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Conducting and dissipative rubbers, vulcanized or thermoplastic — Measurement of resistivity

Caoutchoucs vulcanisés ou thermoplastiques conducteurs et dissipants — Mesurage de la résistivité

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 fourth edition cancels and replaces the third edition (ISO 1853:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— in Clause 3, terms have been newly defined;

— in 4.3, the length of the test piece has been changed from 70 mm to 95 mm;

— in Annex A, information on the solid-state electrometer manufacturer has been updated;

— in Annex B, a calibration schedule has been added.

Introduction

Rubber is normally regarded as a material of high electrical resistivity; consequently, it is widely used as an insulator. However, the incorporation of various materials, in particular certain forms of carbon black, greatly reduces the electrical resistance so that volume resistivities between 10^{13} Ω·m and 0,01 Ω·m are obtainable.

There are various technical and industrial purposes for which rubber with a reduced resistivity is a useful material, the most frequent application being for the dissipation of static charges. In certain circumstances, a lower limit of resistance is specified on a product with this latter application, as a safety precaution to prevent its ignition or to prevent severe shock to a person in contact with it, in the event of faulty insulation or nearby electrical equipment.

Products which, while conducting away static charges, are sufficiently insulating to fulfil the safety requirements above are termed “dissipative rubbers” (the description antistatic rubber is also used). Products which do not fulfil the safety requirements are termed “conducting” rubbers. Since the dimensions of the product are involved, it is not possible to define a suitable range of volume resistivity for either of these classes, but only a range of resistance values between defined points. However, conductive materials are generally considered to have a resistivity below 10^6 Ω·m and dissipative materials to have a resistivity between 10^5 Ω·m and 10^{10} Ω·m.

The principal hazard, apart from static electricity, in most buildings and with most electrical equipment is from leakage currents from normal voltage supply mains. To guard against these hazards, it is recommended that the lower limit of resistance for a dissipative rubber product be $5 \cdot 10^4$ Ω for 250 V mains supplies, which is a maximum current of 5 mA. The limit can be proportionally less for lower voltages.

The maximum resistance which will permit the dissipation of static charges depends on the rate of generation of charge required to produce the minimum voltage which can be regarded as a hazard in a particular application.

Effect of temperature changes and strain on conducting and dissipative rubbers

The resistance of rubber and plastics made conductive by the addition of carbon black is very sensitive to strain and temperature history, since resistance depends on the structural configuration of the carbon particles in the matrix.

Under normal conditions of service with varying temperature and strain history, the resistance of a sample of a given material can vary considerably, for example by a hundred or more times, between freshly strained materials at room temperatures and material which has remained unstrained for a short period at 100 °C.

To make valid comparisons on test pieces, a conditioning treatment is specified so that the measurements are made on test pieces brought close to a condition of zero strain.

Electrode systems

Certain types of electrode, when applied to these rubbers, have a contact resistance which can be many thousands of times greater than the intrinsic resistance of the test piece. Dry contacts under light pressure or point contacts are particularly poor.

The definition of a suitable electrode system is therefore an important part of this document and, in order to satisfy the various practical requirements for tests on laboratory-prepared test pieces, several electrode systems have been selected and are described in Clauses 4, 5 and 6.

Conducting and dissipative rubbers, vulcanized or thermoplastic — Measurement of resistivity

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 the applicability of any other restrictions.

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 the requirements for the laboratory testing of the volume resistivity of specially prepared test pieces of vulcanized or thermoplastic rubber compounds rendered conducting or dissipative by the inclusion of carbon black or ionizable materials. The tests are suitable for materials with a resistivity of less than about 10^8 Ω·m.

Method 1 is the preferred method when test pieces with bonded electrodes are not available.

Method 2 is the preferred method when test pieces are moulded with the inclusion of bonded electrodes.

Method 3 is another method that can be used if the apparatus for method 1 or 2 is not available, but it has lower accuracy.

If a reference to this document is made without specifying the method, method 1 is used.

2 Normative reference

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 14309, *Rubber, vulcanized or thermoplastic — Determination of volume and/or surface resistivity*

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 terms and definitions given in ISO 14309 apply.

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

NOTE There is no distinction between surface and volume resistivity for conductive materials.

4 Method 1

4.1 Apparatus and materials

See Figure 1 for a schematic diagram of the test circuit.

4.1.1 Current source, a source of direct current which has a minimum resistance to earth of 10^{12} Ω and which will not cause a dissipation of power greater than 0,1 W within the test piece.

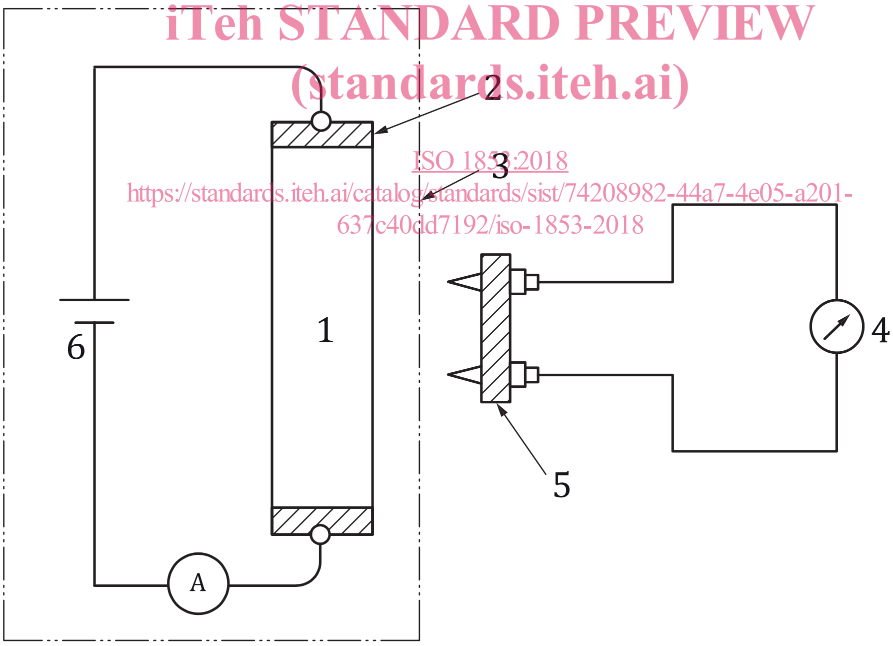
4.1.2 Means of measuring the current to an accuracy of 5 %.

NOTE Very small currents can be computed from measurement of the voltage drop across a known resistance using an electrometer (4.1.5).

4.1.3 Test piece holder and current electrodes, comprising a polystyrene strip of about 10 mm thickness to which the current electrodes are fixed (see Figure 1). The current electrodes shall be of clean metal approximately 5 mm long and running across the full width of the test piece, and be held in place by suitable clamps or grips.

The distance between the current electrodes shall be at least 75 mm, and the resistance between them shall be greater than 10^{12} Ω.

A minimum of three test piece holders shall be available.



Key

1 test piece
2 current electrode
3 sheet of insulating material — resistivity at least 10^{13} Ω
4 electrometer
5 potentiometric electrode
6 adjustable direct-current voltage

Figure 1 — Schematic diagram of test circuit

4.1.4 Potentiometric electrodes, constructed so that they exert a contacting force of approximately 0,65 N for 10 mm wide test pieces or 1,3 N for 20 mm wide test pieces (see Figure 2). The resistance between the potentiometric electrodes shall be greater than 10^{12} Ω.

4.1.5 Electrometer, having an input resistance greater than 10^{11} Ω. References for such instruments are given in Annex A.

4.1.6 Sheet of insulating material, having a resistivity greater than 10^{13} Ω·m.

4.1.7 Oven, capable of being controlled at a temperature of (70 ± 2) °C.

4.2 Calibration

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

4.3 Test piece

Each test piece shall be a strip, (10 ± 0,5) mm or (20 ± 0,5) mm wide, of vulcanized or thermoplastic rubber at least 95 mm long and normally 2 mm, 4 mm or 6,3 mm thick, with a tolerance of uniformity of thickness of ±5 %.

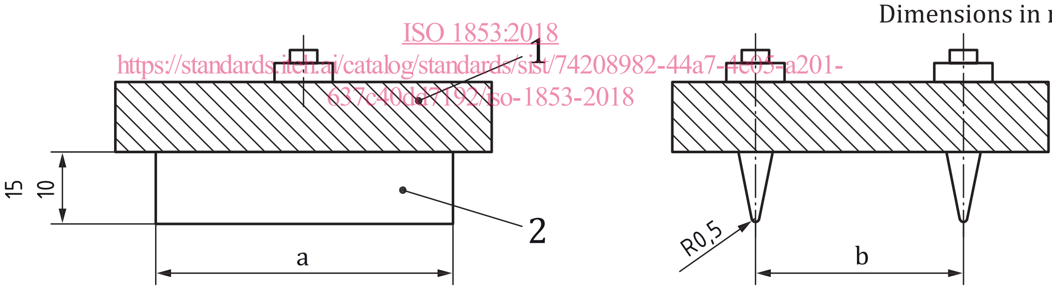
For comparison purposes, test pieces of the same size shall be used.

The test piece can be cut out with a knife or die, but care shall be taken to minimize distortion as this will affect the resistance values.

The surfaces of the test piece shall be clean; if necessary, they can be cleaned by rubbing with Fuller's earth (aluminium magnesium silicate) and water, washing with distilled water and allowing to dry. The surfaces shall not be buffed or abraded.

Do not clean the test pieces with organic materials that would attack or swell the rubber.

Dimensions in millimetres



Key

1 polystyrene strip

2 stainless steel

a Test piece width + at least 10 mm.

b 10 mm to 20 mm measured to ±2 %.

Figure 2 — Potentiometric electrodes

4.4 Number of test pieces

Three test pieces of equal size shall be prepared and tested.

4.5 Procedure

Allow the test piece to rest for not less than 16 h after vulcanization or moulding, in accordance with ISO 23529.

Immediately prior to the start of the test, place the test piece on the test piece holder and clamp the current electrodes to its ends.

Without removing it from the test piece holder, heat the test piece in the oven for 2 h ± 15 min at a temperature of (70 ± 2) °C and then condition for not less than 16 h at a standard laboratory temperature and humidity in accordance with ISO 23529. Place the two potentiometric electrodes in position with a distance of 10 mm to 20 mm between them, ensuring that the knife edges are at right angles to the current flow and that neither is nearer than 20 mm to a current electrode. Measure the distance between the potentiometric electrodes to an accuracy of ±2 %.

Apply the current and, after the current has been passing for 1 min, determine the steady potential between the potentiometric electrodes, using the electrometer at the same standard temperature and humidity as was used to condition the test piece.

Repeat the measuring procedure twice more on the same test piece, moving the potentiometric electrodes each time to obtain measurements over lengths of the test piece evenly distributed between the current electrodes.

Similarly, test the other two test pieces.

4.6 Expression of results

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.

Average the three measurements of resistance for each test piece and calculate the resistivity, ρ, in Ω·m, using Formula (1):

$$\rho = \frac{V \times w \times t}{l \times I} \tag{1}$$

where

V is the measured potential, in V;

w is the width of the test piece, in m;

t is the thickness of the test piece, in m;

l is the distance between the potentiometer electrodes, in m;

I is measured current, in A.

Report the median value of the resistivities of the three test pieces.

4.7 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin,

   2) the method of preparation of the test piece from the sample, for example moulded or cut;

b) test method:

   1) a full reference to the test method used, i.e. ISO 1853:2018, method 1,

   2) the dimensions of the test piece used;

c) test details:

   1) the time, temperature and humidity of conditioning prior to the test,

2) the voltage applied to the current electrodes,

3) the current through the test piece,

4) details of any procedures not specified in this document;

d) test results:

1) the median value of the resistivities of the three test pieces,

2) the individual average resistivity value for each test piece, if required;

e) the date of the test.

5 Method 2

5.1 Apparatus and materials

5.1.1 Brass electrodes, with dimensions as given in Table 1 and Figure 3. These may be either made of solid brass or brass-plated.

5.1.2 Mould, suitable for use with brass electrodes.

5.1.3 Sheets of insulating material, with a volume resistivity greater than 10^{13} Ω·m and a surface resistivity greater than 10^{14} Ω.

5.1.4 Oven, capable of being controlled at (70 ± 2) °C.

5.1.5 Resistance-measuring instrument, any suitable resistance-measuring instrument which does not dissipate more than 0,25 W in the test piece.

5.2 Calibration

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

5.3 Test piece

Each test piece shall be a rectangular strip of rubber bonded during moulding to brass electrodes, with the dimensions given in Table 1 and shown in Figure 3.

Either clean the brass electrodes in dilute nitric acid, wash with distilled water and allow to dry, or clean them with emery cloth. If brass-plated electrodes are used, ensure that the cleaning treatment does not remove the plating. Do not apply cements or rubber solutions to the brass or rubber, as these could affect the electrical resistance.

When moulding the test pieces, ensure that the state of cure is similar to that of the product for which the compound is to be used.

Ensure also that the rubber blanks placed in the mould are of such mass and dimensions as to give the minimum amount of flow and spew in moulding, the spew being of even thickness all round the test piece. Wherever possible, any grain in the material shall run along the length of each test piece.

Remove each test piece from the mould with the minimum of bending and flexing. Remove any adhering spew from the electrodes, taking care not to buff or abrade the test piece, and ensure that the electrodes present a clean surface to the test leads. Reject any test piece showing evidence of insufficient mould filling, poor adhesion to the electrodes or any other defect.

