IEC 62631-3-4

Edition 1.0 2019-03

INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Dielectric and resistive properties of solid insulating materials – Part 3-4: Determination of resistive properties (DC methods) – Volume resistance and volume resistivity at elevated temperatures

Propriétés diélectriques et résistives des matériaux isolants solides – Partie 3-4: Détermination des propriétés résistives (méthodes en courant continu) – Résistance transversale et résistivité transversale aux températures élevées

IEC 62631-3-4:2019-03(en-fr)

IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC
The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications
The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigendum or an amendment might have been published.

IEC publications search - webstore.iec.ch/advsearchform
The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee,…). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished
Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and once a month by email.

IEC Customer Service Centre - webstore.iec.ch/csc
If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

Electropedia - www.electropedia.org
The world's leading online dictionary on electrotechnology, containing more than 22 000 terminological entries in English and French, with equivalent terms in 16 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

IEC Glossary - std.iec.ch/glossary
67 000 electrotechnical terminology entries in English and French extracted from the Terms and Definitions clause of IEC publications issued since 2002. Some entries have been collected from earlier publications of IEC TC 37, 77, 86 and CISPR.

A propos de l'IEC
La Commission Electrotechnique Internationale (IEC) est la première organisation mondiale qui élabore et publie des Normes internationales pour tout ce qui a trait à l'électricité, à l'électronique et aux technologies apparentées.

A propos des publications IEC
Le contenu technique des publications IEC est constamment revu. Veuillez vous assurer que vous possédez l'édition la plus récente, un corrigendum ou amendement peut avoir été publié.

Recherche de publications IEC - webstore.iec.ch/advsearchform
La recherche avancée permet de trouver des publications IEC en utilisant différents critères (numéro de référence, texte, comité d'études,…). Elle donne aussi des informations sur les projets et les publications remplacées ou retirées.

IEC Just Published - webstore.iec.ch/justpublished
Restez informé sur les nouvelles publications IEC. Just Published détaille les nouvelles publications parues. Disponible en ligne et une fois par mois par email.

Service Clients - webstore.iec.ch/csc
Si vous désirez nous donner des commentaires sur cette publication ou si vous avez des questions contactez-nous: sales@iec.ch.

Electropedia - www.electropedia.org
Le premier dictionnaire d'électrotechnologie en ligne au monde, avec plus de 22 000 articles terminologiques en anglais et en français, ainsi que les termes équivalents dans 16 langues additionnelles. Egalement appelé Vocabulaire Electrotechnique International (IEV) en ligne.

Glossaire IEC - std.iec.ch/glossary
67 000 entrées terminologiques électrotechniques, en anglais et en français, extraites des articles Termes et Définitions des publications IEC parues depuis 2002. Plus certaines entrées antérieures extraites des publications des CE 37, 77, 86 et CISPR de l'IEC.

IEC 62631-3-4

Edition 1.0 2019-03

INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Dielectric and resistive properties of solid insulating materials – Part 3-4: Determination of resistive properties (DC methods) – Volume resistance and volume resistivity at elevated temperatures

Propriétés diélectriques et résistives des matériaux isolants solides – Partie 3-4: Détermination des propriétés résistives (méthodes en courant continu) – Résistance transversale et résistivité transversale aux températures élevées

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

COMMISSION
ELECTROTECHNIQUE
INTERNATIONALE

ICS 17.220.99; 29.035.01

ISBN 978-2-8322-6688-5

Warning! Make sure that you obtained this publication from an authorized distributor.
Attention! Veuillez vous assurer que vous avez obtenu cette publication via un distributeur agréé.

CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

____

DIELECTRIC AND RESISTIVE PROPERTIES OF SOLID INSULATING MATERIALS –

Part 3-4: Determination of resistive properties (DC methods) – Volume resistance and volume resistivity at elevated temperatures

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as “IEC Publication(s)”). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.

3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.

5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
6) All users should ensure that they have the latest edition of this publication.
7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 62631-3-4 has been prepared by IEC technical committee 112: Evaluation and qualification of electrical insulating materials and systems.

This edition of IEC 62631-3-4 cancels and replaces IEC 60345 “Method of test for electrical resistance and resistivity of insulating materials at elevated temperatures”, published in 1971. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to IEC 60345:

a) The revised standard becomes part of the series IEC 62631-3-x. Title of the standard is changed and adapted to the series as Part 3-4.
b) Clauses 2 "Normative references", 3 "Terms and definitions", and 4 "Significance" are added.

c) Subclauses 5.2 "Power supply, Voltage", 5.3.1.2 "Number of test specimens" and 5.3.1.3 "Conditioning and pre-treatment of test specimens" are added.

d) In 5.3.5 "Special precautions during measurements", errors analysis in the measurement of current are modified, and aligned with IEC 62631-3-1.

e) In 6.2 "Increasing the temperature by steps (method B)", the method for more than one specimen is removed.

f) The standard atmospheric conditions for testing and conditioning, especially the temperature, are replaced according to IEC 60212.

g) The circuit diagram of test apparatus is modified, and the structure diagram and pictures of test apparatus are added in Annex A.

h) The orders of part clauses are adjusted.

The text of this International Standard is based on the following documents:

CDV	Report on voting
112/406/CDV	112/445/RVC

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62631 series, published under the general title *Dielectric and resistive properties of solid insulating materials*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

DIELECTRIC AND RESISTIVE PROPERTIES OF SOLID INSULATING MATERIALS –

Part 3-4: Determination of resistive properties (DC methods) – Volume resistance and volume resistivity at elevated temperatures

1 Scope

This part of IEC 62631 covers procedures for the determination of insulation resistance and volume resistivity of insulating materials by applying DC-voltage and temperatures up to 800 °C. The typical application materials include high temperature mica plate and alumina ceramics.

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.

IEC 60212:2010, *Standard conditions for use prior to and during the testing of solid electrical insulating materials*

IEC 62631-3-1, *Dielectric and resistive properties of solid insulating materials – Part 3-1: Determination of resistive properties (DC methods) – Volume resistance and volume resistivity – General method*

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:

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

3.1
heating chamber
device which is used for supplying an elevated temperature to the specimen

3.2
volume resistance
part of the insulation resistance which is due to conduction through the volume

Note 1 to entry: Volume resistance is expressed in Ω.

3.3
volume resistivity
volume resistance of a material related to its volume

Note 1 to entry: Volume resistivity is expressed in Ωm.

Note 2 to entry: For insulating materials, the volume resistivity is usually determined by means of measuring electrodes arranged on a sheet of the material.

Note 3 to entry: According to IEC 60050-121: Electromagnetism, "conductivity" is defined as the "scalar or tensor quantity, the product of which by the electric field strength in a medium is equal to the electric current density" and "resistivity" as the "inverse of the conductivity when this inverse exists". Measured in this way, the volume resistivity is an average of the resistivity over possible heterogeneities in the volume incorporated in the measurement; it includes the effect of possible polarization phenomena at the electrodes.

4 Significance

Some types of insulating materials are used under high temperatures up to 800 °C, such as mica plate and alumina ceramics, when mica plate is used for supporting aluminum electrolytic tank and alumina ceramics are used for high temperature crucibles or resistance furnace tubes.

For these purposes, it is generally desirable to have the insulation resistance as high as possible. Volume resistance and volume resistivity can be used as an aid in the choice of an insulating material for a specific application. The change in resistivity with temperature may be great and shall be known when designing for operation conditions.

5 Method of test

5.1 General

This method describes specific types of materials used at elevated temperatures; the typical upper temperature limit is 800 °C. Different types of electrodes can be used, depending on the specific measurement or product demands.

NOTE Thickness changes due to the high temperature can affect the measurement results.

5.2 Power supply and test voltages

A source of very steady DC voltage is required. This can be provided either by batteries or by rectified and stabilized power supply. The degree of stability required is such that the change in current due to any change in voltage is negligible compared with the current to be measured. Commonly specified test voltages to be applied to the complete specimen are 10 V, 100 V, 500 V, 1 000 V. If not otherwise stipulated, a voltage of 500 V shall be used.

5.3 Equipment

5.3.1 Specimens and electrodes

5.3.1.1 Preparation of specimens and electrodes

For insulation resistance measurements, the specimens shall be of any suitable size and shape and shall have electrodes already attached. When volume resistivity is measured, guarded electrodes are suggested. The preferred dimensions of test specimens shall be those given in the test procedures of IEC 62631-3-1. The specimen electrodes shall consist of fired-on conducting paint or a conducting coating evaporated or sprayed onto the specimen surfaces. Platinum is a suitable electrode material.

If other types of metal, such as silver or gold are used, make sure that they will not migrate into the sample or oxidize at test temperature.

The specimens shall be mounted securely among electrode backing plates within the heating chamber. These backing plates and their respective leads shall be made of a metal which is mechanically stable and resistive to oxidation. High-heat-resistant alloys such as stainless steel may also be utilized. The backing plates shall be of sufficient thickness to prevent warping and to provide heat equalization between the specimens and the electrode backing plates.

For mica plate and aluminium oxide specimen, a plate sample is recommended.

5.3.1.2 Number of test specimens

The number of specimens to be tested shall be determined by the relevant product standards. If no such data is available, at least three specimens shall be tested.

5.3.1.3 Conditioning and pre-treatment of test specimen

Conditioning and any other pre-treatment of the test specimen shall be done according to the relevant product standard. If no product standards exist, conditioning shall be done according to IEC 60212 standard conditions for use prior to and during the testing of solid electrical insulating materials.

5.3.2 Heating chamber

For heating the specimen, a suitable electric oven can normally be utilized up to 500 °C; a resistance furnace shall be used if the testing temperature reaches 800 °C. The construction shall be such that the specimen is subjected to a uniform temperature throughout its total volume with temperature fluctuations as small as possible. The atmospheric conditions for testing and conditioning shall be in accordance with IEC 60212. The samples shall be tested during the heat preservation period.

An adequate muffle should be provided to shield the specimen from direct radiation by the heating elements. This muffle may be made of a ceramic such as aluminum oxide or equivalent. A grounded metallic shield of stainless steel or equivalent metal shall also be provided within the oven. The shield shall act as a guard to prevent leakage currents between the heating circuit and the measuring circuit.

In the case of very high resistance specimens, it may be necessary to disconnect the heating element to prevent interference during the measurement.

A typical structure of heating chamber is shown in Annex A. Alternatively, the tests may be performed in an inert atmosphere.

5.3.3 Measuring leads

Insulated measuring leads shall be brought into the furnace through high-resistance ceramic insulators located in a cool zone and adequately guarded so as to prevent leakage current from affecting the test results.

NOTE Alternatively, the leads can be passed through holes in the top or in the wall of the furnace which is earthed. If stiff leads are used, they can be supported externally so as not to touch anything but their supports. The supports will be relatively cool and thus can be made of any rigid insulating material.

5.3.4 Temperature control

The temperature control mode of the heating chamber shall be set by program. A means of temperature control shall be provided which can maintain temperature tolerances according to IEC 60212 standard conditions for use prior to and during the testing of solid electrical insulating materials. The use of two thermocouples is recommended, one in the chamber for control and a second for the direct measurement of the specimen temperature.

The temperature of the specimen shall be measured using a thermocouple mounted as close as possible to the specimen without causing electrical interaction with the measurement of resistance. For example, the thermocouple may be inserted directly into a hole extending almost to the surface of the backing plate adjacent to the specimen. The hole can be drilled from the opposite face of the plate perpendicular to the surface of the specimen or from the side of the plate parallel to the specimen surface. If the thermocouple is mounted within the electrode backing plate, the leads and the temperature-indicating instrument shall be adequately insulated or the thermocouple shall be disconnected or removed when measurements are made.

5.3.5 Special precautions during measurements

Errors in the measurement of current may result from the fact that the current-measuring device is shunted by the resistance between the guarded terminal and the guard system. To ensure satisfactory operation of the equipment, a measurement should be made with the lead from the voltage source to the specimen disconnected. Under this condition, the equipment should indicate infinite resistance within its sensitivity. If suitable standards of known values are available, they may be used to test the operation of the equipment.

If the material insulating the leads into the oven is subjected to heat, the insulation resistance of the lead insulation may become low enough to affect the measurements. The leakage resistance shall be determined by a separate measurement at each temperature. Thermocouple potentials between dissimilar metals, when they are used in leads and electrode holders, can cause measurement errors. A measurement of current, with the supply voltage replaced by a short circuit, will indicate the magnitude of this thermocouple effect.

5.4 Calibration

The equipment shall be calibrated in the magnitude of the volume resistance measured at room temperature.

NOTE Calibration resistors in the range up to 100 TΩ are commercially available.

6 Procedure

6.1 Continuously increasing temperature (method A)

This method is suitable for obtaining quickly an approximate relationship between resistance and temperature of a single specimen over a wide temperature range. The method is suitable only with materials for which the effects of dielectric absorption can be neglected, or for obtaining comparative results for similar materials. The specimen shall be mounted tightly between the electrode backing plates, but not so tightly that the specimen is distorted while being heated. The specified voltage shall be applied to the test specimen and the temperature shall be increased at a rate depending on the thickness of the material and not higher than 5 K/min. A sufficient number of resistance measurements shall be made, as the temperature is increased, so as to define adequately the relationship between resistance and temperature.

6.2 Increasing the temperature by steps (method B)

This method is suitable for obtaining the relationship between resistance and temperature of a single specimen more accurately than the one which is possible with continuously increasing temperature. It is useful also with specimens for which dielectric absorption is a problem.

The test specimen shall be mounted tightly between the electrode backing plates but not so tightly that the specimen is distorted while being heated. The temperature of the test specimen shall be increased from room temperature to the desired test temperature and subsequently from each test temperature step to the next.

NOTE 1 The test chamber is controlled in such a manner that the temperature of the electrode backing plate does not exceed the desired test temperature. Usually the temperature step is kept long enough to ensure the sample temperature meets the requirements in the high temperature range. If a temperature overshoot occurs, it will take some time to wait for the sample end temperature to meet the final test temperature.

When the temperature of the electrode backing plate is within the desired test temperature according to Table 2 of IEC 60212:2010, the voltage specified in the material specification shall be applied to the specimen for 1 min (or for other times as specified) and the resistance shall then be measured. When the measurement is completed, the voltage shall be removed and the high voltage, measuring and guarded electrodes shall be connected to each other (short-circuited).

A sufficient number of test temperatures, but no less than five, shall be selected to define adequately the relationship between temperature and resistance over the desired range of temperatures. At the lower temperatures, the temperature increments should be relatively small, for example 10 K. As the test temperature is increased, the temperature increments should also be increased.

NOTE 2 The logarithm of resistance (or resistivity) is often plotted as a function of the reciprocal absolute temperature.

6.3 Precautions to be taken

When the current has not stabilized in the time specified for measurement, due to dielectric relaxation, it may be necessary to determine the resistance as a function of time so that the resistance value obtained at stabilization can be estimated.

When the resistance of the material being tested is relatively low, it may be necessary to make the measurements at a reduced voltage to avoid the effects of specimen heating.

For those materials in which polarization effects play a part, with concentration of ions at one or both electrodes, the results may be of doubtful value.

Unless the effect of thermal degradation is specially required, the specimen shall be kept at the test temperature only long enough to attain thermal equilibrium. The maximum permissible time of exposure to the test temperature should be determined by comparing values of resistance (one minute of electrification) measured periodically over a time, which are comparable to or longer than that expected in the tests (using an additional specimen).

After a series of tests at progressively higher temperatures, an additional measurement shall be made at the starting temperature to determine whether the exposure to the elevated temperatures has produced a permanent change in the specimens.

6.4 Calculation of volume resistivity

The volume resistivity shall be calculated from the following formula:

$$\rho = R_x \times A / h$$

where

ρ is the volume resistivity in Ωm;

R_x is the volume resistance measured in Ω;

A is the effective area of the electrode in m^2;

h is the thickness of the specimen in m.

7 Report

The report shall include the following:

- complete identification and description of the material tested, including source and manufacturer’s code;
- shape and thickness of test specimens;
- type of electrodes and nature of the electrode backing plates;
- test voltage and time of electrification;

- accuracy of the instrument and calibration method, depending on the measured values of resistance, if necessary;
- curing conditions of the material and any pre-treatment;
- conditioning of samples and climatic conditions under test;
- description of test set-up and instrument used for the test;
- number of samples;
- each single value and the median of volume resistance and volume resistivity respectively at each temperature;
- the method of increasing the temperature, i.e. method A or B;
- date of test;
- any other important observations if applicable.