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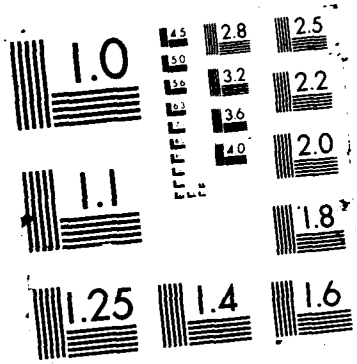
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Historical Overview on Resistivity Measurements

by
J. Covino
Research Department

OCTOBER 1986

NAVAL WEAPONS CENTER
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This report summarizes the theoretical and experimental methods employed in making electrical resistivity measurements of materials. Specific attention has been paid to (1) the theory of the electrical resistivity measurements, (2) instrumentation available for such measurements, (3) electrode design used in resistivity measurements for all types of materials, (4) factors affecting resistivity measurements, and (5) the best method for measuring propellant electrical resistivity.

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CONDUCTOMETRIC METHODS FOR SOLUTIONS

INTRODUCTION

For many solutions, electrical conductance is a descriptive property that lends itself readily and usefully to measurement. Simple electrolyte solutions in polar solvents are particularly well adapted to quantitative analysis by conductometric techniques. The range of applications extends from systems of small conductance and very low ionic concentrations, e.g., AgCl in water at 25°C, to those of high conductance and concentration, e.g., the fused salt mixture KCl-NaCl at 800°C. In this section, the theory of conductance measurements on solutions, as well as experimental setup, will be addressed.

CONDUCTANCE RELATIONSHIPS

Conductance is simply the reciprocal of resistance, a more fundamental property that is a measure of the opposition a substance offers to charge movement. A measured resistance also depends on volume and dimensions. For example, if a sample of regular shape is placed between a pair of parallel electrodes, the resistance measured increases linearly with sample length ℓ and decreases linearly with cross-sectional area A. We remove the dependence on shape and size by defining the specific resistance ρ as the resistance of a unit volume 1 cm^3. In terms of ρ the measured resistance R of a sample is given by the expression $R = \rho\ell/A$. Since R is in ohms, ρ must have units Ω cm. The reciprocal of ρ, the specific conductance κ, is the quantity of interest here. It is defined by the equation

$$\kappa = 1/\rho = \ell/AR \tag{1}$$

and has the units $\Omega^{-1}cm^{-1}$. For measurements on solutions the ratio ℓ/A is fixed by the spacing and size of electrodes in the conductance cell.

In dealing with dissolved electrolytes, it is convenient to define also an equivalent conductance as the conductance associated with one faraday of charge. This is taken as the conductivity of a slab of solution 1 cm thick and of sufficient breadth and length to hold the volume of solution that contains one equivalent of electrolyte. Experimentally, it might be measured by using planar electrodes 1 cm apart and of sufficient surface area just to contain the required volume of solution. The equivalent conductance Λ is related to specific conductance by the formal expression

$$\Lambda = \frac{1000\kappa}{C} \tag{2}$$

where C is the normality of the solution. Since both positive and negative ions will share in carrying the current, we can rewrite Equation 2 in terms of the equivalent ionic conductances λ^+ and λ^-

$$\lambda^- + \lambda^+ = \frac{1000\kappa}{C} \tag{3}$$

It should be noted that only at infinite dilution are the ionic conductances known precisely.

How should we relate conductances to the discussion of mass-transport processes? Conductance is the experimental measure of the transport process called migration. When we apply a potential difference across a pair of electrodes, ions first move to set up electrical double layers at the electrode surface. If the potential is sufficiently large, the oxidation or reduction of electroactive species also begins. As ions are removed by reaction, additional ions move toward the electrodes. Figure 1 shows in simplistic terms the mechanism by which conductance occurs.

Mobility. By definition, the mobility u of an ion is its velocity v under an electric field strength E of 1 V cm^{-1} (E is identical to the electrical gradient in the solution where V volts are applied to plates ℓ centimeters apart). The defining equation is

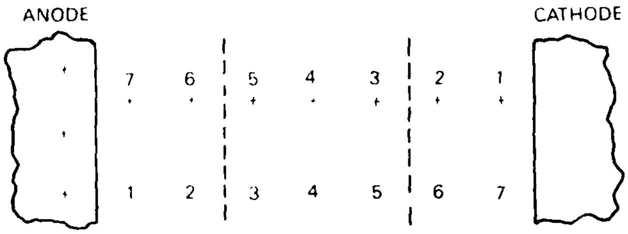
$$v = uE \tag{4}$$

A force $z_i eE$ acts upon each ion, where z_i is the charge on the ion and e the electron charge. As a result of the force, an ion accelerates very rapidly until its motion is just offset by the frictional resistance of the solution. Mobility is a measure of its steady-state motion. So rapidly is a limiting velocity attained by ions that in audio-frequency conductions measurements ions may be assumed to travel at a constant velocity, even though the field is reversing a great many times per second.

The limiting velocity or mobility of an ionic species is determined by the viscosity of the solvent, the solvated size of the ion, the concentration of the solution, and the potential gradient. It has been found that for ions whose radius is about 0.5 nm (5 Å) or greater, Stoke's law describes approximately the relations between the force on the ion F, bulk viscosity η, and mobility u. The statement of the law is

$$u = \frac{F}{6\pi\eta r} \tag{5}$$

where r is the radius of the ion. Equation (5) is of limited validity, but does apply to spherical ions moving in a solvent whose molecules are considerably smaller than the ions.



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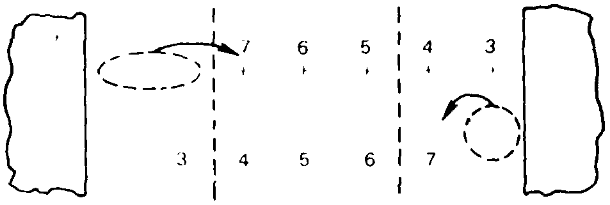


DISTRIBUTION AT TIME t

Figure 1. Transport of Charge by Migration. Electrical double layers at electrode surfaces are omitted. At t = 0, there are equal numbers of positive and negative ions throughout the bulk solution. In time t, two cations and two anions are discharged by electrolysis. If the cations are assumed to have a mobility twice that of the anions, both cations are replaced at the cathode but only one anion is replaced at the anode. Any ionic species not electrolyzed simply tends to accumulate at the electrodes.

Remember that most ions are of the same general size as solvent molecules. They therefore share in the general thermal agitation and have at best a randomly directed type of progress. The instantaneous velocity of any ion in a liquid is of the order of 10^4 cm s^{-1}, but its mean free path is so short that its average velocity toward an electrode is no more than 10^{-3} to $1-^{-4}$ cm s^{-1} when the electrical field strength is of the order of 1 V cm^{-1}.

The current through a unit cube of solution may now be expressed in terms of mobilities. Assume for simplicity that a single electrolyte has completely dissociated. For the positive ions let N_+ be their number per cubic centimeter, u_+ be their mobility, and z_+ be their charge. The related quantities for the negative ions will be denoted by N_-, u_-, and z_-. The total charge arriving at the negative electrode per unit area per second is then $N_+u_+z_+eE$. To convert to cell current, we must include a charge arriving at the positive electrode and multiply by electrode area A. The equation obtained is

$$i = (N_+u_+z_+ = N_-u_-z_-)eEA \tag{6}$$

By use of Ohm's law and Equation 1, we can formulate the specific conductance from Equation 6. We obtain

$$\kappa = (N_+u_+z_+ + N_-u_-z_-)e \tag{7}$$

Concentration Dependence. At very low concentration, ions behave essentially independently. Any given ion moves in a medium where other ions are so distant that they fail to influence its velocity or physical behaviour. But from concentrations of the order of 10^{-6}N upward ions approach each other sufficiently often that interionic forces are important. In addition many kinds of ions begin to associate. Where such a process occurs, conductance decreases proportionally. It is beyond the scope of this book to treat ion association processes. Two interionic effects are of sufficient importance even where there is no association, however, to warrant a brief description.

Definitions

Electrophoretic Effect. In an electrolyte solution, any ion is surrounded by a sheath of solvent molecules, each held with reasonably strong ion-dipole forces. When an ion moves, its solvation sheath tends to accompany it. In this connection remember there is a continuing interchange between "bound" and "free" solvent in the sheath. Since ions of opposite charge move toward different electrodes, a given ion experiences a drag as solvent "bound" to ions of opposite charge moves past. For example, any negative ion moves through solvent that is not stationary but is actually flowing in the opposite direction since it loosely accompanies positive ions.

Relaxation Effect. Another important property of an electrolyte solution that affects ion migration arises from the tendency toward electroneutrality. Any ion may be regarded as surrounded by an atmosphere of ions whose net charge is equal to its charge but opposite in sign. For an ion that is univalent and positive, the essentially spherical atmosphere will include both positive and negative ions but will have an overall charge of -1. The dimensions of an ionic atmosphere can be shown to be inversely proportional to the ionic strength of the solution. At high dilution the radius of the atmosphere is large; in concentrated solutions it may be only a few times the radius of the central ion.

When the ion moves, it tends to leave its atmosphere and a finite time will be required for the thermal and electrical forces to reestablish the randomly arranged atmosphere. Each ion is therefore subjected to a transient restoring force exerted by its old atmosphere as it decays. The opposing force tending to return the central ion to its original location is small at best and because of its time-dependent behavior is termed a relaxation effect. This force also tends to diminish conductance.

According to the general electrostatic theory of electrolytes developed by Fuoss and Onsager (Reference 1), we can represent conductance as a function of concentration C by the following equation

$$\Lambda = \Lambda_0 - SC^{1/2} + EC\log C + JC \tag{8}$$

where Λ_0 is the equivalent conductance at infinite dilution, S is the Onsager coefficient of the limiting conductance law, E is a constant (Both S and E are determined by the absolute temperature, dielectric constant and viscosity of the solvent, valence type of the solute, and universal constants. S also includes Λ_0.), and J is a factor dependent on ion size. Appropriate modifications to Equation 8 can be made to cover association of electrolytes. The equation has been shown to hold generally up to concentrations of about 0.1 N (N = Normal).

ALTERNATING-CURRENT MEASUREMENTS

Because conductance involves the transfer of mass, both solution and electrodes are altered during a measurement. If we impose a DC voltage across a conductance cell there result two immediate, undesirable effects. The electrodes polarize slightly as (a) the solution layer near the electrodes tends to become depleted in the species being oxidized or reduced and (b) the electrode surfaces are altered by the products of electrolysis. The effects are not serious if the current is kept small ($<10^{-7}$ A), but attention must customarily be given to them. If a larger current flows, a DC conductance measurement may well be invalid.

When an AC voltage of audio frequency is applied, the changes described are largely minimized. Because of the frequent reversal of electrolysis, the ionic movement and electrolysis that take place during one half of a cycle can be completely or nearly completely destroyed during the second half of each cycle. Concentrations are maintained essentially constant even though a current exists. The conductance of the solution and the current density at the electrode for a given applied voltage are key variables in arriving at an optimum frequency. If solutions of extremely low conductivity ($\kappa < 10^{-7}$) are being studied or the current density is very low, even DC measurements can be accurate. If the conductance is slightly larger, 60 Hz line current may allow precise measurements. Usually, however, a frequency of about 100 Hz is preferable. Where great precision is required, we find the conductance at several frequencies in the audio range and extrapolate to infinite frequency (Reference 2).

Note that larger conductance values are usually found at radio frequencies (10^5 to 10^7 Hz). The change is a direct consequence of the increased importance of circuit capacitances and inductances. At radio frequencies, the interpretation must thus be broadened to include the bulk capacitance of the cell as well as the resistance of the solution.

A further aid in the elimination of surface polarization effects is the use of platinized platinum electrodes (Reference 2). These are electrodes on which finely divided platinum has been deposited in a thin, adherent layer by electrolysis. As a result of the greatly increased surface area, the reunion of liberated hydrogen and oxygen appears to be catalyzed. The polarization from this source is thus minimized. The large surface area also eliminates concentration polarization.

Electrical Model. A desirable insight into the nature of a conductance measurement is gained by considering an AC circuit that is electrically equivalent to a conductivity cell. Probably the simplest representation is the circuit pictured in bold lines in Fig. 2. The bulk of the solution between the electrodes behaves like an ordinary ohmic resistor and is designated R_3. It is this resistance that is of interest. But at each electrode-solution interface there is an ionic double layer, which must also affect the current. The double layers appear as capacitors of high capacity; without any loss of rigor, these two capacitances may be lumped and labelled C_3. The magnitude of C_3 depends strongly on the amount of platinization of the electrode surface, the extent to which the electrode is polarized, and the time available for the buildup of the ionic layers. For very dilute solutions, that is, those whose concentration is of the order of 10^{-4}M or smaller, this simple representation is quite useful.

The representation can be made more exact and extend it to more concentrated media by including (a) the electrolysis at the metal-solution interfaces, and (b) the cell and lead capacitances. The first appears as an additional resistance R_5 in parallel with the ionic double-layer capacitance C_3. The second may be represented as a capacitance C_5 in parallel with all the other components. Figure 2 shows theses modifications in light lines. Neither R_5 nor C_5 are major factors in influencing circuit behavior, although they cannot be ignored in very precise observations. Since R_5 is usually of the order of from 0.1 to 1 Ω, the measured cell resistance is nearly equal to R_3. Analogously, C_5 is usually no more than from 10 to 100 pF, about 1000 to 10,000 times smaller than C_3. In the next two sections we use the equivalent circuits in considering cell and bridge design.

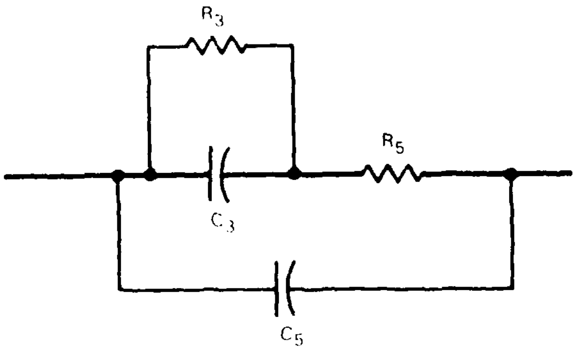


Figure 2. Schematic Representation of an AC Conductance Cell in Terms of its Equivalent Circuit. The simplest equivalent circuit is shown in bold lines. R_3 is the resistance of the bulk solution, C_3 represents the double-layer capacitance at the electrodes, R_5 is the faradaic resistance across the electrode double layer, and C_5 represents the capacitances of the cell electrodes, leads, etc.

CONDUCTANCE CELLS

The usual envelopes for cells with electrodes are made of hard glass. Where ruggedness is required, e.g., in many field and plant applications, other inert, stable dielectrics such as hard rubber and some of the plastics are also in common use. The electrodes are generally square pieces of stiff platinum foil aligned parallel to each other. It is essential that the electrodes be rigidly supported at the desired spacing; by proper design they may be self-supporting like those shown in Fig. 3. If required, the electrodes may be platinized by brief electrolysis in a chloroplatinic acid solution (Reference 3).

Leads. Special attention is always given to the arrangement of the leads to the electrodes, except where an accuracy of from 2 to 5% is adequate. If leads are bare and are brought out close together through the solution, stray electrolytic and capacitive current will pass between them. Accordingly, it is good practice to use insulated lead wires and bring them out of the electrode chamber in opposite directions. Three different designs of conductance cells are illustrated in Fig. 3. Note that all obstructed spaces where mixing will not occur readily have been eliminated in the cells of types (a) and (b).

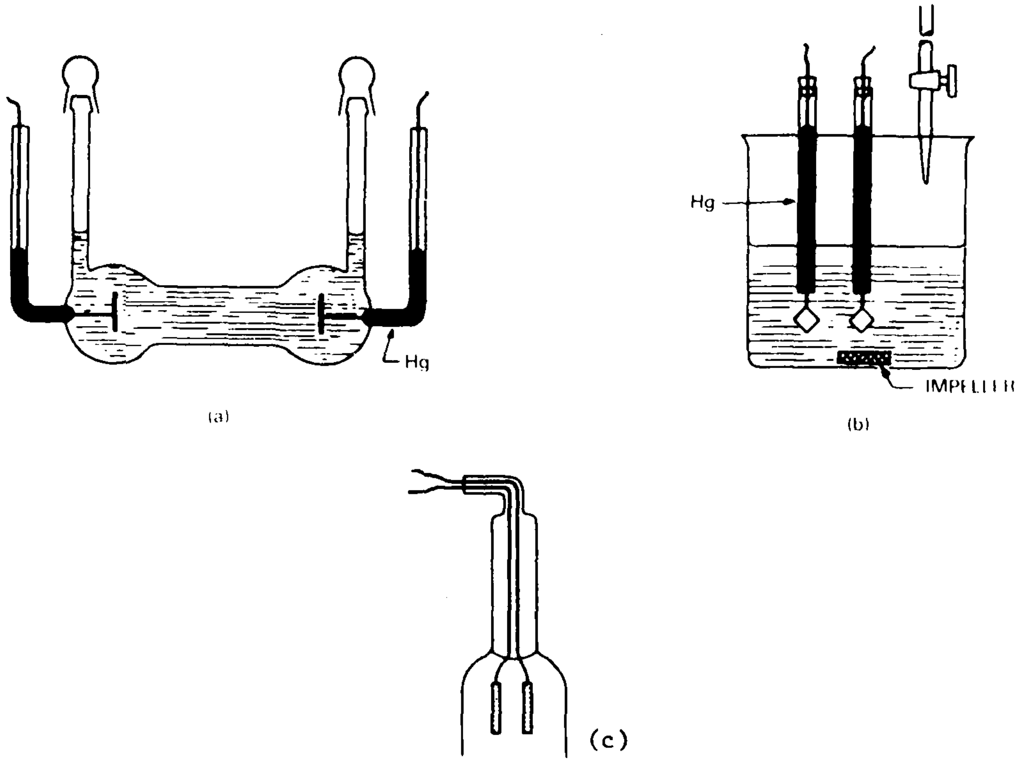


Figure 3. Some Types of Conductance Cells. (a) Jones and Bollinger precision cell, (b) Titration cell, and (c) Dip-type cell.

Cell Constant. The resistance of a solution between the electrodes of a cell is a function not only of solution specific conductance κ but also of the volume of conducting solution between the electrodes. For a pair of parallel electrodes of area A and spacing ℓ, κ may be obtained by rewriting Equation 1 as

$$R = \frac{\ell}{A\kappa} \qquad (9)$$

In practice we determine the ratio ℓ/A, termed the cell constant, for each cell by measuring its resistance when filled with a conductance standard. Solutions of potassium chloride of known concentration are primary standards, their conductances having been accurately determined in cells of known electrode geometry.

For accurate conductance work over a range of concentration it is desirable to use cells of different cell constant. In aqueous work cell constants from about 0.1 to 10 are needed. In nonaqueous media other ranges are called for. The reason is that a bridge of conventional design is capable of greatest accuracy if the cell resistance falls in the range from 1 to 30 kΩ.

Example: A solution of specific conductance of $10^{-5}\Omega^{-1}\text{cm}^{-1}$ has a resistance of 30,000 Ω or below if its cell constant is 0.3 or smaller.

Thermostating. Control of temperature is indispensable if reliable conductance measurements are sought. The specific conductance of electrolytes increases the average about 2% per degree Celsius. To reduce the error from this source to 1% therefore requires regulation to ±0.5°C; to reduce the error to 0.01% requires regulation to ±0.005°C. A constant-temperature bath filled with a light transformer oil is often used to achieve the desired regulation. Water is seldom used as the fluid because of accompanying undesirable capacitance effects between cell and ground.

THE DC AND AC WHEATSTONE BRIDGE

The DC Wheatstone Bridge

This well-known bridge is a simple network for determination of unknown resistances or conductances. The precision inherent in the design of the Wheatstone bridge derives from the fact that, like the potentiometer just discussed, it relies on a comparison procedure. Thus, a measurement is a null determination. In this section the DC operation of the bridge will be taken up.

The basic circuit of a Wheatstone bridge is shown in Fig. 4. Two resistance arms, $R_1 + R_3$ and $R_2 + R_4$, are connected by a shunt BC in which a null detector N (of resistance R_5) is located. Measurements are made by balancing, i.e., varying the resistance of at least one arm, for example R_4, until there is no current in the shunt. In other words, at balance the potential of points B and C must be equal.

To find the relative values of the bridge resistances at balance is a straightforward process. First, voltage V appears across both arms $R_1 + R_3$ and $R_2 + R_4$. Second, points B and C can be equal potential only when the drop across R_1 equals R_4, that is $I_1R_1 = I_2R_2$ and $I_1R_3 = I_2R_4$. Substituting for I_2 in the first equation gives

$$I_1R_1 = R_2(I_1R_3/R_4) \tag{10}$$

Cancelling I_1 and rearranging gives

$$R_1R_2 = R_3/R_4 \tag{11}$$

This equation is the condition of balance.

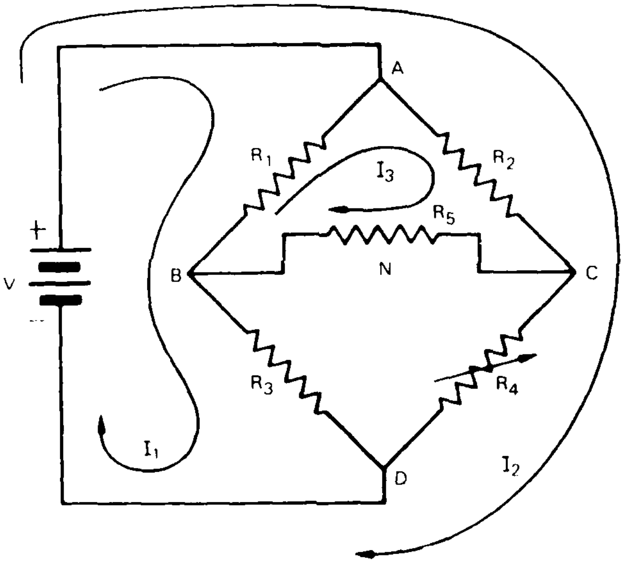


Figure 4. Wheatstone Bridge Circuit. Source V energizes the bridge. The null detector is represented by R_5. Ratio arms are R_1 and R_2. R_3 is an unknown resistance inserted for measurement. Loop currents for analysis of the circuit are also shown.

Design. Because of its wide use, the design of the Wheatstone bridge deserves examination. In any application, values of three of the bridge resistances must be known. (In Fig. 4, R_3 may be taken as unknown.) A much greater range of unknown resistance values can be handled by the bridge if the ratio R_1/R_2 is varied as well as R_4. In most semiprecision designs, fixed ratios of R_1/R_2 from perhaps 0.001 to 1000.0 may be selected by a switch. Such bridges are versatile but seldom accurate to better than ±1%. A current-indicating device is ordinarily used as the null detector, often with some amplification.

What factors must be controlled to ensure high precision, reliability, and sensitivity in a DC Wheatstone bridge? Precision resistors (maximum accuracy about ±0.01%) should be used. They should be designed for high stability and have small temperature coefficients. For best precision, R_1 and R_2 should be identical in value and construction so that they will drift in like fashion with time and temperature and maintain their resistance ratio of unity. The null detector must have sufficient sensitivity for the precision of balance required.

An important advantage of the Wheatstone bridge is that within reasonable limits its balance point is independent of both energizing voltage and resistance in either the power branch or the detector branch. As a result, a possible major source of error, contact resistance in switches and in connections to the unknown resistor can be minimized by arranging that such contacts be in series with the power circuit. In the detector branch the resistance should be whatever value will allow best sensitivity of detection.

Other methods of resistance comparison are possible (Reference 4) and direct types of measurement employing a galvanometer and DC sources are sometimes devised, particularly for resistances of high value (another method was discussed at the end of the last section). Unfortunately, most other procedures require circuits either less accurate or inherently more complicated.

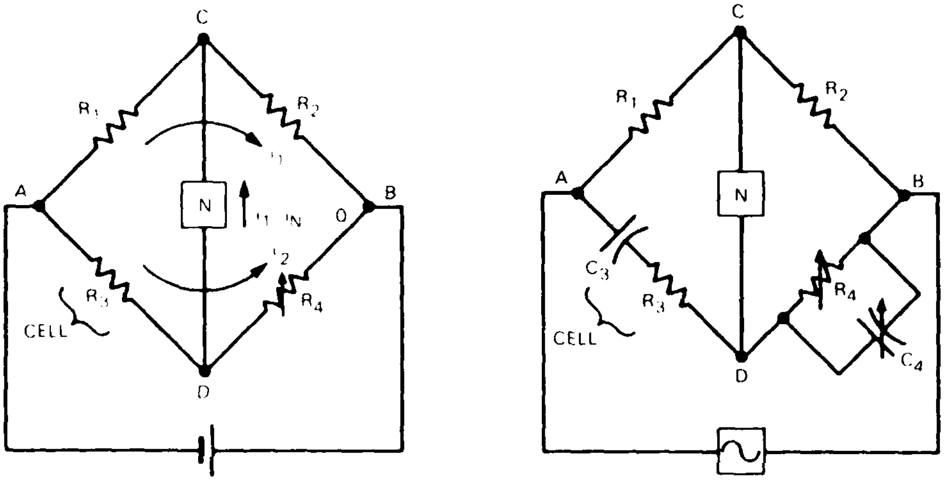
The AC Wheatstone Bridge

This bridge is the basic instrument for determining conductance. In Fig. 5a a schematic circuit of the DC bridge has been shown to permit comparison with the common form of AC Wheatstone bridge shown in Fig. 5b. The condition of balance of the DC bridge is that the potential at points C and D must be equal, yielding the equation

$$\frac{R_1}{R_2} = \frac{R_3}{R_4} \tag{12}$$

Conductance is then obtained by taking the reciprocal of R_3. It is of particular interest that this condition also holds for balance of the AC bridge to within ±0.1%. Some variation is to be expected since the AC bridge is properly an impedance bridge. Sources of error in the AC bridge will be considered below.

Range of Measurement. The range of resistance measurable may be deduced from Equation 12. If $R_1 = R_2$, unknown resistance R_3 can be measured by the bridges shown in Fig. 5 when its value falls within the range $0 < R_3 \leq R_4$. Since this span is short, ways to extend it are important. One method is to vary the ratio R_1/R_2 as well as R_4. Bridges offering several set ratios of R_1/R_2 from 0.001 to 100 are common. Range is traded off for accuracy in these bridges; they are accurate at best to about ±1%.



(a) (b)

Figure 5. Wheatstone Bridge Circuit in Two Versions. In each, R_1 and R_2 are ratio arms and N is a Suitable Null Detector. (a) Simple bridge. (b) Alternating-current conductance bridge. The C_4-R_4 combination provides adequate compensation for both resistance and capacitance of the conductivity cell under most conditions.

Alternatively, range can be extended in conductance measurements by the strategem of use of cells of different cell constant. This approach permits use of equal values for R_1 and R_2, which is necessary to the construction of precision conductance bridges. If R_1 and R_2 have equal values and have been carefully constructed of stable, low-temperature-coefficient alloy, we can assume their resistances will change in like amount with time and temperature and keep the ratio invariant.

Sources of Error. Contact resistance in switches and in leads to the cell, a major potential source of error, can be minimized by keeping every contact possible in series with the power supply (V) or detector. (In any event, there should be sufficient resistance in the power circuit to ensure that bridge resistors dissipate less than the maximum allowable power.)

We must now consider certain sources of error peculiar to the use of AC. The resistors that comprise the bridge arms possess distributed inductance and capacitance, and we must regard the cell itself as equivalent to a model like that in Fig. 2. Second, there is a considerable number of possible stray current paths in an AC bridge. These are of two types. Any part of the bridge has some capacitance with respect to ground and offers a leakage path. Also, by virtue of the inductance of the resistance coils, there exists the possibility of inductive pickup of stray AC currents from power lines or from the oscillator that supplies the bridge power.

The contribution of error from these sources may be reduced considerably by proper resistance, shielding, and physical arrangement (Reference 5). The resistors should be noninductively wound. The bifilar winding, in which the length of wire required to obtain the desired resistance is doubled back on itself and then wound on the form, is widely used to minimize inductance. It is advantageous to have enough residual capacitance so that the capacitive reactance will nearly cancel the inductive reactance at the operating frequency. The cell capacitance C_5 can be compensated by placing a variable capacitor C_4 in the bridge parallel with resistance R_4, as shown in Fig. 5b. Since the problem of eliminating stray leakage paths involves more difficult considerations, it is deferred until the end of this section.

Power Sources. Some industrial and field conductivity instruments operate on 60 Hz AC stepped down from a power line. Much better accuracy is generally secured by operation at audio frequencies in the range of from 500 to 4000 Hz. In this case, an electronic oscillator is usually employed as a generator. The output of the oscillator should ideally be of a single frequency (a pure sine wave) and should be variable in amplitude from zero to several volts. If the harmonic content is minimized, a more precise balance can be obtained, for the problem of phase shifts will be simplified (see below). A variable voltage output allows flexibility of operation.

Phase Relationships. For a true bridge balance, the AC waves must be in phase at points C and D (Fig. 5b). This condition requires either no phase change in either arm or the same phase change in each. Only the latter is a possible solution to the requirement. The capacitance and inductance of the resistors and cell can be minimized but not eliminated.

If accuracies of the order of 1% are satisfactory we may ignore the phase difference, providing the bridge resistors have been wound with reasonable care. For most nonresearch measurements and conductometric titrations, the phase difference can be neglected. On the other hand, work in which the precision must be 0.1% or better calls for a careful examination of the phase dependence of the arms. It is customary to simplify the problem by using matched resistors for the ratio arms R_1 and R_2 so that not only are the resistances equal, but the phase behavior is identical. There remains the question of whether the phase difference introduced by the cell in arm 3 will be equal to that caused by the parallel R_4-C_4 combination. A thorough discussion is beyond the scope of this book, but a limiting case can be considered. In general, if the R_4-C_4 combination introduces a phase shift of less than about 10 minutes of arc, R_4 can be taken as equal to the cell resistance R_3 within 0.1%.

Wagner Ground. Finally, the elimination of stray paths for alternating currents must be treated briefly. The problem is solved by (a) electrostatically shielding the resistance arms by enclosing them in electrically grounded metal covers and (b) incorporating a Wagner ground in the circuit. By use of the Wagner device, points C and D of the bridge are brought to ground potential during balancing. This "grounding" operation allows the detector arm to be at ground potential at balance and virtually eliminates stray pickup at a spot where it would cause the greatest error.

Figure 6 gives a schematic of the Wagner ground. The impedances Z_5 and Z_6 are suitable combinations of variable resistors and capacitors. The balancing of the bridge now has some additional steps. A normal balance is first obtained with the Wagner ground out of the circuit. Then the switch S is thrown to the other position. The circuit is rebalanced using the variable elements in the Wagner ground. Next the switch is returned to the "bridge position," and the bridge circuit is rebalanced. Although a measurement requires more time as a result of the additional steps, the presence of the device greatly refines the precision of a conductance observation.

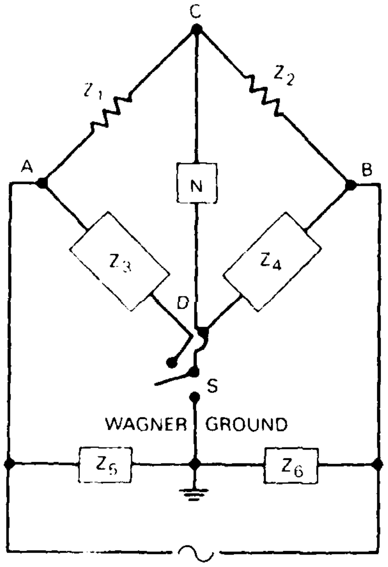


Figure 6. Wagner Ground in an AC Bridge Circuit. It consists of impedances Z_5 and Z_5 and the switch S.

DC RESISTANCE OR CONDUCTANCE OF INSULATING MATERIALS

In this section direct-current measurements for the determination of DC insulation resistance, volume resistance, volume resistivity, surface resistance, and surface resistivity or corresponding conductances and conductivities of insulating materials are addressed.

SUMMARY OF METHODS

The resistance or conductance of a material specimen or of a capacitor is determined from a measurement of current or of voltage drop under specified conditions. By using the appropriate electrode systems, surface and volume resistance or conductance may be measured separately. The resistivity or conductivity can then be calculated when the required specimen and electrode dimensions are known. It should be noted that surface resistance changes rapidly with humidity, while volume resistance changes slowly.

Resistivity or conductivity may be used to predict, indirectly, the low-frequency dielectric breakdown and dissipation factor properties of some materials. Resistivity or conductivity is often used as an indirect measure of moisture content, degree of cure, mechanical continuity, and deterioration of various types. The usefulness of these indirect measurements is dependent on the degree of correlation established by supporting theoretical or experimental investigations. A decrease of surface resistance may result in either an increase of the dielectric breakdown voltage because the electric field intensity is reduced, or a decrease of the dielectric breakdown voltage because the amount of stress they are under is increased.

All the dielectric resistances of conductances depend on the length of time of electrification and on the value of applied voltage (in addition to the usual environment variables). These must be known to make the measured value of resistance or conductance meaningful.

Surface resistance or conductance cannot be measured accurately. They can only be approximated. This is due to the fact that the measured value is largely a property of the contamination that happens to be on the specimen at the time. However, the permittivity of the specimens influences the deposition of contaminants and its surface characteristics affect the conductance of the contaminants. Surface resistivity or conductivity can be considered to be related to material properties when contamination is involved but is not a material property in the usual sense.

ELECTRODE SYSTEMS

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 (Reference 6). The electrode material should be corrosion-resistant under the conditions of test.

Binding-Post and Taper-Pin Electrodes, Figures 7 and 8, provide a means of applying voltage to rigid insulating materials to permit an evaluation of their resistive or conductive properties. Resistance or conductance values obtained are highly influenced by the individual contact between each pin and the dielectric material, the surface roughness of the pins, and the smoothness of the hole in the dielectric material. Reproducibility of results on different specimens is difficult to obtain.

Metal Bars in the arrangement of Fig. 9 were primarily devised to evaluate the insulation resistance or conductance of flexible tapes and thin, solid specimens as a fairly simple and convenient means of electrical quality control. This arrangement is somewhat more satisfactory for obtaining approximate values of surface resistance or conductance when the width of the insulating material is much greater than its thickness.

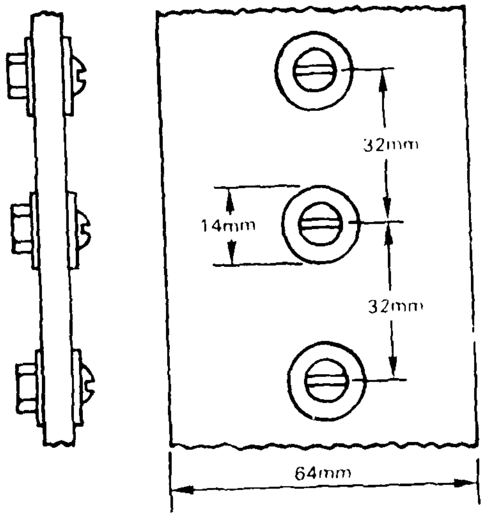
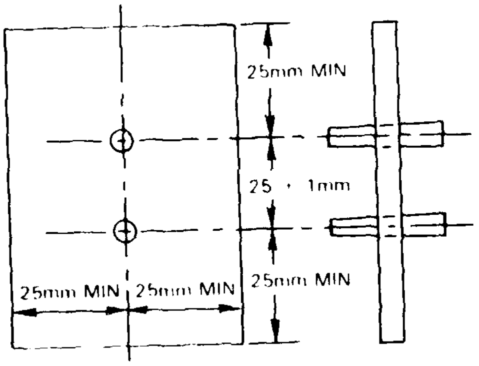
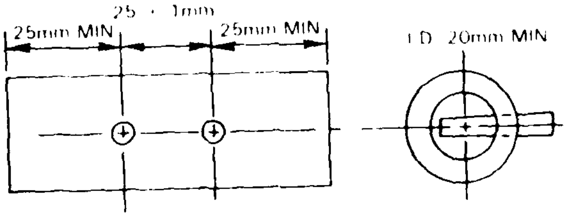


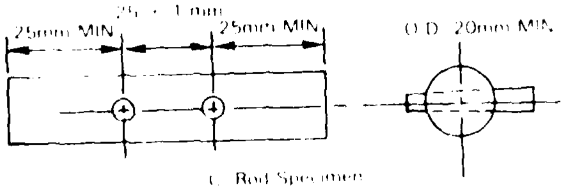
Figure 7. Binding-Post Electrodes for Flat, Solid Specimens.



A Plate Specimen



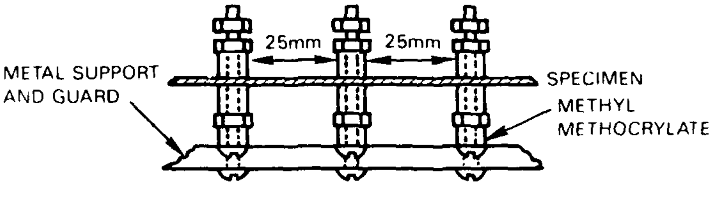
B Tube Specimen



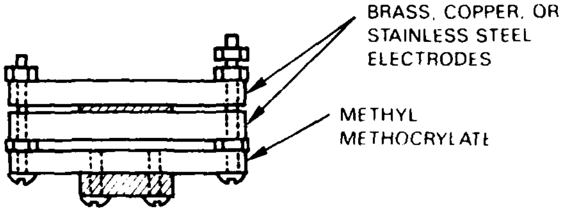
C Rod Specimen

USE PRATT & WHITNEY NO. 3 TAPER PINS

Figure 8. Taper-Pin Electrodes.



SIDE VIEW



END VIEW

Figure 9. Strip Electrodes for Tapes and Flat, Solid Specimens.

Silver Paint, Figures 10, 11, and 12, is available commercially with a high conductivity, either air-drying or low-temperature-baking varieties, which are sufficiently porous to permit diffusion of moisture through them and thereby allow the test specimen to be conditioned after the application of the electrodes. This is a particularly useful feature in studying resistance-humidity effects, as well as change with temperature. However, before conductive paint is used as an electrode material, it should be established that the solvent in the paint does not attach the material so as to change its electrical properties.

Sprayed Metal, Figures 10, 11, and 12, 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. Narrow strips of masking tape or clamp-on masks must be used to produce a gap between the guarded and the guard electrodes. The tape shall be such as not to contaminate the gap surface.

Evaporated Metal may be used under the same conditions given above.

Metal Foil, Figure 10, may be applied to specimen surfaces as electrodes. The usual thickness of metal foil used for resistance of conductance studies of dielectrics ranges from 6 to 80 µm. Lead or tin foil is in most common use, and is usually attached to the test specimen by a minimum quantity of petrolatum, silicone grease, oil, or other suitable material, as an adhesive.

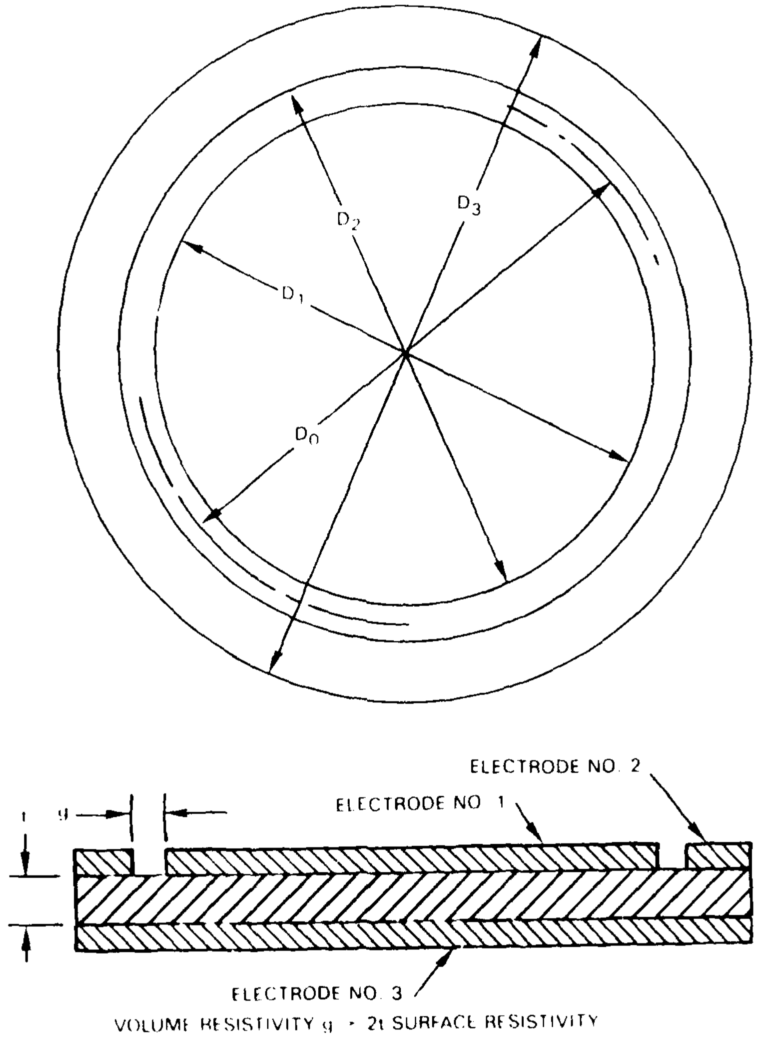


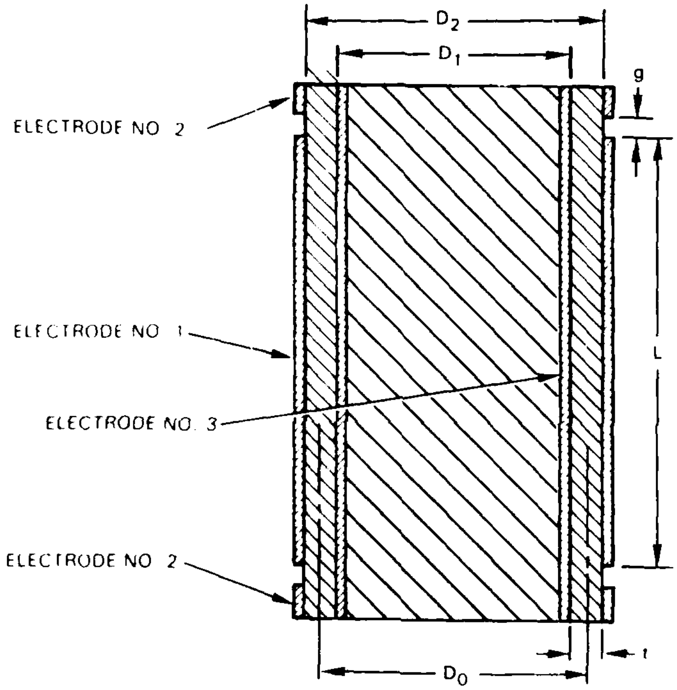
Figure 10. Flat Specimen for Measuring Volume and Surface Resistances or Conductances.

Colloidal Graphite, Fig. 10, dispersed in water or other suitable vehicle, may be brushed on nonporous, sheet insulating materials to form an air-drying electrode. Masking tapes or clamp-on masks may be used. This electrode material is recommended only if all of the following conditions are met:

1. The material to be tested must accept a graphite coating that will not flake before testing,

2. The material being tested must not absorb water readily, and

3. Conditioning must be in a dry atmosphere (Procedure B, Methods D 618), and measurements made in this same atmosphere.

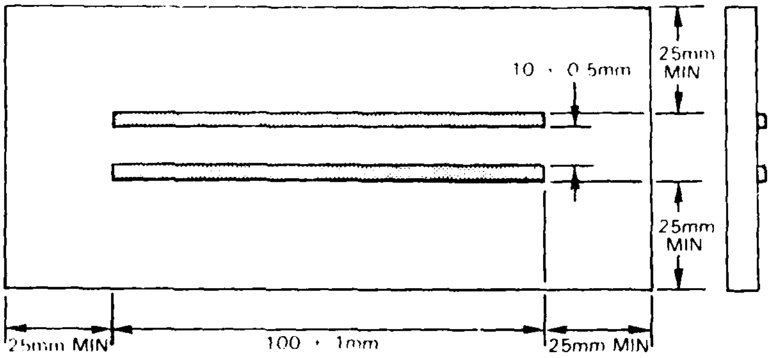


$D_0 = (D_1 + D_2)/2$ $L > 4t$ $g \leq 2t$ VOLUME RESISTIVITY $g > 2t$ SURFACE RESISTIVITY

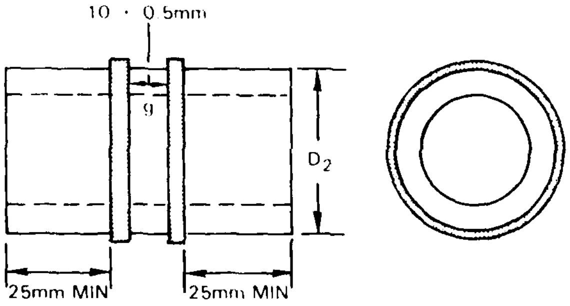
Figure 11. Tubular Specimen for Measuring Volume and Surface Resistances or Conductances.

Mercury or other liquid metal electrodes give satisfactory results. Mercury is not recommended for continuous use or at elevated temperatures due to toxic effects. The metal forming the upper electrodes should be confined by stainless steel rings, each of which should have its lower rim reduced to a sharp edge by beveling on the side away from the liquid metal. Figure 13 shows two electrode arrangements.

Flat Metal Plates, Fig. 10, (preferably guarded) may be used for testing flexible and compressible materials, both at room temperature and at elevated temperatures. They may be circular or rectangular (for tapes). To ensure intimate contact with the specimen, considerable pressure is usually required. Pressures of 140 to 700 kPa have been found satisfactory (see material specifications).



A Plate Specimen



B Tube or Rod Specimen

Figure 12. Conducting-Paint Electrodes.

Conducting Rubber, has been used as electrode material, as in Fig. 10, and has the advantage that it can quickly and easily be applied and removed from the specimen. As the electrodes are applied only during the time of measurement, they do not interfere with the conditioning of the specimen. The conductive-rubber material must be backed by proper plates and be soft enough so that effective contact with the specimen is obtained when a reasonable pressure is applied.

Water is widely employed as one electrode in testing insulation on wires and cables. Both ends of the specimen must be out of the water and of such length that leakage along the insulation is negligible. Guard rings may be necessary at each end. It may be desirable to add a small amount of sodium chloride to the water to ensure high conductivity. Measurements may be performed at temperatures up to about 100°C.

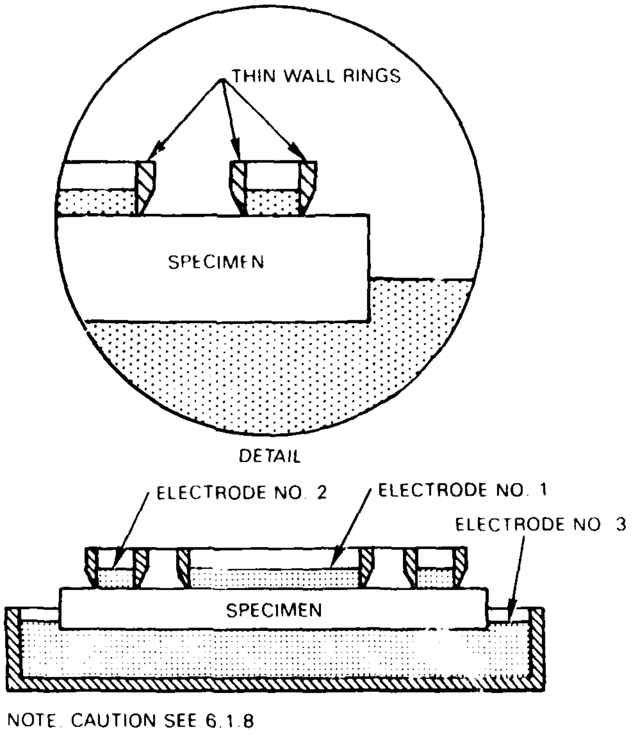
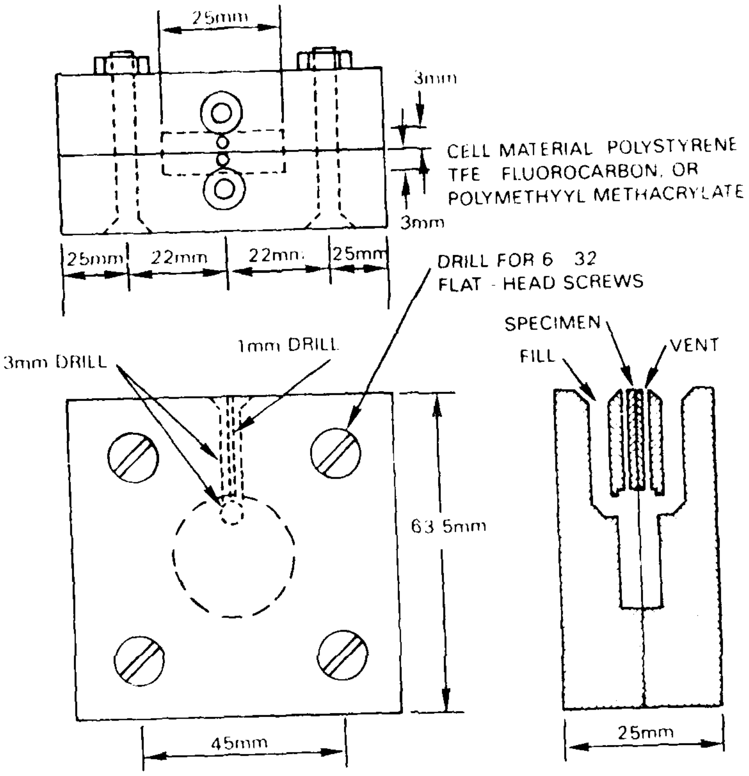


Figure 13a. Mercury Electrodes for Flat, Solid Specimens.



NOTE CAUTION SEE 6 1.8

Figure 13b. Mercury Cell for Thin Sheet Material.

CHOICE OF APPARATUS AND METHOD

Power Supply

A source of very steady direct voltage is required. Batteries or other stable direct voltage supplies may be used.

Direct Measurements

The current through a specimen at a fixed voltage may be measured using any equipment that has the required sensitivity and accuracy (±10% is usually adequate). Current-measuring devices available include electrometers, DC amplifiers with indicating meters, and galvanometers. When the measuring device scale is calibrated to read ohms directly no calculations are required.

Comparison Methods

A Wheatstone-bridge circuit may be used to compare the resistance of the specimen with that of a standard resistor. This was discussed earlier (pp. 11-17).

Direct Measurements:

Galvanometer-Voltmeter. The maximum percentage error in the measurement of resistance by the galvanometer-voltmeter method is the sum of the percentage errors of galvanometer indication, galvanometer readability, and voltmeter indication. As an example, a galvanometer having a sensitivity of 500 pA/scale division will be deflected 25 divisions with 500 V applied to a resistance of 40 GΩ (conductance of 25 pS). If the deflection can be read to the nearest 0.5 division, and the calibration error (including Ayrton Shunt error) is ±2% of the observed value, the resultant galvanometer error will not exceed ±4%. If the voltmeter has an error of ±2% of full scale, this resistance can be measured with a maximum error of ±6% when the voltmeter reads full scale, and ±10% when it reads one-third full scale. The desirability of readings near full scale is readily apparent.

Voltmeter-Ammeter. The maximum percentage error in the computed value is the sum of the percentage errors in the voltages, V_z and V_s, and the resistance, R_s. The errors in V_s and R_s are generally dependent more on the characteristics of the apparatus used than on the particular method. The most significant factors that determine the errors in V_s are indicator errors, amplifier zero drift, and amplifier gain stability. With modern, well-designed amplifiers or electrometers, gain stability is usually not a matter of concern. With existing techniques, the zero drift of direct voltage amplifiers or electrometers cannot be eliminated but it can be made slow enough to be relatively insignificant for these measurements. The zero drift is virtually nonexistent for carefully designed converter-type amplifiers. Consequently, the null method of Fig. 14 is theoretically less subject to error than those methods employing an indicating instrument, provided, however, that the potentiometer voltage is accurately known. The error in R_s is to some extent dependent on the amplifier sensitivity. For measurement of a given current, the higher the amplifier sensitivity, the greater likelihood that lower valued, highly precise wire-wound standard resistors can be used. Such amplifiers can be obtained. Standard resistances of 100 GΩ known to ±2%, are available. If 10-mV input to the amplifier or electrometer gives full-scale deflection with an error not greater than 2% of full scale, with 500 V applied, a resistance of 5000 TΩ can be measured with a maximum error of 6% when the voltmeter reads full scale, and 10% when it reads 1/3 scale.

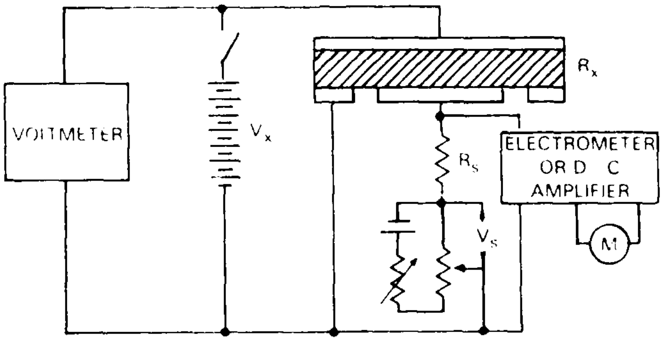


Figure 14. Voltmeter-Ammeter Method Using DC Amplification. Amplifier and indicating meter as null detector.

Comparison - Galvanometer. The maximum percentage error in the computed resistance or conductance is given by the sum of the percentage errors in R_s, the galvanometer deflections or amplifier readings, and the assumption that the current sensitivities are independent of the deflections. The latter assumption is correct to well within ±2% over the useful range (above 1/10 full-scale deflection) of a good, modern galvanometer (probably 1/3 scale deflection for a DC current amplifier having a sensitivity of 10 nA for full-scale deflection, 500 V applied to a resistance of 5 TΩ will produce a 1% deflection. At this voltage, with the above noted standard resistor, and with $F_s = 10^6$, d_s would be about half of full-scale deflection, with a readability error not more than ±1%. If d_s is approximately 1/4 of full-scale deflection, the readability error would not exceed ±4%, and a resistance of the order of 200 GΩ could be measured with a maximum error of ±5-1/2%.

Voltage Rate-of-Change. The accuracy of the measurement is directly proportional to the accuracy of the measurement of applied voltage and time rate of change of the electrometer reading. The length of time that the electrometer switch is open and the scale used should be such that the time can be measured accurately and a full-scale reading obtained. Under these conditions, the accuracy will be comparable with that of the other methods of measuring current.

Comparison Bridge. When the detector has adequate sensitivity, the maximum percentage error in the computer resistance is the sum of the percentage errors in the arms, A, B, and N. With a detector sensitivity of 1 mV/scale division, 500 V applied to the bridge, and R_N = 1 GΩ known to within ±2% and with the bridge balanced to one detector-scale division, a resistance of 100 TΩ can be measured with a maximum error of ±6%.

TEST SPECIMENS

A variety of electrode configurations have been designed in order to measure electrical properties for different sample configurations. These electrode configurations can be obtained from ASTMD257 (Reference 7).

A METHOD OF MEASURING THE RESISTIVITY ON LAMELLAE OF ARBITRARY SHAPE

L. J. Van der Pauw (Reference 8) developed a method of measuring the resistivity on a thin section of material of arbitrary shape. Detailed accounts of this method and theoretical proof of how the method works can be found in References 8 and 9. In summary, the method is based upon a theorem which holds for a flat sample of arbitrary shape if the contacts are sufficiently small and located at the circumference of the sample. Furthermore, the sample must be singly connected, i.e., it should not have isolated holes, pores, electrical discontinuities, or be heterogeneous in composition.

FACTORS AFFECTING INSULATION RESISTANCE OR CONDUCTANCE MEASUREMENTS

In this section factors affecting electrical measurements of insulating materials will be summarized.

INHERENT VARIATION IN MATERIALS

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

TEMPERATURE

The resistance of electrical insulating materials is known to change with temperature, and the variation often can be represented by a function of the form

$$R = Be^{m/T} \tag{13}$$

where R = resistance (or resistivity of an insulating material or system,
B = proportionality constant,
m = activation constant, and
T = absolute temperature in kelvin (K).

This equation is a simplified form of the Arrhenius equation relating the activation energy of a chemical reaction to the absolute temperature; and the Boltzmann principle, a general law dealing with the statistical distribution of energy among large numbers of minute particles subject to thermal agitation. The activation constant, m, has a value that is characteristic of a particular energy absorption process. Several such processes may exist within the material, each with a different effective temperature range, so that several values of m would be needed to fully characterize the material. These values of m can be determined experimentally by plotting the natural logarithm of resistance against the reciprocal of the absolute temperature. The desired values of m are obtained from such a plot by measuring the slopes of the straight-line sections of the plot. This derives from Equation 14, for it follows that by taking the natural logarithm of both sides:

$$\ell n\ R = \ell n\ B + m\ 1/t \tag{14}$$

The change in resistance (or resistivity) corresponding to a change in absolute temperature from T_1 to T_2, based on Equation 17, and expressed in logarithmic form, is

$$\ell n\ (R_2/R_1) = m\ \left(\frac{1}{T_2} - \frac{1}{T_1}\right) = m\ \left(\frac{\Delta T}{T_1 T_2}\right) \tag{15}$$

These equations are valid over a temperature range only if the material does not undergo a transition within this temperature range. Extrapolations are seldom safe since transitions are seldom obvious or predictable. As a corollary, deviation of a plot of the logarithm of R against 1/T from a straight line is evidence that a transition is occurring. Furthermore, in making comparisons between materials, it is essential that measurements be made over the entire range of interest for all materials.

It should be noted that: (1) The resistance of an electrical insulating material may be affected by the time of temperature exposure. Therefore, equivalent temperature conditioning periods are essentially for comparative measurements. (2) If the insulating material shows signs of deterioration after conditioning at elevated temperature, condition periods are essential for comparative measurements.

TEMPERATURE AND HUMIDITY

The insulation resistance of solid dielectric materials decreases both with the temperature and with increasing humidity. Volume resistance is particularly sensitive to temperature changes, while surface resistance changes widely and very rapidly with humidity changes. In both cases the change is exponential. For some materials a change from 25 to 100°C may change insulation resistance or conductance by a factor of 100,000, often due to the combined effects of temperature and moisture content change; the effect of temperature change alone is usually much smaller. A change from 25 to 90% relative humidity may change insulation resistance or conductance by as much as a factor of 1,000,000 or more. Insulation resistance or conductance is a function of both the volume and surface resistance or conductance of the specimen, and surface resistance changes almost instantaneously with change of relative humidity. It is, therefore, absolutely essential to maintain both temperature and relative humidity within close limits during the conditioning period and to make the insulation resistance or conductance measurements in the specified conditioning environment. Another point not to be overlooked is that at relative humidities above 90%, surface condensation may result from inadvertent fluctuations in humidity or temperature produced by the conditioning system. This problem can be avoided by the use of equivalent absolute humidity at a slightly higher temperature, as equilibrium moisture content remains nearly the same for a small temperature change. In determining the effect of humidity on volume resistance or conductance, extended periods of conditioning are required, since the absorption of water into the body of the dielectric is a relatively slow process. Some specimens require months to come to equilibrium. When such long periods of conditioning are prohibitive, use of thinner specimens or comparative measurements near equilibrium may be reasonable alternatives, but the details must be included in the test report.

TIME OF ELECTRIFICATION

Measurements of a dielectric material are not fundamentally different from that of a conductor except that an additional parameter, time of electrification (and in some cases the voltage gradient) is involved. The relationship between the applied voltage and the current is involved in both cases. For dielectric materials, the standard resistance placed in series with the unknown resistance must have a relatively low value, so that essentially full voltage will be applied across the unknown resistance. When a potential difference is applied to a specimen, the current through it generally decreases asymptotically toward a limiting value which may be less than 0.01 of the current observed at the end of 1 min. This decrease of current with time is due to dielectric absorption (interfacial polarization, volume charge, etc.) and the sweep of mobile ions to the electrodes. In general, the relation of current and time is of the form $\ell(t) = At^{-m}$, after the initial charge is completed and until the true leakage current becomes a significant factor. In this relation A is a constant, numerically the current at unit time, and m usually, but not always, has a value between 0 and 1. Depending upon the characteristics of the specimen material, the time required for the current to decrease to within 1% of this minimum value may be from a few seconds to many hours. Thus, in order to ensure that measurements on a given material will be comparable, it is necessary to specify the time of electrification. The conventional arbitrary time of electrification has been 1 min. For some materials, misleading conclusions may be drawn from the test results obtained at this arbitrary time. A resistance-time or conductance-time curve should be obtained under the conditions of test for a given material as a basis for selection of a suitable time of electrification, which must be specified in the test method for the material, or such curves should be used for comparative purposes. Occasionally, a material will be found for which the current increases with time. In this case either the time curves must be used or a special study undertaken, and arbitrary decisions made as to the time of electrification.

MAGNITUDE OF VOLTAGE

Both volume and surface resistance or conductance of a specimen may be voltage-sensitive. In that case, it is necessary that the same voltage gradient be used if measurements on similar specimens are to be comparable. Also, the applied voltage should be within at least 5% of the specified voltage. This is a separate requirement from that given in a later section, which discusses voltage regulation and stability where appreciable specimen capacitance is involved.

Commonly specified test voltages to be applied to the complete specimen are 100, 250, 500, 1000, 2500, 5000, 10,000, and 15,000 V. Of these, the most frequently used are 100 and 500 V. The higher voltages are used either to study the voltage-resistance or voltage-conductance characteristics of materials (to make tests at or near the operating voltage gradients), or to increase the sensitivity of measurement.

Specimen resistance or conductance of some materials may, depending upon the moisture content, be affected by the polarity of the applied voltage. This effect, caused by electrolysis or ionic migration, or both, particularly in the presence of nonuniform fields, may be particularly noticeable in insulation configurations such as those found in cables where the test-voltage gradient is greater at the inner conductor that at the outer surface. Where electrolysis or ionic migration does exit in specimens, the electrical resistance will be lower when the smaller test electrode is made negative with respect to the larger. In such cases, the polarity of the applied voltage shall be specified according to the requirements of the specimen under test.

CONTOUR OF SPECIMEN

The measured value of the insulation resistance or conductance of a specimen results from the composite effect of its volume and surface resistance or conductances. Since the relative values of the components vary from material to material, comparison of different materials, by the use of the electrode systems Figs. 7, 8, and 9, is generally inconclusive. There is no assurance that, if material A has a higher insulation resistance than material B as measured by the use of one of these electrode systems, it will also have a higher resistance than B in the application for which it is intended.

It is possible to devise specimen and electrode configurations suitable for the separate evaluation of the volume resistance or conductance and the approximate surface resistance or conductance of the same specimen. In general, this requires at least three electrodes so arranged that one may select electrode pairs for which the resistance or conductance measured is primarily that of either a volume current path or a surface current path, not both.

DEFICIENCIES IN THE MEASURING CIRCUIT

The insulation resistance of many solid dielectric specimens is extremely high at standard laboratory conditions, approaching or exceeding the maximum measurable limits given in Table 1. Unless extreme care is taken with the insulation of the measuring circuit, the values obtained are more a measure of apparatus limitations than of the material itself. Thus errors in the measurement of the specimen may arise from undue shunting of the specimen, reference resistors, or the current-measuring device, by leakage resistances or conductances of unknown, and possibly variable, magnitude.

Table 1. Apparatus and Conditions for Use.

Method	Maximum Ohms Detectable at 500 V
Voltmeter-ammeter (galvanometer)	10^{12}
Comparison (galvanometer)	10^{12}
Voltmeter-ammeter (DC amplification, electrometer)	10^{15}
	10^{15}
	10^{17}
	10^{17}
Comparison (Wheatstone bridge)	10^{15}
Voltage rate-of-change	$\approx 100\ \mathrm{M\Omega \cdot F}$
Megohmmeter (typical)	10^{15}

Electrolytic, contact, or thermal electromotive forces (emfs) may exist in the measuring circuit itself; or spurious emfs may be caused by leakage from external sources. Thermal emfs are normally insignificant except in the low resistance circuit of a galvanometer and shunt. When thermal emfs are present, random drifts in the galvanometer zero occur. Slow drifts due to air currents may be troublesome. Electrolytic emfs are usually associated with moist specimens and dissimilar metals, but emfs of 20 mV or more can be obtained in the guard circuit of a high-resistance detector when pieces of the same metal are in contact with moist specimens. If a voltage is applied between the guard and the guarded electrodes a polarization emf may remain after the voltage is removed. True contact emfs can be detected only with an electrometer and are not a source of error. The term "spurious emf" is sometimes applied to electrolytic emfs. To ensure the absence of spurious emfs of whatever origin, the deflection of the detecting device should be observed before the application of voltage to the specimen and after the voltage has been removed. If the two deflections are the same, or nearly the same, a correction can be made to the measured resistance or conductance, provided the correction is small. If the deflections differ widely, or approach the deflection of the measurement, it will be necessary to find and eliminate the source of the spurious emf. Capacitance changes in the connecting shielded cables can cause serious difficulties.

Where appreciable specimen capacitance is involved, both the regulation and transient stability of the applied voltage should be such that resistance or conductance measurements can be made to prescribed accuracy. Short-time transients, as well as long-time drifts in the applied voltage, may cause spurious capacitive charge and discharge currents which can significantly affect the accuracy of measurement. In the case of current-measuring methods particularly, this can be a serious problem. The current in the measuring instrument due to a voltage transient is $I_0 = C_s dV/dt$. The amplitude and rate of pointer excursions depend upon the following factors:

1. The capacitance of the specimen.

2. The magnitude of the current being measured.

3. The magnitude and duration of the incoming voltage transient, and its rate of change.

4. The ability of the stabilizing circuit used to provide a constant voltage with incoming transients of various characteristics.

5. The time-constant of the complete test circuit as compared to the period and damping of the current-measuring instrument.

Changes of range of a current-measuring instrument may introduce a current transient. When $R_m < R_s$ and $C_m < C_s$, the equation of this transient is

$$I = (V_0/R_s)\,[1 - e^{-t/R_sC_m}] \tag{16}$$

where V_0 = applied voltage
R_s = apparent resistance of the specimen
R_m = effective input resistance of the measuring instrument
C_s = capacitance of the specimen at 1000 Hz
C_m = input capacitance of the measuring instrument
t = time after R_m is switched into the circuit

For not more than 5% error due to this transient,

$$R_mC_s \geq t/3 \tag{17}$$

Microammeters employing feedback are usually free of this source of error as the actual input resistance is divided, effectively, by the amount of feedback, usually at least by 1000.

RESIDUAL CHARGE

It was pointed out that the current continues for a long time after the application of a potential difference to the electrodes. Conversely, current will continue for a long time after the electrodes of a charged specimen are connected together. It should be established that the test specimen is completely discharged before attempting the first measurement, a repeat measurement, a measurement of volume resistance following a measurement of surface resistance, or a measurement with reversed voltage. The time of discharge before making a measurement should be at least four times any previous charging time. The specimen electrodes should be connected together until the measurement is to be made to prevent any buildup of charge from the surroundings.

GUARDING

Guarding depends on interposing, in all critical insulated paths, 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.

Proper use of the guard system for the methods involving current measurement is illustrated in Figs. 14-17, inclusively, where the guard system is shown connected to the junction of the voltage source and current-measuring instrument or standard resistor. In Fig. 18 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 must* be complete, and must include any controls operated by the observer in making the measurement. The guard system is generally maintained at a potential close to that of the guarded terminal, but insulated from it. This is because, among other things, the resistance of many insulating materials is voltage-dependent. Otherwise, the direct resistance or conductances of a three-terminal network are independent of the electrode potentials. It is usual to ground the guard system and hence one side of the voltage source and current-measuring device. This places both terminals of the specimen above ground. Sometimes, one terminal of the specimen is permanently grounded. The current-measuring device usually is then connected to this terminal, requiring that the voltage source be well insulated from ground.

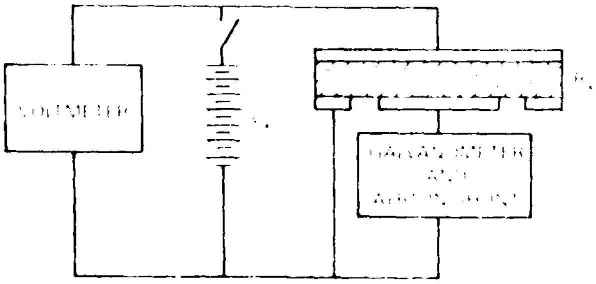


Figure 15. Voltmeter-Ammeter Method Using a Galvanometer.

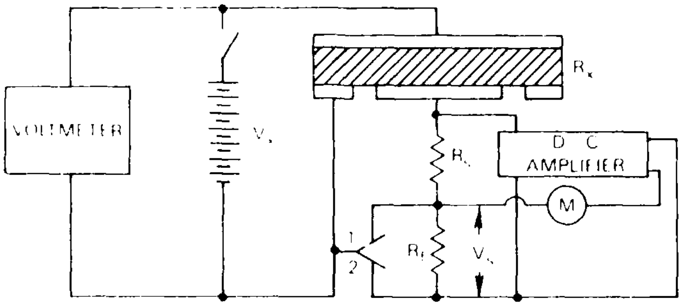


Figure 16. Voltmeter-Ammeter Method Using DC Amplification. Normal use of amplifier and indicating meter.

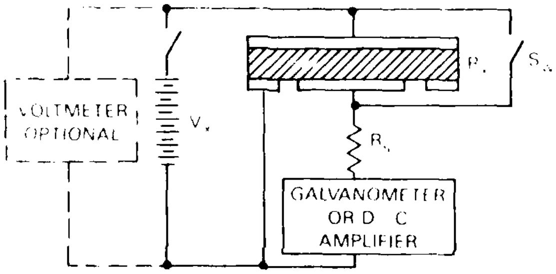


Figure 17. Comparison Method Using a Galvanometer.

Errors in current measurements may result from the fact that the current-measuring device is shunted by the resistance or conductance between the guarded terminal and the guard system. This resistance should be at least 10 to 100 times the input resistance of the current measuring device. In some bridge techniques, the guard and measuring terminals are brought to nearly the same potentials, but a standard resistor in the bridge is shunted between the unguarded terminal and the guard system. This resistance should be at least 1000 times that of the reference resistor.

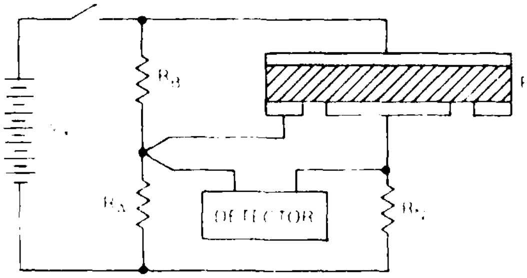


Figure 18. Comparison Method Using a Wheatstone Bridge.

RESISTIVITY MEASUREMENT ON PROPELLANT SAMPLES

After detailed evaluation of the theory and experimental procedures for electrical resistivity measurement the constant voltage, direct-current method (Reference 7) was used for propellants. This method gives the resistance as a function of voltage and information about the ohmic or nonohmic behavior of conduction. The resistance of a sample is determined from the measurement of the DC current which was caused by a known voltage gradient across the sample.

The ideal way to measure the resistance of most materials is to apply a known potential to the sample and measure the resulting current with an electrometer picoammeter. This permits the sample to be measured with different applied potentials to determine any dependency of the resistance on voltage.

When measuring extremely rigid samples such as glass, epoxy, and ceramics, an interface between the stainless steel electrodes of the fixture and the sample surface is required. This enhances the surface contact between the two. Conductive rubber may be used for such an application. Care must be taken since the electrode area becomes the area of the contact medium. The guarding plays an important role when measuring resistance. It minimizes errors due to surface resistivity while making volume resistivity measurements, and vice versa. Since low currents are involved, electrostatic interference can be significant. This problem is avoided by proper sample shielding.

The determination of surface and volume resistivities consists of measuring surface and volume resistances followed by calculations of the corresponding resistivities with the use of known sample and electrode dimensions. The volume resistivity (ρ_V) is defined as the ratio of potential gradient parallel to the current in the material to the current density in units of ohm-cm (References 7 and 10). The surface resistivity (ρ_S) is defined as the ratio of potential gradient parallel to the current along a surface to the current per unit width of the surface in units of ohms (References 7 and 10).

Figure 19 shows electrode configuration to measure surface resistance. The measurement is performed by applying a set voltage on the surface of the sample and obtaining a current reading. The following equation is used to calculate the surface resistivity:

$\rho_S = K_S * (V/I)$
V = voltage (volts)
I = current reading (amperes)
K_S = a geometrical factor arising from electrode geometry (unitless)

The geometric factor is an effective perimeter of the guarded electrode divided by the gap between the guarded electrode and the guard.

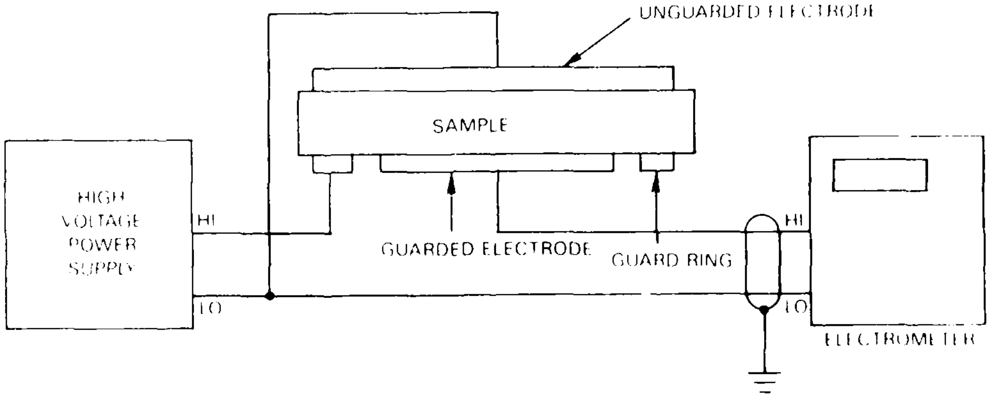


Figure 19. Surface Resistivity Electrical Diagram.

Figure 20 shows electrode configuration to measure volume resistance. The measurement is performed by applying a set voltage through the sample and obtaining a current reading after a set time interval of one minute. The following equation is used to calculate volume resistivity:

$\rho_V = K_V * (V/I)$
v = voltage (volts)
I = current reading (amperes)
K_V = a geometric factor arising from electrode geometry (cm)

The geometric factor is an effective area of measuring electrodes divided by the sample thickness.

In order to calculate the geometric factors, K_S and K_V, specific electrode dimensions and sample thickness are needed. Figure 21 illustrates the electrode geometry used at NWC. To calculate the surface geometric factor, K_S, the following equation is used:

$$K_S = P/g = (\pi D_0)/g \tag{18}$$

where P = the effective perimeter of the guarded electrode for the particular arrangement used (cm)
g = gap (cm)

To calculate the volume geometric factor, K_v, the following equation is used:

$$K_v = A/t \tag{19}$$

$$A = [\pi(D_1 + g)^2]/4 \tag{20}$$

where A = the effective area of the measuring electrode for the particular arrangement used (cm^2)
t = the average thickness of the sample

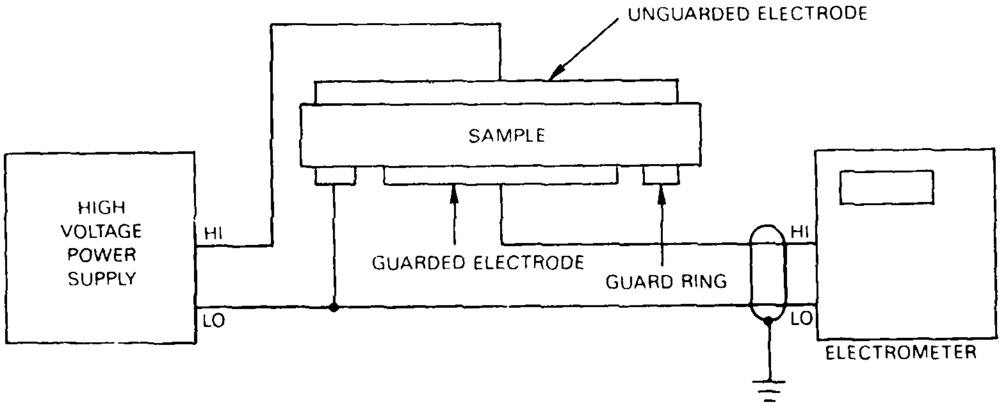


Figure 20. Volume Resistivity Electrical Diagram.

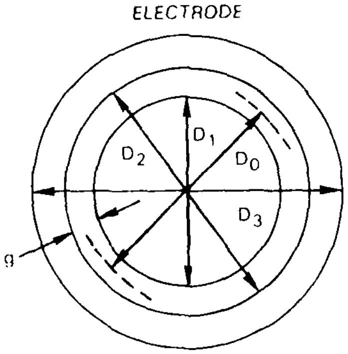


Figure 21. Electrode Configurations and Parameters Needed for the Geometric Calculations for Surface and Volume Resistivity.

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