INTERNATIONAL STANDARD

ISO 14309

Second edition
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Rubber, vulcanized or thermoplastic — Determination of volume and/or surface resistivity

Caoutchouc vulcanisé ou thermoplastique — Détermination de la résistivité transversale et/ou superficielle

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).

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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 second edition cancels and replaces the first edition (ISO 14309:2011), which has been technically revised.

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

— A detailed explanation on the requirement for the electrode gap for volume resistivity has been added in 5.4.2.

— The typical dimension for D_2 has been changed to (60 ± 0,5) mm in 5.4.2.

— To calculate the volume resistivity, the effective area of the main electrode is now derived from $D_1 + B_g$ in 11.1, and the information on B_g has been newly included as Annex D.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Rubber materials are widely used in many industries, either as the major material or forming a part of the product, because of their unique physical properties which can be tailored by compounding to match the particular requirements of the product specification. Although rubbers are generally regarded as insulating materials, they can be made electrically conductive or dissipative by compounding with a certain amount of carbon black or ionizable ingredients. Hence, the range of electrical resistance to be measured is very wide. It is difficult, however, to obtain high accuracy for measurements in the high-resistance range due to a number of factors.

In this document, the guarded-electrode system is used to determine the resistivity of rubber test pieces since it is considered a good compromise between minimizing the errors by shunting away stray currents and using more unwieldy measurement instruments (see also IEC 62631-3-1).

ISO 1853, on the other hand, covers rubber materials with medium to low resistance, i.e. resistivities of 10^8 Ω·m or below. It specifies three methods for determining volume resistivity which minimize or eliminate contact resistance.

The methods specified in this document were originally designed for the determination of both surface and volume resistivity of insulating rubber materials, but their use can be extended to cover the range from high to low resistivity.

It is known that the test results are sensitive to the test conditions, such as temperature and humidity, and to heat and strain history.

Rubber, vulcanized or thermoplastic — Determination of volume and/or surface 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 a method for the determination of the volume and the surface resistivity of vulcanized or thermoplastic rubbers. The method can be applied to materials with a resistivity from 10^1 Ω·m to 10^{17} Ω·m.

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 1382, *Rubber — Vocabulary*

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 1382 and the following 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 http://www.electropedia.org/

3.1
volume resistance
R_v
quotient of a direct-current voltage applied between two electrodes in contact with opposite faces of a test piece and the current between the electrodes, excluding current along the surface

Note 1 to entry: It is expressed in ohms (Ω).

3.2
surface resistance

R_s
quotient of a direct-current voltage applied between two electrodes on the same surface of a test piece and the current between the electrodes

Note 1 to entry: It is expressed in ohms (Ω).

3.3
volume resistivity
ρ_v
measured volume resistance calculated to apply to a cube of unit side

Note 1 to entry: It is expressed in ohm metres (Ω·m).

3.4
surface resistivity
ρ_s
measured surface resistance calculated to apply to a square

Note 1 to entry: It is expressed in ohms (Ω) and the size of the square is immaterial.

3.5
guarded-electrode system
electrode system composed of three electrodes, a guard, and a guarded and an unguarded electrode to reduce measurement errors by protecting the current-measuring electrode from the interfering influences of voltages other than the test voltage, and of stray conductances

Note 1 to entry: Guarding depends on interposing, in all critical insulated parts, guard electrodes which intercept all stray currents that might otherwise cause errors. The guard electrodes are connected together, constituting the guard system and forming with the measurement terminals a three-terminal network. When suitable connections are made, stray currents from spurious external voltages are shunted away from the measurement circuit by the guard system, the insulation resistance from either measurement terminal to the guard system shunts a circuit element which should be of very much lower resistance, and the specimen resistance constitutes the only direct path between the measurement terminals. By this technique, the probability of error is considerably reduced (see 5.3.2 of IEC 62631-3-1:2016 for more details).

4 Principle

The volume and surface resistances of a rubber test piece are determined, using a suitable arrangement of electrodes, from the current flowing when a voltage is applied. The volume and surface resistivities are calculated from the measured resistances, which include the contact resistance.

5 Apparatus

The test equipment consists of a power supply, current-measuring equipment and electrodes:

5.1 Stabilized direct-current power supply, capable of applying a voltage of 1 V to 1 000 V to the test piece.

5.2 Voltmeter, capable of measuring the applied voltage with an accuracy of ±2 %.

5.3 Ammeter or other current-measuring device, capable of measuring a current of 0,01 pA to 100 mA, depending on the resistivity of the test piece to be measured. The accuracy of the current-measuring device shall be better than 5 %.

5.4 Electrodes

5.4.1 Guarded-electrode system

Three electrodes shall be applied to the test piece:

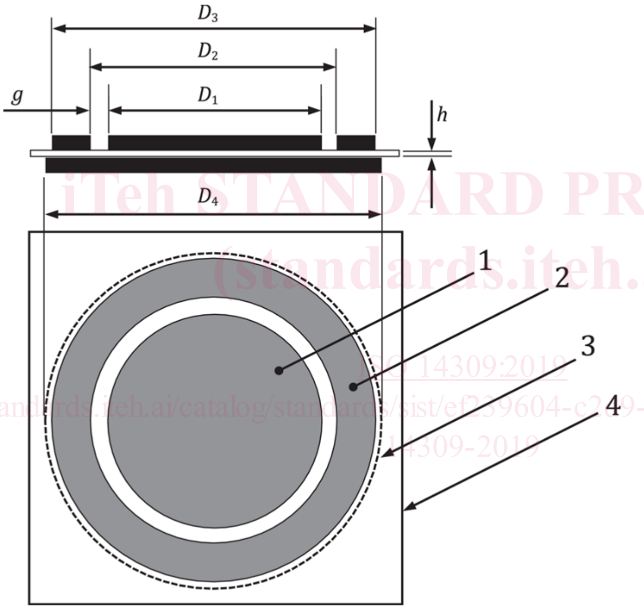
— a main electrode (circular);

— a ring electrode (annular);

— an opposed electrode (circular).

5.4.2 Shapes and dimensions of electrodes

The main (smallest) electrode is circular and is surrounded by the ring electrode. The third electrode is circular and placed on the opposite side of the test piece to the main electrode. The arrangement of the electrodes is shown schematically in Figure 1.



A typical example	
D_1	(50 ± 0,5) mm
D_2	(60 ± 0,5) mm
D_3	(80 ± 0,5) mm
D_4	(83 ± 2) mm
g	5 mm
h	2 mm

Key

1 main electrode

2 ring electrode

3 opposed electrode

4 test piece

Figure 1 — Arrangement of electrodes

The dimensions of the electrodes shall comply with following requirements:

— The diameter D_1 of the main electrode shall be at least ten times the test piece thickness h.

— The gap g between the main electrode and the ring electrode shall be uniform in width. For the measurement of volume resistivity, the gap needs to be such as to give a balance between fringing and current leakage. Fringing is current flowing in a curved path near the electrode and is more prevalent with a wide gap. Leakage current between the main and ring electrodes will be greater with a narrow gap. Gaps between 1 mm and 15 mm have been used depending on the range of resistivity to be measured.

For the measurement of surface resistivity, the gap g shall be at least twice the test piece thickness so that the effect of the volume resistance can be ignored.

— The width of the ring electrode shall be greater than the test piece thickness h.

— The diameter D_4 of the opposed electrode shall be greater than the outer diameter D_3 of the ring electrode.

NOTE The measured volume or surface resistance might depend strongly on the test piece and electrode dimensions. For comparative determinations, the same size of test piece and electrodes need to be used.

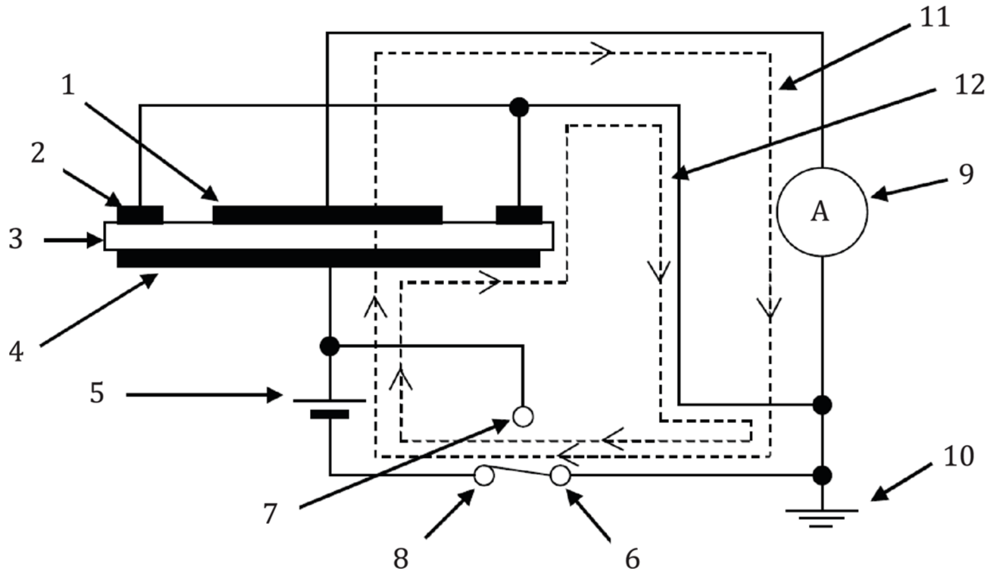
5.4.3 Electrode materials

Electrodes shall be of a conducting material capable of being intimately applied to the test piece. If they are applied before conditioning, the material shall be moisture-permeable. Electrodes other than of rigid metal shall be supplemented by rigid metal backing plates.

NOTE Suitable electrode materials are considered in Annex A.

5.4.4 Electrical circuits

Suitable circuits for testing are shown in Figures 2 and 3.



Key

1 guarded electrode (main electrode)
2 guard electrode (ring electrode)
3 test piece
4 unguarded electrode (opposed electrode)
5 direct-current supply
6 switch
7 connection for short-circuiting electrodes (to discharge test piece)
8 connection for measurement circuit
9 ammeter
10 earth
11 measurement current
12 guard current

Figure 2 — Circuit configuration for volume resistivity

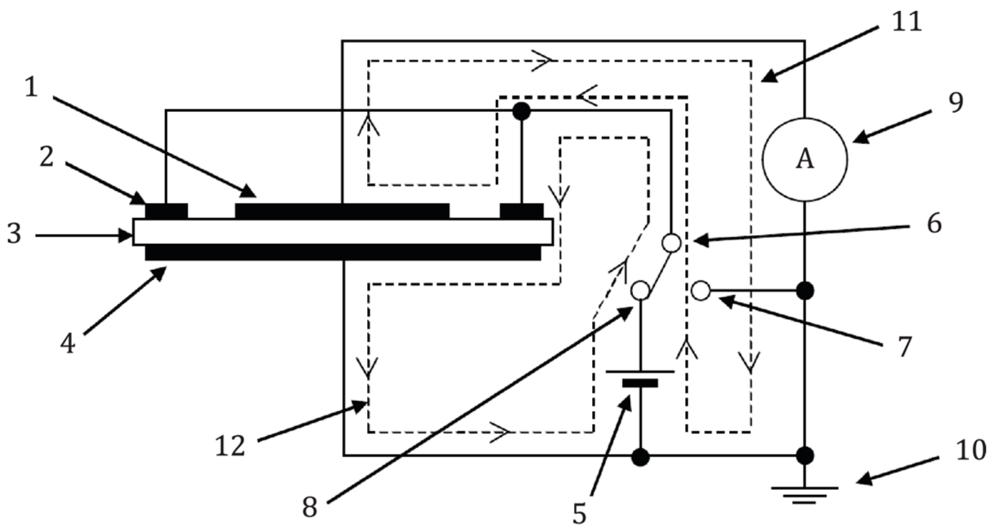


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Figure 3 — Circuit configuration for surface resistivity

6 Calibration

The requirements for calibration of the test apparatus are given in Annex C.

7 Test pieces

7.1 Form

The test piece shall be a flat, smooth sheet of sufficient size that the annular electrode does not reach its edges. The surfaces of the sheet shall not be buffed.

The nominal thickness of the test piece shall be in the range 0,5 mm to 5 mm. Recommended thickness is 1 mm or 2 mm.

The thickness of the test piece shall be measured at several points distributed uniformly over the area covered by the main electrode to the nearest 0,01 mm. The average value shall be used as the test piece thickness. The variation in thickness within a given test piece shall not exceed 10 % of the mean value. Test pieces used for comparative tests shall be, as nearly as practicable, of the same thickness.

7.2 Number of test pieces

Three test pieces shall be used.

8 Conditioning

The time interval between vulcanization and testing shall be in accordance with ISO 23529.

Samples and test pieces shall be stored in accordance with ISO 23529 during the interval between vulcanization and testing.

The material shall be conditioned before testing for a minimum of 16 h at standard laboratory temperature and humidity as specified in ISO 23529.

Metal foil, liquid and conductive elastomeric electrodes shall be applied after conditioning. This shall be carried out either in the conditioning atmosphere or as soon as possible after removal from the conditioning atmosphere. Moisture-permeable electrodes can be applied before conditioning.

9 Test conditions

9.1 Temperature and humidity

Tests are normally performed at a standard laboratory temperature as defined in ISO 23529, although elevated or subnormal temperatures can be used. In the latter case, the test temperature shall be selected from the list in ISO 23529.

With materials for which the results are known to be sensitive to humidity, the test shall be carried out under standard laboratory conditions (temperature and humidity) as defined in ISO 23529.

NOTE An influence of humidity has been observed with polyurethane rubbers and other rubbers containing hydrophilic fillers.

9.2 Applied voltage

The test voltage applied to the test piece shall be in the range 1 V to 1 000 V. The voltage shall be selected in consideration of the resistivity of the test piece and rated current of the ammeter. The power dissipated in the test piece shall not exceed 0,1 W, in order to minimize heat generation. Recommended voltages are 1 V, 10 V, 100 V, 500 V and 1 000 V. Suitable test conditions are given in Annex B.

NOTE Setting the dissipation of electric power at less than 0,1 W is based on the estimation of the heat generated. Assuming that dissipation of electric power within a test piece is 1 W, 60 J is generated during a 60 s current application. Supposing that the mass of the rubber in the heat generation area and the specific heat of the rubber are 3 g and 2 000 J·kg·K, respectively, and that all the heat generated acts to raise the temperature of the test piece, the resulting temperature rise is approximately 10 K. In practice, heat is dissipated out of the test piece and the actual temperature rise will be less than that calculated. If the dissipation power is limited to 0,1 W, the temperature rise is estimated to be approximately 1 K, which can be considered negligible.

10 Test procedure

Measure the electrode dimensions and the width of the gap g to the nearest 0,05 mm.

Apply the electrodes, ensuring that there is intimate contact with the test piece over the entire electrode area. Take care to avoid excessive pressure since the test result can be affected by deformation of the test piece. When conductive paint electrodes are used, ensure that the film is not damaged and not separated from the test piece.