

Specification for

Equipment and general procedure for mixing and vulcanizing rubber test mixes

Spécification pour l'appareillage et le mode opératoire général pour le mélangeage et la vulcanisation des mélanges d'essai à base d'élastomères

Spezifikation für Geräte und allgemeines Verfahren für die Mischung und Vulkanisierung von Elastomer-Prüfmischungen

Committees responsible for this British Standard

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British Railways Board
British Rubber Manufacturers' Association
ERA Technology Ltd.
GAMBICA (BEAMA Ltd.)
Institution of Mechanical Engineers
Institution of Water Engineers and Scientists
Malaysian Rubber Producers' Research Association
Ministry of Defence
National College of Rubber Technology
Rubber and Plastics Research Association of Great Britain
SATRA Footwear Technology Centre

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Foreword

This British Standard has been prepared under the direction of the Plastics and Rubber Standards Policy Committee. It supersedes BS 1674 : 1976 which is withdrawn.

The principal changes introduced in this revision are as follows:

- (a) further alignment with ISO 2393;
- (b) correction of drawings of the stepped test piece mould;
- (c) removal of the description of a mill temperature control system, as it is felt that such items are now sufficiently commercially available;
- (d) detailed review of the requirements for the laboratory mill, internal mixer and mixing procedure.

This British Standard differs from ISO 2393 in the following important respects.

- (1) It does not cover the preparation of ring type test pieces since these are used very little in the United Kingdom.
- (2) It allows a wider range of mill roll speeds than ISO 2393 following considerable laboratory investigations which showed that within fairly wide limits roll speed has only a limited effect on the properties of the vulcanized mix.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Specification

0 Introduction

The processes of mixing and vulcanizing are subject to a number of types of variability which are believed to arise from various causes including the following.

- (a) The source and purity of rubber and compounding ingredients used.
- (b) The accuracy of weighing the ingredients.
- (c) The size and type of mixing equipment.
- (d) The temperature of the rubber during the mastication and mixing processes.
- (e) The manipulation during mixing, including loss of powders.
- (f) The humidity and temperature of the atmosphere in which the unvulcanized mix is stored prior to vulcanization.
- (g) The cooling of the mould during loading of the blank.
- (h) The accuracy and constancy of the mould temperature during the course of vulcanization.
- (i) The time taken to strip the vulcanized rubber from the mould, and to cool it to room temperature.

The contribution of such factors to the total variation depends on the type of mix.

1 Scope

This British Standard specifies the equipment and general procedure to be adopted in mixing and vulcanizing rubber test mixes, other than those for ebonite and cellular rubber, for subsequent laboratory testing.

NOTE. The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this British Standard the definitions given in BS 3558 apply, together with the following.

batch mass

The mass of the test mix prepared in one mixing operation.

3 General safety precautions

Mechanical guarding of apparatus shall comply with current legislation and with BS 5304. Means shall be provided for exhaust ventilation at the apparatus to control injurious or offensive dust or vapour. Air flow quantities shall be adequate to comply with all national and international safety legislation.

4 Equipment

4.1 Laboratory mill

NOTE. For marking, see 4.3.

4.1.1 General

This mixing mill shall be a two roll laboratory mixing mill for polymer with rolls nominally 150 mm in diameter and 300 mm long.

NOTE. This mill is also known as a 6 in × 12 in mill.

The mill shall be capable of operating satisfactorily at temperatures of up to and including 175 °C and at fluid pressures inside the rolls of up to and including 12 bar ¹⁾ (1.2 MPa), and shall be fitted with guides to ensure that material is contained on the rolls.

4.1.2 Mill rolls

The mill shall comply with the following:

Roll diameter (O.D.)	150 ± 5 mm
Roll length between guides (maximum)	280 mm
Speed of front (slow) roll (preferred)	24 ± 1 r/min (see note 1)
Roll speed ratio (preferred)	1 : 1.4
Clearance between rolls	0.2 mm to 8.0 mm (see note 2)

Rolls shall be such that circulation of a fluid medium through the rolls will maintain the surface temperature of the roll at ± 5 °C of that specified for a particular mix. Rotational direction of the rolls shall be such that the ingoing nip is at the top, and shall not be reversible.

NOTE 1. It has been shown that variations in roll speed of the fixed speed roll between 19 r/min and 34 r/min, and of friction ratio between 1.0 : 1 and 1.6 : 1 do not significantly alter the properties of the vulcanized mix.

NOTE 2. The clearance is normally determined by means of two lead strips 10 ± 3 mm in width, at least 50 mm long and 0.25 mm to 0.5 mm thicker than the roll clearance to be measured. The lead strips are inserted, one at each end of the rolls, approximately 25 mm from the guides, while a piece of compound rubber, with a Mooney viscosity, in excess of 50 ML (1 + 4) 100 °C (see BS 903 : Part A58) and measuring approximately 75 mm × 75 mm × 6 mm is passing through the centre portion of the nip. The rolls should be at the temperature specified for mixing. After passing through the nip, the thickness of the lead strips is measured with a micrometer to an accuracy of ± 0.01 mm. Tolerance on roll clearance should be ± 10 % or ± 0.05 mm, whichever is the larger.

4.1.3 Temperature measurement

A suitable surface pyrometer, accurate to ± 1 °C shall be used for measurement of the roll surface temperatures.

¹⁾ 1 bar = 10⁵ N/m² = 10⁵ Pa.

4.2 Internal mixer

4.2.1 General

Internal mixers of type A shall have tangential rotors, and those of type B shall have intermeshing rotors. All types of internal mixer shall be arranged so that machine temperature is controlled by a circulating temperature control fluid.

The internal mixer shall comply with the details given for either type A or type B in table 1.

4.2.2 Mixing control

All internal mixers shall be fitted with the following:

- (a) a system to measure, indicate and record the temperature of the mix during the mixing operation;

NOTE. The actual mix temperature usually exceeds the indicated temperature by an amount dependent on the mixing conditions used.

- (b) a timer to indicate the mixing time;
- (c) a system to indicate and record electrical power demand or torque;
- (d) an efficient temperature control system to control the metal temperatures of the rotors and mixing chamber.

4.2.3 Mixer closure

All internal mixers shall be closed with a ram to contain the mix in the mixing chamber.

4.2.4 Rotor clearance (worn)

Rotor clearances shall be as given in table 1.

NOTE 1. When clearances exceed the values noted in the above table, overhaul is necessary or mixing quality may be adversely affected. This increase in rotor clearance may be equated to an increase of approximately 10 % in mixing capacity.

NOTE 2. For best results, all comparative test mixes should be mixed in the same internal mixer.

4.3 Marking

The mill and the mixer shall both be legibly and clearly marked with the number and date of this British Standard, i.e. BS 1674 : 1991¹⁾.

5 Mixing procedure

5.1 Purpose

NOTE. The objective of the mixing process is the uniform dispersion of the compounding ingredients in the rubber. The general principles of 5.1.1 to 5.1.3 apply to mixing of all types.

5.1.1 Compounding ingredients

Where applicable, ingredients complying with BS 4398 shall be used, and in the special case of inter-laboratory comparisons the ingredients used shall be drawn from the same batch.

Carbon black shall be conditioned as described in appendix A and all other ingredients shall be dry and stored in the dark in a dry atmosphere at a temperature of 23 ± 2 °C prior to weighing and during the period between weighing and mixing.

5.1.2 Weighing tolerances

The rubber and all compounding ingredients shall be weighed to within 0.25 % of the mass specified or to within 10 mg, whichever is the greater.

5.1.3 Mixing technique

The mixing technique used shall be as stated in the appropriate rubber specification.

5.2 Mill mixing

5.2.1 Batch volume

The preferred maximum volume of raw rubber for the standard mill shall be 450 cm³, with a preferred minimum volume of 200 cm³. The maximum volume of the complete mix shall not exceed 1000 cm³.

5.2.2 Batch mass

The mass of the mixed batch and the sum of the masses of all the ingredients shall not differ by an amount exceeding 0.3 % for an unfilled mix or 0.6 % for a filled mix.

Table 1. Machine details of internal mixers

Features	Type A Tangential rotors	Type B Intermeshing rotors
Mixing capacity (cm ³)	1170	1000
Rotor speed (r/min) (fast rotor)	110 ± 10	55
Friction ratio	1.125 : 1	1 : 1
Rotor clearance (new) (mm)	2.38 ± 0.13	2.5 + 0.00, - 0.05
(worn) (mm)	3.7	5.0
Power (fast rotor) (kW/r/min)	0.13	0.227
Ram pressure (MPa) (see note)	0.8	0.4

NOTE. The ram pressure should be specified in the appropriate material mixing specification.

¹⁾ Marking BS 1674 : 1991 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is therefore solely the responsibility of the person making the claim. Such a declaration is not to be confused with third party certification of conformity, which may also be desirable.