INTERNATIONAL STANDARD

ISO 1408

Third edition
1995-10-01

Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods

Caoutchouc — Dosage du noir de carbone — Méthode pyrolytique et méthodes par dégradation chimique

Reference number
ISO 1408:1995(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 1408 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 1408:1987), of which it constitutes a minor revision.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

1.1 This International Standard specifies a pyrolytic method (A) and two chemical degradation methods (B and C) for the determination of the carbon black content of rubber.

1.2 Method A is preferred and should be used for the following polymers, except when certain compounding materials such as lead and cobalt salts, graphitic carbon blacks, phenolic and other resins, bitumen, or cellulose, etc., which cause the formation of a carbonaceous residue during pyrolysis, are present:

— polyisoprene, natural or synthetic;

— polybutadiene;

— styrene-butadiene copolymers;

— butyl rubber;

— acrylate rubber;

— ethylene-propylene copolymer;

— ethylene-propylene terpolymer;

— polyethers;

— polyethylene-derived polymers;

— silicone rubbers;

— fluorosilicone rubbers;

— chlorosulfonated polyethylenes containing less than 30 % (*m/m*) of chlorine.

The precision of this method may be affected if mineral fillers, e.g. alumina or calcium carbonate, are present which decompose or dehydrate, or form volatile halides in the case of halogenated polymers, at the pyrolysis temperature.

The method cannot be used for either chloroprene rubbers or butadiene-nitrile rubbers having an acrylic acid nitrile content greater than 30 % (*m/m*).

1.3 Method B is chiefly intended to be used with samples not amenable to the pyrolytic method A, although it can be used for all samples based on unsaturated rubbers, except for isobutylene-isoprene copolymers.

1.4 Method C is relatively hazardous and should be used only for the analysis of samples based on isobutylene-isoprene copolymers and ethylene-propylene copolymers and related terpolymers when methods A and B fail.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged

to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 1407:1992, *Rubber — Determination of solvent extract.*

3 Principle

3.1 Method A

A weighed test piece of the rubber is extracted with acetone and, if bitumen is present, with dichloromethane. The extracted rubber is pyrolized in a combustion boat at 850 °C in a stream of nitrogen. The boat containing the non-volatile residue is cooled and weighed.

The carbon black is then burnt off in air or oxygen in a furnace at the same temperature. The boat and its contents are cooled and reweighed. The loss in mass represents the carbon black.

3.2 Method B

A weighed test piece of the rubber is extracted with acetone. The organic components are destroyed by oxidation with nitric acid, the acid-soluble inorganic components dissolving simultaneously in the nitric acid. The residue, which consists of carbon black and acid-insoluble mineral fillers is filtered, washed and then dried to constant mass at 850 °C in a nitrogen atmosphere to avoid oxidation of the carbon black.

The weighed residue is reheated at the same temperature (850 °C) (to avoid further change in mass of the inorganic matter) but this time in air so as to cause oxidation of the carbon black to carbon dioxide. The residue is cooled and reweighed. The loss in mass represents the carbon black.

3.3 Method C

After swelling of a test piece by hot *p*-dichlorobenzene, the organic matter is oxidized by *tert*-butyl hydroperoxide. The undissolved carbon black and mineral fillers are filtered, washed and then dried to constant mass at 850 °C in a nitrogen atmosphere to avoid oxidation of the carbon black.

The weighed residue is reheated at the same temperature (850 °C) (to avoid further change in mass of the inorganic matter) but this time in air so as to cause oxidation of the carbon black to carbon dioxide. The residue is cooled and reweighed. The loss in mass represents the carbon black.

4 Method A

WARNING — All recognized health and safety precautions shall be in effect when carrying out this method. All evaporations shall be carried out in a fume cupboard (hood).

4.1 Reagents

During the analysis, unless otherwise stated, use only reagents of analytical reagent grade (or equivalent) and only distilled water or water of equivalent purity.

4.1.1 Nitrogen, dry and free from oxygen.

NOTE 1 Commercial "oxygen-free" nitrogen may require further purification.

4.1.2 Oxygen or **air**, gaseous, dry.

4.1.3 Xylene, general laboratory grade.

4.1.4 Acetone.

4.1.5 Dichloromethane.

4.1.6 Ethanol-toluene azeotrope (ETA).

Mix 7 volumes of absolute ethanol with 3 volumes of toluene. Alternatively, mix 7 volumes of commercial-grade ethanol with 3 volumes of toluene and boil the mixture with anhydrous calcium oxide (quicklime) under reflux for 4 h. Then distil the azeotrope and collect the fraction with a boiling range not exceeding 1 °C, for use in the test.

4.2 Apparatus

Ordinary laboratory apparatus, plus the following:

4.2.1 Combustion boat, made of silica, of length 50 mm to 60 mm, with handle.

4.2.2 Tube furnace assembly see figure 1, comprised of the following component parts:

4.2.2.1 Combustion tube, made of quartz or of impervious aluminous porcelain, and fitted with means for advancing and withdrawing the combustion boat (4.2.1). The inside diameter shall be sufficient to allow the combustion boat to enter the tube and move easily through it. The tube shall be 30 cm longer than the tube furnace (4.2.2.2). One end of the tube