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# Uncertainty Evaluation Problems in Measurements of Volume and Surface Resistivities

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## ABSTRACT

The problems associated with evaluation of uncertainty of volume and surface resistivities were presented in the paper. Special attention was paid to the measurements carried out on extremely high resistivity materials. Different factors affecting the uncertainty were classified and estimated. A simplified method of uncertainty evaluation was proposed.

**Keywords:** dielectrics, volume and surface resistivities, measurements uncertainty.

## 1. INTRODUCTION

There are a lot of factors strongly influencing the results of resistivity measurements on dielectric materials. One can divide them into two groups:
- factors associated with the investigated object,
- metrological factors, associated with the applied method and measuring equipment.

A sample of dielectric with relatively simple geometry, equipped with particular electrodes, is generally used as the investigated object. Simple geometry enables to simplify calculation of resistivity from the results of resistance measurements. Factors, associated with the investigated object, can be classified into three groups:
- factors influencing properties of the material in the sample volume, e.g. material structure, pressure, (pressure exerted by electrodes), temperature, humidity, type and intensity of irradiation, electric field influence (non-linear effects), sample thickness (thin layer effect) and other factors influencing the near-electrode regions, such as type of electrodes, the technology of their deposition, the state of surface before and after deposition, electrode phenomena e.g. mechanisms determining charge carriers transport through the electrode–dielectric interface, space charge storage and potential barrier building in near electrode regions, and others.
- Factors associated with appearance of transient processes like high relaxation time polarisation, space charge time evolution and other processes ( ageing phenomena, electro-cleaning), which are generally closely related to the value of applied electric field value as well as its duration time.

Among the factors of metrological nature one should mention the type of applied measuring method and measuring equipment, noises, disturbances, parasitic and leakage currents [1,2,3] as well as measuring and ambient conditions (voltage, read-out time, temperature, humidity, atmosphere). The mentioned factors are the basic sources of errors in the resistivity measurements.

Application of a particular measuring procedure allows to decrease the influence of the mentioned factors on the measurement results, however the total elimination of their affect is impossible. The influence of non-eliminated components should be taken into consideration by the proper estimation of the measurement uncertainty factors

## 2. RESISTIVITY MEASUREMENTS

Methods of volume and surface resistivity measurements were described in standards: international - IEC 93 [4] and polish - PN-88/E-04405 [5]. The measurements should be carried out on the plate-parallel or cylindrical samples. For the flat samples a concentric three-electrode system was recommended, as it was shown in the Fig. 1.

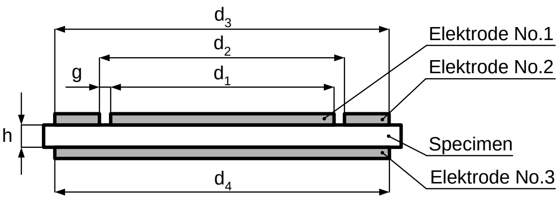


Fig. 1. Three-electrode system recommended for the resistivity measurements on flat dielectric samples.

The electrodes should be made of well conducting material enabling good contact with the sample surface, e.g. vacuum evaporated or sputtered metal electrodes, conducting lacquers, colloidal graphite, conducting rubber and others.

Measurements of high resistances can be carried out using different methods [4,5,6]. The most frequently used is the modified indirect method of resistance measurements [6,7,8]. A scheme of the method was shown in the Fig. 2. Grounding of the Electrode No. 2 (see Fig. 1.) in the circuit for the measurements of volume resistance (Fig. 2a) allows to eliminate influence of the surface current on the results of $I_v$ measurements. Similarly, in the circuit for the measurement of the surface resistance (Fig. 2b.) grounding of Electrode No. 1 (see Fig. 1.) allows to eliminate influence of the volume current on the results of $I_s$ measurements. All connections should be made in a manner enabling to avoid shunting of measured resistance by appropriate insulation resistances. Shielding of the „current"cable, leading to a picoammer, should be kept on the ground potential. In such conditions the insulation resistance of the „current" cable $R_{iA}$ shunts only a relatively small input resistance of the picoammer, not changing its effective input resistance (Fig. 2.b). Similarly, grounding of shielding of the „voltage" cable introduces shunting of the voltage source by an additional loading caused by an insulation resistance $R_{iz}$)

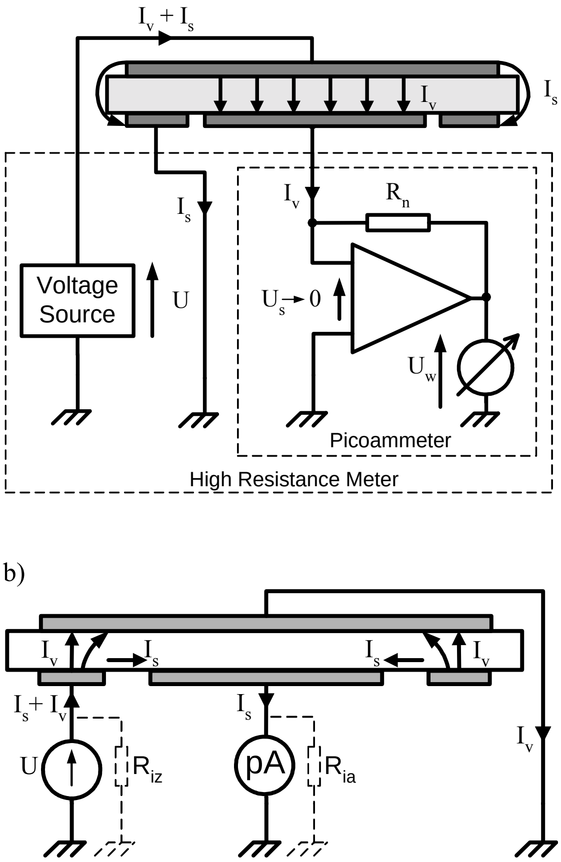


Fig.2. Circuitry for the measurements of resistance of dielectric samples: a) volume resistance, b) surface resistance.

In case of measurements of volume resistance $R_v$ (as well as surface resistance $R_s$) the current flowing through the sample under the voltage $U$, decreases usually in time to the steady state value $I_{vu}$ (Fig. 3.).

For high resistivity dielectrics the time of achieving of the steady-state value $I_{vu}$, for the current $I_v(t)$ may be of the order of minutes, hours or even years (for PTFE). The surface current $I_s(t)$ reaches its steady-state value usually earlier.

According to the standards IEC 93 and PN-88/E-04405 the surface resistivity should be measured after 1 min. For materials with a volume resistivity above $10^{10}$ Ωm the volume current may not reach its steady-state value in 1min. In such situation the volume resistivity should be read-out after 1, 2, 5, 10, 20, 50, 100 min. since the switching on the polarising voltage $U$. Thus because of the transient phenomena, results of resistivity measurements should always be followed by the information about the read-out time.

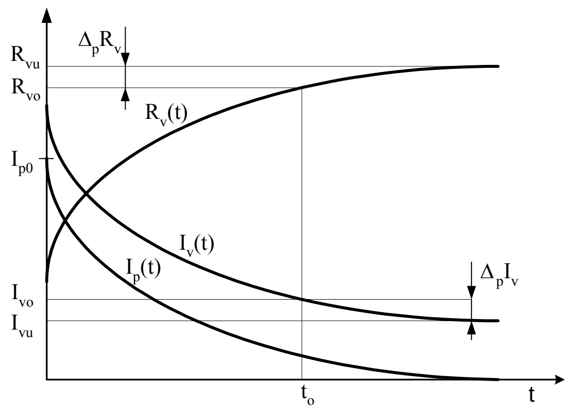


Fig. 3. Time dependencies of the volume current and volume resistance for the sample of dielectric.

For the materials with the highest resistivities (e.g. PTFE) one can expect that the steady state may be reached after time much longer than 100 min. In such situations one can apply a method proposed by Badian [1,9] In the method it is suggested that the steady-state current value $I_{vu}$ can be calculated from the difference between absorption and desorption currents measured for the same read-out time. Experiments confirmed that the estimated current value is only comparable with the steady-state one. Volume and surface resistivities, respectively $\rho_v$ and $\rho_s$, are calculated from the expressions:

$$\rho_v = \frac{\pi (d_1 + g)^2}{4h} R_v , \qquad (1)$$

$$\rho_s = \frac{\pi (d_1 + g)}{g} R_s , \qquad (2)$$

where: $R_v$ – volume resistance, $R_s$ – surface resistance , $d_1$ – measuring electrode diameter (see Fig.1.), $g$ – electrodes gap width, $h$ – sample thickness.

## 3. EVALUATION OF MEASUREMENTS UNCERTAINTY

The „Guide to expression of uncertainty in measurement" [10] proposes to calculate standard uncertainties A and B, and next, on the base of the mentioned values the combined uncertainty. According to the guide, expanded uncertainty should be calculated by multiplication of the combined standard uncertainty by an appropriate coverage factor $k$, depending on the confidence level. For the most frequently used value of the confidence level p=0,95, the coverage factor $k$=2.

The guide [10] recommends to eliminate all systematic errors from the measurement results, including errors introduced by applied measuring devices. According to the mentioned recommendations the measuring apparatus should be standardised for the indications on the level of measured values and the systematic error eliminated by introducing of the appropriate correction. The mentioned calculations of uncertainty are very time-consuming. In case of high precision measurements such procedure is justified, however in case of investigations of dielectric materials it seems to be irrational The acceptable uncertainty for the measurements of volume and surface resistivities is on the level of 10 ÷ 20 % and sometimes more. Thus the measurement uncertainty is weakly affected by inaccuracy of applied measuring devices. As it comes from experiments, the factors mentioned in section 1 belong to those that most seriously affect the uncertainty. That is why it was suggested to simplify calculation of the uncertainty and for the measurements of volume and surface resistivity on the confidence level $p$=0,95 to use the expression:

$$\Delta\rho = \sqrt{\sum_{i=1}^{n}\left(\frac{\partial\rho}{\partial x_i}\Delta x_i\right)^2}\ , \qquad (3)$$

where: $x_i$ – components of the resistivity function $\rho=f(x_i)$, $\Delta x_i$ – uncertainties of components of the function on the confidence level $p$=0,95.

The uncertainties calculated from the equation (3) exhibit a little higher value then the calculated according to the procedure given in a guide [10]. This overvaluation is not a problem and creates a natural protection against uncertainty underestimation by not considering all factors affecting the particular measurement uncertainty. A-type uncertainties are visible during repeated measurements of the same value. The standard uncertainty A-type, equal to the standard deviation of the measured quantity $x$, one can calculate from the expression:

$$\Delta_{sA}\overline{x} = \sqrt{\frac{\sum_{i=1}^{n}(x_i-\overline{x})^2}{n(n-1)}}\ , \qquad (4)$$

where: $x_i$ – measured value of the quantity $x$, $\overline{x}$ – its arithmetic average value, $n$ – number of measurements. The expanded uncertainty A-type of the average value $\overline{x}$ on the confidence level $p$=0,95 is equal:

$$\Delta x = 2\Delta_{sA}\overline{x}\ . \qquad (5)$$

The combined expanded uncertainty can be calculated from the expression:

$$\Delta x = \sqrt{(\Delta_A x)^2 + (\Delta_B x)^2}\ . \qquad (6)$$

## 4. UNCERTAINTY OF THE MEASUREMENTS OF VOLUME RESISTIVITY

The absolute uncertainty of the measured volume resistivity, for the plane-parallel samples, on the confidence level $p$=0,95 can be calculated - according to equation (3) - from the expression:

$$\Delta\rho_v = \sqrt{\left(\frac{\delta\rho_v}{\delta d_1}\cdot\Delta d_1\right)^2 + \left(\frac{\partial\rho_v}{\partial g}\cdot\Delta g\right)^2 + \left(\frac{\partial\rho_v}{\partial h}\cdot\Delta h\right)^2 + \left(\frac{\partial\rho_v}{\partial R_x}\cdot\Delta R_v\right)^2}\ , \qquad (7)$$

Particular partial derivatives in equation (7) one can compute by differentiation of the expression (1). Finally, for the absolute value of uncertainty, one can obtain:

$$\Delta\rho_v = \frac{\pi(d_1+g)}{2h}\sqrt{R_v^{\,2}\left[(\Delta d_1)^2 + (\Delta g)^2 + \left(\frac{(d_1+g)}{2h}\cdot\Delta h\right)^2\right] + \left(\frac{(d_1+g)}{2}\cdot\Delta R_v\right)^2} \qquad (8)$$

The relative uncertainty for the volume resistivity measurements can be calculated from the following expression:

$$\delta\rho_v = \frac{\Delta\rho_v}{\rho_v} = \sqrt{\left(\frac{2\Delta d_1}{d_1+g}\right)^2 + \left(\frac{2\Delta g}{d_1+g}\right)^2 + \left(\frac{\Delta h}{h}\right)^2 + \left(\frac{\Delta R_v}{R_v}\right)^2} \times 100\ \ [\%]\ . \qquad (9)$$

## 5. UNCERTAINTY OF THE MEASUREMENTS OF SURFACE RESISTIVITY

The absolute uncertainty of the measurements of surface resistivity on the confidence level $p$=95 one can calculate from the expression:

$$\Delta\rho_s = \pm\sqrt{\left(\frac{\partial\rho_s}{\partial d_1}\Delta d_1\right)^2 + \left(\frac{\partial\rho_s}{\partial g}\Delta g\right)^2 + \left(\frac{\partial\rho_s}{\partial R_s}\Delta R_s\right)^2}\ , \qquad (10)$$

Where particular partial derivatives can be computed by differentiation of the expression (2). Finally, for the absolute value of uncertainty, one can get:

$$\Delta\rho_s = \pm\frac{\pi}{g}\sqrt{(R_s\Delta d_1)^2 + \left(-\frac{d_1}{g}\Delta g R_s\right)^2 + [(d_1+g)\Delta R_s]^2}\ . \qquad (11)$$

The relative uncertainty for the surface resistivity measurements on the confidence level $p$=0,95 can be calculated from the following expression:

$$\delta\rho_s = \frac{\Delta\rho_s}{\rho_s} = \pm\sqrt{\left(\frac{\Delta d_1}{d_1+g}\right)^2 + \left(-\frac{d_1\cdot\Delta g}{g(d_1+g)}\right)^2 + \left(\frac{\Delta R_s}{R_s}\right)^2} \times 100\ [\%] \quad . \tag{12}$$

## 6. UNCERTAINTY OF THE MEASUREMENTS OF THE SAMPLE DIMENTIONS

Measurements of geometrical dimensions are usually carried out repeatedly and in different parts of the sample. Slide callipers, micrometer screws, microscopes and other high precision instruments are usually used for such measurements. The average value is assumed as the measurement result. Uncertainty of the diameter measurements $\Delta x$ is the combined uncertainty, comprising A-type expanded uncertainty, originating from scattering of results of repeatable measurements $\Delta_A x$ and B-type uncertainty $\Delta_B x$, associated with the applied measuring instrument, and equals its inaccuracy. The expanded combined uncertainty $\Delta x$ for the measurement of the dimension $x$ may be calculated from the expression (6).

## 7. UNCERTAINTY OF THE RESISTANCE MEASUREMENTS

The total uncertainty of the measurements of the resistance $R$ ($R_v$, $R_s$) is determined by the uncertainty of the high resistance meter as well as by uncertainties associated with appearance of the components mentioned in the section 1. Influence of the last component is difficult to estimate. The resistance is generally calculated from the ratio of a polarising voltage value. $U$ and appropriate current value $I$ ($I_v$, $I_s$) So, the relative uncertainty for the resistance measurement will be as follows:

$$\delta R = \sqrt{(\delta U)^2 + (\delta I)^2} \ . \tag{13}$$

In case of measurements on high resistivity materials special attention should be paid to the influences associated with space charge and transient processes appearing in such materials. As it comes from experiments, the electric field values, determined by e.g. space charge, can be much higher then those created by the polarising voltage.

The influence of space charge can be modelled [1,2] by an additional source with a voltage $U_Z$, depending on the value of charge stored in the sample. In the first approximation the equivalent electret voltage (voltage created on the sample capacitance by the effective surface charge) can be assumed as the voltage $U_Z$ [12]. The appropriate investigations confirmed that the equivalent voltages could be higher than 100 V (for samples with thickness in the range of 10-100 micrometers [13]) or even of the order of 1000 V (for samples with thickness of 1 mm). In case of samples equipped with one side electrodes only, the true value of the equivalent voltage can be estimated with electrostatic volt- or field-meters. The equivalent voltage is the basic source of error in the determination of the voltage $U$ enforcing flow of the measured current. The error can be determined from the expression:

$$\delta_z U = \frac{U_z}{U}\cdot 100\ [\%] \tag{14}$$

Standards IEC 93 and PN-88/E-04405 recommend to eliminate the influence of the stored space charge through short-circuiting of the sample before the measurement. The short-circuiting time should be so long that value of the discharge current will become incomparable with the expected value of the measured current. For materials with extremely high resistivities (e.g. PTFE) the discharge process can require such a long time that the measurement will not make sense. Acceleration of discharge process can be achieved through increase of the temperature of the sample. Such treatment can however result in serious changes in the micro-molecular structure of the investigated material.

The voltage $U_Z$, determined by a particular distribution of the space charge in the sample, may act in or against the direction of the polarising voltage $U$. Assuming, that the value of $U_Z$ remains constant throughout the measurement, the error $\delta_z U$ can be eliminated or considerably diminished by application of the procedure [1,2,5] which includes periodical changing of the direction of the polarising voltage. Such procedure can be easily carried out by application of the Keithley, model 6517A electrometer, equipped with the 6517 Hi-R Test software [7].

Incomplete elimination of the error $\delta_z U$ causes uncertainty of the measurement of dielectric resistivity. The second component of uncertainty is associated with appearing of voltage-drops on the electrode-dielectric interface, which are difficult to determine.

Their appearance manifests especially in case of electrodes glued to the samples made of relatively low resistivity materials. The phenomenon is also present in case of evaporated electrodes, but when high values of polarising voltages are applied, the last can be ignored.

Uncertainty of determination of voltage $U$ should be supplemented with additional uncertainty associated with the setting the measured voltage of the internal standard voltage source of the resistance meter.

The next component is the uncertainty $\delta I$ of the measured current. The current flowing through the sample under the voltage $U$ consist of inertial-less, component $I_u$, determined by the conductivity, and the transient one $I_P(t)$. Thus, the measured current I *(t)* is a function of time as it was shown in the Fig. 2. If the time of attaining of the steady state by the measured current exceeds 1 minute, the measurement is made for the values of transient currents and the mentioned standards recommend to present the resistivity by appropriate *R(t)* values, as it was shown in the Fig 2. Thus, the uncertainty of measurements for the resistance determined in transient conditions will be affected by uncertainty of the measurement of time. In case of resistivity measurements with high accuracy digital electrometers the fluctuation of the last 1-2 digits of read-outs is usually observed. Thus, the final read-out usually consists of 3-4 digits and the read-out uncertainty (the basic one in comparison with the inaccuracy of the electrometer) should be combined with uncertainty of the electrometer indicated by its manufacturer.

It is often assumed that the measured current will reach its steady-state value after time $t_o$. However some observations for longer times show that the current will further decrease.

So it is important to determine the expected value of the transient component in the total measured current for the particular read-out time. This component creates uncertainty of the measurement of the real value of conductivity current. Such evaluation can be carried out by introducing simplifying assumptions. Let us assume exponential character of the transient process depicted for one relaxation time $\tau$ (e.g. relaxation polarisation). Thus, for the transient component of the measured current one can write:

$$I_p(t) = I_{po} \exp\left(-\frac{t}{\tau}\right), \tag{15}$$

where: $I_{po}$ - initial value of the transient component for $t$=0 (on the switching-on of the polarising voltage $U$). The total value of the volume current $I_v(t)$ (as well as surface current $I_s(t)$) flowing trough the sample under voltage $U$ is described by the expression:

$$I_v(t) = I_{vu} + I_p(t). \tag{16}$$

When the read-out is made for the time $t=t_o$, the remaining part of the transient component of the current $I_p(t_o)$ will crate an error of determination of the conductivity current. The error may be estimated from the relation:

$$\Delta_p I_v = I_v(t_o) - I_{vu}. \tag{17}$$

Similarly, the relative error of the determination of conductive component of the measured current for the time $t=t_0$ will be given by relation:

$$\delta I_v = \left(\frac{I_{po}}{I_{vu}}\right)\exp\left(-\frac{t_o}{\tau}\right). \quad 18)$$

Relation (18) clearly points that the error $\delta I_v$ decreases exponentially with increase of the read-out time $t_o$. Calculation of the mentioned error requires knowledge of the $I_{po}$ value. Usually the value of the $\delta I_v$ is unknown and should be evaluated and taken into consideration as the uncertainty of the measurement. The mentioned problem can be serious for materials exhibiting relaxation processes with long relaxation time $\tau$.

All the mentioned factors contribute to considerable uncertainty of the measured volume resistance especially in case of materials with high resistivity. Even though inaccuracy of applied resistance meters may be on the level of 1%, the total uncertainty often exceeds the value of 10%. In case of materials with extremely high resistivities, e.g. PTFE, the uncertainty of the resistance measurements may be on the level of 100%.

## 8. EVALUATION EXAMPLE

A shortened example of combined uncertainty evaluation for the volume resistivity measurements, carried out on plane–parallel sample, was shown below. The results of average values of particular measurements, including appropriate uncertainties, were collected in the Tab. 1.

Tab. 1. Results of indirect measurements made for the calculations of volume resistivity

| Measuring Device | Measurements result | Relative uncertainty |
|---|---|---|
| Sliding Calliper | $d_1$ =50,04 mm | $\delta d_1$ = 0,08 % |
| Measuring Microscope | $g$ =1,91 mm | $\delta g$ = 2,6 % |
| Micrometer Gauge | $h$ = 0,78 mm | $\delta h$ = 1,3 % |
| Teraohmmeter | $R_v$ =3,7·10$^{14}$ Ω, | $\delta R_v$ =14 % |

According to the data collected in the Tab.1. the volume resistivity, calculated from the expression (1), is equal to $\rho_v$=1,0·10$^{15}$ Ωm, and the relative uncertainty of the resistivity measurements, calculated from the relation (6) is on the level of $\Delta\rho_v$=15 %. It is worth emphasising that the uncertainty of resistance measurement is the most important component of the combined uncertainty of the resistivity measurements in case of dielectric materials.

## 9. SUMMARY

In general volume and surface resistivities are measured by application of indirect methods, including direct measurements of the sample geometric dimensions and measurements of appropriate resistance. Evaluation of the uncertainty of measurements of geometrical dimensions is a simple procedure. However the appropriate measurement of volume or surface resistance, as well as correct evaluation of particular uncertainties may be a serious problem, not solved until today. Description of some details of uncertainty analysis in case of resistivity measurements can be found in papers [1,2,3]. The author's attention was focused on the emphasising problems arising during evaluation of uncertainty of resistivity measurements. Application of the term ”evaluation” is hereby fully justified, because it is practically impossible to precisely calculate uncertainty of the high resistance measurements. Similarly, it is difficult to calculate or estimate uncertainties associated with an appearance of factors mentioned in the section 1. The influence of some of them can be diminished by application of appropriate measurement procedures. Evaluation of influences of different factors on the results of resistivity measurements and methods of avoiding or decreasing them is the subject of the author's investigations. Because the factors influencing the results of measurements cannot be eliminated totally, their influence should be taken into consideration during evaluation of the measurement uncertainty.

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