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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 132

DETERMINATION OF RESISTANCE TO FLEX CRACKING OF VULCANIZED NATURAL OR SYNTHETIC RUBBER (DE MATTIA TYPE MACHINE)

1st EDITION
September 1959

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 132, *Determination of Resistance to Flex Cracking of Vulcanized Natural or Synthetic Rubber (De Mattia type machine)*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

Work on the preparation of this method was authorized at the first meeting of the Technical Committee held in London, in June 1948, and was entrusted to the Working Group on " Flex Cracking and Cut-Growth ".

It was agreed by the Working Group that the method should be based on that used in the United Kingdom.

The method was considered by the Working Group and by the Technical Committee at the meetings held in The Hague (September 1949), in Akron (October 1950), in Oxford (October 1951), in Paris (June 1953) and was finally approved, as a Draft ISO Recommendation, at the Dusseldorf meeting, in September 1955.

On 17 July 1957, the Draft ISO Recommendation (No. 172) was distributed to all the ISO Member Bodies and was approved, subject to some modifications, by the following 26 (out of a total of 39) Member Bodies:

*Australia	*Greece	Portugal
Austria	Hungary	Romania
Burma	India	Spain
*Canada	*Ireland	Sweden
Czechoslovakia	Italy	Switzerland
*Denmark	Japan	Union of South Africa
Finland	New Zealand	United Kingdom
France	Pakistan	U.S.A.
Germany	Poland	

One Member Body opposed the approval of the Draft: U.S.S.R.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in September 1959, to accept it as an ISO RECOMMENDATION.

* These Member Bodies stated that they had no objection to the Draft being approved.

ISO Recommendation | R 132 | September 1959

DETERMINATION OF RESISTANCE TO FLEX CRACKING OF VULCANIZED NATURAL OR SYNTHETIC RUBBER

(DE MATTIA TYPE MACHINE)

FOREWORD

Repeated bending or flexing of a rubber vulcanizate causes cracks to develop in that part of the surface where tension stress is set up during flexing or, if this part of the surface contains a crack, causes this crack to extend in a direction perpendicular to the stress. Certain soft vulcanizates, notably those prepared from styrene butadiene rubber show marked resistance to crack initiation, but it is possible for these sample compounds to have a low resistance to crack growth.

It is important therefore to measure both the resistance to crack initiation by flexing and the resistance to crack growth. A method for determining the resistance to crack growth is given in ISO Recommendation R 133, *Determination of Resistance to Crack Growth of Vulcanized Natural or Synthetic Rubber (De Mattia Type Machine).*

The test described below is intended for use in comparing the resistance of rubbers to the formation and growth of cracks, when subjected to repeated flexing on the De Mattia type machine.

1. APPARATUS

The essential features of the De Mattia type machine are as follows:

There are stationary parts, provided with grips for holding one end of each of the test pieces in a fixed position, and similar but reciprocating parts for holding the other end of each of the test pieces. The travel is 57.15 ± 0.1 mm (2.250 ± 0.005 in) and is such that the maximum distance between each set of opposing grips is $75.0\,^{+1.2}_{-0.0}$ mm ($3.00\,^{+0.00}_{-0.05}$ in) (see Fig. 1).

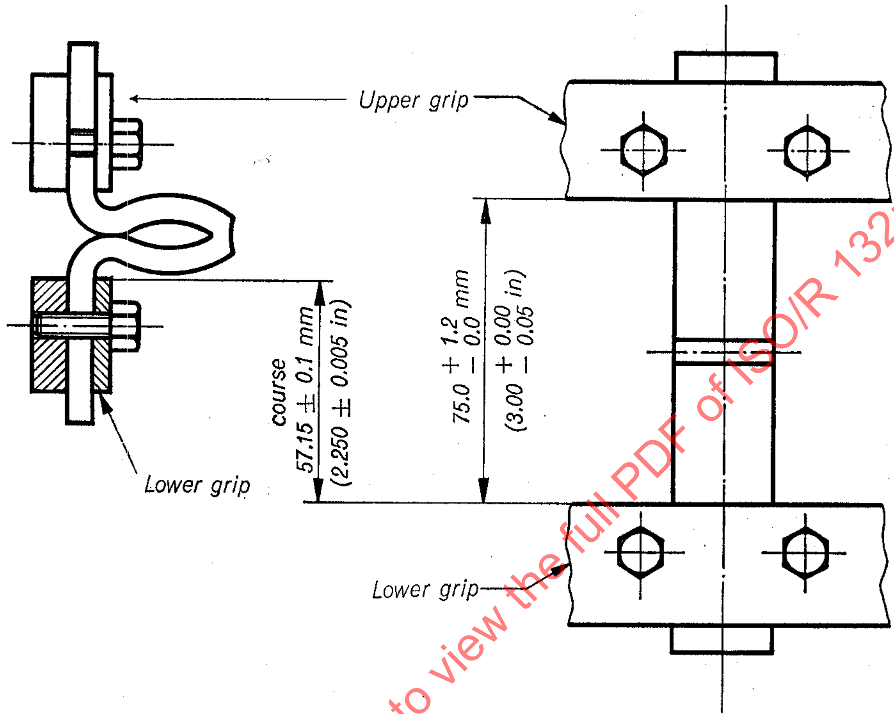

Fig. 1.—**De Mattia type machine**

The reciprocating parts are so arranged that their motion is straight, and in the direction of, and in the same plane as, the common centre-line of each opposing pair of grips. The planes of the gripping surfaces of each opposing pair of grips remain parallel throughout the motion.

The eccentric which actuates the reciprocating parts is driven by a constant-speed motor to give 300 ± 10 flexing cycles per minute, with sufficient power to flex at least six, and preferably twelve, test pieces at one test. The grips hold the test pieces firmly, without undue compression, and enable individual adjustment to be made to the test pieces to ensure accurate insertion.

Note. It is useful to arrange the test pieces in two equal groups, so that one group is being flexed while the other group is being straightened, thus reducing the vibration in the machine.

2. TEST PIECE

The test piece is a strip with a moulded groove, as shown in Figure 2. The strips may be moulded individually in a multiple-cavity mould or may be cut from a wide slab having a moulded groove.

The groove in the test piece should have a smooth surface and be free from irregularities from which cracks may start prematurely. The groove should be moulded into the test piece or slab by a half-round ridge in the centre of the cavity; this half-round ridge should have a radius of 2.38 ± 0.03 mm (0.094 ± 0.001 in).

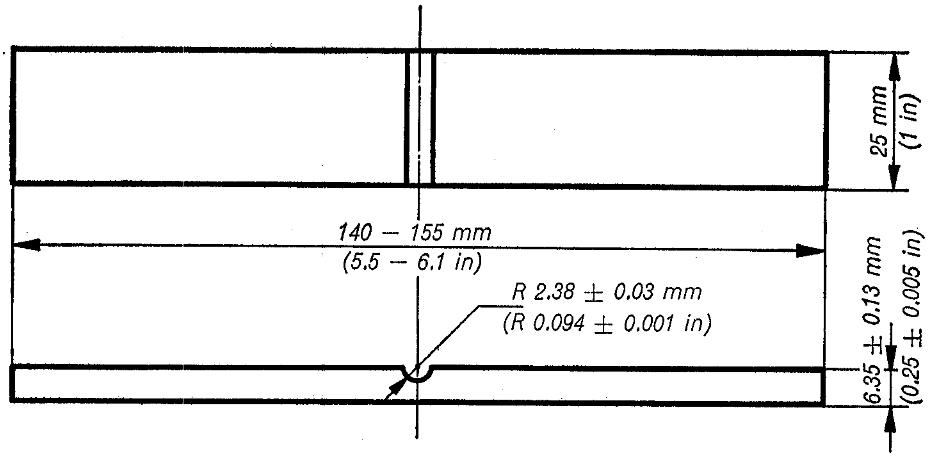

FIG. 2.—Test piece

The results should be compared only between test pieces having thicknesses agreeing within 0.13 mm (0.005 in), when measured close to the groove, because the results of the test are dependent upon the thickness of the test piece.

3. STORAGE OF SAMPLES AND TEST PIECES

The properties of vulcanized rubber change continuously with time, these changes being particularly rapid during the first 24 hours after vulcanization.

Tests should therefore be carried out not less than 24 hours after vulcanization, and for accurate comparisons between different rubbers it may be necessary to ensure that these are tested at substantially the same interval after vulcanization.

Samples and test pieces should be protected from light as completely as possible.

4. NUMBER OF TEST PIECES

At least three, and preferably six, test pieces from each rubber compound are tested, and the results averaged, one or more test pieces being tested simultaneously with those of other rubbers with which the comparison is to be made.

5. PROCEDURE

The pairs of grips are separated to their maximum extent, and the test pieces inserted so that they are flat and not under tension, with the groove in any particular test piece midway between the two grips, in which that test piece is held, and on the outside of the angle made by the test piece, when it is bent.

The machine is started and the test continued with frequent inspection until the first minute sign of cracking is detected; when the number of flexing cycles is recorded, the machine is restarted and stopped after intervals in which the number of flexing cycles is increased in geometric progression, a suitable ratio being 50 per cent on each occasion.

It is not desirable to run the test piece until complete rupture occurs, the preferred method being to grade the severity of cracking by comparison with a standard scale of cracked test pieces, as described in section 7, " Expression of results ". The comparison includes an assessment of the length, depth and number of cracks.

The results should be recorded as follows:

(a) the grade of cracking reached by each test piece on each occasion the machine is stopped;
(b) the flexing cycles which have been run.

6. TEMPERATURE OF TEST

A precise temperature is not specified and tests are normally performed at room temperature, although elevated temperatures may often be used with advantage.

7. EXPRESSION OF RESULTS

The accuracy of the test result can be greatly improved if quantitative values are calculated. The method recommended, based on references 1* and 2**, is to provide the operator with photographs of a series of cracked test pieces and also a description of the effects, as a guide in interpreting the photographs.

A copy of the photographs is provided in Figure 3.

Grade A. A few (less than ten) minute cracks have appeared at scattered points on the surface. A lens is not necessary for examining them, but the unaided eye is unable to detect that they have any depth. They should not be confused with mould-marks or specks of dust on the rubber; the latter should be removed before grading by wiping the test piece with a moistened finger.

Grade B. The number of cracks has increased, but they still appear to have no depth; they tend to concentrate along the centre-line of the groove and extend nearly the full width of the test piece.

Grade C. The cracks begin to show some depth, and their breadth is equal to their length. This grade is regarded as the standard amount of deterioration to which the final result is calculated.

Grade D. The cracks have now become so concentrated along the centre-line that a few have coalesced.

Grade E. Many of the cracks have now coalesced to form about a dozen cracks 1 to 2 mm long with a length/breadth ratio of about 2 or 3. This is the most severe degree of cracking which is regarded as satisfactory for grading purposes.

Although the gradings F to K are much more arbitrary, a brief description follows:

Grade F. Several cracks have coalesced to form one large crack which releases the surface in the centre of the test piece, thus distorting the top and bottom edges of the groove.

Grade G. The large crack has torn its way nearer to the ends of the groove.

Grade H. The crack has grown nearer to the ends and has absorbed a number of small ones, thus making its outline indistinct.

Grade J. The crack has torn nearly to the ends of the groove.

Grade K. The crack has torn right across the groove.

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* Reference 1: R. G. Newton, *Transactions of the Institution of the Rubber Industry*, 1939, **15,** 172 — *Rubber Chemistry and Technology*, 1940, **13,** 59 — *Journal for Rubber Research*, 1947, **25,** 29.

** Reference 2: W. L. Stevens, *India Rubber World*, 1940, **102,** 5, 36 — *Rubber Chemistry and Technology*, 1942, **15,** 159.

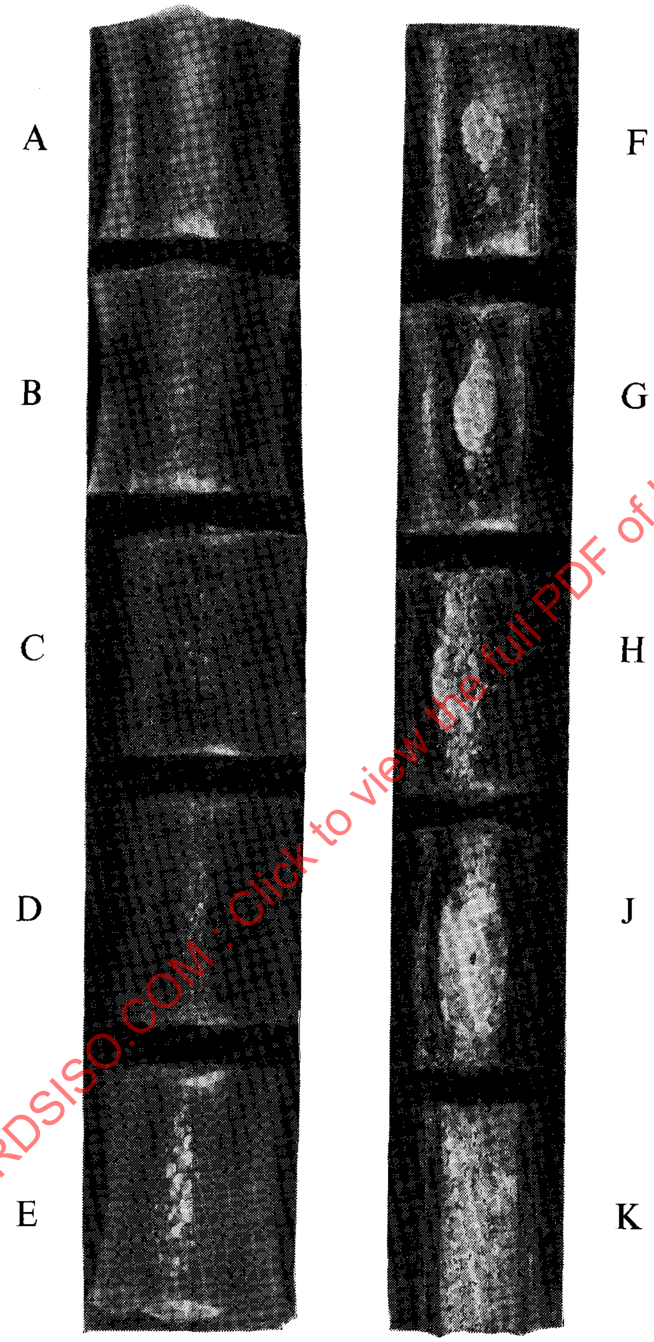

FIG. 3.—Reference standards for grading of cracked test pieces

7.1 *Logarithmic method*

Different observers obtain reproducible results in using these gradings, and numerical constants can be associated with the gradings, representing the proportionate increase in time of running to change from one grade to another. If the number of kilocycles for which the machine has run is expressed as a logarithm to the base 10, these constants can be added or subtracted to change from one grade to another (see references 1* and 2**). Thus several gradings can be obtained on the same test piece for different periods of running; it is convenient to add the constants corresponding to Grades A and B and subtract those for D and E, thus converting all the values to Grade C.

These constants for the gradings are:

A + 0.35
B + 0.15
C 0.00
D − 0.14
E − 0.24

These values were taken from reference 1*, and their use is illustrated by the following examples:

Number of flexing cycles	log number of kilocycles	Grading	Constant	Flex-cracking resistance
13 500	1.13	A	+ 0.35	1.48
22 500	1.35	A	+ 0.35	1.70
31 500	1.50	C	0.00	1.50
40 500	1.61	C	0.00	1.61
49 500	1.70	D	− 0.14	1.56
58 500	1.77	D	− 0.14	1.63
67 500	1.83	D	− 0.14	1.69
85 500	1.93	E	− 0.24	1.69
			Mean =	1.61
			Standard error of mean =	0.03

The mean flex-cracking resistance represents the logarithm of the mean number of kilocycles required to produce Grade C cracking, the eight individual values lying between 1.48 and 1.70, i.e. between 30 000 and 50 000 cycles.

7.2 *Graphical method*

If no assumption is made as to the manner in which cracking progresses, the results may be treated graphically (see reference 3***). Either the experimental results as observed are expressed in kilocycles or the logarithm of the observed results may be used. When results are to be averaged, the arithmetic mean should be taken, it being noted that the arithmetic mean of the logarithm of the experimental results is equivalent to the geometric mean of the experimental results. It is assumed in the following treatment that either the observed experimental results are being used or that all experimental results have been converted into their corresponding logarithms, the logarithms then being treated as " results ".

The method is illustrated in Figure 4, page 10.

7.2.1 Considering the individual results for each test piece obtained in the experiment, the average results corresponding to Grades A, B, C, D and E (call these $\bar{A}$, $\bar{B}$, $\bar{C}$, $\bar{D}$ and $\bar{E}$) are calculated.

* Reference 1: R. G. Newton, *Transactions of the Institution of the Rubber Industry*, 1939, **15**, 172 — *Rubber Chemistry and Technology*, 1940, **13**, 694 — *Journal for Rubber Research*, 1947, **16**, 29.

** Reference 2: W. L. Stevens, *India Rubber World*, 1940, **102**, 36 — *Rubber Chemistry and Technology*, 1942, **15**, 159.

*** Reference 3: J. M. Buist and G. E. Williams, *Transactions of the Institution of the Rubber Industry*, 1951, **27**, 209 — *Rubber Chemistry and Technology*, 1952, **25**, 110.