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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1653

DETERMINATION OF COMPRESSION SET OF VULCANIZED RUBBERS
UNDER CONSTANT DEFLECTION AT LOW TEMPERATURES

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1653, *Determination of compression set of vulcanized rubbers under constant deflection at low temperatures*, 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 led to the adoption of Draft ISO Recommendation No. 1653, which was circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

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

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DETERMINATION OF COMPRESSION SET OF VULCANIZED RUBBERS UNDER CONSTANT DEFLECTION AT LOW TEMPERATURES

1. SCOPE

This ISO Recommendation describes a method for determining the compression set characteristics of vulcanized rubbers which is intended to measure the ability of rubbers to retain their elastic properties at low temperatures.

2. PRINCIPLE

A test piece of known thickness is compressed at room temperature to a defined strain, which is then maintained constant for a specified time at a fixed temperature in the sub-zero region and then released at this test temperature. Measurements of thickness are made at intervals after release of the compression and the compression set characteristics are assessed from a plot of recovery against time.

NOTE. — Two sizes of test pieces are described. These do not necessarily give the same values of compression set, and comparison of values obtained from the two sizes should be avoided. Where possible, the use of the large size of test piece is recommended.

3. APPARATUS

3.1 *Compression apparatus.* The main parts of the compression apparatus are pairs of parallel, flat and highly polished chromium-plated steel or highly polished stainless steel plates between the faces of which the test pieces are compressed. The finish of the surface of the compression plates should be not worse than $0.2 \mu\text{m}$ centre line average. The plates should

- (a) be sufficiently rigid to withstand the stress of the test pieces without bending;
- (b) be of suitable size to ensure that all compressed test pieces will be within the area of the plates;
- (c) remain absolutely parallel when compressing the test pieces.

Steel plates with transverse dimensions of approximately $75 \text{ mm} \times 50 \text{ mm}$ and a thickness of 10 mm are suggested.

The plates should be held together by the quick release holding device described in clause 3.2.

Mild steel spacers, preferably in the form of rings, should be used to provide the specified compression. The spacers should be of such size that contact with the compressed test pieces is avoided. The heights of spacers (t_s) for large and small test pieces should be $9.38 \pm 0.01 \text{ mm}$ and $4.72 \pm 0.01 \text{ mm}$ respectively, so that the applied compression is approximately 25 % of the initial thickness of the test piece. When crystallization studies are being made it is necessary to control the compression strain more accurately. This strain should be as near to 25 % as is practicably possible.

- 3.2 *Handling apparatus.* A quick release device, such as a cam or air-operated vice or pliers, should be provided for holding the plates and test pieces under compression.

A suitable pair of tongs should also be provided.

- 3.3 *Low temperature cabinet.* The cabinet in which the test pieces are exposed may be of the mechanically refrigerated type or may be cooled directly by dry ice or liquid nitrogen. The cabinet should be capable of temperature control within $\pm 1^\circ\text{C}$ of the specified temperature. The test temperature should be measured directly in the plates of the compression apparatus with a precision of $\pm 0.5^\circ\text{C}$.

As all final handling and measurements are to be made within the cabinet, it should be possible to perform these operations while remaining within the permissible temperature variations. This may be done by providing suitable equipment which prevents direct contact of the test chamber with the outside (for example by means of hand holes and gloves through the door or wall of the cabinet).

4. TEST PIECE

4.1 Preparation of test piece

- 4.1.1 *Large type.* A cylindrical disk of diameter 29.0 ± 0.5 mm and thickness 12.5 ± 0.5 mm should be prepared either by moulding or by cutting. Cutting should be done by means of a sharp rotating circular die or revolving knife, lubricated with soapy water, and brought carefully into contact with the rubber, which should preferably be mounted on wood or other suitable backing material, the cutting pressure being kept low enough to avoid "cupping" of the cut surface.

- 4.1.2 *Small type.* A cylindrical disk of diameter 13.0 ± 0.5 mm and thickness 6.3 ± 0.3 mm, should be prepared as described in clause 4.1.1.

4.2 Measurement of thickness of test piece

Thickness of the test piece should be measured by a micrometer dial gauge with two contact members having flat circular surfaces of 9.5 mm diameter.

The gauge should be operated under a dead-weight load of 0.85 ± 0.03 N, load and tolerance also maintaining the same values at low temperatures, and should have a scale graduated in unit divisions of 0.01 mm.

4.3 Number of test pieces

Three pieces should be tested either separately or as a set.

4.4 Conditioning of test pieces

- 4.4.1 The minimum time between vulcanization and testing should be 16 hours.

For non-product tests the maximum time between vulcanization and testing should be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases tests should be made within 2 months of the date of receipt by the customer of the product.

- 4.4.2 Samples and test pieces should be protected from light as completely as possible during the interval between vulcanization and testing.

- 4.4.3 Prepared test pieces should be conditioned immediately before testing for a minimum of 3 hours at a standard laboratory temperature, the same temperature being used throughout any one test or series of tests intended to be comparable.