TC 45

International Standard 1304

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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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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

1 Scope and field of application

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

NOTE — The iodine adsorption number is related to the surface area of carbon black, and is generally in agreement with nitrogen surface area. However, it 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 1126, *Carbon black for use in the rubber industry — Determination of loss on heating.*

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

4 Reagents

During the analysis, use only reagents of recognized analytical quality, and only distilled water, having a pH value within the range of 6,5 to 7,2 (preferably close to 6,9).

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 and a 0,2 to 4 µm membrane filter.

Distilled water shall be protected from atmospheric contamination and from solution of container and tubing materials. Extreme care shall 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.

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 dm^3 of boiling water while stirring.

Boil the resulting starch solution for at least 10 min to ensure proper solution.

Allow to cool to ambient temperature and to settle, and decant the clear portion into glass stoppered bottles (5.7).

4.2 Potassium iodate/iodide, standard reference solution, $c(1/2\ I_2) = 0{,}039\ 41\ mol/dm^3$.

Dry an adequate quantity of potassium iodate in an oven for 1 h at a temperature of 125 ± 1 °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 thiosulfate solution, $c(1/2\ Na_2S_2O_3) = 0{,}039\ 41\ mol/dm^3$.

4.3.1 Preparation

Dissolve 9,79 g of sodium thiosulfate 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 thiosulfate 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 20 % (m/m) sulfuric acid solution to liberate the iodine. Add the sodium thiosulfate from a burette until a pale straw colour is observed. Add approximately 5 cm^3 of the starch solution (4.1) and continue titrating until 1 drop of the sodium thiosulfate solution causes the blue colour to change to colourless. Read the burette to the nearest 0,01 cm^3.

Calculate the concentration c_1 of the sodium thiosulfate solution using the equation

$$c_1 = \frac{25 \times 0{,}039\ 41}{V_1}$$

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

NOTE — The concentration factor is $25/V_1$, but it may be adjusted to 1,00 if so desired.

4.4 Iodine, standard volumetric solution, $c(1/2\ I_2)$ = 0,047 28 mol/dm^3, with 9,5 parts of potassium iodide to 1,0 part of iodine.

4.4.1 Preparation

Weigh, to the nearest 0,01 g, 57,00 g of potassium iodide, and transfer to a 1 000 cm^3 one-mark 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 thiosulfate solution (4.3) as follows :

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

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

Calculate the concentration c_2 of the iodine solution using the equation

$$c_2 = \frac{V_2 \times c_1}{25}$$

where

V_2 is the volume, in cubic centimetres, of sodium thiosulfate solution used in the titration;

c_1 is as defined in 4.3.2.

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

$$\frac{V_3 \times c_2}{c_3} - V_3$$

where

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

c_2 is the concentration of the iodine solution before adjustment;

c_3 is the desired concentration of the iodine solution (0,047 28 mol/dm^3).

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 mol/dm^3, i.e. the concentration shall be within the following range : 0,047 23 to 0,047 33 mol/dm^3.

3 All reagents should be stored in stoppered amber glass bottles (5.8) in a dark cabinet before being used.

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

4 Standardized iodine solution should be discarded when its concentration deviates by more than 0,000 4 mol/dm^3 from the stated concentration.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Analytical balance, accurate to 0,1 mg.

5.2 Oven, preferably of the gravity convection type, capable of being controlled at 125 ± 1 °C.

5.3 Oven, preferably of the gravity convection type, capable of being controlled at 105 ± 2 °C.

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

5.5 Volumetric pipettes, high precision :

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

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

5.6 Burettes, high precision :

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

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

NOTE — High-precision burettes and pipettes should 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 calibration corrections of the same magnitude and of the same sense.

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

5.8 Stoppered bottles, amber glass, with ground stoppers, of capacity 1 dm^3.

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

NOTE — Cork, rubber or aluminium stoppers should 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 r/min.

6 Sample preparation

Dry an adequate amount of the sample of carbon black for 1 h at a temperature of 105 ± 2 °C or 125 ± 1 °C as specified in ISO 1126. Allow to cool to ambient temperature in a desiccator. Keep the dried sample in the desiccator until ready for testing.

NOTE — Pellets of 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 ° 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 a centrifuge tube (5.9), 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 should 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 r/min, 1 min for pelletized black or 3 min for unpelletized black, measured from the moment the centrifuge speed reaches 1 000 r/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 iodine solution with the sodium thiosulfate 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 thiosulfate 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 specified in 8.4.

9 Expression of results

Calculate the iodine adsorption number IAN, expressed in milligrams of iodine per gram of carbon black, to the nearest 0,1 mg/g, using the equation

$$IAN = (V_4 - V_5) \times 126{,}9\, c_1 \times \frac{5}{4\,m}$$

$$= 158{,}6\,(V_4 - V_5) \times \frac{c_1}{m}$$

where

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

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

c_1 is the concentration, expressed in moles per cubic decimetre, of the sodium thiosulfate solution, calculated as in 4.3.2;

m is the mass, in grams, of the test portion of carbon black (8.1).

NOTE — When using 0,5 g of carbon black, the equation simplifies to

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

10 Test report

The test report shall include the following particulars :

a) a reference to this International Standard;

b) the proper identification of the sample;

c) the conditions of test;

d) the mass of test portion used;

e) the results obtained;

f) the drying temperature used.