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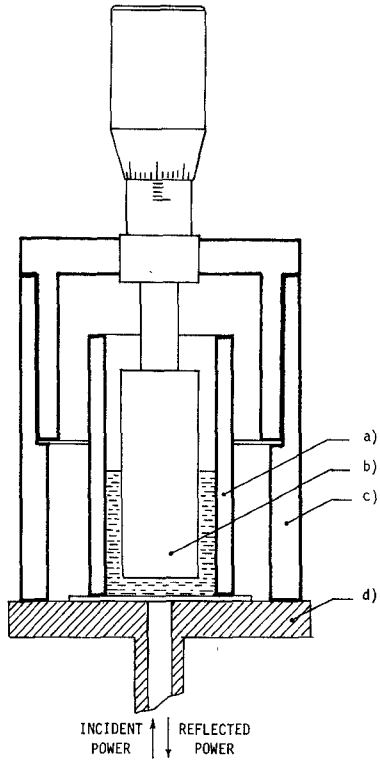


Fig. 1. Cell for lossy liquids permittivity measurements. a) Glass sample holder. b) Teflon piston. c) Micrometer screw socket. d) Waveguide flange.

# Permittivity Measurements of Lossy Liquids at Millimeter-Wave Frequencies

LUIGI ZANFORLIN

*Abstract*—**A measurement system is described which allows the determination of the complex permittivity of high-loss liquids at millimeter waves. Basically, the setup consists of a waveguide interferometer whose unknown arm embodies a liquid holder irradiated by an open-ended rectangular waveguide. The sample thickness is varied by means of a piston driven by a micrometer screw. The bridge output then is read as a function of the liquid thickness. Best fitting between experimental and computed data through a suitable model of the system enables the permittivity to be determined. The system can operate, with high sensitivity, over the whole frequency range of the dominant mode propagating in the waveguide setup employed. System performance is described through a set of experimental results obtained on ethanol, methanol, and pure water at 20° C and 70 GHz.**

## I. Introduction

Measurements of the complex dielectric constant of liquids in the largest possible frequency range have received qualified attention from experimenters of different fields. Thus, Hollecker, Goulon *et al.* performed measurements of 2 and 5 MHz [1], and Goulon, Roussy *et al.* performed measurements of solutions in the far IR [2]. Szwarnowski and Sheppard worked at 70 GHz by using holders designed for this frequency [3].

In this paper, a wide-band measurement system in the millimeter-wave range is presented, following a previous method to measure the permittivity of a living cell's sediment obtained from a suspension into a watery medium [4], [5]. The very high sensitivity experienced with this technique in the range 65–85 GHz, in spite of the large attenuation constant of water, has suggested extending the basic method to lossy liquid permittivity measurements.

For this purpose, a proper sample holder to be kept in contact with the flange of an open-ended waveguide has been studied. Thus, the high-frequency difficulties associated with the use of cavity resonators or waveguides as sample holders have been avoided, and a broad-band precise measurement system has been obtained. The system performance has been evaluated by carrying out measurements on ethanol, methanol, and pure water at 20° C and 70 GHz.

## II. Measuring System

The liquid dielectric to be tested is put in the glass sample holder shown in Fig. 1. The bottom of the sample holder consists of a slide 0.15 mm thick, and its underside is irradiated by a contacting open-ended flanged rectangular waveguide. The signal reflected from the holder is varied by changing the sample thickness by means of a piston driven by a micrometer screw; the piston position can be set to within 5 $\mu$m.

Both the socket of the micrometer and the bottom of the sample holder are trued with the waveguide and are cemented to the waveguide flange. Thus, unwanted movements while turning the micrometer are avoided, and a correct and repeatable placement of the sample is ensured. In the experiments, a teflon piston is used in order to avoid chemical reactions with the liquid sample.

Results can be shown to be independent of the piston material, provided that its electrical characteristics are different enough from those of the sample.

The signal reflected from the sample holder is compared with a constant reference signal by means of the null interferometric

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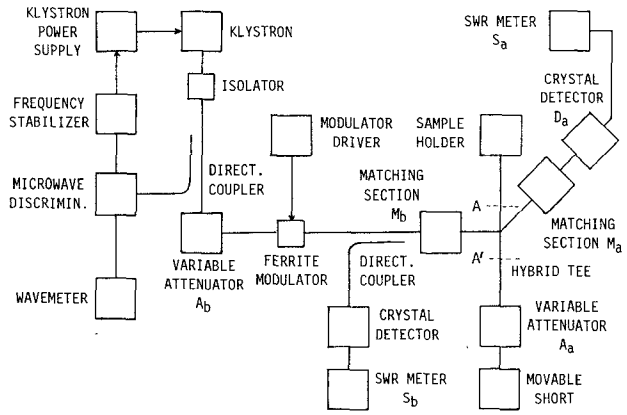


Fig. 2 Measurement system for the determination of electrical parameters of lossy liquids.

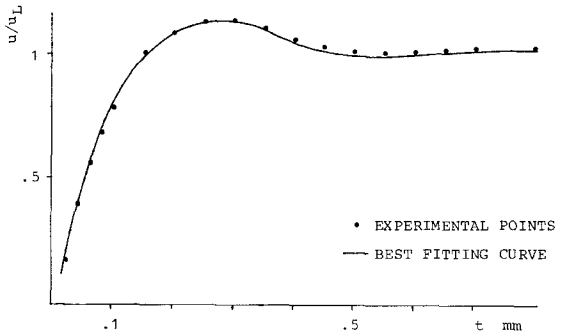


Fig. 3. Experimental points and best fitting curve for distilled water at 20° C and 70 GHz.

bridge shown in Fig. 2. Microwave components are the same as those of the (klystron) system outlined in [5].

For precise permittivity measurements, once having assured accurate reading of the piston travel and mechanical stability of the setup, good frequency stability is also required. This is fulfilled by locking the klystron through a frequency stabilizer and a microwave discriminator which uses a wavemeter as reference cavity (see Fig. 2).

For well-known sensitivity reasons, at the output of the system a 1000-Hz selective voltmeter (SWR meter $S_a$) is used; proper modulation of the microwaves is provided by a ferrite modulator and the associated driver.

The variable attenuator $A_b$ is adjusted to allow the detector $D_a$ to operate in its square-law range.

To insure broad-band operation, a hybrid Tee is used. However, the matching sections $M_a$ and $M_b$ are provided in the $E$- and $H$-arm of the hybrid Tee in order to obtain, at a given frequency, the desirable performance of a magic Tee.

Finally, one of the side arms of the Tee is connected to the sample holder, and the other to a variable load realized by a variable attenuator ended by a movable short.

## III. Measurement Procedure

After tuning and stabilizing the klystron to the working frequency, the piston is placed in contact with the bottom of the sample holder which is filled with the liquid to be tested.

The detector $D_a$ is matched through the section $M_a$ (maximum reading at the output meter $S_a$), and a null at the interferometer output is obtained by means of the movable short and of the variable attenuator $A_a$. Then, by acting on the movable short and on the matching section $M_b$ in sequence, a null at the meter $S_b$ and a simultaneous maximum at the meter $S_a$ are obtained. Such a procedure is to be repeated until the above null and maximum become interchangeable by moving the short only.

Now, by selecting the movable short position corresponding to the null at the output, the piston is turned away by means of the micrometer screw. Thus, a layer of liquid dielectric grows between the piston underside and the sample holder bottom. A bridge unbalance then occurs; therefore, an output signal can be read at meter $S_a$ as a function of the liquid thickness.

## IV. Sample Modeling and Permittivity Determination

The measuring procedure proposed above leads to a quite simple relationship between the permittivity to be determined and the experimental data (bridge output versus sample thickness). To this end, the propagation into the sample is assumed as being plane-wave equivalent.

TABLE I
Real and Imaginary Part of the Complex Permittivity Evaluated from Propagation Constant for Various Liquids at 20°C, 70 GHz.

| Liquid | Re/γ/ mm$^{-1}$ | Im/γ/ mm$^{-1}$ | ε' | ε" |
|---|---|---|---|---|
| Ethanol absolute | .414 | 2.807 | 3.58 | 1.07 |
| Methanol absolute | .801 | 3.268 | 4.66 | 2.43 |
| Distilled water | 3.328 | 5.693 | 9.31 | 17.61 |

Let $W$ be the power delivered to the crystal detector $D_a$ at the output arm. If it is assumed that the output of this detector is proportional to $W$ raised to some power $n$, it can be shown (see Appendix) that the output signal $u$ depends on the sample thickness $t$ as follows:

$$u = u_L \left| \frac{1 - e^{-2\gamma t}}{1 + m e^{-2\gamma t}} \right|^{2n} \tag{1}$$

where $u_L$ is the output signal for $t \to \infty$ (it coincides with the output reached when no variations are observed by moving the piston), $\gamma$ is the propagation constant in the sample to be tested, and $m$ is a complex constant that (for a given frequency and a specified liquid) accounts for the electrical characteristics of the system consisting of waveguide, sample holder, and piston (Appendix). Experimentally, $n$ has been found near unity for the crystal detector used.

By optimizing the four parameters appearing in (1), namely the real and imaginary parts of $\gamma$ and $m$, the proposed model fits the experimental data very well. Then, the optimized values of $\mathrm{Re}(\gamma)$ and $\mathrm{Im}(\gamma)$ allow the complex permittivity to be determined. Fig. 3 shows the experimental points obtained for distilled water, and the best fitting curve computed through the model outlined.

The results of measurements carried out on lossy liquids at 20° C and at 70 GHz are presented in Table I. The best fitting operation has been implemented on a desk-top computer.

The experimental data appear to be in very good agreement with those obtained by Szwarnowski and Sheppard [3], whose setup, however, requires two different cells, one for high-loss liquids and the other for medium-loss liquids, both designed to operate at 70 GHz only.

## V. Considerations on Accuracy

Theoretical analysis (Appendix) allows one to conclude that a residual film of liquid between the piston and the holder bottom does not affect the accuracy of the results, provided that the corresponding piston is taken as the sample thickness reference in performing measurements. This has also been verified experimentally.

The simplificative assumption of plane-wave equivalent propagation corresponds to neglect the near-field nature of the propagation into the sample. Actually, the experimental results have shown that the proposed model is fully adequate for liquids characterized by high values of real and imaginary parts of the complex permittivity.

As far as mismatches and component imperfections are concerned, tests performed by replacing the sample holder with a standard variable attenuator ended by a variable short have shown that the corresponding error remains below a few percent if the return loss ratio at the input and output arms is less than $-50$ dB, and the output detector follows a square law within 1 dB.

In order to cut down errors made in the determination of the sample thickness, as well as those depending on source amplitude fluctuations, the experimental points are taken as the mean of several output values. For each point, these values correspond to predetermined positions of the micrometer indicator, reached either when unscrewing or screwing down the device.

## VI. Conclusions

The measurement method presented here enables one to obtain readily the complex permittivity of high-loss liquids over the whole frequency range of the dominant mode propagating in the waveguide setup employed.

The system exhibits high sensitivity, resulting in a dynamic range of about 40 dB on the output signal when performing measurements on water.

The simplified model of the system, implemented on a desk-top computer, enables precise results for dielectrics, like water, with high real and imaginary parts of the complex permittivity. When testing dielectrics characterized by low values of the above parameters, an improved model of the system is needed (see, for example, [6]). In these cases, however, the computational procedures become quite involved, requiring high-capability hardware equipment.

## Appendix

The system has been studied by assuming that the Tee is matched looking in the input and output arms, and that the propagation into the sample is a plane-wave equivalent.

Diffraction effects are negligible, provided that the glass sample holder and the piston are sufficiently larger than the reciprocal of the attenuation constant of the glass and of the liquid to be tested. In the described setup, the piston diameter is 15 mm and the inside diameter of the holder is 22 mm.

The measurement theory refers to the schematic view of the sample holder shown in Fig. 4, where $A$ is a reference plane in the bridge measuring arm (Fig. 2), $B$ is the reference plane for the measurement of the sample thickness $t$, and $\delta$ is the thickness of a residual film of liquid between the holder bottom and the piston at initial position.

In the plane-wave model, the part between sections $A$ and $B$ has been represented as in Fig. 5, where a linear two-port represents a) a part of the waveguide of the measurement arm, b) the discontinuity at the open end of the waveguide, and c) the bottom of the sample holder, together with a residual film of liquid.

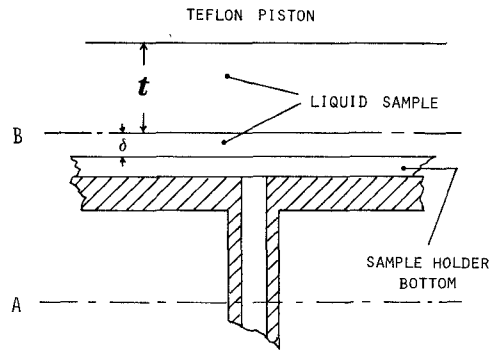


Fig. 4. Schematic view of the sample holder.

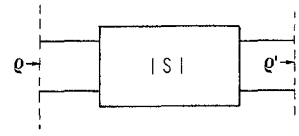


Fig. 5. Electrical model of the measuring arm of the Tee including the lower part of the sample holder.

The reflection coefficient $\rho$ at the section $A$ is related to that $\rho'$ at the section $B$ as follows:

$$\rho = S_{11} + \frac{S_{12}^2 \rho'}{1 - S_{22}\rho'} \tag{A1}$$

where $S_{ij}$ are the scattering parameters of the two-port.

The reflection coefficient $\rho'$ can be written as

$$\rho' = \frac{\rho_\infty + \rho'' e^{-2\gamma t}}{1 + \rho_\infty \rho'' e^{-2\gamma t}} \tag{A2}$$

where $\rho_\infty$ is the value of $\rho'$ for $t \to \infty$, $\rho''$ is the reflection coefficient at the liquid-piston interface, and $\gamma$ and $t$ are defined as in (1). The output $u$ of the interferometer can be written as

$$u \cap |\rho - \rho_0|^{2n} \tag{A3}$$

where $n$ is defined as in (1) and $\rho_0$ is the reflection coefficient in the section $A'$ (symmetrical of section $A$) in the side arm connected to the attenuator $A_a$ (Fig. 2).

The initial bridge balance makes $\rho_0$ equal to $\rho$ yielded by (A1) through (A2) with $t = 0$. Consequently, (1) is reached; the parameter $m$ depends, therefore, on $\rho_\infty$, $\rho''$, and the $S_{ij}$ parameters.

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