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Differential and double-differential dielectric spectroscopy to measure complex permittivity in transmission lines

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This article presents and compares two differential methods for measuring the complex permittivity of dielectric materials: In the first method, two measuring cells built as coaxial transmission lines of identical cross section and terminations but different lengths are filled with a sample of the dielectric material. The complex dielectric permittivity is determined from the scattering parameter measurements and the length difference between the two cells, neglecting the resistive losses due to the cells. The second method is a double-differential one: Repeating measurements on the same cells empty, no other knowledge or limiting assumption is required. © 2002 American Institute of Physics. [DOI: 10.1063/1.1494870]

I. INTRODUCTION

The knowledge of complex dielectric permittivity of materials is very important in many areas of applications of science and engineering. In particular, in the field of complex fluids the knowledge of the permittivity allows the characterization of the internal microstructures of the system.¹⁻³ A number of techniques are available for determining the complex dielectric permittivity, each technique having its advantages and drawbacks. In the microwave frequencies range, the most widely used ones are free space,⁴ cavity resonators,⁵ wave guides,⁶ open- or short-ended coaxial probes,⁷ and transmission lines.⁸ The transmission line methods are the simplest ones for the electromagnetic characterization of fluids in wideband measurements from low frequencies up to frequencies of several gigahertz. They include both one-port measurements, using open or shorted lines, and two-ports measurements: a sample fills a section of a transmission line whose complex permittivity is obtained from the measurements of the line section scattering parameters and an accurate knowledge of the line geometry.

In waveguide measurements, a differential method has been recently devised in order to measure the complex propagation constant γ in a waveguide, avoiding the need of a detailed knowledge of the guide geometry.⁹ This method is based on the use of two waveguides identical but for the length of a central segment of uniform cross section. In our work, we apply this differential method to wideband measurements on coaxial transmission lines to obtain the complex dielectric permittivity of a sample that fills a section of the lines. Moreover, we propose an improvement introducing a double-differential scheme in order to avoid the simplifying assumption that is often used in neglecting the conductive losses in the metal of the cell (see, e.g., Ref. 6): The complex dielectric permittivity of the sample is obtained by comparison of the propagation constant γ as measured with the method described in Ref. 9 on a couple of cells of different length filled with the sample and the value γ_0 obtained with the same cells empty. The great advantage of this tech-

nique is that there is no need for an accurate knowledge of the geometry of the cells; even the knowledge of the length difference between the two cells is not required. The only requirements for the method to be used are: (i) a very regular cross section in the central segment of the coaxial line cells and (ii) relative magnetic permeability of the sample under study $\mu_r = 1$.

II. DESCRIPTION OF METHOD

A. Determination of the propagation constant γ

Following the differential method described in Ref. 9 to measure the propagation constant γ of a waveguide from the scattering parameter measurements, we have used two cells built as coaxial transmission lines, with identical characteristic impedance, but different lengths, h_s and h_ℓ . We define four 2×2 cascade scattering matrices that describe the several segments of the cells:¹⁰ L and R account for the mismatching and the losses at the connection between a network analyzer and the cell's terminations and H_s and H_ℓ describe the central segment of the short and long cell, respectively (Fig. 1). Central segments have a regular and uniform cross section that ensures a reflectionless propagation; hence, matrices H_s and H_ℓ can be written as

$$H_s = \begin{pmatrix} e^{-\gamma h_s} & 0 \\ 0 & e^{\gamma h_s} \end{pmatrix}, \quad H_\ell = \begin{pmatrix} e^{-\gamma h_\ell} & 0 \\ 0 & e^{\gamma h_\ell} \end{pmatrix}. \quad (1)$$

By this way, h_s and h_ℓ are not uniquely defined, but their difference $h_\ell - h_s$ is invariant with respect to the position of the ideal separation planes a and b between the homogeneous central segment and the left and right regions of discontinuity. In other words, we assume that L and R are the same for the two cells, as terminations are machined identically to a great accuracy.

The cascade matrix F_i of a complete transmission line, according to the definition of cascade matrix, is

$$F_i = LH_iR, \quad (2)$$

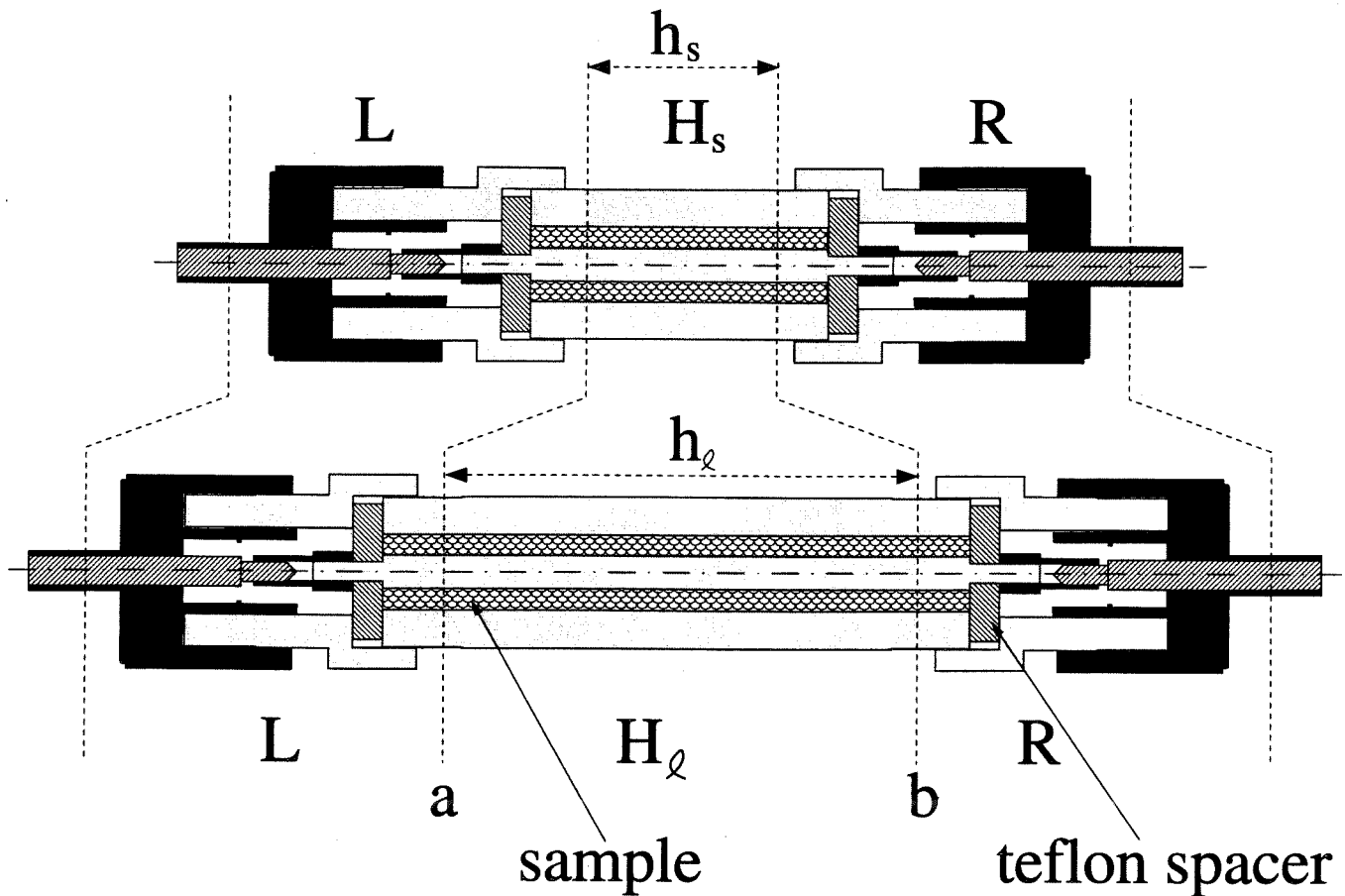


FIG. 1. Schematization of the two coaxial cells. Segments L and R account for mismatching and losses at the connection between a network analyzer and the cell's terminations; they are identical in the short and long cell and are described by the cascade matrices L and R . Segments H_s and H_ℓ are, respectively, the central part of the short and long cell; they have identical cross sections and different lengths and are described by cascade matrices H_s and H_ℓ . Ideal planes a and b , that separate the segments, lay inside the central homogeneous sections but their position is otherwise arbitrary; difference $h_\ell - h_s$ is invariant with respect to the planes position.

where i is, respectively, s for the short cell and ℓ for the long one. Matrices F_i can be expressed in terms of the measured scattering parameters S^i :¹⁰

$$F_i = \frac{1}{S_{21}^i} \begin{pmatrix} S_{12}^i S_{21}^i - S_{11}^i S_{22}^i & S_{11}^i \\ -S_{22}^i & 1 \end{pmatrix}. \tag{3}$$

Combining the expressions of F_s and F_ℓ we obtain

$$F_s F_\ell^{-1} = L \Delta L^{-1}, \tag{4}$$

$$F_\ell F_s^{-1} = L \Delta^{-1} L^{-1}, \tag{5}$$

$$F_s F_\ell^{-1} + F_\ell F_s^{-1} = L (\Delta + \Delta^{-1}) L^{-1}, \tag{6}$$

where

$$\Delta = H_s H_\ell^{-1} = \begin{pmatrix} e^{\gamma \delta} & 0 \\ 0 & e^{-\gamma \delta} \end{pmatrix}, \tag{7}$$

$$\Delta^{-1} = H_\ell H_s^{-1} = \begin{pmatrix} e^{-\gamma \delta} & 0 \\ 0 & e^{\gamma \delta} \end{pmatrix}, \tag{8}$$

and

$$\delta = h_\ell - h_s. \tag{9}$$

As matrices $F_s F_\ell^{-1} + F_\ell F_s^{-1}$ and $\Delta + \Delta^{-1}$ are similar, they share the same trace t^{11} :

$$t = \text{Tr}[F_s F_\ell^{-1} + F_\ell F_s^{-1}] = \text{Tr}[\Delta + \Delta^{-1}] = 2(e^{\gamma \delta} + e^{-\gamma \delta}). \tag{10}$$

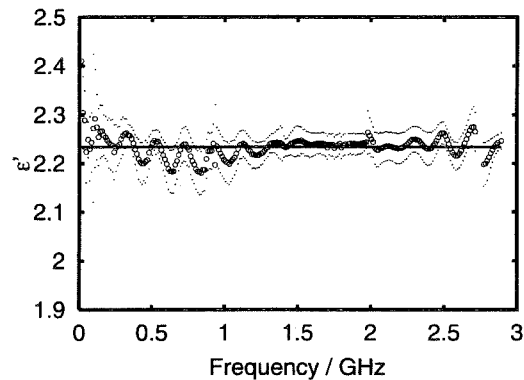


FIG. 2. Real part of dielectric permittivity as a function of frequency as obtained with the (simple) differential method. Points are the experimental values; dotted lines are mean-square deviations, and the solid straight line is the reference value from NIST.

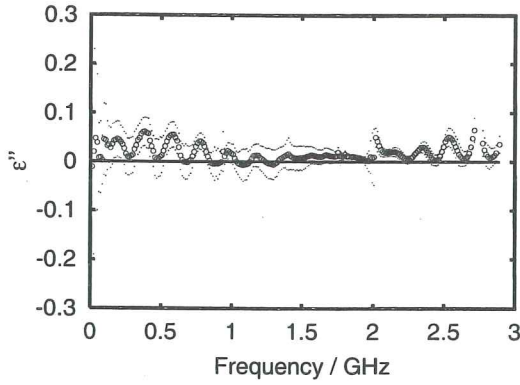


FIG. 3. Imaginary part of dielectric permittivity as a function of frequency as obtained with the (simple) differential method. Points are the experimental values; dotted lines are mean-square deviations, and the straight solid line is the reference value from NIST.

The propagation constant is obtained from relationship (9) as

$$\gamma\delta = \ln \left[\frac{1}{2} \left(\frac{t}{2} \pm \sqrt{\left(\frac{t}{2} \right)^2 - 4} \right) \right]. \quad (10)$$

B. Determination of complex dielectric permittivity

The expression of γ as a function of the physical properties of a transmission line cell is

$$\gamma = \sqrt{(r + j\omega l)(g + j\omega c)}, \quad (11)$$

where r , l , g , and c are the resistance, inductance, conductance, and capacitance per unit length of the cell and ω is the angular frequency.

Assuming $\mu_r = 1$, as usual with most complex fluids of interest, the constants r and l depend only on the cell material and do not change between an empty and a filled cell.

When the cell is filled with a dielectric material only g and c are affected by the material properties

$$g = \frac{1}{K}(\sigma + \omega\epsilon_0\epsilon''), \quad (12)$$

$$c = \frac{1}{K}\epsilon_0\epsilon'. \quad (13)$$

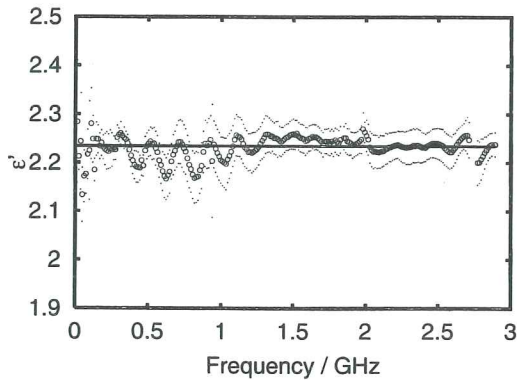


FIG. 4. Real part of dielectric permittivity as a function of frequency as obtained with the double-differential method. Points are the experimental values; dotted lines are mean-square deviations, and the straight solid line is the reference value from NIST.

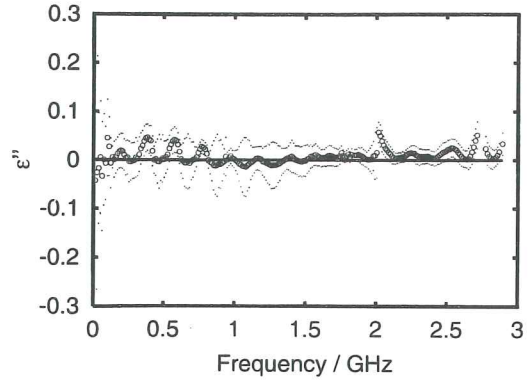


FIG. 5. Imaginary part of dielectric permittivity as a function of frequency as obtained with the double-differential method. Points are the experimental values; dotted lines are mean-square deviations, and the straight solid line is the reference value from NIST.

In these expressions

$$\epsilon' - j\epsilon'' = \epsilon \quad (14)$$

is the complex dielectric permittivity of the material, σ is its dc ohmic conductivity, and K is a cell geometric constant.

1. Differential method

We make two assumptions: (i) we can neglect r in Eq. (11) and (ii) we know δ , the difference in length of the two cells.

We obtain $\gamma\delta$ from Eq. (10); then from Eqs. (11)–(13), and considering that $l = K\mu_0$ [where K is the same geometric constant as in Eqs. (12) and (13)]:

$$\epsilon' - j \left(\epsilon'' + \frac{\sigma}{\omega\epsilon_0} \right) = -\gamma^2 \frac{\epsilon_0\mu_0}{\omega^2}.$$

2. Double-differential method

The whole procedure that leads to $\gamma\delta$ as described above is repeated twice, with both cells empty and filled with the sample, obtaining, respectively, $\gamma_0\delta$ and $\gamma\delta$. Then, considering that $r + j\omega l$ has the same value in the two cases, we obtain

$$\left(\frac{\gamma\delta}{\gamma_0\delta} \right)^2 = \left(\frac{\gamma}{\gamma_0} \right)^2 = \epsilon' - j \left(\epsilon'' + \frac{\sigma}{\omega\epsilon_0} \right). \quad (15)$$

It is worth noting that δ in Eq. (10) elides in Eq. (15) so that its value is no more needed in the computations.

TABLE I. Mean-square deviation and mean deviation of the experimental points from the value indicated by NIST. v_i is the experimental value at the i th frequency, v is the value indicated by NIST, and N is the number of experimental points.

Measured item	Method	$\sqrt{\frac{\sum_i (v_i - v)^2}{N-1}}$	$\frac{\sum_i (v_i - v)}{N}$
ϵ'	Simple differential	2.3×10^{-2}	
	Double differential	2.3×10^{-2}	
ϵ''	Simple differential	2.4×10^{-2}	1.8×10^{-2}
	Double differential	1.6×10^{-2}	7.5×10^{-3}

III. EXPERIMENT

Measurements have been performed in the range 100 kHz to 3 GHz using an Anritsu MS4661A Network Analyzer.

The cells were coaxial transmission lines machined from ISO316 steel and shaped as female type-*N* connectors at both ends. The external and internal diameters of the coaxial cavity were 7.70 ± 0.05 and 3.35 ± 0.05 mm, respectively. Two Teflon spacers placed at the ends of the cell kept the internal conductor centered. These spacers also made the cell watertight as required when working with fluids.

Cells length were 30 and 80 mm (± 0.05 mm).

Two coaxial cables Sucoflex 104 with type-*N* male terminations at both ends were used to connect the cells to the network analyzer.

Carbon tetrachloride Chromasolv grade (form Riedel-de Haen, Germany) has been used as test liquid.

All measurements have been performed at temperature $T = 22 \pm 2$ °C.

IV. RESULTS AND DISCUSSIONS

The two techniques described in Sec. II have been tested measuring the dielectric permittivity of a standard liquid, namely, carbon tetrachloride, and comparing the results with values from National Institute of Standards and Technology (NIST) (formerly National Bureau of Standards).¹² Measurements have been repeated six times; each time the measuring cell has been completely disassembled, cleaned, and refilled with fresh liquid. Values for ϵ' and ϵ'' from the six measurements have been averaged and plotted: in Figs. 2 and 3, the results of the simple differential method are reported, whereas in Figs. 4 and 5, the results of the double-differential method are plotted. The error band delimited by the dotted lines represents the standard deviation of the six measurements for each frequency. The experimental error due to the measuring instrument accuracy resulted in being much smaller than the spread due to the cell filling procedure and has been neglected. Analogously, errors due to temperature fluctuations, evaluated on the basis of the temperature coefficient given by NIST, resulted negligible.

In Table I, third column, the mean-square deviation of the experimental points from the value indicated by NIST is reported. The values obtained for ϵ' with the two methods are comparable whereas the value obtained for ϵ'' with the double-differential method is significantly better with respect to the value obtained with the simple differential method. This is due to the fact that neglecting r in the simple differential method we obtain an overestimated value for ϵ'' . This is in agreement with the fact that ϵ'' accounts for the losses in the dielectric, whereas the ϵ' value is not significantly affected by this approximation. With the simple differential method, the average deviation of experimental points from the reference value is not significant with respect to the data spread for ϵ' , while for ϵ'' a significant systematic error appears (column 4 in Table I).

In conclusion, the results obtained with the two methods are equivalent as long as the real part of the dielectric permittivity is concerned; when dealing with the imaginary part, instead, the simple differential method is affected by a systematic error that can be significantly reduced adopting the double differential procedure.

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