Calibration of a Coaxial-loaded Stepped Cut-off Circular Waveguide and Related Application of Dielectric Measurement for Liquids

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SUMMARY A novel jig structure for $S_{11}$ calibration with short/open conditions and one reference material (referred to here as SOM) in dielectric measurement of liquids using a coaxial feed type stepped cut-off circular waveguide and a formula for exact calculation of $S_{11}$ for the analytical model of the structure using the method of moments (MoM) was proposed. The accuracy and validity of $S_{11}$ values calculated using the relevant formula was then verified for frequencies of 0.50, 1.5 and 3.0 GHz, and $S_{11}$ measurement accuracy with each termination condition was verified after calibration with SOM by combining the jig of the proposed structure with the study’s electromagnetic (EM) analysis method. The relative complex permittivity was then estimated from $S_{11}$ values measured with various liquids in the jig after calibration, and differences in results obtained with the proposed method and the conventional jig, the analytical model and the EM analysis method were examined. The validity of the proposed dielectric measurement method based on a combination of the above jig structure, numerical $S_{11}$ calculation and the calibration method was thus confirmed.

key words: Dielectric measurement, liquid, cut-off circular waveguide, $S_{11}$, calibration

1. Introduction

Research has recently advanced in the field of microwave chemistry, which involves heating using electromagnetic (EM) waves with an accelerating liquid solvent to synthesize new chemicals [1]. Accordingly, there is a need for a method enabling accurate determination of dielectric constants at the irradiation frequency of the material being heated for efficient work. Against this background, Stuchly et al. (1980) overviewed dielectric measurement for various lossy media based on reflected and transmitted EM waves with a sample in the middle or at the tip of a transmission line [2]. In the above research, dielectric measurement based on reflection coefficients with a sample in a cut-off circular waveguide placed at the tip of a coaxial line was also examined, but no actual dielectric measurement example with this jig shape was included in the report [2]. Belhadj-Tahar (1986) proposed a method enabling estimation of dielectric constants for materials based on reflected and transmitted waves with a sample in a circular cavity between two coaxial lines [3]. However, the structure used needs to be improved so that the sample can be inserted into the cavity without gaps. Göttmann et al. (1996) proposed dielectric measurement based on a reflection coefficient with a sample in both the coaxial line and the cut-off circular waveguide, which is placed at the tip of the coaxial line of the connector [4], [5]. In this method, movement of the observation plane from the SOL calibration plane to the front of the sample (the electrical length offset) is therefore required. Meanwhile, the coaxial line transmission wave method, in which complex permittivity can be estimated from $S_{21}$ with the sample in the coaxial line, is also used for dielectric measurement of powders and liquids [6], [7]. A theory has also been proposed to eliminate the influence of variations in liquid insertion into the transmission line to improve measurement accuracy in this approach [8]. However, this involves liquid insertion into the jig with connection to a measurement cable to eliminate liquid-surface meniscus effects. Accordingly, jig structure improvement (such as the inclusion of a liquid insertion hole in the outer conductor of the coaxial line) is needed.

Meanwhile, the coaxial probe method [9] – [13] is commonly used to measure the dielectric constant of high-loss solids and liquids in the high-frequency band. However, the estimated relative permittivity is significantly affected by EM waves reflected from the boundary between the sample and air, or by the container for small samples [14]. Accordingly, a large sample is needed to prevent these issues.

Nishikata (2009) proposed dielectric measurement in liquids based on transmitted waves ($S_{21}$) with a sample in a hollow resin tube penetrating the H-plane of a rectangular waveguide [15]. However, the measurable frequency is generally limited to the band in which only the fundamental mode of the rectangular waveguide can be propagated. Moreover, the cavity resonator method can be used for high-precision dielectric measurement [16], [17], but does not support broadband measurement with continuous frequency because it involves the determination of material constants at discrete frequencies in response to the resonant frequency of the cavity resonator.

Against this background, Shibata (2010) previously outlined the effectiveness of high-precision broadband dielectric measurement for small amounts of certain liquids based on a reflection constant using a coaxial-feed-type open-ended cut-off circular waveguide [18]. To develop this method, the potential for dielectric measurement in liquids in the low frequency band, estimation using a simple formula, calculation for uncertainty and ways to improve measurement accuracy.
have been presented [19] – [23]. Moreover, calibration of $S_{11}$ at the front of the sample using three reference materials and SOM (short/open conditions and a known material) with a jig in the measurement system using a VNA (vector network analyzer) and before dielectric measurement has also been proposed [24], [25]. After calibration using the above methods, $S_{11}$ calculated with various liquids inserted and the relative complex permittivity estimated from $S_{11}$ were compared with those from the conventional method and similar approaches, with results showing the validity of related $S_{11}$ calibration and relative permittivity estimation [26]. In the future, it will be necessary to confirm the validity of this method by conducting Round-robin testing at other institutions. Moreover, a jig structure for more stable $S_{11}$ calibration and subsequent dielectric measurement is required for future standardization of the open-ended cut-off circular waveguide reflection method. However, with a conventional jig, the outer conductor of the coaxial line and the waveguide have the same diameter, thereby hindering complete electrical contact.

This paper proposes a novel jig structure for $S_{11}$ calibration with reliable short termination to be performed before estimation of liquid dielectric permittivity based on cut-off waveguide reflection. Specifically, the inner diameter of the jig’s circular waveguide was made slightly larger than that of the outer conductor of the coaxial line to allow complete contact between the short element and the outer conductor (ground plane) of the coaxial line. $S_{11}$ for the front of the sample was calibrated via SOM termination [24] using a VNA, a jig with the above structure for sample insertion, a round metal bar and a jig for bar press-fitting. In contrast to the conditions with $\phi 4.40$ jig, $S_{11}$ calculated using the method of moments (MoM) [27], [28] for the analytical model, in which the step between the coaxial line and the cut-off waveguide is faithfully reproduced, was substituted into the formula for $S_{11}$ calculation. With pure water as a reference material, after calibration with the new jig structure and the procedure described above, $S_{11}$ values measured at frequencies of 0.50, 1.5 and 3.0 GHz with various termination conditions (including an unknown material in the jig) were compared with those measured after calibration with the conventional jig structure and the analytical model with the mode-matching technique (MMT) [18]. The results showed a quantitative difference associated with analytical model differences. The relative complex permittivity was also estimated from $S_{11}$ values measured with various liquids inserted after calibration of jig with the proposed structure and SOM condition. Estimated values were compared with the relative complex permittivity estimated using a conventional jig in which the inner diameter of the outer conductor of the coaxial line was the same as the inner diameter of the circular waveguide. Quantitative evaluation of errors in liquid permittivity estimated with the conventional structure and the EM analysis method also indicated the effectiveness of $S_{11}$ calibration and dielectric estimation using the proposed structure and the $S_{11}$ calculation technique.

2. $S_{11}$ calibration with a jig and related dielectric measurement

In the proposed method, the relative complex permittivity is estimated by comparing $S_{11}$ measurements with those of various liquids in a stepped cut-off circular waveguide with a coaxial feed and calculated $S_{11}$ values for the analytical model (Figs. 1 to 3). Figure 2 shows $S_{11}$ calibration for a jig at the sample front surface of the coaxial line in the pre-stage of this work with short/open conditions and one reference material (SOM) [24]. The specific measurement procedure is as follows:

1. The measurement jig (a coaxial-feed-type cut-off circular waveguide with an SMA connector) is attached to a measurement cable connected to a VNA.
2. $S_{11}$ is calibrated at the front surface of the jig sample with SOM termination.
3. $S_{11}$ at the front of the sample material is measured with various liquids in the jig.
4. The dielectric constant is estimated as an inverse problem so that the calculated $S_{11}$ value for the jig-related analytical model corresponds to the measured value for each frequency.

The proposed $S_{11}$ calibration with SOM (short/open conditions and a reference material) termination is outlined here. Calibration for $S_{11}$ with SOM – as applied in the previous stage of dielectric measurement of materials based on reflection coefficient differences with various samples in the jig – has previously been reported (e.g., [29]). This procedure is applied to commercial products such as dielectric measurement kits involving the use of coaxial probes from Keysight Technologies [30]. However, in [29], the capacitance of the jig required to calculate the equivalent circuit (i.e., the theoretical value) for $S_{11}$ calibration is not calculated from the exact solution of the analytical model. Accordingly, errors relating to differences from actual capacitance (i.e., the theoretical input impedance of the jig) is included in the calibrated $S_{11}$ value. In the proposed method, the $S_{11}$ value calculated from EM analysis of an exact analytical model of the stepped circular waveguide with a coaxial feed is substituted into the formula for $S_{11}$ calibration. The reflection coefficient $\Gamma_{corr}$ for Ref. 2 of the measurement jig (Fig. 1) is then calibrated by substituting the measured $\rho_{meas}$ value of the reflection coefficient for Ref. 1 into Eq. (1) [24], [25].

$$\Gamma_{corr} = \frac{\hat{\rho}_{meas} - E_{DF}}{E_{SF} \cdot \rho_{meas} + E_{RF} - E_{SF} \cdot E_{DF}} \quad (1)$$

Here, $E_{SF}$, $E_{DF}$ and $E_{RF}$ are system error terms required for $S_{11}$ calibration as defined by Eqs. (2) to (4).
\[ E_{SF} = \frac{\hat{\rho}_2 - \hat{\rho}_1 + \hat{\gamma} \cdot (\hat{\Gamma}_3 - \hat{\Gamma}_2)}{\hat{\Gamma}_2 \cdot (\hat{\rho}_2 - \hat{\rho}_1)} \]  
(2)

\[ \hat{E}_{DF} = \hat{\rho}_1 - \hat{\Gamma}_1 \cdot (\hat{E}_{SF} \cdot \hat{\rho}_1 + \hat{\gamma}) \]  
(3)

\[ \hat{E}_{RF} = \hat{E}_{DF} \cdot \hat{E}_{SF} + \hat{\gamma} \]  
(4)

The auxiliary function \( \gamma \) of the above values in Eqs. (2) to (4) is defined by Eq. (5).

\[
\hat{\gamma} = \frac{(\hat{\rho}_2 - \hat{\rho}_1) \cdot (\hat{\Gamma}_3 \cdot \hat{\rho}_2 - \hat{\Gamma}_1 \cdot \hat{\rho}_1) + (\hat{\rho}_3 - \hat{\rho}_1) \cdot (\hat{\Gamma}_1 \cdot \hat{\rho}_3 - \hat{\Gamma}_2 \cdot \hat{\rho}_2)}{(\hat{\Gamma}_1 - \hat{\Gamma}_2) \cdot (\hat{\Gamma}_2 \cdot \hat{\rho}_2 - \hat{\Gamma}_1 \cdot \hat{\rho}_1) + (\hat{\Gamma}_1 - \hat{\Gamma}_3) \cdot (\hat{\Gamma}_3 \cdot \hat{\rho}_3 - \hat{\Gamma}_2 \cdot \hat{\rho}_2)} 
\]  
(5)

Here, \( \hat{\rho} \) and \( \hat{\Gamma} \) in Eqs. (2) to (5) are the measured reflection coefficient for Ref. 1 and the theoretical reflection coefficient for Ref. 2, respectively. The suffix “\( i \)” (= 1, 2 and 3) distinguishes the three standard termination conditions. Moreover, the values calculated using the MoM as described in Section 4 are substituted as the theoretical values \( \hat{\Gamma}_i \) in Eqs. (2) to (5) for Ref. 2 (Fig. 1) with the three standard jig termination conditions. \( S_{11} \) measurement values for Ref. 1 from a VNA are substituted for the reflection constant \( \hat{\rho} \) into the above formula. \( S_{11} \) measurement values on the front surface of the sample with various liquids inserted is thus calibrated.

A new sample holder (jig) was placed in the circular waveguide (at distance d from the boundary plane with the coaxial line), and \( S_{11} \) with the analytical model is calculated from the above reflection coefficient \( \hat{\Gamma}_i \) for Ref. 2 at the discontinuity between the coaxial line and the circular waveguide is defined via the following equation by applying the MoM [27], [28] to the analytical model:

\[ \Gamma_i = S_{11} = V_i - 1 \]  
(6)

Thus, the input impedance of the TEM mode for Ref. 2 at the connection plane between both regions can also be calculated from the above reflection coefficient \( \Gamma_i \). Here, \( V_i \) is the voltage of the TEM mode in the coaxial line. The mode voltage \( V_n \) of TEM and TM_{0m} including higher-order modes, is calculated as

\[ V_n = Y_{mn} \cdot I_m \]  
(7)

where \( I_m \) is the electrical current of the m-th TM (TM_{0m}) mode in the coaxial line section, and is calculated as a 1-

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**Fig. 1 Analytical model (coaxial loaded cut-off circular waveguide)**

**Fig. 2 Auxiliary jig for contact of the short rod with coaxial line (left)**

**Fig. 3 Structure of waveguide for stable calibration with short (right)**

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### 3. Novel waveguide structure and auxiliary jig to ensure electrical shorting for \( S_{11} \) calibration

The realization of a complete electrical short termination using a conventional jig for dielectric measurement [24] in which the inner diameter of the waveguide is the same as that of the outer conductor of the coaxial line is challenging because the calibration surface of \( S_{11} \) is inside the waveguide. In this study, \( S_{11} \) calibration was realized with favorable short termination via slight jig improvement. Specifically, 1. A new sample holder (jig) with the internal structure shown in Fig. 1 (with the inner diameter of the waveguide slightly larger than that of the outer conductor of the coaxial line) was created. 2. A round brass bar for short termination with outer dimensions slightly larger than the inner diameter of the outer conductor of the coaxial line was also created. 3. A round brass bar was placed in the jig insertion hole. 4. Pressure was applied to the brass bar using the auxiliary jig (Fig. 2). 5. The brass bar remained in contact with the front surface of the outer conductor (ground plane) of the coaxial line of the jig (Fig. 3). An electrical short at the front of the sample was then realized via the above procedure. Application of this process enabled stable \( S_{11} \) calibration at the front of the sample in the cut-off circular waveguide. The inner diameter of the outer conductor of the coaxial line was \( \phi 4.10 \) mm, as per manufacturer specifications. The inner diameter of the circular waveguide (jig) and the round metal bar were \( \phi 4.40 \) and \( \phi 4.38 \) mm, respectively. Thus, complete electrical contact between the metal bar and the outer conductor in front of the coaxial line for \( S_{11} \) calibration with short termination was realized (Fig. 3).
by-m matrix using
\[ I_n = \frac{2 \cdot \delta_{mn}}{\eta_0} \]  
\[ (8) \]

Here, \( \delta_{mn} \) is the Kronecker delta function calculated as \( \delta_{mn} = 1 \) (m = 1) and \( \delta_{mn} = 0 \) (m \neq 1). \( \eta_0 \) is the characteristic impedance of the TEM mode in the coaxial line in region A, expressed as
\[ \eta_0 = \sqrt{\frac{\mu_0}{\varepsilon_0}} \]  
\[ (9) \]

Here, \( \varepsilon_0 \) and \( \mu_0 \) represent permittivity and permeability in a vacuum, respectively. \( \eta_{mn} \) in Eq. (7) is the composite admittance at the connection plane as calculated using the following m-by-n matrix:
\[ Y_{mn} = \frac{\delta_{mn}}{\eta_{m-1}} - j\omega \sum_{i=1}^{l} F_{ni} F_{mi} \]  
\[ (10) \]

Here, \( d \) [m] is the length of region B, which is set to \( d \to \infty \) under the condition of infinite length. \( \tan(\xi d) \) is replaced with \( \tan(\xi d) = -j \), and \( \eta_0 \) is the line impedance for the TM\(_0\) mode of the coaxial line in region A, calculated as
\[ \eta_0 = \frac{\gamma_0}{j\omega \varepsilon_0} \]  
\[ (11) \]

Here, \( \gamma_0 \) is the propagation constant of the TM\(_{0m}\) mode for the coaxial line in region A, calculated as
\[ \gamma_0 = \sqrt{k_0^2 + \gamma_0^2} \]  
\[ (n \geq 1) \]  
\[ (12) \]

Here, \( k_0 \) is the n-th root of
\[ J_0(k_0 b) - Y_0(k_0 a) - J_0(k_0 b) = 0 \]  
\[ (13) \]

\( J_0(k_0 b) \) and \( J_0(k_0 a) \) are Bessel functions of the first kind, and \( Y_0(k_0 a) \) and \( Y_0(k_0 b) \) are Bessel functions of the second kind. \( y_0 \) in Eq. (12) is the propagation constant in a vacuum, expressed as
\[ y_0 = j\omega \sqrt{\varepsilon_0 \mu_0} \]  
\[ (14) \]

\( \xi_0 \) in Eq. (10) is the propagation constant in the cut-off circular waveguide in region B, defined as
\[ \xi_0 = \sqrt{\omega \varepsilon_0 \mu_0 - P_c^2} \]  
\[ (15) \]

Here, \( P_c \) is the i-th root of \( J_0(P_c) = 0 \). \( F_{mn} \) in Eq. (10) when the radius \( \rho \) for the outer conductor of the circular waveguide is larger than the inner diameter \( a \) of the coaxial line (\( c > a \)) is calculated as
\[ F_n = \left\{ \begin{array}{ll} \frac{-2\sqrt{\pi} \cdot N_{n-1} \cdot P_c \cdot \left[ J_0(P_c) \cdot a_{n-1} - J_0(P_c) \cdot b_{n-1} \right]}{c \cdot J_0(P_c) \cdot [k_{n-1} - P_c]^2} & (n \geq 1) \end{array} \right. \]  
\[ (16) \]

Here, \( a, b, \) and \( c \) are the radius of the outer conductor’s inner diameter for the coaxial line in region A, the inner conductor’s outer diameter for the coaxial line in region A, and the inner diameter of the circular waveguide in region B. \( n \) is a normalization constant calculated as
\[ n_0 = \frac{1}{\sqrt{\delta_0^2 - \beta_0^2}} \]  
\[ (17) \]

\( \alpha_n \) and \( \beta_n \) are calculated as
\[ \alpha_n = a \left[ J_n(k_n b)Y_n(k_n a) - Y_n(k_n b)J_n(k_n a) \right] \]  
\[ (18) \]

\[ \beta_n = b \left[ J_n(k_n b)Y_n(k_n b) - Y_n(k_n b)J_n(k_n b) \right] \]  
\[ (19) \]

\( F_{mn} \) and \( F_{mi} \) are calculated using Eqs. (16) – (19) to determine \( S_{11} \) for the analytical model (Fig. 1). \( Y_{mn} \) is then calculated by substituting \( F_{mn} \) and Eqs. (11) – (15) into Eq. (10), and the mode voltage \( V_m \) is calculated by substituting \( I_m \) in Eq. (8) and the inverse matrix of \( Y_{mn} \) in Eq. (10) into Eq. (7). The reflection coefficient \( I_1 \) for the TEM mode at the connection surface is then calculated using Eq. (6) from the voltage \( V_1 \) of the dominant TEM mode among the above \( V_m \) values.

5. \( S_{11} \) Calibration for the jig at 0.5, 1.5, and 3.0 GHz

This chapter outlines the effectiveness of \( S_{11} \) calibration for the jig (a coaxial feed stepped coaxial circular waveguide) as described in Chapter 2 for frequencies of 0.50, 1.5, and 3.0 GHz. The error in \( S_{11} \) calibrated using the conventional structure and EM analysis method was also quantitatively evaluated. For this purpose, an experimental setup is shown in Fig. 4. A vector network analyzer (VNA) was used for measurement of \( S_{11} \). Next, a 6dB coaxial attenuator (AT-106 (40), Hirose Electric) was connected to Port 1 of the VNA via a coaxial cable. The measurement jig was then connected to the coaxial attenuator. Moreover, the procedure for \( S_{11} \) calibration with SOM on the material front surface (Ref. 2) of the jig with an inner diameter of \( \phi 4.40 \) mm, \( 2b = 1.30 \) mm, \( d = 5.00 \) mm and \( \varepsilon_1 = 2.05 \) at the sample insertion part was evaluated as follows:

1. The jig is attached after calibration of the coaxial cable tip using a general SMA-type SOL calibration kit. 2. \( S_{11} \) is measured on the SOL calibration plane (Ref. 1) with the jig short, open and with one reference material (pure water) inserted. 3. The theoretical \( S_{11} \) for Ref. 2 with the jig tip open and pure water inserted, as necessary for calibration, is determined using Eq. (6) based on exact calculation via the MoM. 4. The measured values of \( S_{11} \) after calibration at the front surface of the sample under various termination conditions are determined by substituting the measured and theoretical values of the above reflection coefficient into Eqs. (1) – (5).

The validity of the proposed jig structure and \( S_{11} \) calibration procedure was verified as below, and differences in \( S_{11} \) calibration values associated with differences in the calculation methods for theoretical \( S_{11} \) were compared between 1. MoM [28] with a \( \phi 4.40 \) jig, and 2. Mode-matching technique (MMT) [18] with a \( \phi 4.10 \) jig.
with $\varphi 4.40$ and $\varphi 4.10$. In this study, the values with $\varphi 4.40$ jig were substituted into Eqs. (1) – (5) for $S_{11}$ calibration as in Ref. 2 as measured values of $S_{11}$ at the SOL calibration plane (Ref. 1) with SOM jig termination (Table 1). The values with $\varphi 4.40$ jig were also substituted into Eqs. (1) – (5) for $S_{11}$ calibration as measurement values as in Ref. 1 with various termination conditions, including with liquids in the jig (Table 2).

The calculated $S_{11}$ value for the proposed analytical model in Fig. 1 (with exact representation of the inner diameter of the sample insertion part as $\varphi 4.40$) for Ref. 2 using the MoM is substituted into $\Gamma_i$ in Eqs. (1) – (5) as the theoretical value for $S_{11}$ calibration. As shown in Table 3, the calculated value from the Debye dispersion equation [31] at a liquid temperature of 25.0°C for pure water and the theoretical value ($c_r = 1.0 - j 0.0$) for air (open) are set to the relative permittivity of the reference material (required for calculation of the true value $\Gamma_i$, ideal), respectively. Relative permittivity inside the coaxial line was set to $c_r = 2.05$, and the theoretical reflection coefficient $\Gamma_i$, ideal for Ref. 2 was calculated from the relative complex permittivity of the above sample insertion part via EM analysis using the MoM [28].

In the proposed method, a jig ($\varphi 4.40$) with a different inner diameter from the conventional one was used. Accordingly, it is necessary to clarify the difference in the estimated dielectric constant due to the difference in the size of the jig (the inner diameter). The measured values of input impedance after calibration in Case 1 (the diameter of $\varphi 4.40$ mm was used and method of moment (MoM) was applied for computation of input impedance) and Case 2 (the diameter of $\varphi 4.10$ mm was used and mode-matching technique (MMT) was applied for computation of input impedance) were thus compared as a preliminary step as follows:

The results are shown in Table 3 as input impedance. Differences in measurement accuracy with the proposed $S_{11}$ calibration from differences in the analytical model and calculation are compared. Here, the theoretical reflection coefficient calculated with the mode-matching technique (MMT) [18] under each sample insertion condition with the conventional analytical model (with the inner diameter of the sample insertion part as $\varphi 4.10$) is also shown in Table 3 as input impedance ($Z_{r, \text{ ideal}}$ and $Z_{r, \text{ ideal}}$). The results of $S_{11}$ calculation using the MoM [28] with a sample insertion part inner diameter of $\varphi 4.40$ and $S_{11}$ based on the MMT [18] with an inner diameter of $\varphi 4.10$ were compared, with results showing an input impedance difference of around 2.36% associated with the difference in the analytical model and the calculation method with no sample inserted (open). The differences were 1.38 – 1.55% for the real part and 1.33 – 0.45% for the imaginary part with pure water inserted. Here, the theoretical value of the calculated value in both methods needs to be 0 (zero) when air is inserted into the jig. On the other hand, the actual value is shown in Table 3 due to the factor of calculation accuracy at the time of numerical calculation. Accordingly, only the calculation results for the real part of input impedance are shown.

Table 1: Input impedance measurement values with reference material insertion (25.0°C) for the SOL plane (Ref. 1) in $S_{11}$ calibration

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>$Z_{r, \text{ ideal}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Short</td>
<td>0.070838</td>
<td>+j 5.9600</td>
</tr>
<tr>
<td>Open</td>
<td>0.98498</td>
<td>+j 41.04</td>
</tr>
<tr>
<td>Pure water</td>
<td>2.0682</td>
<td>+j 95.084</td>
</tr>
</tbody>
</table>

Table 2: Input impedance measurement values with various termination conditions for the SOL plane (Ref. 1) for $S_{11}$ correction at the front of the sample (Ref. 2) after calibration

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>$Z_{r, \text{ ideal}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>14.176</td>
<td>+j 166.55</td>
</tr>
<tr>
<td>Ethanol</td>
<td>37.129</td>
<td>+j 202.46</td>
</tr>
</tbody>
</table>

Table 3: Reference-material dielectric constants (25.0°C) and theoretical values of input impedance at the front of the sample (Ref. 2) (calculation based on EM analysis)

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>$Z_{r, \text{ ideal}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relative complex permittivity of air</td>
<td>1.0 – j 0.0</td>
<td>+0.38915 – j 9963.6</td>
</tr>
<tr>
<td>Method of moments [28] with pure water $Z_{r, \text{ ideal}}$</td>
<td>+3.03479 – j 3320.0</td>
<td>+3.03479 – j 3320.0</td>
</tr>
</tbody>
</table>

Calibration differences were also examined under the conditions shown in Tables 1 and 2 in regard to measured values for Ref. 1 and theoretical values for Ref. 2. $S_{11}$ was calculated precisely using the MoM [28] with an inner diameter of $\varphi 4.40$ (Fig. 1), and $S_{11}$ was calculated using the MMT [18] with an inner diameter of $\varphi 4.10$. $S_{11}$ values measured with various termination conditions are shown in Table 2.
conditions after SOM calibration based on Eqs. (1) – (5) are shown in Table 4 as input impedance. Differences in $S_{11}$ measurement values (input impedance) with an open condition calibrated via SOM with each calculation condition for the theoretical reflection coefficient for calibration were compared between the MoM [28] with a sample insertion part inner diameter of ϕ4.40 and the MMT [18] with a sample insertion part inner diameter of ϕ4.10.

Small differences in measured input impedance for short (theoretical value $Z_{in} = 0.0 - j 0.0$) after calibration due to differences in reflection constant calculation between the MoM and the MMT were observed, at 1.34 – 0.036% for the real part and 1.30 – 0.525% for the imaginary part. Differences in post-calibration measured input impedance with an open condition (no sample inserted) due to differences between the MoM and the MMT in the calculation of the theoretical reflection coefficient were also compared, with results showing 2.87 – 4.63% for the real part and 2.39% – 2.37% for the imaginary part. Differences in measured values were directly attributed to differences in theoretical impedance values (Table 3) associated with differences in the analytical models and EM analysis methods. Differences in post-calibration measured input impedance with pure water due differences between the MoM and the MMT in the calculation of the theoretical reflection coefficient were also compared, with results showing 1.38 – 1.54% for the real part and 1.33% – 0.44% for the imaginary part. These outcomes indicated that differences in measured values are directly affected by differences in theoretical impedance values (Table 3) associated with differences in analytical models and EM analysis methods. It was thus found that SOM-based $S_{11}$ calibration is directly affected by $S_{11}$ values calculated using the analytical model and the EM analysis method used. Differences in post-calibration measured input impedance with the unknown materials of methanol and ethanol were also compared in relation to the differences in theoretical value calculation using the MoM and the MMT, with results showing 1.40 – 0.90% for the real part and 1.36 – 0.36% for the imaginary part.

6. Dielectric measurement with various liquids at frequencies of 0.50, 1.5 and 3.0 GHz

This chapter outlines estimation to determine the relative complex permittivity of various liquids in the jig at frequencies of 0.50, 1.5 and 3.0 GHz after $S_{11}$ calibration using the procedure described in Chapter 2. The estimated values were compared with those based on a conventional jig in which the inner diameter of the outer conductor of the coaxial line was the same as that of the circular waveguide, and the effectiveness of $S_{11}$ calibration and dielectric measurement with the proposed structure was evaluated. Errors in the complex permittivity of liquid estimated using the conventional structure and measurement procedure were also quantitatively evaluated (Table 5). The relative complex permittivity of pure water was estimated as an inverse problem via the MoM [28] as shown in Table 5 (a) after $S_{11}$ calibration with the SOM using a ϕ4.40 jig with the theoretical analytical model value calculated using the MoM [28] with the inner diameter of the sample insertion part as ϕ4.40. The results matched those estimated as an inverse problem via the MMT [18] after SOM $S_{11}$ calibration with the theoretical value calculated via the MMT with the inner diameter of the sample insertion part of the analytical model as ϕ4.10. This direct match is attributed to the fact that the $S_{11}$ value used to determine the theoretical value in calibration and that used to determine relative complex permittivity were

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>0.50</th>
<th>1.5</th>
<th>3.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Short</td>
<td>Calibrated value (MoM)</td>
<td>-j310.49</td>
<td>-j9915.4</td>
<td>-j5.7690×10^-1</td>
</tr>
<tr>
<td></td>
<td>Calibrated value (MMT)</td>
<td>-j3252.7</td>
<td>-j1671.6</td>
<td>-j5.8439×10^-1</td>
</tr>
<tr>
<td></td>
<td>Difference [%]</td>
<td>+1.348</td>
<td>+1.144</td>
<td>+0.036</td>
</tr>
<tr>
<td>Open</td>
<td>Calibrated value (MoM)</td>
<td>-j140.65</td>
<td>-j134.69</td>
<td>-j4.8606×10^-1</td>
</tr>
<tr>
<td></td>
<td>Calibrated value (MMT)</td>
<td>-j1671.6</td>
<td>-j117.29</td>
<td>-j4.9261×10^-1</td>
</tr>
<tr>
<td></td>
<td>Difference [%]</td>
<td>+2.867</td>
<td>+4.255</td>
<td>+4.634</td>
</tr>
<tr>
<td>Pure water</td>
<td>Calibrated value (MoM)</td>
<td>-j310.49</td>
<td>-j105.26</td>
<td>-j7.7148×10^-1</td>
</tr>
<tr>
<td></td>
<td>Calibrated value (MMT)</td>
<td>-j55.169</td>
<td>-j114.03</td>
<td>-j8.3717×10^-1</td>
</tr>
<tr>
<td></td>
<td>Difference [%]</td>
<td>+1.391</td>
<td>+1.268</td>
<td>+1.543</td>
</tr>
<tr>
<td>Methanol</td>
<td>Calibrated value (MoM)</td>
<td>-j310.49</td>
<td>-j105.26</td>
<td>-j7.7148×10^-1</td>
</tr>
<tr>
<td></td>
<td>Calibrated value (MMT)</td>
<td>-j55.169</td>
<td>-j114.03</td>
<td>-j8.3717×10^-1</td>
</tr>
<tr>
<td></td>
<td>Difference [%]</td>
<td>+1.391</td>
<td>+1.268</td>
<td>+1.543</td>
</tr>
<tr>
<td>Ethanol</td>
<td>Calibrated value (MoM)</td>
<td>-j310.49</td>
<td>-j105.26</td>
<td>-j7.7148×10^-1</td>
</tr>
<tr>
<td></td>
<td>Calibrated value (MMT)</td>
<td>-j55.169</td>
<td>-j114.03</td>
<td>-j8.3717×10^-1</td>
</tr>
<tr>
<td></td>
<td>Difference [%]</td>
<td>+1.391</td>
<td>+1.268</td>
<td>+1.543</td>
</tr>
</tbody>
</table>
calculated using identical EM analysis with MoM [28] and MMT [18]. Thus, individual jig differences were eliminated via calibration even where post-calibration $S_{11}$ measurement values differed due to differences in calculation for the theoretical reflection constant between the MoM with the inner diameter of the sample insertion part as $\phi_4.40$ and the MMT with $\phi_4.10$. Accordingly, the estimated relative complex permittivity values are considered to have matched despite differences in EM analysis and the analytical model.

The estimated values of relative complex permittivity for pure water after SOM $S_{11}$ calibration were also compared between 1. a $\phi_4.40$ jig with a theoretical value calculated via the MoM with the inner diameter of the analytical model as $\phi_4.40$ and relative permittivity estimated as an inverse problem via the MoM [28] after calibration, and 2. a $\phi_4.10$ jig with a theoretical value calculated via the MMT with the inner diameter of the analytical model as $\phi_4.10$ and relative permittivity estimated as an inverse problem via the MMT [18] after calibration. The results showed close agreement, exhibiting differences within 0.051% for the real part and 2.07 – 0.089% for the imaginary part at all frequencies. This close correspondence is attributed to the use of pure water in both cases as the reference material for $S_{11}$ calibration and subsequent measurement of unknown materials. The relative complex permittivity of unknown materials (methanol and ethanol) was also estimated as an inverse problem using each EM analysis method after SOM-based $S_{11}$ calibration. The results (Tables 5 (b) and (c)) compared the estimated values of relative complex permittivity between 1. a $\phi_4.40$ jig with a theoretical value calculated via the MoM with the inner diameter of the analytical model as $\phi_4.40$ and relative permittivity estimated via the MoM, and 2. a $\phi_4.10$ jig with a theoretical value calculated via the MMT with the inner diameter of the analytical model as $\phi_4.10$ and relative permittivity estimated as an inverse problem via the MMT [18] after calibration. This chapter outlines estimation to determine the relative permittivity of various liquids (as described in Chapter 6) at frequencies of 0.50 – 3.0 GHz. The results (Figs. 5 and 6) for pure water indicate close correspondence of measured values for the whole frequency band. The real part of relative permittivity for methanol was around 1.10 smaller with a new $\phi_4.40$ jig than with the previous various liquids inserted.

### Table 5 Differences in estimated relative permittivity based on an inverse problem via the MoM and the MMT with various liquids in consideration of differences in jig hole diameter (25.0°C)

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>Inner diameter of sample insertion part [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\phi_4.40$ (MoM)</td>
</tr>
<tr>
<td></td>
<td>0.50</td>
<td>1.5</td>
</tr>
<tr>
<td>$\phi_4.40$</td>
<td>78.63</td>
<td>75.26</td>
</tr>
<tr>
<td>Difference between the MoM and the MMT at $\phi_4.40$ [%]</td>
<td>-0.052</td>
<td>0.000</td>
</tr>
<tr>
<td>Difference between the MoM at $\phi_4.40$ and the MMT at $\phi_4.10$ [%]</td>
<td>+0.013</td>
<td>+0.051</td>
</tr>
<tr>
<td>Difference between the MMT at $\phi_4.40$ and the Debye dispersion formula [%]</td>
<td>+0.166</td>
<td>+0.192</td>
</tr>
<tr>
<td>(a) Pure water</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 5 Differences in estimated relative permittivity based on an inverse problem via the MoM and the MMT with various liquids in consideration of differences in jig hole diameter (25.0°C) (b) Methanol

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>Inner diameter of sample insertion part [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\phi_4.40$ (MoM)</td>
</tr>
<tr>
<td></td>
<td>0.50</td>
<td>1.5</td>
</tr>
<tr>
<td>$\phi_4.40$</td>
<td>35.82</td>
<td>38.02</td>
</tr>
<tr>
<td>Difference between the MoM and the MMT at $\phi_4.40$ [%]</td>
<td>+0.002</td>
<td>+0.002</td>
</tr>
<tr>
<td>Difference between the MMT at $\phi_4.40$ and the MMT at $\phi_4.10$ [%]</td>
<td>-0.559</td>
<td>-0.411</td>
</tr>
<tr>
<td>Difference between the MMT at $\phi_4.40$ and the Debye dispersion formula [%]</td>
<td>-1.782</td>
<td>-2.653</td>
</tr>
<tr>
<td>(b) Methanol</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 5 Differences in estimated relative permittivity based on an inverse problem via the MoM and the MMT with various liquids in consideration of differences in jig hole diameter (25.0°C) (c) Ethanol

<table>
<thead>
<tr>
<th>Condition</th>
<th>Frequency [GHz]</th>
<th>Inner diameter of sample insertion part [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\phi_4.40$ (MoM)</td>
</tr>
<tr>
<td></td>
<td>0.50</td>
<td>1.5</td>
</tr>
<tr>
<td>$\phi_4.40$</td>
<td>24.36</td>
<td>24.35</td>
</tr>
<tr>
<td>Difference between the MoM and the MMT at $\phi_4.40$ [%]</td>
<td>+0.223</td>
<td>+0.263</td>
</tr>
<tr>
<td>Difference between the MMT at $\phi_4.40$ and the MMT at $\phi_4.10$ [%]</td>
<td>-0.291</td>
<td>-1.141</td>
</tr>
<tr>
<td>Difference between the MMT at $\phi_4.40$ and the Debye dispersion formula [%]</td>
<td>-1.782</td>
<td>-2.653</td>
</tr>
<tr>
<td>(c) Ethanol</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 7. Frequency characteristics of relative complex permittivity in various liquids

This chapter outlines estimation to determine the relative permittivity of various liquids (as described in Chapter 6) at frequencies of 0.50 – 3.0 GHz. The results (Figs. 5 and 6) for pure water indicate close correspondence of measured values for the whole frequency band. The real part of relative permittivity for methanol was around 1.10 smaller with a new $\phi_4.40$ jig than with the previous
4.10 jig at 3.0 GHz, and the imaginary part was around 0.40 smaller. The relative permittivity of ethanol was slightly smaller with the new φ4.40 jig for both the real and imaginary parts, as was the case with methanol. However, these differences are considered to be within the range of variation caused by differences in liquid temperature and individual jigs. Accordingly, the dielectric constants of the various liquids measured with different jigs are considered to have matched within a certain range of variation. In future work, it will be necessary to clarify differences in uncertainty associated with differences in calibration and dielectric measurement methods based on measurement using more jigs. Here, in this method, pure water was used as the standard load when calibrating the jig. As a result, the estimated results of pure water by both methods are in perfect agreement as shown in Table 4 (a). Accordingly, the figure of the frequency characteristics of the relative complex permittivity for pure water was omitted.

Fig. 5 Frequency characteristics of relative complex permittivity for methanol

Fig. 6 Frequency characteristics of relative complex permittivity for ethanol

6. Conclusion

This paper proposed a novel jig structure for $S_{11}$ calibration with reliable short termination to be performed before estimation of liquid dielectric permittivity based on cut-off waveguide reflection. Specifically, the inner diameter of the jig’s circular waveguide was made slightly larger than that of the outer conductor of the coaxial line to allow complete contact between the short element and the outer conductor of the coaxial line. $S_{11}$ for the front of the sample was calibrated via SOM using a VNA, a jig with the above structure for sample insertion, a round metal bar and a jig for bar press-fitting. Moreover, $S_{11}$ calculated using the MoM for the analytical model, in which the step between the coaxial line and the cut-off circular waveguide is faithfully reproduced, was substituted into the formula for $S_{11}$ calibration. With pure water as a reference material, after calibration with the new jig structure and the procedure described above, $S_{11}$ values measured at frequencies of 0.50, 1.5 and 3.0 GHz with various termination conditions were compared with those measured after calibration with the conventional jig structure and the analytical model. The results showed a quantitative difference associated with analytical model differences. Relative complex permittivity was also estimated from $S_{11}$ values measured with various liquids inserted after jig calibration with the proposed structure and SOM. Estimated values were compared with relative complex permittivity estimated using a conventional jig in which the inner diameter of the outer conductor of the coaxial line was the same as the inner diameter of the circular waveguide. Quantitative evaluation of errors in liquid permittivity estimated with the conventional structure and the measurement procedure also indicated the effectiveness of $S_{11}$ calibration and dielectric measurement using the proposed structure and the $S_{11}$ calculation technique. Future work will involve evaluation of actual liquids and their temperature dependence along with extension of the method to examination of liquids in the millimeter range. Round-robin testing at different institutions is also necessary in association with standardization of this method.

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References


Kouji Shibata was born in Shimada City, Shizuoka Prefecture, Japan, in 1970. After graduating from the Engineering Department at the Kanazawa Institute of Technology in Ishikawa, Japan, in 1993, he took up employment with SPC Electronics (Shimada Physical & Chemical Industrial) Co., Ltd. in Chofu, Tokyo, Japan. His roles there included designing and developing passive circuits such as filters, couplers, diplexers and antennas in the microwave/ millimeter band. In 2001 and 2004, respectively, he completed the Master's and Doctoral Programs of Aoyama Gakuin University Graduate School in Tokyo, Japan, and holds Doctor of Engineering status. In 2004 he took up a position as a lecturer at Hachinohe Institute of Technology in Japan and became an associate professor in 2015. He is currently engaged in research on electric constant measurement for passive components in the high-frequency/ microwave band and the development of a small sensor information mobile Internet communication system using an ARM microcomputers running on Linux.