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DETERMINATION OF HARDNESS OF VULCANIZED RUBBERS

2nd EDITION

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This second edition supersedes the first edition

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BRIEF HISTORY

The ISO Recommendation R 48, *Determination of hardness of vulcanized natural and synthetic rubbers,* was drawn up by Technical Committee ISO/TC 45, *Rubber,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1948 and led, in 1953, to the adoption of a Draft ISO Recommendation.

In August 1954, this Draft ISO Recommendation (No. 50) was circulated to all the ISO Member Bodies for enquiry. It was approved by 26 Member Bodies. No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in July 1957, to accept it as an ISO RECOMMENDATION.

BRIEF HISTORY RELATING TO THE 2nd EDITION

Work for the revision of ISO Recommendation R 48-1957 was subsequently started and, in 1963, a draft proposal was adopted by the Members of Technical Committee ISO/TC 45 as a Draft ISO Revision.

In November 1964, this Draft ISO Revision (No. 742) was circulated to all ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Hungary	Poland
Australia	India	Spain
Austria	Iran	Sweden
Canada	Israel	Switzerland
Czechoslovakia	Italy	Turkey
Chile	Japan	U.A.R.
France	Korea, Rep. of	United Kingdom
Germany	Netherlands	U.S.A.
Greece	New Zealand	U.S.S.R.

No Member Body opposed the approval of the Draft.

The Draft ISO Revision was then submitted by correspondence to the ISO Council which decided, in September 1968, to accept it.

The title of ISO Recommendation R 48-1957 is amended as follows : *Determination of hardness of vulcanized rubbers.*

The present edition (2nd edition) supersedes the first edition of ISO Recommendation R 48-1957.

ISO Recommendation R 48 September 1968

DETERMINATION OF HARDNESS OF VULCANIZED RUBBERS

INTRODUCTION

The hardness test is based on a measurement of the indentation of a rigid ball into the rubber test piece under specified conditions. For the normal test the standard test piece is between 8 and 10 mm thick; test pieces less than 8 mm thick give smaller indentation than the standard. For tests on thin pieces of rubber a scaled-down version (herein after referred to as the micro-test) of the normal test is therefore used, in which the apparatus dimensions are reduced to one-sixth. When used on a piece 1.6 to 2 mm in thickness, the result of the micro-test will be about the same as that obtained by the normal test.

It is considered unrealistic to fix a precise thickness above which the normal test should be used and below which the micro-test should be used, but in general the latter test should be used for thicknesses below about 4 mm. There will, however, be exceptions; for instance, the micro-test would be preferable even on thicknesses above 4 mm if the lateral dimensions of the test piece are much less than those specified for the normal test (see Table 2), because the latter test would then be inaccurate. The micro-test would also be preferable for testing some small awkwardly-shaped rubber articles. The figure of 4 mm has been chosen for the following reasons :

(*a*) at this thickness the normal test will give readings in international rubber hardness degrees (IRHD) higher than the "standard" reading (i.e., on 8 to 10 mm thickness), and the micro-test will give readings lower than this (because this test gives the "standard" reading on a thickness of about 1.6 to 2.0 mm). These two errors are about equal when the thickness tested is 4 mm.

(*b*) 4 mm is the greatest thickness on which the micro-test could be made without increasing the lateral dimensions of the test piece above that now specified (i.e., 2 mm minimum between the indenter and the edge of the test piece).

In either the normal test or the micro-test, the measured indentation is converted into international rubber hardness degrees, the scale of degrees being so chosen that 0 represents the hardness of a material having an elasticity modulus of zero and 100 represents the hardness of a material of infinite elasticity modulus, and so that the following conditions are fulfilled over most of the normal range of hardness :

(*a*) one international rubber hardness degree always represents approximately the same proportionate difference in Young's modulus;

(*b*) for highly elastic rubbers, the scales of international rubber hardness degrees and the Shore A durometer are comparable.

For substantially elastic isotropic materials like well-vulcanized natural rubbers, the hardness in international rubber hardness degrees bears a known relation to Young's modulus, although for markedly plastic or anisotropic rubbers the relationship will be less precisely known.

1. SCOPE

The method of test described in this ISO Recommendation should preferably be used for rubbers of a hardness range between 30 to 85 IRHD, but may be used for those rubbers with a hardness between 30 and 95 IRHD.

Methods for very hard or very soft rubbers will be the subject of separate ISO Recommendations and the range of applicability of each is indicated in the following figure :

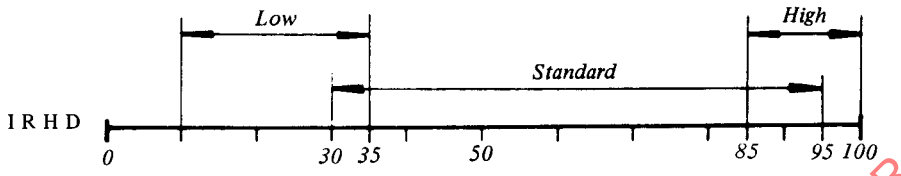

FIGURE – **Range of applicability of hardness tests**

2. PRINCIPLE OF METHOD

The hardness test consists in measuring the difference between the depths of indentation of the ball into the rubber under a small contact force and a large total force. From this difference, multiplied when using the micro-test by the scale factor 6, the hardness in international rubber hardness degrees is derived by using either Table 3, or a graph based on this table, or a scale reading directly in international rubber hardness degrees and derived from the table, fitted to the indentation-measuring instrument.

The relation between the difference of indentation and the hardness expressed in international rubber hardness degrees is based on :

(1) the known relation, for a perfectly elastic isotropic material, between indentation P, expressed in hundredths of a millimetre, and Young's modulus M, expressed in bar, namely :

$$F/M = 0.00038\ R^{0.65} p^{1.35}\ *$$

where

F is the indenting force, expressed in newtons,

R is the radius of ball, expressed in millimetres;

(2) the use of a probit (integrated normal error) curve to relate $\log_{10}$M and the hardness in international rubber hardness degrees, as shown in the diagram on page 9. This curve is defined as

(*a*) *value of $\log_{10}M$ corresponding to midpoint of curve*
= 1.364 (M expressed in bar),

(*b*) *maximum slope*
= 57 international rubber hardness degrees per unit increase in $\log_{10}$M.

* **This formula is approximate and is included as an indication.**

3. APPARATUS

The essential parts of the apparatus are as follows, the appropriate dimensions and forces being shown in Table 1.

(*a*) A vertical plunger, having a rigid ball or spherical surface on the lower end, and means for supporting the plunger so that the spherical tip is kept slightly above the surface on the annular foot prior to applying the contact force.

(*b*) Means for applying a contact force and an indenting force to the ball, allowing for the weight of the plunger and of any fittings attached to it, and for the force of any spring acting on it, in order that the forces actually applied to the ball shall be as specified.

(*c*) Means, which may be mechanical, optical or electrical, for measuring the increase in depth of indentation of the plunger caused by the indenting force, either in metric units, or reading directly in international rubber hardness degrees.

(*d*) A flat annular foot, normal to the axis of the plunger and having a central hole for the passage of the plunger. The foot rests on the test piece and exerts a pressure on it of 0.3 ± 0.05 bar, provided the total force on the foot does not fall outside the values given in Table 1. The foot is rigidly connected to the indentation-measuring device, so that a measurement is made of the movement of the plunger relative to the foot (i.e., the top surface of the test piece), not relative to the surface supporting the test piece.

(*e*) Means, for example, an electrically operated buzzer, for gently vibrating the apparatus to overcome any slight friction (this can be omitted in instruments where friction is completely eliminated).

(*f*) A chamber for the test piece when tests are made at temperatures other than a standard laboratory temperature. This chamber is equipped with means of maintaining the temperature within 2 deg C of the desired value. The foot and vertical plunger extend through the top of the chamber, and the portion passing through the top is constructed from a material having a low thermal conductivity. A sensing device is located within the chamber near or at the location of the test piece for measuring the temperature.

TABLE 1 – Forces and dimensions of apparatus

Test	Diameters	Force on ball			Force on foot
		Contact	Indenting	Total	
	mm	N	N	N	N
Normal	either ball 2.38 ± 0.01 foot 20 ± 1 hole 6 ± 1	0.30 ± 0.02	5.23 ± 0.01	5.53 ± 0.03	8.3 ± 1.5
	or ball 2.50 ± 0.01 foot 20 ± 1 hole 6 ± 1	0.30 ± 0.02	5.40 ± 0.01	5.70 ± 0.03	8.3 ± 1.5
	mm	mN	mN	mN	mN *
Micro	ball 0.395 ± 0.005 foot 3.35 ± 0.15 hole 1.00 ± 0.15	8.3 ± 0.5	145 ± 0.5	153 ± 1	235 ± 30

NOTE. – All possible combinations of dimensions and forces in Table 1 will not meet the pressure requirements of section 3 (*d*).

* In the micro-test using instruments in which the test piece table is pressed upwards by a spring (see Scott and Soden, Trans. I.R.I. 1960, 3b, 1, Fig. 8), the values of foot pressure and force on foot are those acting during the period of application of the total force. Before the indenting force of 145 mN is applied, the force on the foot is greater by this amount, and hence equals 380 ± 30 mN.

4. TEST PIECE

4.1 Dimensions of test piece

The test piece should have its upper and lower surfaces flat, smooth and parallel to one another.

Tests intended to be comparable should be made on test pieces of the same thickness.

To obtain the necessary thickness it is permissible to superimpose two pieces of rubber (but not more than two), provided these have flat parallel surfaces.

4.1.1 *Normal test.* The standard test piece should be 8 to 10 mm thick. Non-standard test pieces may be either thicker or thinner, but usually the thickness should not be less than 4 mm. The lateral dimensions of both standard and non-standard test pieces should be such that no test is made at a distance from the edge of the test piece less than the appropriate distance shown in Table 2.

TABLE 2

Total thickness of test piece	Minimum distance from point of impact to edge of test piece
mm	mm
4	7.0
6	8.0
8	9.0
10	10.0
15	11.5
25	13.0

4.1.2 *Micro test.* The standard test piece should have a thickness of 2.0 ± 0.5 mm. Because of the variable effects of surface hardening of the rubber and of any slight surface roughness (e.g., produced by buffing), no standard thickness of test piece will always give results agreeing with those of the normal test using its standard test piece. A thickness in the range 1.6 to 2.0 mm will most often give such agreement, but this will not always be exact. Thicker or thinner test pieces may be used, but in no case less than 1 mm thick. On such test pieces the readings will not in general agree with those given by the normal test. The lateral dimensions should be such that no test is made at a distance from the edge less than 2 mm.

When test pieces thicker than 4 mm are tested on the micro-test instrument because lateral dimensions or area of flatness do not permit testing on a normal instrument, the test is made at a distance from the edge as great as possible.

Curved test pieces, O-rings for example, may be tested with the micro-instrument but the values may not be comparable with those obtained on flat test pieces.

4.2 Conditioning of test pieces

Tests should not be carried out less than 16 hours after vulcanization and, for arbitration, not less than 72 hours after vulcanization.

When a test is made at a standard laboratory temperature the test pieces should be maintained at the conditions of test for at least 3 hours immediately before testing. When tests are made at higher or lower temperatures the test pieces should be maintained at the conditions of test for a period of time sufficient to reach temperature equilibrium with the testing environment, or for the period of time required by the specification covering the material or product being tested.

5. TEMPERATURE OF TEST

The test should normally be carried out at a standard laboratory temperature. The standard laboratory temperature should be 20 ± 2 °C, 23 ± 2 °C or 27 ± 2 °C, the same temperature being used throughout any one test or series of tests intended to be comparable.

6. PROCEDURE

The test piece should first be conditioned as described in clause 4.2. The upper and lower surfaces of the test piece should be lightly dusted with talcum powder, and the test piece supported on a horizontal rigid surface. The foot should first be brought into contact with the surface of the test piece. The plunger and indenting ball should be pressed for 5 seconds onto the rubber, the force on the ball being the contact force.

(*a*) If the gauge is graduated in international rubber hardness degrees it should be adjusted to read 100 at the end of the 5 seconds period. The additional indenting force should then be applied and maintained for 30 seconds, when a direct reading of the hardness in international rubber hardness degrees is obtained.

(*b*) If the gauge is graduated in metric units, the difference in indentations D (expressed in hundredths of a millimetre) of the plunger caused by the additional indenting force, applied for 30 seconds, should be noted. This (after multiplying by the scale factor of 6.0 when using the apparatus for the micro-test) should be converted into international rubber hardness degrees by using Table 3 or a graph constructed therefrom.

During the loading periods the apparatus may be gently vibrated to overcome any friction.

7. NUMBER OF READINGS

One measurement should be made at either three or five different points distributed over the test piece and the median of the results should be taken, i.e., the middle value when these are arranged in increasing order.

8. EXPRESSION OF RESULTS

Hardness should be reported to the nearest whole number as the median of the three or five measurements in international rubber hardness degrees.

9. TEST REPORT

The test report should include the following :

(*a*) dimensions of test piece and whether made up of one or two pieces;

(*b*) temperature of test;

(*c*) type of surface tested, i.e., moulded, buffed or otherwise;

(*d*) type of apparatus used, i.e., normal or micro.