# Measurement of Complex Permittivity in the UHF Band Using a Perturbed Cavity Resonator without Sample Insertion Holes \*

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#### Synopsis

The standard method of measuring complex permittivity ( $\varepsilon$ ) with a perturbed cavity resonator at microwave frequencies inevitably involved loading and unloading a sample through an insertion hole. This method is used because of the simplicity of the sample insertion process as specified by JIS (Japanese Industrial Standards) and ASTM (American Society for Testing Materials). However, there is a strong possibility that sample insertion holes cause undesirable effects during actual measurements, especially in the UHF band where the height of the resonator is not as great as that of the SHF band. In this reason, we proposed that a resonator without the sample insertion holes is necessary for accurate permittivity measurements, thus some 20 years ago we proposed a simple cavity resonator design without sample insertion holes for the X band<sup>1)2</sup>.

Recently, the cavity perturbation method has been reevaluated because the measurement process and data reduction are simple. With recent research of cavity perturbation method, the accuracy of the measurements becomes better, and also the limitation of sample size and magnitude of permittivity have become clear through rigorous theoretical analysis of cavity resonance mode. In order to obtain high accuracy data, it is desirable to measure under the simple boundary condition which is same as a capacitor at low frequencies, so that we must make use of the resonator which does not have insertion holes.

We think that there is no better method for measuring complex dielectric constant of low loss materials at UHF band. We have now constructed a new and improved perturbed resonator operating in the UHF band (1 GHz and 2.34GHz) and present some measurement examples using this equipment.

### 1. Introduction

In order to determine electrical and magnetic parameters, such as complex permittivity ( $\mathcal{E} = \mathcal{E}' - j\mathcal{E}''$ ) and permeability ( $\mu = \mu' - j\mu''$ ), a simple and accurate measurement technique is required. In the microwave region, the perturbed resonator method<sup>3)-9)</sup> for separately measuring complex permittivity and permeability is widely used.

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In the case of nonmagnetic dielectric materials, there are many measurement methods<sup>10)</sup>. The standing wave (S-parameter) method <sup>11)12)</sup> has commonly been used because of its capability for broadband measurements. However, this method cannot be applied with low-loss dielectric materials. Recently, measurements of complex permittivity in low loss materials having large dielectric constants have been conducted using the dielectric resonator method<sup>13)14)</sup>. In this method, the sample itself is a resonator, and precision disk samples and standard materials can be prepared for precise measurements<sup>15)</sup>. Although the cavity perturbation method is very simple, we had to use a small sample so that the perturbation formula would hold. For example, the diameter of rod samples must be less than 1-1.5 mm for the X band<sup>1) 2)</sup>. However, with the UHF band, limits on the sample size become less strict making this method more effective.

If it were possible to perform measurements at microwave frequencies using capacitors, as is done at low frequencies (Fig. 1), a simple and reliable measurement method could be developed. For example, if a dielectric rod or stick sample is positioned so that only the electric field of the cavity resonator exists, the effect is identical to that of a capacitor, as shown in Fig. 1. The measurement of complex permittivity in the microwave region using a perturbed resonator is an application of this theory.

# Measurement of $\boldsymbol{\varepsilon}$ using a capacitor at *low frequencies*



The changes of complex admittances ( *capacitance* C *and* Q *of the capacitor* ) are measured with and without sample by a impedance meter or a Q-meter with LC-Resonator Circuit.

Fig. 1. Principles of measuring at low frequencies using a capacitor. The measurement formula to obtain complex permittivity is same as perturbation formula of cavity perturbation method.

A conventional perturbed resonator for measurement at microwave frequencies has a hole for insertion of a long bar-shaped sample as specified by JIS<sup>16)</sup> and ASTM<sup>17)</sup>. This is noted in Fig. 2 which shows the introduction of a bar-shaped sample all the way through the resonator. The insertion holes correspond to elimination of the original electrodes used in low-frequency capacitor measurements as shown in Fig. 1. Therefore, when compared with the case where no insertion hole is provided, it is expected that the measured value will be lower than the

actual value. As is apparent from Fig. 3 (a), the occurrence of low values can be virtually eliminated by setting guidelines for the size of the insertion hole and the sample<sup>16)-18)</sup>. Regarding the effect of the insertion hole, in 1969 K. Bethe et al.<sup>19</sup> pointed out that test results indicated a lower value (Fig. 3).



Fig. 2. Resonator for measuring at microwave frequencies with a rod sample positioned in the middle of the resonator. In both standard cavities the sample is inserted through the upper and lower holes.



diameter in H<sub>103</sub> resonator (K. Bethe et al., 1969)

Fig. 3. Effect of sample insertion holes on measurements using a dielectric bar (K. Bethe [et al.]<sup>17)</sup>.

Recently, the cavity perturbation method has been reevaluated because of its simplicity. The accuracy of the measurements has improved using the Resonance Area Method by T. Miura et al. in 1994<sup>20</sup>, and limitations of the perturbation method have become clear through rigorous theoretical analysis of cavity resonance modes<sup>21</sup>. In 2001, M. Ikeda et al. in TDK<sup>22</sup> also pointed out that the measured value is lower than the actual value using the cavity perturbation method with a conventional  $TM_{010}$  cavity resonator having insertion holes, especially for a cavity having low height.

This has led us to conclude that a perturbed resonator without sample insertion holes is essential for the accurate measurement of complex permittivity. We have already designed and manufactured such a resonator 20 years ago for the X Band<sup>1)-2)</sup>. Recently, we improved the structure of the sample insertion holder and manufactured a new  $TM_{010}$  cavity resonator operating at 1 GHz in the UHF band. We plan to design a new large cavity operating below 1 GHz (300 MHz).

# 2. Measurement of Complex Permittivity in Dielectric Materials using the Cavity Perturbation Method.

Table 1 shows methods for measuring complex permittivity in the microwave region. For materials having low permittivity, the cavity resonator and dielectric resonator methods are available.

Method	£'	tanδ	Material	Specimen
Cavity Perturbation Method	1 ~ 20	$10^{-2} \sim 10^{-4} \sim -5$	Low <i>ε'</i> material Low loss material <u>Separable<i>ε</i> and μ</u>	Small sample (rod, stick and plate sample)
S-parameter Method	2 ~ 30	$10^{-1} \sim 10^{-2}$	High loss material <u>Wide frequency band</u>	Toroidal or plate sample
Dielectric Resonator Method	10 ~ 100	$10^{-3} \sim 10^{-5}$	Large material Low loss material <u>Need standard sample</u>	Disc sample
Free Space Method (reflection and transmittion)	-		Measurement <i>in situ</i> <u>Non-destructive</u>	Large sample Long sample for low loss material

Table 1. Comparison of the methods for measuring complex permittivity in microwave region.

The perturbed cavity resonator method was studied from the 1940s to 1950s and is widely used<sup>1)-9)</sup>. It is an excellent technique for measuring separately the complex permittivity ( ) and complex permeability ( $\mu$ ) of magnetic materials such as ferrite. Using the perturbed cavity resonator method at microwave frequencies corresponds to measuring with a capacitor and  $\mu$  with a coil at low frequencies.

Measuring at low frequencies involves determining the value of from the rate of capacitance change. A disk sample is inserted into a capacitor consisting of two parallel electrodes, as shown in Fig. 1. The rate of capacitance change is proportional to (-1) of the material as expressed by

$$\frac{Y - Y_0}{Y_0} = (\varepsilon - 1)\frac{\Delta V}{V} \quad , \tag{1}$$

where Y and Y0 are the admittance of the capacitor with and without the dielectric sample, respectively, and  $\Delta V/V$  is the filling factor of the sample (equal to 1 in this case). Capacitance change is measured using an impedance meter (Bridge method). This corresponds to the standing wave method used for microwave frequencies.

Using an LC resonator (consisting of an inductor connected to a capacitor) to determine the value of from resonance frequencies and Q variations (Q-meter method) corresponds to using the perturbed cavity resonator method at microwave frequencies.

It is possible to measure independent of  $\mu$  by positioning a small sample so that only the electric field of the cavity resonator is present in the sample. This fact indicates that the perturbed cavity resonator method is superior. The sample shape and sample position for various types of resonators have already been addressed by N. Ogasawara [et al.]. Using a bar-shaped sample exploits the simplicity of measuring with a low frequency capacitor as illustrated in Fig. 1.

For the X band, we constructed and used both a rectangular  $TE_{10n}$  mode cavity and a cylindrical  $TM_{010}$  cavity. But for the UHF band, because the size of the waveguide is so large and the Q value of the  $TE_{10n}$  cavity is not as high as that of the  $TM_{010}$  cavity, we designed a cylindrical  $TM_{010}$  cavity resonator only. Fig. 4 shows the case in which a cylindrical bar sample is positioned in the middle of the  $TM_{010}$  cavity resonator as specified in the JIS method, shown in Fig. 2.

Using this configuration in which the sample is placed at the center of the cavity, only the electric field is present and, as such, the device behaves like a capacitor for low frequency measurements. If the sample radius (r) and value are small, the perturbation formula holds.

The insertion holes in the cavity wall essentially form the maximum point of the electric field and mimic a capacitor. The former does not pose any problem. The shift of resonant frequencies is insignificant since the leakage of electromagnetic waves is eliminated as a result of the cutoff design of the insertion hole. However, the latter reduces the measured value of as explained above. It is desirable to eliminate the insertion hole entirely and develop a system that takes better advantage of the perturbation theory, thus obtaining the value from the perturbation of a capacitor. The authors therefore concluded that eliminating the sample insertion holes would provide an improved means of measuring values at microwave frequencies while simplifying the technique. As a result, we have designed and successfully manufactured a resonator without insertion holes and, taking advantage of this opportunity, devised a new design for the sample adapter to simplify the sample insertion process.

#### 3. Perturbed Cavity Resonator without Sample Insertion Holes

As discussed earlier for the measurement of at microwave frequencies, a perturbed cavity resonator without sample insertion holes is required. This section presents our design method for the new resonator. It is important that the simplicity of the resonator is not lost through elimination of the insertion holes. Twenty years ago, to maintain this simplicity, we designed a cassette-type insertion mount for simplified sample insertion and removal (Photo. 1).

The essential requirements in the design of the new resonator are as follows:

(1) The sample can be positioned accurately in the center of the resonator.

(2) Both upper and lower electrodes can be moved outside when a sample is inserted.

(3) There is a method for eliminating the gap between the end of the sample and the plate electrode.

(4) The plate electrode can be positioned identically whether measuring with or without a sample



Photo. 1. X-band rectangular  $TE_{10n}$  cavity resonator for measuring complex permittivity without sample insertion holes. The sample position was determined with a micrometer having a precision of  $\pm 1 \mu m$ .

In the previous cavity, the sample position was determined with a micrometer having a precision of  $\pm 1 \,\mu$  m as shown in Photo. 1. This mechanism makes it difficult to place the upper electrode in the same position for measurements both with and without a sample.

Fig. 6 and Photo. 1 show the newly designed cylindrical  $TM_{010}$  resonator meeting the above-mentioned requirements. The sample must be positioned at the middle of the resonator so that only the electric field is present. This condition (1) is very important. The sample alignment adapter is prepared as shown in Photo. 2. Conditions (2)-(4) are satisfied by the improved sample insertion holder. The sample insertion/removal holder is designed so that the parallel plate capacitors having upper and lower electrodes are fixed to the external frame. The upper plate electrode ( diameter:10mm ) has a screw for position adjustment to eliminate the air gap

between the sample and the electrode. This holder can be transferred to the interior of the resonator with the sample already inserted. Sample height, even when the sample is carefully manufactured to just fit the inner height (30.0 mm) of the resonator, can still have a slight error. For this reason, the upper plate electrode is designed so that it can be moved slightly with the screw to eliminate any gap. The actual sample height is memorized by the positioning screw and fixed by the locking screw during experiments. Although adjustment results in a slight misalignment of upper plate electrode with cavity height, it is possible to measure only the perturbation of the sample by measuring under the same conditions when the sample is not inserted.

Our resonant cavity was made out of oxygen free copper with an inner diameter of 225.0 mm . The height of the cavity is 30.0mm. In order to make Q of the cavity high, it is necessary the height of the resonator to enlarge (50mm). But we determined the height to 30.0mm because it is difficult to make the long samples of the natural rock salt which is our measuring objects. The resonance frequency of the  $TM_{010}$  cavity resonator is  $f_0 = 1.02$  GHz. Magnetic field couplings are used to excite the  $TM_{0n0}$  modes of cavity for the input and output couplings. The loaded quality factor of the cavity is high enough;  $Q_{0L} = 10000$ , which is the almost same value as a theoretical value of it, or 12000. The cavity insertion loss (IL expressed as dB) is less than -30 dB to reduce the effect of external Q. If the value of IL is not less than -30dB, true internal Q (Q<sub>0</sub>) of the cavity is derived by

$$Q_o = Q_{0L} \frac{1}{1 - 10^{-\frac{H}{20}}} \quad , \tag{2}$$

This cavity also resonates at 2.34GHz using  $TM_{020}$  mode. In our cavity, it is easy to measure complex permittivity at higher  $TM_{0n0}$ mode, because the ratio of a cavity diameter D and a height L (D/L) is very large so that the influence of the other extraneous TE modes of the cavity decreases. In the measurement at 2.34GHz or higher frequencies, large samples do not hold the perturbation condition. In this case, the exact solution, which had been already derived and expressed in JIS<sup>16</sup>, would be used to derive complex permittivity. In order to do such compensation, the cavity resonator without insertion holes is needed so that the boundary condition is exactly same as the theory.

We plan in future to design a new large cavity operating below 1 GHz (300 MHz). At this frequency, the size of the cavity will be much larger and heavier. For example, the diameter of a cavity resonating at 300 MHz is about 700 mm  $\,$ . To maintain a high Q equivalent to that of a 1 GHz resonator, the height must be 100 mm. In this case, oxygen-free copper cannot be used because of its heaviness. To solve the weight problem, a cavity resonator using aluminum for the side wall operating at 1 GHz is now manufactured and tested by Keycom Co., ltd. This resonator is shown in Photo. 3. The results of the tests at 1GHz aluminum cavity were good, we ordered large cavity for 300MHz to get the data of complex permittivity of rock salts. The design of the cavity without of insertion holes would be different from those of the 1GHz cavity because its size would be very large.



Photo. 2. Cylindrical  $TM_{010}$  resonator for measuring at 1 GHz without sample insertion holes. A sample stick of rock salt (Hockley mine, Texas, U.S.A.) is shown with alignment equipment.



Fig. 6. Cross-sectional view of the sample insertion holder of a cylindrical  $TM_{010}$  cavity resonator. The sample position is memorized by the adjustment screw and fixed by the upper locking screw.



Photo. 3. Test cavity resonator operating at 1 GHz using aluminum side wall. (Courtesy of Keycom Co., ltd.)

#### 4. Experimental System and Procedure for Measurement.

Fig. 7 shows a conventional circuit for measuring at microwave frequencies. Using this apparatus, complex permittivity  $\boldsymbol{\varepsilon}$  is given by

$$\varepsilon = \varepsilon' - j\varepsilon'' = \varepsilon'(1 - j\tan\delta), \tag{3}$$

where permittivity loss angle  $tan\delta$  is

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'}.\tag{4}$$

The principle of this measurement is to derive the real and imaginary parts of the complex permittivity,  $\varepsilon'$  and  $\varepsilon''$  respectively, from changes in the center frequency and width of resonance, respectively. Measurements are made both with and without sample insertion in the cavity.

Real  $\varepsilon'$  is derived from the change in resonance frequency with the sample inserted in the cavity by

$$\frac{-(f-f_0)}{f_0} = \alpha_{\varepsilon}(\varepsilon'-1)\frac{dV}{V} \quad , \tag{5}$$

where f and  $f_0$  are the resonance frequencies with and without the sample inserted,  $\Delta V$  and V are the volumes of the sample and the cavity resonator, respectively, and  $\alpha_{\varepsilon}$  is a constant determined by the resonance mode and the sample position relative to the electric field maximum

(equal to 1.855 in this case with a  $TM_{010}$  mode cavity). Note that the presence of  $\Delta V/V$  indicates that the size of the sample will impact the perturbation of the resonance behavior and hence a small stick-shaped sample or rod sample should be used.

Imaginary  $\varepsilon''$  depends on the change in the *Q* factor,

$$\frac{1}{2}\left[\left(\frac{1}{Q}\right) - \left(\frac{1}{Q_0}\right)\right] = \alpha_{\varepsilon}\varepsilon''\frac{dV}{V} \quad , \tag{6}$$

where Q and  $Q_0$  are f/df and  $f_0/df_0$ , respectively, and df and  $df_0$  are the resonance widths measured at a height of half the peak height. The inverse Q difference  $(1/Q - 1/Q_0)$  is defined as  $1/Q_s$ . The measured  $Q_0$  was found to be around 10000 for this cavity.

A radio frequency signal was supplied to the cavity resonator by a synthesized CW generator (Anritsu 68x47C) and Q was measured by a HP 8755B swept amplitude analyzer or power meter (HP E4418B). A vector network analyzer (VNA) system (HP 8510B) was also used. The VNA system makes the measurement setup and procedure very easy.

The absolute uncertainty of the frequency measurements was well under  $1 \times 10^{-5}$ . Measurement uncertainty in  $\varepsilon'$  comes from an uncertainty of ~10 kHz in measured resonance peak frequencies and an uncertainty of ~0.001 mm<sup>3</sup> in measured cavity and sample volumes. The largest contribution to uncertainty in  $\varepsilon'$  was from the measurement of  $\Delta V$ . The sample volume was measured using a microscope with movable x-y micrometers. We estimated an uncertainty in  $\varepsilon'$  of 3%.

Uncertainty in  $\varepsilon''$  is due mainly to uncertainty in measuring the resonance width, ~100 kHz, with and without the sample. After calculation, the estimated uncertainty in  $\varepsilon''$  is 2.5 × 10<sup>-4</sup>. As mentioned above, more precise measurements were conducted by TDK<sup>20</sup>.



Fig. 7. Conventional circuit for measuring complex permittivity at microwave frequencies. Can be replaced with a simpler vector network analyzer (VNA) system.

#### 5. Examples of Measurement in Dielectric Materials.

Table 2. shows an example of measurement using the cylindrical  $TM_{010}$  resonator without sample insertion holes at 1 GHz. The standard material samples were Teflon and synthetic NaCl. Samples considered candidates for the ultra-high energy neutrino detector<sup>20)23)</sup> were materials such as rock salts, limestone and granite. Measurements were made with diameters less than 10 mm for rod samples and less than 6 x 6 mm square for stick samples. Sample length was 30.0 mm, the same as the cavity height. The restrictions on sample size were determined by experiments in the X band (below 1 mm) where the perturbation formula holds.

Table 2 indicates that =  $(2.05 \pm 0.06) - j(5x10^{-4} \pm 0.5)$  or  $\tan \delta = (2.5 \pm 0.5) \times 10^{-4}$  for Teflon. The measured value is the same as for standard permittivity data. For synthetic rock salt we found an average of  $\varepsilon = (5.9 \pm 0.2) - j(2.5 \pm 0.3) \times 10^{-4}$  or  $\tan \delta = (4.3 \pm 0.5) \times 10^{-5}$  which is lower than the values given in Science Chronological Tables and the book by Hippel<sup>10</sup>.

The attenuation length (L $\alpha$ ) of rock salt from Hockley Mine, Texas, U.S.A. is tan =  $2.3 \times 10^{-4}$ , L $\alpha$  = 180 m at 1 GHz. If tan is constant with respect to frequency, then L $\alpha$  becomes 900 m at 200 MHz. The limestone and granite from the Kamaishi mine in Japan have a very large loss tangent, or a short attenuation length.

Sample	Cross section	ε'	<i>€"</i> ×10 <sup>-4</sup>	$Tan\delta  imes 10^{-4}$
Teflon	4 - 5 mm	$2.05\pm0.06$	$5.3 \pm 0.5$	$2.6\pm0.5$
Teflon (2.4 GHz)	4 - 5 mm	$2.06\pm0.06$	$5.6 \pm 0.5$	$2.7\pm0.5$
Single Crystal NaCl	4 - 9 mm	$5.9 \pm 0.2$	$2.5 \pm 0.5$	$0.43\pm0.05$
Rock Salt (NaCl) (Hockley Mine, Texas, U.S.A.)	6×6 mm	5.9 ± 0.2	1.5 ± 0.5	2.5 ± 0.5
Limestone (CaCO <sub>3</sub> ) Kamaishi, Japan	3 - 6 mm	8.7 ± 0.3	$180 \pm 20$	21 ± 2
Granite Kamaishi, Japan	3 - 6 mm	$6.6 \pm 0.2$	2700 ± 300	400 ± 30

TABLE 2. Comparison among Teflon, NaCl single crystal, Hallstadt rock salt, Kamaishi limestone and Kamaishi granite for  $\varepsilon'$ ,  $\varepsilon''$ ,  $tan\delta = \varepsilon''/\varepsilon'$  at 1.02 GHz except for the data of Teflon at 2.4GHz.

The samples were formed into small sticks of length 30.0 mm equal to the height of the cavity resonator. For this perturbation method, small samples should be used to avoid changing the resonance behavior significantly, e.g., inducing only a small shift in the resonance frequency and resonance width. In addition, the electric field strength should be uniform over a cross section of the sample. Although difficult to cut fragile samples to the size needed, it is easy to fabricate small samples for low frequency. Mechanical cutting using a milling machine was unsatisfactory for the natural rock salt samples. For stick samples, we cut from raw materials and then used a

grinding process. It was relatively easy to fabricate rod samples from single crystals of synthesized rock salt. Limestone and granite were strong enough so that we could cut those materials with a milling machine.

For the rock salt and NaCl samples, because they are weak in high humidity, we created a dry-air circulating system that employs a moisture removal machine. This system is shown in Fig. 9.



Fig. 9 Dry-air circulating system using moisture removal machine.

#### 6. Conclusion

The perturbed resonator method has been established as a suitable technique for separate measurements of complex permittivity and complex permeability of magnetic materials such as ferrite. For this reason, this method was used here with an emphasis placed on the measurement of complex permeability. Originally, this method was not accepted as an accurate way of measuring complex permittivity at microwave frequencies due to slight changes in measurement conditions resulting in low C values. In view of this, the authors proposed construction of a resonator such that permittivity could be measured using a capacitor at low frequencies. Based on this idea, a new improved resonator without sample insertion holes was designed and manufactured for operation at 1 GHz. The new resonator was used to measure values.

In this study, the attenuation length of various rock salts and limestone were measured at 1 GHz by the cavity perturbation method with precision ten times better than previous measurements at 10 MHz and 25 GHz. Synthesized NaCl showed '=5.9, tan  $=4.3 \times 10^{-5}$ , at 1 GHz and tan five times smaller than the upper limit measured previously at 10 MHz. The attenuation length (L $\alpha$ ) of rock salt from Hockley Mine, Texas, U.S.A. yielded tan  $=2.3 \times 10^{-4}$ , L $\alpha = 180$  m, at 1 GHz. When tan is constant with respect to frequency, L $\alpha$  becomes

900 m at 200 MHz. L $\alpha$  is sufficient for the salt neutrino detector. These measurements show that natural rock salt is a candidate material for the ultra-high energy neutrino detector. In future we would like to measure complex permittivity at low frequencies (100 MHz – 300 MHz) where the attenuation length increases.

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