

Chemical tests for raw and vulcanized rubber —

Part 24: Determination of free sulfur

ICS 83.060

Confirmed September 2010

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

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 May 1996

© BSI 10 December 2002
First published September 1990
Second edition May 1996

The following BSI references relate to the work on this standard:
Committee reference PRI/23
Draft announced in *BSI News* February 1996

ISBN 0 580 25611 1

Amendments issued since publication

Amd. No.	Date	Comments
13980 Technical Corrigendum No. 1	10 December 2002	Changes to formulae in 5.4 and 7.4

Contents

	Page
Committees responsible	Inside front cover
National foreword	ii
Introduction	1
1 Scope	1
2 Normative references	1
3 Principle	1
4 Limitations	2
5 Copper spiral method A	2
6 Copper spiral method B	4
7 Sulfite method	5
8 Test report	6
Annex A (normative) Determination of free-sulfur content — Effect of various commercial accelerators on the copper spiral and sulfite methods of analysis	10
Figure 1 — Typical apparatus for extraction of free sulfur by copper spiral method A	7
Figure 2 — Typical apparatus for determination of free sulfur by copper spiral method A	8
Figure 3 — Typical apparatus for determination of free sulfur by copper spiral method B	9

National foreword

This Part of BS 7164 has been prepared by Technical Committee PRI/23 and is identical with ISO 7269:1995 *Rubber — Determination of free sulfur*, including Technical Corrigendum 1 May 2002, published by the International Organization for Standardization (ISO).

It supersedes BS 7164-24:1990 which is withdrawn.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

The Technical Committee has reviewed the provisions of ISO 383:1976 to which normative reference is made in the text, and has decided that it is acceptable for use in conjunction with this standard. A related British Standard to ISO 383:1976 is BS 572:1991 *Specification for interchangeable conical ground glass joints*.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its 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 10, an inside back cover and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

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.

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.