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INFLUENCE OF MOISTURE AND SALINITY OF SOIL ON ITS DIELECTRIC PERMITTIVITY

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LIST OF SYMBOLS

FDR	– Frequency Domain Reflectometry
FFT	– Fast Fourier Transform
PLL	– Phase Locked Loop
TDR	– Time Domain Reflectometry
VNA	– Vector Network Analyser
a, b, c	– complex coefficients of equation for model of two-terminal-pair network type T
a', b', c'	– modified complex coefficients of equation for model of two-terminal-pair network type T
a_0, a_1	– trend line coefficients
c	– velocity of light in vacuum
C_f	– internal threshold capacity of measuring probe
C_0	– capacity of coaxial or tw0-rod probe in air
f	– frequency of measurement signal
j	– imaginary unit equal to $\sqrt{-1}$
L	– planar spacing of points of complex coefficient of reflection S_{11}
P	– vector of polarisation
P_s	– vector of polarisation of medium in constant electric field
$\mathrm{Re}(\varepsilon)$, $\mathrm{Im}(\varepsilon)$	– real and imaginary parts of dielectric permittivity ε
R, L, G, C	– unit parameters of long line
S_{11}	– complex coefficient of reflection
$S_{11\mathrm{air}}$	– measured coefficient of reflection for probe in air
$S_{11\mathrm{cal}}$	– coefficient of reflection determined from calibration with two-terminal-pair network type T
$S_{11\mathrm{m}}$	– measured coefficient of reflection for probe placed in soil
$S_{11\mathrm{model}}$	– coefficient of reflection determined for model of lossy transformer
$\lvert S_{11}\rvert, \varphi$	– module and phase of measured complex coefficient of reflection S_{11}
t	– time
t_a, t_b	– recorded time of characteristic reflections of TDR signal
x	– length of probe rods
Z	– probe impedance for model Z_f, Z_0
Z_A	– impedance of probe rods in acetone calculated from measurement
Z_{air}	– probe impedance in air
Z_c	– characteristic impedance of connection cable

Z_f	– threshold impedance within measurement probe
Z_L	– impedance for model of lossy transformer
Z_K	– impedance of tips of probe rods
Z_p	– probe impedance for capacitance model C_0, C_f
Z_P	– impedance of probe rods in air, calculated from measurement
Z_T	– equivalent impedance of two-terminal-pair network type T measured by VNA
Z_0	– impedance of capacitor C_0 filled with lossy dielectric
Z_0	– impedance of rods of two-rod probe in air
Z_1, Z_2, Z_3	– component impedances of two-terminal-pair network type T
Z_Z	– fault impedance of probe rods at the base $0+j0$
α	– phase constant
β	– attenuation constant
ε	– complex dielectric permittivity of material studied
ε_A	– dielectric permittivity of acetone from Debye's model
ε_{air}	– dielectric permittivity of gaseous phase
ε_b	– apparent dielectric permittivity of soil determined with TDR method
ε_d	– dielectric loss
ε_P	– dielectric permittivity of air equal to $1+j0$
ε_s	– dielectric permittivity for static field
ε_{solid}	– dielectric permittivity of solid phase
ε_{wat}	– dielectric permittivity of liquid phase
ε_0	– vacuum permittivity
ε_∞	– dielectric permittivity above relaxation frequency
γ	– propagation constant
θ	– volumetric moisture determined with oven-dry method
θ_{TDR}	– volumetric moisture of material determined with TDR method
λ	– wavelength
σ_{DC}	– DC conductivity
ρ	– soil density
τ	– relaxation time
ω	– angular velocity equal to $2\pi f$

1. INTRODUCTION

In the present times the protection of the natural environment is assuming a particular importance due to the violent development of industry and to the growing civilization threats. Effective protection against the degradation of the environment can be ensured by suitable methods of monitoring of physical, chemical and biological parameters that will permit the modelling of processes taking place in nature, which will allow the prediction of the places of occurrence and counteracting further degradation. Thanks to modelling based on archivised information it is possible to predict approaching threats on local and global scales, which entails measurable economic benefits. The development of agrophysical metrology, and especially inexpensive and energy-saving methods of measurement, becomes a necessity due to the continual expansion of both the areas covered with monitoring and of the amount and diversity of acquired data. Increase in the demand for agrophysical measurements results also from the development of satellite monitoring of the soil environment, being the cheapest form of acquisition of information but requiring, as in the case of the requirements of metrology, an extensive network of land-based measurement points. Water is a key element requiring monitoring, and having a direct and immediate effect on all the processes that take place in the soil environment. The monitoring of parameters concerning the quality and quantity of water occurring in various places on our planet provides an abundance of valuable information, analysis of which permits evaluation of the status of the soil environment. The undertaking of suitable actions permits counteracting the harmful effects of human activity before the undesirable phenomena of an excess or a deficit of water may occur. Of particular importance is the measurement of soil moisture in places threatened with mudslides, floods, and under conditions of strong soil contamination. Thanks to the unique dielectric properties of water it is possible to perform selective measurement of the liquid phase volumetric fraction of porous materials with the use of electronic methods. At present the most popular method of measurement of moisture and salinity of porous materials, and of soil in particular, is the TDR method (Time Domain Reflectometry). That method gained popularity in the nineteen eighties, and due to its versatility and ease of application is now one of the most frequently used in research laboratories as well as in monitoring systems. There is also an ongoing search for alternative and cheaper methods, with comparable parameters of selectivity and accuracy that would permit their widespread application.

This paper presents the results of a study on the effect of soil moisture and salinity on measurements performed with the method of Frequency Domain Reflectometry (FDR), as well as an evaluation of a probe developed for the measurement of soil moisture and salinity, operating on the basis of the method. It permits

the application of probes in the form of a parallel waveguide, with probe rod lengths like in the TDR method, but also considerably shorter. Measurements with the FDR method permit direct determination of the frequency spectrum of complex dielectric permittivity, with the measurement signal having a controlled amplitude for the whole spectrum, which is something the TDR method cannot provide. Spectrum measurement permits the application of the methods of dielectric spectroscopy in the analysis of the results, thanks to which it is possible to obtain also information other than that on moisture and salinity of the medium studied, e.g. on the content of bound water or on the particle size distribution. For this reason the method can be applied for the study and estimation of quality of porous materials of agricultural origin on the basis of their dielectric properties. In remote-sensing measurements of soil moisture the measurement signal frequencies in the range of 1-1.5 GHz are the least attenuated by the atmosphere and the least susceptible to the effect of the vegetation cover (Walker *et al.* 2004, Kerr 2007). Therefore, the ease of selection of the measurement signal frequency in the FDR method permits measurements that are easier to correlate with remote-sensing measurements conducted by means of satellites. The method is characterised by low energy requirements (Veldkamp and O'Brien 2000), and its development can contribute to the appearance of new sensors for the measurement of soil moisture and salinity, operating on battery power and permitting maintenance-free operation with wireless transmission of the measurement results.

2. ELECTRICAL METHODS OF MEASUREMENT OF POROUS MATERIAL PROPERTIES

Agrophysics is a branch of science created from the need to get insight into physical properties and phenomena happening in biological environment of plants conditioning the process of photosynthesis (light, heat, water, nutrients), and therefore the yield. The application of research and interpretation tools characteristic to physics makes Agrophysics original and interdisciplinary branch of science.

The key problem in Agrophysics is water status in soil-plant-atmosphere system, because every phenomenon examined in its scope of interest relays somehow on the water status. The number of variables necessary to describe the water status in porous materials depends on the examined problem and is practically arbitrary. In majority of practical situations the status of water is described only by one variable – water content. Soil is the material where water status should be described by minimum five variables: water content (moisture), water potential, salinity, oxygenation and temperature. The most demanding biological material for monitoring the status of water contained in it is soil, because soil structure is

diversified and its properties are not stable in time. Also, soil water content seems to be the most difficult water status variable to determine because the response of soil water content sensors is not dependent only on soil water content. It may depend on soil salinity, temperature and texture. Therefore there has been much effort put down on the soil water content metrology. If there exist a reliable method of soil water content measurement it could be applicable to other biological materials with less differentiated structure.

There are three international standards for soil water content measurement. Two of them relate to the thermogravimetric methods: ISO11461 – for water content on volume basis, ISO11465 for water content on the mass basis and the norm describing neutron scattering method for soil water content determination in unsaturated zone – ISO10573. Thermogravimetric methods enable to determine the amount of water in the soil sample on the mass or volume basis in the direct way and they are the most popular. These methods are very laborious, time consuming and require laboratory equipment. In the indirect neutron scattering method the value of soil water content is correlated with the energy loss of neutrons emitted by neutron probe, located in the access tube inserted vertically into the soil, bumping into the hydrogen nuclei having similar mass. Neutron scattering method is fast and precise but is becoming less popular because of radiation hazard.

The soil conductivity measurement method is described by the international standard ISO 11265. After drying the soil sample according to ISO 11464 standard, it is mixed with water in proportion 1:5 and then the filtered extract conductivity is measured by a liquid conductometer. This method is time consuming, laborious and its application *in situ* is limited.

The reflectometric method for soil water content and electrical conductivity has become popular since the publication of Topp *et al.* (1980). This paper started research in TDR application in soil metrology and now recognized distributors of soil metrology offer the TDR meters for soil water content and electrical conductivity measurement. Time Domain Reflectometry technique was known even earlier; it was successfully applied by Fellner-Feldegg (1969) for the determination of dielectric properties of liquids. The researchers from the Institute of Agrophysics took part in the development of this technique (Malicki and Skierucha 1989) as well as in the construction of TDR meters which are being produced on the license of IAPAS (Easy Test 2004) The main advantages of TDR over other soil water content measurement methods are:

- superior accuracy to within 1 or 2% volumetric water content;
- calibration requirements are minimal – in many cases soil-specific calibration is not needed;
- lack of radiation hazard associated with neutron probe technique;

- TDR has excellent spatial and temporal resolution; and
- measurements are simple to obtain, and the method is capable of providing continuous measurements through automation and multiplexing.

The common use of TDR method and above mentioned advantages place it as a possible standard of porous material, including soil, water content measurement method.

2.1. Dielectric permittivity

Dielectric permittivity ε (relative permittivity) is a dimensionless value describing the properties of a dielectric placed in an electric field. It is defined as the ratio of the capacity of a capacitor filled with the dielectric studied to the capacity of the same capacitor but with vacuum as the dielectric (Agilent 2006, Chełkowski 1993). Unfortunately, it is often referred to as the dielectric constant, although it is a known fact that it depends on temperature, pressure, and on the intensity of the electric field. Dielectric measurements of moisture make use of the specific dielectric properties of water particles, related with its polar structure, i.e. with the occurrence of an unbalanced dipole moment with the value of $6.2 \cdot 10^{-30}$ C m^{-1}. Dipoles of water particles, due to the thermal movement, are arranged in a chaotic manner. After the application of an external electric field, water particles get partially polarized and change their orientation so that their dipoles become parallel to the lines of the field. The value of dielectric permittivity of water is 80.14 at temperature of 20°C, which makes it distinct from other materials – components of the porous medium that is the soil, the permittivity of which is considerably lower. Therefore the essence of dielectric measurements of moisture of porous materials is the utilization of the strong relation between the water content in the material studied and its dielectric permittivity.

Most agricultural materials and products containing water are characterised by ionic electrical conductivity, which, in low frequency measurements, has a significant effect on measurement accuracy or even precludes measurement of permittivity. This problem has been solved through the application of variable electric field for which ionic conductivity decreases with increase of frequency (White *et al.* 1994). The application of high frequencies causes a change in the character of dielectric permittivity which in a constant electric field (frequency equal to zero) is an real value. In a variable electric field it becomes a complex value, dependent on the frequency of changes of the field.

$$\varepsilon = \mathrm{Re}(\varepsilon) - j\,\mathrm{Im}(\varepsilon)\,. \tag{1}$$

Water particles placed in an electric field of a given frequency tend to orient in accordance with the vector of the variable external electric field. The thermal movements of the molecules cause that their dipoles oscillate around the direction of the external field, which is the cause of the appearance of thermal dependence of dielectric permittivity. Due to inertia, that movement takes place with a certain delay with relation to the electric field applied. Above the frequency of 18 GHz (relaxation frequency) water molecules cannot follow the speed of the fast changes of the electric field. The real part Re(ε) of the complex permittivity describes the capacity of a medium to store the energy of the external electric field, while the imaginary part Im(ε) represents energy losses related with the thermal movement of vibrating dipoles.

Measurement of moisture of porous materials with dielectric methods makes use of the commonly known relations between permittivity and moisture. Those relations describe the effect of the particular phases of soil on its permittivity which can be measured in the time domain (TDR) and in the frequency domain (FDR). Measurement in time domain allows to calculate the so-called apparent permittivity for a frequency spectrum occurring in a pulse of step-wise function (Malicki and Skierucha 1989, Topp *et al.* 1980). It is assumed that dielectric loss and ionic conductivity do not affect the speed of propagation of electromagnetic pulse in the medium studied, but only the amplitude relations. Whereas, measurements performed in frequency domain provide direct information about both components of the complex permittivity, allowing a much broader analysis of dielectric properties, determined not only by moisture but also by phenomena related with the occurrence of bound water or with the polarisation of colloidal particles of soil, leading to the determination of their relaxation time. The additional information, such as the relaxation time, can be correlated with other physical properties of soil, e.g. specific surface area, content of clay particles or of organic matter.

2.2. Electrical measurement of soil water content

Concerning the problem of electrical soil water content measurement, the medium conditioning water content is water and its specific property is the polar structure of water molecules (a water molecule has a permanent dipole moment of 1.87 D). Polarity of water molecules is the reason that dielectric permittivity (dielectric constant) of water is much higher than permittivity of soil solid phase (the relative dielectric constant of water in the electric field of frequency below 10 GHz and 18°C temperature is 81, while the relative dielectric constant of solid phase is 4-5 at the same conditions). The dielectric constant of soil strongly depends on its water content, therefore it may be concluded that electrical measure-

ment of soil water content should be based on the measurement of its dielectric constant.

The efforts to measure soil water content indirectly, on the base of the resistance or electrical capacitance between the electrodes inserted into the soil, has been continuously undertaken since the end of nineteen age (Whitney *et al.* 1897).

2.3. Electrical measurement of soil salinity

Similarly, referring to the problem of electrical measurement of soil salinity, the medium conditioning its salinity is salt present in the soil and the unique property is their ionic nature. The ability to conduct electrical charge by ions present in the soil water is the reason that the soil conducts current. Therefore, the electrical measurement of soil salinity should be based on the measurement of its electrical conductivity.

Soil salinity is expressed by a total concentration of salts in the soil, and practically their ions in the "soil water". Due to technical reasons connected with the determination of such a defined salinity, its measure by indirect quantity – electrical conductivity of the soil extract, named as a soil electrolyte, which is taken from the soil after its saturation by distilled water (Marshall and Holmes 1979). This procedure proposed by US Salinity Laboratory (US Salinity Laboratory 1954), has been recognized as standard. However this method cannot be used in *in* situ measurements and also in automatic monitoring systems of soil salinity.

The *in situ* and non-destructive alternative is the measurement of the soil apparent electrical conductivity, EC_a, as the basis for determination of the electrical conductivity of the soil electrolyte, EC_w (Rhoades *et al.* 1976, Nadler 1981). For this purpose a four-electrode method is often used (Kirkham and Taylor 1949, Gupta and Hanks 1972). It consists in the measurement of the voltage drop between a pair of measuring electrodes placed in the current path, which is forced by other, pair of supply electrodes. Such a solution enables to avoid disturbances caused by electrochemical polarization of at the interface electrode-electrolyte. However the EC_a depends also on soil water content, θ, and to determine EC_w it is necessary to measure EC_a and θ, simultaneously. Rhoades *et al.* presented a model relating EC_a, EC_w and θ as follows:

$$EC_a = EC_w\theta(a_0\theta + a_1) + EC_s \tag{2}$$

where: a_0 and a_1 are empirical indexes different for various soils and profile layers of the given soil, EC_s, is the electrical conductivity of the soil solid phase dependent on the concentration of cations adsorbed on the surface of clay minerals and the presence of ferrite minerals.

2.4. Basics of TDR technique

Time Domain Reflectometry (TDR) method for the measurement of water content of isotropic and homogenous media becomes popular for the simplicity of operation, accuracy and the non-destructive, as compared to other methods, way of measurement (Malicki 1993, O'Connor and Dowding 1999). This measurement technique takes advantage of four physical phenomena characteristic to the soil:

- in the frequency range of 1 GHz the complex dielectric constant of the medium can be approximated by its real value and the electromagnetic wave propagation velocity, v, in the medium can be calculated from Eq. (3):

$$v = \frac{c}{\sqrt{\varepsilon(\theta)}} = \frac{c}{n} = \frac{2L}{\Delta t} \tag{3}$$

  where: c is a velocity of light in free space, $\varepsilon(\theta)$ is a real part of the complex dielectric constant dependent on its water content, θ; $n = \sqrt{\varepsilon(\theta)}$ is the medium refractive index; L is the length of TDR probe rods inserted into the soil; Δt is the time distance between the reflections of TDR pulse from the beginning and the end of the probe rods, inserted into the medium (Skierucha 1996),
- the dielectric constant of the medium liquid phase has much higher value than the other medium phases, *i.e.* about 80 against 2-4 for the solid and 1 for the gas phase,
- the relation between the water content of the medium and its dielectric constant is highly correlated for the majority of the porous media [Davis and Annan 1977, Malicki and Skierucha 1989, Skierucha 1996],
- the attenuation of the amplitude of electromagnetic wave traveling along the parallel transmission line inserted into the medium depends on its apparent electrical conductivity, EC (Dalton *et al.* 1984):

$$EC[\mathrm{Sm}^{-1}] = \frac{\sqrt{\varepsilon}}{120\pi L} \ln\left(\frac{U_{in}}{U_{out}}\right) \tag{4}$$

where: U_{in} and U_{out} are the amplitude of the pulse before and after attenuation caused by the pulse travel twice a distance of the probe length, L.

Therefore, water content in the soil, which is assumed to be an isotropic and homogenous medium, is the main reason determining its bulk dielectric constant.

The basic formula (3) is derived from more general one:

$$v = \frac{c}{\sqrt{\frac{\varepsilon'}{2}\left(1+\sqrt{1+\mathrm{tg}^2\,\delta}\right)}} \tag{5}$$

where: c is the light velocity of propagation in free space, ε' is the real value of the complex dielectric permittivity of the medium, tgδ is the dielectric loss defined as (Chełkowski 1993):

$$\mathrm{tg}\delta = \frac{\varepsilon'' + \frac{\sigma}{2\pi f \varepsilon_0}}{\varepsilon'} \tag{6}$$

The complex dielectric permittivity of the medium, ε, is:

$$\varepsilon = \varepsilon' - j\left(\varepsilon'' + \frac{EC}{2\pi f \varepsilon_0}\right) \tag{7}$$

where: ε'' represents dielectric loss connected with the dielectric polarization of soil particles, EC is the medium electrical conductivity, f is the frequency of the electromagnetic field, ε_0 is dielectric permittivity of free space, j jest equal to $\sqrt{-1}$.

2.5. General idea of the TDR measurements

The TDR method is at present the most popular dielectric method for the measurement of moisture in porous materials, soil in particular, ensuring very high repeatability and notable independence of results from the type of soil measured (Dirksen and Dasberg 1993, Robinson *et al.* 2003). After taking into account individual calibration for a given soil, it enables measurement accuracy at the level of 1%. The method is based on measurement of the speed of propagation of an electromagnetic pulse in a transmission line placed within the porous medium studied. The method requires high time resolution of measurement, of the order of 10^{-11} s, and complicated analysis of the reflectogram, taking into account the broad range of attenuation of signal and elimination of undesirable reflections caused by the non-homogeneity of soil. The high technical requirements cause that the commercially available instruments of this type are relatively expensive.

Figure 1 presents a pictorial diagram of a device for the measurement of the speed of propagation and attenuation of electromagnetic waves in porous materials, based on the reflectometric technique of measurement.

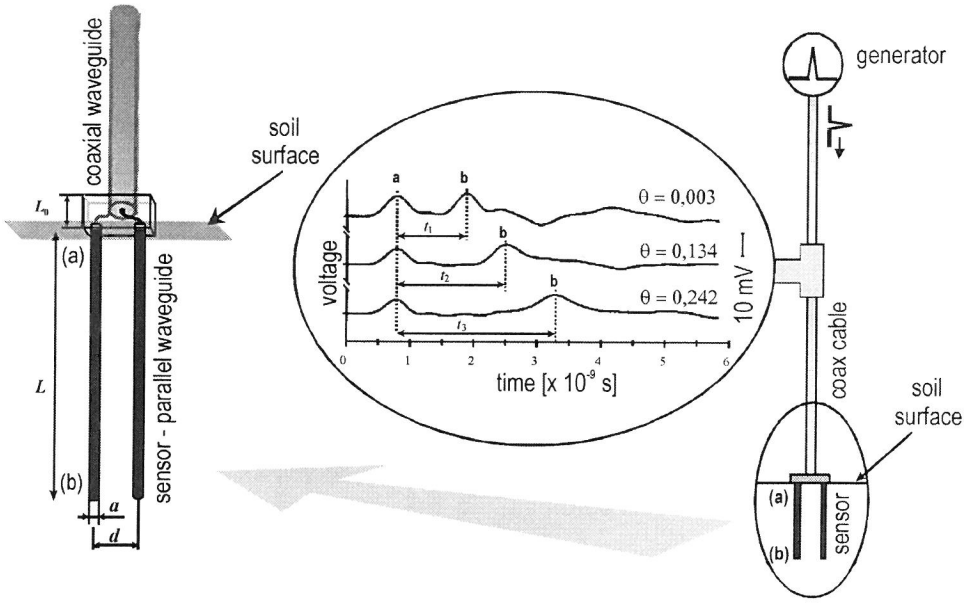


Fig. 1. Hardware setup for simultaneous measurement of soil water content and electrical conductivity using Time Domain Reflectometry method (Skierucha and Malicki 2004)

The idea of simultaneous measurement soil water content and electrical conductivity is presented in Figure 1. The TDR probe consists of two waveguides connected together: a coaxial one, called the feeder, and a parallel one, called the sensor, made of two parallel metal rods inserted into the measured medium.

A needle-type electric pulse, with pulse rise and fall time of about 250 ps, produced in a generator, runs along a 50 Ω coaxial cable to a measurement probe in the form of a waveguide with a dielectric of unknown permittivity. The waveguide can be formed of two (e.g. the FP/mts probe (Easy Test 2004), the probe used by Malicki and Skierucha (1989) or three e.g. the probes used by Heimovaara (1993), Wright and Or (1999), Zegelin *et al.* (1989) parallel rods of stainless steel, whose length equals x.

Between the generator and the probe there is the head of a stroboscopic oscilloscope sampling the condition of the transmission line. Due to the discontinuity of impedance between the coaxial cable and the probe, a part of the pulse from the generator is reflected, which is registered as (a). That part of the pulse that is not reflected continues along the probe rods and gets reflected on their ends (b).

Reflectograms obtained when the probe was placed in dry, wet and saturated sand indicate that time t between reflections (a) and (b) increases with increasing level of soil moisture. Assuming negligible dielectric loss of the medium, the

speed of propagation of the electric pulse along the probe rods can be expressed with the formula:

$$v = \frac{2x}{t_b - t_a} = \frac{c}{\sqrt{\varepsilon_b}} \tag{8}$$

The reason for that is the change of electromagnetic propagation velocity in media of different dielectric constants, according to Eq. (3).Therefore, having a probe that permits measurement of the time between two characteristic reflections of electromagnetic pulse, and knowing the length of probe rods x, we can use the method to determine the apparent permittivity of a material, ε_b. The volumetric moisture of the material, θ, i.e. the value which is the objective of reflectometric measurement, is determined from the empirical calibration $\theta_{TDR} = f(\varepsilon_b)$ or using dielectric mixing models.

Also, the amplitude of the pulse at the point (b) decreases with the increase of soil electrical conductivity, according to Eq. (4).

2.6. Calibration of TDR soil water content measurement

There are two approaches that allow calculating the soil water content from the TDR measurements: empirical and theoretical - using dielectric mixing models.

The results of TDR measurements of mineral, organic soils as well as mineral/organic mixtures are presented in Figure 2. The coefficients of linear regression equations fitted to empirical data points differ slightly showing that mineral composition of the soil significantly influences the TDR calibration curve: $n(\theta_{grav}) = a_0 + a_1\theta_{grav}$, where θ_{grav} is the soil water content determined by standard thermogravimetric method ISO 11461. Reconfiguration of the calibration curve allows the calculation of θ when TDR meter measures n. The accuracy of the TDR soil water content measurement is about ±2% of the true thermogravimetrically determined value, Eq. (9).

$$\theta = 0.13\sqrt{\varepsilon} - 0.18 \tag{9}$$

The most popular calibration curve for TDR soil (mineral soils) water content measurement was given by Topp *et al.* (1980) and is presented below:

$$\theta = -5.3 \cdot 10^{-2} + 2.92 \cdot 10^{-2} \cdot \varepsilon - 5.5 \cdot 10^{-4} \cdot \varepsilon^2 + 4.3 \cdot 10^{-6} \cdot \varepsilon^3 \tag{10}$$

The introduction in the Eq. (9) the correction on soil bulk density, ρ, that accounts for the influence of soil solid phase in the TDR readout, decreases the measurement error to about ±1% giving a new calibration curve presented by Eq. (11).

$$\theta = \frac{\sqrt{\varepsilon} - 0.57 - 0.58\rho}{7.75 + 0.92\rho} \tag{11}$$

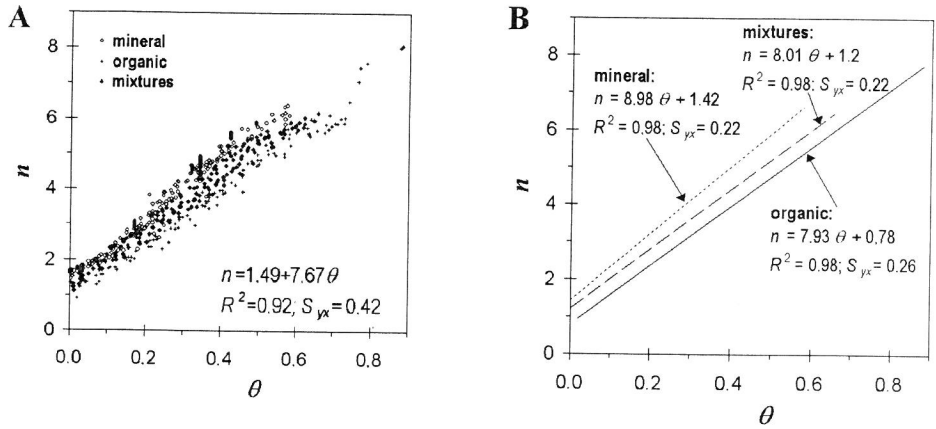


Fig. 2. Relation between the soil refractive index, n, and its water content, θ, for mineral and organic soil samples as well as their mixtures. A – experimental data, B – regression $n(\theta)$

Applying theoretical approach of the soil as a three phase mixture, its dielectric constant ε, can be presented as:

$$\varepsilon^{\alpha} = \theta \cdot \varepsilon_w^{\alpha} + (1-\phi) \cdot \varepsilon_s^{\alpha} + (\phi - \theta) \cdot \varepsilon_a^{\alpha} \tag{12}$$

where: ε, ε_w, ε_s and ε_a are the dielectric constants of soil as a whole, soil water, soil solids, and air, α is a constant interpreted as a measure of the soil particles geometry. On the base of the measured data collected on various soils, it has been found by Roth *et al.* (1990) that for the three phases dielectric model of the soil the average value of α constant is 0.5. For typical values of $\alpha = 0.5$, $\varepsilon_w = 81$, $\varepsilon_s = 4$, $\varepsilon_a = 1$ and $\phi = 0.5$ the Eq. (12) has the form of Eq. (13) with coefficients values close to the ones from Eq. (9).

$$\theta = 0.125 \cdot \sqrt{\varepsilon} - 0.19 \tag{13}$$

TDR method of simultaneous measurement of soil water content and electrical conductivity (salinity) becomes more popular because of its advantages: simplicity of operation, accurate and rapid readout, usually does not need calibration, non-destructive operation, availability of portable systems, availability to multiplex probes makeing automatic measurements possible. Although it has also disadvantages because it requires excellent probe to soil contact, probe installation may disturb soil, and TDR meters are still expensive, it may become the standard of

water content measurement applied not only for soil but also other porous materials like food products and building materials.

Analyzing of the TDR method waveform enables to obtain information on the spectrum of the dielectric permittivity of the soil (Lin 2003). For a step pulse used in the Tektronix 1502B device Heimovaara (1994) found the frequency band 0-1.5 GHz. For the device FOM / mts produced in IAPAN (Skierucha *et al.* 2006), an analysis of the Fourier spectrum of the needle pulse shows that the frequency band for which the signal amplitude does not fall below 3 dB is 1.42 GHz (Fig. 3).

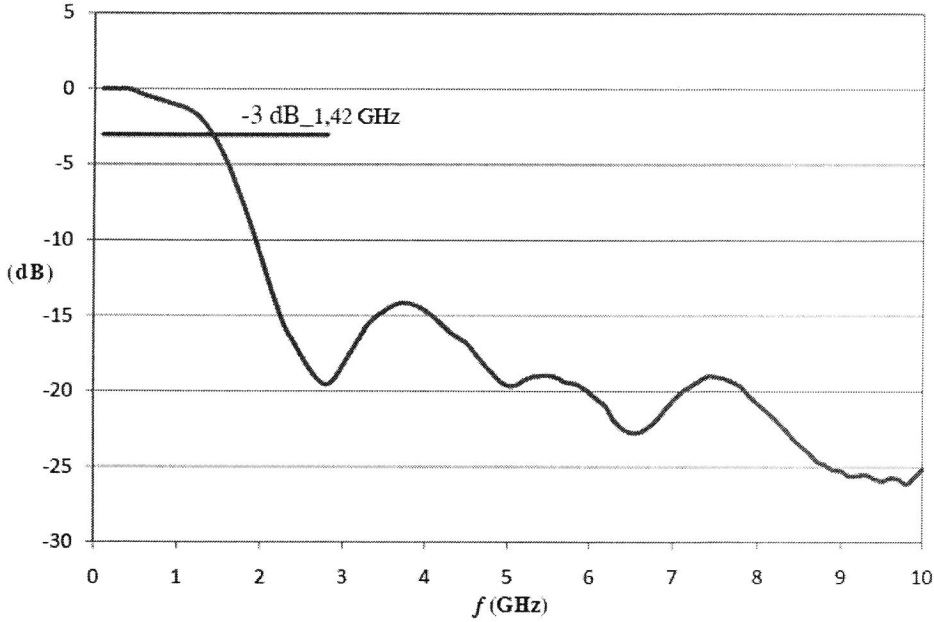


Fig. 3. Spectrum of the TDR pulse from FOM/mts device obtained by fast Fourier transform method

The spectrum of a Gaussian impulse should have the form of the Gaussian curve. In the pulse spectrum one can see characteristic maxima, decreasing with frequency, which indicate deformations of the impulse which in fact is not Gaussian. In spite of such a broad frequency band, Fourier analysis of reflectograms is hard to interpret as it requires the introduction of correction of amplitude-phase errors and the performance of calibrations used in studies involving the use of vector network analysers. Also a special design of the probe is required, minimising the occurrence of impedance discontinuity. Therefore, the determination of the frequency spectrum of the complex dielectric permittivity from analysis of time runs generated with the TDR method is burdened with a considerable error.

2.7. The influence of soil electrical conductivity on the soil water content values measured by TDR method

Topp *et al.* (1980) did not find the influence of soil salinity, or soil electrical conductivity, EC_a, on the electromagnetic wave velocity of propagation for mineral soils wetted with NaCl and $CaSO_4$ solutions of conductivity values practically found for soil solutions. However they found the increase of TDR measured water content, θ_{TDR}, measurement error for high saline soils. They explained this as a result of high attenuation of the TDR pulse and the problems with finding the correct reflection point.

Dalton *et al.* (1984) measured simultaneously water content and electrical conductivity in the range 0.03-0.14 (S m^{-1}) of saturated soil samples ($\theta = 0.34 \pm 0.01$) using the TDR method. They found that TDR determined soil water content is not influenced by the soil electrical conductivity.

Both components of the complex dielectric constant (7) can be represented by loss angle, δ, tangent of which is the ratio of the imaginary to real part.

$$\delta = \operatorname{arc\,tg}\left(\frac{\operatorname{Im}(\varepsilon)}{\operatorname{Re}(\varepsilon)}\right) \tag{14}$$

The frequency dispersion of the real part, ε', of the complex dielectric constant and the loss angle, δ, for the electrolyte having different conductivity values are presented in Figure 4. The figure, based on the Cole-Cole formulas, takes into account the electrical conductivity of the electrolyte ($EC_w/2\pi f\varepsilon_0$ from equation (7).

For the defined frequency range, dependent on the electrical conductivity, EC_w, of the applied electrolyte, the loss angle decreases from 90° to zero. Therefore for the frequency close to 1 GHz the complex dielectric constant of electrolyte reduces to its real part, ε', only when the tested material does not have too high value of electrical conductivity. In the electromagnetic field of high frequency the soil electrical conductivity does seem to influence the velocity of EM propagation. Figure 4 shows that in the defined frequency range the loss angle decreases to the values close to zero. This means that for the frequencies in this range the imaginary part of the complex dielectric permittivity (7) is negligible.

Consequently for the TDR measurement technique of soil water content determination based on the measurement of the electromagnetic wave propagation velocity in the soil can be simplified from Eq. (5) to Eq. (3). The following will practically verify these conclusions.

The soil material consisted of two soils (Tab. 1) and their mixtures prepared in the proportion, so as to have the density of soil samples equally distributed in the range 1.23-1.91 (g cm^{-3}).

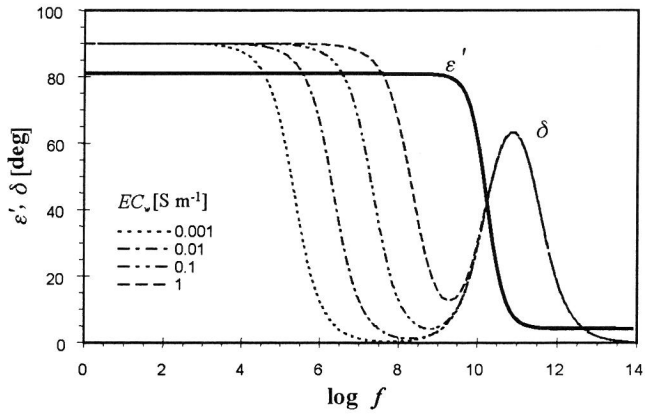


Fig. 4. Frequency dispersion of the real part, ε', of the complex dielectric constant and the loss angle, δ, for the electrolyte for its various electrical conductivities, EC_w

Table 1. The parameters of the soils used for the preparation of soil mixtures

Localization	Soil type (FAO)	Level	Depth (cm)	ρ (g cm^{-3})	ρ_s (g cm^{-3})	Solid texture (%) (FAO)			C(%)
						sand	silt	clay	
Markuszów	Orthic Podzol	Bh1	40-60	1.58-1.8	2.64	85	12	3	0.89
Janów Lub.	Eutric Cambisol	Bh	20-30	1.14-1.53	2.43	1	31	68	0.35

The measurements were performed in laboratory conditions. The measured soil parameters were: soil water content, θ, and bulk density, ρ, performed by thermo-gravimetric method; soil refractive index, $n = \sqrt{\varepsilon}$, and soil apparent electrical conductivity, EC_a, performed by the reflectometric method (Easy Test 2004); electrical conductivity, EC_w, of the solution of KCl used for wetting the soil samples.

The air dry soil was sieved through 2 mm holes and moistened by the KCl solutions of the conductivity values, EC_w (S m^{-1}): 0.00, 0.127, 0.488, 0.825 and 1.17. This way the soil apparent conductivity, EC_a, values were in the range 0.04-0.5 (S m^{-1}). The soil samples were moistened uniformly and were placed in glass containers cautiously to have the soil sample of uniform density.

The results of the measurements performed on the prepared soil samples can be presented in tabular form (Tab. 2) as (n, θ, ρ, EC_w). The influence of the soil electrical conductivity in the TDR refractive index determination can be checked using by multiple regression method.

$$n = a_0 + a_1\theta + a_2\rho + a_3 EC_w \tag{15}$$

where: $a_0 - a_3$ are empirical coefficients and EC_w is the electrical conductivity of the KCl solutions.

Table 2. The results of multiple regression method (15) Syx is the standard error of estimate

$a_3 \neq 0$ $n = a_0 + a_1\theta + a_2\rho + a_3 EC_w$		$a_3 = 0$ $n = a_0 + a_1\theta + a_2\rho$	
R^2	0.99	R^2	0.99
S_{YX}	0.138	S_{YX}	0.139
no of samples	225	no of samples	225
a_0	0.536	a_0	0.539
$\rho(a_0)$	<0.01	$\rho(a_0)$	<0.01
a_1	8.819	a_1	8.792
$\rho(a_1)$	<0.01	$\rho(a_1)$	<0.01
a_2	0.575	a_2	0.569
$\rho(a_2)$	<0.01	$\rho(a_2)$	<0.01
a_3	–0.031		
$\rho(a_3)$	0.164		

The results of R^2, S_{yx} and the coefficients a_0, a_1 and a_2 have similar values in the both cases. The *p-value* for a_3 $p(a_3)$>>0.01 and it can be concluded that the electrical conductivity of the solution (determining the soil salinity) influences the TDR determined soil refractive index in the statistically not significant way.

Consequently the TDR determined water content does not depend on soil electrical conductivity in the soil salinity range covered by the experiment.

Figure 5 presents the relation $n(\theta)$ for the analyzed soil samples wetted by the KCl solutions of different electrical conductivity values, EC_w. In the range of 0-1.17 (S m^{-1}), that is in the range corresponding to the natural soil salinity values, the soil electrical conductivity does not influence this relation. Practically this means, that reflectometric measurement of soil water content by the TDR method is not sensitive to the soil electrical conductivity, EC_a, and its salinity.

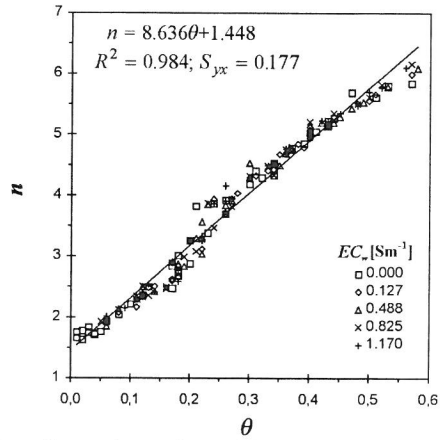


Fig. 5. The relation $n(\theta)$ for soil samples moistened by KCl solutions of different electrical conductivities, EC_w

2.8. Measurement of the spectrum of complex permittivity of porous materials in frequency domain – FDR method

At present, the most frequently used instrument permitting the measurement of the spectrum of complex permittivity is the vector network analyser (VNA). VNA is a measurement instrument permitting the measurement of complex amplitude-phase relations and transmitted sinusoidal signals of a set frequency between two or more input/output ports. It is most often used for the measurement of frequency characteristics of antennas and for the analysis of electronic microwave circuits. VNA is used in observing the frequency characteristics of electronic elements and telecommunication systems, as well as the dielectric spectroscopy tool for characterization of materials treated by electrical field of variable frequency. Increase in the frequency of the measurement signal causes a decrease in the accuracy of measurement of amplitude and phase. Therefore, in modern analysers operating in the range of high frequencies, of the order of GHz, a lowering of the frequency supplied to the detector is applied with the use of the process of mixing. Through that process, the resultant amplitude of the intermediate frequency signal is largely proportional to the amplitude of the high frequency signal and the phase relations are preserved.

The basic functional elements of a simplified VNA (Fig. 6) are the following: a PLL (Phase Locked Loop) frequency synthesiser, directional couplers, an amplitude and phase detector, and a control unit – a microcontroller connected to a PC (Agilent Application Note 2000). The operation of the analyser consists in the generation of a signal with a given frequency and supplying it to measurement port 1. Next, the directional couplers are used to isolate signals going towards the

port and the reflected signals. A typical analyser has two ports for which a four-element complex scattering matrix is defined, describing all measurement combinations of signals supplied to and reflected from the particular ports. For reflectometric measurements a single port is sufficient, for which only one element of the scattering matrix is measured – the coefficient of reflection S_{11}. Parameter S_{11} is a complex number which describes the ratio of the amplitude of the reflected signal to the supplied signal with their mutual shift in phase.

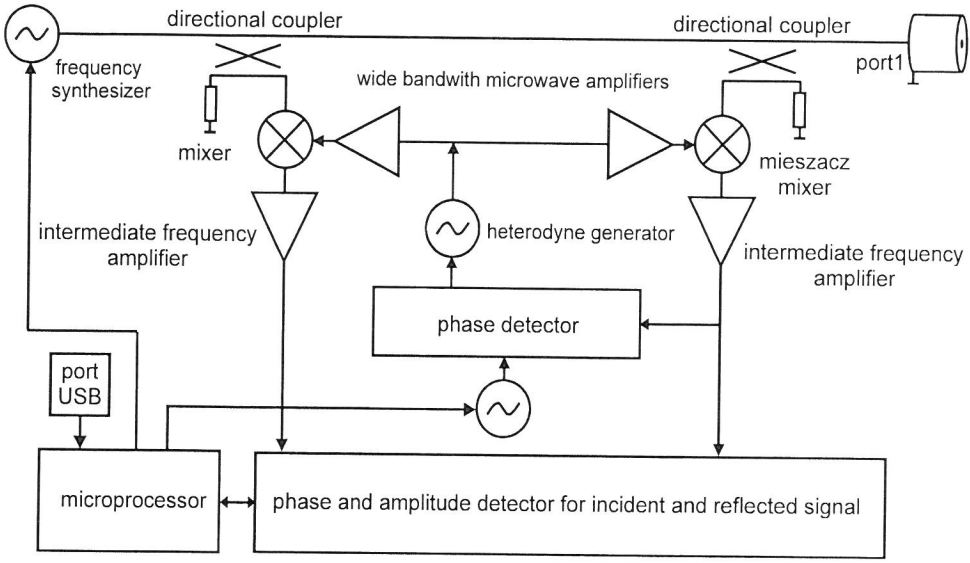


Fig. 6. Block Schematic of Vector Network Analyser VNA

One of the applications of the vector network analyser is dielectric spectroscopy (Logsdon 2005, Nelson 2005) which permits the study of dielectric properties of materials in the function of the frequency and amplitude of applied electric field in the form of a sinusoidal signal. In particular, the instrument is equipped with dedicated software and with probes or calibration-measurement sets (Agilent 85070D 2002) that permit the determination of the complex permittivity of the material studied. Only certain specialised types of analysers are factory equipped with such functionalities. The most frequently used measurement methods are: open-ended coaxial probe and the transmission method in a waveguide (Saed *et al.* 1989). The measuring capabilities of those instruments are limited to a specified volume or thickness of samples due to the need of placing them in the waveguide, or to a specific state of matter of the sample tested, e.g. measurements conducted exclusively on liquids. The software is strictly integrated with the set

of measurement probes and there is no possibility of transferring it to another analyser or of using it with non-typical probes in combination with an instrument from a specific manufacturer. The fundamental problem in the development of methods of dielectric spectroscopy as applied to the study of properties of porous materials is the lack – in the product range offered by manufacturers of VNA instruments – of suitable measurement probes that would permit non-destructive testing of large-volume samples, comparable to those measured with TDR probes within the frequency range of 0-1 GHz.

2.9. Open-ended coaxial probe

One of the methods of measurement of dielectric permittivity, with measurement signal frequency range of 200 MHz-20 GHz, is the method of open-ended coaxial probe extensively applied for the measurement of dielectric properties of liquids and homogeneous solids. At present, Agilent is the only company known to the author offering commercial probes of this type, cooperating with impedance analysers or with vector network analysers. An example here can be the set for the measurement of dielectric properties 85070E, working with vector network analysers series 87XXET/ES (Agilent 85070D 2002). Another design is the range of coaxial probes built on the basis of a modification of calibration air lines through their cutting and filling with a dielectric, or made of connectors of SMA or N types designed for the connection of microwave printed circuit boards and coaxial cables. Those probes and suitable electrical models were developed in the nineteen eighties and they were used primarily for the measurement of dielectric properties of biological materials and agricultural products within the range of radio and microwave frequencies (Gajda and Stuchly 1983, Skierucha *et al.*2005). Open-end coaxial probes do not measure permittivity directly, but the complex coefficient of reflection S_{11} converted to the impedance of the probe placed in the material tested. Only the process of calibration with the use of a suitable model and calibration points selected for it permits an accurate measurement of the permittivity of the material tested. The literature (Behari 2005, Bérubé *et al.* 1996) provides descriptions of numerous models of open-end coaxial probes, the most frequently encountered being the following:

- capacitance model (Application note 1287-2 2000),
- two-terminal-pair network type "T" (Yan-Zhen Wei and Sridhar 1989),
- antenna model (Stuchly *et al.* 1994),
- virtual line model (Ghannouchi and Bosisio 1989).

The measurement of dielectric properties of materials in frequency domain, with the application of microwave frequencies, needs expensive instrumentation. The basic elements of a simplified Vector Network Analyser (VNA), working

only in reflection mode, for determination of reflection parameter S_{11} is presented in Figure 6. The device consists of a very stable frequency synthesizer generating sinusoidal waveforms of variable frequency feeding a sensor – Open-Ended Coax probe connected to the output/input port of the VNA. For a defined frequency the directional couplers sense the amplitudes and the phases of the waveforms produced by the generator and reflected from the probe. The detected difference in amplitude and phase depends on the dielectric properties of the tested material at the end of the open-ended coaxial probe. The signals from the directional couplers are mixed with the signals from another generator, heterodyne, which frequency is adjusted by the phase detector. The reason for this frequency conversion is to have the constant frequency, not dependent on the synthesized frequency, of the signals reaching the measuring detector. The detected signals are characterized by constant frequency but their amplitudes and phase shifts do not change. The whole process of waveforms generation and detections is controlled by a microprocessor that changes the frequency of the synthesizer and registers the detected signals. All elements used in the presented system work in broad frequency range, from kHz to GHz, and they must have linear characteristics. Moreover, the measuring system accomplishes continuous recalibration to maintain parameters stable in time and temperature.

Fundamental to use the Open-Ended Coax probe (Fig. 7) is an accurate model relating the reflection coefficient at its end to the permittivity of the material contacting with the probe. The lumped capacitance circuit model described by Stuchly and Stuchly (1980) is applied in the presented example.

The capacitance model is built on the assumption that a probe placed in a dielectric with the complex permittivity ε can be described by means of a parallel connection of two capacities C_f and C_0.

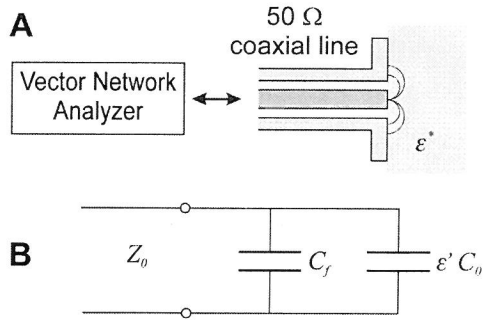


Fig. 7. A – Open-ended coaxial probe in the form of coaxial line open to the space with material of unknown dielectric permittivity ε^*, B – modeling the discontinuity of electromagnetic field by lumped capacitances C_f and C_0

Capacity C_0 describes a capacitor whose electric field lines close outside of the probe in the material tested. The capacity and the loss of that capacitor change in a manner corresponding to changes in real part, Re(ε), and the imaginary part, Im(ε), of dielectric permittivity ε. Capacity C_f, so-called fringe capacity, describes a capacitor whose field lines close within the dielectric of the probe. The values of parameters C_f and C_0 are determined for every measurement frequency in the process of calibration, based on a measurement in a dielectric with well characterised dielectric properties. Probe impedance Z_0 for parallel connection of the capacitors equals:

$$Z_0 = \frac{1}{j\omega(C_f + C_0\varepsilon)} \tag{16}$$

Knowing that the complex coefficient of reflection S_{11} measured with the vector analyser is given by the formula:

$$S_{11} = \frac{Z_p - Z_C}{Z_p + Z_C} \qquad \text{dla } Z_c = 50\Omega \tag{17}$$

Substituting relation (16) into (17) we can calculate the real and the imaginary parts of dielectric permittivity of a material from the formula below (Athey 1982):

$$\mathrm{Re}(\varepsilon) = \frac{-2\,|S_{11}|\sin\varphi}{\omega C_0 Z_C(1 + 2\,|S_{11}|\cos\varphi + |S_{11}|^2)} - \frac{C_f}{C_0} \tag{18}$$

$$\mathrm{Im}(\varepsilon) = \frac{1 - |S_{11}|^2}{\omega C_0 Z_C(1 + 2\,|S_{11}|\cos\varphi + |S_{11}|^2)} \tag{19}$$

where: Z_c – characteristic impedance of cable connecting probe the probe and the VNA, equal to 50Ω, f – frequency of electric field, ω – angular velocity equal to $2\pi f$, $|S_{11}|$,φ – module and phase of the complex coefficient of reflection S_{11} measured by the VNA.

The model considered does not take into account electromagnetic radiation increasing with the frequency of the measurement signal f. Such radiation causes overestimation of loss of capacitor C_0, which is reflected directly in an overestimated value of the imaginary part Im(ε) of dielectric permittivity of the material studied.

Before performing measurements on unknown materials the Open-Ended Coax probe should be calibrated on media with known dielectric properties, usually distilled water, methanol or air to find the values of C_f and C_0. The described measurement method and the lumped capacitance model were verified by measuring the dielectric permittivity of polytetrafluoroethylene PTFE (teflon). It was found that the C_f value is negligible and C_0 calculated from (19) equals to the C_0 coefficient of the correction polynomial obtained during calibration. The value of C_0 describes the geometry of the probe and the frequency range of its application. The larger dimensions of the probe, the bigger its value and the better accuracy in applications for the materials of small values of dielectric permittivity, and simultaneously the high frequency range of measurement is limited. This limitation comes from the fact that in case of materials of high dielectric permittivity and at high frequencies, the changes of the dielectric permittivity of the material causes very small changes of the phase shift of the reflection coefficient. This drastically decreases the accuracy of measurement and the need to apply calibration media of small value of dielectric permittivity.

Below the dielectric permittivity values of three soils were compared and Table 3 presents the basic physical parameters of these soils.

Table 3. Localization and selected physical parameters of tested soil samples

No.	Localization	Soil type	Density (g cm^{-3})	Soil texture ISSS			Specific surface (m^2 g^{-1})
				sand	silt	clay	
				2-0.02 mm	0.02-0.002 mm	< 0.002 mm	
611	Olempin	brown soil	1.59	94	5	1	9
606	Wólka kątna	muck	1.42	97	2	1	23
619	Saharyń	chernozem	1.16	87	12	1	69

Dry soils were mixed with appropriate amount of distilled water to achieve four samples for each analyzed soil with different water content values and taking care to get homogeneous distribution of water in the soil samples. Twelve containers with soil samples covered with plastic foil (to minimize evaporation) were left for 24 hours in room temperature for water distribution in the samples in natural way. The gravimetric water content, θ, and bulk density, ρ, were determined according to ISO standard 16586 (2003) for each soil sample directly after the dielectric permittivity measurements were completed. The values of ρ in Table 3 are the mean for all applied water contents for each tested soil. Soil texture was determined by standard Bouyoucos method (Turski 1993). The values of soil specific surface, S, was determined with water vapour adsorption method (Ościk 1983).

Time Domain Reflectometry (TDR) and Open-Ended Coax Probe methods of determination of the complex dielectric permittivity of porous materials were applied.

TDR measurements were performed using the oscilloscope frame HP54120 with the TDR unit HP54121A, featured by 20 ps rise-time step pulse. The TDR probe was based on the standard coaxial connector (type N jack) with three stainless steel wires soldered to the centre contact and two others symmetrically to the connector flange (Fig. 8A). The diameter of the rods was 2 mm, the distance to the central rod – 13 mm and the length of the rods – 114 mm. The characteristic impedance in air Z_0, of the parallel waveguide measured from the reflection coefficient by the TDR unit was 165 ohm (Agilent Application Note 2002).

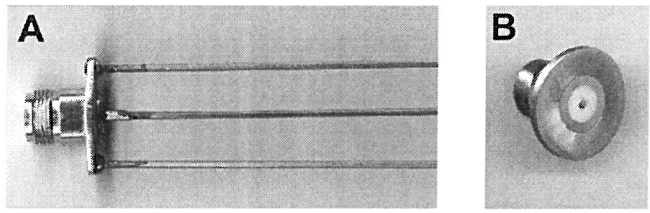


Fig. 8. The three-rod TDR probe – **A**, and an Open-Ended Coax probe – **B,** used in the measurement of dielectric permittivity of soils

The data from the Open-Ended Coax probe were collected by the 20kH-8GHz Rohde&Schwarz ZVCE Vector Network Analyser (Blackham and Pollard 1997, Stuchly and Stuchly 1980). It enables to calculate, on the base of the measured S_{11} parameter, the complex reflection coefficient and the complex admittance at the end of the probe. The probe was constructed on the base of type N coaxial connector machined flat at the side where it contacts with the tested material (Fig. 8B).

The Open-Ended Coax Probe method was verified by comparing the measured data to Cole-Cole model (Blackham and Pollard 1997). The Cole-Cole equation models the permittivity of free water and other polar substances:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + (j\omega\tau_{rel})^{1-\alpha}} \tag{20}$$

where: ε_∞ is the relative high frequency permittivity, ε_s is the relative static permittivity and $\tau_{rel} = 1/f_{rel}$ is the relaxation time (inverse of relaxation frequency f_{rel}) of orientation polarization defined as the time at which the permittivity equals $(\varepsilon_s + \varepsilon_\infty)/2$, α is an experimental correction. The Cole-Cole model values of $\varepsilon_\infty, \varepsilon_s, \tau_{rel}$ and α are 4.45, 33.7, $4.95 \cdot 10^{-11}$s and 0.036, respectively.

The comparison of measured and the modeled data, using Cole-Cole model of complex dielectric permittivity of methanol is presented in Figure 9.

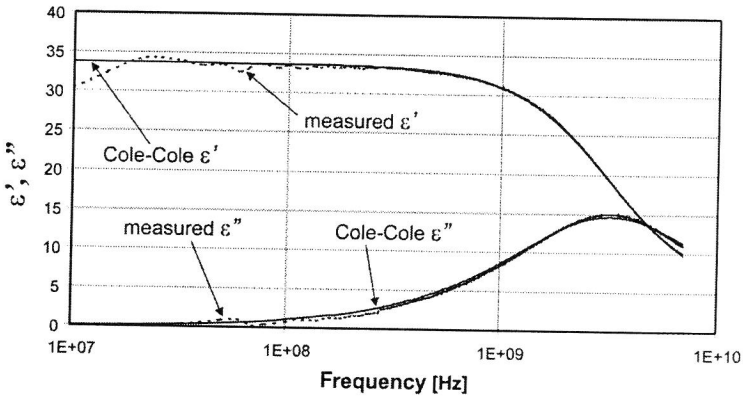


Fig. 9. Frequency change of real and imaginary parts of the complex dielectric permittivity of methanol for Cole-Cole modeled data and measured using open-ended coaxial probe

The measured and modeled data are very close to each other proving the applied measurement procedure to be adequate. The comparison of the measured real and imaginary parts of the complex dielectric permittivity of the analysed soils is presented in Figure 10.

The curves representing the relation $\varepsilon'(f)$ for soils of different water content, θ, determined by gravimetric method, are collected by the Open-Ended Coax probe. In the lower frequency limit of 10 MHz the real part of the complex dielectric permittivity is relatively high. This is attributed to the influence of ionic double layers associated with colloidal soil particles. This effect is often referred to as the "Maxwell-Wagner" effect (Hilhorst and Dirksen 1994). There was no orientation relaxation found in the measured frequency range from 10 MHz to 7 GHz because the tested soils have very small clay content. There is slight decrease of the measured ε' with the frequency increase.

The real part of dielectric permittivity of tested soils, $\varepsilon'_{\text{Open-Coax}}$, taken for the comparison with the soil bulk dielectric constant determined by TDR method, $\varepsilon_{b\text{-TDR}}$, are values for 1 GHz frequency. There is high correlation between the values $\varepsilon'_{\text{Open Coax}}$ and $\varepsilon_{b\text{-TDR}}$, except for high water content, where the measurements in the frequency domain show higher values than time domain measurements.

More differences between the measurements from the both probes can be found for the imaginary parts of the ε^*, where the values from TDR measurements were much lower than for frequency domain. This inconsistency may result from two reasons:

- problems during the calibration of the Open-Ended Coax probe (the Vector Network Analyser calibration kit was not perfectly fitted for the performed measurements),
- the assumption that the dielectric loss, ε_d in Eq. (21), was negligible in TDR measurements is false.

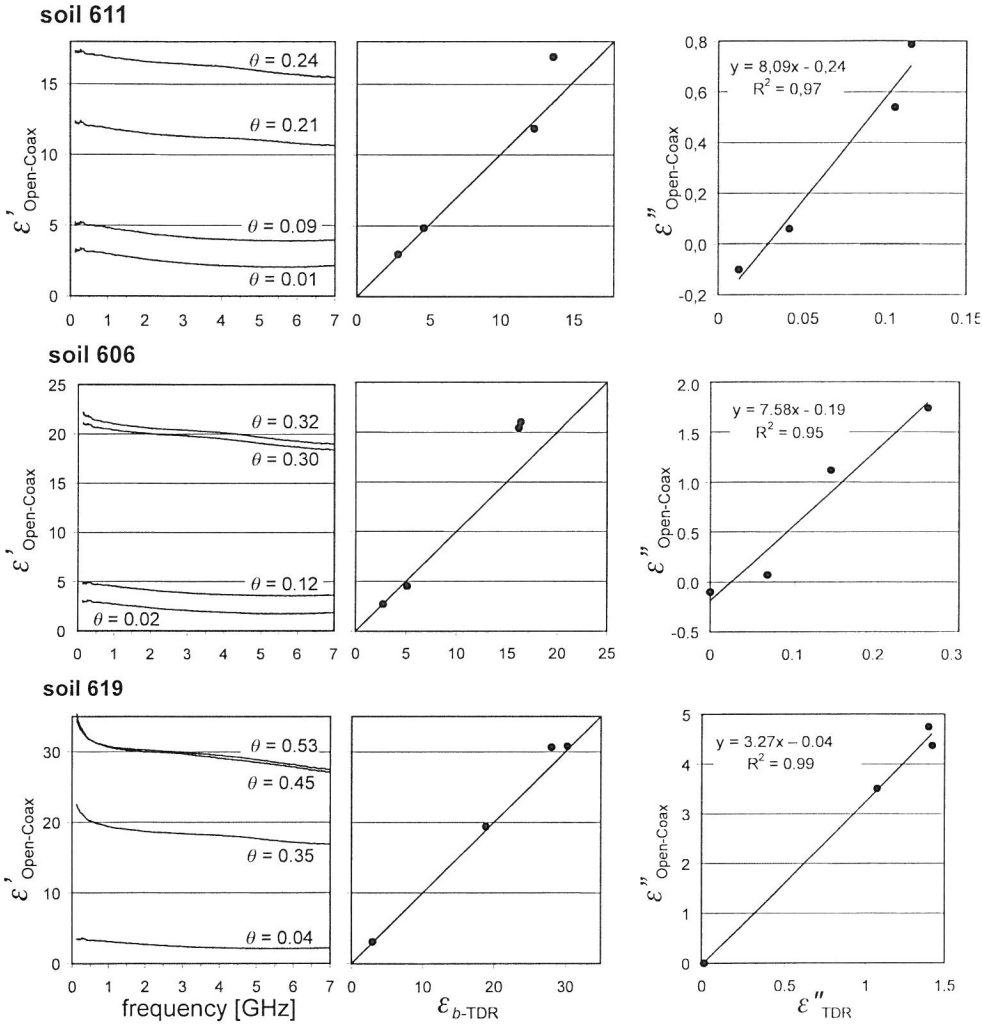


Fig. 10. Comparison of real, ε', and imaginary, ε'', parts of the complex dielectric permittivity for the selected soils, calculated from the TDR and Open-Ended Coax probe measurements. $\varepsilon_{b\text{-TDR}}$ is the bulk dielectric constant measured by TDR

The both methods of the determination of the dielectric permittivity of porous materials have advantages and drawbacks. The parallel waveguide can have the length ranging from 5 cm to 50 cm or more and the tested material constitutes its propagation medium, therefore as opposite to the Open-Ended Coax method, the propagation velocity is an average along the probe rods and the volume of measurement is much larger. For small samples of material or in cases when it is not possible to insert the rods into it, the Open-Ended Coax method is more suitable. The frequency of the TDR measured ε_b is not precisely defined, as it is a superposition of sinusoidal waves making a final step or a needle pulse. Also, for the frequencies in the range 0.5-1.5 GHz the real and imaginary parts of the dielectric permittivity do not change for the majority of agricultural materials. In the case of the Open-Ended Coax probe the user has the whole available frequency spectrum for analysis.

The meters for the TDR and Open-Ended Coax measurements must work in high frequency to sense the physical phenomena associated with dipolar polarization of water molecules. The TDR meter working in time domain (Fig. 1) is much simpler to construct as compared to the meter working in frequency domain (Fig. 6), although both require much effort and test instrumentation. The temperature and long-term stable frequency conversion in the required range of change, from kHz to GHz, need the selection of electronic elements, which requires time and investments. However, for the selected material only one frequency can be applied. The choice of this frequency can be determined by the preliminary tests with the use of a professional VNA. As the summary of this part, it can be concluded that:

- Real parts of $\varepsilon^*$of the tested soils, determined by the both measurement methods, are highly correlated and the measured values are close to each other. However for soils of high water contents the value of the real part ε^* determined by the Open-Ended Coax probe is higher than by TDR method. The imaginary parts are highly correlated but differ significantly.
- The difference of the imaginary parts of ε^* of the tested soils needs additional research for explanation, it may result from the not adequate calibration tools applied.
- Open-Ended Coax and Time-Domain Reflectometry methods need further developments in the field of modeling (to provide models of tested media and identify the measured quantities with indicators of material properties) and hardware (to select appropriate geometry of applied sensors for different porous materials and to decrease the price of the meters).

2.10. Multi-rod probe

Examples of commercially available two-rod probes include probes CS616, Campbell Scientific, or SM200, Delta–T Devices, and of multi-rod probes - probe ML2, Delta–T Devices, which are probes integrated with a gauge (Fig. 11).

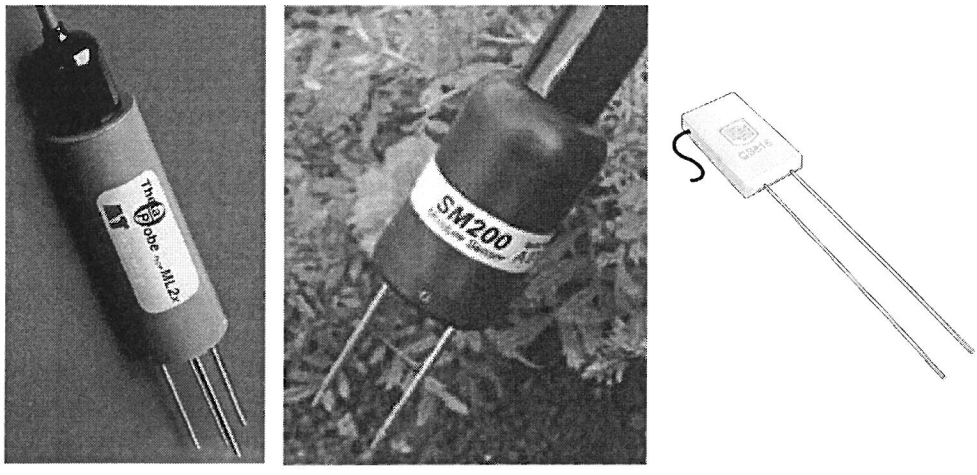


Fig. 11. Commercial milti-rod FDR probes

Probe CS616 (CS616 Instruction Manual, Campbell Scientific, 2006) with 30 cm rods length, is unsuitable for the measurement of moisture in soils with electrical conductivity exceeding the value of 0.6 dS/m. It makes use of delayed reflection of differential signal from probe ends, which triggers the generation of the next signal. This creates a generator whose vibration cycle is proportional to the volumetric moisture of the soil studied. The generated frequency, of the order of 10^8 Hz, is then brought down by means of frequency dividers to the range of 30-60 kHz so that it can be easily registered by the recorder. Probes SM200 (User Manual SM200, 2006) and ML2 (User Manual ML2x, 1999) work at a constant measuring signal frequency of 100 MHz and their operation is based on the principle of measurement of standing wave coefficient. This method does not provide satisfactory selectivity of measurement as for that frequency dielectric permittivity is modified by electrical conductivity, causing overestimation of the measurement results. The probes provide a voltage signal proportional to the root of dielectric permittivity, and after calibration – to the volumetric moisture of soil. The manufacturers recommend that the calibration of the volumetric moisture of soil should be performed with the oven-dry method. Measurements performed with

the probes described do not provide data for the acquisition of the frequency spectrum of the complex permittivity of soil.

One of the methods of measurement of the spectrum of the complex permittivity with a two-rod probe is the application of the TDR method and performing the Fourier transform, but this requires the knowledge of the exact form of the impulse supplied, so as to be able to determine the complex coefficient of reflection S_{11}. Next, with the help of suitable algorithms and calibrations (coaxial probe description – chapter 0) it is possible to determine the complex dielectric permittivity. This subject-matter is described in publications concerned with the calculation of dielectric permittivity from the spectrum of TDR signal (Lin 2003, Heimovaara 1994). Studies on the application of multi-rod probes for the measurement of the spectrum of the complex permittivity of soils in field conditions with the use of the FDR method have not been conducted to date.

2.11. Index of soil salinity

It is known that the imaginary part of dielectric permittivity for an electrolyte with a specific electrical conductivity (Hasted 1973) is equal to the sum of two components described by the formula:

$$\mathrm{Im}(\varepsilon) = \varepsilon_d + \frac{\sigma_{DC}}{2\pi f \varepsilon_0} \tag{21}$$

where: ε_d – dielectric loss, ε_0 – dielectric permittivity of vacuum, σ_{DC} – low frequency conductivity.

The value of dielectric loss ε_d results from the behaviour of polar molecules of water in a variable electric field. The behaviour of that component in the function of frequency depends on a number of factors, such as the amount of unbound water described with the Debye model, the amount of bound water dependent on the surface area of the solid phase of soil and the content of colloidal particles causing the Maxwell–Wagner effect. Molecules of bound water situated close to the slid phase of soil have limited freedom of movement due to the intermolecular effect which causes that a part of energy of electric field in transformed into heat. Dielectric loss for free water increases with increase in the frequency of electric field, whereas for bound water it decreases. For soil, in the frequency range of 10^8 MHz both processes may be balanced but also one of them can dominate over the other.

Another element of equation (21), dominating in the range of low frequencies, is the direct current conductivity σ_{DC} of the material studied, representing the ionic conductivity. That component decreases in reverse proportion to the frequency of external electric field.

The concept of the salinity index (*SI*) of soil has been introduced by Malicki (Malicki 1993) as the value of a partial derivative of the electrical conductivity of soil after dielectric permittivity (22). In that study the TDR method was applied. The apparent permittivity and the electrical conductivity of soil were determined from the rate of propagation of electromagnetic impulse and from its attenuation in a waveguide placed in soil, respectively. The salinity index as a function of electrical conductivity of soil electrolyte, does not depend on the volumetric moisture of soil.

$$SI = \frac{\partial \sigma_{DC}}{\partial \varepsilon} \tag{22}$$

In the study it was observed that *SI* is dependent on the texture of soil. The author gave also the relationship between soil salinity (taken as the electrical conductivity of soil electrolyte), salinity index, and the content of sand fraction in the soil.

Index *SI* is a convenient measure of soil salinity also in relation to other dielectric methods permitting the determination of complex dielectric permittivity, e.g. the FDR method.

3. DIELECTRIC MODELS

3.1. Debye's model

The Debye model (Chełkowski 1993) describes the frequency dispersion and absorption for molecules with a permanent dipole moment. On the base of the model it is possible to determine the behaviour of the complex dielectric permittivity in the function of electric field frequency. The Debye model assumes that the molecules are free and there is no interaction among them. A constant external electric field applied, acting on molecules with a permanent dipole moment, causes their orientation polarisation. That polarisation, however, is not instantaneous, as it depends on the inertia and temperature of the molecules. The thermal movement effectively counteracts the achievement of polarisation of all molecules simultaneously. Hence the temperature dependence of dielectric permittivity that decreases with increase if temperature. When the electric field disappears, the vector of orientation polarisation changes in time in an exponential manner according to the function described by the formula:

$$P(t) = P_s e^{-\frac{t}{\tau}} \tag{23}$$

where: P_s – vector of polarisation in constant electric field, t – time, τ – relaxation time.

The relaxation time depends on the temperature of the medium and on its viscosity. The maximum value of the polarisation vector is related with the dielectric permittivity of the medium. For low frequency fields, the polarisation vector is phase-coincident with the external electric field causing it. Dielectric permittivity is then a real value. With increase of frequency, a phase shift may appear between the direction of the polarisation vector of dipoles in the electric field and the direction of the electric field applied, related with the lag of the polarisation vector. Then the dielectric permittivity becomes complex in character, its imaginary part describing the thermal losses in the dielectric. The relation of the complex dielectric permittivity to the frequency of electric field for gas or for diluted solutions was given by Debye and is known as the Debye dispersion equation:

$$\varepsilon = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + j\omega\tau} \tag{24}$$

where: ε_s – dielectric permittivity for a static field, ε_{∞} – dielectric permittivity for field frequency above the relaxation frequency, f – electric field frequency, ω – angular velocity equal to $2\pi f$.

To obtain the frequency characteristics of dispersion one should consider the runs of the real and imaginary components of dielectric permittivity in relation to the frequency of the applied electric field. For water those characteristics are presented in Figure 12.

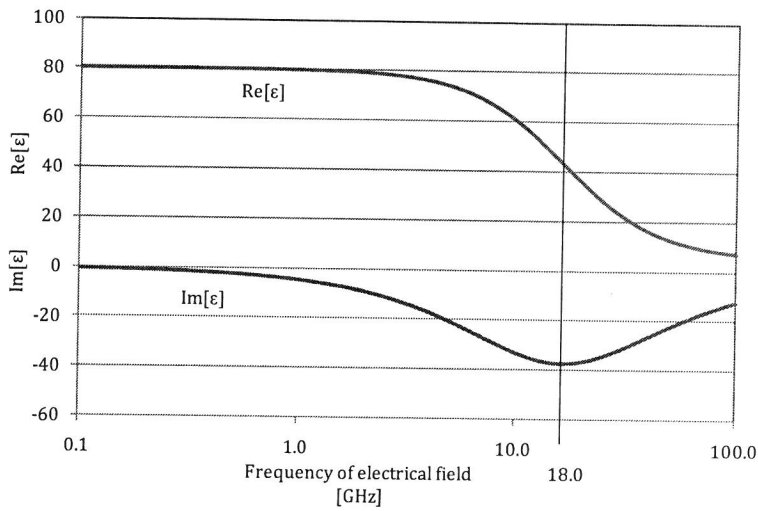


Fig. 12. Spectrum of both component of complex dielectric permittivity of water calculated from Debye'a model for coefficient from Table 6

The Figure shows the appearance of relaxation of water molecules for frequency of ca. 18 GHz, at which the maximum dielectric loss occurs, and a sudden decrease of the real part of dielectric permittivity of orientation polarisation. Above the relaxation frequency the orientation polarisation disappears.

3.2. Evaluation of dielectric mixing models of mineral soils

The increasing popularity of Time Domain Reflectometry (TDR) technique in the dielectric determination of soil water content (Or and Wraith 1999, Topp *et al.* 1988) implies the necessity of verification of the dielectric mixing models. The commonly used models describing the soil dielectric constant as the mixture of solid matter, water and air are: α model (Birtchak 1997, Bohl *et al.* 1994, Steru 1959), the model proposed by *de Loor* (1964) and empirical models (Malicki and Skierucha 1989, Malicki *et al.* 1996, Skierucha 1996, Topp *et al.* 1980).

The soil water content sensor used in TDR method and presented schematically in Figure 13, consists of two parallel metal rods or three metal rods (Heimovaara 1994, Wright and Or 1999, Zegelin *et al.* 1989) of the length L, inserted into the tested soil (Gardner *et al.* 1991, Heimovaara 1993). The time, t, for the electromagnetic (EM) wave to cover the distance L may be calculated as the sum of propagation times along the individual soil phases (Kraszewski *et al.* 1976):

$$t = t_s + t_a + t_w \tag{25}$$

where the indexes s, a and w stand for solid, gas and water phases, respectively.

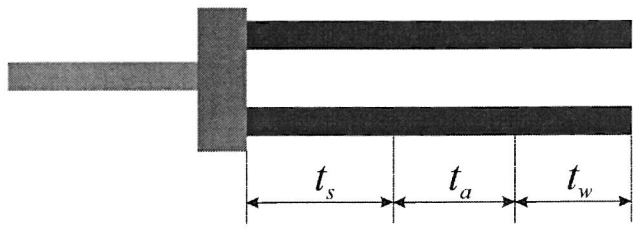


Fig. 13. TDR interpretation of soil as a tree phases medium in dielectric mixing model α

In most practical conditions the velocity of EM wave propagation, v, in the soil may be presented as:

$$v = \frac{c}{\sqrt{\varepsilon}} = \frac{2L}{t} \tag{26}$$

where c is the velocity of light in free space, ε is the soil dielectric constant. The total propagation time, t, of EM wave can be expressed by dividing it into ele-

mentary propagation times along the solid, water and air phases of the soil expressed by (25) by means of (26):

$$t = t_s + t_a + t_w = \frac{2L\sqrt{\varepsilon}}{c} = \frac{2L_s\sqrt{\varepsilon_s}}{c} + \frac{2L_a\sqrt{\varepsilon_a}}{c} + \frac{2L_w\sqrt{\varepsilon_w}}{c} \tag{27}$$

with: L_s, L_a, L_w are idealized by the presented model parts of the probe inserted into an elementary soil phase. After conversion the Eq. (27), multiplying by the elementary cross-section S and then dividing by the whole volume of soil in the cylinder defining the sphere of influence of the TDR measurement method one receives:

$$\sqrt{\varepsilon} = \sqrt{\varepsilon_s}\frac{V_s}{V} + \sqrt{\varepsilon_a}\frac{V_a}{V} + \sqrt{\varepsilon_w}\frac{V_w}{V} = \sqrt{\varepsilon_s}(1-\phi) + \sqrt{\varepsilon_a}(\phi-\theta) + \sqrt{\varepsilon_w}\theta \tag{28}$$

or using a parameter α, which summarizes the geometry of the medium with relation to the applied electric field (Roth *et al.* 1990):

$$\varepsilon = \left((1-\phi)\varepsilon_s^{\alpha} + (\phi-\theta)\varepsilon_a^{\alpha} + \theta\,\varepsilon_w^{\alpha}\right)^{\frac{1}{\alpha}} \tag{29}$$

which is the 3-phase dielectric mixing model α, expressing the square root of the apparent dielectric constant of soil in the terms used by soil scientists: ϕ – soil porosity and θ – volumetric water content. Whalley (1993) presented the similar discussion.

Following Birchak *et al.* (1974), Alharthi and Lange (1987) who assumed $\alpha = 0.5$, gives the Eq. (28). After introducing adsorbed water as a fourth phase (Dobson *et al.* 1985) the authors determined α by regression data for different frequencies (ranging from 1.4 to 18 GHz) and soil types (ranging from sandy loam to silty clay) and obtained $\alpha = 0.65$. The four-phase dielectric mixing model α, accounting for the bound water adsorbed to soil solid phase has the following form:

$$\varepsilon^{\alpha} = (1-\phi)\varepsilon_s^{\alpha} + (\phi-\theta)\varepsilon_a^{\alpha} + (\theta-\theta_{bw})\varepsilon_w^{\alpha} + \theta_{bw}\varepsilon_{bw}^{\alpha} \tag{30}$$

where water exists in two phases: as free water, w, like capillary water and bound water, bw, like molecular or film water. Generally the α-model of soil dielectric mixture may be presented as follows:

$$\varepsilon^{\alpha} = \sum_i V_i \varepsilon_i^{\alpha} \tag{31}$$

where V_i is the volumetric concentration of i-th phase and ε_i is the dielectric constant of this phase.

The four-phase model proposed by *de Loor* (de Loor 1964,1990) and used by Dirksen and Dasberg (1993) and Dobson and Ulaby (1885) is:

$$\varepsilon = \frac{3\varepsilon_s + 2\theta_w(\varepsilon_w - \varepsilon_s) + 2\theta_{bw}(\varepsilon_{bw} - \varepsilon_s) + 2\theta_a(\varepsilon_a - \varepsilon_s)}{3 + \theta_w\left(\frac{\varepsilon_s}{\varepsilon_w} - 1\right) + \theta_{bw}\left(\frac{\varepsilon_s}{\varepsilon_{bw}} - 1\right) + \theta_a\left(\frac{\varepsilon_s}{\varepsilon_a} - 1\right)} \tag{32}$$

where the indexes s, w, bw and a refer to solid phase, water, bound water and air, respectively. This model describes heterogeneous mixture where foreign granules with dielectric constant ε_i are imbedded in a homogeneous and isotropic dielectric with dielectric constant ε. The imbedded foreign granules in Eq. (32) represent free water, bound water and air. The host medium is the solid phase.

The empirical models $\varepsilon(\theta)$ and $\varepsilon(\theta,\rho)$, where ρ is the soil bulk density, form the formula of the appropriate regression curves (Malicki *et al.* 1996, Malicki and Skierucha 1989, Skierucha 1996, Topp *et al.* 1980). The formula (33) presented below and discussed in Skierucha (1996) will be used in this paper as the reference:

$$\varepsilon = \left(0{,}573 + 0{,}582\rho + (7{,}755 + 0{,}792\rho)\,\theta\right)^2 \tag{33}$$

where ρ (g cm^{-3}) is the soil bulk density.

The verification of the discussed 3-phase and 4-phase models is presented in the following part.

Sixteen mineral soils of different texture, volumetric water content and bulk and particle density were investigated (Tab. 4).

The following variables were measured: dielectric constant, ε, bulk density, ρ, particle density, ρ_s, and gravimetrically determined volumetric water content, θ. Dielectric constant, ε, was determined using TDR (LOM/m from *Easy Test, Ltd.* Easy Test 2004). The remaining variables were measured with the application of standard methods.

The solid phase dielectric constant, ε_s, was calculated from the formula (derived from the formula (29)):

$$\varepsilon_s = \overline{\varepsilon_s} = \frac{1}{n}\sum_i^n \left(\frac{(\varepsilon(\theta = 0))^\alpha - \phi_i}{1 - \phi_i}\right)^{\frac{1}{\alpha}} \tag{34}$$

where α = 0,5.

The solid phase dielectric constant was calculated on the base of three-phase model α applied to all soil samples from table 4 fitting the exponential function to the experimental data.

Table 4. Selected properties of the investigated mineral soils, where ρ (g cm^{-3}) is the soil bulk density, ρ_s (g cm^{-3}) is the soil particle density

No	Localization	Soil type (FAO)	Level (depth in cm)	ρ (g cm^{-3})	ρ_s (g cm^{-3})	Texture % (FAO)		
						Sand	Silt	Clay
1	Parana. Brazil	Haplic Ferrasol	Ah (0-10)	1.27-1.60	2.62	82	6	12
2	Parana. Brazil	Haplic Ferrasol	Bws (120-140)	1.31-1.77	2.67	72	8	18
3	Grunewald. Germany	Haplic Luvisol	Bt (90-120)	1.35-1.55	2.53	2	75	23
4	Grunewald. Germany	Haplic Podzol	Bhs (30-45)	1.21-1.60	2.54	88	10	2
5	Elba. Italy	Chromic Luvisol	Ah (0-20)	1.34-1.64	2.63	46	31	23
6	Elba. Italy	Chromic Luvisol	Bt (150-180)	1.33-1.73	2.65	35	21	34
7	Elba. Italy	Luvic Calcisol	Ah (0-20)	1.25-1.68	2.57	50	26	24
8	Elba. Italy	Luvic Calcisol	Btk (80-100)	1.14-1.52	2.53	26	28	46
9	Werbkowice. Poland	Haplic Chernozem	Ap (0-20)	1.18-1.40	2.28	2	52	46
10	Parana. Brazil	Rhodic Ferrasol	Ah (0-10)	1.01-1.37	2.88	4	16	80
11	Parana. Brazil	Rhodic Ferrasol	Bws (120-160)	1.04-1.24	2.88	3	16	81
12	Ohlendorf. Germany	Haplic Luvisol	Bt (60-80)	1.31-1.62	2.70	2	75	23
13	Ohlendorf. Germany	Arenic Cambisol	C (70-120)	1.59-1.70	2.63	98	2	0
14	Markuszów. Poland	Orthic Podzol	Bh1 (40-60)	1.58-1.80	2.64	85	12	3
15	Czesławice. Poland	Orthic Luvisol	Ck (150)	1.25-1.61	2.48	0	68	32
16	Janów Lubelski. Poland	Eutric Cambisol	Bh (20-30)	1.14-1.53	2.43	1	31	68

For dry soil ($\theta = 0$) only solid and air phases are present, therefore knowing the porosity of the most dry soil from the tested samples it is possible to estimate the ε_s as the average of all tested mineral soil samples according to Eq. (34).

This extrapolation gives the average value of dielectric constant soil solid phase equal to 4.72, which is in agreement with the published data (Dirksen and Dasberg 1993, Friedman and Robinson 2002). The detailed statistics of the performed calculations are in Figure 14.

It is assumed that for each soil type there is a transition water content value, θ_{WP}, which makes the distinction between soil water as bound or free. This water content can be determined from the empirical formula (Wang and Schmugge 1980) as:

$$\theta_{WP} = 0{,}0674 - 0{,}00064 \cdot SAND + 0{,}00478 \cdot CLAY \qquad (35)$$

where *SAND* and *CLAY* are percents of sand and clay in the tested soils. The index *WP* stands for wilting point as Wang and Schmugge correlated the transition water content with the wilting point water content.

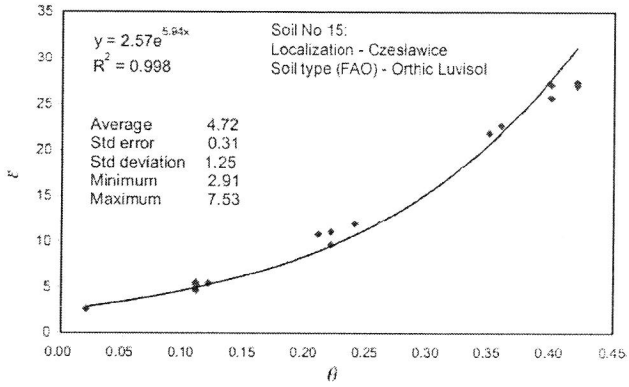


Fig. 14. Determination of the dielectric constant of soil solid phase using extrapolation and three-phase soil dielectric mixing model α

Further discussion concerning 4-phase models assumes:

- If $\theta > \theta_{WP}$, the soil dielectric constant is represented by the 3-phase models, that is the formula (29) for the α-model, and the formula (32) for the *de Loor* model at $\theta_{bw} = 0$.
- If $\theta > \theta_{WP}$, the soil dielectric constant is represented by the 4-phase models, that is the formula (30) for the α-model, and the formula (32) for the *de Loor* model, at $\theta_{bw} = \theta$ and $\varepsilon_{bw} = \varepsilon_s + (80{,}2 - \varepsilon_s)\frac{\theta_{bw}}{\theta_{WP}}$, where ε_s is the soil solid phase dielectric constant, according to (34) for $\alpha = 0{,}5$.

The relation between the soil dielectric constant, ε, and soil water content, θ, is presented in Figure 15. It comes that for water content below 0.3 the values calculated from the empirical model (33) are higher than the measured ones. This is particularly evident for samples with high clay content (Tab. 4, no. 10, 11 and 16), *i.e.* for clay content more than 60%. High clay content is positively correlated with the amount of bound water surrounding soil solid particles (Or and Wraith 1999). These particles of water have the dielectric constant lower than that of free water. Therefore the model (29) is not adequate for clayey soils.

The value of dielectric constant of bound water, ε_{bw}, increases with the distance from the solid particles. Dirksen and Dasberg (1993) confirmed this for soils of high specific surfaces.

The comparison of ε values calculated from the 3-phase model (29) and measured by TDR device is presented in Figure 16. There are two distinct ranges of ε, below and above the transition value of $\varepsilon_{TDR} \cong 15$, and this distinction generally does not depend on the value of the parameter α.

Below the transition value, the soil dielectric constants values calculated from the model are higher than the measured ones. For each α parameter the distribution of data is similar ($R^2 = 0.96$), the slope of the trend line is near 1 for $\alpha = 0.6$. For ε greater than the transition value, the 3-phase α model generates numbers close to the measured ones.

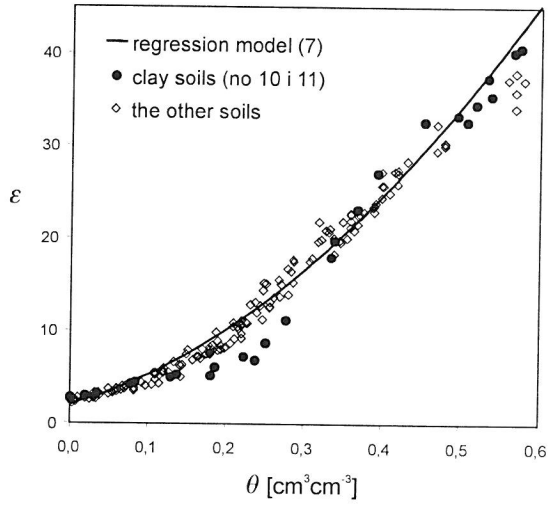


Fig. 15. Relation between the soil relative dielectric constant, ε, and soil volumetric water content, θ, for the investigated mineral soils. The solid line represents the empirical model (33) calculated for the average bulk density of the soil samples 1.42 (g cm^{-3})

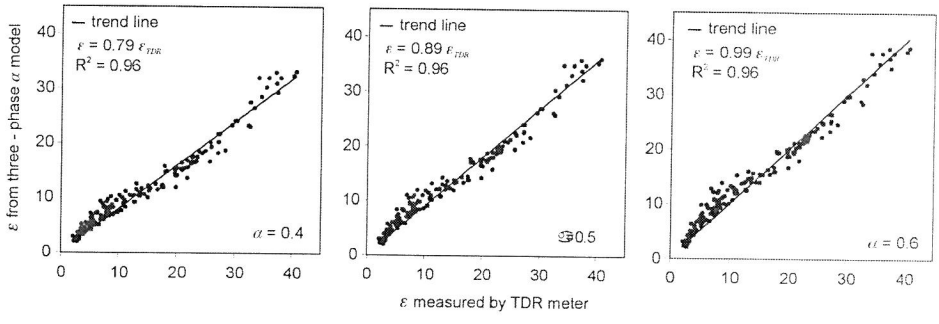


Fig. 16. Comparison of soil relative dielectric constants of the investigated soil samples measured by TDR method (horizontal axis) and calculated from 3-phase model α (29) for different values of α parameter

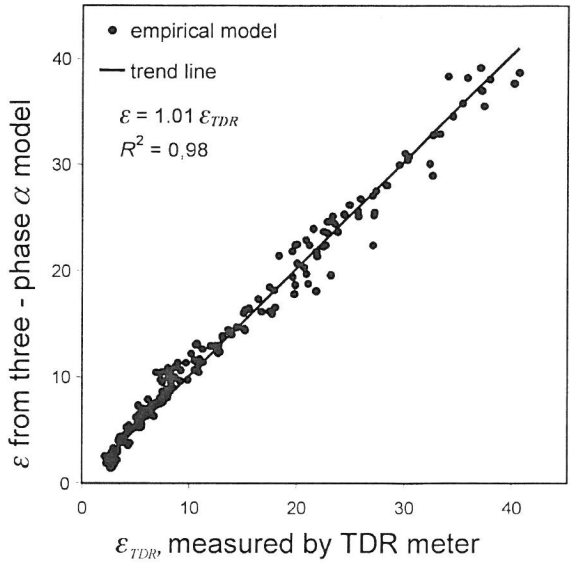


Fig. 17. Comparison of the soil dielectric constants measured by TDR method (horizontal axis) and calculated by the empirical model (33) accounting for soil bulk density

The empirical regression model (33), accounting for the soil density, does not eliminate the characteristic decline of data around the transition value of ε, although it makes it smaller (Fig. 17)

The inclusion of soil bulk density to the empirical model of $\varepsilon(\theta)$ significantly increases the accuracy of TDR soil water content determination (Malicki *et al.* 1996, Skierucha 1996).

The 4-phase models (30) and (32) account for the presence of soil bound water, θ_{bw}, the magnitude of which can be expressed as:

$$\theta_{bw} = x \cdot S \cdot \rho \tag{36}$$

where x(m) is the distance from the solid phase surface and S [m^2/g] is the soil specific surface depending mainly on the soil clay content. According to Or and Wraith (1999) and Sposito (1982) the bound water is formed by no more than 3 monomolecular layers of water, where the single layer thickness is $3 \cdot 10^{-10}$ (m).

The dielectric constant of the bound water is lower than that of capillary water because of hindering effect of solid phase induced to water dipoles. The relation between the bound water dielectric constant and the distance from the solid phase surface is unknown.

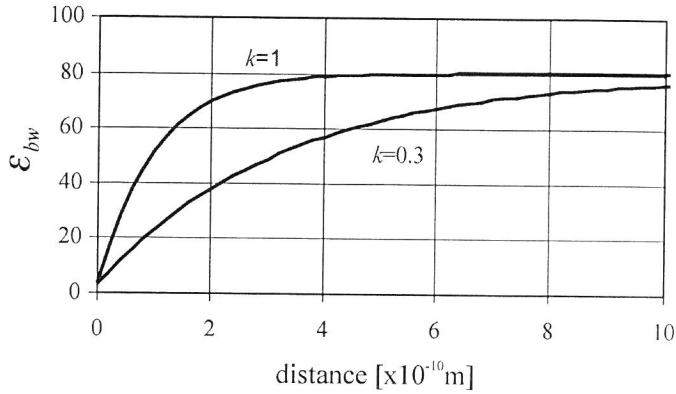


Fig. 18. Values of dielectric constant of bound water, ε_{bw}, related to the distance, x, from the phases interface

Or and Wraith (1999) assume the exponential function, similar to the presented below (Fig. 18):

$$\varepsilon_{bw} = 80{,}2 - 77 \cdot e^{-kx} \tag{37}$$

where k is the parameter influencing the change of ε_{bw} with the distance of water particles from the phases interface.

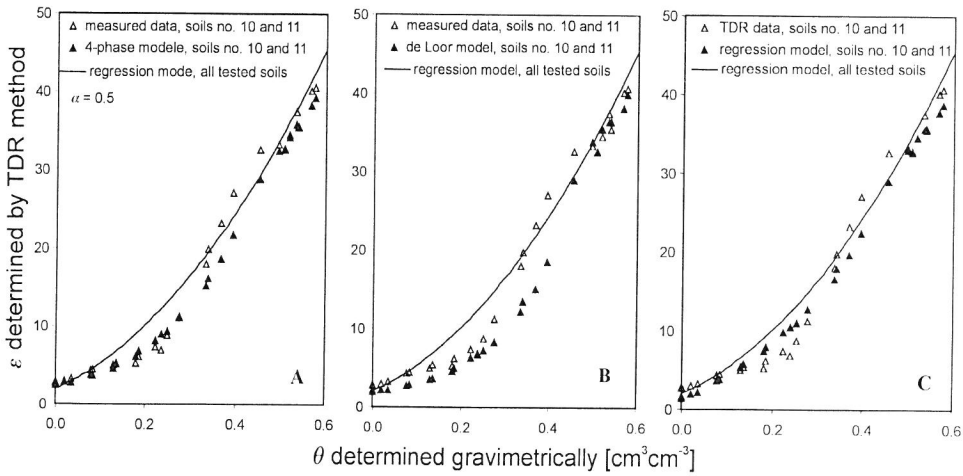


Fig. 19. Effects of the 4-phase models for the investigated clay soils (Tab. 4, no. 10 and 11): A – for model α, B – for *de Loor* model and C – effect of regression model (33) accounting for soil bulk densities, the solid line represents model (33) for $\rho = 1{,}42$ (g cm^{-3})

To verify the observations concerning the influence of bound water on the $\varepsilon(\theta)$ relation the soils 10 and 11 (Tab. 4) are examined. They have the highest clay content from among the investigated soils and the transition water content is distinct on the $\varepsilon(\theta)$ curve (Fig. 15).

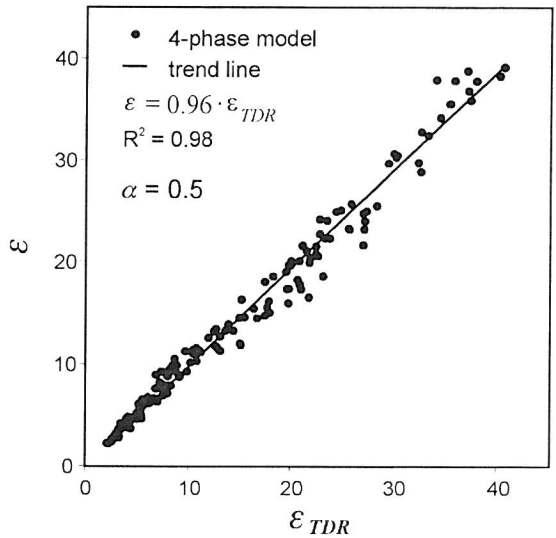


Fig. 20. Comparison of the analysed soil dielectric constant measured by TDR method (horizontal axis) and calculated from the 4-phase model α

The value of transition water content, θ_{WP}, is calculated from the formula (35) presented after Wang and Schmugge (1980). The assumptions presented earlier are applied to 4-phase models (30) and (32) for two selected soils (Fig. 19).

The regression model shows higher values as compared to the measured ones for water contents below 0.35 (Fig. 19C).

The data calculated from the modified 4-phase models follow the measured data in the range where the regression model produces higher values. In the range of $\varepsilon < 15$ the 4-phase models work in agreement with the TDR measured data. Better correlation is achieved after application of α model than the model of *de Loor*. For all tested soils the values of ε produced by 4-phase modified α model as compared to the TDR measured ones are presented in Figure 20.

Scatter of data in Figure 20 is comparable with that from the empirical model (33) accounting for the soil bulk density. It can be concluded that:

- Effect of bound water on the dielectric mixing model can be accounted for if clay content is known.

- The modified model α with the clay content as the corrective parameter of bound water works equally well as the regression model with soil bulk density as the corrective parameter.

4. MODELS DESCRIBING MEASUREMENT PROBES IN BROADBAND FREQUENCY DOMAIN

Measurements with the TDR method provide information on the apparent dielectric permittivity and on the electrical conductivity of soils, with their values being averaged for a broad range of frequency. To obtain accurate frequency relations of the complex dielectric permittivity it is necessary to apply the FDR technique and models of suitable measurement probes, which is described below.

4.1. Coaxial open-ended probe modeled as a two-terminal-pair network type T

The model of two-terminal-pair network type T (Wei and Sridhar 1989) is a modified capacitance model, in which capacity C_f was replaced with a two-terminal-pair network type T built of three impedances Z_1, Z_2, Z_3 (Fig. 21). The application of such a system was based on the properties of a two-terminal-pair network type T used as a fitting system of transmission antennas. As applied for a coaxial probe, it permits the elimination of such sources of error as the attenuation effect and the phase shift of the cable and the connectors between the vector analyser and the probe.

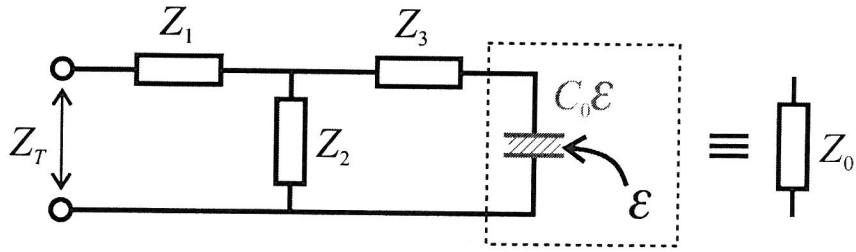


Fig. 21. Two-terminal-pair type T model of a coaxial open-ended probe

The impedance of capacitor C_0 filled with a lossy dielectric can be described as:

$$Z_0 = \frac{1}{j\omega C_0 \varepsilon} \tag{38}$$

The equivalent impedance Z_T of a serial-parallel connection (Horowitz and Hill 1997) can be described as:

$$Z_T = Z_1 + \frac{Z_2(Z_3 + Z_0)}{Z_2 + Z_3 + Z_0} \tag{39}$$

Next, the formula can be transformed to the form of a linear equation:

$$a + bZ_0 - cZ_T = Z_T Z_0 \tag{40}$$

gdzie $a=Z_1Z_2+Z_2Z_3+Z_3Z_1$, $b=Z_1+Z_2$, $c=Z_2+Z_3$ are complex coefficients.

Knowing the impedance Z_0 is dependent on dielectric permittivity in a manner described by formula (38), for air ($\varepsilon_{air}=1+j0$) it assumes the form:

$$Z_{air} = \frac{1}{j\omega C_0} \tag{41}$$

Substituting (41) to (38), impedance Z_0 can be written as:

$$Z_0 = Z_{air}\frac{1}{\varepsilon} \tag{42}$$

Formula (40) can be transformed so that its complex equation coefficients will also include impedance Z_{air}.

$$a' + \frac{b'}{\varepsilon} + c' Z_T = \frac{Z_T}{\varepsilon} \tag{43}$$

where:

Z_T – equivalent impedance of two-terminal-pair network type T measured with a VNA, ε – complex dielectric permittivity of the material studied.

To determine parameters a', b', c' we need to build and solve a system of three equations (43), using the data obtained from measurements of impedance of the modelled open-ended coaxial probe at three calibration points described below. The probe impedance Z_T is determined from measurements of the complex coefficient of reflection S_{11} with a vector analyser, based on the formula:

$$Z_T = Z_C \frac{1+S_{11}}{1-S_{11}} \tag{44}$$

where: Z_C – characteristic impedance of connecting cable between the probe and the VNA ($Z_C = 50\ \Omega$).

Most frequently adopted calibration points are the following:

- probe shorting, e.g. with mercury, for which $\mathrm{Re}(\varepsilon) = \infty$, $\mathrm{Im}(\varepsilon) = \infty$ was adopted,
- measurement in the air – $\mathrm{Re}(\varepsilon) = 1$, $\mathrm{Im}(\varepsilon) = 0$,
- measurement in a liquid with known dielectric properties described with the models of Debye or Cole–Cole, e.g. distilled water, acetone or methanol.

In the process of calibration the parameters a', b', c' are calculated, on the basis of which the complex dielectric permittivity of studied material is determined in accordance with the transformed formula (43):

$$\varepsilon = \frac{Z_T - b'}{a' + c' Z_T} \tag{45}$$

4.2. Probe rods as a lossy capacitor – capacitance model C_0

Analysing the structure of the two-rod probe one can note that, with relation to the open-ended coaxial probe, it has a considerably greater capacity C_0 relative to capacity C_f (Stuchly and Stuchly 1980) which, therefore, can be considered as negligible.

Making use of the analogy to a flat capacitor, parallel rods of the probe can be described as a capacitor filled with a lossy dielectric, *i.e.* soil (Fig. 22). The probe is connected to the vector analyser by means of a coaxial cable with characteristic impedance of $Z_C = 50\Omega$.

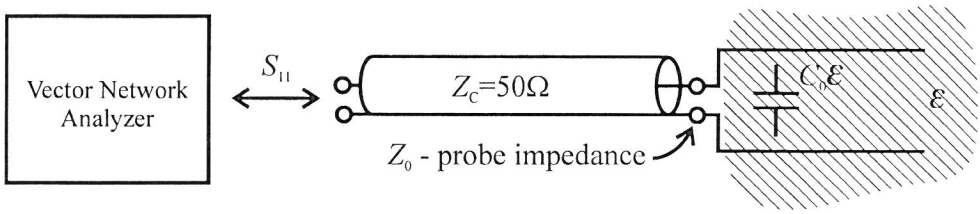


Fig. 22. Capacitance model C_0 two-rods probe inserted in soil

The capacity of a capacitor filled with a lossy dielectric with complex permittivity ε is equal to the product of a capacitor filled with air C_0 and ε. If we adopt the assumption that the electrical capacity of the probe rods in the air is C_0, then the impedance Z_0 of a two-rod probe placed in soil with complex permittivity ε can be described by means of the formula:

$$Z_0 = \frac{1}{j\omega C_0 \varepsilon} \tag{46}$$

for $\omega = 2\pi f$, where f – frequency of the measurement signal.

If we write that the impedance of the probe in the air Z_{air} is:

$$Z_{air} = \frac{1}{j\omega C_0} \tag{47}$$

then the impedance of the probe can be written as:

$$Z_0 = \frac{Z_{air}}{\varepsilon} \tag{48}$$

From this formula we can directly obtain ε as the ratio of the impedance of the probe in air to the impedance of the probe placed in soil. The vector network analyser measures the complex coefficient of reflection given by the formula:

$$S_{11} = |S_{11}| e^{j\varphi} \tag{49}$$

where $|S_{11}|$,φ – module and phase of the measured complex coefficient of reflection.

Knowing that the impedance of the probe with the cable can be determined from the formula:

$$Z_0 = Z_C \frac{1 + S_{11}}{1 - S_{11}} \qquad \text{dla } Z_C = 50\ \Omega \tag{50}$$

substituting into (48) the relation (50) after transformation we obtain:

$$\varepsilon = \frac{(1 + S_{11air})(1 - S_{11m})}{(1 - S_{11air})(1 + S_{11m})} \tag{51}$$

where: S_{11air} – measured coefficient of reflection for the probe in the air, S_{11m} – measured coefficient of reflection for the probe placed in soil.

4.3. Impedance model Z_f, Z_0

The description of capacitors by means of the single parameter of electrical capacity within the range of high frequencies is insufficient. This is evident from the data given in manufacturers' catalogues of those elements, that give frequency characteristics of impedance of capacitors which behave as parallel-serial connections of resistance, induction and capacitance elements.

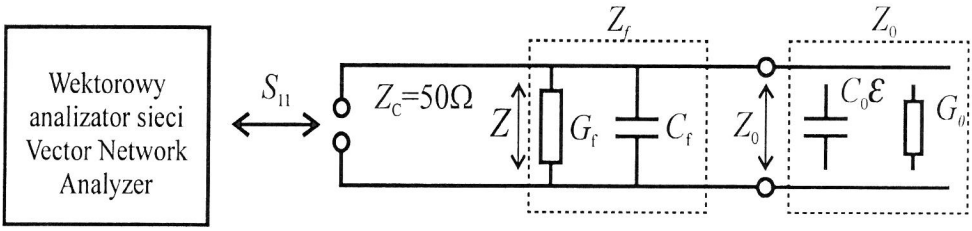


Fig. 23. Location of impedance for two-rods probe model Z_f, Z_0

The impedance model (Fig. 23) has been developed in relation to the ineffectiveness of determination of the imaginary part of dielectric permittivity when using the model of C_f and C_0. It was assumed that both capacities can be replaced with impedances Z_f and Z_0 which provide a much better description of the behaviour of the end of the line, and in the considered case of the two-rod probe they take into account the conductivities G_f, G_0 which describe the probe attenuation and the dielectric loss of the soil, respectively. The values of parameters Z_f, Z_0, as in the case of the capacitance model, are determined for each frequency in the process of calibration. The equivalent probe impedance Z of parallel connection of impedances Z_f, Z_0 is equal to:

$$Z = \frac{1}{\frac{1}{Z_f} + \frac{\varepsilon}{Z_0}} \tag{52}$$

Using formula (20), from the parameter S_{11} measured with the VNA we can determine impedance Z. The calibration of the model consists in solving a system of two equations, (53) and (54), from which the parameters sought, Z_f, Z_0, are determined.

$$Z_P = \frac{1}{\frac{1}{Z_f} + \frac{\varepsilon_P}{Z_0}} \tag{53}$$

$$Z_A = \frac{1}{\frac{1}{Z_f} + \frac{\varepsilon_A}{Z_0}} \tag{54}$$

where: Z_P – probe rods impedance in the air calculated from measurement, Z_A – probe rods impedance in acetone calculated from measurement, ε_P=1+j0 – dielectric permittivity of air, ε_A – dielectric permittivity of acetone determined from Debye model with parameters presented in Table 6.

The dielectric permittivity of an unknown dielectric can be determined in accordance with the transformed formula (52):

$$\varepsilon = \frac{Z_0 (Z_f - Z)}{Z \cdot Z_f} \tag{55}$$

5. MEASUREMENT AND CALIBRATION METHODS IN BROADBAND FREQUENCY DOMAIN

5.1. Configuration of vector network analyser

In the study we used the vector network analyser type ZVCE, Rohde& Schwarz (Operating Manual 2001) (Fig. 24), with measurement signal frequency range of 20 kHz – 8 GHz. The VNA was applied for the measurement of the complex coefficient of reflection S_{11} of a two-rod probe placed in the material studied. In accordance with the recommendations of the manufacturer, the instrument was warmed prior to the measurements for at least an hour, following which all calibration and measurement procedures were performed. During the measurements the air temperature in the room was 20.6±1°C.

The measurements were performed at the following configuration of the VNA:

- range of frequency change 10-500 MHz,
- measurements for 99 values of frequency in the set range of changes, with 5 MHz step,
- 11-fold averaging of results,
- Switched on active filter with band width of 300 Hz,

- output signal power level of –4 dBm, which corresponds to power of 0.39 mW,
- calibration of port 1 performed with OSM method (opening, shorting, mating) using a calibration set made by ROSENBERGER,
- calibration of the electrical length of the probe cable performed after fixing the probe in the stand,
- format of data registration and display – POLAR COMPLEX,
- measurement results recorded in ASCII format for ease of import by calculation program described in section 5.7.

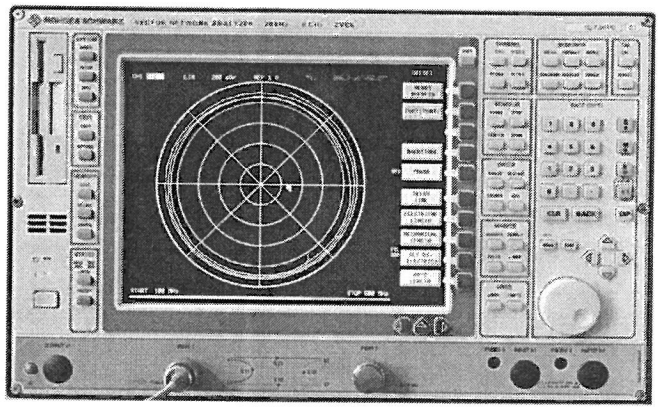


Fig. 24. Vector Network Analyser used for determine dielectric permittivity of investigated soils

The configuration parameters were selected with a view to obtain the optimum accuracy and speed of measurements. During preliminary measurements it was noted that changes in the position of the connecting cable of the probe reduce the repeatability of measurement causing an increase of error of measurement of the coefficient of reflection S_{11} at the level of 0.3 degree, which could be the cause of serious measurement errors. Therefore, the probe was mounted on a stand and the connecting cable was fixed in position. It was also observed that in the graph of the measured complex coefficient of reflection S_{11} (Fig. 25) there appear repeatedly characteristic loops and deflections, known from literature (Sheen and Woodhead 1999) for open-ended coaxial probes. Signal frequency at which the described phenomenon appears changes slightly with relation to the dielectric permittivity of the material tested in which the probe is placed and to the length of the probe rods. It was also noted that the length of the connection cable of the probe has an effect on the frequency value at which the phenomenon

appears. This led to the conclusion that it was a phenomenon related with resonance in the open line. Such resonance appears when the electrical length of the cable together with the probe equals an odd multiple of a half of the measurement signal wavelength (Dobrowolski 2001).

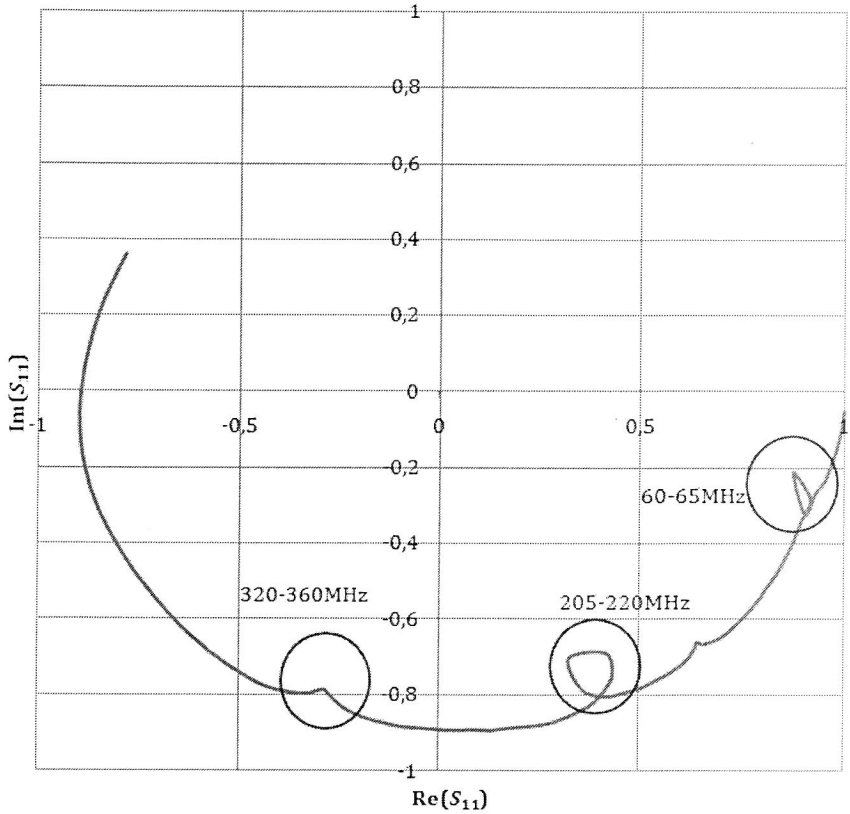


Fig. 25. Resonance distortion of S_{11} parameter measured by VNA marked by circle

The electric length is a parameter describing the length of the transmission line of the cable with relation to the electromagnetic length in vacuum (air). The electric length of a cable with air insulation is equal to its physical length. A dielectric filling the space between the cable wires is responsible for the shortening of the wavelength or the extension of the electric length. Therefore, to define phase delays or shifts introduced by a transmission line it is necessary to know its electric length. Stability of that parameter in the function of temperature and frequency is highly important during with work with the use of the vector network analyser.

The resonance behaviour of measurement probes described above caused a limitation of the frequency range of changes in the measurement signal to 10-500 MHz. Too broad a range of frequency changes caused incorrect calibration of the electrical length of the cable with the probe by the VNA, which led to the appearance of large errors in the measurement of the phase of the complex reflection coefficient S_{11}. Calibration of the electrical length performed by the vector network analyser consisted in measurement of the shift of the signal phase in the function of frequency. The narrowing of the band permitted the acquisition of satisfactory results of measurements of dielectric permittivity with the use of the models developed, and the upper frequency of the band chosen is higher by 300 MHz from the highest measurement frequency of commercially available FDR instruments (CS616 Campbell Scientific 2006, User Manual SM200 2006, User Manual ML2x 1999). For the reason of stability of resonance frequency bands, for further processing of the measurements a spectrum with removed bands indicated in Figure 25 was used.

5.2. Two-rod measurement probes

A specially designed two-rod probe was used for the study. The probe is built of two stainless steel rods with diameter of 2 mm and spaced at 13 mm. Three kinds of probes were built, with rod lengths of 1, 2 and 3 cm (Fig. 26). The spacing of the rods and their diameter were selected following the design of the field TDR probe manufactured at the IA PAS (Skierucha and Malicki 2004).

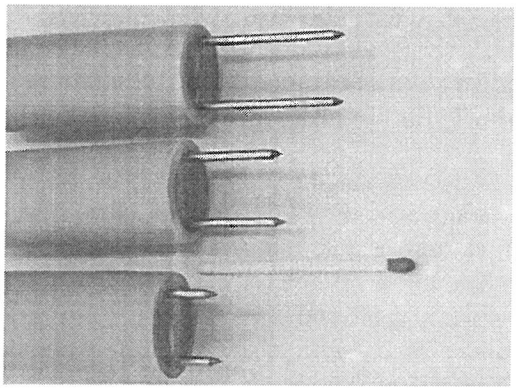


Fig. 26. Two-rods probes used in measurements

That choice was based on the possibility of application of probes with mechanical parameters identical to those of the field TDR probes type FP/mts, but

with a new interface and measuring instrument, which permit the comparison of measurement results obtained with the FDR and TDR methods. TDR probes with so short rods are not used due to the imitations posed by the measurement method. Commercially available TDR instruments with impulse rise time of 200 ps can be used to work with probes of lengths not less than 5 cm. FDR probes with short rods can fill the gap for that type of dielectric sensors appearing between the method of open-ended coaxial line (Skierucha *et al.* 2004) and the TDR probes.

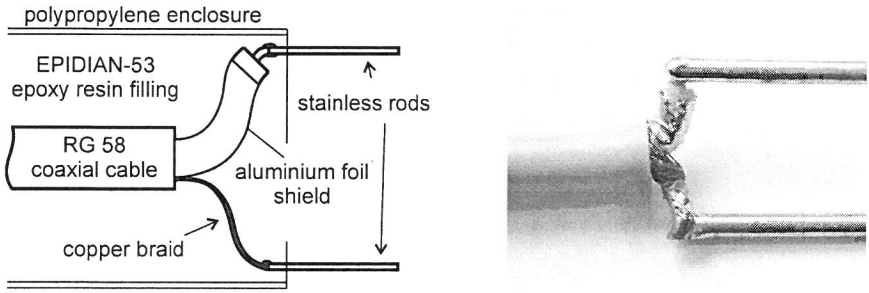


Fig. 27. The Interface of two-rods probe FDR: picture (left side), photo (right side)

The reflection interface of the probes was designed so as to achieve the shortest possible zone of impedance discontinuity, thanks to which commonly known theoretical formulae can be applied for the description of reflection (Dobrowolski 2001). The design and a photograph of the probe interface are presented in Figure 27.

5.3. Method of calibration of two-rod probe with two-terminal-pair network type T– model T_Z_0

Calibration with the use of a two-terminal-pair network type T permits the elimination of the sources of errors that are not taken into account in the capacitance model, such as the variable frequency characteristics of the connection cable between the VNA and the probe rods and the discontinuities of impedance of the connection of coaxial cable and probe rods.

The method under consideration, described in section 4.1, was used for the calibration of the two-rod probe. Figure 28 presents a schematic of the connection of the vector network analyser with the probe, indicating the measured impedance Z_T and the probe impedance Z_0 at the point of entry of the rods into the material tested.

To determine parameters a', b', c' of equation (45) three calibration points were adopted:

- probe shorting, for which $\mathrm{Re}(\varepsilon) = \infty$, $\mathrm{Im}(\varepsilon) = \infty$ was adopted,
- measurement in the air, $\mathrm{Re}(\varepsilon) = 1$, $\mathrm{Im}(\varepsilon) = 0$,
- measurement in acetone with permittivity determined from the Debye model (Tab. 6).

Acetone as the probe calibration material was selected because the values of both components of its complex dielectric permittivity are characterised by small changes both in the function of temperature and in the function of frequency within the considered frequency range of 10-500 MHz. Acetone can be a dielectric substitute of soil with a volumetric moisture of about 43% from equation (33).

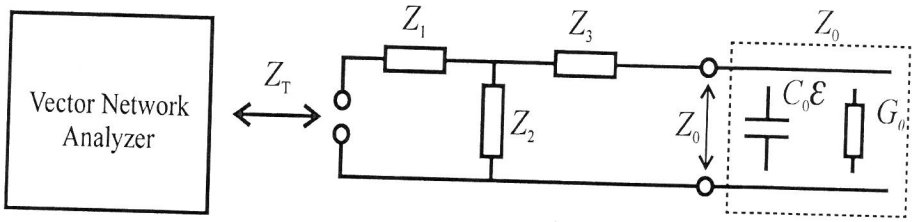


Fig. 28. Two-rods probe calibration used two-terminal-pair network type T

5.4. Probe rods as a lossy transformer of impedance – model T_T

Another method of describing a probe composed of two parallel metallic rods with length comparable to the wavelength of the measurement signal is to use the model of a long line with dispersed parameters (Osiowski and Szabatin 2006). Analysis of phenomena taking place in a long line is best performed by modelling it as a chain of mutually connected sections of length Δ_x, that can be substituted with two-terminal-pair networks built of compact elements with unit values of resistance R, inductivity L, conductivity G and capacity C (Fig. 29). The values of R, L, G and C are given as converted to a unit of line length.

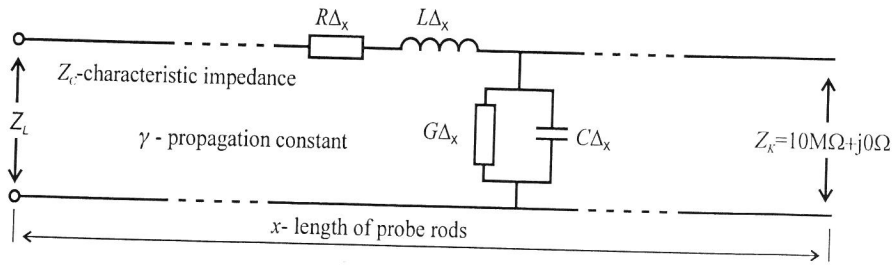


Fig. 29. Probe rods as a long line

Secondary parameters describing the behaviour of voltages and currents in a long line are the phase constant α expressed in rad/m and the attenuation constant β (attenuation) expressed in dB/m, being the real and imaginary components of the propagation constant γ (56), respectively, and characteristic impedance Z_C (57). The values can be expressed by means of formulae on the basis of the unit parameters of the line and of the angular velocity $\omega = 2\pi f$. The propagation constant is expressed by the formula:

$$\gamma = \alpha + j\beta = \sqrt{(R + j\omega L)(G + j\omega C)} \tag{56}$$

Whereas, the characteristic impedance:

$$Z_C = \sqrt{\frac{R + j\omega L}{G + j\omega C}} \tag{57}$$

The description of the input impedance of the probe was done with the use of the phenomenon of transformation of the probe open-end impedance Z_K in a long line with length x and complex parameters of propagation constant γ and characteristic impedance Z_C. The formula describing the transformation of impedance Z_K to the impedance at the beginning of the probe rods has the form:

$$Z_L = Z_C \frac{Z_K + Z_C \operatorname{tgh} \gamma x}{Z_C + Z_K \operatorname{tgh} \gamma x} \tag{58}$$

Assuming that:

- the open end of the probe presents high resistance $Z_K = 10\ \mathrm{M\Omega} + j0\ \Omega$,
- soil is a paramagnetic medium with relative magnetic permeability $\mu_r = 1$, therefore the unit inductivity of the rods L is constant and independent of the king of material tested,
- unit resistance R of the probe rods equals zero,
- unit capacity C can be replaced with unit lossy capacity equal to $C_0\varepsilon$, the introduction of which permits to consider conductivity G as negligible (C_0 – unit capacity in the air (vacuum), ε – complex dielectric permittivity of soil).

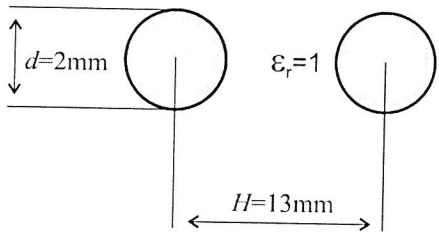
For the above assumptions the formulae (56) and (57) can be transformed into:

$$\gamma = \frac{j\omega}{c}\sqrt{\varepsilon} \tag{59}$$

where c is the velocity of light in vacuum.

$$Z_C = \frac{Z_{air}}{\sqrt{\varepsilon}} \tag{60}$$

Impedance of the probe rods in the air Z_{air}, treated as a symmetric line (Nowakowski 1982), is calculated from formula (61), for geometric parameters of the tested probes with rods with diameter of 2 mm and spacing of 13 mm and amounts to 307 Ω (Fig. 30). Knowing the long line parameters γ and Z_C and using the relation (58) describing the transformation of impedance by means of a section of the long line with length x, we can determine the impedance of probe rods Z_L placed in a lossy dielectric described by the complex permittivity ε.



$$Z_{air} = \frac{120}{\sqrt{\varepsilon_r}} \ln\left(\frac{H}{d} + \sqrt{\left(\frac{H}{d}\right)^2 - 1}\right) \tag{61}$$

Fig. 30. Impedance of two wire symmetrical line in air

After inserting (59) and (60) into formula (58) it assumes the form:

$$Z_L = \frac{Z_K + \frac{Z_{air}}{\sqrt{\varepsilon}} \operatorname{tgh}\left(\frac{j\omega x}{c}\sqrt{\varepsilon}\right)}{1 + \frac{Z_K}{Z_{air}}\sqrt{\varepsilon}\operatorname{tgh}\left(\frac{j\omega x}{c}\sqrt{\varepsilon}\right)} \tag{62}$$

From formula (62) we can easily determine the impedance of rods with length x knowing the complex permittivity ε, but it is much more difficult to determine the inverse function i.e. $\varepsilon = f(Z_L)$.

Treating the probe rods as a lossy transformer of impedance ensured independence of the result of the dielectric permittivity obtained from the length of the rods, but problems appeared with the uniqueness of the measurement results obtained. As follows from analysis of the described model performed in section 5.5, with a single frequency of measurement signal more than one value of dielectric permittivity can be determined for the coefficient of reflection measured. Such behaviour appears

when the wavelength of the measurement signal is shorter than the electrical length of the probe rods in a medium with the highest dielectric permittivity encountered in the tests. To solve the problem of the non-uniqueness, the authors used the fact that for each probe, up to a certain limit frequency, there is only one solution of equation (62), and above that frequency solution tracing can be applied through narrowing the limits of its variability on the basis of the solution determined at a lower frequency. The narrowing of the limits of variability ensures the achievement of uniqueness of determination of dielectric permittivity.

Basing on the above considerations, a computer program was developed that makes use of the method of successive approximations, described in section 5.7.

5.5. Mathematical analysis of lossy transformer of impedance

To find an analytical solution of $\varepsilon = f(Z_L)$ the software Mathematica 4.0 was used (Drwal *et al.* 2000). However, when we tried to solve equation (62) the program returned a message proposing the application of a numerical solution. To enable finding an analytical solution, the type of functions was unified to the power functions. Formula (62) was modified, substituting the hyperbolic tangent with the quotient of Taylor series of functions $\sinh(x) / \cosh(x)$ described with the formulae:

$$\sinh(x) = \sum_{n=0}^{\infty} \frac{1}{(2n+1)!} x^{2n+1} \tag{63}$$

$$\cosh(x) = \sum_{n=0}^{\infty} \frac{1}{(2n)!} x^{2n} \tag{64}$$

That approach should permit an analytical solution giving approximated results, but after the above modification the program Mathematica generated analytical solutions only for the first three terms of the series (63,64). The solutions had highly complicated analytical form, and due to the small number of terms in the series the results obtained had low accuracy. Ultimately, numerical methods were applied to determine the complex dielectric permittivity ε from equation (62).

As it is known from mathematical analysis, the function hyperbolic tangent is a periodic function for the imaginary component of the argument. For equation (62) we can find the condition of periodicity, and thus also of non-uniqueness of solution, which can be expressed with the formula:

$$\lambda < 2 \cdot x \qquad \text{dla} \quad \lambda = \frac{c}{f\sqrt{\varepsilon}} \tag{65}$$

where: c – velocity of light, f – frequency of measurement signal, λ – signal wavelength in tested material with refractive index $\sqrt{\varepsilon}$, x – electrical length of probe rods in the air. If that condition is met, for the whole range of variability of Z_L it is possible to obtain multiple solutions of ε. For a better illustration of the problem, a transformation of impedance Z_L was performed, substituting it as Z_p into formula (17) which expresses the complex coefficient of reflection S_{11}.

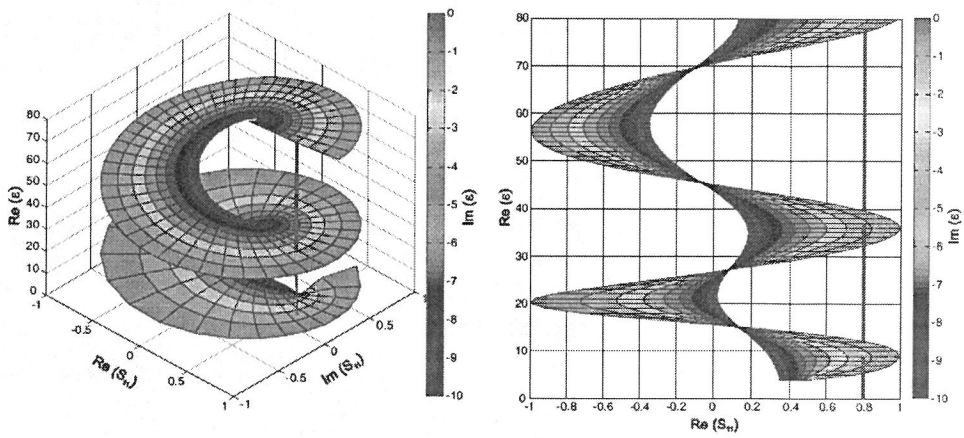


Fig. 31. Four-dimension chart of the complex dielectric permittivity ε (left side), obtain from analysis of loss transformer model, view in Re(ε), Re(S_{11}) plane (right side)

Using the program Matlab R2008b (Pratap 2007), a graph of the complex function $\varepsilon = f(S_{11})$ was plotted (Fig. 31), with the following assumptions: x = 10 cm – electrical length of probe rods in the air, f = 500MHz – frequency of measurement signal.

In the Figure 31 one can see that if from the plane S_{11} a perpendicular line is created, and it will intersect the graph of ε in several points. For this reason, in the numerical method a tracing function was added, consisting in prior calculation of the value of ε, for such signal frequency that the condition (65) could not be met; then there is the certainty of only one solution. After exceeding the measurement signal frequency by the values of the limit frequency, two or more solutions may appear. Those can be identified using the value obtained from the previous calculation, thanks to which it is possible to properly determine the limits of the range

of variation of the function. This approach permits the interpretation of measurements at high frequencies, for which the wavelength is shorter than the electrical length of the probe rods.

5.6. Calibration of lossy transformer of impedance with two-terminal-pair network type T

The method of calibration with a two-terminal-pair network type T requires the determination of at least three calibration points, for which we know the exact values of impedance of the probe modelled (section 5.3). This requirement is related with the necessity of solving a system of three equations.

The calibration under consideration consists in the substitution, in formula (40), of impedance Z_0 of the capacitance model with the lossy transformer impedance Z_L described with formula (62), where x is the measured length of the probe rods. The formula then assumes the following form:

$$a + bZ_L - cZ = ZZ_L \tag{66}$$

For three calibration points we obtain a system of three complex equations.

$$a + bZ_Z - cZ_1 = Z_1 Z_Z \tag{67}$$

$$a + bZ_P - cZ_2 = Z_2 Z_P \tag{68}$$

$$a + bZ_A - cZ_3 = Z_3 Z_A \tag{69}$$

where: $Z_Z = 0+j0$ – impedance of shorting of the rods at the point of their connection with the probe body, Z_P – impedance of probe rods in the air calculated from formula (62) for $\varepsilon = 1+j0$, Z_A – impedance of probe rods in acetone calculated from formula (62) for the Debye model (Tab. 6), Z_1, Z_2, Z_3 – impedances measured by the VNA with the probe rods shorted, and for rods placed in the air and in acetone, respectively.

Parameters a, b, c are calculated for a given measurement signal frequency and for a single probe rod length x. Due to the degree of complexity of function $Z_L = f(\varepsilon)$ it is not possible to express the value of ε directly as in the case of the capacitance model (section 4.1), but only the value of impedance Z_L which, for the ε value sought, should be equal to the value of impedance Z_T calculated from calibration with a two-terminal-pair network type T.

$$Z_L = Z_T = \frac{a - cZ}{Z - b} \tag{70}$$

The substitution of the capacitance model with a lossy transformer of impedance involves an additional difficulty related with the effect of the line open end, that was neglected by adopting the assumption that x is the physical length of the probe rods. In reality, a lossy transformer which is a long line with a specific, physically measurable, length x requires additional calibration of electrical length in air. The electrical length x of a long line placed in air and open-ended is different from its physical length. The difference results from the effect of the open end of the line, where there is both energy radiation identified with lossy, and electrical capacity. The capacity at the end of the line causes its apparent electrical elongation, therefore the electrical length should be determined at the stage of the probe calibration. For the determination of the electrical length of the probe an additional, fourth, calibration point should be used. The problem of correct and accurate determination of the electrical length of a measurement probe, which requires mathematical and numerical analysis and additional calibration measurements, can be the subject of a separate dissertation.

5.7. Software for calibration and for determination of measurement results

The designed two-rod probe connected to port 1 of the vector network analyser permits the determination of the complex coefficients S_{11} for probe placed in soil solutions being tested or in reference solutions. The result of measurement of a single soil sample is a file that contains 99 values of the measured complex coefficient of reflection for the frequency range of 10–500 MHz.

To process that file and obtain the spectrum of dielectric permittivity it is necessary to perform the calibrations described in section 5.6, acquiring from them the calibration parameters. Those parameters should be determined for each frequency of the measurement signal and they can be considered as constant, until the moment of probe change or of a change in the position of the cable connecting the probe with the VNA. The obtained measurement data were processed by means of the software (Fig. 32) developed with the help of the compiler C++ Builder from the Borland Developer Studio 2006 package. The function of the software is the import of files from the VNA, created during the calibrations, and the calculation of calibration parameters for the measurement frequencies applied, depending on the model used. The software permits the determination of the complex dielectric permittivity for all the models described herein.

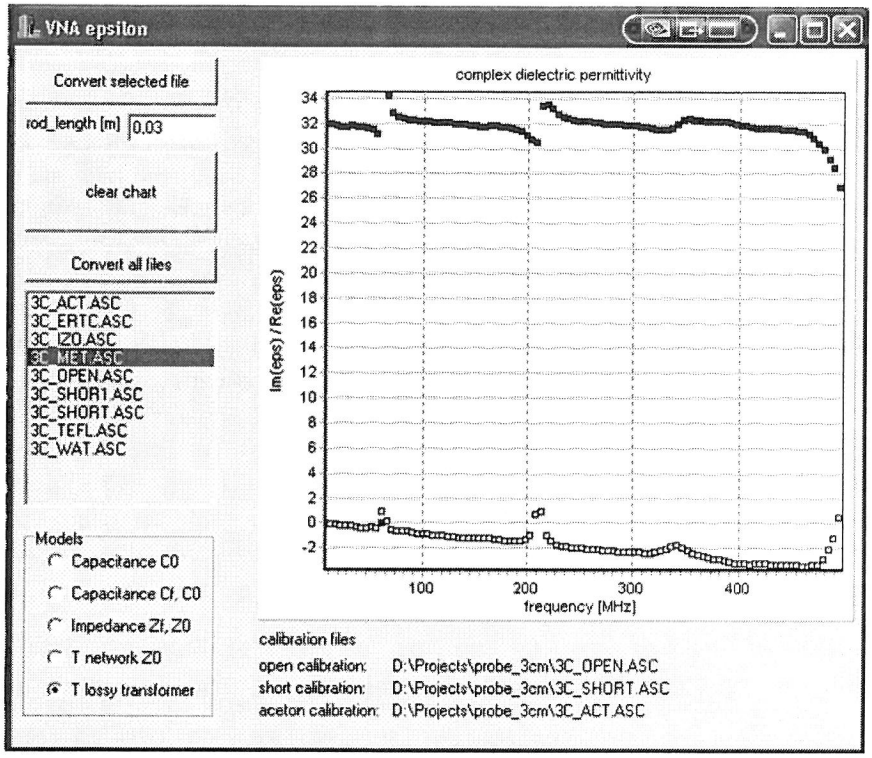


Fig. 32. The window of program for conversion data from VNA using various model

The operation of the numerical method used to find the frequency spectrum of dielectric permittivity of the material studied from the solution of equation (62) is described below.

(1) We select the lowest measurement frequency (10 MHz) for which the uniqueness of solution is ensured, *i.e.* condition (65) is not fulfilled.

(2) Equation (62) is solved numerically for the selected frequency, assuming the maximum limits of variability of ε for the material studied and of parameter Z_L calculated after the calibration (70) from the value of S_{11} measured by the VNA. The effects of this operation are the real and the imaginary values of ε for the selected frequency.

(3) The initial frequency is then increased by 5 MHz and the limits of the search for ε are modified with relation to the solution obtained in the preceding step. Next the procedure from item (2) is repeated.

(4) Items (2) and (3) are repeated until the moment of reaching the maximum frequency within the measurement range (500 MHz).

5.8. Validation of the model

The validation procedure was performed with the use of four mineral soils with varied physicochemical parameters which are presented in Table 5. The soils were selected so that the values of the measured dielectric permittivity could be affected to a minimum extent by factors other than moisture and salinity. Such other factors could be e.g. soil swelling of high levels of organic matter. The soils came from the bank of Polish soils collected at the IA PAS (Gliński *et al.* 1991) and have individual numbering assigned to them by the bank. For soils No. 569, 610 and 622 ten samples each with varied moisture were prepared. At the start the maximum water capacity was determined for each of the soils, and then doses of water were calculated that should be added to the containers with the soil samples to achieve moisture levels changing in steps of 0.1 of the maximum water capacity. Prior to the dosage of water, the soil samples were placed in plastic containers with capacity of 125 ml, and then dried at 105°C for 24 hours.

Table 5. Localization and selected properties of investigated soils

No.		569	610	622	529
Soil type		Brown	Brown	Chernozem	Black soil
Soil localization		Majdan Skierbieszowski	Kol. Olempin	Terebin	Annopol
Dry bulk density (gcm^{-3})		1.33	1.59	1.4	1.76
Soil texture by FAO (%)	Sand	71	94	60	100
	Silt	25	5	34	0
	Clay	4	1	6	0
Specific surface ($m^2 g^{-1}$)		21	9	37	8
Water holding capacity ($kg\ kg^{-1}$)		0.39	0.13	0.36	0.16

After the drying the soil samples were closed tight and weighed. Varied moisture levels of the soil were obtained through the dosage of distilled water amounts calculated earlier to a specific volume of dry soil. To achieve uniform water distribution in each of the soil samples, the samples were closed tight in the containers and warmed at 45°C for three days. Soil samples prepared in this manner were tested with probes (section 5.2) with various rod lengths, beginning with the shortest.

For testing the effect of soil salinity, 5 series of 10 samples of black earth No. 529 were prepared. To achieve varied salinity of the soil, moistening of each series of samples was made using KCl solutions with electrical conductivity values

of 0, 5, 10, 15 and 20 dS/m. As in the case of soils No. 569, 610 and 622, the moisture of the soil samples was modified uniformly at every 0.1 of the maximum water capacity.

Due to the non-homogeneity observed in porous materials, and in soil in particular, the accepted practice in the estimation of the accuracy of a given method and probe model is to use reference liquids with known frequency characteristics of dielectric permittivity at a given temperature. Also the calibration of probes requires the use of media with known parameters. Those liquids are most often described by the models of Debye and Cole–Cole. For the study we selected liquids well described in the literature (National Bureau of Standards 1958), with parameters as presented in Table 6.

The tests were conducted also in solids, such as polyoxymethylene and teflon, with dielectric permittivity of 3.8 and 2.1, respectively, for which the effect temperature and dielectric loss for frequencies below the level of several GHz is negligible.

Table 6. Parameters of Debye'a model of applied reference liquids

Name	Temperature (°C)	ε_s	ε_∞	τ (ps)
Distilled water	20	80.4	5.2	9.45
Acetone	20	21.2	1.9	3.34
Methanol	20	33.64	5.7	53
Isopropanol	20	29	3.2	292

Estimation was performed of the applicability of two-rod probes for the measurement of the frequency spectrum of the complex dielectric permittivity of soil, and measurements were made aimed at testing the effect of soil moisture and salinity on its dielectric permittivity determined with the method of frequency reflectometry – FDR. The work consisted in designing and constructing suitable two-rod probes and developing for them the following:

- a mathematical model,
- methods of calibration and measurement,
- software for calibrations and numerical determination of measurement results.

Based on the validation tests with the use of reference liquids and comparison of the measurement results with literature data suitable for the Debye model, the selection of the best mathematical model was performed. Next, laboratory ex-

periments were conducted with the use of the developed FDR probes and soil samples with varied salinity and moisture. Investigation was performed of the effect of measurement signal frequency on the correlation between the volumetric moisture of the soil samples, determined with the gravimetric method, and the refractive index for the selected range of measurement signal frequencies. Also the effect of salinity of the soil samples on the accuracy of measurement of the real dielectric permittivity was studied, and the possibility of determination of the salinity index (SI) from measurements of the spectrum of the complex dielectric permittivity was examined.

5.9. Comparison of results obtained with various models

To test the applicability of a given probe model for the determination of the complex dielectric permittivity of the material studied measurements were made in the calibration liquids and then in methanol.

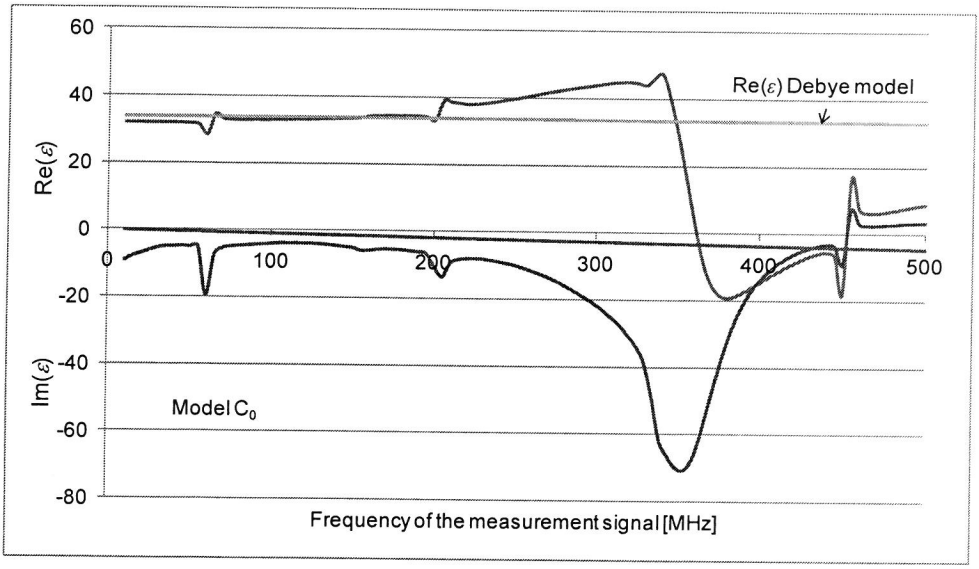


Fig. 33. Measured complex dielectric permittivity of methanol for C_0 model against reference Debye'a model background

Methanol the most frequently referenced in literature medium for the verification of dielectric methods. The results of those measurements were not processed by the software described in section 5.7. The obtained values of dielectric permittivity of methanol were compared with values obtained from the Debye model.

The tests were performed for probes with rod length of 3 cm, and then, after satisfactory results were obtained, probes with lengths of 1, 2 and 10 cm were tested.

Figure 33 presents the results obtained for the capacitance model C_0, described in section 4.2, which assumes that probe rods are an element with compact parameters and that they can be substituted by a capacity with a lossy dielectric. That model permits correct determination of only the real component Re(ε) of dielectric permittivity within measurement signal frequency range up to 200 MHz. Above that frequency both components of dielectric permittivity are burdened with error exceeding 10%. The next model to be analysed was the model C_0, C_f. The results were coincident with those obtained for the model C_0, which was to be expected in view of the assumed negligible effect of the additional capacity C_f. To achieve better results, it was decided to improve the model by replacing capacities C_0, C_f with impedances Z_0, Z_f. As can be seen in Figure 34, while the accuracy of measurement of the real component remained unchanged, the imaginary component became measurable within the same range of frequencies.

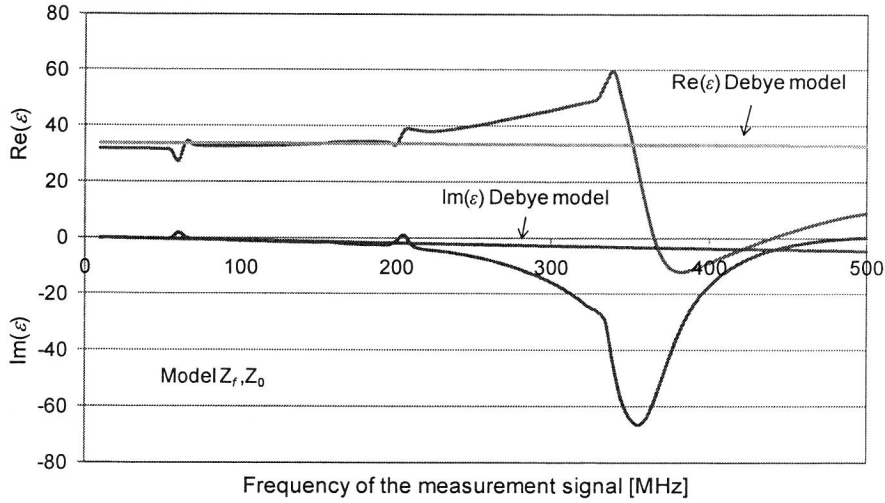


Fig. 34. Measured complex dielectric permittivity of methanol for Z_f, Z_0 model against reference Debye'a model background

Searching for a model ensuring higher accuracy of measurement in the range of high frequencies, the model of the two-terminal-pair network type T described in section 4.1 was applied. It is used for the calibration of open-ended coaxial probes in tests of the dielectric permittivity of liquids. The calibration consists in the substitution of the unknown parameters of the cable and of areas of impedance

discontinuity with a two-terminal-pair network composed of three impedances in the "T" layout. The measurement results presented in Figure 35 permit the conclusion that this model provides an improvement of the accuracy of measurement of both components of dielectric permittivity, but still measurements above 200 MHz for the 3 cm probe and above 350 MHz for the 2 cm probe are totally different from the reference values from the Debye model. The performance of the model is satisfactory for the 1 cm probe.

In view of the unsatisfactory performance of the models in question in the frequency range up to 500 MHz and of the reduction of the maximum frequency of the measurement signal with increase in the length of the probe rods, an attempt was made at developing a new model in which the advantages of the earlier calibrations based on the two-terminal-pair network type T were utilised (section 5.6).

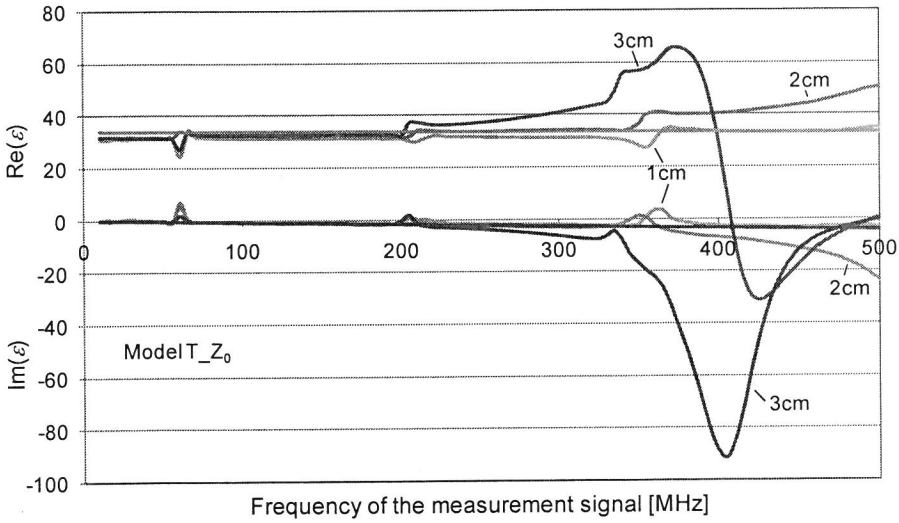


Fig. 35. Complex dielectric permittivity of methanol for T_Z_0 model measured by probes of different length against reference Debye'a model background

In the new model the capacity reactance was replaced with the terminals of a lossy transformer of impedance (section 5.4), formed of probe rods treated as a long line. Figure 36 presents the results obtained after the application of the model. As can be seen, the real component of dielectric permittivity is independent of the length of the probe rods for the whole range of measurement signal frequency. There is only a certain constant underestimation of results, which was observed also for the other models, but at higher frequencies it was masked by the growing error resulting from the imperfect performance of the models. The effect

of underestimation of results can be attributed to probable overestimation of the dielectric permittivity of acetone used for the calibration. The length of the probe rods causes a deterioration of the accuracy of measurement of the imaginary component, *i.e.* the shorter the rods the lower the accuracy.

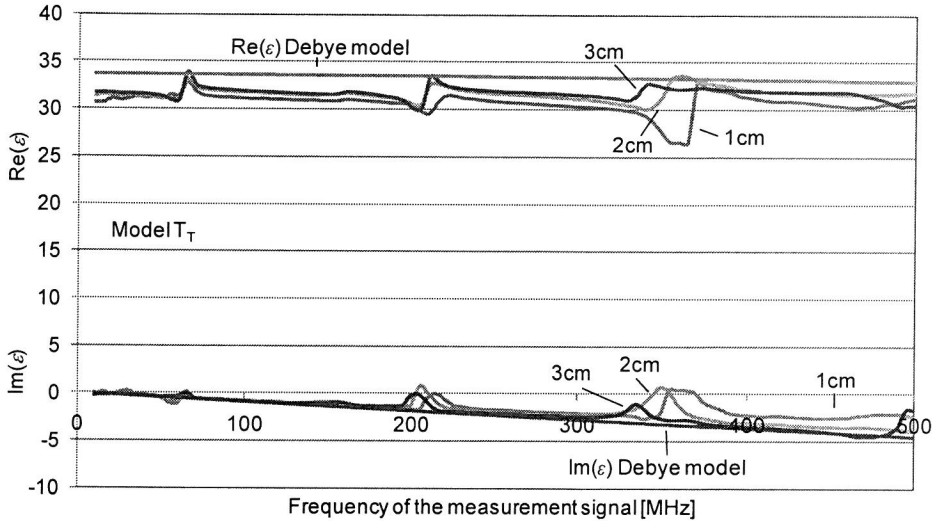


Fig. 36. Complex dielectric permittivity of methanol for lossy transformer T_T model measured by probes of different length against reference Debye'a model background

Table 7. Combination of calibration points

Calibration points	1	2	3
1 (SAW)	short	air	water
2 (SAA)	short	air	acetone
3 (WAA)	water	air	acetone
4 (WAM)	water	air	methanol
5 (SAI)	short	air	isopropyl
6 (SAM)	short	air	methanol
7 (WAI)	water	air	isopropyl
8 (MAA)	methanol	air	acetone

To estimate the accuracy of the lossy transformer model applied, calibrations of the 3 cm probe were performed in various combinations, presented in Table 7. The results are given in Figure 37.

In the graphs one can see the changes in the accuracy of measurement of the real component of dielectric permittivity resulting from the adopted calibration points. Out of the eight combinations, the lowest measurement error was obtained for calibration 3_WAA (water, air, acetone) where for low values of the root $\sqrt{\mathrm{Re}(\varepsilon)} < 2$ the error is less than 4%, and for $\sqrt{\mathrm{Re}(\varepsilon)} > 2$ it is below 2.2%.

Due to the fact that positive effects of performance of the model of the lossy transformer T_T were obtained for the probe with 3 cm rods, its performance was tested for the 10 cm probe (Fig. 38).

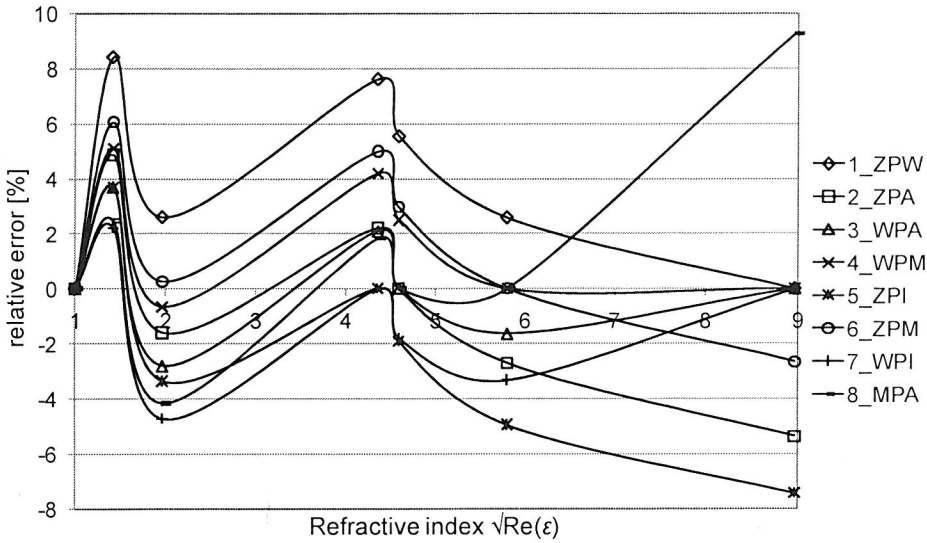


Fig. 37. The percentage relative measurement error of the real part of dielectric permittivity dependent on combination calibration points

As can be seen from the graph, within the band of 125-375 MHz the real component of dielectric permittivity of methanol is burdened with a large measurement error reaching 20% of the measured value, but its mean value is very close to the model value. The behaviour of the imaginary component is similar. This can be attributed to a strong effect open line resonance phenomena (p. 52) that intensify with increase in the length of the probe rods. The large discrepancies in the measured dielectric permittivity do not permit at present for such a long probe to be applied in studies of soils. The application of 10 cm probes in

the FDR method described appears to be possible after the elimination of the resonance phenomena and after developing a new method of calibration of the probe.

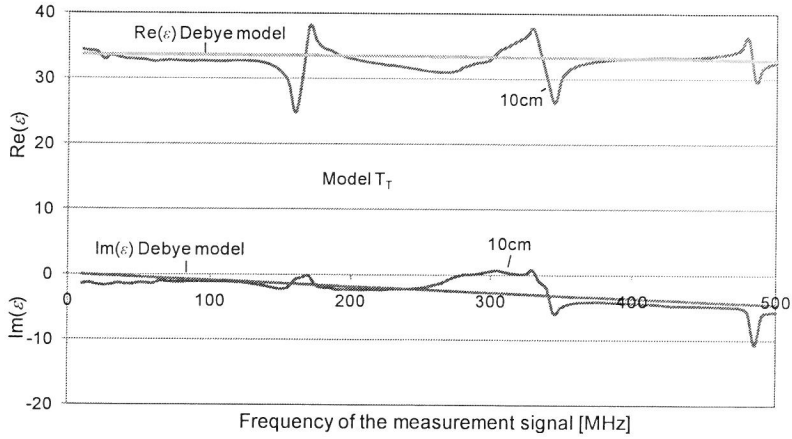


Fig. 38. The Complex dielectric permittivity of methanol for lossy transformer T_T model measured by probe of 10 cm length against reference Debye'a model background

5.10. Effect of measurement signal frequency on measurement of refractive index

The frequency of the measurement signal has a notable effect on the result of measurement of the refractive index of soil with the FDR method (Hilhorst and Dirksen 1994). Depending on the measurement signal frequency an effect of relaxation of colloidal molecules or of bound water molecules is observed.

Analysis of the effect of frequency on the result of measurement was conducted by comparing the averaged values of the refractive index for six selected bands of the spectrum, *i.e.* 10-30, 35-50, 80-120, 125-190, 245-300, 390-485 MHz. The bands were selected so as to exclude those ranges in which the open line resonance appears.

The analysis was performed for results obtained for soil No. 529, measured with the probe with rod length of 3 cm with the use of the lossy transformer model.

In Figure 39 one can observe that with a decrease of frequency the value of the refractive index increases by a constant value over the whole range of moisture (fitted trend lines are almost parallel), its linear relation to moisture being maintained, which indicates that for the non-saline sandy soil studied the results obtained with low-frequency methods can be comparable with those obtained with high-frequency methods. For all the bands trend lines were determined, for which the coefficient of

determination R^2 was not lower than 0.989. In Figure 39 only the trend lines for the extreme bands of frequency are shown for clarity of representation.

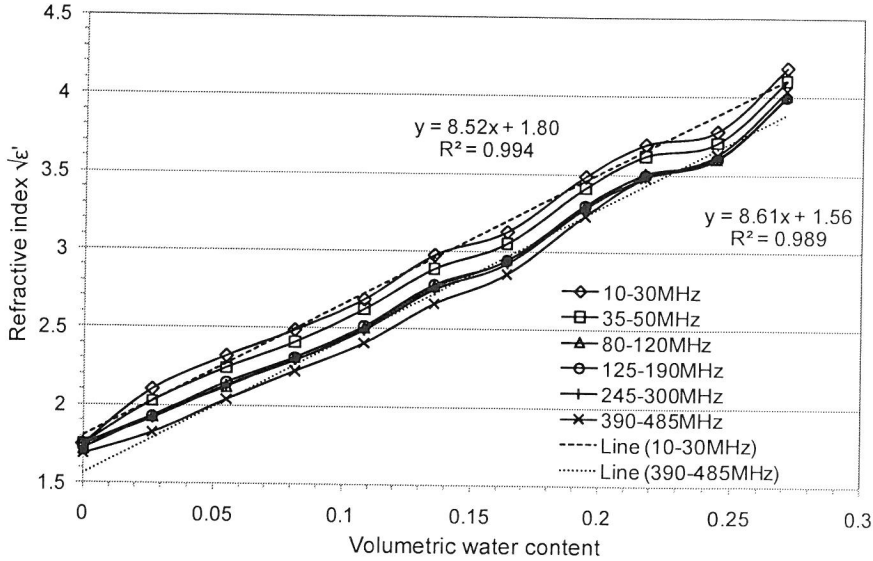


Fig. 39. Influence of the frequency measurement band on correlation between refractive index and volumetric water content of 529 soil moisturized by distilled water

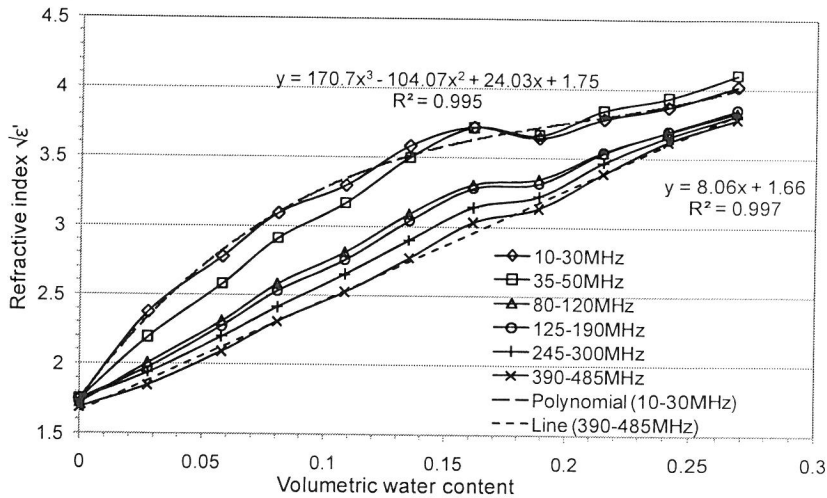


Fig. 40. Influence of the frequency measurement band on correlation between refractive index and volumetric water content of 529 soil moisturized by KCl solution with electrical conductivity 20 dS m^{-1}

In the case of soil moistened with KCl solution with conductivity of 20 dS m^{-1} the behaviour of the refractive index is different.

As can be seen in the Figure 40, the lower the measurement frequency the more distorted and overestimated the value of the refractive index for the same moisture of the soil samples. For the range of 390-485 MHz the relation to moisture is described by a linear function with the coefficient of determination of $R^2 = 0.997$, while for the band of 10-30 MHz we need to use a 3rd degree polynomial to obtain a similar value of the coefficient of determination $R^2 = 0.994$. This means that in the case of the saline sandy soil measurement with low-frequency methods is burdened with notably greater error than is the case with the same soil but non-saline. The distortion of the low-frequency characteristics (10-30 MHz band) can be eliminated through individual calibration for a given type of soils. It should be kept in mind, however, that the form of the characteristics depends on the level of salinity. While the salinity itself (taken as the electrical conductivity of the soil extract) may not change during the experiment, the electrical conductivity of the soil, strongly dependent on temperature, will change. This clearly demonstrates the superiority of high-frequency dielectric methods of soil moisture measurement, the selectivity of which largely eliminates the sensitivity of the results obtained to the effect of electrical conductivity. This feature is especially important in field measurements, for which it is impossible to maintain constant temperature.

5.11. Correlation between volumetric moisture of soil samples determined with the gravimetric method and the refractive index

For analysis we selected the data obtained for the probe with 3 cm rod length and for the measurement signal frequency band of 390-485 MHz, for which the real component of dielectric permittivity undergoes the least changes in the function of frequency. Data from 20 measurement points within that range were averaged for each soil sample. Next, the square root of the dielectric permittivity, i.e. the refractive index, was calculated. The standard deviation for all measurements did not exceed 0.16 of the absolute value. The result obtained in this manner was correlated with the volumetric moisture obtained from gravimetric measurement. The results are presented in Figure 41. In the graph the linear functions were fitted for comparison with literature data. Deviations from the linear characteristics can be attributed to the non-homogeneity of the soil, i.e. to moisture distribution within the soil sample tested or to the effect of density for the particular soil samples.

The soils studied are characterised by varied density, which was the cause of the differences in the slope of the characteristics. The trend line with the form of

$\sqrt{\mathrm{Re}(\varepsilon)} = a_0\theta + a_1$, fitted to all the measurement points, has coefficients $a_0 = 8.86$; $a_1 = 1.48$.

Those coefficients determined for the models of Malicki (1993), elaborated from measurements with the TDR method and presented in table 8, are similar to the above. It can also be noted that correction for soil density improves also the level of agreement of the coefficients. This can lead to the conclusion that results obtained with the FDR probe are in agreement with results obtained with the TDR technique.

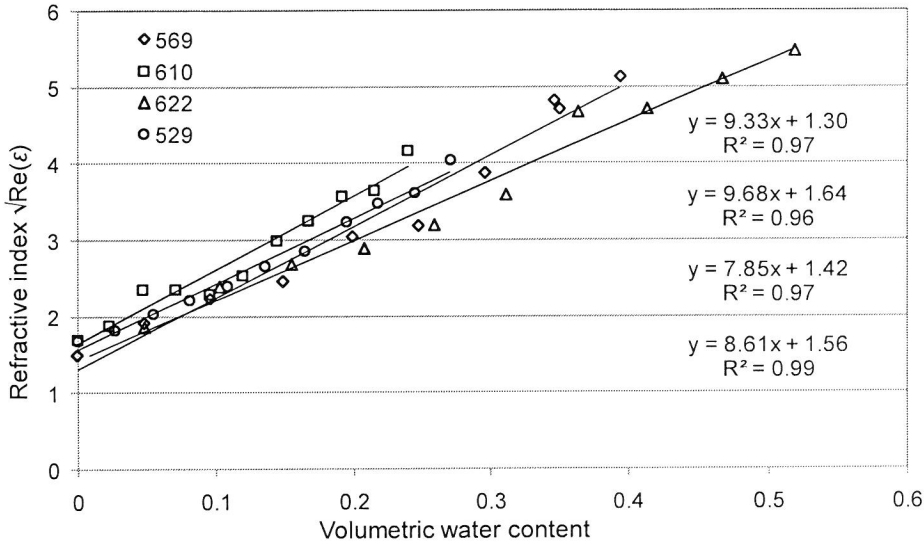


Fig. 41. Dependence of refractive index on volumetric water content investigated soils

Table 8. Comparison of FDR calibrations coefficient of the analyzed sensors with Malicki model for the TDR method

coefficient	$\sqrt{\mathrm{Re}(\varepsilon)} = a_0\theta + a_1$		
	FDR measurements (Fig. 41)	Malicki Model	Malicki model (correction on soil density)
a_0	8.86	7.16	8.96
a_1	1.48	1.44	1.44

5.12. Accuracy of measurement of the real component of dielectric permittivity and the salinity of soil samples

Selectivity of a measurement method is one of the most important methodological features. The determination of selectivity requires measurement performed under conditions of controlled variation of the interfering value. In the case under consideration the verification of selectivity of measurement of the refractive index for soil No. 529 was performed at variable salinity of samples, which was achieved by means of variable conductivity of the moistening solution within the range of 0-20 dSm^{-1}. The same measurement procedure was applied as that described in the preceding section. The results of the measurements are presented in Figure 42.

As can be seen in the graph, the refractive index is correlated in a linear manner with the volumetric moisture of the soil. High values of coefficient of correlation for all five levels of soil salinity and absence of variation in the slope of the lines indicate the lack of effect of salinity on the measured refractive index, confirming high selectivity of the method, as in the case of the TDR method (Malicki and Walczak 1999).

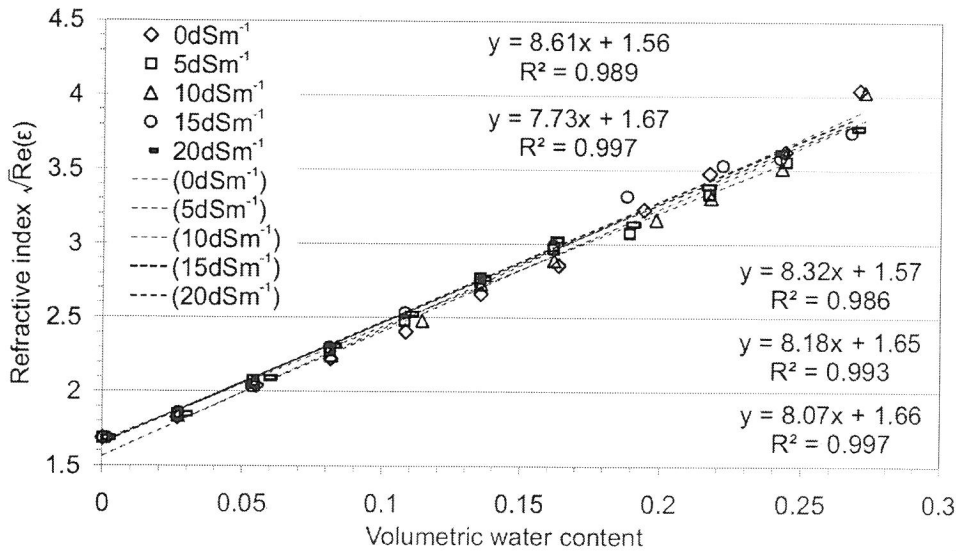


Fig. 42. Dependence of refracive index on volumetric water content of 529 soil moisturized by KCl solution with different value of electrical conductivity

5.13. Measurement of salinity based on the imaginary component of dielectric permittivity of soil, determination of salinity index

Obtaining information on salinity from measurement of the frequency spectrum of the imaginary component of dielectric permittivity requires analysis of both of its components (section 2.11). If the measurement is performed for a single frequency the two components are indistinguishable. Is there is a possibility of analysis of the frequency spectrum of the response of the material tested to external electrical stimulation then it is possible to separate the components on the basis of knowledge of their behaviour in the function of frequency. Measurement with the TDR method permits such an analysis with the assumption that the dielectric loss ε_d is constant within the range of changes of the measurement signal frequency f. For correct performance of the analysis three frequency bands were removed from the spectrum, at which there was resonance interfering with the measurement, i.e. 60-65, 205-220, 320-360 MHz (section 5.1).

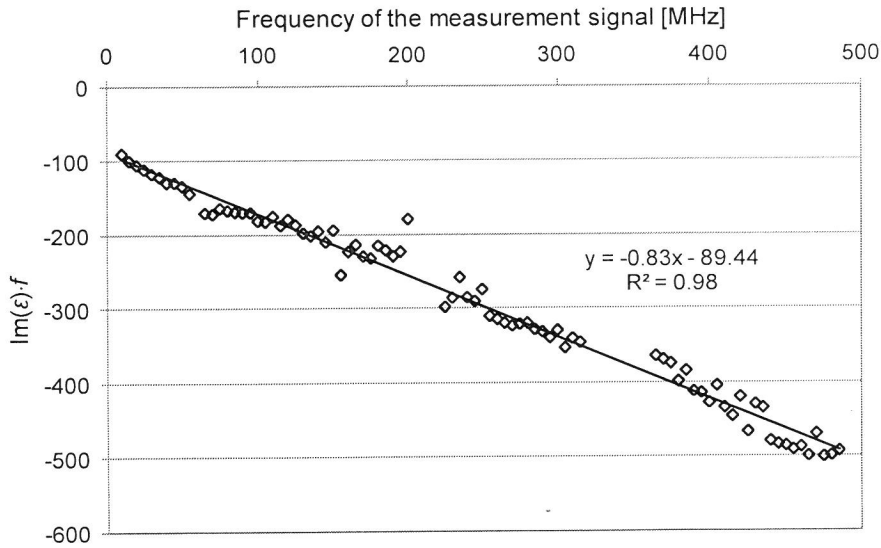


Fig. 43. Calculate dielectric loss ε_d and σ_{DC} direct current conductivity from z linear characteristic of Im(ε)·f on example selected sample of 529 soil with 27% volumetric water content moisturized by distilled water

To permit the determination of both elements ε_d and σ_{DC} from formula (21), it was transformed through multiplying both sides of the formula by f, obtaining a relation in the form:

$$\mathrm{Im}(\varepsilon)\cdot f = af + b \tag{71}$$

where: $a = \varepsilon_d$–dielectric loss, $b = \sigma_{DC}/2\pi\varepsilon_0$.

Thus a linear relation was obtained, for which the slope a and the translation b were determined with the least squares method, as presented in Figure 43.

To verify whether the coefficients obtained give well fitted characteristics, they were substituted in formula (21), and the resultant graph was superimposed on the measurement results, as presented in Figure 44.

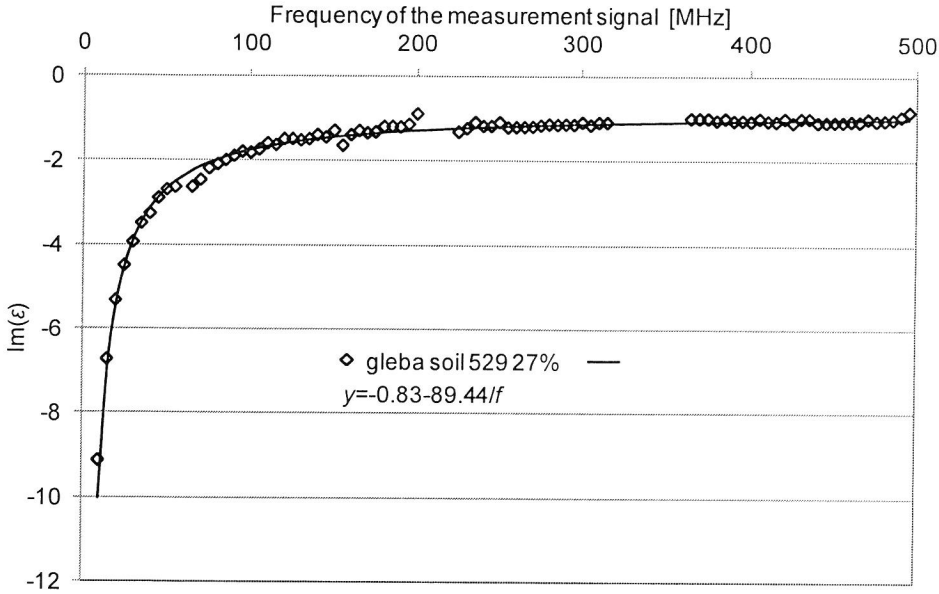


Fig. 44. Imaginary part of dielectric permittivity with overlapped function y = –a– b/f for selected sample of 529 soil with 27% volumetric water content moisturized by distilled water

We can see very good fitting both for the low and the high frequencies, which permits the conclusion that the developed method for the determination of DC conductivity from the spectrum of the imaginary component of dielectric permittivity can be used for further data processing. This method was applied to process all results for 50 samples of soil No. 529 moistened with solutions of KCl. Analysing the results of conductivity obtained for those samples we can determine index *SI* – salinity index described in section 2.11 as the slope of the linear relationships between the determined direct current conductivity and the real compo-

nent of dielectric permittivity (determined for the frequency band of 390-485 MHz). These relations are presented in Figure 45.

The graphs show high coefficients of determination at the level of 0.99, which indicates good selectivity and suitability of the FDR method for measurement of the salinity index f soil.

In the case of values of conductivity the coefficient of determination drops o the level of 0.96, which indicates a lower accuracy of the method with relation to non-saline soils.

In accordance to literature data (Malicki 1993), the index of salinity should have a linear relation to the conductivity of the moistening solution, which is supported by Figure 46. Analysing the slope and translation of the fitted trend line one can find that those values do not differ from those given in the literature for mineral soils. This supports the applicability of the FDR method described above for the measurement of the salinity index.

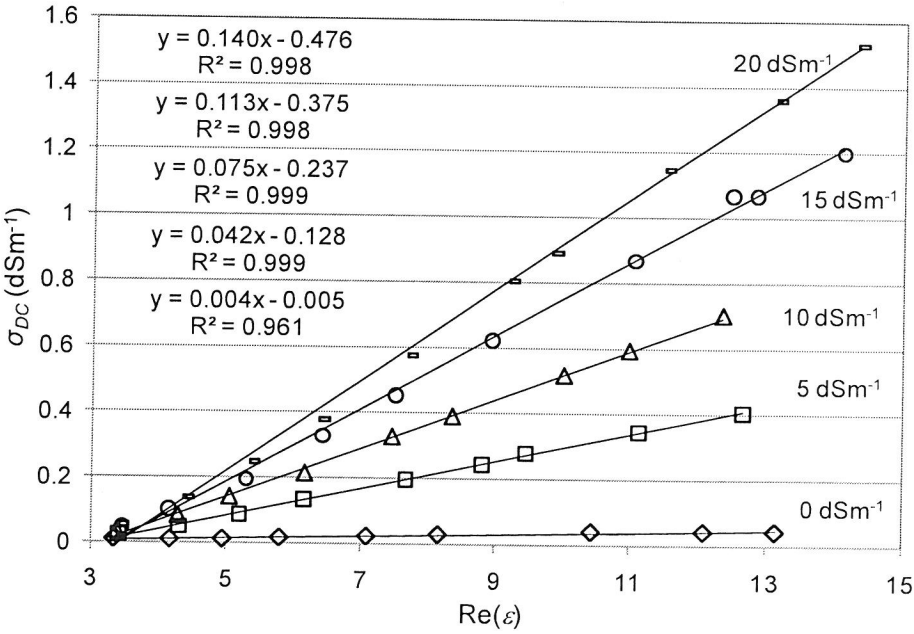


Fig. 45. Salinity index as slope of linear characteristics of electrical conductivity of soil versus real part of dielectric permittivity

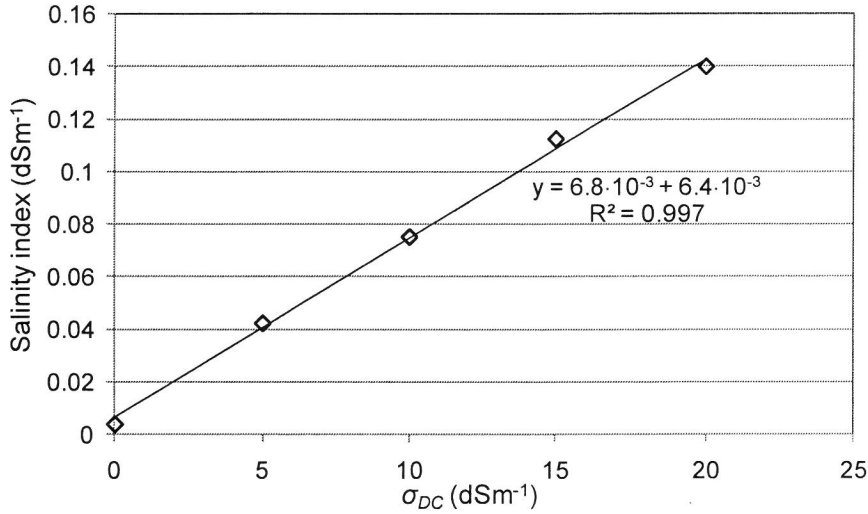


Fig. 46. Dependence of salinity index on electrical conductivity KCl solutions used to moisturize soil samples

6. CONCLUSION

The two-rod probes can be used for the study of the spectrum of complex dielectric permittivity in frequency domain. In this paper we presented an example of the application of probes with three different rod lengths. In the experiments we used a vector network analyser and developed models of the probe types considered. Due to the specific nature of measurements in porous materials, related with the occurrence of non-homogeneity, we focused on improving the model of probe with 3 cm rod length. The best model proved to be the model of lossy transformer of impedance with calculation algorithm and method of calibration. The performance of the model was positively verified by the results of measurements in methanol. Table 9 presents a comparative compilation of the FDR method described against the TDR method.

The developed model of FDR probe permitted the study of frequency spectrum within the range of 10-500 MHz, hitherto unattainable for commercial instruments in application for the measurement of soil moisture and salinity. A number of spectra of the complex dielectric permittivity in soil samples with varied salinity and moisture were examined. The two-rod FDR probes applied and the method of study of the spectrum of complex dielectric permittivity permitted selective and simultaneous determination of soil moisture and salinity for various ranges of measurement signal frequency. Particularly interesting were the results

Table 9. Comparison of dielectric measurement method TDR and FDR

Method	TDR	FDR with spectrum analysis
Calibration	2-point; water – air, or water – known dielectric	3-point; water – air –known dielectric
Amount of data	Reflectogram, 1024 points	Spectrum, 99 points of coefficient of reflection S11 from the range of 10-500MHz
Availability of field instruments	Available	Unavailable
Possibility of obtaining the spectrum of frequency	Necessary modification of the probe and Fourier transform after performing 3-point calibration typical for the FDR method	Possibility of direct determination with the use of the lossy transformer model
Determination of dielectric permittivity of material studied	On the basis of analysis of reflectogram taking into account blurring and drop of amplitude of reflected impulse the apparent dielectric permittivity is obtained	Directly for selected frequency band or for a single value from the spectrum, separately the real and the imaginary components of dielectric permittivity
Determination of electrical conductivity	From attenuation of reflected signal amplitude	From imaginary component of spectrum.
Determination of dielectric loss	Negligible only after modifications aimed at obtaining frequency spectrum	From imaginary component of spectrum.
Level of complexity of measurement instrument	Very complicated, electronic elements hardy available	Complicated, elements easily available
Measurement of soil moisture	Comparable with oven-dry method	Comparable with oven-dry method
Cost of instrument	~2000 Euro	Estimated cost of prototype ~200 Euro
Energy saving in monitoring systems	1.68 Ws/measurement	0.4 Ws/measurement
Application in remote sensing systems	Possible	Possible
Multi-channel measurements	Necessary use of multiplexers	Integrated probe

of study of the effect of measurement signal frequency on the correlation between the volumetric moisture of samples, determined with the gravimetric method, and the refractive index. It was found that for the soils tested with the developed FDR probe the values of the real component of dielectric permittivity in the range of low frequencies were overestimated relative to those determined in the range of high frequencies, especially in the case of soils with high electrical conductivity. No effect was observed of the salinity of the soil samples on the accuracy of measurement of the real component of dielectric permittivity measured within the range of 390-485 MHz. The results confirmed the favourable effect of the application of high frequency f measurement signal on the selectivity of measurement of the real component of dielectric permittivity and, in consequence, of the volumetric moisture of soil. It was also found that the obtained results of measurement of the imaginary component of dielectric permittivity permit the determination of soil salinity with the use of the salinity index.

It was demonstrated that the results of measurement of the moisture and salinity of the soils studied, determined with the developed FDR method, are comparable to corresponding results obtained with the TDR method. Additionally, the measurements of the frequency spectrum of the complex dielectric permittivity create the possibility of estimating other physical parameters of soil, *e.g.* the amount of bound water.

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8. SUMMARY

The study explores methodology of soil moisture and salinity measurement based on FDR frequency domain reflectometry technique. As compared to the TDR time domain reflectometry this technique has potential possibility of wider application by building more economical measurement devices. The fundamental part of the study concerns the design of two-rod probes and methodology allowing to obtain the frequency spectrum of soil complex dielectric permittivity. Especially interesting were results of frequency dependent correlation between volumetric water content of soil measured by thermogravimetric method and the soil refractive index. The results confirmed the advantage of using high frequencies of signal on the measurement selectivity of the real part of soil dielectric permittivity. It was found that the results obtained for low frequencies were dependent on soil salinity. The obtained results of imaginary part of dielectric permittivity allow to calculate soil salinity by using salinity index. The measured data of moisture and salinity of investigated soils determined by FDR technique were comparable with the corresponding ones determined by TDR technique. Moreover, the measurement of frequency spectrum of complex dielectric permittivity give possibility to evaluate other physical parameters of soil, for example the volume fraction of bound water.

Keywords: agrophysics, dielectric permittivity, reflectometry, soil moisture, soil salinity

9. STRESZCZENIE

WPŁYW WILGOTNOŚCI I ZASOLENIA GLEBY NA JEJ PRZENIKALNOŚĆ ELEKTRYCZNĄ

Praca podejmuje badania nad metodyką pomiaru wilgotności i zasolenia gleby w oparciu o technikę reflektometrii w dziedzinie częstotliwości (FDR – frequency domain reflectometry). W odróżnieniu od techniki reflektometrii w dziedzinie czasu (TDR – time domain reflectometry) metoda FDR ma potencjalne możliwości szerszego zastosowania poprzez zbudowanie tańszych urządzeń pomiarowych. W pracy przedstawiono opracowane sondy i metodykę pomiaru widma częstotliwościowego zespolonej przenikalności dielektrycznej gleby. Szczególnie interesujące okazały się wyniki badań wpływu częstotliwości sygnału pomiarowego na korelację pomiędzy wilgotnością objętościową próbek wyznaczoną metodą grawimetryczną i współczynnikiem załamania. Wyniki potwierdziły korzystny wpływ zastosowania wysokiej częstotliwości sygnału pomiarowego na selektyw-

ność pomiaru części rzeczywistej przenikalności dielektrycznej i w konsekwencji wilgotności objętościowej gleby. Wykazano również, że pomiary uzyskane dla niskich częstotliwości podatne są na wpływ zasolenia badanych gleb. Stwierdzono, że uzyskane wyniki pomiarów części urojonej przenikalności dielektrycznej pozwalają na wyznaczenie zasolenia gleby z wykorzystaniem wskaźnika zasolenia. Wykazano, że wyniki pomiaru wilgotności i zasolenia badanych gleb wyznaczone opracowaną metodą FDR są porównywalne z odpowiednimi wyznaczonymi metodą TDR. Dodatkowo, dzięki pomiarom widma częstotliwościowego zespolonej przenikalności dielektrycznej stwarzane są możliwości do oceny innych parametrów fizycznych gleby, np. ilości wody związanej.

Słowa kluczowe: agrofizyka, przenikalność elektryczna, reflektometria, wilgotność gleby, zasolenie gleby

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