International Standard 1138

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Rubber compounding ingredients — Carbon black — Determination of sulphur content

Ingrédients de mélange du caoutchouc — Noir de carbone — Dosage du soufre total

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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.

International Standard ISO 1138 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products.*

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 1138-1975), which had been approved by the member bodies of the following countries :

Australia
Austria
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Egypt, Arab Rep. of
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Hungary
India
Iran
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Italy
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Switzerland
Thailand
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USA
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No member body had expressed disapproval of the document.

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Rubber compounding ingredients — Carbon black — Determination of 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

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

3.1 Barium chloride, 100 g/dm^3 solution.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in water and dilute to 1 dm^3.

3.2 Hydrochloric acid, ϱ_{20} 1,19 g/cm^3.

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 from 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 cm^3 capacity.

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

4.5 Muffle furnace, capable of being controlled at 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 in the oven (4.4), controlled at 105 ± 2 °C.

5.2 Weigh, to the nearest 0,1 mg, about 0,5 g of the dried sample in the fuel capsule (see 4.1). 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 cm^3 of water into the bomb cylinder. Assemble the bomb and fill with oxygen (3.4) (pressure : 3 MPa).

5.4 Add 2 000 cm^3 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 cm^3 beaker. Filter the solution into a 400 cm^3 beaker, washing the filter paper with three 5 cm^3 portions of water. Add 5 cm^3 of the hydrochloric acid (3.2), 5 cm^3 of the saturated picric acid solution (3.3), and 10 cm^3 of the 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 cm^3 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 the porcelain crucible (4.3), weighed to the nearest 0,1 mg. Char and ignite over the low flame of a high-temperature gas burner. Transfer to the muffle furnace (4.5), controlled at 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, expressed as a percentage by mass, is given 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 :

a) a reference to this International Standard;

b) the proper identification of the sample;

c) the 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 standard volumetric potassium iodate-potassium iodide solution. Calculation of the percentage of sulphur.

9 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

9.1 Hydrochloric acid.

Mix 2 volumes of concentrated hydrochloric acid (ϱ_{20} 1,19 g/cm^3) with 13 volumes of water.

9.2 Potassium iodate-potassium iodide, standard volumetric solution.

Dissolve 1,112 5 g of potassium iodate (KIO_3), 100 g of potassium iodide (KI), and 5 g of potassium hydroxide in approximately 500 cm^3 of water. Dilute to 1 dm^3 in a volumetric flask.

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

9.3 Starch, indicator solution.

Dissolve 2,5 g of starch and 5 mg of mercury(II) iodide (HgI_2) in 1 dm^3 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 should 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 (H_2SO_4, ϱ_{20} 1,84 g/cm^3) 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 cm^3 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 being controlled at 105 ± 2 °C.

11 Procedure

11.1 Dry an adequate amount of carbon black for 1 h in the oven (10.9), controlled at 105 ± 2 °C. Weigh, to the nearest 1 mg, about 1 g of the dried sample and transfer to a combustion boat (10.3).

11.2 Fill a titrating flask (10.7) one-third full with the hydrochloric acid solution (9.1), add 2 cm^3 of the starch indicator solution (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 (10.6) in the titrating flask.

11.3 With the combustion furnace at a temperature of 1 425 °C and the oxygen flowing at 1 dm^3 per minute (see the 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 should be purged for 30 min with the oxygen flowing and with the furnace temperature at 1 425 ± 25 °C.

12 Expression of results

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

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

where

V is the volume, in cubic centimetres, of standard volumetric potassium iodate-potassium iodide solution (9.2) 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) a reference to this International Standard;

b) the proper identification of the sample;

c) the results obtained.

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