

Chemical tests for raw and vulcanized rubber —

Part 14: Methods for determination of carbon black content

ICS 83.060

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee PRI/23, Chemical testing of rubber, upon which the following bodies were represented:

- Malaysian Rubber Producers' Research Association
- Ministry of Defence
- RAPRA Technology Ltd.
- Royal Society of Chemistry

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National foreword

This Part of BS 7164 has been prepared by Technical Committee PRI/23 and is identical to ISO 1408:1995 *Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods* published by the International Organization for Standardization (ISO). It supersedes BS 7164-14:1990 which is withdrawn.

Cross-reference

Publication referred to	Corresponding British Standard
ISO 1407:1992	BS 7164 <i>Chemical tests for raw and vulcanized rubber</i> Part 3:1992 <i>Methods for determination of solvent extract</i>

The Technical Committee has reviewed the provisions of ISO 383:1976 to which normative reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

ISO 383:1976 is related to BS 572:1991 *Specification for interchangeable conical ground glass joints*.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 pyrolyzed 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.