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

*Caoutchouc — Détermination de la teneur en noir de carbone — Méthode pyrolytique et
méthodes par dégradation chimique*

Reference number
ISO 1408 : 1987 (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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

This second edition cancels and replaces the first edition (ISO 1408 : 1976), of which it constitutes a technical revision, two chemical degradation methods having been added.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

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, corresponding to ISO 1408 : 1976, 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) 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 References

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

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

3 Principle

3.1 Method A (section one)

Extraction of a weighed test portion of the rubber with acetone and, if bitumen is present, with dichloromethane. Pyrolysis of the extracted rubber in a combustion boat at 850 °C in a stream of nitrogen. Cooling and weighing of the boat containing the non-volatile residue.

Burning off the carbon black in air or oxygen in a furnace at the same temperature. Cooling and reweighing of the boat and its contents. The loss in mass represents the carbon black.

3.2 Method B (section two)

Extraction of a weighed test portion of the rubber with acetone. Destruction of the organic components by oxidation with nitric