INTERNATIONAL
STANDARD

ISO
2781

Fifth edition
2018-06

Rubber, vulcanized or thermoplastic — Determination of density

Caoutchouc vulcanisé ou thermoplastique — Détermination de la masse volumique

Reference number
ISO 2781:2018(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 2781:2008), which has been technically revised. It also incorporates the Amendment ISO 2781:2008/Amd.1:2010.

The main changes compared to the previous edition are as follows:

- precision data have been incorporated in Annex A;
- a calibration schedule has been added in Annex B.

Rubber, vulcanized or thermoplastic — Determination of density

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine applicability of any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies two methods of test for the determination of the density of solid vulcanized and thermoplastic rubbers.

Such determinations are of importance in the control of the quality of a rubber compound and in the calculation of the mass of rubber required to produce a given volume of material.

This document does not cover the determination of the relative density of rubber, which is the ratio of the mass of a given volume of rubber to the mass of an equal volume of pure water at a given temperature.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
density
mass of unit volume of the rubber at a stated temperature

Note 1 to entry: It is expressed in megagrams per cubic metre (Mg/m^3).

4 Principle

Two methods, A and B, are given.

In method A, the mass of the test piece and the mass of water equal in volume to the volume of the test piece are determined using a balance equipped with a pan straddle. The apparent mass of the test piece when immersed in water is less than that in air by the mass of water displaced, the volume of water displaced being equal to that of the test piece.

Method B is intended to be used only when it is necessary to cut up the test piece into small pieces to eliminate air spaces, as in the case of narrow-bore tubing and electric-cable insulation. Measurements are made using a balance and a density bottle.

5 Test apparatus

Ordinary laboratory apparatus and the following.

5.1 Analytical balance, accurate to ±1 mg.

5.2 Balance pan straddle, of convenient size to support the beaker and permit determination of the mass of the test piece in water (for method A).

5.3 Beaker, 250 cm^3 capacity (or smaller if necessitated by the design of the balance) (for method A).

5.4 Density bottle (for method B).

6 Test piece

6.1 The test piece shall consist of a piece of the rubber with smooth surfaces, free from cracks and dust, and having a mass of at least 2,5 g. For method B, the shape of the test piece shall be such as to permit cutting into suitable pieces (see 11.3).

6.2 A minimum of two test pieces shall be made.

7 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex B.

8 Time-interval between forming and testing

8.1 Unless otherwise specified for technical reasons, the following requirements for time-intervals shall be observed.

8.2 For all test purposes, the minimum time between forming and testing shall be 16 h.

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

8.4 For product tests, whenever possible, the time between forming and testing shall 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.

9 Conditioning of test pieces

9.1 Samples and test pieces shall be protected from direct sunlight during the interval between vulcanization and testing.

9.2 Samples, after such preparation as is necessary, shall be conditioned at a standard laboratory temperature (that is, 23 °C ± 2 °C or 27 °C ± 2 °C) for at least 3 h before the test pieces are cut from them.

These test pieces may be tested immediately but, if not, they shall be kept at the standard laboratory temperature until tested. If the preparation involves buffing, the interval between buffing and testing shall not exceed 72 h.

10 Temperature of test

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

11 Procedure

11.1 Preparation of sample

If fabric is attached to, or embedded in, the sample, it shall be removed before cutting out the test pieces. The method of removal shall preferably avoid the use of a swelling liquid, but, if necessary, a suitable non-toxic liquid of low boiling point may be used to wet the contacting surfaces. Care shall be taken to avoid stretching the rubber during the separation from the fabric, and the liquid, if used, shall be allowed to evaporate completely from the rubber surfaces after separation. Cloth-marked surfaces shall be made smooth by buffing in accordance with ISO 23529.

11.2 Method A

11.2.1 Suspend the test piece (see Clause 6) from the hook on the balance (5.1) using a suitable length of filament so that the bottom of the test piece is about 25 mm above the straddle (5.2). The filament shall be made from a material which is insoluble in water and which does not absorb a significant amount of water. It shall either be counterbalanced or be weighed and, if weighed, its mass shall be deducted from subsequent weighings of the test piece (see 11.2.3).

11.2.2 Weigh the test piece to the nearest milligram in air. Repeat the weighing with the test piece (and sinker, if required, see 11.2.4) immersed in freshly boiled and cooled distilled water or deionized water at a standard laboratory temperature (23 °C ± 2 °C or 27 °C ± 2 °C) contained in the beaker (5.3), placed on the straddle. Remove air bubbles adhering to the test piece (see 11.2.5 and 11.2.6) and weigh to the nearest milligram, watching for a few seconds to make sure that the reading does not drift gradually as a result of convection currents.

11.2.3 Where the filament used has a mass less than 0,010 g, such as is the case with thin nylon filament, the correction to account for its mass is not necessary to ensure the stated accuracy of the final result. However, when the test piece is smaller than the one specified (e.g. when the density of small O-rings is to be measured), this can lead to inaccuracies and the mass of the filament needs to be taken into account in the final calculation. If a means of suspension other than a filament is used, the volume and mass of the suspension shall be taken into account in making the final calculation.

11.2.4 When this procedure is used for rubber having a density less than 1 Mg/m^3, a sinker is necessary; a further weighing of the sinker alone in water is required. Alternatively, a liquid of different density to water, which does not have any interaction with the rubber, may be used in place of the water, in which case Formula (1) and Formula (2) shall be modified to replace the density of water with the density of the liquid used.

11.2.5 The main sources of error are

a) air bubbles adhering to the surfaces of the test piece during weighing in water;

b) surface tension effects on the filament;

c) convection currents in the water in which the test piece is suspended, to minimize which the temperature of the water and of the air in the balance case shall be the same.

11.2.6 In order to minimize the adherence of air bubbles to the test piece, it is permissible either to add a trace (say 1 part in 10 000) of surface-active material such as a detergent to the distilled water or to dip the test piece momentarily into a suitable liquid, such as methyl alcohol or denatured spirit, miscible with water and having a negligible swelling or leaching action on rubber. If the latter method is adopted, precautions shall be taken to minimize the carry-over of alcohol.

11.3 Method B

11.3.1 Weigh the clean, dry density bottle and its stopper (5.4) before and after the insertion of the test piece (see Clause 6) cut into suitable pieces. The exact size and shape of the pieces will depend on the thickness of the original test piece. They should be such that no two dimensions are greater than 4 mm and the third not greater than 6 mm. Within these limitations, the pieces should be as large as possible. All cut edges shall be smooth. Fill the bottle, containing the rubber, completely with freshly boiled and cooled distilled water or deionized water at a standard laboratory temperature (23 °C ± 2 °C or 27 °C ± 2 °C). Remove air bubbles adhering to the rubber or to the walls of the bottle (see 11.2.6 and 11.3.2). Insert the stopper, taking care that there is no air in the bottle or the capillary. Dry the outside of the bottle carefully. Weigh the bottle and contents. Empty the bottle completely and refill with freshly boiled and cooled distilled water or deionized water. After removing the air bubbles, inserting the stopper and drying, weigh the bottle and water. All the above weighings shall be made to the nearest milligram.

11.3.2 The main source of error is air bubbles inside the bottle. It might be necessary to heat the bottle and contents to approximately 50 °C to dislodge bubbles, but in this case the bottle and contents shall be cooled before weighing. Alternatively, the bottle may be placed in a vacuum desiccator and the vacuum applied and released several times until no more air is extracted.

12 Expression of results

12.1 Method A

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

The density, ρ, expressed in megagrams per cubic metre, is given by Formula (1):

$$\rho = \rho_w \frac{m_1}{m_1 - m_2} \tag{1}$$

where

ρ_w is the density of water;

m_1 is the mass of the rubber, determined by weighing in air;

m_2 is the mass of the rubber less the mass of an equal volume of water, determined by weighing in water, both at standard laboratory temperature.

This method is accurate to the nearest 0,01 Mg/m^3.

For most purposes, the density of water at standard laboratory temperature may be taken as 1,00 Mg/m^3. However, for precise work, a factor to take account of the density of water at the test temperature shall be used.

When a sinker has been used, the calculation shall be modified as given by Formula (2):

$$\rho = \rho_w \frac{m_1}{m_1 + m_2 - m_3} \qquad (2)$$

where

ρ_w is the density of water;

m_1 is the mass of the rubber, determined by weighing in air;

m_2 is the mass of the sinker less the mass of an equal volume of water, determined by weighing in water, both at standard laboratory temperature;

m_3 is the mass of the sinker and rubber less the mass of a volume of water equal to their combined volumes, determined by weighing in water, both at standard laboratory temperature.

Report the mean value.

12.2 Method B

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

The density, ρ, expressed in megagrams per cubic metre, is given by Formula (3):

$$\rho = \rho_w \frac{m_2 - m_1}{m_4 - m_3 + m_2 - m_1} \qquad (3)$$

where

ρ_w is the density of water;

m_1 is the mass of the density bottle;

m_2 is the mass of the density bottle plus test piece;

m_3 is the mass of the bottle plus test piece plus water;

m_4 is the mass of the density bottle filled with water.

For most purposes, the density of water at standard laboratory temperature may be taken as 1,00 Mg/m^3. However, for precise work, a factor to take account of the density of water at the test temperature shall be used.

Report the mean value.

13 Precision

See Annex A.

14 Test report

The test report shall include the following particulars:

a) sample details:
   1) a full description of the sample and its origin,
   2) compound details and cure details, where appropriate,
   3) the method of preparation of the test pieces from the sample, for example moulded or cut;

b) test method:
   1) a full reference to the test method used, i.e. the number of this document (ISO 2781),
   2) the test procedure used (method A or method B),
   3) the type of test piece used;

c) test details:
   1) the standard laboratory temperature used,
   2) the time and temperature of conditioning prior to the test,
   3) the temperature of the test,
   4) details of any procedures not specified in this document;

d) test results:
   1) the number of test pieces used,
   2) the individual test results,
   3) the mean density;

e) the date of the test.

Annex A
(informative)

Precision

A.1 Background

An interlaboratory test programme (ITP) for the determination of the precision of the density measurement method specified in this document was conducted in 2008 using the precision procedures and guidelines described in ISO/TR 9272.

A.2 Details of ITP

A.2.1 A total of 15 laboratories participated in the ITP. However, test results were reported by only 13 of the laboratories. A total of four materials or compounds were used, designated compounds A, B, C and D in increasing magnitude of density from 0,97 to 1,86. The ITP was conducted over a period of four sequential weeks. On a specified day in each of these four weeks, five individual density measurements were made by each laboratory on each of the four materials. A test result was taken as the mean of the five individual measurements. The analysis of the data was conducted on the basis of these test results.

A.2.2 Two separate determinations of repeatability and reproducibility were conducted. The results from weeks 1 and 2 were used for repeatability and reproducibility evaluation No. 1 (i.e. these results were obtained by the typical day 1 vs day 2 test protocol specified in ISO/TR 9272). The results of weeks 3 and 4 were used in the same way for repeatability and reproducibility evaluation No. 2. The ISO/TR 9272:2005 option 1 outlier deletion procedure was used.

A.2.3 The precision results as determined by this ITP may not be applied to acceptance or rejection testing of any group of materials or products without documentation stating that the results of this precision evaluation actually apply to the products or materials tested.

A.3 Precision results

A.3.1 The precision results are given in Table A.1. General statements for the use of the precision results are also given. The results listed in Table A.1 are the mean results for evaluations 1 and 2 as indicated above (i.e. for all four weeks). These are given in terms of both the absolute precision, r and R, and the relative precision, (r) and (R).

A.3.2 *Repeatability*: The repeatability, or local domain precision, of the test method has been established for each material as the values given in Table A.1. Two individual test results (obtained by the proper use of this document) that differ by more than the tabulated values of r, in measurement units, or (r), in percent, should be considered suspect, i.e. to have come from different populations. Such a decision suggests that appropriate investigative action be taken.

A.3.3 *Reproducibility*: The reproducibility, or global domain precision, for the test method has been established for each material as the values given in Table A.1. Two individual test results obtained in different laboratories (by the proper use of this document) that differ by more than the tabulated values of R, in measurement units, or (R), in percent, should be considered suspect, i.e. to have come from different populations. Such a decision suggests that appropriate investigative action be taken.

Table A.1 — Precision results (type 1 precision)

Compound	Mean density	Within laboratory			Between laboratories			Number of laboratories[a]
		s_r	r	(r)	s_R	R	(R)	
A	0,966	0,001 86	0,005 21	0,54	0,002 47	0,006 91	0,72	10
B	1,223	0,001 19	0,003 34	0,27	0,001 8	0,005 04	0,41	12
C	1,366	0,000 93	0,002 61	0,19	0,002 02	0,005 65	0,41	11
D	1,857	0,001 16	0,003 25	0,17	0,002 15	0,006 02	0,32	11
Average value[b]		0,001 05	0,002 93	0,18	0,002 08	0,005 84	0,37	

Notation used:

s_r is the within-laboratory standard deviation (in measurement units);

s_R is the between-laboratory standard deviation (in measurement units);

r is the repeatability (in measurement units);

(r) is the repeatability (as a percentage of the mean value);

R is the reproducibility (in measurement units);

(R) is the reproducibility (as a percentage of the mean value).

[a] Number of laboratories after outliers deleted (total number of laboratories in ITP: 13).

[b] Simple averages calculated.

A.4 Additional comments

The absolute repeatability and reproducibility, r and R, values are essentially constant over the density range of 0,97 to 1,86. This results in a slight decrease in relative precision, (r) and (R), over this range. Consult Table A.1 for the actual values.

A.5 Bias

Bias is the difference between a measured average test result and a reference or true value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be determined.