ISSUES OF VOLUME RESISTIVITY MEASUREMENT OF SOLID DIELECTRICS AND EVALUATION OF UNCERTAINTY OF RESULTS BY APPROXIMATE METHOD AT A CONFIDENCE LEVEL OF 0.95

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Abstract

In the work, the issues of measurements of volume resistivity of solid dielectric made in a concentric three-electrode system have been described. The factors affecting the measurement results, especially the charge collected in the specimens in the result of their electrization and contact resistance between the electrode and specimen have been discussed. Special attention was paid to the effect of temperature and humidity. The methods of minimization of the factors were given. A simplified approach to the evaluation of uncertainty of measurement results of volume resistivity by approximate vector sum at a confidence level of 0.95 was presented.

Keywords: dielectric, measurement, volume resistivity, measurement uncertainty.

1. Introduction

Volume resistivity beside surface resistivity is a basic parameter of dielectric materials. In order to assess its value, a dielectric specimen is placed between two electrodes, forming a capacitor and its resistance is measured. The value of the resistivity is evaluated based on the measured resistance value and geometrical dimensions of the electrodes and specimen. Determining geometrical dimensions usually does not cause any problems. However, serious problems may occur with the measurement of resistance, which can be very high. The values of electric current flowing across the tested sample are often comparable with the values of parasitic currents of galvanic couplings and the measurement results are burdened with high uncertainty [1].

The factors affecting accuracy of the measurements of dielectric resistivity can be classified into factors connected with the tested object and metrological factors associated with the employed method and measuring apparatus. Among the factors connected with the tested object, the following can be pointed out:

− factors influencing material properties in a specimen volume: material structure, pressure (of electrodes), temperature, humidity, kind and intensity of radiation, influence of electric field in the form of nonlinear effects;

− factors influencing the properties of the range adjacent to the electrodes: kind of electrodes and the method of their deposition, electronic phenomena determining the transport of charge carriers across the dielectric- electrode interface and development of specific a space charge distribution in three-electrode range;

− factors associated with the occurrence of transition processes like slow-relaxation polarization, space charge evolution and other physical processes, dependent on the strength of electric field and duration of its exposure.

The metrological factors include: the kind of the employed measurement method and measuring apparatus, noise and disturbances, parasitic and leakage currents of insulation and the measurement conditions (voltage, time of polarization after which the result is read out, temperature, humidity *etc.*).

Minimization of the effects is very difficult and requires both a broad expertise in specific measuring technique and extended knowledge about the tested material. The factors can be the source of errors in resistivity measurements. The employment of suitable measuring procedures can minimize the effect of the mentioned factors on measurement results. However, their complete elimination is impossible. The effect of components not eliminated should be assessed in the form of measurement uncertainty.

2. Currents in the dielectric

Switching on a constant voltage source U connected to a capacitor with dielectric without electric charge between the electrodes results in an increase of intense short duration current in the electric circuit, which can approach the maximum value of I_{vo} [2-4] (Fig. 1a). Then the current drops to a steady state value I_{vu}. The volume current $I_v(t)$ flowing across the dielectric is a superposition of three currents: charging $I_c(t)$, conduction (leakage) I_{vu} and absorption $I_a(t)$
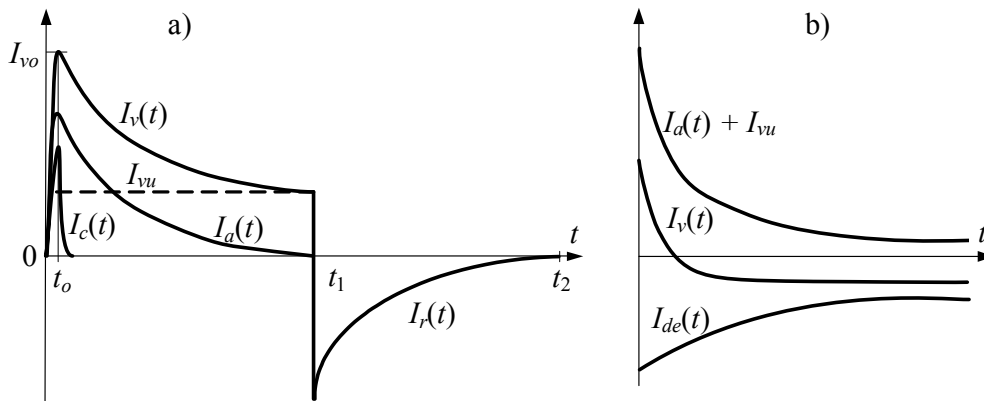
$$I_v(t) = I_c(t) + I_{vu} + I_a(t) . \tag{1}$$

Fig. 1. Volume current $I_v(t)$ versus applied constant voltage. After the time period t_1 the specimen of dielectric was short-circuited for the time period t_2. The dependences concern the specimens: a) without electric charge before the voltage application, b) after the time period t_o with electric charge. Particular current components denote: $I_c(t)$ – charging current, I_{vu} – conduction current, $I_a(t)$ – absorption current, $I_{de}(t)$ – de-electrization current.

The density of steady state current J_{vu} depends on the electric field strength $E,$ according to the following dependence

$$J_{vu} = \gamma E = \frac{E}{\rho_v}, \tag{2}$$

where γ is the dielectric's conductivity and ρ_v its volume resistivity.

Dielectric materials are able to collect and store electric charge even in the absence of an electric field. Some electrification of the dielectric material can be spontaneous and take place during the technological process of material fabrication, its storage, preparation of the

specimens for examinations (deposition of electrodes) as well as during measurements in electric field.

The electrification of the dielectric may also occur in the result of application of an external electric field, for example during measurement of its resistance. The dielectric subjected to an external electric field undergoes electrization. It collects both surface as well as space charge. After switching off the external electric field, the electric charge remains in the dielectric and gradually disappears. The rate of the decay depends on the dielectric's resistivity. In a dielectric with very high resistivity values, the charge may be maintained for several hours, days, weeks or even years (electrets). The value of surface potential associated with the charge can even exceed the value of 1000 V. In such case, the application a voltage of 1000 V to the dielectric specimen results in a current flow in the electric circuit, in the opposite direction compared to the case of uncharged dielectric.

The charge collected in the specimen before the measurement is the source of a de-electrization current $I_{de}(t)$, designated also as background current [1, 2, 5, 7]. Therefore, the volume current $I_v(t)$, flowing across the charged specimen in the result of a constant voltage applied to the electrodes, has also the $I_{de}(t)$ component (Fig. 1b). After the decay time of charging current $I_c(t)$, the volume current is

$$I_v(t) = I_{vu} + I_a(t) + I_{de}(t). \tag{3}$$

The presence of the de-electrization current $I_{de}(t)$, thus the charge in the specimen, can be tested by short-circuiting the specimen through a current gauge, before applying the voltage to the specimen. The de-electrization current $I_{de}(t)$ can also have an important influence on the resultant current $I(t)$ and even change its direction (Fig. 1b). If the voltage source in the specimen circuit is switched off and again switched on, for example by changing the voltage polarity, then beside de-electrization current $I_{de}(t)$ also a resorption current $I_r(t)$ will flow and the resultant current in the dielectric is

$$I_v(t) = I_{vu} + I_a(t) + I_{de}(t) + I_r(t). \tag{4}$$

The specimens prepared for resistivity testing should be deprived of electric charge as it forges the measurement results [8]. Elimination of $I_{de}(t)$ and $I_r(t)$ can be achieved by short-circuiting of the electrodes, where the dielectric has been placed, through a current gauge for a longer time period before each measurement. The time should be long enough so that the value of current flowing through the current gauge achieves a value negligible in comparison with the value of steady state current I_{vu}. The method is not always effective, especially for a dielectric with very high resistivities, like for example polytetrafluoroethylene (PTFE). Such dielectric can be discharged by subjecting the dielectric specimen to elevated temperatures, ca 250 °C [1]. Such a temperature may cause, however, some structural changes in the material.

3. Electrodes

Measurements of volume resistance involve placing the dielectric between conducting electrodes, usually made of metal. The electrodes can have an important effect on the measurement results. At the interface electrode-dielectric, a double layer of electric charge, forming a potential barrier, is created [6]. The layer is characterized by electric capacity and contact resistance [1, 8]. Its structure depends mainly on the charge exchange between the first ion layer adjacent to the electrode and the electrode. If the transition resistance between the first ion layer and the electrode is higher than the resistance between two adjacent

dielectric layers, then the electrode is blocking. If it is equal or lower, then the electrode is reversible [8].

Besides the energetic factors, the method of electrode deposition has an important effect on the metal-dielectric contact. During the vacuum evaporation an intermediate layer is created. Metal particles, due to their kinetic energy and temperature penetrate into some depth to the dielectric, forming a kind of mixture dielectric-metal with quite different properties. The intermediate layer may be also form in result of chemical or mechanical surface treatment, made before the actual deposition of electrodes.

Diffusion of atoms from the electrode to the bulk material can also take place [9, 10]. The local field in the vicinity of the electrode can also differ from the estimated field based on specimen thickness and applied voltage [11]. In the diffusion of elements from the electrode to the dielectric, the kind of material of the electrode (emission energy) plays a crucial role. The intermediate layer may also develop in the result of poor adhesion of the electrodes to the specimen (air gap) or it may be formed by a layer of paraffin oil used for attaching the electrodes. The air gap or intermediate layer between the electrode and the tested material is a source of inaccuracy in the measurements of dielectric's characteristics, when the tested material is thin (like insulation foils) [4].

The electrodes should be made of a material which enables precise contact with the specimen surface and does not introduce any significant error resulting from the resistance of the electrode contact pad with the dielectric. The most often used electrode materials are as follows: silver conducting lacquers, sprayed metal layers, vacuum or cathode evaporated metal layers, metal layers deposited by burning out, metal foils, colloidal graphite, conducting rubber. Massive clamping electrodes made of metal are also used.

Vacuum or cathode evaporation of metal layers (silver, gold, platinum, aluminium, copper) offers the possibility of fabrication of the best electrodes, characterized by the lowest capacities and contact resistances with the dielectric. This technology of the electrodes deposition is currently the most popular. However, it requires quite expensive vacuum equipment.

The electrodes made of metal foil with a thickness from 6 to 80 μm are also often used due to their simple fabrication. They are deposited on the tested specimen with a small amount of vaseline (paraffin), grease, oil or other suitable material or binding agent. Since the binding layer is connected in series with the specimen, it always causes some increase in the measured resistance value.

Similar to the conducting lacquer, colloidal graphite, diluted in water or other carrier, can be deposited with a brush or sprayer onto nonporous surfaces of insulating material and then dried in normal atmospheric conditions. The electrodes made of colloidal graphite are not recommended as they have a high contact resistance and may falsify the measurement results at relatively low resistivities.

The electrodes covered with conducting rubber are comfortable in applications. The specimen can be quickly placed in the electrode set. However, the measurement results carried out with this type of electrodes are always burdened with error, *i.e.* they are higher by several tens or even few hundred percent than the values obtained with the electrodes with evaporated silver [1].

The massive clamping electrodes made of bulk metal (chromium plated brass or stainless steel) with ground contact surfaces with a tested specimen can be used for resistivity measurement of the specimen of elastic material (rubber, elastomer, clothes) well adhering to the metal electrodes. They should not be, however, applied for testing rigid materials, which do not adhere well to the electrodes' surfaces since in the case that air bubbles occur, that would considerably falsify the measurement results.

In case of flat specimen, a three-electrode, concentric system of circular electrodes is recommended by the standards [12-14].

4. Measurement of volume resistances

Methods of volume and surface resistances measurement of dielectric solid materials are described in IEC 60093 [12] international standard, ASTM D 257-99 [13] American standard and in PN-88/E-04405 Polish standard [14]. In practice, the modified indirect method of volume resistance measurement, illustrated in Fig. 2 is employed [1, 15-19]. The circuit is supplied from a calibrated voltage source and the current measurement is carried out with a pico-ammeter with an electrometric amplifier at the input. The input voltage U_s of the pico-ammeter is actually equal to zero whereas the output voltage is proportional to the measured current ($U_w = I_v R_n$). So, at a constant voltage of the source, the indications of the voltage meter U_w are reversely proportional to the measured resistance ($R_v = U/I_v = UR_n/U_w = c/U_w$). Modern digital instruments contain microprocessors which perform mathematical operations. The observer obtains the results in a user-friendly digital form [19].
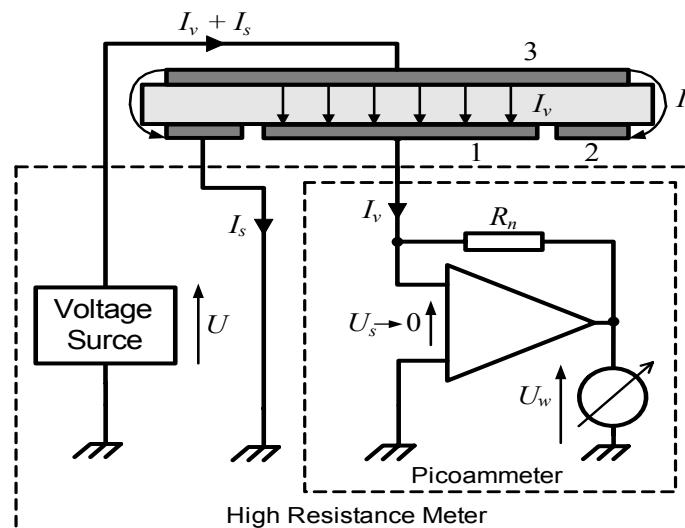

Fig. 2. A system for measuring a volume resistance of solid dielectric materials: 1 – measuring electrode, 2 – guard electrode, 3 – voltage electrode.

Grounding of the ring guard electrode enables elimination of the effect of surface current I_s on the result of I_v current measurement. Connections in the measurement circuit should be made so that the insulation resistance does not shunt the measured resistance whose value can be comparable with the insulation resistance. Therefore, the cable of the pico-ammeter should be absolutely shielded and its shield-guarded. Then, the insulation resistance of the supplying cable shunts the low resistance of the pico-ammeter without affecting the resultant resistance.

The current flowing across the specimen in the result of supplied voltage U decays asymptotically in time down to a steady state value I_{vu} (Fig. 1). In case of dielectrics with high resistivity, the period of time after which the volume current $I_v(t)$ approaches the steady state value I_{vu} may last several minutes, hours and even years (in case of PTFE). Since the measured resistance is the quotient of voltage U to current $I_v(t)$, its value increases asymptotically in time, up to the steady state value (Fig. 1). In case of a material with volume resistivity above 10^{10} Ωm, when the current does not approach its steady state value within 1 min, the characteristics $R_v(t)$ up to $t = 100$ min should be given.

5. Assessment of volume resistivity

Volume resistivity of a flat specimen can be estimated from the following relationship

$$\rho_v = R_v \frac{A}{h},$$ (5)

where A is the effective area of the measuring electrode. The area, in the result of extension of electric field lines outside the range of the measuring electrode, is larger than the actual surface of the electrode. For estimation of the effective area of the measuring electrode, the standards IEC 60093 [12], ASTM 257-99 [13] and PN-88/E-04405 [14] recommend an extension of the diameter of the electrode by the gap width g (Fig. 3). Then, in the case of a flat specimen with circular concentric electrodes, the effective area of the electrode can be estimated from the following dependence

$$A = \frac{\pi(d_1 + g)^2}{4}.$$ (6)

It means that the extension of electric field lines recommended by the standards occurs exactly at the width-half of the gap g. Such assumption would be justified only in the case if the gap width were negligibly small in relation to the specimen thickness ($g \ll h$). Actual distribution of the electric field in the gap space between the measuring and guard electrodes is illustrated in Fig. 3.
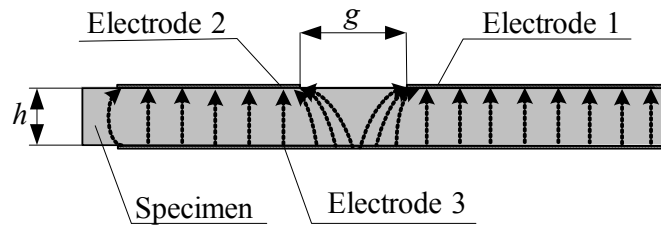

Fig. 3. Electric field distribution in the volume of dielectric specimen in three-electrode arrangement during the measurements of volume resistance: 1 – measuring electrode, 2 – guard electrode, 3 – voltage electrode.

Therefore, the effective area of the circular measuring electrode should be calculated from the relationship [1, 20, 21]

$$A = \frac{\pi(d_1 + Bg)^2}{4},$$ (7)

where B is the expansion coefficient of the electric field beyond the edge of the measuring electrode. The coefficient is less than unity ($B<1$). It can be estimated from Amey's dependence [1, 13, 20, 21]

$$B = 1 - \frac{4}{\pi} \frac{h}{g} \ln \cosh\left(\frac{\pi}{4} \frac{g}{h}\right).$$ (8)

For very thick specimens, when $h \gg g$, the value of $B \to 1$. For very thin specimens, when $h \ll g$, the value of $B \to 0$. For instance, if $h = 0.1g$, i.e. for a gap width $g = 1$ mm and a foil with a thickness of $h = 100$ μm, B takes on the values below 0.1. So, the assumption $B = 1$

may have an important effect on the calculation results of the effective area A and consequently on the results of measurement of volume resistivity of particularly thin materials, like foils. If we assume $B = 1$, then the relative error of estimation of effective area of a circular measuring electrode is given by

$$\delta A = \frac{8h}{\pi d_1} \ln \cosh\left(\frac{\pi}{4}\frac{g}{h}\right).$$

(9)

The dependence (9) is illustrated in Fig. 4. It should be pointed out that for the specimen thickness smaller than the gap width, the errors exceed 1 %, whereas for extremely thin specimens they can even approach 16%. Therefore, for very thin foils, the value of $B = 0$ may be used in the calculations of the effective area and in any case $B = 1$ cannot be assumed.
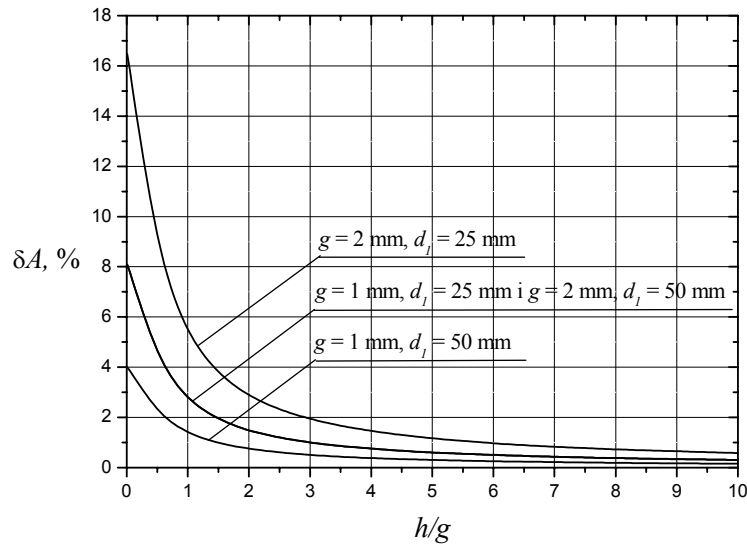

Fig. 4. Relative calculation error of effective area of the measurement electrode when $B = 1$ is assumed.

If the measuring and voltage electrodes do not adhere completely to the specimen (there are air bubbles between the specimen and electrodes), then the effective contact area decreases. It may cause measurement errors whose values are difficult to estimate. Therefore, electrodes ensuring the best contact with the specimen should be used.

6. The influence of temperature and humidity

The results of volume resistivity measurement of dielectrics are highly influenced by specimen temperature and humidity. If the specimen is conditioned and measured in different environmental conditions than required, then the theoretical discrepancies can be taken into account in form of corrections. In such case the dependence (5) for volume resistivity can be written as

$$\rho_v = \frac{A}{h} R_v + \Delta\rho_{vT} + \Delta\rho_{vW},$$

(10)

where $\Delta\rho_{vT}$ is a temperature correction taking into account the change in the specimen temperature, and $\Delta\rho_{vW}$ – is a humidity correction taking into account the change in humidity.

To estimate the correction values, the knowledge of characteristics of change in volume resistivity versus temperature and humidity are necessary. Temperature correction which

takes into account deviations in temperature of a specimen ΔT from the standard temperature, can be estimated from the dependence

$$\Delta\rho_{vT} = k_{Tv}\Delta T,\qquad(11)$$

where: k_{Tv} – temperature coefficient of change in volume resistivity, ΔT – the difference between the ambient temperature during the measurements and the temperature for which the measurement results are estimated.

Humidity correction which takes into account deviations in specimen humidity ΔW from standard humidity, can be estimated from the expression

$$\Delta\rho_{vW} = k_{Wv}\Delta W,\qquad(12)$$

where: k_{Wv} – humidity coefficient of change in volume resistivity, ΔW – the difference between the relative ambient humidity during the conditioning and measurements and the humidity for which the measurement results are estimated.

In practice, the temperature and humidity coefficients k_{Tv} and k_{Wv} are usually unknown with such accuracy which would be sufficient for the corrections estimation. For this reason, the measurements are usually conducted in standardized environmental conditions (usually temperature 23 °C and air humidity 50 %). If the measurements are conducted in such conditions then the values of temperature and humidity corrections should be assumed as zeroes. If the temperature and humidity in the laboratory vary within (23±2) °C and (50±10) % respectively, then the zero corrections are defined with uncertainties which should be estimated.

7. General rules of estimation of measurement uncertainties by approximate method

The accurate method of estimation of measurement uncertainties, recommended by ISO Guide [22, 23] is very laborious and not keenly used in industrial laboratories. Therefore, the laboratories are looking for simpler and not so laborious methods of estimation of measurement uncertainty. One of such methods is the method of geometric sum of particular components of extended uncertainties [24, 25].

If value quality $y = f(x_j)$, then for every value x_j, the uncertainty of B-type is estimated from a limit of permissible error $\Delta_g x_j$ with rectangular distribution, then the extended uncertainty $U_B(x) = 0.95\Delta_g x_j$ at the confidence level of 0.95. If the uncertainties of A and B type are comparable and none of them can be neglected, then the extended uncertainty at the confidence level 0.95 can be estimated from the simplified expression

$$U(x_j) = \sqrt{U_A^2(\overline{x}_j) + (\Delta_g x_j)^2}\ ,\qquad(13)$$

with the extended uncertainty of A type

$$U_A(\overline{x}_j) = t_{0,95;n}u_A(\overline{x}_j),\qquad(14)$$

where: $t_{0,95;n}$ is a quantile of t-Student distribution for the probability of 95 %, depending on the number of measurements n, $u_A(\overline{x}_j)$ – a standard uncertainty of type A for average value of x_j , equal to the standard deviation of the average value $s(\overline{x}_j)$ – estimated from the commonly known expression

$$u_A(\bar{x}_j) = s_A(\bar{x}_j) = \sqrt{\frac{\sum_{i=1}^{n}(x_{i_j} - \bar{x}_j)^2}{n(n-1)}}, \tag{15}$$

where: x_{ij} is the value of i-th measurement of the quantity with the value x_j, n − is the number of measurements of the quantity.

If $y = f(x_j)$, then for $u_A(x_j) < u_B(x_j)$ a simplifying assumption that the uncertainties $U(x_j)$ have a rectangular distribution can be accepted and the extended uncertainty at the confidence level 0.95 can be estimated from the dependence

$$U(y) = \frac{k_p}{k_B} \sqrt{\sum_{j=1}^{m} c_j^2 U^2(x_j)} = \frac{2}{\sqrt{3}} \sqrt{\sum_{j=1}^{m} c_j^2 \left[U_{Aj}^2(\bar{x}_j) + (\Delta_g x_j)^2 \right]} = 1.15 \sqrt{\sum_{j=1}^{m} c_j^2 \left[U_{Aj}^2(\bar{x}_j) + (\Delta_g x_j)^2 \right]}, \tag{16}$$

where: k_p is a coverage factor for normal distribution, k_B − coverage factor for a rectangular distribution.

8. Uncertainty of measurement results of volume resistivity

If the tested specimen does not contain any electric charge before the measurement and the electrodes resistance is negligibly low, then the volume resistivity may be evaluated from the dependence (10). The quantities included in the dependence are not correlated with each other. An extended uncertainty at the confidence level $p = 0.95$ can be assessed by an approximate method based on the equation (16), which for the volume resistivity has the form

$$U(\rho_v) = 1.15 \sqrt{c_A^2 U^2(A) + c_h^2 U^2(h) + c_{R_v}^2 U(R_v) + c_T^2 U^2(\Delta\rho_{vT}) + c_W^2 U^2(\Delta\rho_{vW})}, \tag{17}$$

where the particular coefficients of sensitivity are as follows

$$c_A = \frac{\partial \rho_v}{\partial A} = \frac{R_v}{h}, \tag{18}$$

$$c_h = \frac{\partial \rho_v}{\partial h} = -\frac{A}{h^2} R_v, \tag{19}$$

$$c_{R_v} = \frac{\partial \rho_v}{\partial R_v} = \frac{A}{h}, \tag{20}$$

$$c_T = 1 \quad \text{and} \quad c_W = 1.$$

The effective area of measuring electrode A can be evaluated from the equation (7) and the uncertainty of the effective area of the measuring electrode can be expressed by the relationship

$$U(A) = \sqrt{c_{d_1}^2 U^2(d_1) + c_g^2 U^2(g) + c_B^2 U^2(B)}, \tag{21}$$

where the particular coefficients of sensitivity are

$$c_{d_1} = \frac{\partial A}{\partial d_1} = \frac{\pi}{2}(d_1 + Bg), \tag{22}$$

$$c_g = \frac{\partial A}{\partial g} = \frac{\pi}{2} B(d_1 + Bg), \tag{23}$$

$$c_B = \frac{\partial A}{\partial B} = \frac{\pi}{2} g(d_1 + Bg), \tag{24}$$

B coefficient is defined by the dependence (8) so the uncertainty of the coefficient is

$$U(B) = \sqrt{c_h^2 U^2(h) + c_g^2 U^2(g)}, \tag{25}$$

where the particular coefficients of sensitivity are

$$c_h = \frac{\partial B}{\partial h} = \frac{g}{h^2} \left[\frac{4}{\pi} \ln \cosh\left(\frac{\pi}{4} \frac{g}{h} \right) + \frac{g}{h} \tgh\left(\frac{\pi}{4} \frac{g}{h} \right) \right], \tag{26}$$

$$c_g = \frac{\partial B}{\partial g} = -\frac{1}{h} \left[\frac{4}{\pi} \ln \cosh\left(\frac{\pi}{4} \frac{g}{h} \right) + \frac{g}{h} \tgh\left(\frac{\pi}{4} \frac{g}{h} \right) \right]. \tag{27}$$

8.1. Uncertainty of measurement of specimen thickness

A specimen thickness h is measured before deposition of electrodes with a micrometer at different points of the surface of the measuring electrode. The uncertainty of type A of average specimen thickness

$$U_A(\bar{h}) = t_{0,95;n} \sqrt{\frac{\sum_{i=1}^{n}(h_i - \bar{h})^2}{n(n-1)}}, \tag{28}$$

where: h_i – results of successive measurements of specimen thickness, $\bar{h}$ value of arithmetic average, n – number of measurements, $t_{0,95;n}$ – quantile t-Student distribution determined at the confidence level $p = 0.95$ for n measurements.

The uncertainty of specimen thickness measurement result is

$$U(h) = \sqrt{U_A^2(\bar{h}) + (\Delta_g h)^2}, \tag{29}$$

where $\Delta_g h$ is the limit of permissible error of the measuring instrument used for the measurement of specimen thickness.

8.2. Uncertainty of measurement of diameter of measuring electrode

The measurements of the diameter of the measuring electrode d_1 are carried out repeatedly at different points with a slide calliper. The type A uncertainty of the average value of diameter is

$$U_A(\bar{d}_1) = t_{0,95;n} \sqrt{\frac{\sum_{i=1}^{n}(d_{1i} - \bar{d}_1)^2}{n(n-1)}}, \tag{30}$$

where: d_{1i} – results of successive measurements, $\overline{d}_1$ –average of the diameter measurement results.

The uncertainty of diameter measurement is

$$U(d_1) = \sqrt{U_A^2(\overline{d}_1) + (\Delta_g d_1)} ,$$

(31)

where $\Delta_g d_1$ is a limit of permissible error of measuring gauge used for the diameter measurement.

8.3. Uncertainty of measurement of gap width

The gap width g between the measuring and guard electrodes is usually measured with a measuring microscope at several points. The type A uncertainty of the gap width

$$U_A(\overline{g}) = t_{0,95;n} \sqrt{\frac{\sum_{i=1}^{n}(g_i - \overline{g})^2}{n(n-1)}} ,$$

(32)

where: g_i – results of successive measurements, $\overline{g}$ – average of the measurement results.

The uncertainty of measurement of the gap width

$$U(g) = \sqrt{U_A^2(\overline{g}) + (\Delta_g g)^2} ,$$

(33)

where $\Delta_g g$ is the limit of permissible error of the measuring instrument applied for the gap width measurement.

8.4. Uncertainty of measurement of volume resistance

The uncertainty of measuring instruments is given in the form of a limit of permissible error. The uncertainty of resistance measurements is also affected by instability of indications, caused by fluctuation of the occurrences in the specimen and other disturbances. The uncertainties of measurement of specimen resistance are also influenced by the errors connected with the transition resistance between electrodes and the examined specimen as well as the electric charge gathered in the specimen. The errors are difficult to evaluate and they should be minimized as much as possible to keep them negligible.

If the volume current $I_v(t)$ does not approach a steady state value within 1 min, the result of resistance measurement should be given in the form of $R(t)$ characteristics, after its multiplied measurement. However, before each new measurement the sample should be completely discharged and it should be checked if the value of the short-circuit current is negligibly small. The results of $R_v(t)$ measurements are average values of repeated characteristics of $R_v(t)$. Uncertainty of type A of volume resistance at a certain time is calculated from the relationship

$$U_A(\overline{R}_v) = t_{0,95;n} \sqrt{\frac{\sum_{i=1}^{n}(R_{vi} - \overline{R}_v)^2}{n(n-1)}} ,$$

(34)

where: R_{vi} – successive results of resistance measurements, $\overline{R}_v$ – average value of the results of resistance measurements.

Extended uncertainty of R_v measurement can be assessed from the expression

$$U(R_v) = \sqrt{U_A^2(\overline{R}_v) + (\Delta_g R_v)^2},$$ (35)

where $\Delta_g R_v$ is the limit of permissible error of the measuring instrument used for resistance measurement.

8.5. Uncertainty of temperature correction of volume resistance

Temperature correction of volume resistance taking into account a change in temperature of the specimen ΔT in comparison with a standard temperature ($T = 23°C$) can be estimated from the equation (11), and its uncertainty from the relationship

$$U(\Delta\rho_{vT}) = \sqrt{(\Delta T)^2 U^2(k_{Tv}) + k_{Tv}^2 U^2(\Delta T)},$$ (36)

where: k_{Tv} – temperature coefficient of volume resistance change, ΔT – temperature difference between the ambient temperature during measurements and the temperature for which the volume resistance is estimated, $U(k_{Tv})$ – uncertainty of the temperature coefficient of volume resistance change, $U(\Delta T)$ – uncertainty of estimation of the temperature difference.

If the temperature in the laboratory during the measurements corresponds to the required standard value, then the correction $\Delta\rho_{vT} = 0$ is assumed. However, the zero correction does not mean zero uncertainty since in practice the ambient temperature in the laboratory always undergoes some fluctuations.

8.6. Uncertainty of humidity correction of volume resistance

Humidity correction of change in volume resistance, taking into account a change in humidity of air surrounding the tested sample of ΔW in comparison with a standard humidity $W = 50\%$ can be estimated from equation (12) and its standard uncertainty, from the relationship

$$U(\Delta\rho_{vW}) = \sqrt{(\Delta W)^2 U^2(k_{Wv}) + k_{Wv}^2 U^2(\Delta W)},$$ (37)

where: k_{Wv} – humidity coefficient of change in volume resistivity, ΔW – difference between relative humidity of ambient during the measurements of volume resistance and the relative humidity for which the volume resistivity is estimated, $U(k_{Wv})$ – uncertainty of the humidity coefficient of change in volume resistance, $U(\Delta W)$ – uncertainty of estimation of humidity difference.

If the humidity in the laboratory during the measurements corresponds to required standard humidity, then the correction $\Delta\rho_{vW} = 0$ is assumed. However, the zero correction does not mean its zero uncertainty since in practice the air humidity in laboratory always undergoes some fluctuations.

9. An example of calculation of measurement uncertainty of volume resistivity

Volume resistivity of polyamide was estimated based on the measurement of dimensions of a flat specimen and concentric circular electrodes and the measurement of volume resistance. The measurements were carried out after appropriate conditioning of the specimen in the laboratory, where the temperature was kept at 23 °C ± 1 °C and relative humidity of air

at 50% $\pm$ 5 %. Vacuum evaporation of silver electrodes was preceded by measurement of specimen's thickness h with a micrometer. Then, the silver electrodes were vacuum evaporated and the gap width g was measured with a measuring microscope Additionally, the measurement of the diameter of the measuring electrode d_1 was made with a slide calliper.

Before the resistance measurement, the specimen was short-circuited by a pico-ammeter for a period after which the de-electrization current approached the 0.01 value of the expected volume current.

Then the measurement was carried out at the voltage of 1000 V after stabilization of the value of indication. The indication was stabilized after 3 min. at the value $R_v = 2,80 \times 10^{13}$ Ω. Relative limit of permissible error of the volume resistance measurement was 6.2 %.

The balance of uncertainty calculations at the confidence level of 0.95 by the described approximate method is presented in Tables 1, 2, and 3. At first, the uncertainty of the coefficient of extension of the effective area of the measuring electrode was estimated (Table 1), then the uncertainty of estimation of the effective area of the measuring electrode was calculated (Table 2). In Table 3, a balance of calculations of extended uncertainty of volume resistance, based on the results in Table 2, is presented.

Table 1. Balance of uncertainty of the coefficient of extension of effective area of measuring electrode $U(B)$.

Measured quantity X_j	Mean value $\overline{X}_j$	Sensitivity coefficient c_j	Extended uncertainty $U(X_j)$	$c_j^2 \cdot U^2(X_i)$
Thickness h	$1{,}668 \times 10^{-3}$ m	-189 m^{-1}	$0{,}028 \times 10^{-3}$ m	$28{,}01 \times 10^{-6}$
Width g	$1{,}887 \times 10^{-3}$ m	167 m^{-1}	$0{,}014 \times 10^{-3}$ m	$5{,}47 \times 10^{-6}$
Coefficient B	$0{,}6042$		$\Sigma c_j^2 \cdot U^2(X_j) = 33{,}48 \times 10^{-6}$	
Uncertainty of coefficient B			$U(B) = 0{,}0058$	

Table 2. Balance of uncertainty of effective area of measuring electrode $U(A)$.

Measured quantity X_j	Mean value $\overline{X}_j$	Sensitivity coefficient c_j	Extended uncertainty $U(X_j)$	$c_j^2 \cdot U^2(X_j)$
Diameter d_1	$49{,}974 \times 10^{-3}$ m	$80{,}29 \times 10^{-3}$ m	$0{,}041 \times 10^{-3}$ m	$10{,}84 \times 10^{-12}$ m^4
Width g	$1{,}887 \times 10^{-3}$ m	$48{,}52 \times 10^{-3}$ m	$0{,}014 \times 10^{-3}$ m	$0{,}47 \times 10^{-12}$ m^4
Coefficient B	$0{,}6042$	$1{,}515 \times 10^{-4}$ m^2	$0{,}0058$	$0{,}78 \times 10^{-12}$ m^4
Area A	$2{,}05 \times 10^{-3}$ m^2		$\Sigma c_j^2 \cdot U^2(X_j) = 12{,}19 \times 10^{-12}$ m^4	
Uncertainty of the area of measuring electrode A			$U(A) = 0{,}004 \times 10^{-3}$ m^2	

Table 3. Balance of uncertainty of volume resistance $U(\rho_v)$.

Measured quantity X_j	Mean value $\overline{X}_j$	Sensitivity coefficient c_j	Extended uncertainty $U(X_i)$	$c_j^2 \cdot U^2(X_i)$
Area A	$2{,}052 \times 10^{-3}$ m^2	$1{,}68 \times 10^{16}$ Ω/m	$0{,}004 \times 10^{-3}$ m^2	$0{,}5 \times 10^{22}$ Ω^{2}m^2
Thickness h	$1{,}668 \times 10^{-3}$ m	$-2{,}07 \times 10^{21}$ Ω	$0{,}028 \times 10^{-3}$ m	$33{,}6 \times 10^{22}$ Ω^{2}m^2
Volume resistance R_v	$2{,}80 \times 10^{13}$ Ω	$1{,}23$ m	$0{,}2 \times 10^{13}$ Ω	$605{,}2 \times 10^{22}$ Ω^{2}m^2
Temp. correction $\Delta\overline{\rho}_{vT}$	0	1	$2{,}0 \times 10^{12}$ Ωm	400×10^{22} Ω^{2}m^2
Hum. correction $\Delta\overline{\rho}_{vW}$	0	1	$2{,}0 \times 10^{12}$ Ωm	400×10^{22} Ω^{2}m^2
Volume resistivity ρ_v	$3{,}45 \times 10^{13}$ Ωm		$\Sigma c_j^2 \cdot U^2(X_j) = 1439{,}3 \times 10^{32}$ Ω^{2}m^2	
Extended uncertainty of volume resistivity			$U(\rho_v) = 0{,}44 \times 10^{13}$ Ωm	

Uncertainty of measurement of volume resistivity of a polyamide specimen at the confidence level $p = 0.95$, estimated by approximate method has almost the same value as the extended uncertainty calculated by an exact method *i.e.* $U(\rho_v) = 0{,}44 \times 10^{13}$ Ωm. It should be pointed out that in spite of maintaining all possible measures for proper measurement accomplishment, the relative uncertainty of volume resistance measurement is quite high and amounts to 13 % in the considered example. Estimation of the volume or surface resistance with relative uncertainty below 10 %, seems to be, according to the author's experience, practically impossible.

10. Summary

Volume resistance is always measured by an indirect method, by the measurement of geometrical dimensions and appropriate resistances. Appropriate estimation of uncertainty of the measurements of geometrical dimension of the tested specimen is relatively simple.

Correct accomplishment of resistance measurements as well as appropriate assessment of the measurement uncertainty is quite a serious problem which has not been completely solved up to now. Correct estimation of volume resistivity based on the measurement results is also very important. The expressions, given in the international standard IEC 60093 [12] as well as in the Polish standard PN/E-04405 [14] are simplified and the resistivity calculated with the expressions is burdened with significant errors (see section 5). The person performing the measurements is often unconscious of the errors and does not take them into account neither in the resistivity calculations nor in uncertainty evaluation. Therefore, the author together with R. Kasprzyk proposed some changes in the international standard IEC 60093, concerning the measurement of volume and surface resistivity [20].

The results of resistivity measurement of dielectric materials are affected by numerous factors. Many of them can be avoided by the application of appropriate measurement procedures. The relative uncertainty of estimation of volume resistivity of dielectric materials is of the order of several tenth per-cents and can be successfully estimated by the presented approximate method at the confidence level of 0.95. The method is much simpler than the exact method and in practice, it gives similar results.

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