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Microwave method for reference-plane-invariant and thickness-independent permittivity determination of liquid materials

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An attractive transmission-reflection method based on reference-plane invariant and thickness-independent expressions has been proposed for accurate and unique retrieval of complex permittivity of dielectric liquid samples. The method uses both branch-index-independent expressions and a restricted solution set for determining unique and fast complex permittivities. A 2D graphical method has been applied to demonstrate the operation and validation of the proposed method. A uncertainty analysis has been performed to monitor how the accuracy of the proposed method can be improved by a correct selection of sample holder properties. Scattering parameter measurements of two tested reference liquids (distilled water and methanol) have been carried out for comparison of various techniques with the proposed one when the reference-planes and sample thickness are not precisely known. We note from the comparison that whereas other techniques are seriously affected by imprecise knowledge of both reference-planes and sample thickness, the proposed method removes this restriction. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4862047>]

I. INTRODUCTION

Knowledge of electromagnetic properties (relative complex permeability, ϵ_r , relative complex permeability, μ_r , magneto-electric coupling coefficient, chiral coefficient, etc.) of materials is an essential input for modeling and analyzing wave matter interaction of complex structures composing many different substances. In addition, chemical, mechanical, and biological properties of materials can be analyzed by correlating these properties with their electromagnetic properties (indirect characterization). This type of characterization is especially an important issue in production, processing, and management applications in many areas including agriculture, food engineering, medical treatments, and so on.^{1,2} Besides, the introduction of new type of materials exhibiting negative refractive index (named metamaterial) and their exotic applications in various applications (e.g., negative refractive index, super lens^{3,4}) have attracted a huge interest in materials characterization.

Materials have different responses with frequency to a stimulus of either electric origin or magnetic origin, or both, depending on the type of the material. In this respect, as different from resonant methods,^{5,6} to fully characterize electromagnetic properties of materials, their properties should have been measured over a broad frequency range. To meet this demand, many different broadband (non-resonant) techniques each with various advantages and disadvantages have been proposed in the literature (our interest in this study is characterization of materials at microwave frequencies). The most notable ones among these non-resonant techniques are open-ended (coaxial or waveguide) probes,^{7,8} parallel-plate

waveguide,⁹ free-space measurement,^{10,11} and transmission-line techniques (microstrip, waveguide, coaxial, etc.).^{12–14} Aside from the advantages of these techniques, they have some definite drawbacks of which our main interest is their elimination.

First, measured transmission scattering (S –) parameters produce multiple solutions for extraction of ϵ_r and/or μ_r of thick materials as well as for dispersive materials due to the periodicity of complex propagation factor (T) (This is our first interest in this paper). A variety of techniques have been devised for determining the correct and unique solution for constitutive parameters of materials.^{15–27} Whereas some of them necessitate two samples of different lengths, thin samples, or additional measurements,^{15,16} others require measurements over a broad frequency range or assume that the sample be non-dispersive (weakly dispersive).^{17–27}

The second problem in broadband non-resonant methods is the appearance of undesired ripples in the extracted constitutive parameters. These ripples arise from the decreased accuracy in the measured reflection S -parameters (S_{11} and S_{22}) when the material length is an integer multiple of one-half guided wavelength.^{14,28,29} The following powerful techniques have been proposed to remove this problem for magnetic and nonmagnetic materials.^{14,28,30–33} While some of them require a good initial guess,^{14,28} others are applicable only to non-dispersive materials,^{30,31} and yet others can be applicable to both ϵ_r and μ_r extraction.^{32,33}

The third problem, which is of our second interest in this paper, at high-frequencies in broadband measurements is the increased uncertainty in reference-plane positions of the sample in its cell. The following methods can be utilized as a

remedy.^{34–37} The method in Ref. 34 can only be applied for high-loss dielectric materials. The methods in Refs. 35 and 36 are applicable to both low-loss and high-loss materials as well as thinner and thicker samples and magnetic or dielectric materials. Nonetheless, they are restricted to solid materials. To extend these methods for ϵ_r and μ_r measurement of liquid materials, we have recently proposed another technique.³⁷ Although this technique measures ϵ_r and μ_r of liquid materials, it has two shortcomings. First, it requires that the electromagnetic properties and lengths of the plugs used for holding the liquid sample in place within the measurement cell be identical.^{38,39} In real measurements, such a restricting assumption would produce undesired measurement errors if not considered. Second, there might be present some air region between the liquid sample and the plugs in the measurement cell, which could also affect the measurement accuracy especially at higher frequencies. To eliminate these problems, a measurement cell composed of only one plug positioned vertically could be utilized.^{32,40,41}

As a final shortcoming of broadband non-resonant methods, which is of our third concern in this paper, the sample thickness is assumed to be precisely known.^{12–16,20–40} Spectroscopic ellipsometry,⁴² wavelength scanning,^{43–45} spectral reflectometry/interferometry,^{46–50} and full spectra fitting techniques^{51–53} can be utilized for determining the sample thickness. Ellipsometry technique may not be so accurate if angle arrangement is not so precise. Wavelength scanning technique needs a wide frequency range to determine sample thickness in addition to ϵ_r and/or μ_r . Besides, spectral reflectometry/interferometry method is based on extremes and/or envelopes of reflectance and/or transmittance and can be applied only for non-dispersive materials. Finally, full spectra technique requires an information about the behavior ϵ_r and μ_r to model their wavelength/frequency dependence. While spectroscopic ellipsometry, spectral reflectometry/interferometry, and wavelength scanning methods necessitate a broad frequency band for thickness measurements, full spectra fitting technique requires some knowledge on behavior of material dispersive character.

As discussed above for the first, third, and last drawbacks of broadband non-resonant measurements, to our best knowledge, there is no measurement technique that simultaneously eliminates the information of reference-plane and sample thickness without resorting to a correct branch index value to determine ϵ_r of liquid materials. Our motivation in this study is to propose a measurement method for ϵ_r measurements of liquid materials using a vertically positioned waveguide sample holder based on its aforementioned advantages. Such an analysis (electromagnetic characterization and some related properties of liquid materials) is an important field because liquid samples can be used as phantom materials (tissue-equivalent materials) due to the fact that their electromagnetic properties match to those of biological tissues.⁵⁴ In addition, a number of medical treatment techniques in medicine based on radio frequency and microwave radiation such as diathermy and hyperthermia, fast fixation, denaturation, and sterilization, would get benefit from reliable knowledge of electromagnetic properties of biological tissues involved.^{55–57} Finally, the correct information about

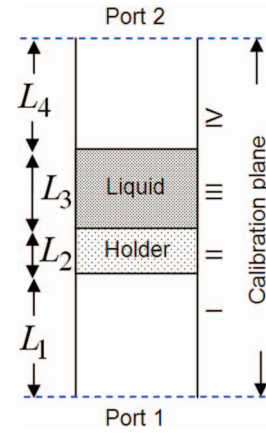


FIG. 1. A typical configuration for complex permittivity measurement of liquid sample within a vertically oriented waveguide cell.

electromagnetic properties of liquids is needed for determination of dissipated power from microwave irradiation through measurements of specific absorption rate.⁵⁸

II. THE METHOD

The problem for ϵ_r measurement of liquid samples using a vertical measurement cell is demonstrated in Fig. 1. The cell has a sample holder with length L_2 , a liquid sample whose length L_3 and ϵ_{r3} are to be determined, and two air regions with lengths L_1 and L_4 . Although the configuration of the problem in Fig. 1 is similar those in Refs. 32 and 41, the proposed technique in this study is different from those in Refs. 32 and 41 in that the presented study determines reference-plane invariant (RPI) ϵ_r without resorting to the correct branch index value and thickness information of liquid sample. This is accomplished by using RPI expressions containing no thickness information.

A. Forward and backward S-parameters

To derive forward and backward reflection and transmission S -parameters (S_{11} , S_{21} , S_{12} , and S_{22}), we apply the wave matrix technique.^{59–64} Based on this technique, assuming that only the dominant mode exists within the rectangular waveguide, S_{11} , S_{21} , S_{12} , and S_{22} parameters at calibration plane are derived in matrix form as

$$\begin{bmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{bmatrix} = \frac{1}{M_{22}} \begin{bmatrix} M_{12} & (M_{11}M_{22} - M_{12}M_{21}) \\ 1 & -M_{21} \end{bmatrix}, \quad (1)$$

where

$$M_t = \begin{bmatrix} M_{11} & M_{12} \\ M_{21} & M_{22} \end{bmatrix} = \prod_{k=1}^N \begin{bmatrix} m_{11}^k & m_{12}^k \\ m_{21}^k & m_{22}^k \end{bmatrix}, \quad (2)$$

$$m_{11}^k = \frac{T_k}{\Lambda_k}, \quad m_{12}^k = \frac{1}{T_k \Lambda_k} \Gamma_k, \quad (3)$$

$$m_{21}^k = \frac{T_k}{\Lambda_k} \Gamma_k, \quad m_{22}^k = \frac{1}{T_k \Lambda_k}. \quad (4)$$

Here, N is the number of total layers between calibration planes ($N = 4$), and the explicit expressions of the wave matrix M_i elements (m_{11}^k , m_{12}^k , m_{21}^k , and m_{22}^k) are

$$\Lambda_k = \sqrt{1 - \Gamma_k^2}, \quad T_k = \exp(-i2\pi\chi_k L_k/\lambda), \quad (5a)$$

$$\Gamma_k = \frac{\chi_{(k-1)} - \chi_k}{\chi_{(k-1)} + \chi_k}, \quad \chi_k = \sqrt{\varepsilon_{rk} - \lambda^2/\lambda_c^2}. \quad (5b)$$

In Eqs. (5a) and (5b), it is assumed that $\varepsilon_{r0} = \varepsilon_{r1}$, all layers between calibration planes are dielectric, and the time reference $\exp(+i\omega t)$ is applied. Here, $\varepsilon_{rk} = \varepsilon'_{rk} - i\varepsilon''_{rk}$ is the relative complex permittivity of layer k ; and λ and λ_c are the operating and cut-off wavelengths.

Performing the product in Eq. (2) and after some manipulations, we derive

$$S_{11} = T_1^2 \left(\frac{\xi_1 - \xi_2 T_3^2}{\xi_6 - \xi_7 T_3^2} \right), \quad S_{22} = T_4^2 \left(\frac{\xi_3 - \xi_4 T_3^2}{\xi_6 - \xi_7 T_3^2} \right), \quad (6)$$

$$S_{21} = S_{12} = T_1 T_4 \left(\frac{\xi_5 T_3}{\xi_6 - \xi_7 T_3^2} \right), \quad (7)$$

where

$$\xi_1 = (1 + \Gamma_2 \Gamma_3)(\Gamma_2 + \Gamma_3 T_2^2), \quad (8a)$$

$$\xi_2 = (\Gamma_2 + \Gamma_3)(\Gamma_2 \Gamma_3 + T_2^2), \quad (8b)$$

$$\xi_3 = (\Gamma_2 + \Gamma_3)(1 + \Gamma_2 \Gamma_3 T_2^2), \quad (8c)$$

$$\xi_4 = (1 + \Gamma_2 \Gamma_3)(\Gamma_3 + \Gamma_2 T_2^2), \quad (8d)$$

$$\xi_5 = (1 - \Gamma_2^2)(1 - \Gamma_3^2)T_2, \quad (8e)$$

$$\xi_6 = (1 + \Gamma_2 \Gamma_3)(1 + \Gamma_2 \Gamma_3 T_2^2), \quad (8f)$$

$$\xi_7 = (\Gamma_2 + \Gamma_3)(\Gamma_3 + \Gamma_2 T_2^2). \quad (8g)$$

Comparing Eqs. (6)–(8g) for $T_1 = T_4 = 1$ with Eqs. (9)–(11) in Ref. 41, we note that they are identical. Assuming that electromagnetic and physical properties of the sample holder are known (Γ_2 and T_2 are both known), we note from Eq. (6) through Eqs. (8a)–(8g) that ε_{r3} of the liquid sample depends on its thickness and reference-plane transformation factors (T_1 and T_4). Our aim in this study is to determine ε_{r3} when T_1 , T_4 , and L_3 are all not known.

B. Reference-plane invariant and thickness-independent expressions

In Subsection II A, we presented the reference-plane and thickness dependent expressions for ε_{r3} measurement of liquid samples. Now, in this section, our aim is to determine ε_{r3}

from Eqs. (6) to (8g) when T_1 , T_4 , and L_3 are not known. Toward this end, we first obtain the following expression involving L_3 as only the unknown parameter^{14,35–37}

$$A = \frac{S_{11} S_{22}}{S_{21} S_{12}} = \frac{1}{1 - \frac{M_{11} M_{22}}{M_{21} M_{12}}} = \frac{(\xi_1 - \xi_2 T_3^2)(\xi_3 - \xi_4 T_3^2)}{\xi_5^2 T_3^2}. \quad (9)$$

From Eq. (9), we can extract T_3 as

$$T_3^2 = \frac{\xi_8 \mp \sqrt{\xi_8^2 - 4\xi_1 \xi_2 \xi_3 \xi_4}}{2\xi_2 \xi_4}, \quad (10)$$

where $\xi_8 = \xi_1 \xi_4 + \xi_2 \xi_3 + A \xi_5^2$. The correct sign for T_3^2 for passive materials can be ascertained by enforcing the passivity condition $|T_3^2| \leq 1$.

Substituting correct T_3^2 into S_{11} and S_{22} in Eq. (6), and then taking their magnitudes, we obtain two expressions with only $\Gamma_3 = \Gamma'_3 + i\Gamma''_3$ (and hence ε_{r3}) as the unknown

$$|S_{11}(\Gamma_3)| = \left| \frac{\xi_1 - \xi_2 T_3^2}{\xi_6 - \xi_7 T_3^2} \right|, \quad |S_{22}(\Gamma_3)| = \left| \frac{\xi_3 - \xi_4 T_3^2}{\xi_6 - \xi_7 T_3^2} \right|. \quad (11)$$

Real and imaginary parts of Γ_3 (Γ'_3 and Γ''_3) can be determined from simultaneous solution of $|S_{11}|$ and $|S_{22}|$ in Eq. (11) by using any 2D zero-search numerical techniques. Some of these techniques are the Newton's method,⁶⁵ the Muller's method,⁶⁶ the Davidenko's method,⁶⁷ the Cauchy integral method,⁶⁸ genetic algorithms, sequential quadratic programming, and globalized Nelder–Mead methods,⁶⁹ and the graphical method.^{11,30,70,71} In Subsection II C, we will apply the graphical method to demonstrate the possibility of unique Γ'_3 and Γ''_3 determination. After determination of Γ_3 by a suitable technique, ε_{r3} can be uniquely calculated from the expression of Γ_3 in Eq. (5b),

$$\varepsilon_{r3} = (\varepsilon_{r2} - \lambda^2/\lambda_c^2) \left(\frac{1 - \Gamma_3}{1 + \Gamma_3} \right)^2 + \lambda^2/\lambda_c^2. \quad (12)$$

We note that the transformation from ε_{r3} space (ideally infinite) to Γ_3 space (a finite space to be shown later) through Eqs. (9)–(11) allows not only RPI and thickness-independent (TI) determination of Γ_3 but also extraction of Γ_3 without resorting to the correct branch index value.^{15–27} Furthermore, to be discussed in Subsection II C such a transformation restricts the range of possible ε_{r3} solution^{70,71} which is especially an important issue in electromagnetic characterization of new materials whose electromagnetic properties are yet known.

C. Range for possible solutions of complex permittivity

Since all 2D methods^{65–71} require a solution set (or region) for determination of Γ_3 , it is therefore beneficial to investigate the range of this set before starting a numerical analysis. First, considering the fact that the impedance of the waveguide housing the sample (and its holder) is not highly capacitive, while the sample (and its holder) possesses a not-highly inductive property,⁷² the range of Γ_3 can be restricted by using $|\Gamma_3| \leq 1$. Furthermore, from the expression of Γ_3 in

Eq. (5b), we note that

$$\Gamma'_3 + i\Gamma''_3 = \frac{[(c^2 + d^2) - (a^2 + b^2)] + 2i(bc - ad)}{(c + a)^2 + (d + b)^2}, \quad (13)$$

where

$$a - ib = \sqrt{\varepsilon_{r3} - \lambda^2/\lambda_c^2}, \quad c - id = \sqrt{\varepsilon_{r2} - \lambda^2/\lambda_c^2}. \quad (14)$$

It is noted from Eqs. (13) and (14) that, for a low-loss sample holder ($d \rightarrow 0$), Γ''_3 becomes a positive quantity. However, there is no definite physical information about assigning the positiveness or negativeness of Γ'_3 . As a result, for low-loss sample holders, we determine $-1 \leq \Gamma'_3 \leq 1$ and $0 \leq \Gamma''_3 \leq 1$ provided that $|\Gamma_3| \leq 1$.

D. Numerical validation

In this subsection, we will apply the graphical method^{11,30,70,71} which shows, if any, the multiple-solutions problem, to analyze whether we can extract unique solution for Γ_3 . In the application of the graphical method, an initial guess for Γ_3 is substituted into Eqs. (8a)–(8g), (10), and (11) for computation of $|S_{11}|_c$ and $|S_{22}|_c$ where the subscript c denotes computed quantities. These computed values are then compared with measured $|S_{11}|_m$ and $|S_{22}|_m$ where the subscript m designates the measured quantities. Next, if the differences are less than desired tolerances ($\delta_{S_{11}} \ll 1$ and $\delta_{S_{22}} \ll 1$), we save this Γ_3 (yielding a possible correct Γ_3). This whole process is repeated for all possible Γ_3 values within the pre-defined range. The graph of saved Γ_3 values produces the constant value curves (C_{11} and C_{22}). Finally, the intersection of these curves is analyzed for Γ_3 determination of the liquid sample. For example, Fig. 2 illustrates the dependencies of C_{11} and C_{22} for a sample with $\varepsilon_r \cong 62.74 - i30.12$ (representative of the distilled water) and $L_3 = 5$ mm. It is assumed that $\varepsilon_{r2} = 2.04 - i0.005$ (representative of a typical Teflon sample), $L_1 = L_2 = 10$ mm, $L_4 = 12$ mm, $f = 10$ GHz, and $f_c = 6.555$ GHz (X-band waveguide).

From Fig. 2, we note that there is one intersection point (one Γ_3 value) of constant value curves C_{11} and C_{22} , and this corresponds to $\Gamma_3 \cong -0.74 + i0.05$, as can clearly be seen from the inset of Fig. 2. After determination of Γ_3 by using the graphical method, the computed ε_{r3} from Eq. (12) is al-

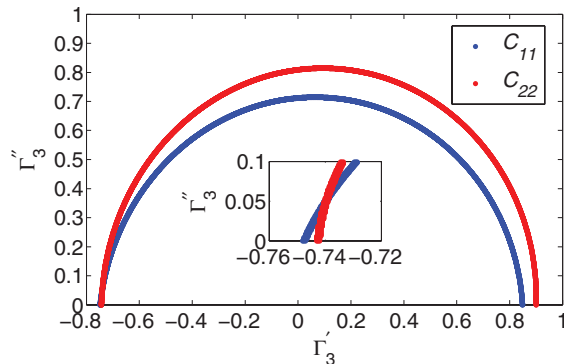


FIG. 2. Constant value curves C_{11} and C_{22} to demonstrate the possibility of the unique solution for the analyzed problem.

most identical to that assigned before analysis ($\varepsilon_r \cong 62.74 - i30.12$). This shows that it is possible to determine unique Γ_3 and thus ε_{r3} by using the presented method. We note that we have used the graphical method for determination of unique Γ_3 . Although this method shows the dependence of analyzed expressions ($|S_{11}|$ and $|S_{22}|$) with respect to independent parameters (Γ'_3 and Γ''_3) over a defined region (whether a unique solution is possible), it is not so fast. For instance, more than 1 min (70 s) has passed to obtain the dependencies of C_{11} and C_{22} in Fig. 2 for iteration steps $\Delta\Gamma'_3 = \Delta\Gamma''_3 = 0.0005$ and for resolutions $\delta_{S_{11}} = \delta_{S_{22}} = 0.005$. Therefore, the graphical method is only suitable for analyzing the possibility of one solution. Having assigned that unique solution becomes possible by the proposed method, any faster 2D numerical methods (discussed above) can be applied to determine ε_{r3} of liquid samples over a broad frequency band.

It might be possible that the analyzed cell in Fig. 1 possesses reflection-symmetric property ($S_{11} = S_{22}$). In such a cases, from Eq. (6) it is noticed that $\xi_1 = \xi_3$ and $\xi_2 = \xi_4$ when $L_2 = L_3$, $\chi_2 = \chi_3$, and $T_2 = T_3$, yielding $\Gamma_3 = 0$. Although practically this situation is rare, our proposed method will not work and cannot be used to determine ε_{r3} . To eliminate this drawback of our method, different sample holders with various properties can be used.

III. UNCERTAINTY ANALYSIS

In Sec. II, we have derived expressions for RPI and TI ε_{r3} of liquid samples without resorting to the correct branch index value. However, we have not considered the effect of some independent parameters (L_2 , L_3 , ε_{r2} , and so on) on the measurement accuracy and on performance of the proposed method. In this section, we perform an uncertainty analysis for analyzing the effect of some parameters on the accuracy level of measurements before starting to experiments. Several factors contribute to the uncertainty in ε_{r3} determination in waveguide measurements,^{14,28,31,35,36,39–41} namely: (1) the uncertainty in measured S-parameters; (2) errors in the sample and holder lengths; (3) the uncertainty in reference-plane positions; (4) guide losses and conductor mismatches; (5) air gaps between the external surface of the sample (holder) and inner walls of waveguides; and (6) presence of spurious higher order modes. Care can be given to limit the uncertainties due to calibration, guide losses, and conductor mismatches by using appropriate calibration techniques and machining the sample holder with no scratches, nicks, or cracks.^{14,73} In addition, the effect of higher order modes can be eliminated by using a longer measurement cell in Fig. 1.^{31,41}

In this study, our purpose is to analyze the effects of inaccurate sample length and holder length on the determined ε_{r3} of liquid materials. We utilize the well-known differential uncertainty model which examines the effect of minute changes in the independent variables on dependent ones. Following the procedure in Ref. 73, we find

$$\frac{\partial \varepsilon_{r3}}{\partial L_3} = \frac{\Omega_1}{\Omega_3}, \quad \frac{\partial \varepsilon_{r3}}{\partial L_2} = \frac{\Omega_2}{\Omega_3}, \quad (15)$$

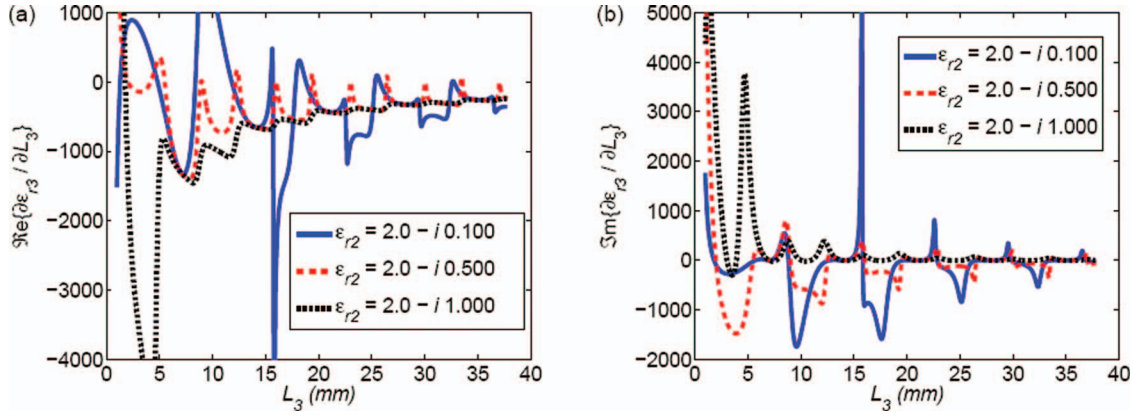


FIG. 3. Dependencies of (a) real part and (b) imaginary part of $\partial\epsilon_3/\partial L_3$ versus L_3 for various ϵ_{r2} values [Assumed parameters are: $\epsilon_{r3} = 5.0 - i0.001$, $L_1 = 8.0$ mm, $L_2 = 7.0$ mm, $L_4 = 6.0$ mm, $f = 10.0$ GHz, and $f_c = 6.555$ GHz].

where

$$\Omega_1 = -\frac{\partial A}{\partial T_3} \frac{\partial T_3}{\partial L_3}, \quad \Omega_2 = -\left(\sum_{u=1}^5 \frac{\partial A}{\partial \xi_u} \frac{\partial \xi_u}{\partial T_2} \right) \frac{\partial T_2}{\partial L_2}, \quad (16a)$$

$$\Omega_3 = \left(\sum_{u=1}^5 \frac{\partial A}{\partial \xi_u} \frac{\partial \xi_u}{\partial \Gamma_3} \right) \frac{\partial \Gamma_3}{\partial \epsilon_{r3}} + \frac{\partial A}{\partial T_3} \frac{\partial T_3}{\partial \epsilon_{r3}}. \quad (16b)$$

The intermediate derivatives in Eqs. (16a) and (16b) can be easily obtained from Eqs. (5a) to (9), and thus they are not given here for brevity. In the analysis, it is assumed that the dependent variables are analytic (or satisfy the Cauchy–Riemann equations in the general sense) over the region of interest with respect to the differentiation parameters.

Using Eqs. (15) and (16a) and (16b), we draw $\partial\epsilon_{r3}/\partial L_3$ over L_3 ($\epsilon_{r3} = 5.0 - i0.001$ and $L_2 = 7.0$ mm), $\partial\epsilon_{r3}/\partial L_2$ over L_2 ($\epsilon_{r2} = 2.0 - i0.100$ and $L_3 = 10.0$ mm), and $\partial\epsilon_{r3}/\partial L_3$ over L_3 ($\epsilon_{r3} = 20.0 - i10.0$ and $L_2 = 7.0$ mm) in Figs. 3–5, respectively, using different ϵ_{r3} and ϵ_{r2} values. For the dependencies in Figs. 3–5, it is assumed that $L_1 = 8.0$ mm, $L_4 = 6.0$ mm, $f = 10.0$ GHz, and $f_c = 6.555$ GHz. We note the following main results from uncertainty analysis. First, $\Re\{\partial\epsilon_{r3}/\partial L_3\}$ and $\Im\{\partial\epsilon_{r3}/\partial L_3\}$ over L_3 (Fig. 3) oscillate for

smaller sample lengths due to increased uncertainty in sample length. Second, dependence of ϵ_{r3} over the length of the sample holder (Fig. 4) reduces for lossy samples ($\epsilon_{r3} = 10.0 - i2.00$) as compared to that of low-loss samples ($\epsilon_{r3} = 10.0 - i0.50$). Third, the oscillatory behavior of $\Re\{\partial\epsilon_{r3}/\partial L_3\}$ and $\Im\{\partial\epsilon_{r3}/\partial L_3\}$ over L_3 of a low-loss sample (Fig. 3) decreases with an increase in the loss of ϵ_{r2} . This means that, for low-loss samples (if the loss character of the sample is known), an uncertainty in sample length can be reduced using a lossy sample holder. Fourth, for a high-loss sample, the dependencies of $\Re\{\partial\epsilon_{r3}/\partial L_3\}$ and $\Im\{\partial\epsilon_{r3}/\partial L_3\}$ over L_3 (Fig. 5) generally do have slightly oscillatory behavior and are not much affected by electromagnetic properties of the sample holder, as compared to the dependencies in Fig. 3. Besides, for high-loss samples and lossy sample plugs, the uncertainty in S_{21} and S_{12} greatly increase, restricting to use low-loss sample holders for ϵ_{r3} measurement of lossy samples.

The dependencies in Figs. 3–5 are representative values and are demonstrated with the intent to give a general behavior of $\partial\epsilon_{r3}/\partial L_3$ and $\partial\epsilon_{r3}/\partial L_2$. It is surely certain that these dependencies are subject to change in complex manner for different electromagnetic properties of sample and its holder. For samples whose electromagnetic properties are yet unknown, as a prescription, it is recommended in measurements

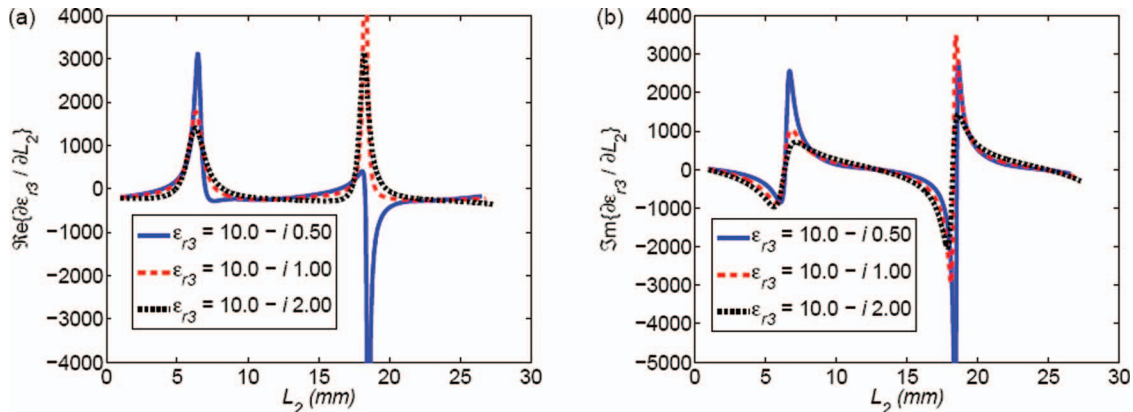


FIG. 4. Dependencies of (a) real part and (b) imaginary part of $\partial\epsilon_3/\partial L_2$ versus L_2 for various ϵ_{r3} values [Assumed parameters are: $\epsilon_{r2} = 2.0 - i0.100$, $L_1 = 8.0$ mm, $L_3 = 10.0$ mm, $L_4 = 6.0$ mm, $f = 10.0$ GHz, and $f_c = 6.555$ GHz].

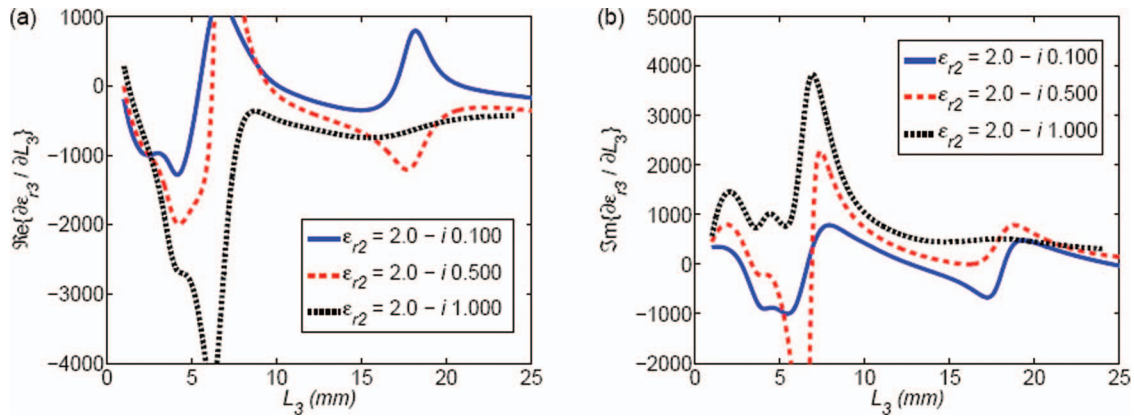


FIG. 5. Dependencies of (a) real part and (b) imaginary part of $\partial\epsilon_{r3}/\partial L_3$ versus L_3 for various ϵ_{r2} values [Assumed parameters are: $\epsilon_{r3} = 20.0 - i10.0$, $L_1 = 8.0$ mm, $L_2 = 7.0$ mm, $L_4 = 6.0$ mm, $f = 10.0$ GHz, and $f_c = 6.555$ GHz].

to utilize few sample holders with different electromagnetic properties and then measure ϵ_{r3} . Such an approach will first allow determination of the loss character of the sample and then its accurate ϵ_{r3} measurement provided that appropriate sample holder is used via the uncertainty analysis in this section.

IV. MEASUREMENTS

In this section, our aim is to validate the proposed method by carrying out S-parameter measurements of some reference liquid materials. Distilled water and methanol are selected for validation since they are reference samples in many measurements. A general purpose waveguide measurement setup^{32,40,41} is used for validation of the proposed method, as shown in Fig. 6. A HP8720C Vector network analyzer (VNA) is connected as a source and measurement equipment. It has a 1 Hz frequency resolution (with option 001) and 8 ppm (parts per million) frequency accuracy. The waveguide used in measurements has a width of 22.86 mm ($f_c \cong 6.555$ GHz).

The extra waveguide sections with lengths greater than 70 mm, located at Ports 1 and 2 in Fig. 6, are utilized to sup-

press any spurious reflections inside the measurement cell. For each liquid sample measurements, first the cell and upper extra waveguide section are unbolted (Port 2), then the liquid sample is poured onto the sample holder, and finally the cell and upper waveguide section are bolted tightly (Port 2). For waveguide measurements, bolting and unbolting process could be repeated many times without loss of measurement accuracy. Before the pouring process, the empty measurement cell is cleaned and dried.

For calibration of the set-up, the thru-reflect-line (TRL) calibration technique^{30,31,41} is utilized. We used a waveguide short and the 44.38 mm long waveguide spacer for reflect and line standards, respectively. The line has a $\pm 70^\circ$ maximum offset from 90° between 9.7 GHz and 11.7 GHz. In order to assess the accuracy of measurements, we measured the magnitude of S_{11} for waveguide through measurements and noted that it ranges from -40 dB to -75 dB.

Based on the uncertainty analysis in Sec. III, a polytetrafluoroethylene (PTFE) material with length 8.06 mm is chosen as a sample holder since its length is large enough to sufficiently hold the sample and since it will not attenuate electromagnetic wave propagation through the cell. Since our proposed method assumes that the thickness and electromagnetic properties of the sample holder be known, we measured its relative permittivity ($\epsilon_{r2} \cong 2.04 - i0.005$ and $\mu_{r2} = 1$) using amplitude-only measurements of S_{11} and complex S_{21} over the 9.7–11.7 GHz.³¹

For validation of the presented method, we use the measurement data of distilled water ($L_3 \cong 5.04$ mm) and methanol ($L_3 \cong 10.02$ mm) taken from Ref. 41. Lengths of the liquid samples are determined as follows. We first measure masses of sampled (some amount of) distilled water and methanol, respectively, as $m_d \cong 1.166$ g and $m_m \cong 1.842$ g using a high precision scale. Then, we calculate corresponding volumes of distilled water and methanol, respectively, as $v_d \cong 1.168$ cm³ and $v_m \cong 2.326$ cm³ using their density values: $\rho_d = 0.9982$ g/cm³ for distilled water and $\rho_m = 0.7918$ g/cm³ for methanol (both are values for room temperatures). Next, using these volume values, we calculate the thicknesses of distilled water and methanol, respectively, as $L_3 \cong 5.04$ mm and $L_3 \cong 10.02$ mm for known cross section area (22.86×10.16 mm²) of X-band waveguides. To be demonstrated in

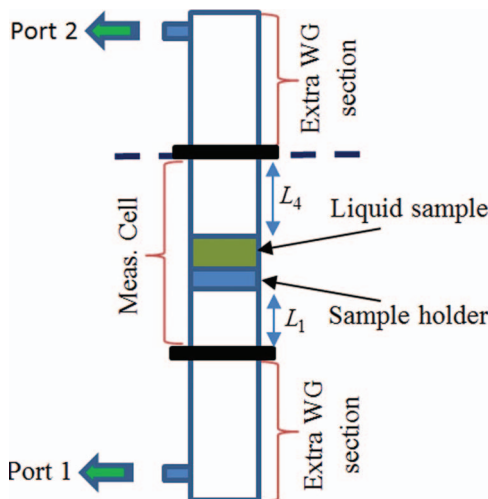


FIG. 6. Schematic view of the measurement setup. Here WG denotes the acronym of waveguide.

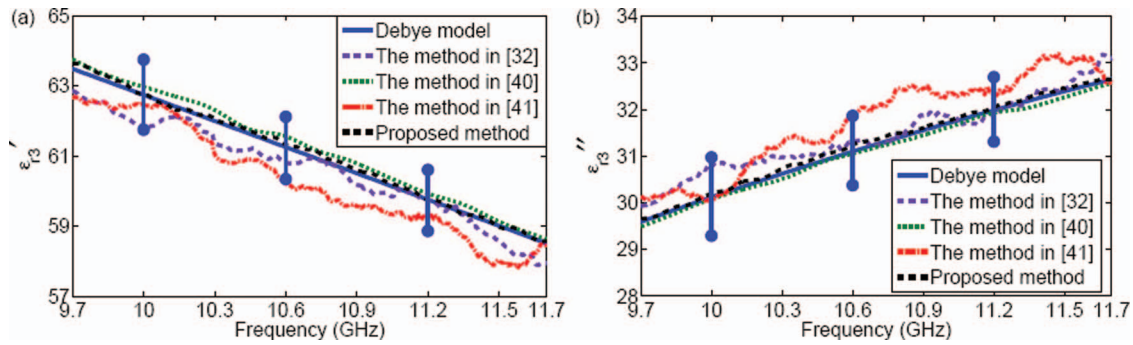


FIG. 7. Measured (a) real part and (b) imaginary part of ϵ_{r3} of distilled water over frequency by the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ the noniterative transmission-reflection method,⁴¹ and the proposed method.

Figs. 11(a) and 11(b) that if the density (or the sample length) of the sample under test is not known precisely, measurement accuracy greatly degrades. Our proposed method eliminates this requirement by using new formulas in Eqs. (9)–(11).

Theoretical ϵ_{r3} of distilled water and methanol over 9.7–11.7 GHz are obtained from the Debye model:⁷⁴

$$\epsilon_{r3} = \epsilon_{r3\infty} + (\epsilon_{r3s} - \epsilon_{r3\infty}) / (1 + i\omega\tau_3), \quad \omega = 2\pi f, \quad (17)$$

with the following parameters: $\epsilon_{r3\infty} = 5.2$, $\epsilon_{r3s} = 78.5$, and $\tau_3 = 8.33$ ps (the relaxation time) for distilled water, and $\epsilon_{r3\infty} = 5.6$, $\epsilon_{r3s} = 32.6$, and $\tau_3 = 48$ ps for methanol.

In the application of our method, we first determine Γ_3 of both distilled water and methanol at $f = 9.7$ GHz by using the graphical method discussed in Sec. II. We applied the graphical method to monitor occurrence of multi-valued solutions for Γ_3 for both samples. After deciding that one solution exists for the tested samples at the analyzed frequency, we then compute ϵ_{r3} of both samples. Next, using computed ϵ_{r3} of both samples at $f = 9.7$ GHz as initial guess for Newton's method (other multi-dimensional methods are also applicable, see Sec. II), we retrieved ϵ_{r3} of distilled water and methanol for the remaining frequency band.

To check whether accuracies of each method are within the limits of error bars, we superimposed total relative uncertainties $\Delta\epsilon_{r3}/\epsilon_{r3}$ (denoted by vertical bars with solid brown circles at each end) of distilled water and methanol at $f = 10.00$ GHz, 10.6 GHz, and 11.2 GHz. In the determination of $\Delta\epsilon_{r3}/\epsilon_{r3}$, theoretical ϵ_{r3} values for distilled water and methanol obtained from the Debye model have been used.

Measurements of ϵ_3 of both distilled water and methanol are carried out at an ordinary room temperature around 23 °C. Throughout measurements, we tried to keep the temperature conditions constant (23 °C \pm 5%) since ϵ_3 depends on temperature. From the measured data in Figs. 7–11, we note the following key points.

1. First, we note from Figs. 7 and 8 that measured ϵ_{r3} by our proposed method and by the methods in Refs. 32, 40, and 41 are in good agreement with each other and the permittivity data obtained from the Debye model.⁷⁴ However, retrieved ϵ_{r3} values by the methods^{32,41} have more fluctuations over frequency than those extracted by the proposed method and that in Ref. 40. Furthermore, it is noted from Figs. 7 and 8 that retrieved ϵ_{r3} values of distilled water and methanol by all applied methods are within the range of uncertainty levels, showing partly the accuracy of measured S-parameters.
2. We note from Figs. 9 and 10 that measured ϵ_{r3} values by the reference-plane-dependent methods^{32,40,41} greatly diverge, as noted how large retrieved ϵ_{r3} deviates from the uncertainty limits (vertical blue lines) calculated from the Debye model, by a small offset (+0.5%) in the reference-plane transformation factor (L_1) from theoretical ϵ_{r3} values obtained from the Debye model. For example, measured ϵ'_{r3} at $f = 10.6$ GHz of distilled water by the noniterative transmission-reflection method⁴¹ alters approximately from 60.26 to 63.04 (a %4.4 increase) by this +0.5% small offset. This shows

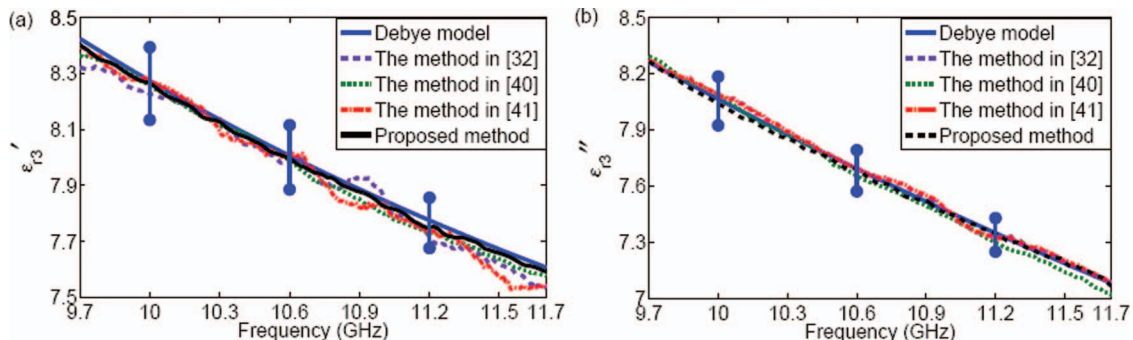


FIG. 8. Measured (a) real part and (b) imaginary part of ϵ_{r3} of methanol over frequency by the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ the noniterative transmission-reflection method,⁴¹ and the proposed method.

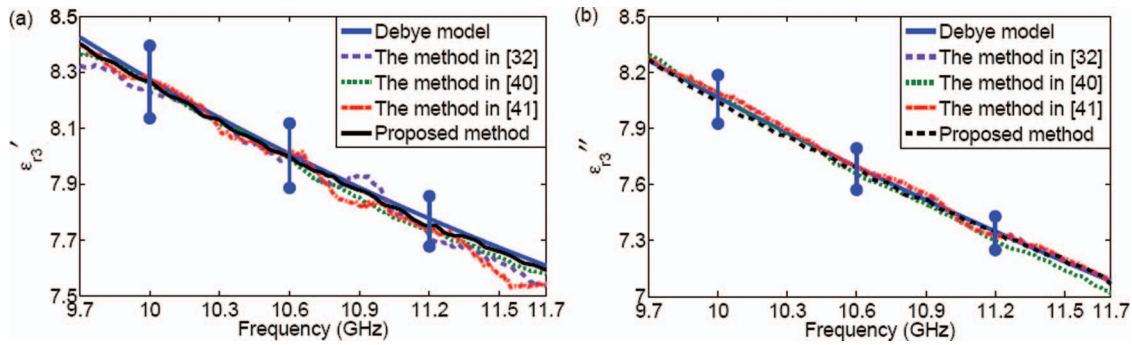


FIG. 9. Measured (a) real part and (b) imaginary part of ϵ_{r3} of distilled water over frequency by the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ the noniterative transmission-reflection method,⁴¹ and the proposed method. There is a +0.5 % offset in the reference-plane transformation factor L_1 ($L_1 = 10.05$ mm).

the importance of the application, in the retrieval of electromagnetic properties, of a method which is not a function of reference-plane-dependent expressions. We note from Fig. 10 that an offset in L_1 alters extracted ϵ'_{r3} more than ϵ''_{r3} . Additionally, we note from Figs. 9 and 10 that ϵ''_{r3} of methanol is less affected by the offset in L_1 than ϵ'_{r3} of distilled water. We think that this arises from the fact that loss tangent of methanol is higher than the loss tangent of distilled water. On the other hand, since our proposed method does not require the precise information on L_1 (or L_2) value so as to retrieve correct ϵ_{r3} values, its accuracy is not affected.

- We note from Fig. 11 that the offset (+1%) in sample lengths of each sample significantly changes measured ϵ'_{r3} of both samples extracted from the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ and the non-iterative transmission-reflection method.⁴¹ For instance, we note from Figs. 7(a) and 11(a) that measured ϵ'_{r3} at $f = 10$ GHz of distilled water by the method³² changes approximately from 61.83 to 61.28 (−0.9% decrease) for this +1% length offset. For conciseness purposes, only are retrieved ϵ'_{r3} values shown in Fig. 11, since a similar observation also applies to variations of ϵ''_{r3} with the sample length. The dependences in Fig. 11 present the importance of using precise sample length in the extraction of electromagnetic

properties of materials. Our proposed method eliminates this constraint by using thickness-independent expressions in Eqs. (9)–(11).

- Proper branch indices are used in extraction of electromagnetic properties of distilled water and methanol by the methods in Refs. 32, 40, and 41. These indices are estimated from the theoretical dependencies of liquid samples by the Debye model. For liquid samples whose range of electromagnetic properties are yet unknown, the methods in Refs. 32, 40, and 41 may produce erroneous results especially for electromagnetic properties of thick samples. The methods in Refs. 15–27 can be utilized as a remedy to some degree. In particular, for measurements of electromagnetic properties of highly dispersive liquids in a limited frequency range (or at some certain frequencies), these methods cannot help much. Nonetheless, our proposed method resolves this problem by using branch-index-independent expressions in Eqs. (9)–(11).

Although the proposed method has many advantages as shown from the dependencies in Figs. 7–11, it only retrieves ϵ_r of liquid samples and requires a numerical technique (iterative method). As a future study, we would like to extend this powerful technique for non-iterative constitutive parameters retrieval of magnetic materials.

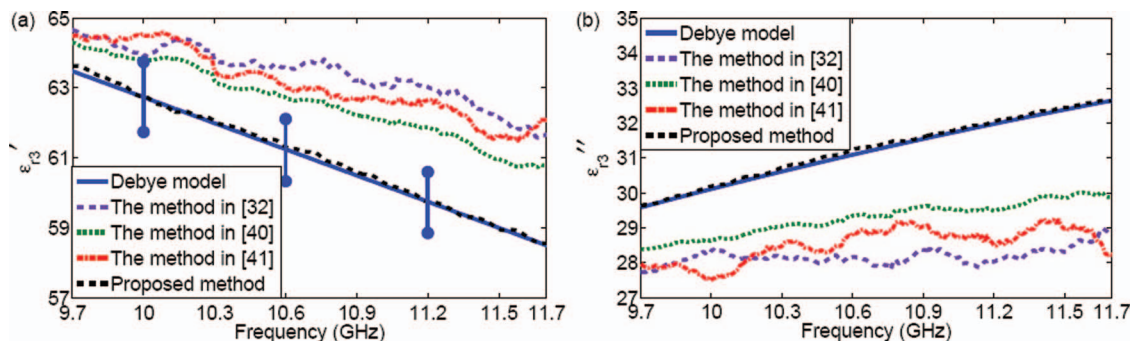


FIG. 10. Measured (a) real part and (b) imaginary part of ϵ_{r3} of methanol over frequency by the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ the noniterative transmission-reflection method,⁴¹ and the proposed method. There is a +0.5 % offset in the reference-plane transformation factor L_1 ($L_1 = 10.05$ mm).

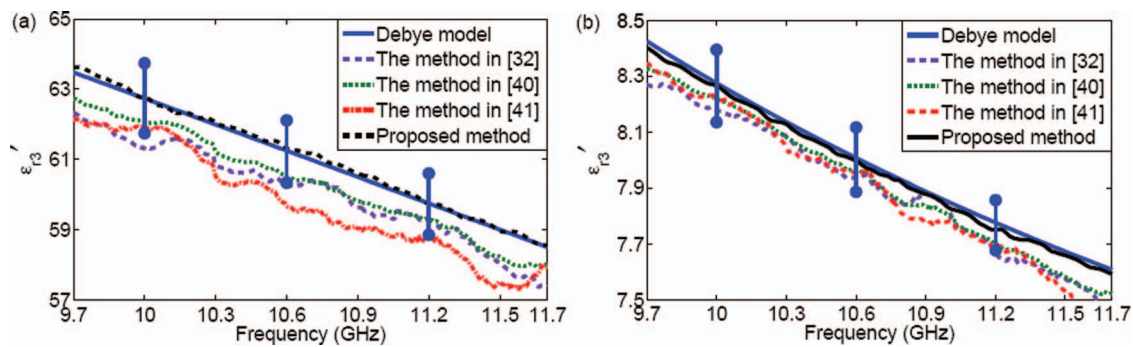


FIG. 11. Measured real parts of ϵ_{r3} of (a) distilled water and (b) methanol over frequency by the accurate and stable method,³² the modified transmission-reflection method,⁴⁰ the noniterative transmission-reflection method,⁴¹ and the proposed method. There is a +1 % offset in the lengths of each sample ($L_3 \cong 5.09$ mm for distilled water and $L_3 \cong 10.12$ mm for methanol).

V. CONCLUSIONS

An attractive transmission-reflection method has been proposed for accurate extraction of complex permittivities of dielectric liquid samples poured over a sample holder inside a vertically positioned waveguide cell. The method uses thickness-independent and reference-plane-invariant expressions as shown in Eqs. (9)–(11). In addition, it retrieves complex permittivity without the need of correct branch index value. This is especially an important feature in extraction of electromagnetic properties of newly fabricated materials (or materials whose electromagnetic properties are not yet known). Finally, we note that the method uses a very restricted domain for solutions of the reflection coefficient Γ_3 , facilitating fast computations of electromagnetic properties of liquid materials. A graphical method has been consulted for validation of the proposed method. We have then applied the well-known differential uncertainty model to assess the accuracy level of the proposed method for various uncertainty parameters. We note from this analysis that the selection of appropriate sample holder (its thickness and electromagnetic properties) can increase the accuracy level of retrieved complex permittivity of liquid samples by the proposed method. We validated the proposed method by using S-parameter measurements of distilled water and methanol and by comparing their retrieved electromagnetic properties by the proposed method with those by other similar methods in the literature. In the comparison, we monitored the effects of inaccurate reference-plane transformation factors and the sample thickness on measured electromagnetic properties. We note from measurements of tested liquid samples that while the accuracy of proposed method is not altered by any imprecise knowledge of reference-plane transformation factors and sample thickness, those of other similar methods are drastically changed. These results demonstrate that our proposed method is a good candidate especially for measurements of electromagnetic properties of dielectric liquid samples when their densities (or equivalently thicknesses) are not known precisely and when their location inside the sample holder is unclear.

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