PD 4056

Amendment No. 1, published 1 February, 1961

to B.S. 903, Part B 19 : 1958

Methods of testing vulcanized rubber

Part B 19. Preparation and examination of water extract

Revision

Section 2. 2.2 Procedure. Delete the last sentence of the first paragraph.

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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 31st December, 1958.

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B.S. 903, first revision October 1950.
First published as B.S. 903 : Part B 19, December, 1958.

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 3000, indexed and cross-indexed for reference, together with an abstract of each standard, will be found in the Institution's Yearbook, price 15s.

This standard makes reference to the following British Standards:

B.S. 572	Interchangeable conical ground glass joints.
B.S. 612	Nessler cylinders.
B.S. 1647	pH scale.
B.S. 2087	Chemical requirements for treated textiles.
B.S. 2690	Methods for testing water used in industry.

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 B.S.I. references relate to the work on this standard:—
Committee references RUC/10, RUC/10/5
Draft for comment CY(RUC) 2957

CO-OPERATING ORGANIZATIONS

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

Board of Trade
*British Rubber Producers' Research Association
*Federation of British Rubber and Allied Manufacturers
*Institution of the Rubber Industry
*Ministry of Supply
Natural Rubber Development Board
*Research Association of British Rubber Manufacturers
*Rubber Growers' Association

The Government departments 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:—

Admiralty
Air Ministry
Association of British Chemical Manufacturers
British Chemical Plant Manufacturers' Association
British Electrical and Allied Industries Research Association
British Railways, The British Transport Commission
British Rubber and Resin Adhesive Manufacturers' Association
Cable Makers' Association
Department of the Government Chemist
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Ministry of Housing and Local Government
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Society for Analytical Chemistry
Society of Motor Manufacturers and Traders Ltd.

BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Part B 19. Preparation and Examination of Water Extract

FOREWORD

This British Standard has been published under the authority of the Rubber Industry Standards Committee. In deciding to issue a revision of the 1950 edition of B.S. 903, it has also been considered desirable to publish it in separate parts and the present part replaces Part 12 of 1950.

The main difference is in the inclusion of more detailed procedures on the water extract.

The letter B indicates the parts concerned with chemical analyses. The following parts have already been published:

Parts B 1 to B 5	Preparation of material and extraction methods.
Parts B 6 to B 10	Determination of sulphur.

SECTION 1 EXPLANATORY NOTE

Water extracts more or less completely certain non-rubber constituents of the raw rubber: quebrachitol, proteins and mineral salts from natural rubber; emulsifiers, stabilizers, coagulants, etc., from synthetic rubbers; and from compounded rubbers, glue, gelatine and other proteins, certain accelerators, starch, glycerin, alkalis, soap, calcium sulphate, and non-volatile residues from blowing agents, such as sodium carbonate and sodium chloride.

Owing to the very slow penetration of water into rubber, the method given, which consists of boiling the comminuted rubber with water for one hour, will not in general give complete extraction, and comparisons should therefore be made only between results obtained on samples comminuted in the same way.

The water extract can be used for determining any of the following:

> total water-soluble matter; free acid or alkali; chloride; sulphate; pH value and electrical resistivity of the extract; and the presence of ammonium salts.

In many cases the water extract is substantially unbuffered and the determination of pH value may then be subject to error. Determination of the electrical resistivity indicates when such conditions may be met and it is also a useful rapid test for the presence of electrolytes.

The procedures specify a test portion of 5 to 15 g of the prepared sample and require water 20 times the weight of the test portion. The actual weight of test portion used will depend on the number and type of tests which are required, and the following list indicates the volume of water needed for the tests where this is specified, and suggests suitable amounts elsewhere, though the actual apparatus used may determine the volume necessary.

Section 3	Total water soluble material	10 ml	(suggested)
,, 4	Electrical resistivity	5 ml	(suggested but will depend on cell used)
,, 5	Free acid or free alkali	20 ml	(required minimum)
,, 6	pH value	10 ml	(suggested but will depend on vessel used)
,, 7	Chloride	10 ml	(required)
,, 8	Sulphate	10 ml	(required)
,, 9	Ammonium salts	45 ml	(required, but extract used for resistivity and pH value can be used for this test so that the minimum requirement here is really 30 ml)
Total *minimum* requirement for all tests		95 ml	

SECTION 2 METHOD

2.1 Apparatus. The extraction apparatus shall consist of a borosilicate glass flask, 250 or 500 ml and B.24 neck,* fitted with an efficient reflux condenser, to the top of which shall be fitted a guard tube with a suitable reagent for the exclusion of carbon dioxide and atmospheric impurities.

Borosilicate glass flask of suitable size, for storage purposes. This may be similar to the extraction flask but shall be fitted with stopper and soda lime guard tube.

2.2 Procedure. Clean the surface of the sample with a cotton wool pad, moistened with demineralized water, to remove contamination, and then dry between filter papers. Cut the sample into cubes with sides of about 3 mm or (when the thickness is less than 3 mm) into 3 mm squares. If the sample is cellular rubber, instead of cutting as described above, crumb the sample by passing through the tightly closed cold rolls of a laboratory mill.

To prepare the water extract, weight 5–15 g of the prepared sample into the flask and add to the test portion 20 times its weight of demineralized water.

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* B.S. 572, ' Interchangeable conical ground glass joints '

If the electrical resistivity or pH value is to be determined, the resistivity of this water shall be not less than 10^6 ohm cm.[*] Fit the reflux condenser and guard tube, heat the water to boiling point and continue gentle boiling for one hour, at the end of which time immediately decant into the storage flask, cool to room temperature, and pipette as required.

The pH, if required, shall be determined within thirty minutes from the time of decanting.

If electrical resistivity or pH value is to be determined, the following check test shall be carried out on the apparatus:

> Boil at least 100 ml of the same demineralized water in the apparatus for one hour. Stopper the flask and cool. The resistivity shall be not less than 5×10^5 ohm cm.

SECTION 3 TOTAL WATER SOLUBLE MATERIAL

Evaporate to dryness an aliquot portion of the water extract and dry the residue for one hour at 100 ± 2°C. Cool in a desiccator and weigh. Express the result as a percentage of the initial weight of the test portion.

SECTION 4 ELECTRICAL RESISTIVITY OF THE WATER EXTRACT

The electrical resistivity of the aqueous extract shall be determined by measurements made with an alternating current bridge circuit employing a frequency of about 1000 c/s and with a cell to contain a portion of the aqueous extract. The cell shall be of borosilicate glass with electrodes of platinum coated with platinum black, and shall have a known cell constant. Suitable cells are described in B.S. 2087[†] and B.S. 2690.[‡]

Immediately before using the cell for the test it shall be rinsed with the demineralized water. The result shall be reported in ohm cm, the degree of comminution of the sample being stated.

SECTION 5 FREE ACID OR FREE ALKALI

5.1 Reagents. All reagents used shall be of recognized analytical reagent quality, and demineralized water shall be used wherever water is specified.

Sodium hydroxide	0·02N
Sulphuric acid	0·02N
Indicator solution	Methyl orange screened with Xylene Cyanol FF, 0·05 per cent solution in industrial methylated spirit.[§]

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[*] A suitable apparatus and procedure for preparing water of this quality is given in B.S. 2087, ' Chemical requirements for treated textiles '.

[†] B.S. 2087, ' Chemical requirements for treated textiles '.

[‡] B.S. 2690, ' Methods of testing water used in industry '.

[§] It should be noted that the use of industrial methylated spirit is governed by the Methylated Spirits Regulation 1952 (S.I. 1952, No. 2230).

5.2 Procedure. Add a few drops of the indicator solution to an aliquot consisting of at least one-fifth of the total aqueous extract and then titrate with acid or alkali as is indicated. If only 0·5 ml or less of acid or alkali is required to change the indicator colour a negative result shall be reported. If the titration is greater than 0·5 ml the free acid or alkali shall be reported as equivalent to the total millilitres of 0·02N solution per gramme of unextracted rubber.

SECTION 6 pH VALUE

The pH value of the aqueous extract shall be determined electrometrically with a glass electrode used in accordance with the principles laid down in B.S. 1647.* If the pH of the aqueous extract lies above or below the range 2·5 to 9·5, then this fact shall be recorded instead of a pH value.

SECTION 7 CHLORIDE

(Determination by direct titration)

7.1 Reagents. All reagents used shall be of recognized analytical reagent quality, and demineralized water shall be used whenever water is specified.

Nitric acid	5N
Silver nitrate	0·025M
Indicator solution	0·2 g *p*-dichlorofluorescein in 100 ml of industrial methylated spirit.†

7.2 Procedure. Take an aliquot portion of the aqueous extract consisting of at least one-tenth of the total, acidify with dilute nitric acid, and titrate with silver nitrate using a 5 ml burette graduated in 0·01 ml and using the adsorption indicator. Express the result as percentage chloride on the initial weight of the test portion.

SECTION 8 SULPHATE

(Determination by direct titration)

8.1 Reagents. All reagents used shall be of recognized analytical reagent quality, and demineralized water shall be used whenever water is specified.

Acetic acid	5N
Barium chloride	0·01M solution with pH of 3·0 to 3·5 adjusted by the addition of hydrochloric acid
Magnesium acetate	0·25M solution, approximately
Methanol	
Potassium sulphate	0·01M

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* B.S. 1647, ' pH scale '.

† It should be noted that the use of industrial methylated spirit is governed by the Methylated Spirits Regulation 1952 (S.I. 1952, No. 2230).

Indicator	0·2 per cent solution of Alizarin red S in water
Universal indicator paper	

8.2 Procedure. To 10 ml of the aqueous extract add 2 ml of potassium sulphate solution and 12 ml of methanol. Adjust the pH of the solution to between 3·0 and 4·0 with the aid of indicator paper using acetic acid or magnesium acetate solution as may be necessary. Commence the titration, using a 5 ml burette graduated in 0·01 ml, by adding 0·5 ml of barium chloride from the burette and only after this first addition add 3 drops of indicator solution. Continue the titration to the first permanent pink shown in the precipitate. Carry out a blank determination by repeating the titration with the same quantities of reagents but using 10 ml of demineralized water in place of the aqueous extract.

$$\text{Sulphate, per cent} = 0{\cdot}192 \times (A - B)$$

where A = ml of barium chloride solution used with aqueous extract
B = ml of barium chloride solution used with blank.

SECTION 9 AMMONIUM SALTS

9.1 Introductory note. This test is the familiar Nessler test for the presence of ammonia or amines. It is used here only to establish the absence of such compounds.

9.2 Reagents. All reagents used shall be of recognized analytical reagent quality, and demineralized water shall be used wherever water is specified.

Potassium iodide	
Mercuric chloride	Saturated aqueous solution
Sodium hydroxide	9N
Nessler's reagent	Dissolve 5 g of potassium iodide in 5 ml of water. Add a saturated solution of mercuric chloride dropwise until a slight precipitate appears and then add 40 ml of sodium hydroxide solution. Dilute to 100 ml, place in darkness for 24 hours and use after decanting from any precipitate. The reagent should be stored away from light.

9.3 Procedure. Place 45 ml of the aqueous extract in a 50 ml glass Nessler cylinder* and add 5 ml of the reagent. An orange-brown colour develops if ammonia is present and, if it is present in excess of about 0·02 per cent NH_3 on the test portion, some turbidity develops immediately. In the absence of this turbidity it is reasonable to infer that an ammonium salt has not been used in the preparation of the rubber compound.

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* B.S. 612, ' Nessler cylinders '.