

A Method for Dielectric Loss Measurements by a Microwave Cavity in Fixed Resonance Condition

M. MARTINELLI, P. A. ROLLA, AND E. TOMBARI

Abstract—A method for the measurement of dielectric losses at microwave frequencies for small samples inserted in a resonating cavity is discussed. The method allows static and dynamic determinations of ϵ'' by power reflection coefficient measurements in resonance condition and is suitable for experimental setups that use either active or passive frequency techniques. An X-band microwave apparatus based on the passive frequency technique and built up with a right cylindrical cavity resonating in the TE_{011} mode is presented. By means of this apparatus, simultaneous measurements of ϵ' and ϵ'' can be performed. Since each determination of the complex permittivity requires times of the order of 1 ms, and the setting is carried out at the beginning of the experiment only, the apparatus is very suitable for dynamic process measurements. The error sources, the overall accuracy, and resolution are discussed and compared with performance achievable with the basic cavity perturbation method. The efficacy of the proposed method is demonstrated by measurements performed on polar liquid mixtures in perturbed cavity conditions.

I. INTRODUCTION

INTEREST IN dielectric measurements at microwave frequencies has grown in the last few decades in order to characterize the properties of materials, in both applied and fundamental research. The availability of more and more sophisticated instruments has permitted the improvement of well-known techniques and the implementation of other methods using resonant or nonresonant networks.

The cavity perturbation method [1] has proved very suitable for studying the dielectric properties of several materials and for characterizing some processes; therefore, many measurement techniques of the resonant frequency and merit factor Q of a cavity have been developed [2]. Here, we want to recall the most significant techniques that have recently been substantially improved to obtain more rapid and accurate dielectric measurements over the microwave region.

In general, the resonance frequency of a cavity is measured at the minimum (maximum) of reflected (transmitted) power in phase-lock [3]–[6] or in frequency sweep conditions [7]–[9]. The Q -factor is measured directly by finding out the frequency or equivalent time difference between the 3-dB points of the resonance curve [7]–[10], or the phase shift with a frequency tracking system [3]–[5],

[11], [12], or also the transmission coefficients at two different resonance frequencies of a bimodal cavity [13].

Measurements performed in phase-lock conditions employing both passive or active techniques are more accurate and faster than those performed in frequency sweep conditions. In fact, in this latter case, the uncertainties in the frequency assignment to the characteristic points of the resonance curve limit the achievable accuracy. However, the choice of the method and technique is determined by the specific application requested, along with a thorough knowledge of the performance of each apparatus. In [4], [5], [7], [13]–[17], the characteristics of some apparatus and measurement techniques are closely examined, so they will be considered for a comparison with results given here.

In the present paper, it is shown that for a small dielectric sample, the imaginary part ϵ'' of the dielectric constant is a linear function of the voltage standing-wave ratio (VSWR) at the cavity input in resonance conditions.

An apparatus based on the above method and working at X-band is presented. It allows simultaneous determinations of the real (ϵ') and imaginary (ϵ'') parts of the relative dielectric constant of samples with an acquisition time as short as 1 ms. High-accuracy experiments confirm the validity of the method and its capabilities, which are particularly useful in the study of dynamic processes.

II. PRINCIPLE OF OPERATION

For a small dielectric sample inserted in a cavity, the cavity perturbation method (CPM) gives ϵ' and ϵ'' in terms of the resonance frequency shift and Q -factor variation as expressed by the equations [1]

$$\epsilon' - 1 = \frac{2}{\eta} \frac{\nu_0 - \nu_1}{\nu_1} \quad (1a)$$

$$\epsilon'' = \frac{1}{\eta} \left(\frac{1}{Q_1} - \frac{1}{Q_0} \right) \quad (1b)$$

where ν_0 and Q_0 are the resonant frequency and the merit factor of the cavity without sample, respectively, ν_1 and Q_1 are the corresponding quantities with a sample present.

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The authors are with the Dipartimento di Fisica dell'Università, Gruppo Nazionale and Centro Interuniversitario di Struttura della Materia, Piazza Torricelli 2, 56100 Pisa, Italy.

The filling factor η is given by [6]

$$\eta = \frac{\int_{V_s} |\vec{E}|^2 dv}{\int_{V_c} |\vec{E}|^2 dv}.$$

Here, V_s and V_c denote the sample and cavity volume, respectively, and \vec{E} is the microwave electric field. These equations are obtained by means of a first-order perturbative expansion under the assumptions that the electromagnetic energy stored or dissipated in the sample volume is negligible in comparison of the energy stored in the whole cavity and the electromagnetic-field distribution is not affected by the sample insertion.

At the second order, the development involved in the CPM implies an influence of the Q -factor on ϵ' determination expressed by

$$\frac{\eta}{2}(\epsilon' - 1) = \frac{\nu_0 - \nu_1}{\nu_1} + \frac{1}{4Q_1} \left(\frac{1}{Q_1} - \frac{1}{Q_0} \right). \quad (2)$$

Then the Q -change contributes for an additional frequency change $\Delta\nu$ which, by substituting (1b) in (2), can be written as

$$\frac{\Delta\nu}{\nu_1} = \frac{\eta\epsilon''}{4Q_1}. \quad (3)$$

This equation gives the error affecting (1a); such an error has to be taken into account when more precise measurements are required.

For the determination of ϵ'' , the CPM requires Q -factor measurements that in most cases cannot be performed with sufficient rapidity and precision. Therefore, we introduce a different method allowing the ϵ'' determination by measurements of the power reflection coefficient of the cavity at resonance.

Let us consider an inductance-coupled cavity connected to a microwave bridge (Fig. 1). The equivalent circuit for the cavity input impedance z_i can be represented, according to [18]–[20], as shown in Fig. 2, where R_c is the characteristic impedance of the transmission line and X_1 denotes the constant reactance expressing the “external” impedance of the cavity near to the resonance. For this circuit, the coupling coefficient K is given by the expression [20]

$$K = R/R_c(1 + X_1^2/R_c). \quad (4)$$

In the apparatus of Fig. 1, a phase-lock loop forces the klystron frequency to the minimum of the power reflection coefficient $|\Gamma_i|^2$. In this condition, the VSWR at plane $(i-i)$, indicated as $(VSWR)_i$, is related to the coupling coefficient by the equation

$$(VSWR)_i = \frac{1 + |\Gamma_i|}{1 - |\Gamma_i|} = 1 \sqrt{K} = \frac{1}{R} \left(\frac{R_c^2 + X_1^2}{R_c} \right). \quad (5)$$

From the definition of the unloaded Q -factor for the cavity equivalent circuit, it follows that

$$\frac{1}{Q} = \frac{\omega L}{R} = \frac{P_T}{\omega W_s} \quad (6)$$

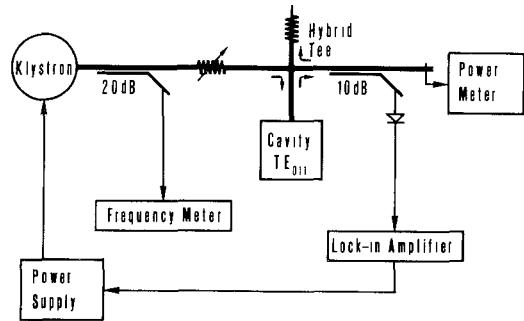


Fig. 1. Schematic diagram of the experimental apparatus.

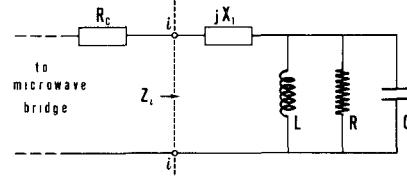


Fig. 2. Equivalent circuit of the cavity and the coupling system.

where P_T and W_s are the total average power dissipated and the total average energy stored in the cavity, respectively, and ω is the angular frequency. By assuming that the cavity contains a small homogeneous, nonmagnetic (i.e., relative permeability $\mu_r = 1$) dielectric sample, (6) gives

$$\frac{1}{R} = \frac{1}{\omega^2 L W_s} \left(P_0 + \frac{1}{8\pi} \epsilon'' \omega \int_{V_s} |\vec{E}|^2 dv \right) \quad (7)$$

provided that the field distribution in the cavity is not changed. Here, P_0 is the average power dissipated in the whole cavity, except the sample, and the second term within the brackets is the average power loss in the dielectric sample. The right-hand side of (7) can be developed in terms of the relative frequency shift $\Delta\omega/\omega$; by substituting with (1a), and neglecting second-order terms, one obtains the equation

$$\frac{1}{R} = \frac{1}{R_0} + \frac{\eta}{\omega_0 L} \epsilon'' \quad (8)$$

where R_0 and ω_0 are the equivalent resistance and resonance frequency of the cavity without sample, respectively.

Combining (8) with (5), it follows that

$$(VSWR)_i = \eta \frac{Q_0}{K_0} \epsilon'' + \frac{1}{K_0} \quad (9)$$

where Q_0 and K_0 are the unloaded Q -factor and coupling coefficient for the cavity without sample, respectively. The quantity $1/K_0$ can be determined from measurements of the reflection coefficient of the cavity without sample; in particular, $K_0 = 1$ when the cavity is critically coupled. The factor (ηQ_0) can be measured as a whole from the dielectric loss of a reference sample, or from a separate Q_0 measurement and η determination employing a reference dielectric sample, according to (1a).

Equation (9) is very useful in the determination of the ϵ'' value and its variation against different physical and chem-

ical parameters in both static and dynamic measurements. The precision and sensitivity of this method favorably compare with the results of the Q -factor measurements performed according to (1b).

III. EXPERIMENTAL ERROR ANALYSIS

Different apparatuses can be built for different frequency ranges; in our measurements we employed an X -band experimental setup, whose diagram is shown in Fig. 1.

The circular right cylindrical cavity resonates in the TE_{011} mode and liquid samples are inserted in a narrow tube [6], extending out of the cavity, so it can be filled and emptied avoiding any mechanical displacement. The cavity temperature is controlled by a thermostatic bath and measured with a precision of 0.1°C . The overall cavity temperature drift can be taken into account by means of the resonance frequency shift. The effective experimental conditions require a different evaluation of the frequency stability and drift. The first one can be measured from Allan's variance [21], and the second one from a maximum long-term shift of the resonance frequency value, averaged over a short time interval. With accurate phase-locking by frequency modulation of the microwave source, Allan's variance in a bandwidth from 10^{-3} to 1 Hz is about 25 Hz. In a time interval of 5×10^3 s, the cavity temperature drift causes a frequency shift of 2×10^4 Hz. This drift, determined by the cavity temperature coefficient, is about 0.1°C .

A very reliable and complete review of the accuracy of dielectric measurements and a careful specification of reference samples is given in [16] and [22]. Concerning ϵ' measurements, the errors due to the CPM approximations have to be considered by evaluating the contribution of (3). In our typical experimental conditions and using a chlorobenzene sample ($\epsilon'' = 1.44$), this error is about 10^{-7} , that is, of the order of 10^{-3} with respect to the entire frequency variation ($(\nu_0 - \nu_1)/\nu_1$). This contribution can be neglected for a relatively low-loss sample. When higher loss samples are measured, a further optimization can be achieved by assuming smaller filling factors, thus increasing the Q -factor according to (8).

The determination of the ϵ'' value by means of VSWR measurements is affected by a relative error, depending on the reflection coefficient value. This dependence is given by the expression

$$\frac{\Delta(\text{VSWR})}{(\text{VSWR})} = \frac{2\Gamma}{(1 - \Gamma^2)} \frac{\Delta\Gamma}{\Gamma}. \quad (10)$$

The relative error in the reflection coefficient measurements performed with a chlorobenzene sample is better than 10^{-3} and the maximum value of $|\Gamma|$ is about 0.7. The corresponding worst relative error, calculated from (10), is 3×10^{-3} .

When the value of Q_0 is determined by a reference sample, as for instance chlorobenzene [23], a proper choice of $|\Gamma|$ must be performed, by means of the coupling circuit, in order to minimize errors in VSWR measurements. By

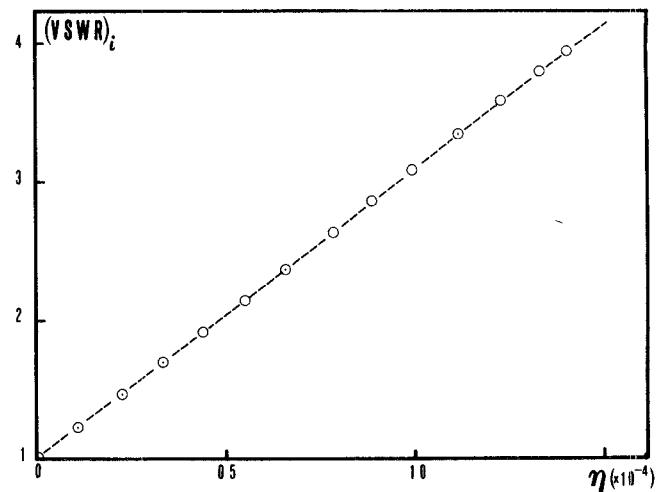


Fig. 3. $(VSWR)_i$ at resonance as a function of the filling factor η . Dashed line represents the linear data-fitting according to a least-squares method, circles indicate experimental values.

this way, an overall relative error of about $2-3 \times 10^{-3}$ in ϵ'' measurements can be determined.

In the case that ηQ_0 is determined from separate measurements of η and Q_0 , the greatest error arises from the Q_0 measurement as discussed in [5], [11]-[13], [19].

All the quantities of interest depend on the filling factor, which is an intrinsic parameter of the chosen sample configuration in the cavity. We have determined this parameter according to the particular experimental arrangement by an ϵ' measurement of a reference low-loss dielectric material. Using a purified and dried sample of cyclohexane, the accuracy of this determination turns out to be about 10^{-3} .

Accuracies previously discussed are reached by our apparatus in a single measurement and with overall acquisition times as short as 10^{-3} s. This latter result can be improved using faster phase-lock techniques.

IV. EXPERIMENTAL RESULTS

Fig. 3 shows the $(VSWR)_i$ at resonance for different filling factors. These are measured, according to (1a), from the resonance frequency shift corresponding to different quantities of chlorobenzene introduced in the cavity by the method discussed in [6]. The experimental results fit very well the theoretical behavior represented by (9). The data fitting has been performed by a standard least-squares method for a linear function written as

$$y = px + q.$$

Here, the y and x variables correspond to $(VSWR)_i$ and η , respectively, and errors in values of both y and x have been taken into account. The relative error in both p and q parameters is 10^{-3} . According to the χ^2 -test, the S value of the χ^2 -function, calculated from experimental data, is 15.5. Since each sample volume spans different field locations in the cavity, the accuracy of measurements confirms also that the boundary conditions are well satisfied and the sample does not perturb the field distribution.

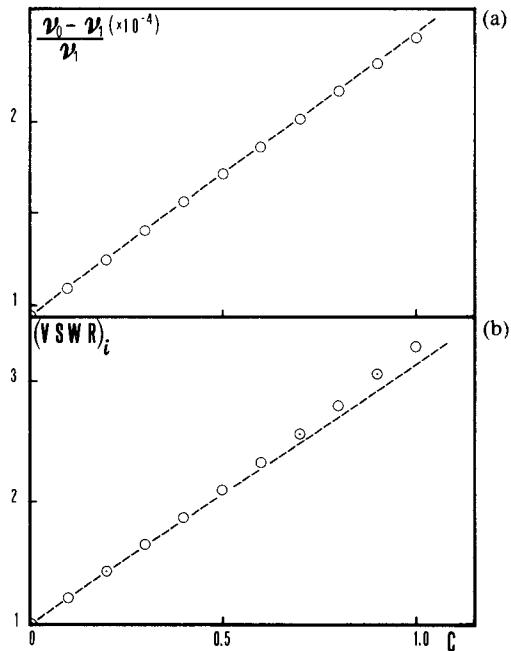


Fig. 4. Measurements on solutions of n.butyl acrylate in benzene solvent. Dashed line corresponds to the behavior of an ideal solution, circles indicate experimental values. (a) The relative frequency change $((v_0 - v_1)/v_1)$ at resonance versus the n.butyl acrylate volume fraction C . (b) The $(VSWR)_i$ variation at resonance versus C .

TABLE I
PARAMETERS OF DATA-FITTING FOR ϵ' AND ϵ'' OF MIXTURES OF
N.BUTYL ACRYLATE IN BENZENE.

	$\Delta p/p$	$\Delta q/q$	S
ϵ'	8×10^{-3}	10^{-3}	8.6
ϵ''	2×10^{-3}	1.2×10^{-2}	9.15

The check of the linearity can be performed with dielectric sample mixtures; in this case, particular care has to be taken into the dipolar interaction effect. Dilute solutions of proper polar liquids in nonpolar solvents at different concentrations must be employed.

Measurements of the frequency shift (Fig. 4(a)) and the $(VSWR)_i$ (Fig. 4(b)), performed on solutions of n.butyl acrylate (BA) in benzene in the whole volume fraction range, are presented. At highest volume fraction values, a small deviation from the linear behavior is observed. This can be mainly ascribed to the dipolar interactions [24].

If we restrict our analysis to a lower volume fraction range, the results reported in Fig. 5 are obtained. During all these measurements, the coupling coefficient K_0 is kept fixed and the concentration values are the greatest error source. In order to remove the effect of the concentration error on the linearity check of (9), the concentration variations in the plot of ϵ'' are measured by frequency shift according to (1a). According to the above procedure, the results summarized in Table I are obtained by a linear data fitting. In the ϵ' fitting, the value of $\Delta p/p$ is just due to errors in the concentration values. In the ϵ'' fitting, $\Delta q/q$ is relatively large; it can be ascribed to effects of measure-

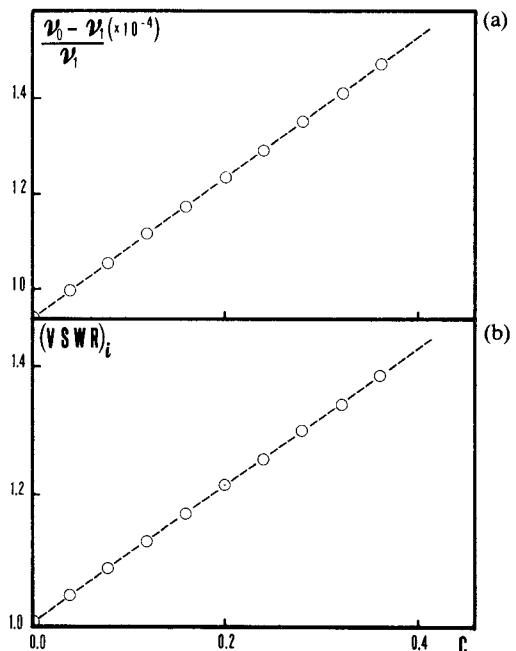


Fig. 5. Measurements on dilute solutions of n.butyl acrylate in benzene solvent. Dashed line corresponds to the linear data-fitting according to a least-squares method, circles indicate experimental values. (a) The relative frequency change $((v_0 - v_1)/v_1)$ at resonance versus the n.butyl acrylate volume fraction C . (b) The $(VSWR)_i$ variation at resonance versus C .

ment errors at very low concentrations. The error on determination of p is very low and the precision of (9) for a linear trend versus ϵ'' is verified with a very good accuracy.

V. CONCLUSIONS

The new method discussed here for measuring dielectric loss has proved useful and accurate. The performance of the experimental apparatus presented here is very suitable in the absolute determination of both real and imaginary parts of the relative dielectric constant.

The setting of the apparatus is very simple and is performed once at the beginning of the experiments. Measurements are very fast and accurate, as previously discussed. The apparatus appears very useful in the dielectric analysis of dynamic processes.

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†

M. Martinelli obtained the laurea in physics from the University of Pisa in 1969.

Since then he has been involved in research activity sponsored by the National Research Council (C.N.R.) at the Dipartimento di Fisica of the University of Pisa through the Gruppo Nazionale di Struttura della Materia (G.N.S.M.). Presently, he has a permanent position as a Research Scientist of C.N.R.. His main scientific activity has been concerned with the experimental research in the field of magnetic resonance and microwave spectroscopy. He has realized or contributed to several microwave and electronic apparatuses of current use in experimental research.

†

P. A. Rolla received the laurea in physics from the University of Pisa in 1971.

From 1971 to 1973, he worked for the Istituto di Elaborazione dell'Informazione of the National Research Council (C.N.R.) of Italy. Since 1973, has been with the University of Pisa at the Dipartimento di Fisica (at first as Assistant Professor then as Associate Professor), where he has been involved in research activity sponsored by the C.N.R. through the Gruppo Nazionale di Struttura della Materia (G.N.S.M.). He has

worked in electronic instrumentation for the research laboratory, magnetic resonance techniques, and microwave spectroscopy. His current interests are in the field of dielectric theories and measurements.

†

E. Tombari was born in Pesaro, Italy, on June 1959. He obtained the laurea from the University of Pisa in 1984.

He is working in the field of dielectric characterization of polymerization processes. His interests includes dielectric theories and dielectric measurement techniques in the microwave region.

