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Rubber compounding ingredients — Carbon black — Determination of sulfur content

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

Reference number
ISO 1138:2007(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1138 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 1138:1981). The temperature of the oven in Method A has been changed to 125 °C, a method using an automatic analyser has been added, and the text has been up-dated editorially.

Rubber compounding ingredients — Carbon black — Determination of sulfur content

1 Scope

This International Standard specifies three methods for the determination of the total sulfur 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;
- Method C, using an automatic analyser.

With respect to safety aspects and test precision, it is preferable to use automatic systems. Classical chemical analysis (Method A and Method B) is acceptable if automatic equipment is not available.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15671, *Rubber and rubber additives — Determination of total sulfur content using an automatic analyser*

3 Method A: Oxygen bomb calorimeter

3.1 Principle

A weighed test portion of dried carbon black is ignited in an oxygen bomb calorimeter. After the bomb is opened, the inner surfaces are washed with water, and the washings collected in a beaker. The sulfur in the washings is precipitated as barium sulfate, which is collected and weighed. The percentage of sulfur is calculated.

3.2 Reagents

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

3.2.1 Barium chloride, 100 g/l solution.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in water and dilute to 1 l.

3.2.2 Hydrochloric acid, ρ_{20} 1,19 g/ml.

3.2.3 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.

3.2.4 Oxygen.

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

3.3 Apparatus

Ordinary laboratory apparatus and the following.

3.3.1 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 °C to 35 °C in intervals of 0,02 °C.

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

3.3.3 Crucible, porcelain, 30 ml capacity.

3.3.4 Oven, gravity-convection type, capable of temperature regulation within ± 1 °C at 125 °C and temperature uniformity within ± 5 °C.

3.3.5 Muffle furnace, capable of being controlled at 925 °C ± 25 °C.

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

3.4 Procedure

3.4.1 Dry an adequate amount of the sample for 1 h in the oven (3.3.4) set at 125 °C.

3.4.2 Weigh, to the nearest 0,1 mg, about 0,5 g of the dried sample in the fuel capsule (see 3.3.1). 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.

3.4.3 Pipette 5 ml of water into the bomb cylinder. Assemble the bomb and fill with oxygen (3.2.4.) (pressure: 3 MPa).

3.4.4 Add 2 l 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.

3.4.5 Open the bomb and wash all the inner surfaces of the bomb with water, collecting all the washings in a 250 ml beaker. Filter the solution into a 400 ml beaker, washing the filter paper with three 5 ml portions of water. Add 5 ml of the hydrochloric acid (3.2.2), 5 ml of the saturated picric acid solution (3.2.3) and 10 ml of the barium chloride solution (3.2.1) 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 ml portions of boiling water. Continue washing until a test with silver nitrate shows absence of chloride in the filtrate.

3.4.6 Transfer the filter paper with the precipitate to the porcelain crucible (3.3.3), 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 (3.3.5), controlled at 925 °C ± 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.

3.5 Expression of results

The total sulfur 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.

3.6 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) identification of the sample;

c) the results obtained.

4 Method B: Combustion furnace

4.1 Principle

A weighed test portion of dried carbon black is heated at a temperature of 1 425 °C in a combustion furnace in a stream of oxygen. The sulfur compounds evolved from the test portion are collected in a titrating flask containing hydrochloric acid solution. These compounds are titrated with standard volumetric potassium iodate-potassium iodide solution. The percentage of sulfur is calculated.

4.2 Reagents

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

4.2.1 **Hydrochloric acid**.

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

4.2.2 **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 ml of water. Dilute to 1 l in a volumetric flask.

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

4.2.3 **Starch**, indicator solution.

Dissolve 2,5 g of starch and 5 mg of mercury(II) iodide (HgI_2) in 1 l of water.

4.2.4 Oxygen.

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

4.3 Apparatus

Ordinary laboratory apparatus and the following.

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

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

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

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

4.3.4 Pressure regulator and **flowmeter**.

4.3.5 Oxygen purification train, consisting of a gas-washing bottle containing concentrated sulfuric acid (H_2SO_4, ρ_{20} 1,84 g/ml) and a water-absorbing bottle filled with a mixture of soda asbestos and anhydrous calcium sulfate that has been saturated with carbon dioxide.

4.3.6 Gas-dispersion tube, coarse-fritted glass.

4.3.7 Titrating flasks, about 500 ml capacity.

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

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

4.4 Procedure

4.4.1 Dry an adequate amount of carbon black for 1 h in the oven (4.3.9), controlled at 105 °C ± 2 °C. Weigh, to the nearest 1 mg, about 1 g of the dried sample and transfer to a combustion boat (4.3.3).

4.4.2 Fill a titrating flask (4.3.7) one-third full with the hydrochloric acid solution (4.2.1), add 2 ml of the starch indicator solution (4.2.3) and, while agitating the solution, add just enough of the potassium iodate-potassium iodide solution (4.2.2) to give a perceptible colour. Place the gas-dispersion tube (4.3.6) in the titrating flask.

4.4.3 With the combustion furnace at a temperature of 1 425 °C and the oxygen flowing at 1 l per minute (see 4.4.4), 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 (4.2.2) 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.

4.4.4 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 °C ± 25 °C.

4.5 Expression of results

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

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

where

V is the volume, in millilitres, of standard volumetric potassium iodate-potassium iodide solution (4.2.2) required for the titration;

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

4.6 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) identification of the sample;

c) the results obtained.

5 Method C: Automatic analyser

See ISO 15671 for the specification of the method. Three different options for detection of the generated sulfur dioxide are given. A thermal conductivity detector may also be used, provided it is used in accordance with the manufacturer's instructions.

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