
INTERNATIONAL STANDARD



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Carbon black for the rubber industry — Determination of total sulphur content

Noir de carbone pour l'industrie des élastomères — Dosage du soufre total

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45 has reviewed ISO Recommendation R 1138 and found it technically suitable for transformation. International Standard ISO 1138 therefore replaces ISO Recommendation R 1138-1969 to which it is technically identical.

ISO Recommendation R 1138 was approved by the Member Bodies of the following countries :

Australia	Hungary	Portugal
Austria	India	Spain
Brazil	Iran	Sweden
Canada	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Japan	United Kingdom
France	Netherlands	U.S.A.
Germany	New Zealand	U.S.S.R.
Greece	Poland	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1138 into an International Standard.

Carbon black for the rubber industry — Determination of total sulphur content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two methods for the determination of the total sulphur in all types of carbon black for use in the rubber industry :

- Method A, using an oxygen bomb calorimeter;
- Method B, using a combustion furnace.

SECTION ONE : METHOD A — OXYGEN BOMB CALORIMETER

2 PRINCIPLE

Ignition of a weighed test portion of dried carbon black in an oxygen bomb calorimeter. After the bomb is opened, washing of the inner surfaces with water, and collection of the washings in a beaker. Precipitation of the sulphur in the washings as barium sulphate which is collected and weighed. Calculation of the percentage of sulphur.

3 REAGENTS

All reagents shall be of recognized analytical quality.

Distilled water shall be used wherever water is specified.

3.1 Barium chloride, 100 g/dm³ solution.

Dissolve 100 g of barium chloride (BaCl₂·2H₂O) in water and dilute to 1 dm³.

3.2 Hydrochloric acid, ρ_{20} 1,19 g/cm³.

3.3 Picric acid, saturated solution.

Add an excess of picric acid to water in a glass-stoppered bottle. Shake the bottle well to dissolve enough picric acid to saturate the solution. A few crystals of picric acid shall remain in contact with the solution after saturation.

3.4 Oxygen.

A supply of oxygen, such as from commercial cylinders, of adequate purity (free of sulphur compounds).

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Oxygen bomb calorimeter, complete with ignition transformer, ignition wire, fuel capsule crucibles, oval-type bucket, calorimeter jacket, electric stirrer for water circulation around bomb, and calorimetric thermometer covering the range 19 to 35 °C in intervals of 0,02 °C.

4.2 Oxygen cylinder, with regulator and connector for oxygen bomb calorimeter.

4.3 Crucible, porcelain, 30 cm³ capacity.

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

4.5 Muffle furnace, capable of maintaining a temperature of 925 ± 25 °C.

4.6 Filter funnels, bunsen filtering, long stem, 60° angle fluted.

5 PROCEDURE

5.1 Dry an adequate amount of the sample for 1 h at a temperature of 105 ± 2 °C.

5.2 Weigh, to the nearest 0,1 mg, about 0,5 g of the dried sample in the fuel capsule. Place the crucible in the loop electrode and attach a fuse wire as described in the instructions for the oxygen bomb calorimeter. Bend the fuse wire so that the wire extends just below the surface of the carbon black.

5.3 Pipette 5 cm³ of water into the bomb cylinder. Assemble the bomb and fill with oxygen (pressure : 3 MN/m²).

5.4 Add 2 000 cm³ of water, at ambient temperature, to the calorimeter bucket. Place the bucket in the calorimeter, set the bomb in the bucket, attach the thrust terminal to the bomb, close the calorimeter with the thermometer in place, and start the electric stirrer.

Allow the electric stirrer to operate for 2 min, then record the water temperature. Close the ignition circuit momentarily to fire the charge. If the ignition system has

no pilot light, hold the ignition switch button for 5 s before releasing. Observe the rise in water temperature. The temperature should rise about 2 °C in approximately 5 min. (If the temperature fails to rise, the test shall be abandoned and a fresh determination begun.) Allow the electric stirrer to operate for a total of 10 min after ignition.

Remove the bomb, and release the pressure gradually over a period of time of at least 1 min.

5.5 Open the bomb and wash all the inner surfaces of the bomb with water, collecting all the washings in a 250 cm³ beaker. Filter the solution into a 400 cm³ beaker, washing the filter paper with three 5 cm³ portions of water. Add 5 cm³ of hydrochloric acid (3.2), 5 cm³ of saturated picric acid solution (3.3), and 10 cm³ of barium chloride solution (3.1) to the filtrate. Digest the mixture on an electric hot-plate at low temperature for approximately 15 min. Filter through a low-ash paper and wash the precipitate in the paper with 10 cm³ portions of boiling water. Continue washing until a test with silver nitrate shows absence of chloride in the filtrate.

5.6 Transfer the filter paper with the precipitate to a porcelain crucible, weighed to the nearest 0,1 mg. Char and ignite over the low flame of a high-temperature gas burner. Transfer to a muffle furnace at a temperature of 925 ± 25 °C for 30 min. Remove the crucible and contents to a desiccator, allow to cool to ambient temperature, and weigh to the nearest 0,1 mg.

6 EXPRESSION OF RESULTS

The total sulphur content is given, as a percentage by mass, by the formula :

$$\frac{0,137\ 3\ (m_2 - m_1)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the dried test portion;

m_1 is the mass, in grams, of the crucible;

m_2 is the mass, in grams, of the crucible and precipitate after ignition.

7 TEST REPORT

The test report shall include the following particulars :

- proper identification of the sample;
- results obtained.

SECTION TWO : METHOD B — COMBUSTION FURNACE

8 PRINCIPLE

Heating of a weighed test portion of dried carbon black at a temperature of 1 425 °C in a combustion furnace in a

stream of oxygen. Collection of sulphur compounds evolved from the test portion in a titrating flask containing hydrochloric acid solution. Titration of these compounds with potassium iodate-potassium iodide solution. Calculation of the percentage of sulphur.

9 REAGENTS

All reagents shall be of recognized analytical quality.

Distilled water shall be used wherever water is specified.

9.1 Hydrochloric acid.

Mix 2 volumes of concentrated hydrochloric acid (ρ_{20} 1,19 g/cm³) with 13 volumes of water.

9.2 Potassium iodate-potassium iodide solution.

Dissolve 1,112 5 g of potassium iodate (KIO₃), 100 g of potassium iodide (KI), and 5 g of potassium hydroxide in approximately 500 cm³ of water. Dilute to 1 dm³ in a volumetric flask.

NOTE — The solution shall be stored in a brown or green glass bottle. It is advisable to prepare a fresh solution every 30 days.

9.3 Starch indicator.

Dissolve 2,5 g of starch and 5 mg of mercury(II) iodide (HgI₂) in 1 dm³ of water.

9.4 Oxygen.

A supply of oxygen, such as from commercial cylinders, of adequate purity (free from sulphur compounds).

10 APPARATUS

Ordinary laboratory apparatus and

10.1 Combustion furnace, suitable for use at a temperature of 1 425 ± 25 °C.

10.2 Combustion tube about 75 cm in length, with one end tapered.

10.3 Combustion boats, either of fused aluminium oxide (alundum) or of porcelain.

NOTE — All new combustion boats shall be fired for 1 h at 1 425 ± 25 °C prior to use.

10.4 Pressure regulator and flowmeter.

10.5 Oxygen purification train, consisting of a gas-washing bottle containing concentrated sulphuric acid (H₂SO₄, ρ_{20} 1,84 g/cm³) and a water-absorbing bottle filled with a mixture of soda asbestos and anhydrous calcium sulphate that has been saturated with carbon dioxide.

10.6 Gas-dispersion tube, coarse-fritted glass.

10.7 Titrating flasks, about 500 cm³ capacity.

10.8 Rubber stopper equipped with a high-temperature heat reflector or baffle made of either metal or a refractory.

10.9 Oven, preferably gravity convection type, capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$.

11 PROCEDURE

11.1 Dry an adequate amount of carbon black for 1 h at a temperature of $105 \pm 2^\circ\text{C}$. Weigh, to the nearest 1 mg, about 1 g of the dried sample and transfer to a combustion boat.

11.2 Fill the titrating flask one-third full with the hydrochloric acid (9.1), add 2 cm³ of the starch indicator (9.3) and, while agitating the solution, add just enough of the potassium iodate-potassium iodide solution (9.2) to give a perceptible colour. Place the gas-dispersion tube in the titrating flask.

11.3 With the combustion furnace at a temperature of $1\,425^\circ\text{C}$ and the oxygen flowing at 1 dm³ per minute (see note), insert the combustion boat containing the carbon black into the hot zone of the combustion tube. Stopper the combustion tube immediately so as to pass the combustion gases evolved through the gas-dispersion tube. Titrate the solution in the titrating flask with the potassium

iodate-potassium iodide solution (9.2) to maintain the blue colour in the titrating flask. Consider the end point to be reached when, after 3 min of bubbling, it is not necessary to add potassium iodate-potassium iodide solution to maintain the blue colour in the titrating flask.

NOTE — Prior to each series of determinations, the combustion system shall be purged for 30 min with the oxygen flowing and with the furnace temperature at $1\,425 \pm 25^\circ\text{C}$.

12 EXPRESSION OF RESULTS

The total sulphur content is given, as a percentage by mass, by the formula :

$$\frac{V}{20 m}$$

where

V is the volume, in cubic centimetres, of potassium iodate-potassium iodide solution required for the titration;

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

13 TEST REPORT

The test report shall include the following particulars :

- a) proper identification of the sample;
- b) results obtained.

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