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Méthodes pour la mesure de la résistivité transversale et de la résistivité superficielle des matériaux isolants électriques solides

Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

METHODS OF TEST FOR VOLUME RESISTIVITY AND SURFACE RESISTIVITY OF SOLID ELECTRICAL INSULATING MATERIALS

FOREWORD

1) The formal decisions or agreements of the IEC on technical matters, prepared by Technical Committees on which all the National Committees having a special interest therein are represented, express, as nearly as possible, an international consensus of opinion on the subjects dealt with.
2) They have the form of recommendations for international use and they are accepted by the National Committees in that sense.
3) In order to promote international unification, the IEC expresses the wish that all National Committees should adopt the text of the IEC recommendation for their national rules in so far as national conditions will permit. Any divergence between the IEC recommendation and the corresponding national rules should, as far as possible, be clearly indicated in the latter.

PREFACE

This standard has been prepared by Sub-Committee 15A: Short-time Tests, of IEC Technical Committee No. 15: Insulating Materials.

It forms the second edition of IEC Publication 93.

A first draft was discussed at the meeting held in Toronto in 1976. As a result of this meeting, a draft, Document 15A(Central Office)35, was submitted to the National Committees for approval under the Six Months' Rule in November 1977.

Amendments, Document 15A(Central Office)39, were submitted to the National Committees for approval under the Two Months' Procedure in October 1979.

The National Committees of the following countries voted explicitly in favour of publication:

Austria
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Brazil
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Italy
Korea (Republic of)
New Zealand
Norway
Poland
Spain
Sweden
Switzerland
United Kingdom
United States of America
Yugoslavia

Other IEC publications quoted in this standard:

Publications Nos. 167: Methods of Test for the Determination of the Insulation Resistance of Solid Insulating Materials.

212: Standard Conditions for Use Prior to and during the Testing of Solid Electrical Insulating Materials.

260: Test Enclosures of Non-injection Type for Constant Relative Humidity.

Contents

National foreword

This British Standard is identical with IEC Publication 93 (Second edition) *"Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials"* published in 1980 by the International Electrotechnical Commission (IEC). This British Standard is published under the direction of the General Electrotechnical Engineering Standards Committee.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards.

Cross-references

International Standard	Corresponding British Standard
IEC 212:1971	BS 2844:1972 *Memorandum on conditioning of solid electrical insulating materials prior to and during testing* (Technically equivalent)

The technical committee has reviewed the provisions of IEC 167 and IEC 260 to which reference is made in this standard, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard for IEC 260 is BS 3718 *"Laboratory humidity ovens (non-injection type)"*.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 14, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

These methods of test cover procedures for the determination of volume and surface resistance and calculations for the determination of volume and surface resistivity of solid electrical insulating materials.

Both volume resistance and surface resistance tests are affected by the following factors: the magnitude and time of voltage application, the nature and geometry of the electrodes, and the temperature and humidity of the ambient atmosphere and of the specimens during conditioning and measurement. Recommendations are made for these factors.

2 Definitions

2.1
volume resistance

the quotient of a direct voltage applied between two electrodes placed on two faces (opposite) of a specimen, and the steady-state current between the electrodes, excluding current along the surface, and neglecting possible polarization phenomena at the electrodes

NOTE Unless otherwise specified, the volume resistance is determined after 1 min of electrification.

2.2
volume resistivity

the quotient of a d.c. electric field strength and the steady-state current density within an insulating material. In practice it is taken as the volume resistance reduced to a cubical unit volume

NOTE The SI unit of volume resistivity is the ohm metre. In practice the unit ohm centimetre is also used.

2.3
surface resistance

the quotient of a direct voltage applied between two electrodes on a surface of a specimen, and the current between the electrodes at a given time of electrification, neglecting possible polarization phenomena at the electrodes

NOTE 1 Unless otherwise specified, the surface resistance is determined after 1 min of electrification.

NOTE 2 The current generally passes mainly through a surface layer of the specimen and any associated moisture and surface contaminant, but it also includes a component through the volume of the specimen.

2.4
surface resistivity

the quotient of a d.c. electric field strength, and the linear current density in a surface layer of an insulating material. In practice it is taken as the surface resistance reduced to a square area. The size of the square is immaterial

NOTE The SI unit of surface resistivity is the ohm. In practice this is sometimes referred to as "ohms per square".

2.5
electrodes

measuring electrodes are conductors of defined shape, size and configuration in contact with the specimen being measured

GENERAL NOTE. *Insulation resistance* is the quotient of a direct voltage applied between two electrodes in contact with a specimen and the total current between the electrodes. The insulation resistance depends on both volume and surface resistivity of the specimen (see IEC Publication 167: Methods of Test for the Determination of the Insulation Resistance of Solid Insulating Materials).

3 Significance

3.1 Insulating materials are used in general to isolate components of an electrical system from each other and from earth; solid insulating materials may also provide mechanical support. For these purposes it is generally desirable to have the insulation resistance as high as possible, consistent with acceptable mechanical, chemical and heat-resisting properties. Surface resistance changes very rapidly with humidity, while volume resistance changes only slowly, although the final change may be greater.

3.2 Volume resistivity can be used as an aid in the choice of an insulating material for a specific application. The change of resistivity with temperature and humidity may be great and must be known when designing for operating conditions. Volume resistivity measurements are often used in checking the uniformity of an insulating material, either with regard to processing or to detect conductive impurities that affect the quality of the material and that may not be readily detectable by other means.

3.3 When a direct voltage is applied between electrodes in contact with a specimen, the current through it decreases asymptotically towards a steady-state value. The decrease of current with time may be due to dielectric polarization and the sweep of mobile ions to the electrodes. For materials having volume resistivities less than about $10^{10}\ \Omega \cdot m$ ($10^{12}\ \Omega \cdot cm$), the steady-state is in general reached within 1 min, and the resistance is then determined after this time of electrification. For materials of higher volume resistivity the current may continue to decrease for several minutes, hours, days, or even weeks. For such materials, therefore, longer electrification times are used, and, if relevant, the material is characterized by the time dependence of the volume resistivity.

3.4 Surface resistance or surface conductance cannot be measured accurately, only approximated, because more or less volume conductance is nearly always involved in the measurement. The measured value is largely a property of the contamination of the surface of the specimen at the time of measurement. However, the permittivity of the specimen influences the deposition of contaminants, and their conductive capabilities are affected by the surface characteristics of the specimen. Thus surface resistivity is not a material property in the usual sense, but can be considered to be related to material properties when contamination is involved.

Some materials, such as laminates, may have quite different resistivities in a surface layer and in the interior. It may therefore be of interest to measure the intrinsic property of a clean surface. Cleaning procedures aimed at producing consistent results should be fully specified bearing in mind the possible effect of solvents and other factors of the cleaning procedure on the surface characteristics.

The surface resistance, especially when high, often changes in an erratic manner, and in general depends strongly on the time of electrification; for measurements, 1 min of electrification is usually specified.

4 Power supply

A source of very steady direct voltage is required. This may be provided either by batteries or by a 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 100 V, 250 V, 500 V, 1 000 V, 2 500 V, 5 000 V, 10 000 V and 15 000 V. Of these the most frequently used are 100 V, 500 V and 1 000 V.

In some cases, the specimen resistance depends upon the polarity of the applied voltage.

If the resistance is polarity dependent, this should be indicated. The geometric (arithmetic mean of the logarithmic exponents) mean of the two resistance values is taken as the result.

Since the specimen resistance may be voltage dependent, the test voltage should be stated.

5 Measuring methods and accuracy

5.1 Methods

The methods commonly in use for measuring high resistances are either direct methods or comparison methods.

The direct methods depend upon simultaneous measurement of the direct voltage applied to the unknown resistance and the current through it (voltmeter-ammeter method).

The comparison methods establish the ratio of the unknown resistance to the resistance of a known resistor, either in a bridge circuit, or by comparison of currents through the resistances at fixed voltage.

Examples illustrating the principles are described in Appendix A.

The voltmeter-ammeter method requires a reasonably accurate voltmeter, but the sensitivity and accuracy of the method depend mainly on the properties of the current measuring device, which may be a galvanometer, an electronic amplifier instrument, or an electrometer.

The bridge method requires only a sensitive current detector as null indicator, and the accuracy is mainly determined by the known bridge arm resistors, which are obtainable with high precision and stability over a wide range of resistance values.

The accuracy of the current comparison method depends on the accuracy of the known resistor, and on the stability and linearity of the current measuring device, including associated measuring resistors, etc., whereas the exact values of current are insignificant, as long as the voltage is constant.

Determination of volume resistivity in accordance with Sub-clause **10.1** using a galvanometer in the voltmeter-ammeter method is feasible for resistances up to about 10^{11} Ω. For higher values, the use of a d.c. amplifier or electrometer is recommended.

In the bridge method, it is not possible to measure the current directly in the short-circuited specimen (see Sub-clause **10.1**).

The methods utilizing current measuring devices permit automatic recording of the current to facilitate determination of the steady state (Sub-clause **10.1**).

Special circuits and instruments for measuring high resistance are available. These may be used, provided that they are sufficiently accurate and stable, and that, where needed, they enable the specimen to be properly short-circuited, and the current measured before electrification.

5.2 Accuracy

The measuring device should be capable of determining the unknown resistance with an overall accuracy of at least ± 10 % for resistances below 10^{10} Ω, and ± 20 % for higher values.
See also Appendix A.

5.3 Guarding

The insulation of the measuring circuit is composed of materials which, at best, have properties comparable with those of the material under test. Errors in the measurement of the specimen may arise from:

a) stray current from spurious external voltages which are usually unknown in magnitude and often sporadic in character;

b) undue shunting of the specimen resistance, reference resistors, or the current measuring device by insulation, having resistance of unknown, and possibly variable magnitude.

An approximate correction of these difficulties may be obtained by making the insulation resistance of all parts of the circuit as high as possible under the conditions of use. This may lead to unwieldy apparatus which is still inadequate for measurement of insulation resistances higher than a few hundred megohms. A more satisfactory correction is obtained by using the technique of guarding.

Guarding depends on interposing, in all critical insulated parts, guard conductors which intercept all stray currents that might otherwise cause errors. The guard conductors are connected together, constituting the guard system and forming with the measuring terminals a three terminal network. When suitable connections are made, stray currents from spurious external voltages are shunted away from the measuring circuit by the guard system, the insulation resistance from either measuring 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 measuring terminals. By this technique the probability of error is considerably reduced. Figure 1, page 10, shows the basic connections for guarded electrodes used for volume resistance and surface resistance measurements.

Proper use of the guard system for the method involving current measurement is illustrated in Figure 5 and Figure 7, pages 12 and 13, where the guard system is shown connected to the junction of the voltage source and current-measuring device. In Figure 6, page 13, for the Wheatstone bridge method, the guard system is shown connected to the junction of the two lower-valued resistance arms. In all cases, to be effective, guarding shall be complete, and shall include any control operated by the observer in making the measurement.

Electrolytic, contact, or thermal e.m.f.'s. existing between guard and guarded terminals can be compensated if they are small. Care must be taken that such e.m.f.'s do not introduce appreciable errors in the measurements.

Errors in current measurements may result from the fact that the current-measuring device is shunted by the resistance between the guarded terminal and the guard system. This resistance should be at least 10 and preferably 100 times that of the current-measuring device. In some bridge techniques, the guard and measuring terminal are brought to nearly the same potential but a standard resistor in the bridge is shunted by the resistance between the unguarded terminal and the guard system. This resistance should be at least 10 and preferably 100 times that of the reference resistor.

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.

6 Test specimens

6.1 Volume resistivity

For the determination of volume resistivity the test specimen may have any practicable form that allows the use of a third electrode to guard against error from surface effect. For specimens that have negligible surface leakage, the guard may be omitted when measuring volume resistance, provided that it has been shown that its omission has negligible effect on the result.

The gap on the surface of the specimen between the guarded and guard electrodes should be of uniform width and as narrow as possible provided that the surface leakage does not cause error in the measurement. A gap of 1 mm is usually the smallest practicable.

Examples of electrode arrangements with three electrodes are shown in Figure 2 and Figure 3, pages 10 and 11. In the measurement of volume resistance, electrode No. 1 is the guarded electrode, No. 2 is the guard electrode, and No. 3 is the unguarded electrode. The diameter d_1 (Figure 2), or length l_1 (Figure 3) of the guarded electrode should be at least ten times the specimen thickness h and for practical reasons usually at least 25 mm. The diameter d_4 (or length l_4) of the unguarded electrode, and the outer diameter d_3 of the guard electrode (or length l_3 between the outer edges of the guard electrodes) should be equal to the inner diameter d_2 of the guard electrode (or length l_2 between the inner edges of the guard electrodes) plus at least twice the specimen thickness.

6.2 Surface resistivity

For the determination of surface resistivity the test specimen may have any practicable form that allows the use of a third electrode to guard against error from volume effects. The three electrode arrangements of Figure 2 and Figure 3 are recommended. The resistance of the surface gap between electrodes Nos. 1 and 2 is measured directly by using electrode No. 1 as the guarded electrode, electrode No. 3 as the guard electrode and electrode No. 2 as the unguarded electrode. The resistance so measured includes the surface resistance between electrodes Nos. 1 and 2 and the volume resistance between the same two electrodes. With suitable dimensioning of the electrodes, however, the effect of the volume resistance can be made negligible for wide ranges of ambient conditions and material properties. This condition may be achieved for the arrangement of Figure 2 and Figure 3 when the electrodes are dimensioned so that the surface gap width g is at least twice the specimen thickness; 1 mm is normally the smallest practicable. The diameter d_1 (or length l_1) of the guarded electrode should be at least ten times the specimen thickness h, and for practical reasons usually at least 25 mm.

Alternatively, straight electrodes or other arrangements with suitable dimensions may be used.

NOTE Due to the influence of current through the interior of the test specimen the calculated value of surface resistivity may depend strongly on the specimen and electrode dimensions. For comparative determinations it is therefore recommended to use specimens of identical form with the electrode arrangement of Figure 2 with d_1 = 50 mm, d_2 = 60 mm, and d_3 = 80 mm.

7 Electrode material

7.1 General

The electrodes for insulating materials should be of a material that is readily applied, allows intimate contact with the specimen surface and introduces no appreciable error because of electrode resistance or contamination of the specimen. The electrode material should be corrosion resistant under the conditions of the test. The following are typical electrode materials that may be used. The electrodes shall be used with suitable backing plates of the given form and dimensions.

It may be advantageous to use two different electrode materials or two methods of application to see if appreciable error is introduced.

7.2 Conductive silver paint

Certain types of commercially available, high-conductivity silver paints, either air-drying or low-temperature-baking varieties are sufficiently porous to permit diffusion of moisture through them and thereby allow the test specimens to be conditioned after application of the electrodes. This is a particularly useful feature in studying resistance-humidity effects as well as changes with temperature. However, before conductive paint is used as an electrode material, it should be established that the solvent in the paint does not affect the electrical properties of the specimen. Reasonably smooth edges of guard electrodes may be obtained with a fine-bristle brush. However, for circular electrodes, sharper edges may be obtained by the use of a compass for drawing the outline circles of the electrodes and filling in the enclosed areas by brush. Clamp-on masks may be used if the electrode paint is sprayed on.

7.3 Sprayed metal

Sprayed metal may be used if satisfactory adhesion to the test specimen can be obtained. Thin sprayed electrodes may have certain advantages in that they are ready for use as soon as applied. They may be sufficiently porous to allow the specimen to be conditioned, but this should be verified. Clamp-on masks may be used to produce a gap between the guarded electrode and the guard electrode.

7.4 Evaporated or sputtered metal

Evaporated or sputtered metal may be used under the same conditions as given in Sub-clause **7.3** where it can be shown that the material is not affected by ion bombardment or vacuum treatment.

7.5 Liquid electrodes

Liquid electrodes may be used and give satisfactory results. The liquid forming the upper electrode should be confined, for example, by stainless steel rings, each of which should have its lower rim reduced to a sharp edge by bevelling on the side away from the liquid. Figure 4, page 12, shows the electrode arrangement. Mercury is not recommended for continuous use or at elevated temperatures due to toxic effects.

7.6 Colloidal graphite

Colloidal graphite dispersed in water or other suitable medium, may be used under the same conditions as given in Sub-clause **7.2**.

7.7 Conducting rubber

Conducting rubber may be used as an electrode material. It has the advantage that it can be applied and removed from the specimen quickly and easily. As the electrodes are applied only during the time of measurement they do not interfere with the conditioning of the specimen. The conducting rubber material shall be soft enough to ensure that effective contact to the specimen is obtained when a reasonable pressure, for example 2 kPa (0.2 N/cm^2), is applied.

7.8 Metal foil

Metal foil may be applied to specimen surfaces as electrodes for volume resistance measurement, but it is not suitable for surface resistance measurement. Lead, antimonial lead, aluminium, and tin foil are in common use. They are usually attached to the specimen by a minimum quantity of petrolatum, silicone grease, oil or other suitable material, as an adhesive. A pharmaceutically obtainable jelly of the following composition is suitable as a conductive adhesive:

Anhydrous polyethylene glycol of molecular mass 600	800 parts by mass
Water	200 parts by mass
Soft soap (pharmaceutical quality)	1 part by mass
Potassium chloride	10 parts by mass

The electrodes shall be applied under a smoothing pressure sufficient to eliminate all wrinkles and to work excess adhesive towards the edge of the foil where it can be wiped off with a cleansing tissue. Rubbing with a soft material such as the finger, has been used successfully. This technique can be used satisfactorily only on specimens that have very smooth surfaces. With care, the adhesive film can be reduced to 0.0025 mm or less.

8 Specimen handling and mounting

It is important that stray currents between the electrodes or between the measuring electrodes and earth do not have a significant effect on the reading of the measuring instrument. Great care shall be used in applying the electrodes, in handling the specimens, and in mounting the specimens for measurement to avoid the possibility of creating stray paths that may adversely affect the result of the measurement.

When surface resistance is to be measured, the surface shall not be cleaned unless agreed or specified. That part of the surface which is to be measured shall not be touched by anything other than an untouched surface of another specimen of the same material.

9 Conditioning

The conditioning that a specimen should receive depends upon the material being tested and should be specified in the material specification.

Recommended conditions are given in IEC Publication 212: Standard Conditions for Use Prior to and during the Testing of Solid Electrical Insulating Materials, and the relative humidities associated with various salt solutions are given in IEC Publication 260: Test Enclosures of Non-injection Type for Constant Relative Humidity. Mechanical vaporization systems may be used.

Both volume resistivity and surface resistivity are particularly sensitive to temperature changes. The change is exponential. It is therefore necessary to measure the volume resistance and surface resistance of the specimen while under specified conditions. Extended periods of conditioning are required to determine the effect of humidity on volume resistivity since the absorption of water into the body of the dielectric is a relatively slow process. Water absorption usually decreases volume resistance. Some specimens may require months to reach equilibrium.

10 Test procedure

A number of specimens as prescribed in the relevant specification are prepared in accordance with Clauses **6**, **7**, **8** and **9**.

The specimen and electrode dimensions, and the width of the surface gap g are measured with an accuracy of ± 1 %. For thin specimens, however, a different accuracy may be stated in the relevant specification, when appropriate.

For the determination of volume resistivity, the average thickness of each specimen is determined in accordance with the relevant specification, the measuring points being distributed uniformly over the area to be covered by the guarded measuring electrode.

NOTE For thin specimens, at least, the thickness should be measured before applying the electrodes.

In general, the resistance measurements should be made at the same humidity (except for conditioning by immersion in a liquid) and temperature as used during conditioning. In some cases however it may be sufficient to make the measurements within a specified time after stopping the conditioning.

10.1 Volume resistance

Before measurement the specimen shall be brought into a dielectrically stable condition. To obtain this, short-circuit the measuring electrodes Nos. 1 and 3 of the specimen, (Figure 1a) through the measuring device and observe the changing short-circuit current, while increasing the sensitivity of the current-measuring device as required. Continue until the short-circuit current attains a fairly constant value, small compared with the expected steady-state value of the current under electrification, or if relevant, the current at 100 min of electrification. As there is a possibility of a change in the direction of the short-circuit current, the short circuit should be maintained even if the current passes zero. The magnitude and direction of the short-circuit current I_0 are noted when it becomes essentially constant, which may require several hours.

Then apply the specified direct voltage and start a timing device simultaneously. Unless otherwise specified, make a measurement after each of the following times of electrification: 1 min, 2 min, 5 min, 10 min, 50 min, 100 min. If two successive measurements give the same results, the test may be terminated, and the value thus found used to calculate the volume resistance. The electrification time until the first of the identical measurements is recorded. If the steady state is not reached within 100 min, the volume resistance is reported as a function of electrification time.

For acceptance tests, the value after a fixed time of electrification, for example 1 min, is used, as specified in the relevant specification.

10.2 Surface resistance

Apply the specified direct voltage, and determine the resistance between the measuring electrodes on the specimen surface (Nos. 1 and 2, Figure 1b). The resistance shall be determined after 1 min of electrification, even though the current has not necessarily reached a steady-state value within this time.

11 Calculation

11.1 Volume resistivity

The volume resistivity shall be calculated from the following formula:

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

where:

- ρ is the volume resistivity in ohms metres (ohms centimetres)
- R_x is the volume resistance in ohms measured as specified in Sub-clause **10.1**
- A is the effective area of the guarded electrode in square metres (square centimetres)
- h is the average thickness of the specimen in metres (centimetres)

Formulae for calculating the effective area A for some particular electrode arrangements are given in Appendix B.

For some materials with high resistivity, the short-circuit current I_0 prior to electrification (see Sub-clause **10.1**) may not be negligible compared with the steady-state current I_s during electrification. In such cases the volume resistance is determined as

$$R_x = U_x/(I_s \pm I_0)$$

where:

- R_x is the volume resistance in ohms
- U_x is the applied voltage in volts
- I_s is the steady-state current in amperes during electrification, or the values of current in amperes after 1 min, 10 min and 100 min if the current changes during electrification
- I_0 is the short-circuit current in amperes prior to electrification

The minus sign is used when I_0 is in the same direction as I_s, otherwise the plus sign is used.

11.2 Surface resistivity

The surface resistivity shall be calculated from the following formula:

$$\sigma = R_x \cdot p/g$$

where:

- σ is the surface resistivity in ohms
- R_x is the surface resistance in ohms measured as specified in Sub-clause **10.2**
- p is the effective perimeter in metres (centimetres) of the guarded electrode for the particular electrode arrangement employed
- g is the distance in metres (centimetres) between the electrodes

11.3 Reproducibility

Because of the variability of the resistance of a given specimen with test conditions, and because of non-uniformity of the material from specimen to specimen, determinations are usually not reproducible to closer than ± 10 % and are often even more widely divergent (a range of values of 10 to 1 may be obtained under apparently identical conditions).

In order that measurements on similar specimens are to be comparable, they must be made with approximately equal voltage gradients.

12 Report

The report shall include at least the following information:

a) description and identification of the material (name, grade, colour, manufacturer, etc.);

b) shape and dimensions of the specimen;

c) type, material and dimensions of the electrodes and guards;

d) conditioning of the specimen (cleaning, pre-drying, conditioning time, humidity and temperature, etc.);

e) test condition (specimen temperature, relative humidity);

f) method of measurement;

g) applied voltage;

h) volume resistivity (when relevant);

NOTE 1 When a fixed electrification time is specified, state this time, give the individual results, and report the central value as the volume resistivity.

NOTE 2 When measurements have been made after different electrification times report as follows:
Where specimens reach a steady state in the same electrification time, give the individual results, and report the central value as the volume resistivity. Where some specimens do not reach the steady state in this electrification time, report the number failing to do so and give the results on them separately. Where results are dependent on electrification time, report this relationship, e.g., in the form of a graph, or as the central value of the volume resistivity after 1 min, 10 min and 100 min.

i) surface resistivity (when relevant):

give the individual values after 1 min of electrification, and report the central value as the surface resistivity.

Appendix A Examples of measuring methods and their accuracy

A.1 Voltmeter-ammeter method

This direct method employs the circuit shown in Figure 5, page 12. The applied voltage is measured by the d.c. voltmeter. The current is measured by a current-measuring device, which may be a galvanometer (now seldom used), an electronic amplifier instrument, or an electrometer.

In general, while the specimen is being charged, the measuring device should be short-circuited to avoid damage to it during this period.

The galvanometer should have high current sensitivity and be provided with a universal shunt (also known as Ayrton shunt). The unknown resistance in ohms is calculated as

$$R_x = U/k\alpha$$

where:

- U is the applied voltage in volts
- k is the sensitivity of the shunted galvanometer in ampere per scale division
- α is the deflection in scale divisions

Resistances up to about 10^{10} to 10^{11} Ω can be measured at 100 V with the required accuracy by means of a galvanometer.

An electronic amplifier instrument or an electrometer with high input resistance shunted by a resistor of known, high resistance R_s, may be used as the current-measuring device. The current is measured in terms of a voltage drop U_s across R_s. The unknown resistance R_x, is calculated as

$$R_x = U \cdot R_s/U_s$$

where:

U is the applied voltage (provided $R_s \ll R_x$)

A number of different resistors R_s may be included in the instrument case, and the instrument is then often graduated directly in amperes or submultiples thereof.

Here also the maximum resistance that can be measured with the required accuracy depends on the properties of the current-measuring device. The error in U_s is determined by the indicator error, the amplifier zero drift and gain stability. In adequately designed amplifiers and electrometers the instability in gain is negligible, and the zero drift can be held so low that it is of no concern in relation to the times involved in these measurements. The indicator error for high gain electronic voltmeters is typically ± 2 % to 5 % of full-scale deflection, and resistors up to 10^{12} Ω with about the same degree of accuracy are feasible. If the voltage-measuring device has an input resistance greater than 10^{14} Ω, and full-scale deflection at an input voltage of 10 mV, a current of 10^{-14} A can be measured with an accuracy of about ± 10 %.

A resistance of 10^{16} Ω can thus be measured at 100 V with the required accuracy by means of a precision resistor with very high resistance and an electronic amplifier voltmeter or electrometer.

A.2 Comparison methods

A.2.1 *Wheatstone bridge method*

The test specimen is connected in one arm of a Wheatstone bridge as shown in Figure 6, page 13. The three known arms shall be of as high a resistance as practicable, limited by the errors inherent in such resistors. Usually, the resistance R_B is changed in decade steps and the resistance R_A is used for fine balance adjustment, and R_N is fixed for the duration of measurement. The detector shall be a d.c. amplifier, with an input resistance high compared with any of these arms. The unknown resistance R_x is calculated as follows:

$$R_x = R_N \cdot R_B/R_A$$

where R_A, R_B and R_N are as shown in Figure 6.

The maximum percentage error in the computed resistance is the sum of the percentage errors in R_A, R_B and R_N, when the null detector has adequate sensitivity. If R_A and R_B are wire-wound resistors with values below, for example, 1 MΩ, their errors can be made negligible, and for measuring very high resistances, R_N could be, for instance, 10^9 Ω, which may be known with an accuracy of ± 2 %. The accuracy with which the ratio R_B/R_A can be determined depends essentially on the sensitivity of the null detector. If the unknown resistance $R_x \gg R_N$, the uncertainty Δr in the determination of the ratio $r = R_B/R_A$ is determined by $\Delta r/r = I_g R_x/U$, where I_g is the minimum perceivable null detector current and U the voltage applied to the bridge. If, for example, an electronic amplifier instrument with input resistance 1 MΩ and full-scale deflection for an input voltage of 10^{-5} is used, the lowest perceivable current will be about $2 \cdot 10^{-13}$ A, corresponding to 2 % of full-scale deflection. With this value of I_g, U = 100 V, and $R_x = 10^{13}$ Ω, $\Delta r/r$ = 0.02 or 2 % is obtained.

Resistances up to 10^{13} to 10^{14} Ω can thus be measured at 100 V with the required accuracy by the Wheatstone bridge method.

A.2.2 ***Ammeter method***

This method employs the circuit shown in Figure 7, page 13, and the components are the same as those described in Clause **A.1** with the addition of a resistor R_N of known value, and a switch to short-circuit the unknown resistance. It is very important that the resistance of this switch in the open position be much higher than the unknown resistance R_x in order not to affect the measurement of the latter. This is most easily obtained by short-circuiting R_x with a copper wire, which is removed when measuring R_x. In general, it is preferable to leave R_N in the circuit at all times in order to limit the current in case of failure of the specimen and thus protect the current-measuring device.

With the switch open, the current through R_x and R_N is determined as specified in Clause **10** by noting the instrument deflection α_x and the shunt ratio F_x, the shunt being adjusted to give as near as possible maximum scale deflection. Thereafter, R_x is short-circuited and the current through R_N determined by noting the instrument deflection α_N and the shunt ratio F_N, the shunt again being adjusted to give as near as possible maximum scale deflection, starting from the least sensitivity. Provided the applied voltage U does not change during the measurement, R_x can be calculated from

$$R_x = R_N[(\alpha_N F_N/\alpha_x F_x) - 1]$$

if $\alpha_N F_N/\alpha_x F_x > 100$, the approximated formula may be used

$$R_x = R_N(\alpha_N F_N/\alpha_x F_x)$$

This method allows R_x to be determined with about the same accuracy as by the direct method described in Clause **A.1**, but has the advantage that the current-measuring device is checked *in situ* by the measurement of R_N, the error of which can be made negligible by using a wire-wound resistor, which is readily obtainable with an accuracy of 0.1 % or better. The measurement of the current through R_x may thus be more reliable.

Appendix B Formulae for calculating *A* and *p*

For most purposes, the following approximate formulae are sufficiently accurate for calculating the effective area A and the effective perimeter p of the guarded electrode.

B.1 The effective area *A*

a) Circular electrodes (Figure 2, page 10)	$A = \pi(d_1 + g)^2/4$
b) Rectangular electrodes	$A = (a + g)\ (b + g)$
c) Square electrodes	$A = (a + g)^2$
d) Tubular electrodes (Figure 3, page 11)	$A = \pi(d_0 - h)\ (l_1 + g)$

where d_0, d_1, g, h and l_1 are the dimensions indicated in Figure 2 and Figure 3, and a and b are the length and width, respectively, of the guarded electrode when rectangular or square. The dimensions are expressed in metres (centimetres).

B.2 The effective perimeter

a) Circular electrodes (Figure 2)	$p = \pi(d_1 + g)$
b) Rectangular electrodes	$p = 2(a + b + 2g)$
c) Square electrodes	$p = 4(a + g)$
d) Tubular electrodes	$p = 2\pi d_0$

where the meaning of the symbols is the same as in Clause **B.1**.

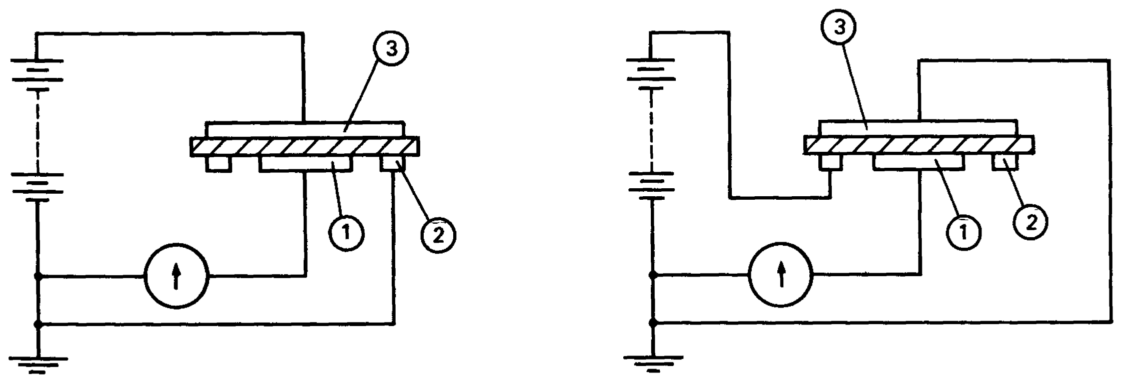

Figure 1a

Figure 1b

Figure 1 — Basic connections for guarded electrodes used for:
a) volume resistivity b) surface resistivity

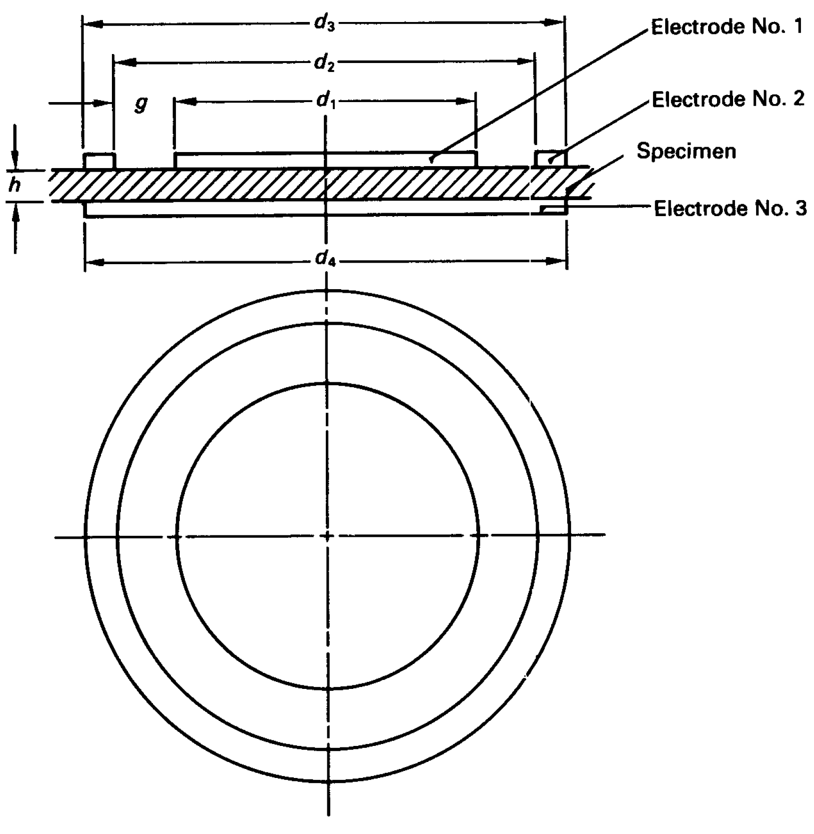

Figure 2 — Example of electrode arrangement on flat specimen

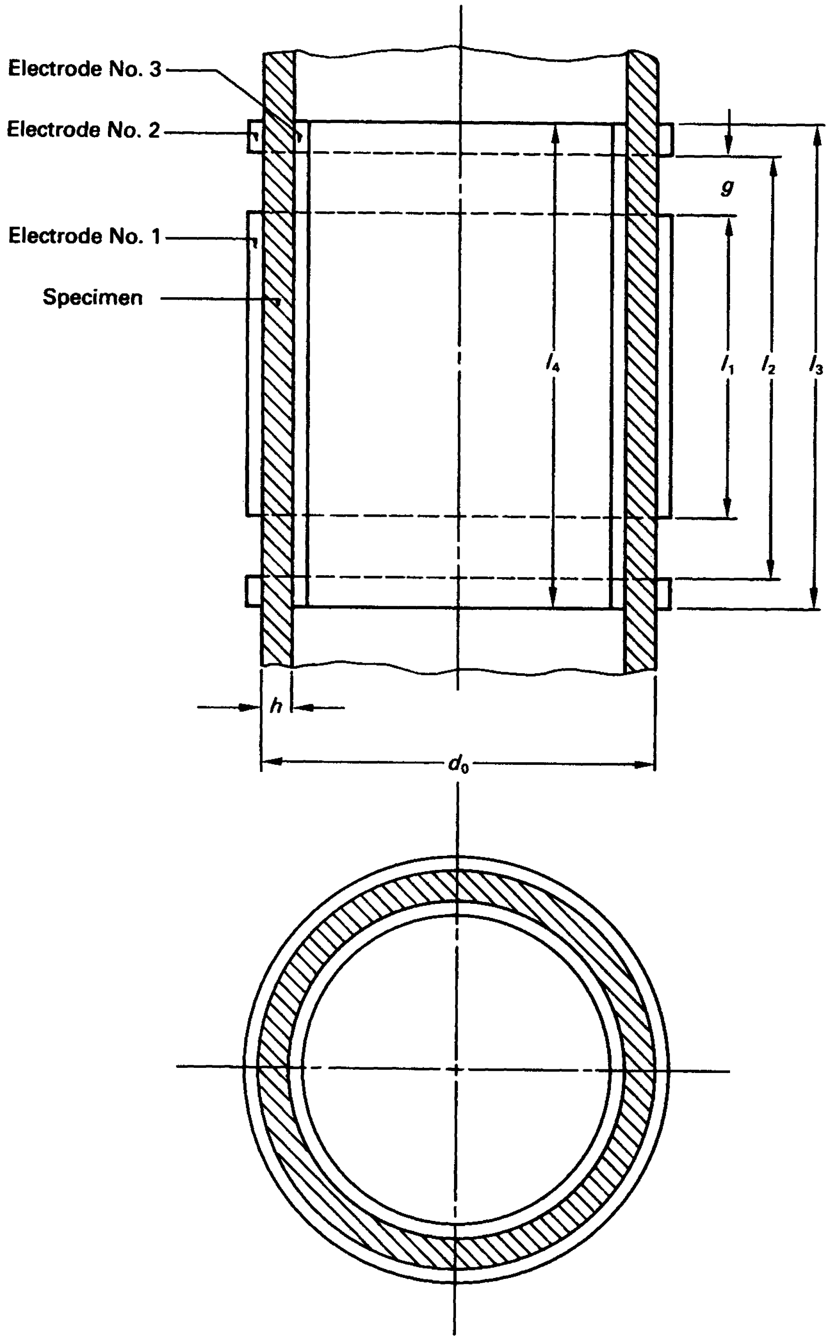

Figure 3 — Example of electrode arrangement on tubular specimen

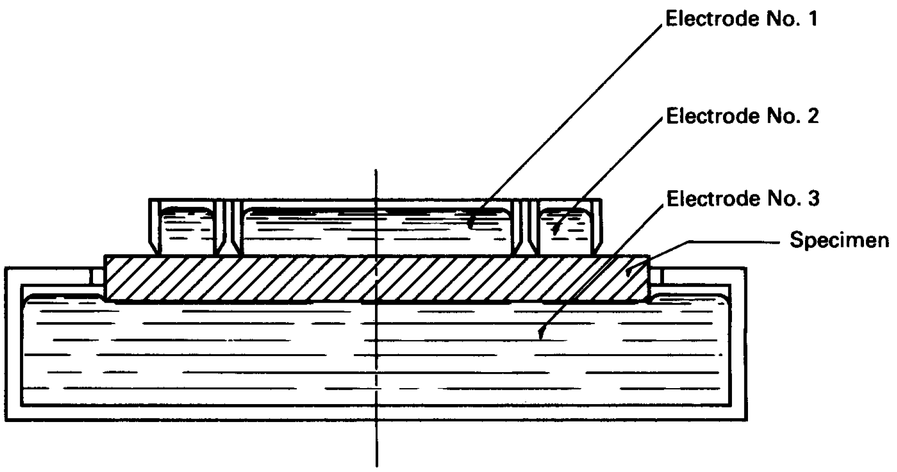

Figure 4 — Arrangement of liquid electrodes

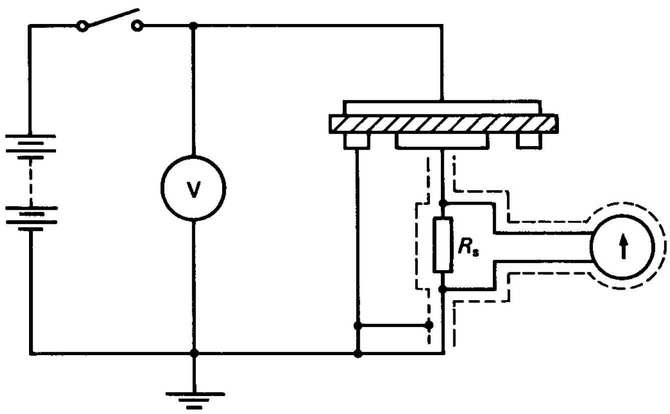

Figure 5 — Voltmeter-ammeter method used for measuring volume resistance. For surface resistance measurements, the connections to the specimen are made as shown in Figure 1b

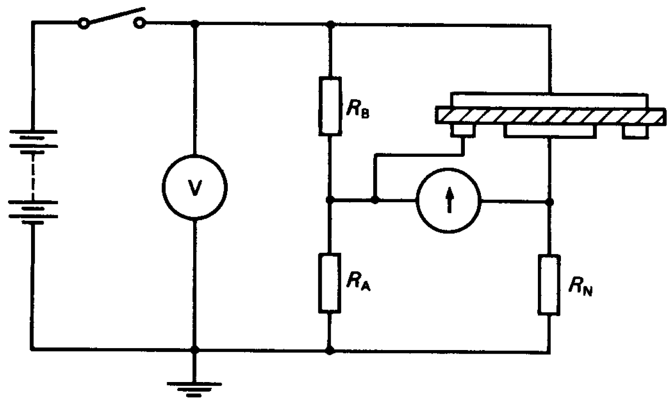

Figure 6 — Wheatstone bridge method used for measuring volume resistance. For surface resistance measurements, the connections to the specimen are made as shown in Figure 1b

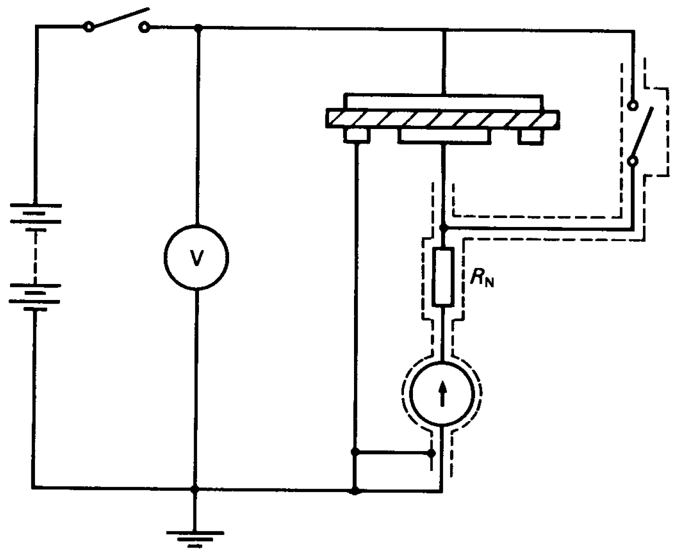

Figure 7 — Ammeter method used for measuring volume resistance. For surface resistance measurements, the connections to the specimen are made as shown in Figure 1b

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