INTERNATIONAL STANDARD 1653

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Vulcanized rubbers — Determination of compression set under constant deflection at low temperatures

Élastomères vulcanisés — Détermination de la déformation rémanente après compression sous déformation constante à basse température

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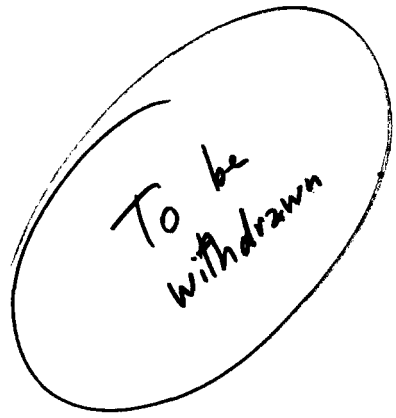

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45 has reviewed ISO Recommendation R 1653 and found it technically suitable for transformation. International Standard ISO 1653 therefore replaces ISO Recommendation R 1653-1971 to which it is technically identical.

ISO Recommendation R 1653 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1653 into an International Standard.

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Vulcanized rubbers — Determination of compression set under constant deflection at low temperatures

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies 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

Compression of a test piece of known thickness 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. Measurement of thickness at intervals after release of the compression. Assessment of the compression set characteristics from a plot of recovery against time.

NOTE — Two sizes of test pieces are given. 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 on the surface of the compression plates shall be not worse than 0,2 μm centre line average. The plate shall

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 mm × 50 mm and a thickness of 10 mm are suggested.

The plates shall be held together by the quick-release holding device described in 3.2.

Mild steel spacers, preferably in the form of rings, shall be used to provide the specified compression. The spacers shall be of such size that contact with the compressed test pieces is avoided. The heights of spacers for large and small test pieces shall be 9,38 ± 0,01 mm and 4,72 ± 0,01 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 shall 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, shall be provided for holding the plates and test pieces under compression.

A suitable pair of tongs shall 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. It shall be possible to control the temperature of the cabinet within ± 1 °C of the specified temperature. The test temperature shall be measured directly in the plates of the compression apparatus with a precision of ± 0,5 °C.

As all final handling and measurements are to be made within the cabinet, it shall 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

4.1.1 *Large type*

A cylindrical disk of diameter 29,0 ± 0,5 mm and thickness 12,5 ± 0,5 mm shall be prepared either by moulding or by cutting. Cutting shall 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 shall be prepared as specified in 4.1.1.

4.2 Measurement of thickness

The thickness of the test piece shall be measured by a micrometer dial-gauge with two contact members having flat circular surfaces of 9,5 mm diameter.

The gauge shall 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 shall have a scale graduated in unit divisions of 0,01 mm.

4.3 Number

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

4.4 Conditioning

4.4.1 The minimum time between vulcanization and testing shall be 16 h.

For non-product tests the maximum time between vulcanization and testing shall 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 shall be made within 2 months of the date of receipt by the customer of the product.

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

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

5 PROCEDURE

5.1 Initial thickness measurements

Measure the initial thickness at the central portion of the test piece to the nearest 0,01 mm at the standard laboratory temperature.

5.2 Strain application

The compression apparatus shall be kept at the standard laboratory temperature. Its operating surfaces shall be carefully cleaned and then, if desired, lubricated. Where a lubricant is applied it shall consist of a thin coating of a fluid having substantially no action on rubber. For most purposes a silicone fluid is suitable.

NOTE – Lubrication of the operating surfaces of the compression apparatus is optional. While giving more reproducible results, lubrication may somewhat alter the compression set values.

Place the test pieces between the pairs of plates together with the requisite spacer(s). Tighten the holding device so that the plates are drawn together parallel until they are in contact with the spacer(s).

5.3 Exposure at low temperature

Approximately 30 min after the compression apparatus has been loaded, place it in the low-temperature cabinet.

All apparatus designed to be used in contact with test pieces shall also be introduced into the cabinet at least 30 min before it is used.

5.4 Final thickness measurements

At the end of the exposure period, release the holding device as quickly as possible and simultaneously start a stop-watch.

Take the thickness readings inside the cabinet at the central portion of the test piece at time intervals, beginning as quickly as possible and ending after 2 h, which make it possible to plot thickness against logarithm of time (10 s; 30 s; 1 min; 3 min; 10 min; 30 min; 2 h are suggested). All handling of test pieces shall be accomplished with the tongs.

Cut the test pieces that have been used for the test in two along a diameter; if any internal defects such as gas bubbles are found, discard the test result.

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

5.5 Exposure time

The exposure time shall be either $24 \, {}^{0}_{-2}$ h or $72 \, {}^{0}_{-2}$ h measured from the moment of placing the compression apparatus in the low-temperature cabinet. Longer times may be used when studying crystallization or plasticizer migration at a specified test temperature.

5.6 Temperature of test

Unless otherwise specified, the test shall be carried out at one of the following temperatures :

0 ± 1 °C

− 10 ± 1 °C

− 25 ± 1 °C

− 40 ± 1 °C

− 55 ± 1 °C

− 75 ± 1 °C

6 ASSESSMENT AND EXPRESSION OF RESULTS

Test results shall be presented for each test piece by plotting on graph paper with semi-logarithmic scales, the thickness being the ordinate, the time being the (logarithmic) abscissa. In the time-range of the recovery process, an approximately straight line results in most cases, which permits the calculation of thickness value after any desired time of recovery by extrapolation (by more than two decades of time) or interpolation.

The compression set, C, expressed as a percentage of the initial deflection, is given by the formula :

$$C = \frac{d_0 - d_2}{d_0 - d_1} \times 100$$

where

d_0 is the initial thickness of the test piece;

d_1 is the height of the spacer;

d_2 is the thickness of the test piece after recovery.

Normally, compression set values are calculated after a recovery period of 10 s (C_{10}) and of 1 800 s ($C_{1\,800}$).

The results for the three test pieces shall agree within 5 % of the mean compression set value; if they do not, the test shall be repeated.

Large fluctuations in test results are to be expected by this method, when measurements are performed in the transition region of the tested materials or in case of low compression set values (for example less than 15 %).

7 TEST REPORT

The test report shall include the following particulars :

a) the mean compression set values (for example C_{10}, $C_{1\,800}$) for the three test pieces after the specified times of recovery;

b) the laboratory temperature;

c) the duration and temperature of test and recovery;

d) the initial dimensions of the test pieces, including the initial thickness d_0;

e) the thicknesses d_2 of the test pieces after specified times of recovery (for example 10 s, 30 min) calculated from the semi-logarithmic curve;

f) whether the test pieces are moulded or cut;

g) the nature of lubricant, if used;

h) whether the test pieces are tested separately or as a set.