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Carbon black for use in the rubber industry — Determination of iodine adsorption number

Noir de carbone destiné à l'industrie des élastomères — Détermination de l'indice d'adsorption d'iode

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Australia	Ireland	Sri Lanka
Austria	Italy	Sweden
Canada	Netherlands	Switzerland
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Germany	Romania	U.S.A.
Hungary	South Africa, Rep. of	U.S.S.R.
India	Spain	

No Member Body expressed disapproval of the document.

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Carbon black for use in the rubber industry — Determination of iodine adsorption number

1 SCOPE AND FIELD OF APPLICATION

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

NOTE — The iodine adsorption of carbon black is significantly depressed in the presence of a high content of volatile or solvent extractable matters; the iodine adsorption number therefore should not be considered as providing a measure of the specific surface area of carbon black.

2 REFERENCE

ISO/R 1126, *Determination of loss on heating of carbon black for the rubber industry.*

3 PRINCIPLE

A sample 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 thiosulphate. From this titration value and the sample mass, the iodine adsorption number of the carbon black is calculated.

4 REAGENTS

All reagents shall be of recognized analytical quality, and distilled water, having a pH value within the range of 6,5 to 7,2 (preferably close to 6,9), shall be used whenever water is specified.

NOTE — It is recommended that freshly re-boiled water be used, obtained from water distilled then polished with a mixed bed of ion-exchange materials.

Distilled water shall be protected from atmospheric contamination and from solution of container and tubing materials. Extreme care must be exercised in handling the distilled water. Containers and tubing shall be made of polytetrafluorethylene, block tin, quartz, 18-8 stainless steel, polyethylene, or other material proven to be sufficiently resistant to chemical attack.

4.1 Starch indicator solution, prepared as follows :

Stir 2,5 g of powdered water-soluble starch and 2 mg of mercury(II) iodide (HgI_2) in 25 cm^3 of water in a 50 cm^3 beaker.

Add the starch/mercury(II) iodide suspension immediately to 1 l of boiling water while stirring.

Boil the resulting starch solution for at least 5 min to ensure proper solution.

Allow to cool to ambient temperature and to settle, and decant the clear portion into glass stoppered bottles.

4.2 Potassium iodate/iodide solution, 0,039 41 N, prepared as follows :

Dry an adequate quantity of potassium iodate in an oven for 1 h at a temperature of 120 ± 5 °C. Allow to cool to ambient temperature in a desiccator.

Dissolve 45 g of potassium iodide in about 200 cm^3 of water in a 1 000 cm^3 one-mark volumetric flask.

Add 1,405 8 g, weighed to the nearest 0,1 mg, of the freshly dried potassium iodate. When solution is complete, dilute to 1 000 cm^3 with water.

4.3 Sodium thiosulphate solution, 0,039 41 N.

4.3.1 *Preparation*

Dissolve 9,79 g of sodium thiosulphate pentahydrate ($NA_2S_2O_3.5H_2O$), weighed to the nearest 0,005 g, in approximately 500 cm^3 of water in a 1 000 cm^3 one-mark volumetric flask.

Add 5 cm^3 of pentanol (*n*-amyl alcohol) and shake the solution in the flask to mix thoroughly.

Dilute to 1 000 cm^3 with water. Shake the solution in the flask vigorously to ensure uniform dilution.

4.3.2 *Standardization*

After a resting period of 24 h, standardize the sodium thiosulphate solution with the potassium iodate/iodide solution (4.2) as follows :

Pipette 25 cm^3 of the iodate/iodide solution (4.2) into a 250 cm^3 conical flask and add 3 cm^3 of approximately 4 N sulphuric acid solution to liberate the iodine. Add the sodium thiosulphate from a burette until a pale straw colour is observed. Add approximately 5 cm^3 of starch solution (4.1) and continue titrating until 1 drop of the sodium thiosulphate solution causes the blue colour to change to colourless. Read the burette to the nearest 0,01 cm^3.

Calculate the normality (N_1) of the sodium thiosulphate from the formula

$$N_1 = \frac{25 \times 0{,}039\ 41}{V_1}$$

where V_1 is the volume, in cubic centimetres, of sodium thiosulphate solution used in the titration.

NOTE – The normality factor is $25/V_1$, but the normality factor may be adjusted to 1,00 if so desired.

4.4 Iodine solution, 0,047 28 N, with 9,5 parts of potassium iodide to 1,0 part of iodine.

4.4.1 *Preparation*

Weigh 57,00 g of potassium iodide to the nearest 0,01 g and transfer into a 1 000 cm^3 volumetric flask.

Add approximately 30 cm^3 of water to dissolve it. Weigh rapidly 6,01 g of iodine to the nearest 0,005 g, transfer rapidly to the same flask and slowly dilute to 1 000 cm^3 with water.

4.4.2 *Standardization*

After a resting period of 24 h, standardize the iodine solution with the already standardized sodium thiosulphate solution (4.3) as follows :

Pipette exactly 25 cm^3 of the unstandardized iodine solution with an accurate pipette into a 250 cm^3 flask.

Titrate the contents of the flask with the previously standardized sodium thiosulphate solution (4.3). When the yellow colour of the iodine has almost disappeared, add about 1 cm^3 of starch solution (4.1) and continue the titration until the blue colour disappears.

Calculate the normality (N_2) of the iodine solution from the formula

$$N_2 = \frac{V_2 \times N_1}{25}$$

where V_2 is the volume, in cubic centimetres, of sodium thiosulphate solution used in the titration.

Determine the volume of distilled water necessary to add to the stock solution to adjust it to the desired normality from the formula

$$\frac{V_3 \times N_2}{N_3} - V_3$$

where

V_3 is the remaining volume, in cubic centimetres, of iodine solution before adjustment (see note 1);

N_2 is the normality of the iodine solution before adjustment;

N_3 is the desired normality of the iodine solution (0,047 28 N).

Add the calculated volume of distilled water to the stock solution, stopper the bottle and shake to ensure uniform dilution.

NOTES

1 Measure the amount of iodine solution taken from the stock solution before standardization so that the remaining volume will be known.

2 The iodine solution shall be standardized to ± 0,000 05 N, i.e. the normality shall be within the following range : 0,047 23 to 0,047 33 N.

3 All reagents shall be stored in stoppered amber glass bottles in a dark cabinet before being used.

Iodine and sodium thiosulphate solutions shall be preferably prepared, standardized and stored at an ambient temperature either 23 ± 2 °C or 27 ± 2 °C (see also clause 7).

4 Standardized iodine solution shall be discarded when its normality deviates by more than 0,000 4 from the stated normality.

5 APPARATUS

5.1 Analytical balance, accurate to 0,1 mg.

5.2 Oven, preferably of the gravity convection type, capable of maintaining a temperature of 120 ± 5 °C.

5.3 Oven, preferably of the gravity convection type, capable of maintaining a temperature of 105 ± 2 °C.

5.4 Stoppered volumetric flasks, capacity 1 000 cm^3 with a tolerance of 0,20 cm^3.

5.5 Volumetric pipettes, high precision :

1) 20 cm^3, with a tolerance of ± 0,015 cm^3.

2) 25 cm^3, with a tolerance of ± 0,02 cm^3.

5.6 Burettes, high precision :

1) 25 cm^3, with a tolerance of ± 0,025 cm^3.

2) 50 cm^3, with a tolerance of ± 0,04 cm^3.

NOTE – High precision burettes and 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 8.2, 8.4 and 8.6) it is recommended that the 20 cm^3 and 25 cm^3 pipettes have a calibration corrections of the same magnitude and of the same sense.

5.7 Stoppered bottles, with ground glass stoppers, capacity 250 cm^3 and 500 cm^3.

5.8 Stoppered bottles, amber glass, with ground stoppers, capacity 1 000 cm^3.

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

NOTE – Cork, rubber or aluminium stoppers shall not be used.

5.10 Mechanical shaker, capable of vigorous shaking at a rate of approximately 240 strokes per minute.

5.11 Centrifuge, capable of speeds above 1 000 rev/min.

6 SAMPLE PREPARATION

Dry an adequate amount of the sample of carbon black for 1 h at a temperature of 105 ± 2 °C as described in ISO/R 1126. Allow to cool to ambient temperature in a desiccator. Keep the dried sample in the desiccator until ready for testing.

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

7 CONDITIONS OF TEST

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

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

The testing room shall be free from fumes or vapours which would contaminate reagents and testing equipment used, and therefore alter the results.

8 PROCEDURE

8.1 In the presence of a desiccating agent, weigh, to the nearest 0,1 mg, as rapidly as possible into the centrifuge tube, the following test portion of the dried carbon black :

– 0,500 g for carbon blacks with iodine adsorption numbers not greater than 135;

– 0,250 g for carbon blacks with iodine adsorption numbers greater than 135 but not greater than 500;

– 0,125 g for carbon blacks with iodine adsorption numbers greater than 500.

NOTE – If the iodine adsorption numbers are close to 135 or 500, the actual sample mass used may be chosen on the basis of the accepted grade classification of the carbon black. In this case the grade classification adopted shall also be included in item d) of the Test Report (clause 10).

Stopper the centrifuge tube until the addition of the iodine solution.

8.2 Using a high precision volumetric pipette (5.5), add exactly 25 cm^3 of the iodine solution (4.4) to the centrifuge tube containing the test portion and stopper immediately. Fix the centrifuge tube to the holder of the mechanical shaker (5.10). Allow the iodine/carbon black mix to shake for 1 min at about 240 vigorous strokes per minute.

8.3 Immediately after shaking, centrifuge at a speed above 1 000 rev/min, 1 min for pelletized black or 3 min for unpelletized black, measured from the moment the centrifuge speed reaches 1 000 rev/min.

NOTE – Keep the centrifuge tube stoppered until removing the solution.

8.4 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 high precision pipette (5.5) 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.

8.5 Using a high precision burette (5.6), titrate the solution with the sodium thiosulphate solution (4.3) until a pale yellow colour remains. Add approximately 5 cm^3 of the starch indicator solution (4.1) and continue titrating until 1 drop of the sodium thiosulphate solution causes the blue colour to change to colourless. Read the burette to the nearest 0,01 cm^3.

8.6 Carry out a blank determination on 25 cm^3 of the iodine solution (4.4), and after shaking and centrifuging pour into a beaker from which 20,00 cm^3 are drawn for titration, or pipette directly from the tube as described in 8.4.

9 EXPRESSION OF RESULTS

Calculate the iodine adsorption number (I) (in milligrams of iodine per gram of carbon black), to the nearest 0,1 mg/g, from the formula

$$I = (V_4 - V_5) \times 126{,}9 \times N_1 \times \frac{5}{4\,m}$$

i.e. $I = 158{,}6\,(V_4 - V_5) \times \frac{N_1}{m}$

where

V_4 is the volume, in cubic centimetres, of sodium thiosulphate solution required for the titration of the blank iodine solution;

V_5 is the volume, in cubic centimetres, of sodium thiosulphate solution required for the titration of the iodine solution which has been in contact with the test portion of carbon black;

N_1 is the normality of the sodium thiosulphate solution;

m is the mass, in grams, of the test portion of carbon black.

NOTE – When using 0,5 g of carbon black the formula simplifies to :

$I = (V_4 - V_5) \times 12{,}5 \times$ normality factor (see 4.3.2)

10 TEST REPORT

The test report shall include the following particulars :

a) the proper identification of the sample;

b) the conditions of test;

c) the mass of test portion used;

d) the results obtained;

e) a reference to this International Standard.