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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
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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 third edition cancels and replaces ISO 1138:2007 and ISO 1138:2007/Amd 1:2012, which have been technically revised.

The main changes are as follows:

— withdrawal of the method using an oxygen bomb calorimeter (previously called Method A) due to the use of hazardous chemicals, and withdrawal of Amendment 1:2012 related to that method;

— addition of CAS numbers to the chemical reagents used in the current Method A.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber compounding ingredients — Carbon black — Determination of sulfur content

1 Scope

This document specifies two methods for the determination of the total sulfur in all types of carbon black for use in the rubber industry:

— Method A, using a combustion furnace;

— Method B, using an automatic analyser.

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Method A: 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; CAS 7647-01-0) with 13 volumes of water; CAS 7732-18-5.

4.2.2 Potassium iodate-potassium iodide, standard volumetric solution.

Dissolve 1,112 5 g of potassium iodate (KIO_3; CAS 7758-05-6), 100 g of potassium iodide (KI; CAS 7681-11-0) and 5 g of potassium hydroxide (CAS 1310-58-3) 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 (CAS 9005-25-8) and 5 mg of mercury(II) iodide (HgI_2; CAS 7774-29-0) in 1 l of water.

4.2.4 Oxygen.

A supply of oxygen (CAS 7782-44-7), 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; CAS 7664-93-9) and a water-absorbing bottle filled with a mixture of soda asbestos and anhydrous calcium sulfate (CAS 7778-18-9) that has been saturated with carbon dioxide (CAS 124-38-9).

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