METHODS OF TESTING VULCANIZED RUBBER

PART A6. DETERMINATION OF COMPRESSION SET

BS 903 : Part A6 : 1969

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BRITISH STANDARDS INSTITUTION

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This standard makes reference to the following British Standards:

BS 907. Dial gauges for linear measurement.
BS 1134. Centre-line average height method for the assessment of surface texture.

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 references RUC/10 and RUC/10/4
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CO-OPERATING ORGANIZATIONS

The Rubber Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government department and scientific and industrial organizations:

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*Federation of British Rubber and Allied Manufacturers
*Institution of the Rubber Industry
*Ministry of Technology
Natural Rubber Bureau
*Natural Rubber Producers' Research Association
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BRITISH STANDARD METHODS OF

TESTING VULCANIZED RUBBER

Part A6. Determination of compression set

FOREWORD

This standard was originally published in 1940 and has since been issued twice in revised form. A further revision has become necessary as progress has been made towards the adoption of the basic method by the International Organization for Standardization (ISO) with modified sizes of test piece. An important change is the optional use of a lubricant on the plates. Lubrication gives more reproducible results but may somewhat alter the compression set value.

METHODS

1. SUMMARY AND EXPLANATORY NOTE

These methods cover test procedures for determining the compression set characteristics of vulcanized rubbers of hardness between 30 and 94 IRHD; such tests are intended to measure the ability of rubbers to retain elastic properties after prolonged compression. Two methods are described:

Method A. Compression set after constant strain.
Method B. Compression set after constant stress.

The appropriate method to be used in a particular case will depend on the service conditions. Method A maintains a definite deformation and gives a measure of the amount of such deformation that is retained by the rubber, while Method B applies a constant force and shows the residual deformation produced in the rubber after such an application. Method A is preferred because of simpler and less bulky apparatus and because it is the method being considered by the International Organization for Standardization (ISO).

The effect of rapidly repeated deformation and recovery resulting from intermittent compressive forces is not covered by these methods.

2. DEFINITIONS

For the purposes of this British Standard the following definitions apply:

Compression stress. Stress applied so as to cause shortening or contraction of the test piece in the direction of the stress.

Compression strain. The proportionate contraction produced in the test piece by a compression stress.

Compression set. The proportionate residual contraction in the test piece after it has been released from compression.

3. METHOD A: CONSTANT STRAIN (25%)

3.1 Apparatus

3.1.1 *Dies and knives.* Dies and knives used for cutting the test pieces shall be carefully maintained so that the cutting edges are sharp and free from nicks to avoid leaving ragged edges on the test piece. Details of a suitable form of cutter are shown in Fig. 1.

3.1.2 *Thickness gauge.* The instrument for measuring the thickness of compression set test pieces shall consist of a micrometer dial gauge, the two contact members of which shall be either a raised platform of 9·5 mm diameter and a spherical contact member of 6·35 mm diameter or preferably and for referee purposes contact members having spherical surfaces of 12·5 mm radius formed on rods of about 10 mm diameter. The gauge shall have a scale graduated in unit divisions of 0·01 mm, and shall comply where relevant with the requirements of BS 907* for a Type A gauge, particularly in respect of the accuracy of calibration. The dial gauge shall operate under a force of 235 ± 30 mN.

NOTE. The two types of contact members may give different results owing to concavity of the test piece surfaces after compression and recovery.

For the most accurate results, the instrument shall be used as a comparator as recommended in BS 907*, which also includes notes on the care and use of dial gauges.

3.1.3 *Compression apparatus.* The compression apparatus shall consist of two or more parallel, smooth, flat highly polished chromium plated or highly polished stainless steel plates between the faces of which the test pieces are compressed; these plates shall be sufficiently rigid to withstand the stress without bending, and of sufficient size to ensure that all the compressed test pieces are within the area of the plates. The surface finish of the working surfaces shall be not worse than 8 CLA surface roughness in accordance with BS 1134†.

The plates shall be held together by a bolt or bolts of suitable size, provided with steel spacers, preferably in the form of rings of such size as to avoid contact with the compressed test piece, and of thickness to give the specified compression. The thickness of these spacers shall be 4·73 ± 0·01 mm for Type 1 test pieces and 9·38 ± 0·01 mm for Type 2 test pieces‡.

When lubrication is to be used, the contact surfaces of the plates shall be

* BS 907, ' Dial gauges for linear measurement '.
† BS 1134, ' Centre-line average height method for the assessment of surface texture '.
‡ For test pieces nominally 6·0 and 13·0 mm thick (see Note after 3.2.2) the thickness shall be 4·50 mm and 9·75 mm respectively.

lubricated with a thin coating of a lubricant having substantially no action on the rubber; for most purposes a fluorosilicone fluid of viscosity about 10 000 cS is suitable.

Suitable apparatus is shown in Figs. 2 and 3.

3.1.4 *Oven.* Any well designed, uniformly heated air oven, capable of maintaining the compression apparatus and test pieces within the tolerance given in 3.3.4.

3.2 Test piece. The test piece shall be of one of the two types described below. Where possible the use of Type 2 is recommended for maximum accuracy. The two types of test piece do not necessarily give the same values of compression set, and comparison of values obtained from the two types should be avoided.

3.2.1 *Type* 1. A cylindrical disk 13·0 ± 0·5 mm diameter and 6·3 ± 0·3 mm thickness prepared either by moulding or by cutting. Cutting shall be done by means of a rotating circular die or a revolving knife, lubricated with soapy water and brought carefully into contact with the rubber, which is preferably rigidly mounted on wood or other suitable backing material, the cutting pressure being kept light enough to avoid ' cupping ' of the cut surface.

NOTE. It has been found with silicone rubbers that different results may be obtained depending on whether the test pieces are moulded or cut, and with the latter, whether they are cut before or after post-curing.

3.2.2 *Type* 2. A cylindrical disk 29·0 ± 0·5 mm diameter and 12·5 ± 0·5 mm thickness prepared as for Type 1.

Laminated test pieces conforming to 3.2.1 or 3.2.2 but made up of 2 to 4 (for Type 1) or 2 to 7 (for Type 2) superimposed disks cut from sheets, may be used; these do not necessarily give the same values of compression set as test pieces of the same dimensions formed of one disk.

NOTE. As a temporary measure it is permissible, except for referee purposes, to use test pieces of the thickness previously specified in this standard, i.e. 6·0 ± 0·2 mm for Type 1 and 13·0 ± 0·5 mm for Type 2, since these give approximately the same diameter/thickness ratios (2·17 ± 0·16 and 2·23 ± 0·12 respectively) as those now specified (2·07 ± 0·18 and 2·32 ± 0·14) and hence give substantially the same results. All new moulds should, however, be made to give test pieces of the thickness specified in 3.2.1 and 3.2.2.

3.3 Procedure

3.3.1 *Preparation of samples and test pieces.* If fabric is attached to or embedded in the sample, it shall be removed before cutting the test pieces. The method of removal shall preferably avoid the use of a swelling liquid, but benzene, chloroform or carbon tetrachloride may be used to wet the contacting surfaces if necessary. Care shall be taken to avoid stretching the rubber during the separation from the fabric, and swelling liquid, if used, shall be allowed to evaporate completely from the rubber surfaces after separation.

Cloth-marked surfaces shall be made smooth by buffing, and a sample of

uneven thickness or of thickness above the maximum specified for the test pieces which are to be cut from it, shall also be buffed if necessary.

Moulded test pieces shall be cleaned free of any mould lubricant or dusting powder.

3.3.2 *Conditioning of samples and test pieces.* The properties of vulcanized rubber change continuously with time, these changes being particularly rapid in the period immediately following vulcanization. Therefore no tests should be made less than 16 hours after vulcanization. For non-product tests the maximum interval between vulcanization and testing should be four weeks, and for evaluations intended to be comparable the tests should, as far as possible, be made at the same interval. For products the maximum interval should be three months where the date of vulcanization is known; if this is not known, it should be six weeks from the date of receipt by the customer, unless otherwise specified.

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

Test pieces shall be conditioned at 20 ± 2 °C for at least three hours immediately before being measured and tested. If the preparation of the test piece involves buffing, the interval between buffing and testing shall not exceed 72 hours.

3.3.3 *Measurement of test pieces.* The thickness of each test piece shall be measured at the centre with the gauge specified in 3.1.2.

3.3.4 *Determination of compression set.* Three Type 1 test pieces shall be placed symmetrically (see Fig. 2), or one Type 2 test piece exactly in the centre, (see Fig. 3), together with the requisite spacer(s) between each pair of plates at 20 ± 2 °C, allowing sufficient clearance from the spacers for the bulging of the rubber when compressed; in either case, three test pieces shall be used for each determination. The bolt or bolts shall be tightened so that the plates are drawn together uniformly until they are in contact with the appropriate spacers. The compression apparatus containing the test pieces is introduced without delay into the central part of an oven which is operating at the test temperature.

The temperature during the compression period shall normally be 150 ± 2 °C, 100 ± 1 °C, 70 ± 1 °C or 20 ± 2 °C.

The duration of the compression period shall be 24 (+0−2) hours for tests at elevated temperatures and 72 (+0−2) hours for tests at 20 °C.

At the end of the compression period the bolts of the compression apparatus shall be loosened and the test pieces removed. They shall be quickly transferred to a wooden bench where they are allowed to recover for a period of 30 ± 3 minutes at a temperature of 20 ± 2 °C. The thickness of the test pieces shall then again be measured as in 3.3.3.

The test pieces shall then be cut in two along a diameter; if any internal defects such as air bubbles are found, the test result shall be discarded.

3.3.5 *Calculation of results.* The compression set at constant strain is the difference between the original thickness of the test piece and that after recovery expressed as a percentage of the initially applied compression.

Compression set at constant strain

$$= \frac{t_o - t_r}{t_o - t_s} \times 100$$

where t_o = original thickness of test piece,
t_r = thickness of test piece after recovery,
t_s = thickness of the spacer.

The results from the three test pieces shall be averaged.

3.4 Report. The report shall state:

(1) The compression set calculated as in 3.3.5.
(2) The type of test piece used, whether moulded or cut, and if the latter, whether formed of one or more (and if so, how many) disks.
(3) The original thickness of the test piece (t_o) and its thickness after recovery (t_r).
(4) The duration and temperature of test.
(5) Whether or not the operating surfaces of the plates are lubricated, and if so, the lubricant used.
(6) Type of thickness gauge.

4. METHOD B: CONSTANT STRESS

4.1 Apparatus

4.1.1 *Dies and knives.* Dies and knives used for cutting the test pieces shall be carefully maintained so that the cutting edges are sharp and free from nicks to avoid leaving ragged edges on the test piece. Details of a suitable form of cutter are shown in Fig. 1.

4.1.2 *Thickness gauge.* The instrument for measuring the thickness of compression set test pieces shall consist of a micrometer dial gauge, the two contact members of which shall be either a raised platform of 9·5 mm diameter and a spherical contact member of 6·35 mm diameter or preferably and for referee purposes contact members having spherical surfaces of 12·5 mm radius formed on rods of approximately 10 mm diameter. The gauge shall have a scale graduated in unit divisions of 0·01 mm, and shall comply where relevant with the requirements of BS 907* for a Type A gauge, particularly in respect of the accuracy of calibration. The dial gauge shall operate under a force of 235 ± 30 mN.

NOTE. *The two types of contact members may give different results owing to concavity of the test piece surfaces after compression and recovery.*

* BS 907, ' Dial gauges for linear measurement '.

For the most accurate results, the instrument shall be used as a comparator as recommended in BS 907*, which also includes notes on the care and use of dial gauges.

4.1.3 *Compression apparatus.* The compression apparatus shall consist of two or more parallel, smooth, flat, highly polished chromium plated or highly polished stainless steel plates between the faces of which the test pieces are compressed; these plates shall be sufficiently rigid to withstand the stress without bending, and of sufficient size to ensure that all the compressed test pieces are within the area of the plates. The surface finish of the working surfaces shall be not worse than 8 CLA surface roughness in accordance with BS 1134†.

The plates, together with a calibrated loading spring, shall be mounted in a suitable housing having a screw mechanism for compressing the spring to apply the force to the plates, and equipped with one or more (preferably three) scales graduated to read to the nearest 0·25 mm for measuring the deflection of the spring. The device shall be capable of applying the desired force gradually and maintaining it uniformly over the entire top and bottom surfaces of the test pieces for the duration of the test.

The loading spring shall be made of properly heat-treated spring steel with ends ground flat and perpendicular to the longitudinal axis of the spring (to help in keeping the plates parallel throughout the test), and shall comply with the following requirements:

(1) The spring shall be calibrated at 20 ± 5 °C by applying successive increments of force of 225 ± 25 N and measuring the corresponding deflection to the nearest 0·25 mm. The force-deflection curve shall have a slope of 70 ± 9 N/mm at all points over the required range.

(2) The deflection under a given force shall not change, as a result of fatigue, by more than 0·25 mm over a period of a year, as indicated by weekly check tests.

(3) The force required to close the spring solid shall be not less than 9 000 N.

When lubrication is to be used, the contact surfaces of the plates shall be lubricated with a thin coating of a lubricant having substantially no action on the rubber; for most purposes a fluorosilicone fluid of viscosity about 10 000 cS is suitable.

A suitable apparatus is shown in Fig. 4.

4.1.4 *Oven.* Any well designed, uniformly heated air oven, capable of maintaining the compression apparatus and test pieces within the tolerance given in 4.3.4.

4.2 Test piece. The test piece shall be of one of the two types described below. Where possible the use of Type 2 is recommended for maximum accuracy. The two types of test piece do not necessarily give the same values of compression set, and comparison of values obtained from the types should be avoided.

* BS 907, 'Dial gauges for linear measurement'.
† BS 1134, 'Centre-line average height method for the assessment of surface texture'.

4.2.1 *Type* 1. A cylindrical disk 13·0 ± 0·5 mm diameter and 6·3 ± 0·3 mm thickness prepared either by moulding or by cutting. Cutting shall be done by means of a rotating circular die or a revolving knife, lubricated with soapy water and brought carefully into contact with the rubber, which is preferably rigidly mounted on wood or other suitable backing material, the cutting pressure being kept light enough to avoid ' cupping ' of the cut surface.

NOTE. It has been found with silicone rubbers that different results may be obtained according as the test pieces are moulded or cut, and with the latter, according as they are cut before or after post-curing.

4.2.2 *Type* 2. A cylindrical disk 29·0 ± 0·5 mm diameter and 12·5 ± 0·5 mm thickness prepared as for Type 1.

Laminated test pieces conforming to 4.2.1 or 4.2.2 but made up of 2 to 3 (for Type 1) or 2 to 7 (for Type 2) superimposed disks cut from sheets, may be used; these do not necessarily give the same values of compression set as test pieces of the same dimensions formed of one disk.

NOTE. As a temporary measure it is permissible, except for referee purposes, to use test pieces of the thickness previously specified in this standard, i.e. 6·0 ± 0·2 mm for Type 1 and 13·0 ± 0·5 mm for Type 2, since these give approximately the same diameter/thickness ratios (2·17 ± 0·16 and 2·23 ± 0·12 respectively) as those now specified (2·07 ± 0·18 and 2·32 ± 0·14) and hence give substantially the same results. All new moulds should, however, be made to give test pieces of the thickness specified in 4.2.1 and 4.2.2.

4.3 Procedure

4.3.1 *Preparation of samples and test pieces.* If fabric is attached to or embedded in the sample, it shall be removed before cutting the test pieces. The method of removal shall preferably avoid the use of a swelling liquid, but benzene, chloroform or carbon tetrachloride may be used to wet the contact surfaces if necessary. Care shall be taken to avoid stretching the rubber during the separation from the fabric, and swelling liquid, if used, shall be allowed to evaporate completely from the rubber surfaces after separation.

Cloth-marked surfaces shall be made smooth by buffing, and a sample of uneven thickness or of thickness above the maximum specified for the test pieces which are to be cut from it, shall also be buffed if necessary.

Moulded test pieces shall be cleaned free of any mould lubricant or dusting powder.

4.3.2 *Conditioning of samples and test pieces.* The properties of vulcanized rubber change continuously with time, these changes being particularly rapid in the period immediately following vulcanization. Therefore no tests should be made less than 16 hours after vulcanization. For non-product tests the maximum interval between vulcanization and testing should be four weeks, and for evaluations intended to be comparable the tests should, as far as possible, be made at the same interval. For products the maximum interval should be three months

where the date of vulcanization is known; if this is not known, it should be six weeks from the date of receipt by the customer, unless otherwise specified.

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

Test pieces shall be conditioned at 20 ± 2 °C for at least three hours immediately before being measured and tested. If the preparation of the test piece involves buffing, the interval between buffing and testing shall not exceed 72 hours.

4.3.3 *Measurement of test pieces.* The thickness of each test piece shall be measured at the centre with the gauge specified in 4.1.2.

4.3.4 *Determination of compression set.* Three Type 1 test pieces shall be placed exactly symmetrically (see Fig. 5), or one Type 2 test piece exactly in the centre, between each pair of plates at 20 ± 2 °C; in either case, three test pieces shall be used for each determination. The spring shall be compressed until the required force has been applied; the force to be used will depend on the temperature of test and the type of test piece as specified in Table 1.

TABLE 1. FORCES

Temperature of test	Force for test piece	
	Type 1	Type 2
	N	N
70 °C	1100	1825
20 °C	4400	7300

The temperature during the compression period shall normally be 70 ± 1 °C or 20 ± 2 °C.

The duration of the compression period shall be 24 (+0−2) hours for tests at 70 °C and 72 (+0−2) hours for tests at 20 °C.

At the end of the compression period, the spring shall be released and the test pieces removed. They shall be quickly transferred to a wooden bench for a period of 30 ± 3 minutes at a temperature of 20 ± 2 °C. The thickness of the test pieces shall then again be measured as in 4.3.3.

The test pieces shall then be cut in two along a diameter; if any internal defects such as air bubbles are found, the test result shall be discarded.

4.3.5 *Calculation of results.* The compression set at constant stress is the difference between the original thickness of the test piece and that after recovery expressed as a percentage of the initial thickness.

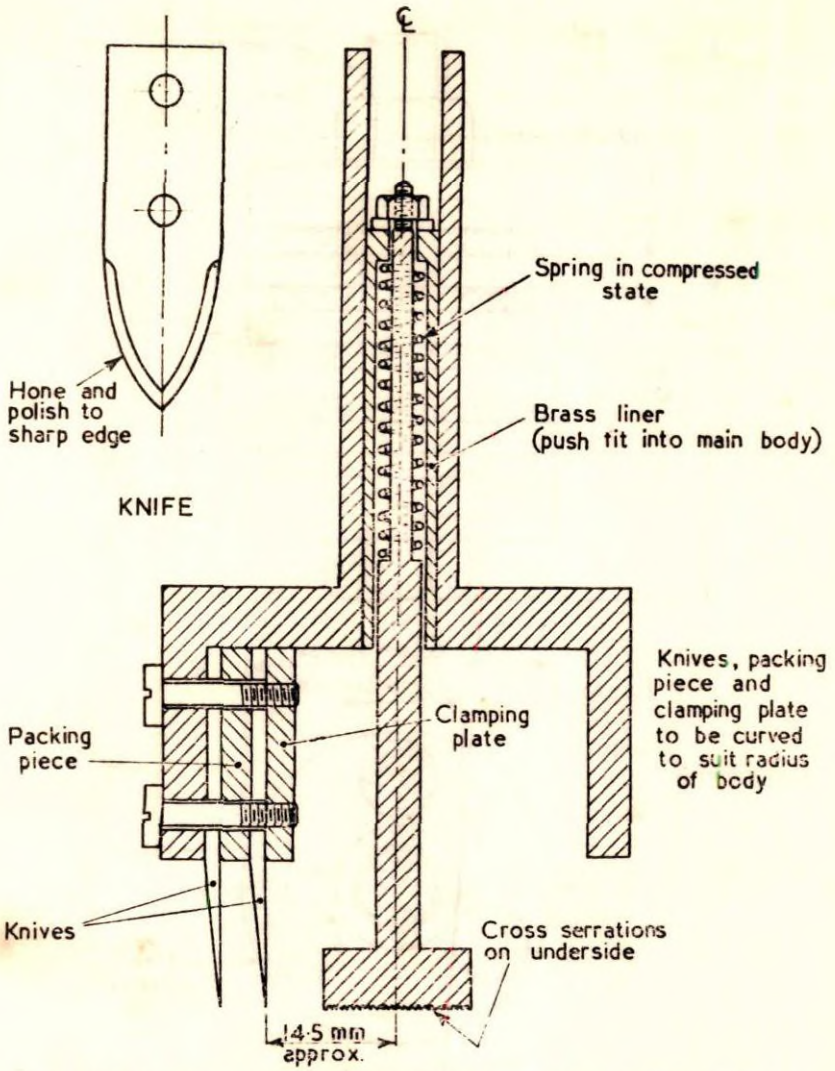
Compression set at constant stress

$$= \frac{(t_o - t_r) \times 100}{t_o}$$

where t_o = original thickness of test piece,
t_r = thickness of test piece after recovery.

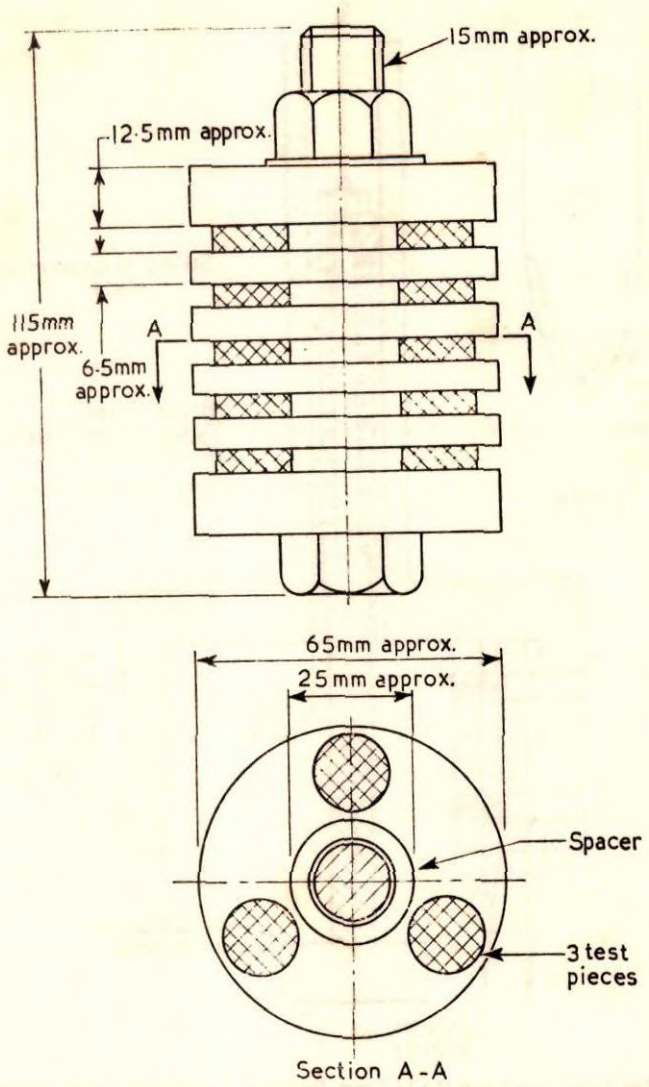
4.4 Report. The report shall state:

(1) The compression set calculated as in 4.3.5.
(2) The type of test piece used, whether moulded or cut, and if the latter, whether formed of one or more (and if so, how many) disks.
(3) The original thickness of the test piece (t_o) and its thickness after recovery (t_r).
(4) The duration and temperature of test.
(5) Whether or not the operating surfaces of the plates are lubricated, and if so, the lubricant used.
(6) Type of thickness gauge.



NOTE 1. For Type 1 test pieces the 14·5 mm dimension must be reduced to 6·5 mm.
NOTE 2. The outer blade minimizes the outward displacement of the rubber due to the force exerted by the inner blade, and thus helps to ensure that the cut made by the latter is vertical.

Fig. 1. Cutter for test piece Type 2



The top and bottom plates shall be keyed to the centre bolt

Fig. 2. Apparatus for compression set, Method A, using Type 1 test piece

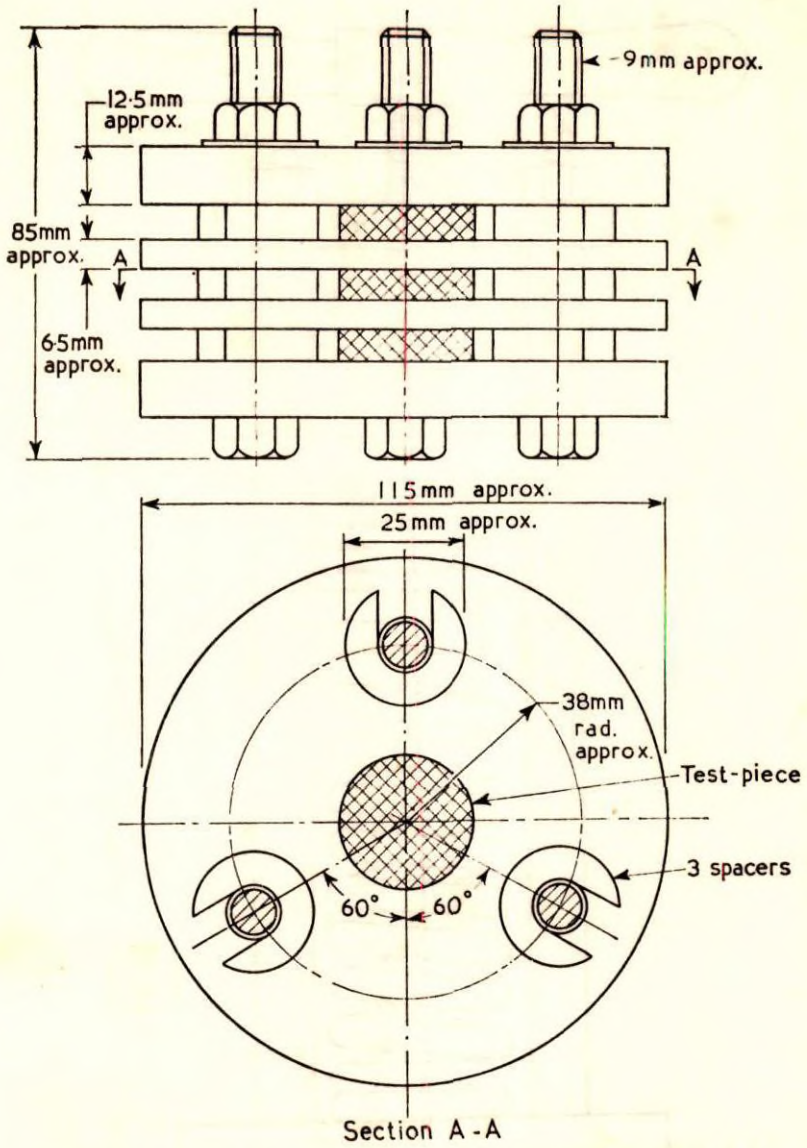


Fig. 3. Apparatus for compression set, Method A, using Type 2 test piece

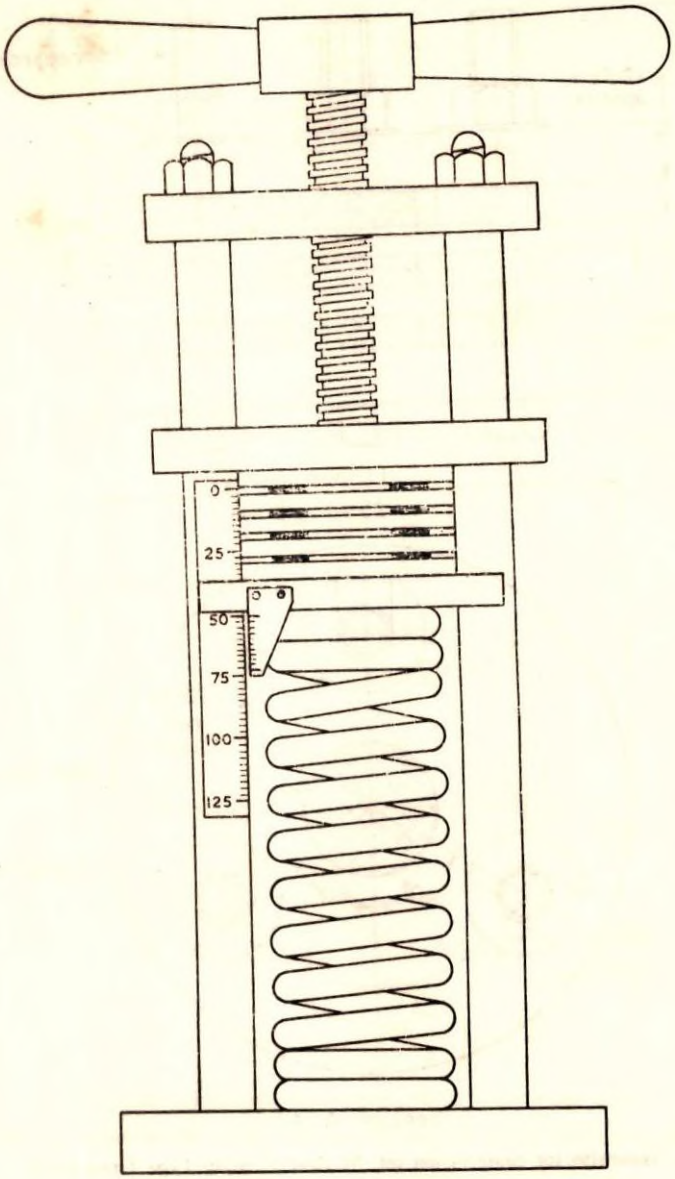


Fig. 4. Apparatus for compression set, Method B

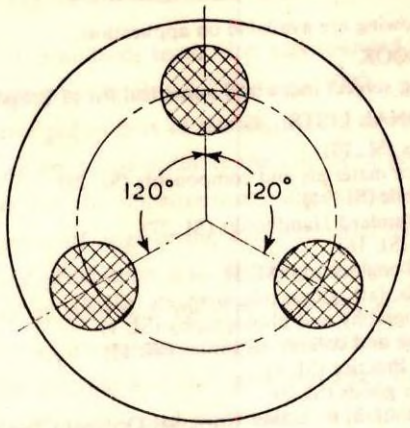


Fig. 5. Plan view of test pieces in position in apparatus

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