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Complex Permittivity Measurement of High Loss Liquids and its Application to Wine Analysis

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1. Introduction

The microwave dielectrometry of high loss liquids is discussed in the present work. For such liquids the ratio of the imaginary to the real complex permittivity parts is of the order of one or more. High loss liquids are water and water solutions of organic and non-organic substances. Water is the most investigated liquid during long time. The reason for the impressive interest to water is due to its ubiquity in our environment. Usually water is used as the reference liquid for differential measurement of water-containing liquids under test, for instance, grape wines and musts. The present paper is directed to the control of naturalness (authenticity) of grape wines and musts by their complex permittivity determination. This complex permittivity determination will be performed in the millimeter wave band where the frequency dispersion of complex permittivity of water has a maximum.

It is necessary to underline that the wine trade is a multi-million-income industry in which frauds can have enormous financial repercussions and the quality of products directly impacts on human health. A universal wine and must authenticity identification method does not exist. Each of known methods (the methods of analytical chemistry, wine sampling, physical-chemical methods) have own advantages and disadvantages and assume a definite area of usage [Organisation internationale de la Vigne et du Vin, 2009]. Thus, the development of alternative methods of wine product quality determination remains actual.

One feature of the wine trade in Ukraine and other former Soviet Union countries is the increase of fraud. The main types of fraud are unregulated sugar use, substitution of the sort of vine, not keeping the ripening terms for the wines, water addition, blending of cheap wines instead the best wines, addition of artificial sweeteners, dyes, aromatizers, and fake wine production without vine processing. In Europe and USA there is fraud growth by bottling inferior wine under a quality label – with the wine originating from a different region or even country than that it is stated.

Despite the rigor of wine quality demands fake wine production did not decrease due to lack of reliable methods for identification and suitable reference data [Ezhov et al., 1999].

The main part of fraud (up to 90-95%) is associated with wines that result of the fermentation of sugar-water or diluted grape must solution. Organoleptic determination of such wines is possible but is not reliable. For reliable examination there are both classical analytical methods (the determination of the density, extract, mineral substance and so on) and methods based on isotope patterns and nuclear magnetic resonance. The major inconvenience of these techniques is that they are generally time-consuming and/or expensive and not suitable for continuous monitoring. Therefore, the development of the reliable methods of wines and fraud identification is still actual.

The authenticity control of wine and fruit musts is a quite complicate problem. At present the great number of parameters is used for that purpose, such as the alcohol content, sugar, acidity, and extractable substances with and without sugar etc. Additional parameters used are the number of anions (Cl^-, SO_4^{2-}) and cations (K^+, Na^+, Ca^{++}, Mg^{++}), organic acids (tartaric, malic, lactic, citric), pH etc. In grape, fruit must, and wine production the measurement of density, pH, buffer capacity, conductivity, viscosity, and optical density are used to manage the quality as well.

The great number of parameters required determining the authenticity and the time for their determination make the testing of wine production during the technological process is really complicated. European scientists have known for almost a decade that analysis of various chemical elements in wine can help to identify roughly the geographic origin of wines, but the tests have been too complex and not conclusive enough to be used on a large scale.

The necessity to measure small changes of complex permittivity of high loss liquids at millimeter wave band arises at biophysical research of native albuminous molecule conformation. At the preparation of calibration data during distance sounding of water surfaces and interpretation of sounding results, at physical-chemical study of properties of water solutions and so on. One of the important practical usages of dielectrometry of high loss liquids is the identification of wines and musts and their authenticity. Differential complex permittivity measurement has an advantage towards to absolute complex permittivity measurement when it is necessary to measure small complex permittivity changes in liquids under study.

2. Techniques for high loss liquid complex permittivity measurement

Numerous techniques for complex permittivity determination of high loss liquids are applied at microwave frequencies [Sato & Buchner, 2004; Masaki at al., 2007; Agilent 85070E, 2008; Buckmaster et al., 1985; Hu, et al., 1994; Cherpak et al., 2004]. Among of them it is well known that both resonator [Afsar & Ding, 2001] and waveguide [Afsar & Suwanvisan, 2005] methods are used for the complex permittivity measurement of high loss liquids.

The resonator methods use the complex resonant frequency of a resonator to obtain complex permittivity of liquid. And the waveguide methods use the complex wave propagation factor in a waveguide segment under study. The significant limitation of the above-mentioned methods is a high attenuation observed in the high loss liquid measurements. Moreover, the high loss liquid has large real and imaginary complex permittivity parts (much greater than 1, specifically, for water) and these values are of the same order. Therefore, measured parameters (the resonant frequency and Q-factor in a resonator or the wave phase and wave attenuation in a waveguide) depend simultaneously both on real and imaginary complex permittivity parts of high loss liquids. Owing to this fact the complex permittivity determination becomes considerably complicated while using measured

parameters. For the resonator method, the Q-factor is usually rather small (it is approximately 10-100 for enough complex permittivity measurement sensitivity) and as a result it is difficult to determine the resonant frequency with needed accuracy. Concerning to the waveguide method, the wave attenuation is high at the propagation of the wave even via a small length (compare to skin layer in liquid) waveguide section at presence of high loss liquid like water or water solutions.

There are a number of papers where the complex permittivity determination of high loss liquids was studied using different resonator methods. For example, in Cherpak's papers whispering-gallery modes in cylindrically shaped dielectric resonators were studied to obtain complex permittivity of high loss liquids. A resonator as a radially two-layered dielectric disc placed between conducting endplates with an internal layer filled with air or loss liquid such as water, ethyl alcohol, benzene and aqueous solutions of ethyl alcohol was studied [Barannik et al., 2007]. In the paper [Shaforost, 2009] it was described a novel approach of high sensitivity liquid analysis for volumes in the nanolitre range with challenging perspectives for practical sensor applications in chemistry, biology and medicine. Whispering-gallery modes in cylindrically shaped dielectric disks machined from low-loss single crystalline materials such as sapphire or quartz allow having very high quality factors. The interaction of extremely small volumes of the liquid under test with the evanescent field located in the vicinity of the dielectric disk surface at micro-to-millimeter wave frequencies was employed for the investigation of aqueous solutions with relevance to biological applications. Based on this resonator type, three different liquid sensing approaches were developed and analyzed at 10, 35 and 170 GHz with emphasis on the determination of the complex permittivity of liquids of nanolitre volumes. In [Cherpak, 2006] electrodynamics' properties of quasi-optical dielectric resonators of two types with liquid-filled small cavities have been studied. One of the quasi-optical dielectric resonators types is a two semi-disc resonator with a diametrical slot in which a cavity with a thin ($0.01 \div 0.1$ mm) flat liquid layer is placed and the other one is a disc resonator with a small diameter ($0.3 \div 2$ mm) cylindrical capillary. Measurements have been carried out at room temperature in K_a-waveband by using the resonators made of Teflon material. The obtained results allow to conclude that the given approaches to the development of measuring technique for characterization of liquids (for example, water and aqueous solutions) are quite perspective. In [Shaforost, 2007] they showed that whispering-gallery mode dielectric resonators from 10 GHz to 3 THz are attractive for highly sensitive liquid detection and identification of small droplets down to volumes of picolitres. Since droplets are usually generated by computer controlled microinjection pipettes being moved on a 2D scanning table, free access to the sensitive resonator surface from above is essential.

To obtain small complex permittivity differences of various substances, in particular, high loss liquids differential measurement methods are widely used. The necessity of small complex permittivity difference registration for high loss liquids appears, for instance, during biological study of conformation changes of native protein molecules [Pethig, 1992] or reference data preparation for the device calibration of water surface remotely sensing [Ellison, 2007]. The dielectric properties of free water are well described by the Debye formula of the second order. Dissolved molecules in water can change the relaxation time both due to coupling of part of free water molecules (hydration) and due to the rebuilding of hydrogen bonds in water clusters. The latter mechanism is common for water - ethanol mixtures [Sato & Buchner, 2004]. Owing to mentioned reasons of complex permittivity measurement of water solutions is sensitive to the variation of their chemical composition at Ka band.

Known waveguide-differential complex permittivity measurement methods distinguished by type of the measurement cell usage due to dielectric properties of liquids and measurement conditions. So, the cells based on waveguides completely filled with high loss liquid [Buckmaster et al., 1985], and also cells with waveguides with capillary with liquid put inside of the waveguide [Masaki et al., 2007] are used. It is known the usage of dielectric waveguides for complex permittivity measurement of high loss liquid [Meriakri & Parkhomenko, 2000]. In a set of Buckmaster's papers for the development and improvement of complex permittivity measurement technique for light and heavy water at 9.335 GHz it is shown the possibility to reach high accuracy of absolute measurements [Buckmaster et al.,1985; Hu et al., 1994]. The measurement setup in [Hu et al., 1994] was a microwave bridge contained a waveguide cavity with a precise length change of a part of rectangular waveguide filled with water. They used thermo stabilization for the cavity with the error of the order of 0.005°C and a high sensitivity superheterodyne receiver with double frequency transformation. After this transformation the precise measurements carried out at frequency 1 kHz. As a result they reached the relative measurement error for the real and imaginary complex permittivity parts the order of 0.1% and 0.2%, respectively. The 1σ standard deviations both for real and for imaginary complex permittivity parts do not exceed 0.02%. According to own metrological characteristics the setup in [Hu et al., 1994] can be used for study the dielectric properties of wines and musts with needed accuracy. It is necessary to have laboratory conditions and the cavity of this device does not correspond to carry out mass measurements.

The upper limit frequency for waveguide methods usage with reasonable errors is 140 GHz [Jain & Voss, 1994]. The majority of such methods are based on the impedance measurement of waveguide system where a sample under study is placed. Possible study at wide frequency band both solid and liquid matter. The most suitable objects are media with average dielectric losses ($0.1<\mathrm{tg}\delta<1$, where δ is angle of dielectric losses). The measurement error real complex permittivity part ε' is often 0.5%, and $\mathrm{tg}\delta$ - 3-5%. It is known several types of waveguide methods. So, completely filling with liquid here the waves propagation is studied in waveguide [Brandt, 1963]. If a sample is absent in the line there is pure standing wave. In [Van Loon & Finsy, 1974] they used the similar method to measure liquid samples at the frequency range from 5 to 40 GHz and from 60 to 150 GHz. At that they underlined the practical difficulties at waveguide usage at the frequencies up 40 GHz due to not enough mechanical power and attenuation increase in waveguide. The authors saw two main obstacles to reach high accuracy: there is undesirable reflection and attenuation in waveguide walls and there are inhomogeneities where higher modes are excited. In the papers [Afsar et al., 2005] and [Fuchs & Kaatze, 2002] the authors are also paid great attention on the analysis of possible errors and recommendations to increase the measurement accuracy. For long wave part of millimeter wave band they decreased the error of $\Delta\varepsilon'/\varepsilon'$ and $\Delta\varepsilon''/\varepsilon''$ up to 0.02. In the paper [Alekseev & Ziskin, 2001] measured the reflection, propagation and attenuation of electromagnetic waves at the interaction with film samples (water and water-ethanol solutions). At 42.25 GHz and 53.35 GHz it is studied the influence of film thickness and the character of attenuation in the cell depending on the thickness and design of isolated interlayer as well. The authors found out that optimal thickness of a sample when the straight-line attenuation of electromagnetic waves takes place is interval of 0.28 - 0.33 mm.

Alison and Sheppard [Alison & Sheppard, 2001] designed a dielectrometer where the liquid layer was changed using short-cut plunger that moved into the liquid. The layer thickness was measured with the step of 0.06 mm. Complex permittivity was obtained at 29 – 44 GHz using the measurement the reflection coefficient for two different thicknesses. The liquid separated from the empty part of a waveguide by means of windows made of organic glass. The appearance of higher modes in transmission section the authors did not observed. With the same technique the authors measured complex permittivity of human blood at 29 – 90 GHz [Alison & Sheppard, 1993]. They found out the existence of the field of additional (except Debay's ones) high frequency dispersion.

In the paper [Zanfolin, 1983] they described the measurement device that allows to obtain complex permittivity of high loss liquid at millimeter wave band. The basis of a dielectrometer is waveguide interferometer (bridge) where in measurement shoulder there is a cell with liquid that is irradiated from open waveguide end. The thickness of a sample is changed using a plunger moving by micrometer screw. As a result, an input signal in the bridge is a function of the thickness of liquid. The authors give the results obtained for ethanol, methanol and pure water at 20°C and 70 GHz. Later the dielectrometer was improved. New variant [Buckmaster et al, 1985] contained two channel superheterodyne system with twice frequency transformation. The authors analyzed the origin of errors and elaborated the ways of their decrease. For that purpose they carried out maximum exact measurement of length of a sample, there was increased the frequency and stability of the signal.

In spite of the various considered methods of high loss liquids complex permittivity measurements, the problem now still actual due to the difficulties of measurement accuracy and it must be at least the order of 0.01% as our measurement showed to identification of water solutions of chemical and biological media and their differences between each others.

One of the possible practical usages of the dielectrometry of high loss liquids is wine and must identification in order to determine their authenticity [Watanabe, 2009]. As it was mentioned above, wines and musts are high loss liquids, because of mass fraction of water in their content is the order of 75-90%. It was proposed to use complex permittivity measurement data to control the stages of sake fermentation - the main biochemical process of wine-alcohol industry [Masaki et al., 2007]. As known the change of quantitative chemical wine composition, for example, for dry (table) natural wines in specification standard product limit influences on complex permittivity values within the scope the order of some percents. Therefore, for the solving of wine identification problem it is necessary to have a dielectrometer that has much higher sensitivity in comparison with complex permittivity deviation for high loss liquids. It is desirable to have a device with differential sensitivity at least the order of 0.1% or higher. Authors in [Watanabe, 2009] could not identify the change of different samples of dry wines components except ethanol at Ka waveband just because of small differential sensitivity. The experimental tool in [Watanabe, 2009] was standard probes for the measurement of complex reflection coefficient in liquids by means of vector analyzer [Agilent 85070E, 2008].

Our goal is to develop a dielectrometer for carrying out the express wine product analysis at industrial chemical laboratories by staff with average technical secondary education level. The device must possess enough high differential sensibility (at least the order of 0.1%). High accuracy of absolute wines and musts complex permittivity measurement does not obligate, because the main requirement is the measurement of wave propagation coefficients difference in two measurement liquid-filled cells with similar dielectric properties. We

reached the assigned task by the use of a measurement cavity [Ganapolskii et al., 2009] and made complete automated measurements.

There are no restrictions for liquid under test for a volume size during wine and must complex permittivity measurement i.e., the cavities of rather big volume can be used and the cavity clearing can be made by means of the liquid under test as well. As a result, measurement errors caused by a cavity with small size or inhomogeneity of cavity filling are minimized. The decrease of accuracy at the measurement in small volume of the liquid is common for a set of known cavities, for instance, a capillary with a liquid under test in a rectangular waveguide [Masaki et al., 2007] or a thin ring belt cavity with a liquid under test in a shielded cylinder dielectric resonator [Eremenko et al., 2009].

A measurement waveguide type cavity proposed in [Ganapolskii et al., 2009] consists of two open measurement cells with a dielectric (quartz) cylinder rod inside of them. These two cells are filled with a reference liquid and a liquid under test, respectively. The operating principle of these cells is based on the dependence of electromagnetic wave propagation coefficients on dielectric properties of a surrounded dielectric rod liquid. The difference between the attenuation and phase coefficients of the wave propagation in two measurement cells is the measured data. The attenuation coefficient of the wave propagated along the quartz rod immersed into water is rather small (the order of 10 dB/cm). The influence of the reflection off a cell body can be neglected during the wines and musts complex permittivity measurements, if the diameter of the cell is two times more than a rod diameter [Ganapolskii et al., 2009]. These reasons create comfortable conditions for the measurement open cavity design to carry out precise express complex permittivity measurements of high loss liquids.

The main goal of our measurements is to detect small complex permittivity differences of two liquids with similar dielectric properties. For such a goal the knowledge of absolute complex permittivity liquid values has reference character only and we can limit ourselves by small difference of two wave propagation coefficients values for two cells. It is preferentially to minimize measurement errors. The errors of indirect complex permittivity measurements can be calculated if we know the errors of direct measurements of wave attenuation and phase coefficients in the measurement cell. For example, relative errors of the real and imaginary complex permittivity parts are associated with absolute standard deviations of independent measurements of phase and attenuation coefficients $\delta h'$ and, $\delta h''$ respectively. So, we obtain for 10% ethanol in water: $\delta\varepsilon'/\varepsilon' = 0.31\%$ and $\delta\varepsilon''/\varepsilon'' = 0.046\%$; for 30% ethanol solution - $\delta\varepsilon'/\varepsilon' = 0.27\%$ and $\delta\varepsilon''/\varepsilon'' = 0.097\%$. Here the indirect complex permittivity measurement errors are in several times higher than direct measurement errors of wave propagation coefficients. The errors of indirect complex permittivity measurement in [Hu et al., 1994] are also higher than the errors of wave propagation during the direct measurement.

We proposed an original measurement cell with an innovation part as a dielectric rod immersed into high loss liquid [Ganapolskii et al., 2009]. The dielectric rod is an open waveguide system where the complex electromagnetic wave propagation constant depends on complex permittivity of liquid. The electrodynamic waveguide structure similar to the proposed one was studied during the electromagnetic wave propagation in tunnels [Holloway et al., 2000]. We elaborated and tested the laboratory sample of a waveguide differential dielectrometer where the designed cell was used. The device, analysis of its performance, and measurement results are present in this report. We analyzed the influence of different reasons that impact on the measurement errors and we elaborated the

conditions to minimize these errors. As a result the error was decreased in ten times in comparison with the data presented in [Jain & Voss, 1994].

3. High precision computer-aided dielectrometer for high loss liquid

Fig.1 presents the appearance of the computer-aided dielectrometer. An operator only fills in and out liquids in the cavity cells. We designed this instrument in order to make up routine high precision measurement for qualitative control of wines and must in biochemical laboratories [Eremenko & Skresanov, 2010]. The determination of high loss liquid complex permittivity is done by means of a computer program. An initial data for computation are the measurement difference of wave attenuation and phase coefficients in two dielectrometer cavity cells. These cells filled with a liquid under test and a reference liquid, e.g. the distilled water.

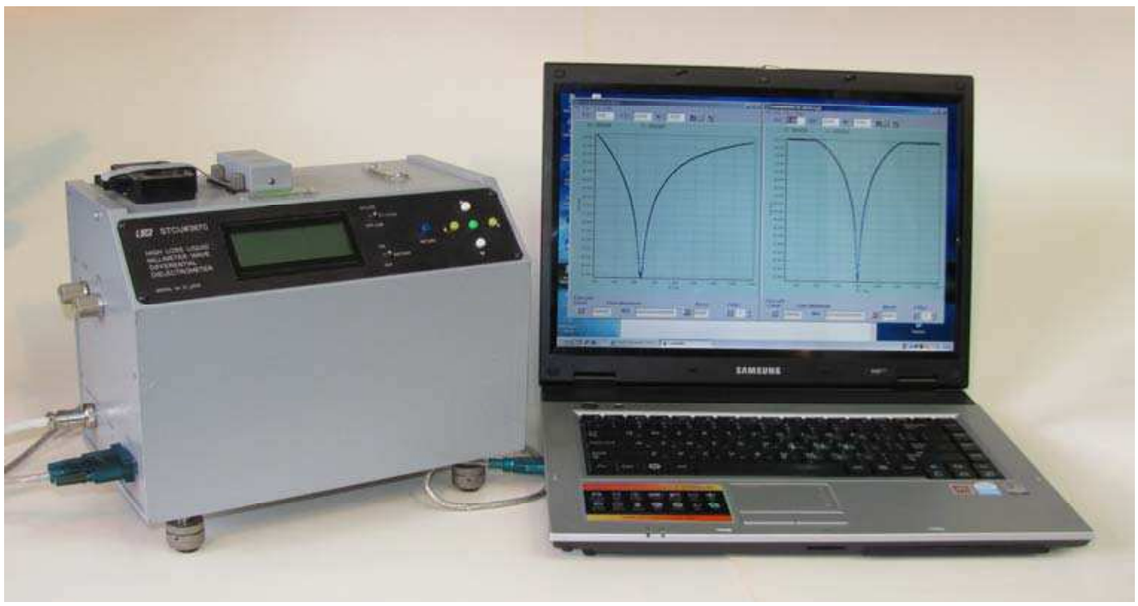


Fig. 1. The appearance of the differential dielectrometer connected with a notebook.

For such measurements we proposed the novel differential cavity [Ganapolskii et al., 2009]. This cavity consists of two identical cells that are cylinder glasses of diameter $D = 20$ mm that are made of a common copper body 1 (Fig.2). Quartz rods 2 were inserted perpendicularly to the side walls. The glasses were filled with liquids 3 (the water solution under test and its solvent – distilled water) at open thermo isolated covers 4 up to the level of overflow holes 5. After measurements liquids were poured out using draining holes 6. The temperature sensors 7 placed into covers 4 are for the temperature control in the cells. Microwave power comes to measurement cells via standard millimeter wave rectangular waveguide 8. The cells are matched with rectangular waveguides by round waveguides sections 9 filled with Teflon. The standing-wave ratio does not exceed 1.05 for the cells input and output at operating frequency 31.82 GHz. The cavity principle of operation is based on the dependence of the wave propagation coefficients in quartz rods on dielectric properties of an outer medium [Ganapolski et al., 2009]. The attenuation coefficient of the wave propagated along the quartz rods inserted into high loss liquid is rather small (the order of

10 dB/cm). The influence of the glass side faces can be neglected at the distance at least of the order of wavelength from the rod surface [Eremenko & Skresanov, 2010].

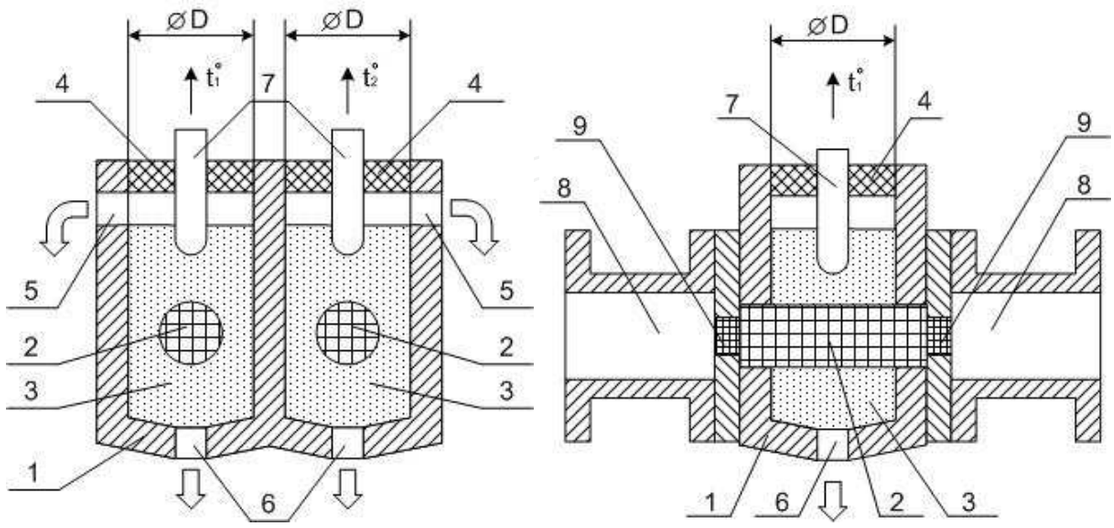


Fig. 2. The schematic picture of dielectrometer differential measurement cavity: the cross section of the cavity (left) and longitudinal section of one of the cavity cells (right). 1– the differential cavity body; 2 – the quartz cylinders; 3 – the liquids; 4 – the covers; 5 – the overflow holes; 6 – the drain holes; 7– the temperature sensors; 8 – the rectangular waveguide sections; 9 – the round waveguides section filled with Teflon.

We can obtain the complex permittivity values from the characteristic equation for the infinite rod in high loss medium [Ganapolskii et al., 2009]

$$\left(\frac{J'_m(g_1a)}{g_1aJ_m(g_1a)}-\frac{H'_m(g_2a)}{g_2aH_m(g_2a)}\right)\left(\frac{\varepsilon_1 J'_m(g_1a)}{g_1aJ_m(g_1a)}-\frac{\varepsilon_2 H'_m(g_2a)}{g_2aH_m(g_2a)}\right)=\frac{m^2h^2(g_2^2-g_1^2)^2}{k^2g_1^4g_2^4a^4}, \quad (1)$$

where $J_m(x), H_m(x)$ – Bessel and Hankel of the order of m and of the first kind; $g_1=\sqrt{k^2\varepsilon_1\mu_1-h^2}$, $g_2=\sqrt{k^2\varepsilon_2\mu_2-h^2}$; k – the wave number in vacuum; h – the longitudinal wave number; $\varepsilon_1=\varepsilon_1'+j\varepsilon_1''$, $\varepsilon_2=\varepsilon_2'+j\varepsilon_2''$ – the complex permittivity of the rod and liquid, respectively; a – the radius of the rod; $\mu_1,\mu_2=1$ – the permeability of the rod and liquid. The typical relations between real and imaginary complex permittivity parts at our measurements are the following

$$\varepsilon_1''<<\varepsilon_1', \quad \varepsilon_1'<\varepsilon_2', \quad \varepsilon_1''<<\varepsilon_2'', \quad \varepsilon_2''/\varepsilon_2'\geq 1. \quad (2)$$

The ratio between the impedance of dielectric material of the rod and the liquid (water solutions) $\zeta=\sqrt{\varepsilon_1'}/\sqrt{\varepsilon_2'}$ is approximately equal to 0.5. We can obtain a set of complex roots h_{mn} at definite operating frequency, complex permittivity of liquid, radius of the rod, and its complex permittivity. The azimuth index m is equal to the number of half-wavelengths placed along azimuth coordinate φ from 0 to 2π; the radial index n is equal to the number of half-wavelengths placed along radial coordinate inside the rod from 0 to $r=a$. The

analysis of electromagnetic field distribution for the corresponding wave number h_{mn} shows that a set of four wave types can be excited in the dielectric rod immersed into high loss liquid. Two of them are as follows. The transverse-electric waves (TE_{on}) and transverse-magnetic waves (TM_{on}) have no z- field component of electric or magnetic field, respectively. Two other types have non-zero z- field components and they are quasi-TE_{mn} or quasi-TM_{mn}. In general, any of the mentioned above waves can be used for our measurement. We used quasi-TE_{11} wave type, because it can be easily excited in the rod by a rectangular or a round waveguide with the basic wave types H_{10} or H_{11}.

The technique of complex permittivity determination is as follows. The wave attenuation $h_r''[\mathrm{dB/cm}]$ and phase $h_r'[\mathrm{rad/cm}]$ coefficients are calculated from the characteristic equation (1) using known complex permittivity of solvent for the cell with solvent (the reference liquid). We measure the difference of attenuation coefficients $\Delta h''[\mathrm{dB/cm}]$ and phase coefficients $\Delta h'[\mathrm{rad/cm}]$ for the cells with solvent and the liquid under test. The attenuation coefficient $h_t''[\mathrm{dB/cm}]$ and the phase coefficient $h_t'[\mathrm{rad/cm}]$ of the wave in the cell with solvent are calculated using formulas: $h_t'' = h_r'' + \Delta h''$ and $h_t' = h_r' + \Delta h'$. And, finally, using equation (1) the complex permittivity of the liquid under test ($\varepsilon_t = \varepsilon_t' + i\varepsilon_t''$) is calculated with the help of obtained h_t'' and h_t'.

It is suitable to use the distilled water as the reference liquid at the complex permittivity determination of water solutions of wines and wine model liquids. In [Ellison, 2007] there is a formula to calculate complex permittivity of water at 0-25 THz and at the temperature band 0-100°C. We use this formula for the complex permittivity calculation of the distilled water at known room temperature as a liquid in the cavity.

We use the principle of differential measurement of the difference in wave attenuation coefficient $\Delta L = \Delta h'' l_{eff}^{(1)}$ and phase shift coefficient $\Delta\varphi = \Delta h' l_{eff}^{(2)}$ in the cavity cells as it was done in [Ganapolskii et al, 2009]. So-called "cell effective lengths" $l_{eff}^{(1)}$ and $l_{eff}^{(2)}$ approximately equal to glass diameter D of the cells. The measurement scheme (Fig. 3.) is a microwave bridge. The signal splitting between bridge arms is done at an oscillator output by means of E-joint, and the signal summation is done at a detector input by means of H-joint. The local oscillator at 31.82 GHz is a phase-locked loop transistor VCO at the frequency 7955 MHz with a reference quartz frequency standard and further multiplication by four. A power amplifier on the basis of the chip CHA3093c was implemented. To increase a signal to noise ratio, the amplitude modulation of a microwave carrier with frequency 100 kHz and a synchronous demodulation were used.

The amplitude of the signal at one of the bridge arms is controlled by the measurement P-I-N attenuator. The high precision short-circuiting plunger was designed for the phase shifter. This plunger is controlled by a step motor. The discrete step of the plunger motion is 2.5μm that corresponds to phase change 0.144°. The tuning of the attenuator and the phase shifter in the bridge arms is done in accordance with a microcontroller program. The microcontroller block was worked out on the basis of AT90USB1287 chip. Its main function is amplitude and phase level control in the microwave bridge arms. Besides, we measure the signal level at the receiver output, the temperature of the liquids in the cells and the temperature of the P-I-N attenuator body. We also control the level of output oscillator signal by the controller. The microcontroller block provides a user interface in manual mode and the data exchange with PC.

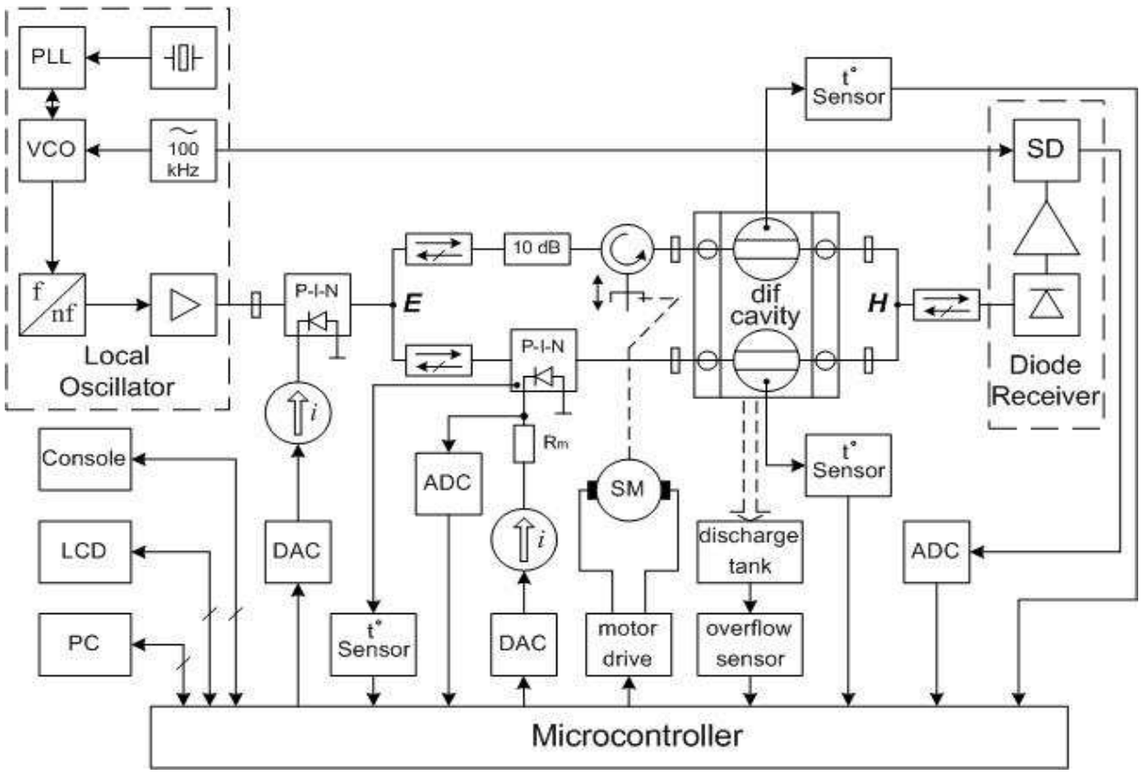


Fig. 3. The structural scheme of the differential dielectrometer.

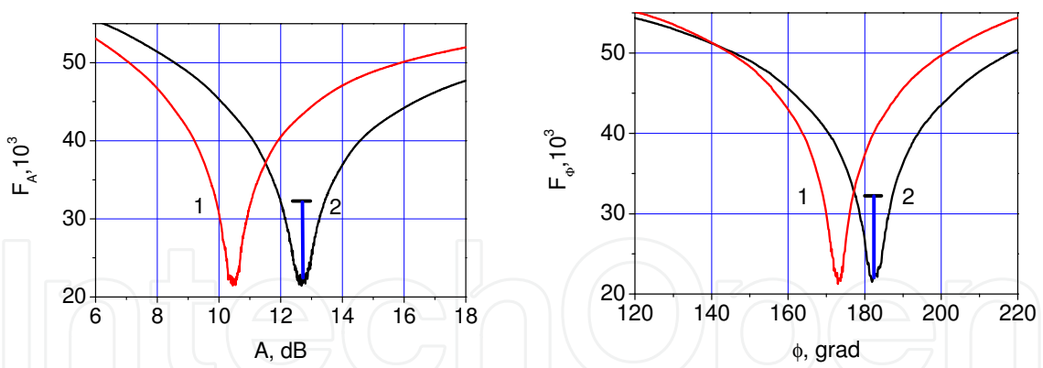


Fig.4. The amplitude (left) and phase (right) dielectrometer functions for the distilled water in two cavity cells (1) for the distilled water and (2) for the table wine in the different cells. The vertical line is the position of a minimum using "bracket" technique.

The readings $F(\delta L, \delta\varphi)$ of an analog-to-digital converter of the receiver in logarithmic units as functions of the differences in amplitude δL[dB] and phase $\delta\varphi$[grad] at the bridge arms can be written

$$F(\delta L, \delta\varphi) = \frac{\partial F_A(L_0, \varphi_0)}{\partial L}\delta L + \frac{\partial F_\Phi(L_0, \varphi_0)}{\partial \varphi}\delta\varphi\,, \tag{3}$$

where L_0 and φ_0 are the attenuation and phase in the arms of the balanced bridge.

The PC program algorithm for the recording F_A and F_Φ functions is as follows. During the first iteration the phase scanning is carried out at arbitrary fixed amplitude; the phase function minimum F_Φ is calculated; the phase shifter is returned to a minimum position; the amplitude scanning is carried out; the amplitude function minimum F_A is calculated; the attenuator is returned to a minimum position. That is the end of the first iteration. Our testing showed that in order to reach maximum accuracy of the bridge balancing it is necessary to do three iterations. In Fig.4 we present the amplitude and phase functions of the dielectrometer. These plots are displayed on PC screen in a real time scale. After curves registration the digital low frequency data filtration is made and the minimum position is calculated according to the "bracket" technique. The minimum position is the average attenuation (phase shift) at the instrumental function slopes where the signal-to-noise ratio is of the order of 10 dB. In Fig.4 the calculated minimum position for a dry table wine with respect to the distilled water is shown by vertical lines.

We carried out the detail analysis of origins and values of random and systematic measurement errors of attenuation and phase coefficients $\delta h'$ and $\delta h''$ for the designed dielectrometer. The random errors determine so-called differential sensitivity of our device i.e., the ability to recognize minimal possible differences of phase $\Delta h' \approx 2\delta(\Delta\varphi)$ or attenuation $\Delta h'' \approx 2\delta(\Delta L)$ coefficients of two liquids with close complex permittivity values. The systematic errors determine the absolute complex permittivity measurement errors.

In the designed dielectrometer we have made a number of schematic and design improvements in order to minimize random measurement errors. They are as follows: 1) the usage of the high power signal oscillator (of the order of 100 mW); 2) the usage of a high modulation frequency (100 kHz); 3) the usage of synchronous detection at the modulation frequency; 4) the usage of a low noise current controller of a P-I-N attenuator; 5) the realization of play-free mechanism of the short-circuiting plunger moving by the small discrete step. The dynamic technique of the minimum position determination of the instrumental functions of the dielectrometer leads to the minimization of random measurement errors as well. The mentioned above steps provide root-mean-square random measurement errors of attenuation ΔL and phase shift $\Delta\varphi$ that are of the order of $\delta(\Delta L) \approx \pm 0.001 dB$ and $\delta(\Delta\varphi) \approx \pm 0.05°$, respectively. This error values were estimated by recoverable measurements with the same liquid at stable ambient conditions. As a result these random errors determine the limit of differential sensibility R_h of our dielectrometer. For the liquid with dielectric properties close to the distilled water ($h' = 11.1$ rad/cm and $h'' = 8.8$ dB/cm) the differential sensibility $R_{h'} = 2\delta(\Delta\varphi)/h' \cdot 100\% \approx 0.02\%$ for the phase shift values and $R_{h''} = 2\delta(\Delta L)/h'' \cdot 100\% \approx 0.02\%$ for the attenuation ones.

Another origin of random errors is random temperature deviation for liquids in the cells. The measured mean-square temperature difference in the cells during entire measurement cycle does not exceed 0.1°C after thermal balance achievement. The entire measurement cycle consists of the microwave bridge balancing with the solvent in two cells, the replacement of the solvent in one of the cell by the liquid under test, thermal equality

reaching, and one more microwave bridge balancing. The approximate time of entire cycle is about 3 minutes. The direct calculation of temperature coefficients of real and imaginary parts of the complex wave propagation coefficient h was made. It gives $\partial h' / \partial T = -0.00566(\text{rad/cm})/^0\text{C}$ and $\partial h'' / \partial T = -0.0462(\text{dB/cm})/^0\text{C}$ in the cell with distilled water at the operating frequency. Thus, the differential sensibility caused by the temperature fluctuation in the cells will be $R_{h'} \approx 0.01\%$ for the phase coefficient and $R_{h''} \approx 0.09\%$ for the attenuation coefficient. Several measurement sets of the wave propagation coefficients in the cells with water and with 10% ethanol solutions in water were made. Each measurement was made according to the entire measurement cycle. We found out that 1σ standard deviations both for $\Delta h'$ and $\Delta h''$ does not exceed 0.06 grad/cm and 0.02 dB/cm, respectively, in absolute units or $R_{h'} \approx 0.05\%$ and $R_{h''} \approx 0.2\%$ in relative units. Obtained measurement data approximately correspond to the given theoretical estimation. If we use a cavity thermostat for the temperature of liquid stabilization, for example, with the accuracy of the order of ±0.01°, then the differential measurement sensitivity will be of the order of 0.01% both for real and imaginary parts of complex wave propagation coefficient.

The absolute complex permittivity measurement error consists of mean-square random errors mentioned above and a number of systematic errors. We analyzed the following systematic errors: 1) a method error $(\delta h)_1$ due to uncertainty of effective length of the cavity. This error exists owing to diffraction effects at excitation of the quartz cylinder in the liquid by the waveguide; 2) an error of absolute calibration $(\delta h)_2$ of the attenuator and the phase shifter; 3) an error $(\delta h)_3$ due to ambient space temperature deviation; 4) an error $(\delta h)_4$ due to parasite phase (attenuation) deviations at attenuation (phase) turning in the microwave bridge arms. One more origin of a method error $(\delta h)_5$ does not have direct connection to quality of measurements. This is the statistical complex permittivity uncertainty of the reference liquid (the distilled water).

The key contribution in absolute measurement accuracy is the error of the uncertainty of the effective length of the cell, which was estimated numerically by 'CST Microwave Studio'. We obtained $(\delta h')_1 / h' \le 1\%$ for the phase coefficient and $(\delta h'')_1 / h'' \le 0.5\%$ for the attenuation coefficient at whole measurement range of any table wines and musts. But this error does not impact on the differential sensibility of our device for the liquids under test with complex permittivity values difference is less than 5 units. The measured value of the temperature attenuation coefficient of the P-I-N attenuator does not exceed 0.03 dB/°C. In order to minimize $(\delta h)_3$ we inserted a temperature numerical correction by the PC program based on a measured temperature deviation of the attenuator body. The final calibration P-I-N attenuator error does not exceed 0.1% at the total attenuation deviation range and the ambient temperature. The most essential origins of the systematic error of phase shift measurement are parasite deviation of the wave phase passed via the P-I-N attenuator at the attenuation control. It is minimized by our PC program as well. According to our estimations the maximal phase shift measurement error due to all reasons does not exceed 0.4° or 0.06%. Summing up all systematic errors $(\delta h)_i$, $i = 1,2,3,4$ we obtain the total absolute phase

coefficient measurement error $(\delta h')_{\Sigma} \approx 6.2^{0}$ or 1.1% and the total absolute attenuation coefficient measurement error $(\delta h'')_{\Sigma} \approx 0.05$ dB or 0.6%.

4. Results of complex permittivity measurement of wine and wine model liquids

All results presented in this section were obtained by means of our designed dielectrometer. We carried out a set of complex permittivity measurement wines and musts (some results were published in [Eremenko, 2009, Anikina, 2010]. More than 100 dry table wines samples were under test. As an example, the measurement results are presented in Fig.5. We obtained histograms for the increment of real $\Delta h'$ and imaginary $\Delta h''$ parts of complex wave propagation in the cell with dry table wines and musts relative to the wave propagation in the cell with the distilled water. In Fig.5 the calculation results of absolute complex permittivity values for the same wine and must samples are presented as well. All wines satisfying to the nowadays quality standard for the dry natural wines were made of musts-self-flowing using the following types of grapes: Chardonnay, Aligote, Riesling Rhine and Rkatsiteli of 2007 harvest that were obtained using microvinification technique.

We observed small but valid distinctions in the complex permittivity and the complex wave propagation coefficients for various sample wines (musts). We also obtained 100 % correlation of the complex permittivity and the wave propagation coefficients of wine samples and corresponding samples of musts. It is interesting to note, that we can recognize distinctions in the complex permittivity and the wave propagation coefficients for wines and musts of the same sort of grapes (Riesling Rhine) with different vintage dates. The additional study has been shown that it can be explained by different sugar content in these musts.

We carried out quantitative analysis of wines and musts chemical content. The essential correlation between the complex permittivity and wines (musts) chemical content was obtained. The possibility to identify wines according to grapes growing regions or a wine sample with wrong production technology was shown. For complex permittivity measurement method it is necessary to have the data of complex permittivity of model liquids: water solution of chemical wine composition elements that are combined in different proportions. The complex permittivity measurement of model liquids allows establishing cause-and-effect relations between concentrations of the solution components and complex permittivity of solutions.

As an example of the complex permittivity of model liquids in Fig.6 we present the measurement results of the differences between the complex permittivity of water and water solutions of glucose, glycerol, and ethanol at 31.82 GHz at temperature 25°C. We apply the complex permittivity of the distilled water (25.24+i31.69) at the same conditions. The concentration of solution components is presented in the mole ratio, i.e. the number of diluted substance molecules on one molecule of a solvent (water). The confidence measurement interval is ±0.007 dB/cm for the attenuation coefficient and is ±0.05 grad/cm for the phase coefficient. Errors of substances concentrations in solutions are higher, but they do not exceed some tenth of percents. It is necessary to note that we compared with other authors complex permittivity data of water-ethanol solutions presented in [Ganapolskii et al, 2009].

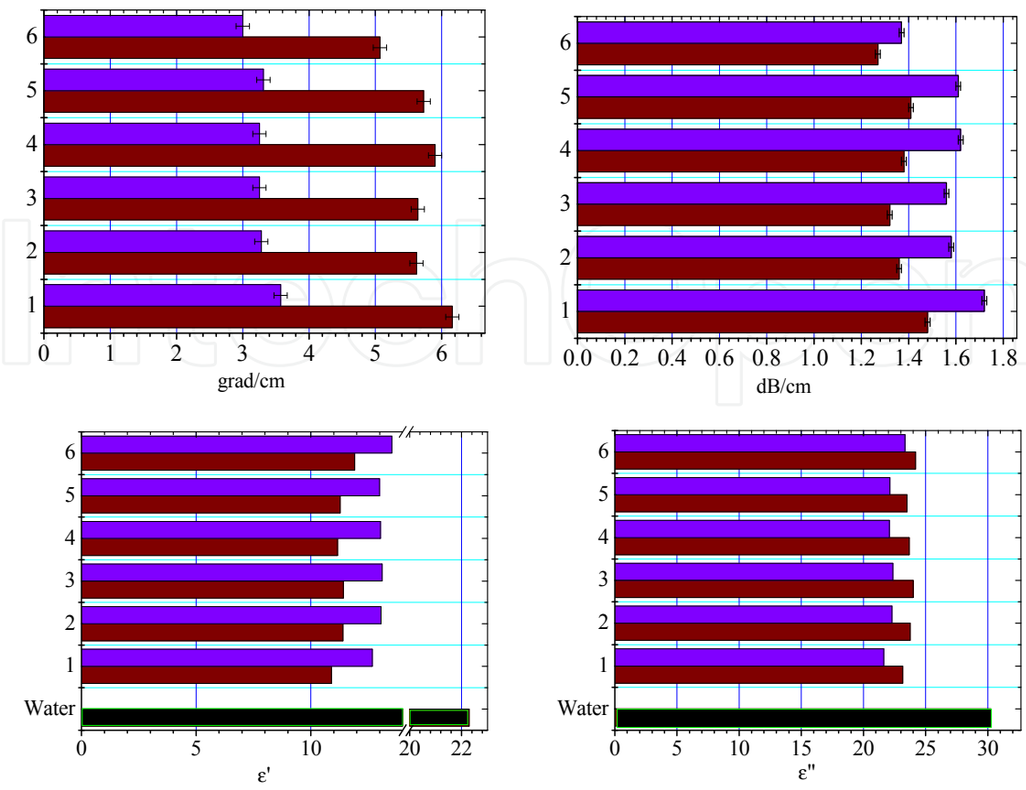


Fig. 5. The increment of the wave phase (upper left) and the attenuation (upper rigth) coefficients in the cell with water and in the cell with table wines (musts) with respect to the distilled water are presented. There are the real (bottom left) and imaginary (bottom rigth) complex permittivity parts of wines and musts samples, respectively. In blue there are data for musts, in brown there are data for wines. The data of grapes vintage are shown on the vertical axis such as 1 - Chardonnay 8 Sept. 07, 2 - Aligote 14 Sept. 07, 3 - Riesling Rhine12 Sept. 07, 4 - Riesling Rhine 19 Sept. 07, 5 - Riesling Rhine 20 Sept. 07, 6 - Rkatsiteli 27 Sept. 07.

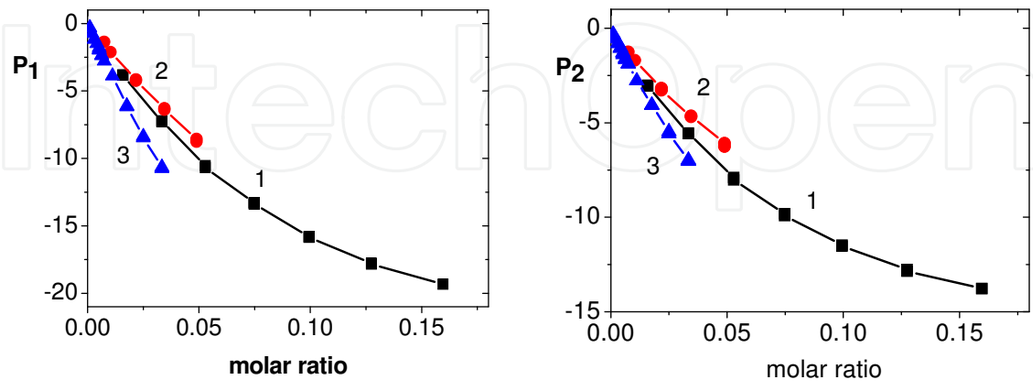


Fig. 6. The differences of the real (left) and imaginary (right) complex permittivity parts of water and water solutions of ethanol, glycerol, and glucose on their concentration in mole ratio. $P_1 = \varepsilon'_x - \varepsilon'_{water}$, $P_2 = \varepsilon''_x - \varepsilon''_{water}$, x is one of components of solutions. The numbers denote 1- ethanol, 2- glycerol, 3 - glucose.

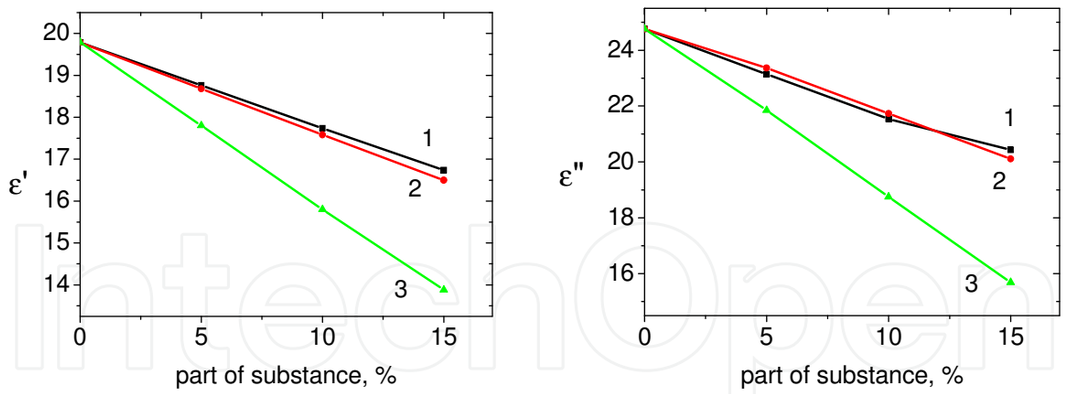


Fig. 7. The influence of wine components on the real (left) and imaginary (right) complex permittivity parts of 10% water - ethanol solutions with additive components: 1 – saccharose, 2 – glycerol, 3 –saccharose and glycerol mixed.

The values of the real and imaginary complex permittivity parts of their water solutions are reduced at the concentration increase of any of three substances. This reduction is approximately linear at small concentrations. Therefore, at mole ratios $r < 0.05$ there is a summation of the contributions of different complex permittivity components of wines and musts (hypothesis of additivity). In Fig.7 there are dependences of complex permittivity of water solutions of saccharose, glycerol, and also their mixture. It validates the hypothesis of additivity. The concentration of quantity of substances is in mass percents.

The water, ethanol, sugars (glucose, saccharose, fructose), and glycerol are chemical components that have the strongest impact on complex permittivity of wines and musts at 8- millimeter wave band in comparison with the other wine components. For instance, in Fig.8 there are dependences of complex permittivity of malic, tartaric, and citric acids diluted with 10% water - ethanol solutions on mass concentration of acids. It presents that the influence of organic acids concentration change on the complex permittivity of wines in several times less than the influence of the mentioned above wine components and these dependences have non-monotonic behavior.

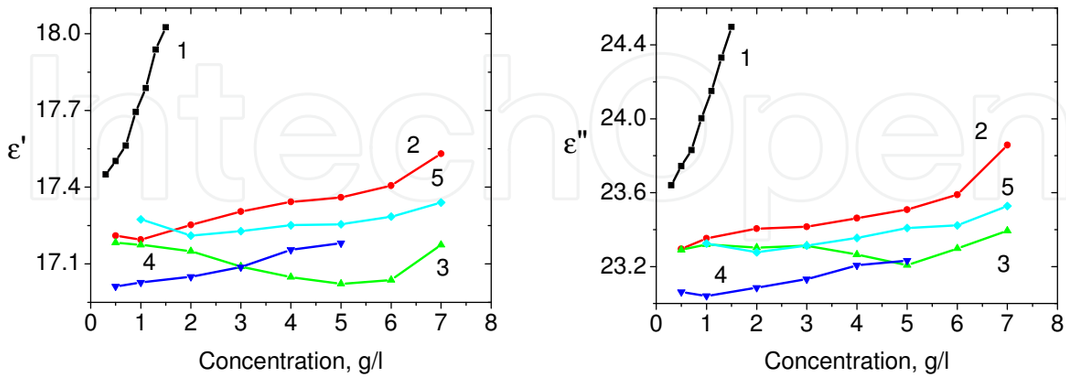


Fig. 8. The dependences of the real (left) and the imaginary (right) complex permittivity parts of organic acids diluted with 10% water - ethanol solutions on mass concentration of organic acids. The numbers denote: 2 – malic acid, 3 – tartaric acid, 4 – citric acid, 5 – tartaric, and malic acids; they are in equal amount. The curve 1 is the dependence of the complex permittivity of potassium diluted with 10% water - ethanol solutions on mass potassium concentration (g/l).

The deviation of cations concentration has enough strong influence on the complex permittivity of wines (the dependence for potassium cations is in Fig.8). However, their absolute quantity in wines and musts is small. Apparently, the influence of cations on the complex permittivity of wines is the reason to have the application possibility of the correlations between complex permittivity and a region of wine-growing.

The results of experimental complex permittivity determination of wines and musts with a different quantity of added water are presented as well. Our objects of research were samples of the natural and diluted with water musts and wines made of the grapes of the following grades: Aligote, Riesling Rhine, Rkatsitely, Cabernet-Sauvignon. It was a crop of 2007-2008. The modeling samples of wines were received by entering water and sugars in the must and squash before the fermentation. Diluted must samples were made by adding the water in the must from 10 % up to 50 %. Diluted wine samples were made by adding the water in natural wine from 5 % up to 30 %.

We defined the following parameters of musts and wines samples: the volume fraction of ethanol, mass concentration of sugars, the total extract, total acidity, viscosity, conductivity, pH, buffer capacity, mass concentration of chlorides, sulfates, potassium, sodium, magnesium, calcium, glycerol, glucose, and saccharose. It was done by the methods accepted in winemaking. Glycerol and separate sugars were defined by high-performance liquid chromatography (HPLC) method on liquid chromatograph Shimadzu LC-20AD. Cation of metals were defined by the method of nuclear absorption on spectrophotometer C115-M1. The viscosimetry, densitometry, titrometry, conductimetry, and pH methods were used for other parameters.

The chemical composition of water added in trial samples, and the monitoring samples of musts and table wines made of grapes growing in a foothill zone of Crimea are presented in Table 1 as well.

Parameters	Riesling Rhine (must)	Aligote (wine)	Cabernet-Sauvignon (wine)	Water
Ethanol, vol. %	-	10.9	13	-
Mass concentration, g/l: Sugars	215	0.81	2.5	-
Total acidity	10.1	8.3	9.8	-
Total extract	239.0	19.0	28.0	-
Phenolic substances	0.524	0.174	1.940	-
Glycerol	0.43	7.4	8.7	-
Chlorides	0.026	0.011	0.03	0.004
Sulfates (K_2SO_4)	0.179	0.289	0.283	0.102
Magnesium	0.136	0.92	0.104	0.007
Calcium	0.140	0.12	0.063	0.104
Potassium	1.200	0.400	0.512	0.001
Sodium	0.066	0.015	0.047	0.008
Buffer capacity, mg-eq/l	64	38	44	9
pH	2.9	2.9	3.1	8.1
Specific conductivity, mS/cm	2.32	1.67	1.95	0.38
Kinematic viscosity, mm^2/s	1.94	1.54	1.73	1.06

Table 1. Physical and chemical parameters of water and natural grape musts and wines.

We performed the analysis of interrelationship between the degree of dilution, chemical composition, and physic-chemical parameters of the diluted by water musts and wines. These results show that the part of bivalent cations grows, and the content of other components decreases with the increasing of the amount of added in wine water.
The change of componential structure in must and wine diluted with water influence on their dielectric properties. Fig. 9 shows the dependence of the complex permittivity on the water added in a must and table wine at temperature 25 °C (31.82 GHz).

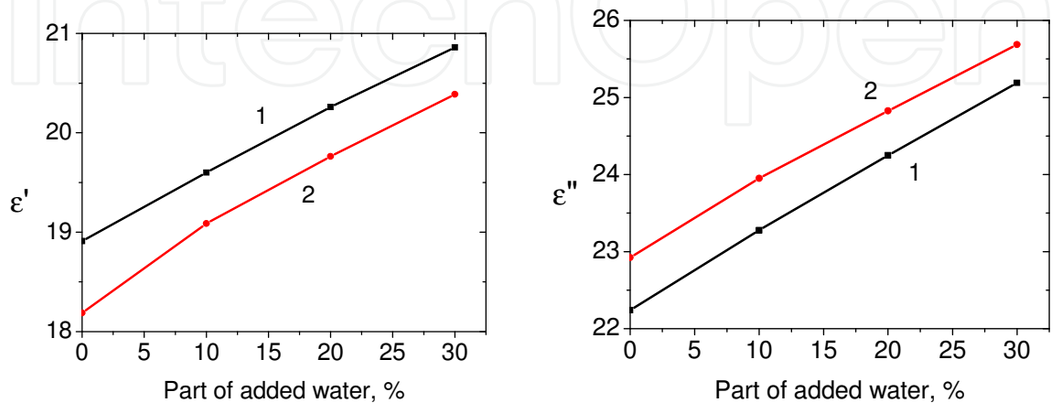


Fig. 9. The complex permittivity of the grape table wine (Rkatsitely) and must (from white Rkatsitely grape) with different degree of water dilution. The real (left) and the imaginary (right) complex permittivity parts. The numbers denote: 1 – must, 2 – wine.

The increasing of the added water part results in the growth of the real and imaginary parts of complex permittivity of wine and must samples under test. The similar effect is observed when the addition of water in must or squash before a fermentation takes place. Close correlation of the complex permittivity with the part of water, added in must and wine indicates the high sensitivity of this parameter to the amount of added water in comparison with traditional parameters of wine composition (Table 2).

Sample	The additive of water in	ε′	ε"	Ethanol	Sugar	Total extract	Total acidity	Phenolic substan-ces
Must	Must	0.999	1.000	-	0.991	0.999	0.997	0.996
Wine	Wine	0.990	0.995	0.996	0.993	0.968	0.983	0.993
Wine	Must (squash)	0.999	0.998	0.990	0.930	0.977	0.918	0.983

Table 2. Correlation coefficient between the complex permittivity and the basic components of must and wine contents with the amount of the added water.

The differential sensibility of dielectometry method is higher than the sensibility of traditional used chemical methods at the determination of the added water (more than 5%.) The small dielectometry analysis duration is also attractive. These facts allows using the difference in complex permittivity of the wine sample under test relative to the complex

permittivity of the control sample of natural wine or must for identification of its authenticity. To solve this problem we started the formation of database of complex permittivity for musts and table wines of Crimea.

5. Conclusion

We presented the results of the design of the simple in operation differential 8 - millimeter wave range dielectrometer for the express analysis of high loss liquids. Our dielectrometer has the measurement cavity with two identical cells filled with the reference liquid and the liquid under test. The measurements are based on the dependence of wave propagation along quarts rod immersed into high loss liquid on its complex permittivity. The complex permittivity measurement is computer-aided and the entire measurement cycle does not exceed 3 minutes. The differential sensibility is 0.05% for the real complex permittivity part of liquid under test and 0.2% – for its imaginary complex permittivity part. In particular, it allows solving the natural table wine and must identification problem i.e., fraud detection by means of added water of the order of 0.1%. We presented complex permittivity measurement data of water and water - ethanol solutions of a number of substances that are wine components and selected those of them which have strong impact on the complex permittivity of liquid under test value. These data can be in use during the development of techniques of dielectrometry usage in wine industry. This device can be used in biochemical laboratories.

Our future research is as follows: The reduction of the measurement errors using thermostat, design the cavity with the cells of different lengths to remove uncertainty due to cell effective length error determination. It is necessary to note that other type of wine and must frauds can be detected using our dielectrometer, not only the wine dilution by water. Our dielectrometer can be used in wine manufacturing process as well.

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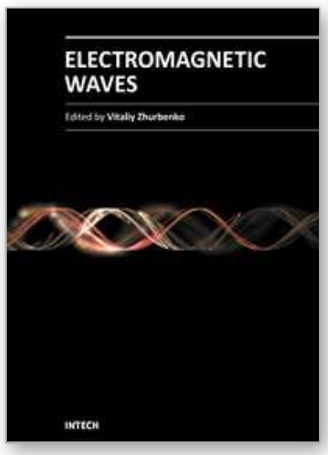
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This book is dedicated to various aspects of electromagnetic wave theory and its applications in science and technology. The covered topics include the fundamental physics of electromagnetic waves, theory of electromagnetic wave propagation and scattering, methods of computational analysis, material characterization, electromagnetic properties of plasma, analysis and applications of periodic structures and waveguide components, and finally, the biological effects and medical applications of electromagnetic fields.

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