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International Standard



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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

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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.

International Standard ISO 1304 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

ISO 1304 was first published in 1974. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

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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, $c(1/2 \text{I}_2) = 0,039 41 \text{ mol/dm}^3$.

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, $c(1/2 \text{Na}_2\text{S}_2\text{O}_3) = 0,039 41 \text{ mol/dm}^3$.

4.3.1 Preparation

Dissolve 9,79 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$), 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 cm³ of the iodate/iodide solution (4.2) into a 250 cm³ conical flask and add 3 cm³ 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 cm³ 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 cm³.

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/dm³, 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 cm³ one-mark volumetric flask.

Add approximately 30 cm³ 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 cm³ 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 cm³ of the unstandardized iodine solution with an accurate pipette into a 250 cm³ 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 cm³ 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/dm³).

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 $\pm 0,000\ 05$ mol/dm³, i.e. the concentration shall be within the following range : 0,047 23 to 0,047 33 mol/dm³.

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/dm³ 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 cm³ with a tolerance of 0,20 cm³.