

Improving the Accuracy of Dielectric Measurements

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Abstract

In modern dielectric spectrometers, the complete measurement procedure including control of devices and evaluation of the data is automatically performed by computer control. Nevertheless, the quality and the accuracy of the results strongly depend on the proper preparation of the sample. This is especially important if one is interested in absolute values of the dielectric function. This article gives an overview on the most important sources of inaccuracies and some practical recommendations concerning the preparation of samples for accurate dielectric material measurements in combination with Novocontrol Technologies broadband dielectric spectrometers.

1 Introduction

The principle of a dielectric measurement is shown in Figure 1. The sample material is placed between

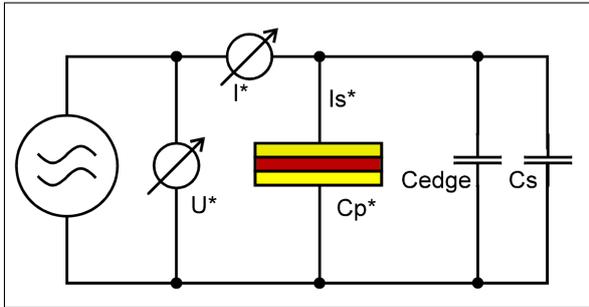


Figure 1: Principle arrangement for a dielectric materials measurement.

two circular metal electrode plates in order to act as a sample capacitor as shown in Figure 2. A generator applies a typically sinusoidal ac voltage $U(\omega)$ of frequency $\omega/2\pi$ to one of the capacitor plates. The sample current $I(\omega)$ is determined at the opposite capacitor electrode by means of a vector current meter that measures not only the current amplitude I_0 but also the phase shift φ of the current signal with respect to the voltage. To simplify calculations and representations of the relations between voltages, currents, and materials properties, it is both

convenient and common to use complex notations:

$$U(\omega) = U_0 \cos(\omega t) = \Re\{U^* \exp(i\omega t)\}. \quad (1)$$

$$I(\omega) = I_0 \cos(\omega t + \varphi) = \Re\{I^* \exp(i\omega t)\}. \quad (2)$$

with

$$U^* = U' + iU'' = U_0 \quad (3)$$

and

$$I^* = I' + iI'' = I_0 \exp(i\varphi) \quad (4)$$

$$I_0 = \sqrt{I'^2 + I''^2} \quad (5)$$

$$\tan(\phi) = \frac{I''}{I'} \quad (6)$$

Since the complex impedance Z^* relates the complex voltage and current and can be expressed by a complex capacity C^* , i.e.,

$$Z^* = \frac{U^*}{I^*} = -\frac{i}{\omega C^*}, \quad (7)$$

we may express the complex measured sample capacity C_m^* as

$$C_m^* = -i \frac{I^*}{\omega U^*} \quad (8)$$

On the other hand, the dielectric function of the sample material is related to the complex capacity of an ideal sample capacitor (see section 2.2.3 for details) by

$$\epsilon^* = \epsilon' - i\epsilon'' = \frac{C_s^*}{C_0} \quad (9)$$

Here, C_0 represents the vacuum capacity of the

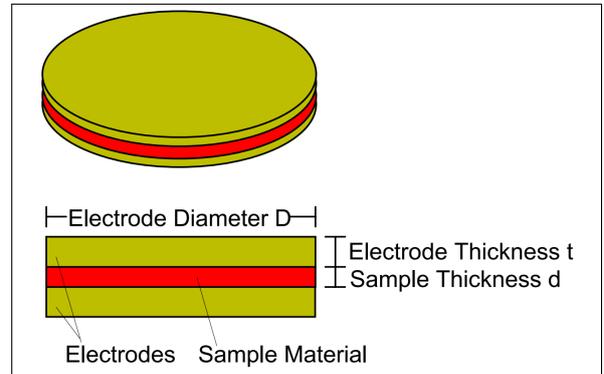


Figure 2: External electrodes with the sample material in "sandwich" arrangement.

empty sample capacitor. In a first approximation,

$$C_s^* \approx C_m^* \quad (10)$$

The accuracy of this method depends on several factors described in the following sections.

2 The Sources of Inaccuracies in Broadband Dielectric Measurements

2.1 Inaccuracies in the Measurement System

The topic of this section is the limits of accuracy of the impedance analyzer itself and the influence of connecting cables and the sample cell. The measurement accuracy is hardly found uniform but strongly depends on the frequency of measurement and the actual sample capacity. Details on measurement accuracy should be specified in the analyzer manual. Most specifications, however, apply only for the measured impedance directly connected to the analyzer input, thereby neglecting the effects of cables and sample cell. For high-quality impedance analyzers, the cables and sample cells may represent the dominant sources of errors, limiting the overall system performance at both the low and high frequency ends of the measurement range.

Results are improved in a system equipped with an active sample cell where the impedance converter is incorporated into an optimized sample cell. Such systems are calibrated for the sample position and thus can be specified for impedance accuracy at the sample position rather than at the analyzer BNC sockets. An example is the Novocontrol Alpha Active Sample Cell (*ZGS* in short) which is specified for the frequency range from 10 MHz to 40 MHz and from -160 °C to 400 °C at the sample position.

2.2 Inaccuracies Resulting from Sample Preparation

Another source of error in the dielectric function evaluation arises from the non-ideal preparation of the sample capacitor. Here we assume that the system accurately measures the complex capacity C_m^* of the sample capacitor, but Eqn. (10) is not fulfilled. From our experience, the following four sources of error are the most important ones:

2.2.1 Inaccuracies in Sample Geometry

These result mostly from uncertainties of the sample thickness. It should be noted that for a sample with a thickness of 0.1 mm, the error in the thickness has to be less than 1 μm for a 1 % accurate result. Moreover, the thickness of most samples will not be

homogeneous within this range. Moreover, pressure applied to the sample when mounted between the electrodes of the sample cell may lower the sample thickness.

2.2.2 Insufficient Electrical Contacts between Sample and Electrodes

Contact problems are frequently due to inhomogeneous sample surfaces. As a consequence, only a part of the sample material will be in contact with the electrodes. In addition, poor contacts may be due to microscopic insulating layers covering either the metal electrodes or the sample material (e.g., oxide layers). Poor contacts, in general, lower the measured capacities C_m^* and, as a consequence, ϵ values and/or introduce additional losses (increased $\tan \delta$).

Suitable contacts are generated by evaporating or sputtering thin metal films (e.g., gold) on both sides of the sample surface. At least the sample should be covered with a layer of silver paint (suspension of colloidal silver). For accurate measurements of absolute values, applying suitable electrodes by metal deposition is mandatory. Nevertheless, even in these cases, the measured results may depend on the metal used for sputtering or evaporation, therefore one should use metals of good conductivity (gold, silver, aluminium). Sample metallization is especially important if materials with large dielectric permittivities are measured. (e.g., a ceramic material of $\epsilon' \approx 1500$ may result in a measured permittivity of only 25 without metallized sample surfaces).

2.2.3 Edge Capacities

In the preparation technique described above, the sample material is directly prepared between two external electrodes. The sample capacitor forms a kind of sandwich structure as shown in Figure 2. After preparation, the complete sandwich is placed between the electrodes of the sample cell. In the standard configuration, we refrain from using so-called guard electrodes (additional ring shaped electrodes intending to reduce the influence of the edge field) thus simplifying the sample preparation and contacting procedures. Disposable electrodes with several diameters can be used for optimal adaptation of the sample material to the impedance measuring system. The usage of disposable electrodes prevents the sample cell electrodes from being contaminated by the sample material. On the other hand, Eqn. (10) will be exact only if the electrical field distribution is homogeneous inside the sample capacitor and zero outside. In practice, there will be a non-homogeneous field contribution at the capacitor edges increasing the sample capacity. In addition, the field outside the capacitor will not be zero, but there will be a stray field, which adds a

contribution to the measured sample capacity as well. Both contributions can be taken into account by an additional capacity contribution, C_{edge} . The measured capacity can thus be written as

$$C_m^* = C_s^* + C_{\text{edge}} \quad (11)$$

where C_m^* is the measured capacity and C_s^* the capacity of the ideal capacitor. The edge capacity could be reduced by using a sample cell with a third so-called guard electrode. Guard-electrode set-ups, however, have the disadvantage of making sample preparation more difficult since no disposable electrodes can be used any longer. In addition, guard electrodes pose additional technical problems at high frequencies. On the other hand, the errors in a non-guarded set-up can be easily kept below other error sources if either a convenient sample cell geometry is chosen and/or the edge capacity is taken into account in the data evaluation. Comparing the benefits of a guarded set-up with its disadvantages, Novocontrol favours the second solution without guarding. C_{edge} can be exactly calculated for two circular capacitor plates centred in an evacuated and grounded metallic sphere with infinite radius (capacitor not filled with sample material) [1].

$$\frac{C_{\text{edge}}}{C_0} = \frac{2d}{\pi D} \left[\ln \frac{8\pi D}{d} - 3 + z(x) \right] \quad (12)$$

$$z(x) = (1+x) \ln(1+x) - x \ln(x) \quad (13)$$

$$x = t/d \quad (14)$$

$$C_0 = \epsilon_0 \frac{\pi D^2}{4d} \quad (15)$$

Here, D , d , and t represent electrode diameter, spacing between electrodes, and electrode thickness, respectively. $\epsilon_0 = 8.854 \cdot 10^{-12}$ As/Vm is the permittivity of free space.

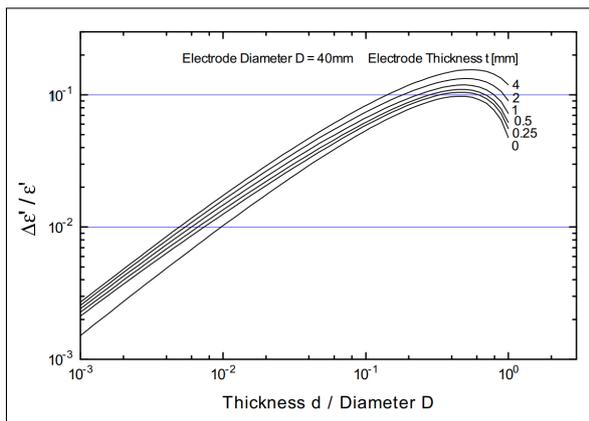


Figure 3: Error due to edge effects (without edge compensation) for an electrode with diameter $D = 40$ mm for several electrode thicknesses t and a dielectric constant $\epsilon' = 3$.

If the volume between the capacitor plates is completely filled with a dielectric material, which does

not overlap the capacitor plates as shown in Fig. 2, the edge capacity will approximately not be changed by the material. In this case the ideal sample capacity C_s^* can be calculated from eqn. (11) and (12). This was verified for several geometries and dielectric materials [2] with the result that the residual errors of this method are $< 1.1\%$ of the measured values for all tested samples. In addition, the ratio C_{edge}/C_0 can be used to estimate the maximum error without edge compensation. The relative error of a measured dielectric function ϵ' due to edge effects (without compensation) is

$$\frac{\Delta\epsilon'}{\epsilon'} = \frac{C_{\text{edge}}}{C_s^*} = \frac{C_{\text{edge}}}{C_0\epsilon'} \quad (16)$$

$\Delta\epsilon'/\epsilon'$ is shown in Fig. 3 as a function of electrode thickness d by diameter D for a material with $\epsilon' = 3$. The error increases with the electrode thickness and decreases with the ratio d/D . Nevertheless, even for $d/D = 1$, the errors remain always below 20%. For $d/D > 0.3$, the error decreases with increasing d/D as the additional edge capacity outside the capacitor is overcompensated by the inhomogenous field distribution inside the sample material which has a negative capacity effect on C_{edge} .

2.3 Electrode Polarisation

For samples having ionic conductivity, mobile ions can migrate within the sample material, hence giving rise to potential differences at the electrode surfaces. This leads to a large increase in both the real and imaginary part of the dielectric function at low frequencies [3, 4, 5, 6].

3 Measurement example

The above techniques were applied to three polyethyleneterephthalate (PET) films with different thickness of 13 μm , 23 μm and 100 μm . The samples were prepared and measured at the University of Leipzig¹ using Novocontrol equipment described in [7, 8]. In order to cover the broad frequency range from 10^{-2} to 10^9 Hz, different measurement systems have to be combined, being based on different measurement principles. At frequencies below 1 MHz, frequency response analysis was applied. At the higher frequencies, coaxial line reflectometry was used. For each method two different samples were prepared. Figure 4 shows the measured complex dielectric function of the three films. The results show the absolute measured values with edge correction applied. The 23 μm and 100 μm films show a nearly perfect match for all four samples over the whole frequency range with a maximum deviation

¹Thanks to H. Groothues, Universität Leipzig, Linnéstr. 5, 04103 Leipzig, Germany

of less than 1%. For the thinnest (13 μm) film, the values of ϵ' and ϵ'' were measured about 6% higher compared to the other two films. Nevertheless, this is well inside the limits of expectation, considering that 6% accuracy corresponds to a uncertainty of the film thickness of 800 nm. The results impressively demonstrate the validity of the measurement technology and systems even for thin films with thickness down to 10 μm .

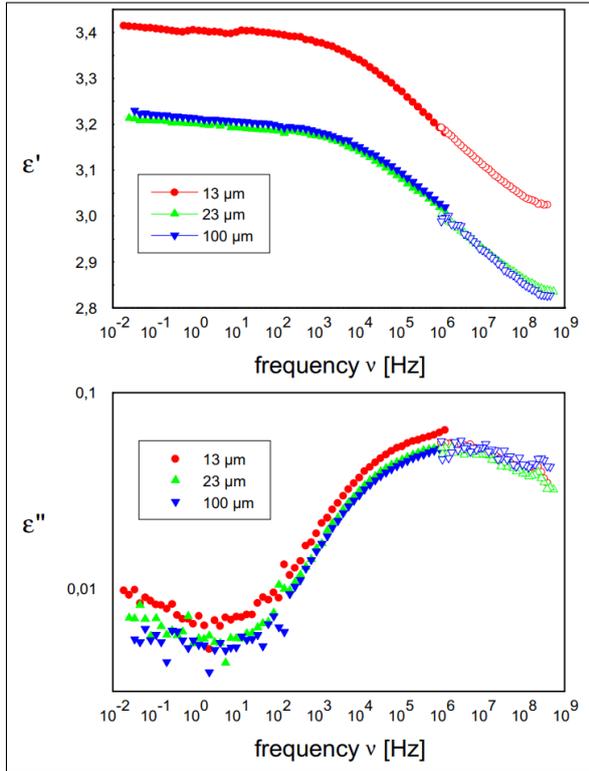


Figure 4: Determination of both real and imaginary parts of the dielectric function of polyethyleneterephthalate (PET). In the frequency range from 10^{-2} Hz to $2 \cdot 10^6$ Hz frequency response analysis was employed, between 10^6 Hz and 10^9 Hz coaxial reflectometry was used. Evidently the results obtained by both measurement system coincide well.

4 Recommendations for Sample Preparation

Both for the Novocontrol low frequency dielectric sample cell BDS 1200 and for the high frequency sample cells BDS 2100 (7 mm diameter) and BDS 2200 (14 mm diameter), the sample material should not be prepared directly between the contacts of the sample cell. Instead, the sample material should be prepared in sandwich structure between additional disposable electrodes as shown in Figure 2. Gold-plated external electrodes are available from Novocontrol Technologies with diameters from 40 mm

to 10 mm for the low frequency systems² (below 10 MHz) and from 12 mm to 3 mm for the RF system (above 1 MHz).

4.1 Recommended Dimensions

For solid samples, the distance between the electrodes is determined by the sample thickness. For optimal measurement results, the sample geometry should be chosen according the following rules:

For the low frequency systems (below 10 MHz), good performance will be reached with sample capacities from 50 pF to 200 pF (optimum around 100 pF) in the frequency range from 100 kHz to 10 MHz. At lower frequencies, higher capacities up to 2 nF will yield good results as well. Note: The system will be able to measure also capacities in a wide range from 0.1 pF up to 1 F depending on frequency, but the accuracy decreases outside the ranges specified above. For the high frequency systems (1 MHz to 3 GHz), a good sample capacity range is from 2 pF to 10 pF (optimum around 4 pF). Smaller capacities yield better results at high frequencies and vice versa. If the dielectric constant ϵ' of the sample is approximately known, the sample capacity can be estimated from

$$C = \epsilon' \epsilon_0 \frac{\pi D^2}{4d} \quad (17)$$

or

$$\frac{C}{\text{pF}} \approx 6.95 \cdot \epsilon' \frac{(D/\text{mm})^2}{d/\mu\text{m}} \quad (18)$$

where D is the diameter of the capacitor electrodes and d the distance between them.

In order to minimize the errors due to edge effects, the ratio d/D should be kept as small as possible. If edge compensation is used, the diameters of both disposable electrodes and the sample material should be equal. Edge compensation is supported the Novocontrol instrument control and evaluation software WinDETA since version 3.5 [9].

4.2 Sample Surface and Contact

4.2.1 Solid Samples

The surface of solid samples should be as flat as possible in order to obtain decent contact with the external electrodes. In addition, it is highly recommended to metallize the sample surface in the contact regions. This can be achieved by evaporation or sputtering a metal film onto opposite sample surfaces. Another easier and cheaper way is to cover the sample surface with a suspension of colloidal silver. Note that this procedure is, however, not suitable for porous materials. The optimum contacting procedure depends both on the particular

²Customized sizes are available as well.

material and its thickness. It is essential to ensure that the bulk sample material to be investigated is not affected by the particular method of electrode preparation.

4.2.2 Liquid Samples

Measurements on liquid samples usually require the use of spacers in order to adjust the electrode spacing, cf. Figure 5. Suitable spacer materials exhibit

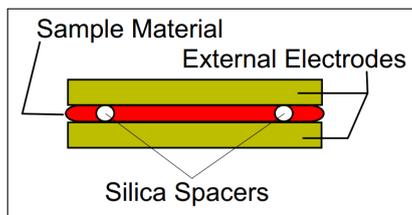


Figure 5: Example liquid sample configuration with spacers (cross sectional view)

low and frequency-independent dielectric constants and low losses. Convenient spacer materials are, e.g., silica fibres (available from Novocontrol) or small stripes of PTFE or Polyethylene. The effect of the spacers on the measurement (stray capacity and sample volume reduction) can be compensated by particular parameters to be entered into the WinDETA software. For some low viscosity liquids with high vapour pressure, sample material may evaporate from the standard sandwich capacitor especially at higher temperatures. In order to prevent evaporation, the sealed liquid sample cell BDS 1308 can be used which is shown in Figure 6. In the open state,

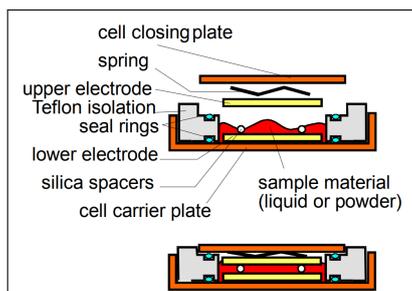


Figure 6: Principle drawing of the Novocontrol liquid sample cell BDS 1308.

the upper electrode is removed from the cell, and the sample material poured out on the bottom of the cell acting simultaneously as the lower electrode. In the closed state, liquid sample material that does not fit between the electrodes can flow around the upper electrode. The two seal rings attached to the teflon isolation prevent evaporation of sample material out of the cell. The BDS 1308 is mounted in the same way as the standard sandwich capacitor between the electrodes of the BDS 1200 or active sample cell ZGS.

References

- [1] G. Kirchhoff. “Zur Theorie des Condensators”. In: *Monatsber. Akad. Wiss.* (1877), pp. 144–163.
- [2] A.H. Scott and H.L. Curtis. “Edge correction in the determination of dielectric constant”. In: *Journal of Research of the National Bureau of Standards* 22.6 (06/1939), p. 747. DOI: 10.6028/jres.022.008.
- [3] H P Schwan. “Electrical properties of tissue and cell suspensions.” In: *Advances in biological and medical physics* 5 (1957), pp. 147–209.
- [4] O.G. Martinsen, S Grimnes, and H P Schwan. “Interface phenomena and dielectric properties of biological tissue”. In: *Encyclopedia of surface and colloid science* 20 (2002), pp. 2643–2653.
- [5] W. Kuang and S. O. Nelson. “Low-frequency Dielectric Properties Of Biological Tissues: A Review With Some New Insights”. In: *Transactions of the ASAE* 41.1 (1998), p. 173.
- [6] G. Blum, H. Maier, F. Sauer, and H. P. Schwan. “Dielectric Relaxation of Colloidal Particle Suspensions at Radio Frequencies Caused by Surface Conductance”. In: *The Journal of Physical Chemistry* 99.2 (01/1995), pp. 780–789. DOI: 10.1021/j100002a048.
- [7] G. Schaumburg. *New broadband dielectric spectrometers*. Dielectrics Newsletter 2. Novocontrol Technologies, 07/1994. URL: <https://www.novocontrol.de/newsletter/DNL02.PDF>.
- [8] G. Schaumburg. *High frequency performance for dielectric measurements to 1.8 GHz improved by the Novocontrol BDS 6000 system*. Dielectrics Newsletter 5. Novocontrol Technologies, 05/1996. URL: <https://www.novocontrol.de/newsletter/DNL05.PDF>.
- [9] G. Schaumburg. *Data evaluation and measurement control software for dielectric research*. Dielectrics Newsletter 3. Novocontrol Technologies, 03/1995. URL: <https://www.novocontrol.de/newsletter/DNL03.PDF>.

