

**METHODS OF TESTING
RAW RUBBER AND UNVULCANIZED
COMPOUNDED RUBBER
PART 2. CHEMICAL ANALYSIS OF RAW
NATURAL RUBBER**

THIS BRITISH STANDARD, having been approved by the Rubber Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 28th March, 1967.

First published, February, 1954.

First revision, March, 1967.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 4000, fully indexed and with a note of the contents of each, will be found in the British Standards Yearbook, The BS Yearbook may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standards:

- BS 245. White spirit.
- BS 410. Test sieves.
- BS 509. Acetone.
- BS 572. Interchangeable conical ground glass joints.
- BS 1673. Methods of testing raw rubber and unvulcanized compounded rubber. Part 1. Sampling.
- BS 1752. Laboratory sintered or fritted filters.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following BSI references relate to the work on this standard:
Committee references RUC/10, RUC/10/5
Draft for comment D65/8243

Amendment No. 1
published and effective from 21 December 1990
to BS 1673 : Part 2 : 1967

Methods of testing raw rubber and unvulcanized compounded rubber
Part 2. Chemical analysis of raw natural rubber

NOTE. This amendment deletes certain methods which have been revised and issued as separately published methods of BS 5923 and BS 7164. In due course Parts of BS 5923 will be superseded by Parts or Sections of BS 7164.

Deletions

2.2 Volatile matter. Delete the entire text and substitute *'Text deleted. Superseded by BS 5923 : Part 3'*.

2.4 Ash. Delete the entire text and substitute *'Text deleted. Superseded by BS 5923 : Part 1'*.

2.5 Nitrogen. Delete the entire text and substitute *'Text deleted. Superseded by BS 7164 : Part 21'*.

2.6 Copper. Delete the entire text and substitute *'Text deleted. Superseded by BS 7164 : Section 28.2'*.

2.7 Manganese. Delete the entire text and substitute *'Text deleted. Superseded by BS 7164 : Section 26.2'*.

2.8 Iron. Delete the entire text and substitute *'Text deleted. Superseded by BS 7164 : Section 27.2'*.

2.9 Dirt. Delete the entire text and substitute *'Text deleted. Superseded by BS 7164 : Part 8'*.

CO-OPERATING ORGANIZATIONS

The Rubber Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government department and scientific and industrial organizations:

- *Federation of British Rubber and Allied Manufacturers
- *Institution of the Rubber Industry
- *Ministry of Aviation
 - Natural Rubber Bureau
- *Natural Rubber Producers' Research Association
- *Rubber and Plastics Research Association of Great Britain
 - Rubber Growers' Association
- *Society of Motor Manufacturers and Traders Limited

The Government department and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Chemical Plant Manufacturers' Association
 British Railways Board
 British Rubber and Resin Adhesive Manufacturers' Association
 Cable Makers' Association
 Chemical Industries Association
 Electric Research Association
 Institution of Mechanical Engineers
 Institution of Municipal Engineers
 Institution of Water Engineers
 Ministry of Defence, Air Force Department
 Ministry of Defence, Army Department
 Ministry of Defence, Navy Department
 Ministry of Housing and Local Government
 National College of Rubber Technology
 Post Office
 Royal Institute of Chemistry
 Rubber Trade Association of London
 Society of Analytical Chemistry

BRITISH STANDARD

METHODS OF TESTING

RAW RUBBER AND UNVULCANIZED
COMPOUNDED RUBBER

PART 2. CHEMICAL ANALYSIS OF RAW NATURAL RUBBER

FOREWORD

This British Standard is one of a series devoted to the chemical testing of raw rubbers.

Two important departures from the 1954 edition of BS 1673 : Part 2 will be noted. First, tests on the raw rubber have been separated from those on unvulcanized compounded rubber. This is felt to be a more rational arrangement since the two forms of rubber have little in common, and the commercial requirements of a standard dealing with them are so different that a unified scheme of chemical testing becomes impracticable. Consequently chemical tests applying to unvulcanized compounded rubber will in future be found in BS 903, 'Testing of vulcanized rubber'.

Secondly, this standard is concerned solely with the chemical testing of natural rubber and no longer includes modified test procedures to be used for various synthetic polymers. This change is a reflection of the fact that many synthetic polymers are no longer regarded as variants of the natural product, but as materials of commerce in their own right. Standard methods of testing synthetic rubbers are in course of preparation.

Since 1954 three ISO recommendations on the chemical testing of raw natural rubber have become available, viz. the determination of ash (R 247), the determination of volatile matter (R 248) and the determination of dirt (R 249), but at the time of writing two of these, R 248 and R 249, are undergoing revision. Another group of chemical tests, the determination of copper, manganese, iron and nitrogen, are also in their final draft form at ISO level. The opportunity has therefore been taken to incorporate the ISO methods in this revised British Standard. The majority of these follow closely the methods given in the 1954 edition of BS 1673, Part 2, and consequently only minor changes have been introduced. In the determination of iron, however, thioglycollic acid has been replaced by 1,10 phenanthroline as a colorimetric reagent since the former has been found unsatisfactory in the presence of copper. Methods for the determination of acid value, of rubber hydrocarbon by chromic acid oxidation and of coarse dirt have been deleted since these tests are no longer required by the industry.

METHODS

2.1 SAMPLING AND SAMPLE PREPARATION

2.1.1 Explanatory note. The importance of adhering to an established recognized procedure for the sampling of natural rubber needs no emphasis; it is sufficient to mention that unless such a procedure is followed, subsequent test results may be invalid or misleading. The procedure recommended here is based on ISO Recommendation R 250 (now under review). To ensure true representation of the original sample (bale or sheets) a piece of rubber of at least 600 g is necessary, of which about 70 g is required for the full range of tests given in this standard. The piece should be taken in accordance with BS 1673, Part 1*, then weighed and homogenized. Further chemical tests are carried out on the homogenized piece.

2.1.2 Procedure. Weigh the piece of rubber cut from the sample bale or the strips from the loose sheets to the nearest 0.1 g. Set the nip of a laboratory mill to 1.25 ± 0.1 mm and the roll temperature to $70 \pm 5^\circ\text{C}$ and homogenize the rubber by passing it ten times between the rolls. The rolls should revolve at even speed, but a friction ratio not exceeding 1.4 : 1 may be used. As the rubber passes through the mill, roll it into a cylinder and present it endways to the mill for the next pass. Collect any solid matter parting from the mass and re-incorporate it in the rubber. Cool the rubber and weigh to the nearest 0.1 g.

Record the weights of the rubber before and after milling since these are required later for the calculation of volatile matter. Store the rubber in an airtight container unless further testing can be carried out immediately.

2.2 VOLATILE MATTER

2.2.1 Explanatory note. This method is primarily intended for the determination of moisture in raw natural rubber, but the result will include the amount of any other material in the rubber which is volatile at 100°C . The method agrees technically with ISO Recommendation R 248.

2.2.2 Procedure. From the homogenized piece prepared as described in 2.1 cut a test portion of approximately 10 g and weigh immediately to the nearest 1 mg. Sheet out the test portion to a thickness not exceeding 2 mm by passing it twice between the tightly closed rolls of a laboratory mill. Dry the test portion at $100 \pm 5^\circ\text{C}$ for one hour and then until the loss in weight on successive weighings after heating periods of 30 minutes is less than 1 mg.

* BS 1673, 'Methods of testing raw rubber and unvulcanized compounded rubber', Part 1, 'Sampling'.

2.2.3 Expression of results. Calculate the volatile matter from the following formula:

$$\text{Volatile matter, per cent} = \left(1 - \frac{BD}{AC}\right) \times 100$$

where A = weight of the piece before homogenization;

B = weight of the piece after homogenization;

C = weight of the test portion before drying at 100°C;

D = weight of the test portion after drying at 100°C.

2.3 SOLVENT EXTRACT

2.3.1 Explanatory note. The usual solvent for the determination of solvent extract in raw natural rubber is acetone. The acetone extract will contain saturated and unsaturated fatty acids, sterols, sterol esters and sterol glucosides and, from some rubbers, amines arising from bacterial maturation of the latex. Additionally, acetone will extract degraded rubber (i.e. rubber of low molecular weight) if the rubber has been exposed to oxidative influences such as strong sunlight.

For the determination a weighed test portion is extracted in an all-glass Soxhlet type apparatus for $16 \pm \frac{1}{2}$ hours. When extraction is complete the solvent is evaporated and the extract is weighed.

2.3.2 Reagent. *Acetone*, to BS 509, 'Acetone', is suitable.

2.3.3 Apparatus. *Extraction apparatus.* The extraction apparatus shall be of the reflux type with the condenser placed immediately above the cup which holds the rubber. The cup shall be situated in the vapour of the boiling solvent and shall be emptied by a siphon. The apparatus shall be wholly of glass except when the extraction cup is suspended from the end of the condenser, in which case platinum wire shall be used for the suspension. The apparatus shall fit together so that loss of vapour during extraction does not exceed 20% of the extracting liquid. Suitable types of apparatus are shown in Figs. 1 and 2.

Filter paper, acid washed hardened, or

Nylon cloth, open weave, continuous filament.

2.3.4 Procedure. From the homogenized piece prepared as described in 2.1 cut a portion weighing about 8–10 g. Pass the rubber once or twice between the rolls of a laboratory mill, then reset the nip to 0.5 mm and sheet the rubber between hot rolls maintained at $95 \pm 5^\circ\text{C}$. It is convenient to run the milled sheet into polythene or glazed linen to prevent sticking as it leaves the mill. Cut from the sheet, avoiding the edges, a test portion estimated to weigh 5 g and weigh to the nearest 0.005 g. Roll in filter paper or cloth to form a loose roll from which the rubber cannot fall and so that the rubber does not contact itself anywhere. This is conveniently done by placing the rubber on a strip of