

SOL Calibration for a Coaxial-Feed-Type Stepped Cut-off Circular Waveguide and Application to Absolute Dielectric Measurement for Liquids Based on the Mode-Matching Technique*

Kouji SHIBATA[†] and Masaki KOBAYASHI[†]

ABSTRACT

In this study, an absolute dielectric evaluation procedure for liquids, including jig S_{11} calibration with no reference materials, was proposed based on cut-off circular waveguide reflection. For this purpose, an open-ended large-bore stepped cut-off circular waveguide with coaxial feed via an N connector, which provides excellent repeatability at low frequencies, was used for the jig. The coaxial tip of a vector network analyzer (VNA) was first calibrated using a commercial SOL (short, open and load) kit, and the commonly used SOL calibration approach for the VNA was also employed for prior jig S_{11} calibration. S_{11} at the front of the jig's coaxial waveguide was measured under SOL (including a matched load) termination conditions. Multiple types of matched termination with different structures were used for this purpose, and theoretical S_{11} values for the open state required for jig's calibration were calculated via mode matching. The jig's S_{11} was then calibrated with no reference liquid. The permittivity was also estimated based on an inverse problem via mode matching from measured S_{11} values with different liquids in the jig after SOL calibration. The results indicated that using the standard matched termination employed for the VNA's SOL calibration in the pre-jig SOL calibration for dielectric evaluation yielded accurate estimates. Permittivity estimated for each liquid after SOL calibration using matched loads of other coaxial waveguide structures significantly differed from the theoretical values, especially at higher frequencies. Based on these results, the jig was SOL calibrated again with different conditions using the measured S_{11} values with each matched termination instead of theoretical S_{11} values. The permittivities for the liquids were then re-estimated based on an inverse problem via the mode-matching technique from the measured S_{11} values after SOL calibration. The results were favorable for the estimated permittivity after jig SOL calibration using all matched terminations. It was then concluded that using high-precision standard matched termination for pre-VNA SOL calibration, or employing measured S_{11} values for matched loads as theoretical values during jig calibration, achieves high-precision absolute dielectric evaluation for liquids. Comparing permittivities estimated based on this absolute measurement with values estimated using a formula involving comparison with SOM termination revealed measurement errors. The proposed technique demonstrates absolute evaluation for small amounts of liquid varied using a convenient, highly accurate measurement method and produces excellent repeatability.

Key Words: Dielectric measurement, Liquid, Impedance measurement, Coaxial line, Cut-off circular waveguide reflection method, Microwave, Radio frequency, S_{11} , calibration, Generalized scattering matrix

* 令和 7 年 1 月 29 日 受付

[†] 工学研究科工学専攻・教授

1. Introduction

In recent years, various methods have been proposed to evaluate complex dielectric properties for liquids [1]. An approach for evaluation of electrolyte solutions with a cut-off circular waveguide fed by a coaxial waveguide was proposed by Bianco in 1979 [2]. The current authors also proposed a valid method using a cut-off circular waveguide fed by a coaxial waveguide via rigorous evaluation based on electromagnetic analysis with mode-matching for an analytical jig model [3]. Various jig calibration methods and a new jig structure for more accurate S_{11} calibration have also been presented [4] – [10]. When a cut-off circular waveguide fed by a coaxial waveguide is used as a jig for this method, input impedance is evaluated with the coaxial tip open. However, in the jig, the outer-conductor inner diameter for the coaxial waveguide of the N connector is the same as that of the inner-conductor outer diameter in the sample holder [2], [3]. As a result, when S_{11} with a metal cylinder in the jig and a short condition is evaluated, the contact state between the flange surface of the connector and the cylinder varies with each evaluation. This causes significant problems with inconsistencies in the values of complex permittivity across multiple evaluations.

Against this background, the authors previously proposed a jig structure with a large-bore coaxial-feed-type stepped cut-off circular waveguide with an N connector [11]. Repeatability in evaluation for the dielectric constant of liquids in the low-frequency MHz range based on the cut-off circular waveguide reflection method was significantly enhanced with this structure. Complex permittivity for each liquid was estimated by substituting the above evaluated S_{11} values into an equation involving comparison with SOM (short/open conditions and a known material) termination. The results were compared with estimated values based on an inverse problem of mode matching using the calibrated S_{11} at the jig's sample front with SOM termination. The results showed good agreement. However, these measurements are performed as relative with comparison to a reference material, with prior S_{11} calibration of the jig. In this case, the discrepancy between the theoretical and measured values of the dielectric constant of the reference material directly affects measurement uncertainty in dielectric constant measurement [4]. Accordingly, relative measurements involve a complex process for determining uncertainty, which hinders standardization.

In this study, an absolute dielectric evaluation method for liquids using a coaxial-feed-type cut-off waveguide was proposed. The method involves no reference materials, even during the prior S_{11} calibration of the jig. An open-ended stepped cut-off circular waveguide fed by a coaxial line with an N connector with excellent repeatability [11] was adopted as the jig. The SOL (short, open and matched-load) calibration method, which is commonly used in VNAs and does not require reference materials, is also applied to the prior S_{11} calibration at the sample front of the jig. A matched load device with the same electrical characteristics as the connector of the measurement jig is required for prior SOL calibration to enable evaluation of the dielectric constant using the coaxial-fed transmission waveguide. In this study, multiple matched terminations with different structures were used to evaluate the impact of their performance differences on calibration. The theoretical values of S_{11} for the analytical model required for calibration with characteristic impedance of coaxial lines other than 50 Ω were calculated using a generalized scattering matrix (GSM) [12], [13]. For S_{11} calibration of jig, the frequency characteristics of the input impedance of each jig structure were initially evaluated after calibration of the coaxial tip of a VNA (vector network analyzer) using a commercially available SOL kit. S_{11} at the coaxial tip of the open-ended circular waveguide with coaxial feeding by N connectors, used for estimation of the dielectric constant of actual liquids, was SOL calibrated using the previously described matched terminations and similar. The theoretical values of S_{11} with the jig in an open state were calculated using mode matching with respect to the jig analysis model. Complex permittivity was then estimated based on an inverse problem via mode matching from the S_{11} measurements with the VNA with each liquid in the jig after SOL calibration. The results showed significant differences, particularly at high frequencies, between the estimated dielectric constants of each liquid after SOL calibration using matched terminations of several coaxial waveguide structures and their theoretical values. Meanwhile, estimated values of complex permittivity that closely matched the theoretical values were observed when the standard matched termination used during pre-SOL calibration of the VNA coaxial tip was subsequently employed in the jig's SOL calibration. Based on these results, the jig used for dielectric evaluation was SOL calibrated at the front end of the coaxial waveguide using the measured S_{11} values of multiple jigs with different coaxial waveguide structures as theoretical values for the matched loads.

Complex permittivity was then estimated again based on an inverse problem via the mode-matching method using the measured S_{11} values with each liquid in the jig after SOL calibration. The estimated values of complex permittivity closely matched the theoretical values when all matched terminations were used in SOL calibration under the conditions of the aforementioned calibration. Accordingly, it was confirmed that accurate absolute dielectric evaluation can be achieved through SOL calibration of the jig preceding the dielectric estimation of various liquids by either: 1. utilizing the standard matched termination during pre-SOL calibration of the VNA, or 2. using the measured S_{11} values of each matched termination as the required theoretical values for calibration. Errors in the estimated values of complex permittivity obtained using the formula involving comparison with SOM termination were revealed through comparison with the complex permittivity obtained in the previous absolute measurements. Thus, convenient, precise and absolute dielectric measurement with excellent repeatability for small amounts of liquid was achieved from the MHz band (low-frequency) based on cut-off circular waveguide reflection.

2. Jig structure and evaluation of solution permittivity

In this study, the dielectric constant of liquids was evaluated using the cut-off circular waveguide reflection method, which involves measuring S_{11} when the electromagnetic field in the sample insertion section is in cut-off mode. In this context, the jig shown in Figure 1 (a) is used for high-precision S_{11} calibration and measurement, especially in the low-frequency band below 100MHz. This jig has an improved structure compared to conventional jigs, providing excellent repeatability and reproducibility. The jig consists of a large-diameter N connector and a metallic sample holder for the sample. In the jig (Fig. 1 (a)), the inner diameter of the sample insertion portion is slightly larger (10.2 mm) than the outer conductor inner diameter (9.80 mm; inner conductor outer diameter: 3.10 mm). The stability of S_{11} evaluation values is greatly improved by ensuring electrical contact with the center conductor and with the flange surface of the connector with a short metal rod inserted. Here, an Orient Microwave CL52-1201-10 was used as the N connector in combination with the sample holder. An absolute measurement method with no reference materials was used for S_{11} calibration and dielectric constant evaluation. The SOL calibration method commonly used for VNA calibration was applied to S_{11} calibration of the measurement jig. However, when calibrating the measurement jig using SOL, it is necessary to use a matched termination that combines connectors with electrical performance equivalent to the N connector used in the jig [14], [15]. Several structures of matched terminations with female connectors were used in matched termination for S_{11} calibration of the jig with the following specifications:

1. Standard matched termination with female connectors used for SOL calibration of the VNA was applied (Figure 2 (a)).
2. 50 Ω matched termination with precision-grade female connectors from Hewlett-Packard (HP) was used (Figure 2 (a)).
3. A device with two N connectors constituting the jig for dielectric evaluation facing each other was used, with standard termination using male connectors on the load side (Figure 2 (b)).
4. A device with a standard load and male connectors was connected to the load side of a finite-length coaxial waveguide with the same inner and outer diameters, fed by N connectors constituting the jig for dielectric evaluation (Figure 2 (c)).
5. A device with standard termination and male connectors was connected to the load side of a finite-length coaxial waveguide with dimensions slightly larger than the outer diameter of the N connector fed by the N connectors constituting the jig for dielectric evaluation (Figure 2(c)).

The internal structures of each matched termination are shown in Figure 2. Actual photographs of each matched termination used are presented in Figure 5 of Chapter 3. Here, the following were used as calibration kits for the high-precision type matched termination shown in Figure 2 (a):

1. 50 Ω impedance standard termination with female connectors from a Keysight 85032F standard mechanical calibration kit, used for SOL calibration of S_{11} for the reference plane of the VNA
2. The Hewlett Packard 909C-Opt.013, a precision-grade 50 Ω impedance standard load with female connectors, was used.

The device shown in Figure 2 (b) was fabricated using the following procedure:

1. The center conductor and PTFE at the tip of the Orient Microwave CL52-1201-10 N connector used for the jig in dielectric evaluation with the cut-off circular waveguide reflection method were cut at the flange plane and filed.
2. The flange surfaces of the two connectors were screwed together.
3. Matched termination with female connectors was achieved by connecting the 50 Ω impedance standard device with male connectors from a Keysight 85032F standard mechanical calibration kit to one side of the aforementioned components.

The device in Figure 2 (c) was constructed by connecting the standard matched termination with male connectors from the Keysight 85032F standard kit to the load side of a finite-length coaxial line with the same inner and outer diameters, sandwiched between the two N connectors (CL52-1201-10) used in the jig for dielectric evaluation. The device in Figure 2 (d) was constructed by connecting the standard matched termination with male connectors from the Keysight's 85032F standard kit to the load side of a finite-length coaxial line with dimensions slightly larger (ϕ 10.20) than the outer diameter (ϕ 9.80), sandwiched between the two N connectors (CL52-1201-10) used in the jig for dielectric evaluation.

S_{11} calibration with the jig using the matched termination evaluated in this study and the method for evaluation of liquid dielectric constants are described below.

1. The S_{11} of the coaxial wave tip (Ref. 1) of the VNA before jig connection is typically SOL-calibrated using a standard N connector kit.
2. After each matched termination in Figure 2 is mounted on the coaxial wave tip (Ref. 1) of the VNA, S_{11} for the SOL calibration plane is measured.
3. The aforementioned matched terminations are removed, and the jig of the cut-off circular waveguide shown in Figure 1 (a) is mounted.
4. The S_{11} value for the SOL calibration plane (Ref. 1) is evaluated with the coaxial line front of the jig shorted (Fig. 1 (b)) in an open state (Fig. 1 (a)) and with a reference material and an unknown material inserted.
5. S_{11} at the sample front of the jig (Ref. 2) is calibrated by substituting the measured values of S_{11} for the SOL calibration plane (Ref. 1) for each of the above termination conditions and the theoretical value of the SOL termination for Ref. 2 into Equation (1).
6. The complex permittivity of the unknown liquid is estimated based on an inverse problem via mode matching from the S_{11} calibrated for Ref. 2 using the above procedure.

Pure water, methanol and ethanol were used as the liquids for which the dielectric constant was evaluated. S_{11} calibration of the evaluation jig for the reference plane is needed using SOL (short, open and load) impedance standards for absolute evaluation of permittivity. The measured values for Ref. 1 and the theoretical values for Ref. 2 are substituted into the formula for S_{11} calibration of the jig. Here, the theoretical value of the reflection coefficient for a short condition was substituted with $\Gamma_1 = -1 - j0$ at each frequency. Calculation of the theoretical reflection constant for an open condition requires consideration of the fringing capacitance at the jig's coaxial tip. The reflection constant in an open condition for the jig's analysis model

was calculated at each frequency using mode matching [7]. The transmission lines of the structures shown in Figures 2 (a) –(c) exhibit theoretically no discontinuities. The theoretical value for the load (matched termination) was substituted as $\Gamma_3 = 0.0 - j0$.

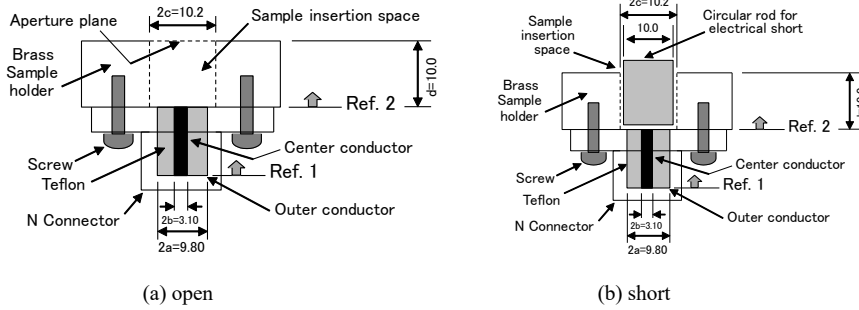
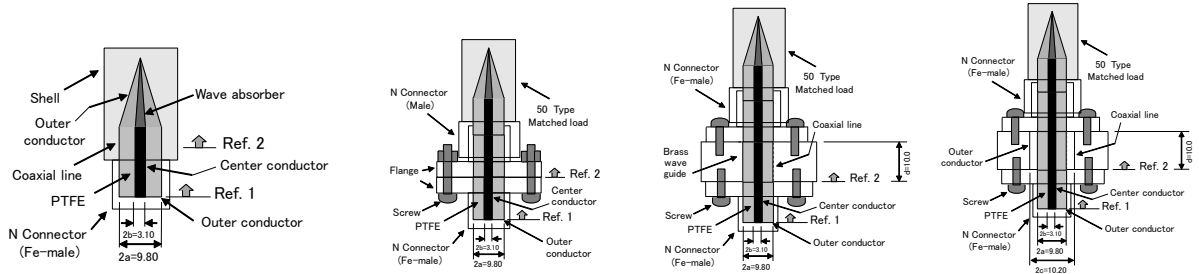


Figure 1. S_{11} calibration for a coaxial-feed-type open-ended cut-off circular waveguide with an N- connector



(a) Standard load of female connector (b) N connector and standard load (c) $\phi 9.8$ mm coaxial waveguide (d) $\phi 10.2$ mm coaxial waveguide

Figure 2. Internal structure of the matching termination set-up used for S_{11} calibration under a load condition

The outer conductor inner diameter of the coaxial line constituting the matching load in Figure 2 (d) is slightly larger at $\phi 10.2$ mm compared to $\phi 9.80$ mm in Figures (b) and (c). In this structure, the PTFE outer diameter is slightly smaller at $\phi 9.8$ mm compared to the outer conductor of the coaxial waveguide. Accordingly, the structure has discontinuities within the transmission line (with characteristic impedance differing from 50Ω in some sections). Theoretical values of the S-matrix for the finite-length transmission lines with the aforementioned discontinuities were calculated based on a generalized scattering matrix (GSM) [12], [13]. The characteristic impedance part in regions 1 and 2 of the device, where a coaxial waveguide with a length of 10.0 mm and a dielectric two-layer structure is connected to the input and output transmission lines as shown in Figure 3, is first calculated using:

$$Z_1 = Z_3 = \frac{\eta_0}{2\pi} \cdot \frac{1}{\sqrt{\epsilon_{r1}}} \cdot \ln\left(\frac{b}{a}\right) \quad (1) \quad Z_2 = \frac{\eta_0}{2\pi} \sqrt{\ln\left(\frac{c}{a}\right) \cdot \left[\frac{\ln\left(\frac{c}{b}\right)}{\epsilon_{r2_bc}} + \frac{\ln\left(\frac{b}{a}\right)}{\epsilon_{r2_ab}} \right]} \quad (2)$$

The propagation constant and characteristic impedance of the free space in region 2 are calculated using:

$$\gamma_2 = \omega \sqrt{\frac{\epsilon_0 \cdot \mu_0 \cdot \epsilon_{r_ab} \cdot \epsilon_{r_bc} \cdot \ln\left(\frac{c}{a}\right)}{\epsilon_{r_ab} \cdot \ln\left(\frac{c}{b}\right) + \epsilon_{r_bc} \cdot \ln\left(\frac{b}{a}\right)}} \quad (3) \quad \eta_0 = \sqrt{\frac{\mu_0}{\epsilon_0}} \quad (4)$$

Here, $\omega = 2\pi f$, and frequency is substituted for f [Hz]. The reflection constant in region 1 is assigned as Γ_1 , and that in region 2 is assigned as Γ_2 . Accordingly, the following are calculated from the relationship known as $S_{11} = \Gamma_1^2$, $S_{21} = 1 - S_{11} = 1 - \Gamma_1^2$, $S_{22} = \Gamma_2^2$, $S_{12} = 1 - S_{22} = 1 - \Gamma_2^2$:

$$\mathbf{S}_{11} = \Gamma_1^2 = \left(\frac{Z_1 - Z_2}{Z_1 + Z_2} \right)^2 \quad (5) \quad , \quad \mathbf{S}_{22} = \Gamma_2^2 = \left(\frac{Z_2 - Z_1}{Z_1 + Z_2} \right)^2 \quad (6)$$

$$\mathbf{S}_{21} = 1 - \Gamma_1^2 = 1 - \mathbf{S}_{11} = 1 - \left(\frac{Z_1 - Z_2}{Z_1 + Z_2} \right)^2 \quad (7) \quad , \quad \mathbf{S}_{12} = 1 - \Gamma_2^2 = 1 - \mathbf{S}_{22} = 1 - \left(\frac{Z_2 - Z_1}{Z_1 + Z_2} \right)^2 \quad (8)$$

The S-matrix of the finite-length coaxial line sandwiched between two discontinuities is calculated using the \mathbf{S}_{11} of the aforementioned discontinuities and the GSM method [12], [13].

The finite-length transmission line in Region 2 is defined by matrix \mathbf{D} :

$$\mathbf{D} = e^{-j\gamma_2 \cdot d} \quad (9)$$

The analytical model in Figure 3 has a structure that is symmetrical in the transmission direction.

The total S-matrix for the analytical model is calculated using Eqs. (5) to (8) representing the S-matrix for the discontinuity for Ref. in Fig. 3, along with Eqs. (10) and (11) below [12], [13].

$$\mathbf{S}_{T_{11}} = \mathbf{S}_{T_{22}} = \mathbf{S}_{11} + \mathbf{S}_{12} \cdot \mathbf{D} \cdot [\mathbf{I} - \mathbf{S}_{22} \cdot \mathbf{D} \cdot \mathbf{S}_{22} \cdot \mathbf{D}]^{-1} \cdot \mathbf{S}_{22} \cdot \mathbf{D} \cdot \mathbf{S}_{21} \quad (10)$$

$$\mathbf{S}_{T_{21}} = \mathbf{S}_{T_{12}} = \mathbf{S}_{12} \cdot \mathbf{D} \cdot [\mathbf{I} - \mathbf{S}_{22} \cdot \mathbf{D} \cdot \mathbf{S}_{22} \cdot \mathbf{D}]^{-1} \cdot \mathbf{S}_{21} \quad (11)$$

\mathbf{I} is the identity matrix. The S-parameters for the structures in Figure 2 (d) and Figure 3 were calculated under the conditions of $a = 3.1$ mm, $b = 9.8$ mm, $c = 10.2$ mm, $d = 10.0$ mm, $\epsilon_1 = 2.05$, $\epsilon_{2_ab} = 2.05$, $\epsilon_{2_bc} = 1.00$, and $\mu_1 = \mu_2 = 1.00$.

As a result, the S-matrix was calculated to be $|\mathbf{S}_{T11}| = 1.324 \cdot 10^{-3}$, $|\mathbf{S}_{T21}| = 0.9987$ at 200MHz. The above \mathbf{S}_{11} was calculated to be $10 \cdot \log|\mathbf{S}_{T11}| = -28.780$ [dB].

A deterioration of approximately 3 dB in \mathbf{S}_{11} was observed compared to the \mathbf{S}_{11} of the individual discontinuity. The calculated results are considered valid. The above \mathbf{S}_{11} was also converted to input impedance. The values for the structures in Figure 2 (d) and Figure 3 were calculated to be $Z = 50.133\Omega$. As a result, the structure was evaluated to have good VSWR characteristics.

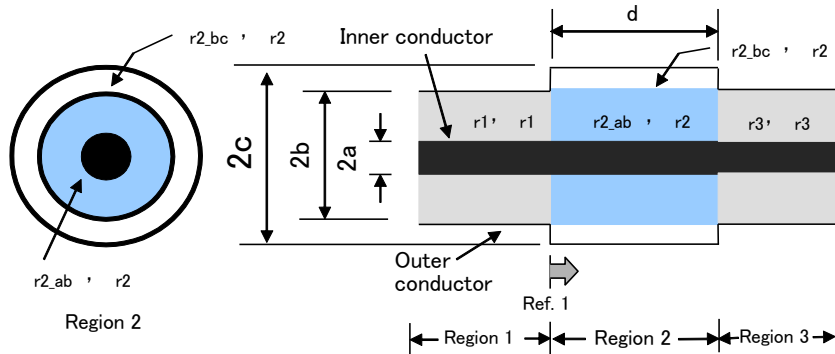


Figure 3. Coaxial line with finite-length discontinuities

3. Evaluation of jig input impedance and complex permittivity

Jig input impedance was evaluated using a Copper Mountain Technologies R60 single-port VNA. The coaxial line front end of the VNA was calibrated before jig attachment using an SOL kit with N connectors in the frequency range of 10 – 1,000MHz. Subsequently, input impedance was evaluated using each matched termination in Figures 2 and 5 with attachment to the VNA. The above matched terminations were removed from the VNA.

Input impedance for each termination condition for the SOL calibration plane (Ref. 1) was evaluated with the coaxial top of the jig short and open, and with pure water, methanol and ethanol at room temperature (25.0°C) in the jig using the R60 VNA with the jig shown in Figure 4 (c) attached. Impedance measurement using the one-port VNA with the jig attached is shown in Figure 4.

During S_{11} evaluation for the short condition, the screw was tightened using an auxiliary jig (Fig. 4 (b)). Pressure applied from above with a short brass rod (Fig. 1 (b)) ensured firm contact with the N connector flange. Contact between the outer conductor and the center conductor at the open end of the coaxial waveguide was ensured. Input impedance at the Ref. 2 plane of the jig was thus calibrated using the above measured S_{11} values with short, open and loaded (matched termination) conditions.

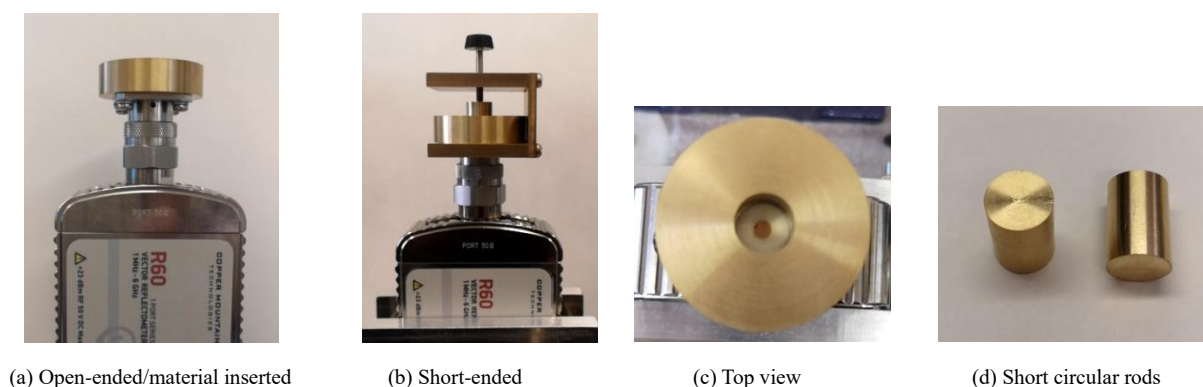


Figure 4. Copper Mountain R60 VNA for impedance data acquisition

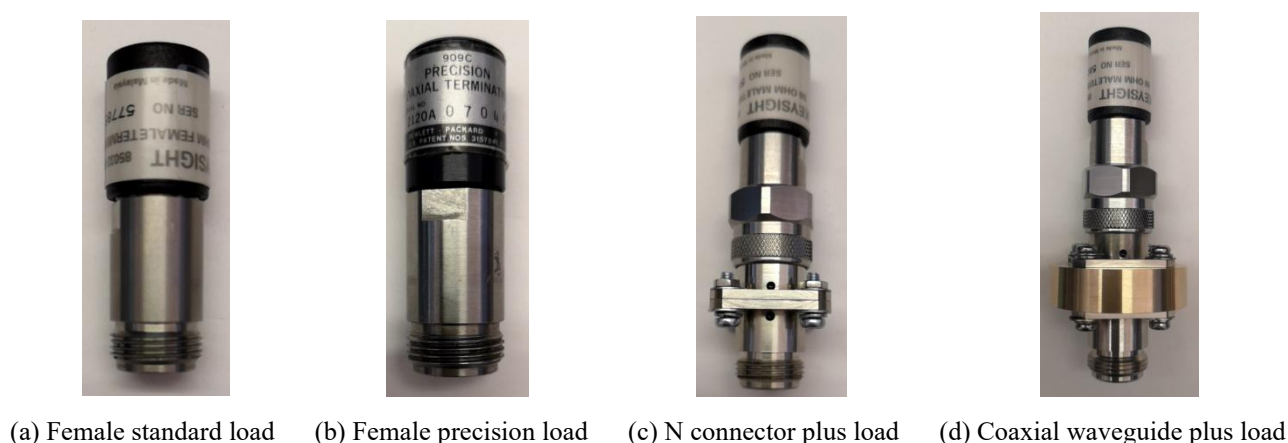


Figure 5. Matched termination for load calibration

The results showed that the measured input impedance of the load after calibration perfectly matched the theoretical value with the Keysight 85032F standard mechanical calibration kit (a standard load calibrator with female connectors used for VNA SOL calibration) and a Hewlett Packard 909C-Opt.013 precision-grade 50Ω impedance standard with female connectors for matched termination.

The complex permittivity of pure water, methanol and ethanol was estimated as an inverse problem based on mode matching from the input impedance for Ref. 2 of the jig when each liquid was inserted after calibration using the matched termination of each structure mentioned above. The estimated values were also compared with the complex permittivity of each liquid calculated using a formula involving comparison with SOM termination. The results are shown in Tables 1 to 5.

The complex permittivity obtained using mode matching was estimated from the input impedance of the jig calibrated for Ref. 2, based on comparison between the measured values for Ref. 1 using five types of matched terminations (two of which are precision-grade 50 Ω impedance standards) and the theoretical value of the load condition. As an example, the results in Tables 1 (a) and 2 (a) for pure water were obtained using the precision-grade 50 Ω impedance standard with the female connectors of Keysight's 85032F standard mechanical calibration kit and a Hewlett Packard 909C-Opt.013 precision-grade 50 Ω impedance standard with female connectors during initial jig SOL calibration.

The complex permittivity estimated as an inverse problem based on mode-matching from the calibrated S_{11} for Ref. 2, based on comparison between the measured values for Ref. 1 and the theoretical values for Ref. 2, was compared with the estimated values obtained using the formula involving comparison with reference materials after SOL calibration for Ref. 2 using the above procedure.

Values for both the real and imaginary parts of estimated complex permittivity were reasonable.

Values closely matching the theoretical ones were thus observed when the standard matched termination used during pre-SOL calibration of the VNA coaxial tip was subsequently employed in calibration.

However, abnormal results were obtained where relative permittivity increased with frequency as shown in Tables 4 (a) to 5 (a) in each of the following conditions:

1. A jig with two female N connectors for dielectric measurement facing each other, with a standard load calibrator and a male connector connected to the load side as shown in Fig. 4 (b).
2. A jig with two female N connectors for dielectric measurement attached at both ends of a coaxial waveguide of finite length with the same dimensions for the inner and outer diameters, with a standard load calibrator and male connectors connected to the load side as shown in Fig. 4 (c).
3. A jig with a coaxial waveguide of finite length and dimensions slightly larger than the outer diameter of the coaxial waveguide fed by two female N connectors used for dielectric measurement jigs, with a standard load calibrator and male connectors connected to the load side as shown in Fig. 4 (d).

The estimated values of complex permittivity for each liquid using the formula involving comparison with SOM after SOL calibration using the above procedure were consistent, even with five different matching terminations.

The reason for these results is that the complex permittivity of the unknown material estimated using the formula involving comparison with SOM termination is simply determined based on the measured input impedance values of short, open and reference materials, regardless of the difference in reference planes.

Table 1. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the Keysight standard in Figure 5 (a) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.03	78.55	+0.611	0.79	0.038	+1978.9
100	79.57	78.55	+1.299	0.50	0.383	+30.548
1000	79.11	78.35	+0.970	4.05	3.816	+6.132

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.02	32.92	+0.304	0.94	0.616	+52.597
100	33.78	33.44	+1.017	0.95	0.884	+7.466
1000	31.57	30.38	+3.917	7.85	7.493	+4.764

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.80	24.76	+0.162	0.52	0.279	+86.380
100	25.41	25.19	+0.873	1.91	1.837	+3.974
1000	16.47	15.77	+4.439	10.40	9.866	+5.413

Table 2. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) with HP's precision 50 Ω load in Figure 5 (b) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.31	78.55	+0.968	0.79	0.038	+1978.9
100	79.86	78.55	+1.668	0.50	0.383	+30.548
1000	79.31	78.35	+1.225	4.18	3.816	+9.539

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.14	32.92	+0.668	0.94	0.616	+52.597
100	33.90	33.44	+1.376	0.96	0.884	+8.597
1000	31.65	30.38	+4.180	7.92	7.493	+5.699

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.88	24.76	+0.485	0.52	0.279	+86.380
100	25.50	25.20	+1.190	1.91	1.836	+4.031
1000	16.50	15.77	+4.629	10.45	9.866	+5.919

Table 3. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (b) and Figure 5 (c) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.06	78.55	+0.649	0.74	0.038	+1847.4
100	79.67	78.55	+1.426	0.12	0.383	-68.668
1000	80.13	78.36	+2.259	1.86	3.816	-51.258

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.03	32.92	+0.334	0.91	0.616	+47.727
100	33.83	33.44	+1.166	0.80	0.884	-9.502
1000	32.17	30.38	+5.892	7.03	7.493	-6.179

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.81	24.76	+0.202	0.50	0.279	+79.211
100	25.45	25.20	+0.992	1.79	1.836	-2.505
1000	16.97	15.77	+7.609	10.06	9.866	+1.966

Table 4. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (c) and Figure 5 (d) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.07	78.55	+0.662	0.75	0.038	+1873.7
100	79.68	78.55	+1.439	0.29	0.383	-24.282
1000	79.49	78.35	+1.455	3.66	3.816	-4.088

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.04	32.92	+0.365	0.92	0.616	+49.351
100	33.83	33.44	+1.166	0.86	0.884	-2.715
1000	31.76	30.38	+4.542	7.72	7.493	+3.029

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.81	24.76	+0.202	0.51	0.279	+82.796
100	25.45	25.20	+0.992	1.84	1.836	+0.218
1000	16.60	15.77	+5.263	10.37	9.866	+5.108

Table 5. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (d) and Figure 5 (d) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.78	78.55	+1.566	0.76	0.038	+1900.0
100	80.39	78.55	+2.322	0.32	0.383	-16.449
1000	80.20	78.35	+2.361	3.34	3.816	-12.474

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.32	32.92	+1.215	0.93	0.616	+50.974
100	34.13	33.44	+2.063	0.88	0.884	-0.452
1000	32.08	30.38	+5.596	7.64	7.493	+1.962

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	25.02	24.76	+1.050	0.51	0.279	+82.796
100	25.67	25.20	+1.865	1.87	1.836	+1.852
1000	16.80	15.77	+6.531	10.39	9.866	+5.311

Based on these results, S_{11} at the coaxial tip (Ref. 2) of the jig was re-calibrated using SOL termination, replacing the theoretical values for loading (matched termination) with measured values under all four matched termination conditions of Figure (2) and Figure (5). The complex permittivity of each liquid was then estimated as an inverse problem using the mode-matching technique from the measured input impedance with each liquid in the jig after the above re-calibration.

The estimated values of complex permittivity, along with those based on the formula, are shown in Tables 6 to 8. As an example, for pure water, the complex permittivity estimated after calibration with all types of matched terminations mentioned above was found to perfectly match both the real and imaginary parts of that estimated based on an inverse problem, with the mode-matching technique from the calibrated S_{11} for Ref. 2 using the measured value for Ref. 1 with Keysight's precision 50 Ω input impedance standard of the 85032F standard mechanical calibration kit or Keysight's male connector using the Hewlett Packard 909C-Opt.013 and theoretical S_{11} values for Ref. 2 for load (matched termination) condition. The estimated permittivity values of all liquids based on the formula involving comparison with SOM termination as shown in Tables 6 to 8 perfectly matched, as shown in Tables 1 to 4. The reason for the good agreement is that the complex permittivity of the unknown material based on the formula involving comparison with SOM termination is simply determined from measured input impedance values of short, open and reference materials, regardless of the difference in reference planes. These results indicated that:

1. S_{11} can be calibrated at the front of the sample (Ref. 2) in the jig using SOL (short, open and load) termination conditions without the need for reference materials, using a coaxial-feed-type stepped cut-off circular waveguide and the high-precision 50 Ω impedance standard used for S_{11} calibration of the VNA as matched termination (load condition).
2. From the measured S_{11} values for Ref. 2 after calibration using the above procedure, the complex permittivity of each liquid can be absolutely evaluated as an inverse problem of the mode-matching technique without comparison to reference materials.

Differences were revealed by comparing the complex permittivity estimated using the above procedure with values determined using the formula involving comparison with SOM termination. Thus, this is confirmed as convenient, precise and absolute dielectric evaluation with excellent repeatability for small amounts of liquids from the MHz band (low-frequency) based on cut-off circular waveguide reflection. In the future, it will be necessary to evaluate the complex permittivity of electrolyte solutions with high conductivity in the low-frequency range using the proposed method.

Table 6. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (b) and Figure 5 (c) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.03	78.55	+0.611	0.79	0.038	+1978.9
100	79.57	78.55	+1.299	0.50	0.383	+30.548
1000	79.09	78.36	+0.932	4.04	3.816	+5.870

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.02	32.92	+0.304	0.94	0.616	+52.597
100	33.78	33.44	+1.017	0.95	0.884	+7.466
1000	31.57	30.38	+3.917	7.84	7.493	+4.631

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.80	24.76	+0.162	0.52	0.279	+86.380
100	25.41	25.20	+0.833	1.91	1.836	+4.031
1000	16.47	15.77	+4.439	10.40	9.866	+5.413

Table 7. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (c) and Figure 5 (d) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.03	78.55	+0.611	0.79	0.038	+1978.9
100	79.57	78.55	+1.299	0.50	0.383	+30.548
1000	79.09	78.35	+0.944	4.06	3.816	+6.394

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.02	32.92	+0.304	0.94	0.616	+52.597
100	33.78	33.44	+1.017	0.95	0.884	+7.466
1000	31.57	30.38	+3.917	7.85	7.493	+4.764

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.80	24.76	+0.162	0.52	0.279	+86.380
100	25.41	25.20	+8.333	1.91	1.836	+4.031
1000	16.47	15.77	+4.439	10.40	9.866	+5.413

Table 8. Complex permittivity of each liquid estimated after SOL calibration at the coaxial waveguide tip (Ref. 2) of the jig in Figure 1 (d) and Figure 5 (d) (25.0°C)

(a) Pure water

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	79.03	78.55	+0.611	0.79	0.038	+1978.9
100	79.57	78.55	+1.299	0.50	0.383	+30.548
1000	79.06	78.35	+0.906	4.04	3.816	+5.870

(b) Methanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	33.02	32.92	+0.304	0.94	0.616	+52.597
100	33.78	33.44	+1.017	0.95	0.884	+7.466
1000	31.55	30.38	+3.851	7.84	7.493	+4.631

(c) Ethanol

Frequency [MHz]	Real			Imaginary		
	Mode-matching	Formula involving comparison with the reference material	Difference	Mode-matching	Formula involving comparison with the reference material	Difference
10	24.80	24.76	+0.162	0.52	0.279	+86.380
100	25.41	25.20	+0.833	1.91	1.836	+4.031
1000	16.46	15.77	+4.375	10.39	9.866	+5.311

4. Conclusion

In this study, an absolute dielectric evaluation method for liquids using a coaxial-feed-type cut-off waveguide was proposed. The method involves no reference materials, even during the prior S_{11} calibration of the jig. An open-ended stepped cut-off circular waveguide fed by a coaxial line with an N connector with excellent repeatability was adopted as the jig. The SOL (short, open and matched-load) calibration method, which is commonly used in VNAs and does not require reference materials, is also applied to the prior S_{11} calibration at the sample front of the jig. A matched load device with the same electrical characteristics as the connector of the measurement jig is required for prior SOL calibration to enable evaluation of the dielectric constant using the coaxial-fed transmission waveguide. In this study, multiple matched terminations with different structures were used to evaluate the impact of their performance differences on calibration. The theoretical values of S_{11} for the analytical model required for calibration with characteristic impedance of coaxial lines other than 50 Ω were calculated using a generalized scattering matrix (GSM). For S_{11} calibration of jig, the frequency characteristics of the input impedance of each jig structure were initially evaluated after calibration of the coaxial tip of a VNA (vector network analyzer) using a commercially available SOL kit. S_{11} at the coaxial tip of the open-ended circular waveguide with coaxial feeding by N connectors, used for estimation of the dielectric constant of actual liquids, was SOL calibrated using the previously described matched terminations and similar. The theoretical values of S_{11} with the jig in an open state were calculated using mode matching with respect to the jig analysis model. Complex permittivity was then estimated based on an inverse problem via mode matching from the S_{11} measurements with the VNA with each liquid in the jig after SOL calibration. The results showed significant differences, particularly at high frequencies, between the estimated dielectric constants of each liquid after SOL calibration using matched terminations of several coaxial waveguide structures and their theoretical values. Meanwhile, estimated values of complex permittivity that closely matched the theoretical values were observed when the standard matched termination used during pre-SOL calibration of the VNA coaxial tip was subsequently employed in the jig's SOL calibration. Based on these results, the jig used for dielectric evaluation was SOL calibrated at the front end of the coaxial waveguide using the measured S_{11} values of multiple jigs with different coaxial waveguide structures as theoretical values for the matched loads.

Complex permittivity was then estimated again based on an inverse problem via the mode-matching

method using the measured S_{11} values with each liquid in the jig after SOL calibration. The estimated values of complex permittivity closely matched the theoretical values when all matched terminations were used in SOL calibration under the conditions of the aforementioned calibration. Accordingly, it was confirmed that accurate absolute dielectric evaluation can be achieved through SOL calibration of the jig preceding the dielectric estimation of various liquids by either: 1. utilizing the standard matched termination during pre-SOL calibration of the VNA, or 2. using the measured S_{11} values of each matched termination as the required theoretical values for calibration. Errors in the estimated values of complex permittivity obtained using the formula involving comparison with SOM termination were revealed through comparison with the complex permittivity obtained in the previous absolute measurements. Thus, convenient, precise and absolute dielectric measurement with excellent repeatability for small amounts of liquid was achieved from the MHz band (low-frequency) based on cut-off circular waveguide reflection.

Acknowledgements

This work was partly supported by a Japan Society for the Promotion of Science (JSPS) Kakenhi Grant (no. 20K04522) for the work titled Establishment of a Broadband Dielectric Measurement Method for Liquids in Temperature-Change Environments for Synthesis of Functional Materials.

References

- [1] Valerica Raicu and Yuri Feldman, "Dielectric Relaxation in Biological Systems: Physical Principles, Methods, and Applications," <https://academic.oup.com/book/27912> ISBN: 9780199686513.
- [2] B. Bianco, G. P. Drago, M. Marchesi, C. Martini, G. S. Mela and S. Ridella, "Measurements of Complex Dielectric Constant of Human Sera and Erythrocytes," IEEE Transactions on Instrumentation and Measurement, Vol. 28, No. 4, pp. 290-295, Dec. 1979, doi: 10.1109/TIM.1979.4314834.
- [3] K. Shibata, "Measurement of Complex Permittivity for Liquid Materials Using the Open-ended Cut-off Waveguide Reflection Method," IEICE Trans. Electron., Vol. E93-C, No. 11, pp. 1,621-1,629, 2010-11.
- [4] K. Shibata and M. Kobayashi, "Simplification of Liquid Dielectric Property Evaluation Based on Comparison with Reference Materials and Electromagnetic Analysis Using the Cut-off Waveguide Reflection Method," IEICE Trans. Electron., Vol. E100-C, No. 10, pp. 908-917, 2017-10.
- [5] K. Shibata, "Dielectric Measurement in Liquids Using an Estimation Equation without Short Termination via the Cut-Off Circular Waveguide Reflection Method," IEICE Trans. Electron., Vol. E101-C, No.8, pp. 627-636, 2018-8.
- [6] K. Shibata, "S11 Calibration of a Cut-off Circular Waveguide with Three Materials and Related Application to Dielectric Measurement for Liquids," IEICE Trans. Electron., Vol. E104-C, No.2, pp. 93- 101, 2021-2.
- [7] K. Shibata, "Calibration of a Coaxial-Loaded Stepped Cut-Off Circular Waveguide and Related Application of Dielectric Measurement for Liquids," IEICE Trans. Electron., Vol. E105-C, No. 4, pp. 163-171, 2022-4.
- [8] K. Shibata, "A Simplified Procedure for Dielectric Measurement of Liquids, Including Calibration via the Cut-Off Circular Waveguide Reflection Method," Proc. of 2023 IEEE Sensors Applications Symposium (IEEE SAS 2023), Ottawa, ON, Canada, 2023-7.
- [9] K. Shibata, E. Otani, J. Han, Y. Umebayashi and K. Fujiwara, "Dielectric Evaluation of Ionic Electrolyte Solutions Based on the Cut-off Waveguide Reflection Method," Proc. of the 14th International Conference on Electromagnetic Wave Interaction with Water and Moist Substances (ISEMA 2023), Brisbane, Australia, 2023-9.
- [10] K. Shibata, "Dielectric Measurement of Liquids Based on Comparison with a Solid Reference Material via the Cut-off Circular Waveguide Reflection Method," IEICE Tech. Rep., Vol. 123, No. 5, MW2023-3, pp. 10-15, 2023-4.

- [11] K. Shibata and M. Kobayashi, "Dielectric Evaluation for Liquids from the MHz Band using a Coaxial-Feed Type Large Bore Stepped Cut-off Circular Waveguide with Good Repeatability," IEICE Tech. Rep., vol. 124, no. 92, EMCJ2024-7, pp. 1 – 5, 2024-6.
- [12] Jaroslav Uher, Jens Bornemann and Uwe Rosenberg, "Waveguide Components for Antenna Feed Systems: Theory and CAD," Artech House, 1993. ISBN: 9780890065822.
- [13] M. Miyazaki, H. Yukawa, T. Nishino, S. Urasaki, T. Katagi and H. Kurebayashi, "Design of an Iris-Coupled Broadband Waveguide Filter Using Modified Reflection-Zero Frequencies," IEICE Trans Electron, Vol. J81-C-I, No. 11, pp. 642-649, 1998-11.
- [14] K. Shibata, "S11 Calibration for a Coaxial-loaded Cut-off Circular Waveguide with SOL (short, open and load) Termination and Related Application to Dielectric Measurement in Liquids," IEICE Tech. Rep., Vol. 120, No. 54, MW2020-13, pp. 11-16, 2024-6.
- [15] K. Shibata, "Calibration for a Coaxial-loaded Cut-off Circular Waveguide with SOL Termination, and Related Application to Dielectric Measurement for Liquids," Bulletin of the Hachinohe Institute of Technology, No. 43, pp. 41-53, 2024-4.

要 旨

本報告書では、遮断円筒導波管導波管反射法による液体の誘電率推定に対し、治具の S_{11} の校正も含め基準物質を用いない絶対測定法を提案する。その為の治具に低周波帯で繰り返し再現性に優れる、N コネクタの同軸給電による終端開放段差付遮断円筒導波管、事前の S_{11} の校正に VNA 等で一般的な SOL (short, open, load) 校正法を用いる。そしてまず、VNA の同軸端を市販の SOL キットで校正後、事前に用意した複数の整合終端と治具にて治具の同軸線路の前面の S_{11} を SOL 校正した。その際、open の S_{11} の理論値はモード整合法で計算した。次に、SOL 校正後の治具に各液体を挿入時の S_{11} の測定値からモード整合法の逆問題で誘電率を推定した。結果、事前の治具の SOL 校正に VNA の SOL 校正に用いた標準 Load 校正器を用いれば、良好な推定値を得ることを確認した。一方、他の同軸線路構造の整合終端で SOL 校正後に推定した各液体の誘電率は、特に高い周波数で理論値と大きく異なった。そこで、整合終端の理論値に先の各整合終端の S_{11} の測定値を用い治具を SOL 校正後に再度、 S_{11} の測定値からモード整合法の逆問題で誘電率を推定した結果、何れの条件でも良好な結果を得た。従い、液体の誘電率推定で事前の治具の SOL 校正に標準 Load 校正器を用いるか、治具を校正時の理論値に整合終端の S_{11} の測定値を用いれば、高精度な誘電率の絶対測定が実現される事を確認した。更に、この絶対測定による誘電率を SOM と比較する公式での推定値と比較し誤差も明らかにした。結果、提案する治具と測定法での、繰り返し再現性に優れる少量の簡便かつ高精度な絶対測定での液体の低周波帯からの誘電率測定法が遮断円筒導波管反射法にて確立された。

キーワード : 誘電率測定, 液体, インピーダンス測定, 同軸線路, 遮断円筒導波管反射法, マイクロ波, 高周波, S_{11} , 校正, モード整合法