BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

PARTS B1 TO B5

PREPARATION OF MATERIAL AND EXTRACTION METHODS

B.S. 903 : Parts B1 to B5 : 1958

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This standard makes reference to the following British Standards:

B.S. 410	Test sieves.
B.S. 509	Acetone.
B.S. 572	Interchangeable conical ground glass joints.

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The following B.S.I. references relate to the work on this standard:—
Committee references RUC/10, RUC/10/5
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*Institution of the Rubber Industry
*Ministry of Supply
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BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Parts B 1 to B 5. Preparation of Material and Extraction Methods

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, it has also been considered desirable to publish B.S. 903 in separate parts and the present part replaces Parts 1 and 2 of B.S. 903 : 1950.

The principal alteration in this document is the deletion of the method for determining paraffin wax and ceresin and the inclusion of an alternative apparatus in the acetone extraction.

This document includes the following parts:—

Part B 1	Preparation of material.
Part B 2	Acetone extract.
Part B 3	Unsaponifiable matter in acetone extract.
Part B 4	Chloroform extract.
Part B 5	Alcoholic potash extract.

PART B 1. PREPARATION OF MATERIAL FOR CHEMICAL ANALYSIS

1 General precautions. Care shall be taken that any test portion is representative of the sample with respect to the property or constituent to be determined. Thus, if it is desired to deduce the composition of the original mix, any surface bloom shall be incorporated, but if the bulk composition is required, bloom shall be excluded.

2 Soft vulcanized rubber. The sample shall be comminuted to pass a No. 10 mesh B.S. test sieve,* with scissors, a rotating rasp, or a suitable grinder. Alternatively, it shall be sheeted to a thickness not exceeding 0·5 mm by passing between the cold, tightly-closed rolls of a laboratory mill. The type of grinder or mill used is immaterial provided the sample does not become contaminated or unduly heated.

3 Ebonite. The sample shall be rasped to a powder which will pass a No. 44 mesh B.S. test sieve.* The powder shall be treated with a magnet to remove any iron particles.

4 Rubberized fabric. If possible, some of the rubber shall be separated from the fabric without wetting; exposure to chloroform vapour is often sufficient to do this and is preferable to wetting. The swollen rubber shall be completely dried in air at room temperature and treated as described in Clause 2.

If it is impossible to separate sufficient rubber from the fabric, the material shall be cut into small pieces and analysed as a whole. In this case the proportion by weight of fabric in the material shall be determined by disintegration of the rubber by a high-boiling solvent and it shall be allowed for in calculating results.

* B.S. 410, 'Test sieves'.

PART B 2. ACETONE EXTRACT

1 Explanatory note. Provided extraction is sufficiently prolonged, acetone completely extracts the following common ingredients, amongst others, of vulcanized rubber:—

Certain non-rubber constituents of the crude rubber (fatty acids, quebrachitol, sterols, etc., from natural rubber, and emulsifying agents, anti-oxidants, and other similar materials used in making synthetic rubbers), some resins, sulphur, mineral oils, wool grease, saponifiable waxes, paraffin wax, ceresin, pine tar, and some dyestuffs, peptizing agents, organic accelerators and anti-oxidants and/or their decomposition products.

Acetone incompletely extracts:—

Fatty oils, oxidized (blown) fatty oils, vulcanized fatty oils (rubber substitutes), mineral rubber (bitumen), oxidized rubber, and some of the polymer from vulcanized thioplasts or butadiene-acrylonitrile copolymers (for these synthetic rubbers it is therefore preferable to use either methanol or light petroleum (b.p. 80–100°C) in place of acetone).

2 Reagent.

Acetone. The acetone used for this determination shall be substantially free from water and polymers of acetone, and shall be of recognized quality.*

3 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 of glass except in patterns where an 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 without the use of cork, rubber, or metal and in such a manner that loss of vapour during extraction does not exceed 20 per cent of the extracting liquid. Suitable types of apparatus are shown in Figs. 1 and 2.

4 Procedure. The sample may be either comminuted or sheeted as described in B.S. 903, Part B 1, and shall be protected from light until the test is completed.

If the sample has been comminuted, place a weighed test portion (2 g to 4 g) in an acetone-extracted paper thimble.

If the sample has been sheeted, wrap loosely a weighed test portion (2 g to 4 g) in filter paper or de-sized linen, previously acetone-extracted, so that no two portions of the surface of the sheet come into contact with each other.

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* Material to B.S. 509, ' Acetone ', is suitable.

Extraction cup	Joint*		Receiver
	A	B	
ml			ml
20–30	B34	B34	150
50–60	B45	B34	250

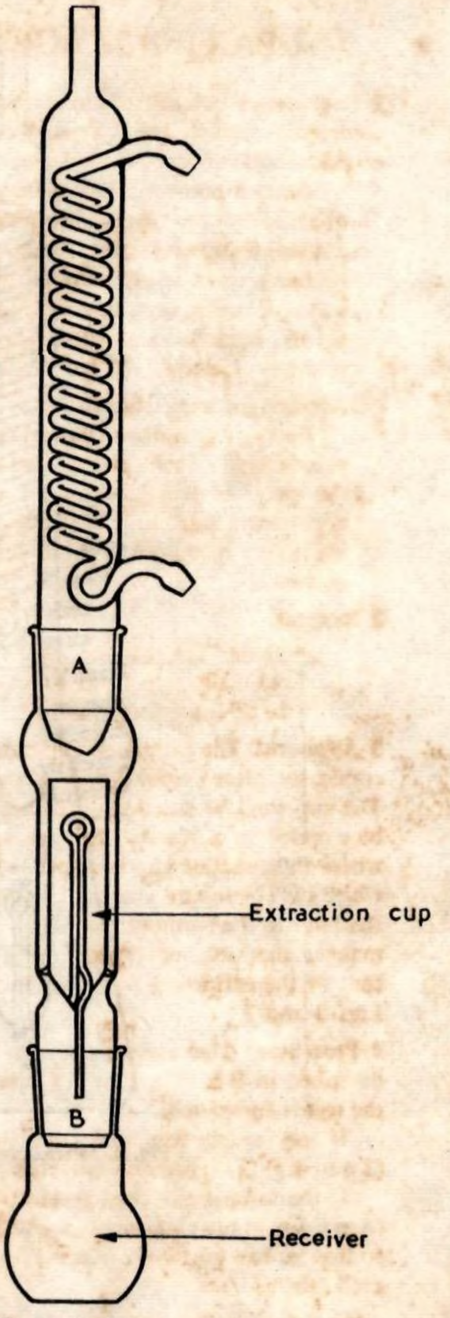

Fig. 1. Extraction apparatus

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* B.S. 752, 'Interchangeable conical ground glass joints'.

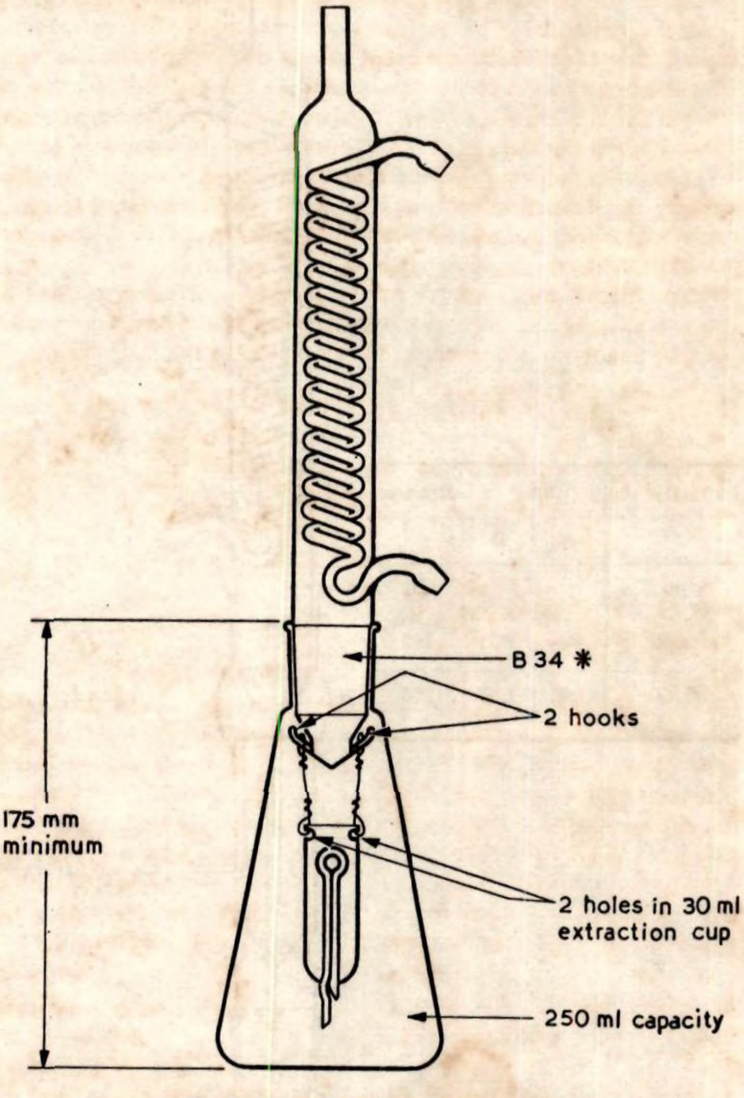

Fig. 2. Alternative extraction apparatus

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

Place the prepared test portion in the extraction cup, and pour into the extraction flask sufficient acetone to fill the extraction cup 2 or 3 times. Subject the test portion to at least 80 hot extractions during a continuous period of not less than 8 or more than 16 hours in the case of soft rubber, and to at least 160 hot extractions during a continuous period of not less than 16 or more than 40 hours in the case of ebonite.

Distil the acetone from the soluble matter in a weighed vessel, preferably the extraction flask, and as soon as all the acetone has been removed, dry the extract at 70–75°C for one hour; cool in a desiccator and weigh.

For precise work and for referee purposes carry out a blank determination, using the same quantities of reagent and the same conditions of test, and apply a corresponding correction to the main result.

5 Expression of results. The weight of the extract (corrected if necessary as described above), expressed as a percentage of the weight of the test portion, shall be taken as the ' acetone extract '.

PART B 3. UNSAPONIFIABLE MATTER IN ACETONE EXTRACT

1 Explanatory note. The unsaponifiable portion of the acetone extract includes unsaponifiable non-rubber constituents of the crude rubber, and any mineral oil, paraffin wax, ceresin, mineral rubber (bitumen) and any unsaponifiable oxidation products of the rubber that are present in the extract.

2 Reagents.

*Acetone.**

Alcoholic potassium hydroxide solution: 5·6 g potassium hydroxide in 100 ml ethanol.

Diethyl ether: freedom from peroxides is essential to avoid risk of explosion in distillation.

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

3 Procedure. To the vessel containing the dried acetone extract add 25 ml of alcoholic potassium hydroxide solution. Boil under a reflux condenser for two hours and then evaporate to dryness on a water bath. Dissolve in about 50 ml of water and transfer to a separating funnel.

Extract with 25 ml of diethyl ether. Separate the ether layer and continue to extract the aqueous layer with three further 10 ml portions of ether. Wash the combined ether extracts repeatedly with half their volume of water until the washings are no longer alkaline to litmus.

Transfer the ethereal solution to a weighed flask and distil off most of the ether. To remove the last traces of water, add about 4 ml of acetone and carefully evaporate on a water bath, blowing out the vapour with a gentle current of air. Repeat the acetone treatment if traces of water still remain (as indicated by mistiness on the sides of the flask); dry the flask and residue for one hour at 70–75°C, cool in a desiccator and weigh.

Carry out a blank determination using the same quantities of reagents and the same conditions of test, and apply a corresponding correction to the main result.

4 Expression of results. The corrected weight of the extract, expressed as a percentage of the weight of the test portion, shall be taken as the ‘ unsaponifiable matter in the acetone extract ’.

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* Material to B.S. 509, ‘ Acetone ’, is suitable.

PART B 4. CHLOROFORM EXTRACT

1 Explanatory note. Chloroform will extract from soft vulcanized rubber, which has been previously subjected to extraction as decribed in Part B 2, about half the mineral rubber (bitumen) which remains after the acetone extraction. As a small amount of the rubber may be extracted, a small chloroform extract (e.g. not exceeding 4 per cent calculated on the rubber content in the case of natural rubber) does not necessarily indicate the presence of bitumen; the latter can, however, usually be recognized by its dark colour. Chloroform extraction should not be applied to thioplasts owing to their relatively high solubility.

2 Reagent.

Chloroform.

NOTE. This shall be of recognized analytical reagent quality.

3 Procedure. Determine the chloroform extract immediately after the acetone extraction, without drying the acetone extracted rubber. Replace the extraction vessel used for the acetone extraction by one containing sufficient chloroform to fill the extraction cup approximately twice, and extract the rubber with this solvent for four hours or until the liquid flowing from the siphon is colourless, whichever is the longer. The rate of distillation of the chloroform shall be such that at least seven hot extractions occur per hour. Distil off the chloroform and dry the extract for one hour at 70–75°C, cool in a desiccator and weigh.

4 Expression of results. The weight of the extract expressed as a percentage of the weight of the test portion shall be taken as the ' chloroform extract '.

PART B 5. ALCOHOLIC POTASH EXTRACT

1 Explanatory note. The alcoholic potash extraction is made on rubber previously extracted with acetone and/or chloroform; if the determination of acetone extract is not required, the rubber may be extracted directly with chloroform; if the acetone extract is required and mineral rubber (bitumen) is known to be absent, the chloroform extraction may be omitted. The alcoholic potash extract consists of the fatty acids derived from saponifiable constituents of the vulcanized rubber, so far as these remain after the previous extraction(s). The most important of these constituents are: vulcanized oils (rubber substitutes), fatty oils, oxidized (blown) oils, and oxidized rubber. The alcoholic potash extract is usually taken as an index of the amount of the vulcanized oils present in the rubber. The method is not applicable to thioplasts, since they are attacked by potassium hydroxide.

2 Reagents.

*Acetone.**

Alcoholic potassium hydroxide solution: 5·6 g potassium hydroxide in 100 ml ethanol.

Ethanol: 95 per cent.

Diethyl ether: freedom from peroxides is essential to avoid risk of explosion during distillation.

Hydrochloric acid: concentrated, sp. gr. 1·18.

Methyl orange indicator solution.

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

3 Procedure. Dry the rubber from the acetone or chloroform extraction at room temperature in a vacuum. It is essential that exposure of the dried extracted rubber to the air should be kept as short as possible. Place the rubber in a suitable flask and add 50 ml of alcoholic potassium hydroxide solution; boil under a reflux condenser for four hours, using a ground glass joint to connect the condenser to the flask.

Decant the solution through a filter and collect the filtrate in a porcelain basin. Wash the rubber with two 25 ml portions of hot ethanol and then thoroughly with hot water, decanting the washings through the filter and collecting with the main filtrate.

Evaporate the combined filtrate and washings to dryness on a boiling water bath and dissolve the residue in 50 ml of water. Transfer the solution to a separating funnel and carefully add concentrated hydrochloric acid until the liquid is slightly acid. Allow the solution to cool.

Extract the acidified liquid completely with three 25 ml portions of diethyl ether and wash the combined ether extracts with water until the washings are no longer acid when tested with methyl orange.

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* Material to B.S. 509, ' Acetone ', is suitable.

Transfer the ether solution to a weighed flask and distil off most of the ether. To remove the last traces of water, add about 4 ml of acetone and carefully evaporate on a water bath, blowing out the vapour with a gentle current of air. Repeat the acetone treatment if traces of water still remain (as indicated by mistiness on the sides of the flask); dry the flask and residue for one hour at 70–75°C, cool in a desiccator and weigh.

Carry out a blank determination, using the same quantities of reagents and the same conditions of test, and apply a corresponding correction to the main result.

4 Expression of results. The corrected weight of the extract, expressed as a percentage of the weight of the original test portion, shall be taken as the ' alcoholic potash extract '.

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