$

where

V_1 is the average titration volume, in cm^3, of the duplicate determinations;

6 is the stoichiometric factor;

0,006 567 is the concentration of the potassium iodate/iodide solution (6.3), expressed in mol/dm^3;

0,039 40 is the concentration of the potassium iodate/iodide solution (6.3), expressed as a normality.

7.2.11 To be acceptable, the concentration c_1 of the sodium thiosulfate solution shall be:

0,039 40 mol/dm^3 ± 0,000 08 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) (2,5 cm^3 of water per 1 000 cm^3 of solution for each 0,000 1 mol/dm^3 over 0,039 4 mol/dm^3) (or in terms of normality: 2,5 cm^3 of water per 1 000 cm^3 of solution for each 0,000 1 N over 0,039 4 N);
- if the solution is too weak, add solid sodium thiosulfate (0,025 g of thiosulfate per 1 000 cm^3 of solution for each 0,000 1 mol/dm^3 under 0,039 4 mol/dm^3) (or in terms of normality: 0,025 g of thiosulfate per 1 000 cm^3 of solution for each 0,000 1 N under 0,039 4 N).

NOTE Any possible adjustment has to take into consideration the actual volume of the solution to be adjusted.

In either case, it is essential to homogenize the adjusted solution thoroughly and to repeat the entire standardization procedure (7.2).

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 (see 4.4), transfer exactly 25 cm^3 of the unstandardized iodine solution into a 250 cm^3 conical flask.

7.3.3 Titrate the contents of the flask with the standardized sodium thiosulfate solution from the burette, following the procedure described in 7.2.4 to 7.2.9, and record the titration volume V_2 to the nearest 0,025 cm^3 (or 0,01 cm^3) (see note to 7.2.8).

7.3.4 Calculate the concentration, in mol/dm^3, of the iodine solution c_2 (or normality n_2) as follows:

$$c_2 = \frac{V_2 \times c_1}{2 \times 25} \quad \left(\text{or } n_2 = \frac{V_2 \times n_1}{25} \right)$$

where

c_1 is the concentration of the standardized sodium thiosulfate solution, expressed in mol/dm^3, as calculated in 7.2.10;

n_1 is the concentration of the standardized sodium thiosulfate solution, expressed as a normality, as calculated in 7.2.10;

V_2 is the average titration volume, in cm^3, of the duplicate determinations;

2 is the stoichiometric factor.

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7.3.5 To be acceptable, the concentration c_2 of the iodine solution shall be:

0,023 64 mol/dm^3 ± 0,000 025 mol/dm^3 (0,047 28 N ± 0,000 05 N)

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) (or in terms of normality: 2,1 cm^3 of water per 1 000 cm^3 of solution for each 0,000 1 N over 0,047 28 N);
- 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) (or in terms of normality: 0,012 7 g of iodine per 1 000 cm^3 of solution for each 0,000 1 N under 0,047 28 N).

NOTE Any possible adjustment has to take into consideration the actual volume of solution to be adjusted.

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 into a centrifuge tube (4.8), 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.

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Table 1

Expected or nominal iodine absorption 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) (see 4.4), introduce 25 cm^3 of the 0,023 64 mol/dm^3 (0,047 28 N) iodine solution into the centrifuge tube containing the test portion and cap immediately.

8.3.4 Place the tube in the mechanical shaker (4.9), 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 (see 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 Using a glass (or digital) burette (see 4.5), titrate with the standardized 0,039 40 mol/dm^3 (0,039 40 N) sodium thiosulfate solution the 20 cm^3 of decanted iodine solution, following the procedure described in 7.2.4 to 7.2.8, and record the titration volume V_S to the nearest 0,025 cm^3 (or 0,01 cm^3) (see note to 7.2.8).

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 note to 7.2.8).

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

NOTE The duplicate blank determination needs to be run only once each day, unless new solutions are introduced during the day.

8.3.10 If both the sodium thiosulfate and the iodine solutions are within acceptable limits, the blank average will be 24,00 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 the equation:

$$\text{IAN} = (V_B - V_S) \times \frac{25}{V_B} \times c_2 \times 253{,}82 \times \frac{1}{m}$$

$$= (V_B - V_S) \times \frac{25}{V_B} \times n_2 \times 126{,}91 \times \frac{1}{m}$$

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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;

n_2 is the concentration of the iodine solution, expressed as a normality;

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 molar mass of iodine;

126,91 is the equivalent mass of iodine.

10 Verification using standard reference blacks

It is recommended that the proper execution of the procedure be checked by using standard reference blacks (see ISO/TR 6809 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 Precision and bias

11.1 Precision

11.1.1 The precision of this test method was determined in accordance with the ISO/TR 9272. Refer to this document for terminology and other statistical details.

11.1.2 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.

11.1.3 A type 1 precision interlaboratory-trials programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions. Ten laboratories tested six standard reference blacks (A4 to F4) twice on each of two different days. Therefore, $p = 10$, $q = 6$ and $n = 4$. The test result is the value obtained from a single determination. Acceptable difference values were not measured.

11.1.4 The results of the precision calculations are given in table 2, with the materials arranged in ascending order of the mean iodine adsorption number.

11.1.5 The precision for the pooled values of the iodine adsorption number may be expressed as follows:

11.1.5.1 Repeatability

The repeatability r for the iodine adsorption number has been established as 1,08 g/kg. Two single test results (or determinations) that differ by more than 1,08 g/kg shall be considered suspect and dictate that some appropriate investigative action be taken.

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11.1.5.2 Reproducibility

The reproducibility R for the iodine adsorption number has been established at 3,35 g/kg. Two single test results (or determinations), produced in separate laboratories, that differ by more than 3,35 g/kg shall be considered suspect and dictate that some appropriate investigative action be taken.

11.2 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.

Table 2 — Precision data

Material	**Mean IAN**	**Within laboratory**			**Between laboratories**		
	g/kg	s_r	r	(r)	s_R	R	(R)
SRB-D4	26,49	0,519	1,470	5,549	1,076	3,046	11,498
SRB-E4	35,98	0,091	0,540	1,501	0,765	2,166	6,021
SRB-F4	37,65	0,352	0,996	2,646	0,966	2,735	7,265
SRB-B4	79,26	0,324	0,916	1,155	0,947	2,680	3,381
SRB-A4	82,67	0,285	0,806	0,975	1,240	3,510	4,246
SRB-C4	121,10	0,504	1,427	1,178	1,822	5,155	4,257
Pooled or averaged values	63,86	0,38	1,08	1,69	1,19	3,35	5,25

The symbols used are defined as follows:

s_r = within-laboratory standard deviation
r = repeatability (in measurement units)
(r) = repeatability (in percent)
s_R = between-laboratory standard deviation
R = reproducibility (in measurement units)
(R) = reproducibility (in percent)

12 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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.

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