

INTERNATIONAL
STANDARD

ISO
7269

Second edition
1995-10-15

Rubber — Determination of free sulfur

Caoutchouc — Dosage du soufre libre



Reference number
ISO 7269:1995(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 7269 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 7269:1987), of which it constitutes a minor revision.

Annex A forms an integral part of this International Standard.

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Introduction

The free-sulfur content of an unvulcanized rubber compound (i.e. the amount of uncombined sulfur present in the compound) should represent all the added, elemental, sulfur. As the vulcanization proceeds and sulfur is gradually combined with the rubber, the free-sulfur content gradually decreases, so its level in any particular product bears some relation to its state of vulcanization, provided the formulation is known and the correct proportion of elemental sulfur (and other ingredients) has been incorporated.

Vulcanizates containing higher proportions of free sulfur than the rubber can dissolve are liable to bloom sulfur, hence certain product specifications include a maximum figure for free sulfur.

Rubber — Determination of free sulfur

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 three methods for the determination of free sulfur in vulcanized rubber: two versions of the copper spiral method, and the sodium sulfite method.

1.2 The copper spiral methods are also applicable, subject to the limitations described in clause 4, to unvulcanized compounds.

1.3 Annex A gives a table listing the sulfur contribution, as a percentage by mass, to these free-sulfur methods for various commercial accelerators.

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 Copper spiral methods A and B

3.1.1 Method A

The free sulfur in a test portion is extracted with acetone as in the determination of solvent extract, described in ISO 1407, except that a spiral of clean copper gauze is placed in the flask containing the acetone. The copper reacts with the extracted sulfur, forming copper sulfide. When extraction is complete, the acetone solution is drawn off through a filter and discarded. The acetone-insoluble copper and copper sulfide remaining in the flask are treated with hydrochloric acid. The resulting hydrogen sulfide is passed through cadmium acetate solution, and the cadmium sulfide formed is determined iodometrically.

3.1.2 Method B

Rapid copper spiral method B is the same as copper spiral method A except that the extraction time is reduced by using a vapour-jacketed Soxhlet of reduced capacity (6 cm³) to increase the extraction temperature.

3.2 Sulfite method

The free sulfur in a test portion is reacted with aqueous sodium sulfite and the sodium thiosulfate so formed is determined iodometrically.