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Method of test

Determination of crystallization effects in rubbers by hardness measurements

Méthode d'essai pour la détermination des effets de crystallisation sur les élastomères au moyen des mesures de la dureté

Prüfverfahren für die Bestimmung der Kristallbildung in Gummi durch Härtemessungen

British Standards Institution

Gr 3

Contents

Foreword

This British Standard has been published under the authority of the Rubber Industry Standards Committee. It is based on the corresponding method described in Draft International Standard ISO/DIS 3387 'Rubbers—Determination of crystallization effects by hardness test' prepared by Technical Committee TC/45 Rubber and Rubber Products of the International Organization for Standardization (ISO).

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British Standard Method of test

Determination of crystallization effects in rubbers by hardness measurements

1. Scope

This British Standard describes a method of test for the determination of the crystallization effects in rubber by hardness measurements made in accordance with BS 903:Part A26. Provided the material has an initial hardness at the test temperature within the range 10 IRHD to 80 IRHD, it is applicable to the following materials:

(a) raw rubber;

(b) unvulcanized (compounded) rubber;

(c) vulcanized rubber.

The method is mainly of use for rubbers with a marked crystallization tendency at temperatures experienced in cold climates.

NOTE. An alternative method for measuring crystallization effects is given in BS 903:Part A29, but it is applicable to vulcanized rubber in the strained state only.

2. References

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

3. Summary

This method describes a hardness test procedure for determining the progressive stiffening of rubber with time, caused by crystallization occasioned by storage at a specified temperature.

The method measures either the time required for half the total hardness increase between the initial and final hardnesses to occur, or the hardness increase after a specified time at the test temperature.

4. Test piece

The test piece shall have its upper and lower surfaces flat, smooth and parallel to one another. The standard test piece shall be between 8 mm and 10 mm thick, with a side length or diameter of at least 42 mm.

Thick or thinner test pieces may be used, but in no case shall the test piece be thinner than 4 mm when method N of BS 903:Part A26 is used, or thinner than 6 mm when method L of that standard is used. In order to avoid edge effects the side length or diameter of the test piece shall be as specified in table 1 and no test shall be made at a distance from the edge of the test piece less than that specified in table 1.

Table 1. Test piece dimensions

Thickness of test piece (mm)	4	6	8	10	15	25
Minimum side length or diameter of test piece (mm)	38	40	42	44	47	50
Minimum distance of test from edge (mm)	7	8	9	10	11.5	13

NOTE. Tests carried out on test pieces of different thicknesses do not necessarily give the same values of hardness. Tests intended to be comparable should be made on test pieces of the same thickness.

5. Apparatus

The following apparatus is required.

5.1 *Cold chamber*, capable of temperature control within ± 1 °C of the specified temperature. A gaseous heat-transfer medium shall be used. The chamber shall be such that the hardness of the test pieces can be determined without removing them from the chamber. The provision of hand holes, fitted with gloves, in the door or walls of the chamber, i.e. 'glove-box' installation, is suitable.

5.2 *Hardness gauges,* complying with the requirements for the apparatus described in BS 903: Part A26 for methods N and L.
Lubricants used in the apparatus shall be of a type not to cause friction in the apparatus at the test temperature.

5.3 *Tweezers or tongs* for handling the test pieces, and gloves for handling the test equipment.

5.4 *Heated press and moulds* for the preparation of test pieces of raw or unvulcanized rubbers.

6. Procedure

6.1 Preparation of test pieces

6.1.1 *Raw rubber.* Prepare test pieces of raw rubber by placing a suitable quantity in a pre-heated mould and then applying pressure.

NOTE. Values of mould temperature and time of application of pressure required to produce a suitable test piece depend upon the type of rubber. 10 min at 150 °C has been found suitable in many cases.

Then cool the mould to 23 °C under pressure for 15 min and remove the test piece.

6.1.2 *Unvulcanized compounded rubber.* Prepare test pieces of unvulcanized compounded rubber by placing a suitable amount of the freshly milled rubber into a pre-heated mould and then applying pressure.

NOTE. Values of mould temperature and time of application of pressure required to produce a suitable test piece depend upon the rubber compound, and can only be arrived at by experience. A temperature of 120 °C applied for 3 min has been found satisfactory for many materials. However, for some materials longer times or higher mould temperatures may be found necessary to ensure a smooth and flat test piece surface. Under no circumstances should conditions be used that cause incipient cure.

Then cool the mould to 23 °C under pressure for 15 min and remove the test piece.

6.1.3 *Vulcanized rubber.* Prepare test pieces of vulcanized rubber by moulding or by cutting from moulded sheet. Two pieces of rubber may be superimposed to obtain the necessary thickness, in which case the thicker piece, if the two are unequal, shall be placed on top.

6.2 Conditioning of test pieces. The minimum time between moulding or vulcanization and testing shall be 16 h. For non-product tests the maximum time between moulding or vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable, the tests as far as possible shall be carried out after the same time interval. For product tests, whenever possible, the time between vulcanization and testing shall not exceed 90 days. In other cases tests shall be made within 60 days of the date of receipt by the customer of the product.

In all cases decrystallize the test pieces immediately before testing by heating them for 45 min in an oven at 70 °C. Then remove them from the oven, condition at 23 ± 2 °C for at least 30 min but not more than 60 min and then place them in the cold chamber.

6.3 Hardness measurements

6.3.1 *General.* Make hardness measurements as described in BS 903:Part A26. Make one measurement at each of three or five different points distributed over the test piece and at least 10 mm from its edge, and take the median of the results. All subsequent readings shall be at points at least 4 mm away from points used for any previous reading.

Use the same hardness apparatus throughout any one test, the appropriate apparatus being determined from the initial hardness at the test temperature. For initial hardnesses between 10 IRHD and 30 IRHD use the apparatus according to method L, and for initial hardnesses between 30 IRHD and 80 IRHD use the apparatus according to method N. If the hardness increase gives values above 30 IRHD for method L determine the hardness readings from an extension of table 6 of BS 903:Part A26:1969 calculated according to the relationship given in appendix A of that standard.

6.3.2 *Initial hardness at test temperature.* Condition the hardness apparatus and the tweezers or tongs in the cold chamber at the desired test temperature for at least 30 min. Place the test piece in the cold chamber at the desired test temperature. After 30 ± 1 min for the standard or thinner test pieces, or 45 ± 1 min for thicker test pieces, take the first hardness reading using the tweezers or tongs for handling the test pieces and gloves for handling the test equipment. If the initial hardness reading is above 80 IRHD the method is not applicable.

NOTE. The hardness apparatus according to this test procedure should normally be conditioned and operated inside the cold chamber. Alternatively, a special device may be used where the body of the hardness apparatus is placed outside the cold chamber and connected with the indentor in the cold chamber by means of a rod with low heat-conductive capacity, and constructed to avoid the introduction of additional friction.

6.3.3 *Hardness increase.* Repeat the hardness measurements after the times of storage at test temperature as specified in **7.2.** Avoid taking measurements at points with 4 mm of those used for previous measurements.

NOTE. After measurements have been taken, it is advisable to dry all apparatus by removing it from the chamber and warming it with circulating air to approximately 40 °C.

7. Test conditions

7.1 Temperature of test. The temperature of test is dependent upon the purpose for which the test is being carried out. This may be specified for special reasons, as for example, the storage temperatures of raw natural rubber.

If not otherwise specified, the temperature of test shall be selected from the following list:

23 ± 2 °C
10 ± 1 °C
0 ± 1 °C
−10 ± 1 °C
−25 ± 1 °C
−40 ± 1 °C
−55 ± 1 °C

7.2 Duration of test. Take hardness measurements at sufficient periods of time after the initial measurement to enable a plot of hardness against time to be prepared (see figures 1 and 2). If possible, these shall be of sufficient duration to allow the final maximum hardness to be determined (see figure 1) but if this time is inconveniently long, then the test shall be terminated after 168 +0, −2 h or 24 +0, −2 h for rapidly crystallizing materials.

Logarithmic time intervals are an advantage.

8. Expression of results

The preferred method of reporting the test results shall be to quote the time required for half of the total hardness increase between initial and final hardness to occur (see figure 1). This time shall be determined from a smoothed plot of hardness versus time; the method assumes that sufficient hardness measurements are made to reach a constant level of final hardness with time.

An alternative method of reporting the test results shall be to quote the increase in hardness from the initial hardness reading after a defined storage time (see figure 2). Normally this shall be 168 +0, −2 h but 24 +0, −2 h may be used for rapidly crystallizing materials.

9. Report

The report shall state the following information.

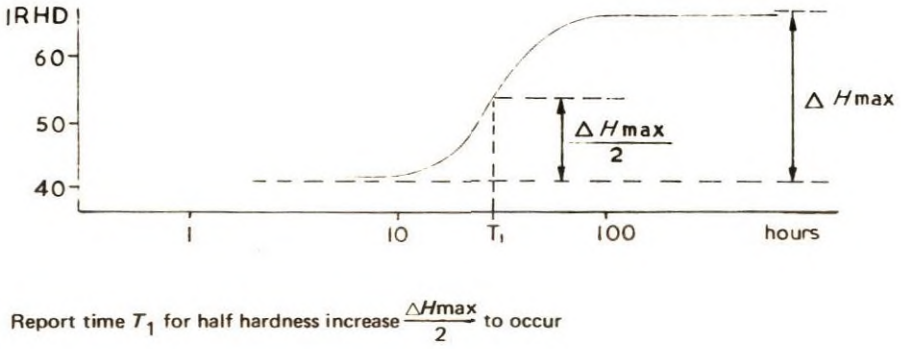
(a) Time in hours for half the hardness increase between initial and final hardness to occur or hardness increase after 168 +0, −2 h, or 24 +0, −2 h for rapidly crystallizing materials (see **7.2**), of storage at the test temperature.

(b) Temperature of test.

(c) Time of storage at test temperature.

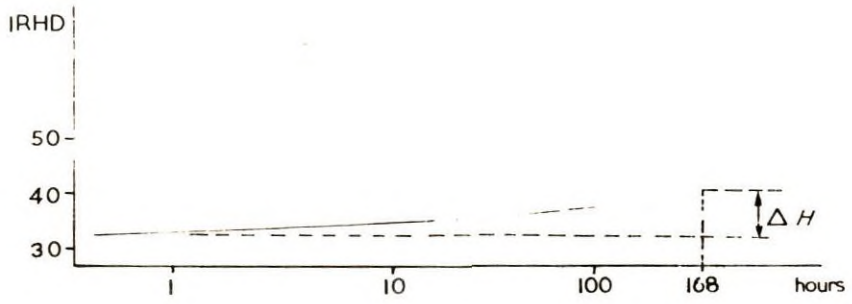
(d) Initial hardness at test temperature and whether method N or L is used.

(e) The thickness of the test piece and whether made up of one or two pieces.



Report time T_1 for half hardness increase $\frac{\Delta H\text{max}}{2}$ to occur

Figure 1. Method of reporting hardness increase



Report actual hardness increase ΔH after 168 h

Figure 2. Alternative method of reporting hardness increase

BSI publications referred to in this standard

This standard makes reference to the following British Standards:

BS 903 Methods of testing vulcanized rubber
Part A26 Determination of hardness
Part A29 Determination of low-temperature retraction (TR test)

This British Standard, having been prepared under the direction of the Rubber Industry Standards Committee, was published under the authority of the Executive Board on 27 February 1976.

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Attention is drawn to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

Revision of British Standards
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 reference RUC/10 and RUC/10/4
Draft for comment 73/53043DC

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:

British Association of Synthetic Rubber Manufacturers
*British Rubber Manufacturers' Association Ltd
Department of Industry
*Malaysian Rubber Producers Research Association
*Ministry of Defence
*Rubber and Plastics Research Association of Great Britain
Rubber Growers' Association
*Society of Motor Manufacturers and Traders Ltd

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 Railways Board
Chemical Industries Association
Department of the Environment
Electrical Research Association
Institution of Mechanical Engineers
Institution of Municipal Engineers
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