

Short Papers

Measurement of the Microwave Absorption for Small Samples in a Coaxial Line

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Abstract—Microwave absorption measurements in terms of power loss per unit volume of very small liquid and solid samples (0.003–2.6 μL) contained in capillaries inserted across the dielectric of a coaxial line have been made between 2 and 26.5 GHz. This perturbation technique makes it possible to measure small differences in the absorption of the samples in a swept fashion. This technique is applicable to biological measurements where the samples are often very small. It is also of use in monitoring and process control environments in biotechnology and polymer technology.

I. INTRODUCTION

Measurement of the microwave absorption of small samples has been done using a number of techniques, including cavity perturbation [1] and time-domain spectroscopy [2], and of somewhat larger solid samples using a strip transmission line [3]. There have been a number of measurements of samples within and at the termini of coaxial lines [4], [5]. In this paper we describe a new technique with the combined benefits of the previously mentioned techniques, i.e. continuous sweepable measurements and simplicity. By placing a small sample contained in a microcapillary tube in the region between the inner and the outer conductor of a coax line and measuring the power transmitted and reflected from the sample, the microwave absorption, expressed in W/m^3 , can be calculated.

II. DESCRIPTION OF MICROWAVE EQUIPMENT

This technique, which is used to measure the absorption and reflection of a sample, is basically an insertion loss measurement. The sample holder was sandwiched between 10 dB broad-band attenuators (pads) to reduce the standing waves. The transmitted power was measured using a calibrated diode. The reflected power was measured using a broad-band (–16 dB) directional coupler and a calibrated diode between the source (Wiltron 6653A programmable sweep generator) and the attenuator on the signal source end of the sample holder. Shown in Fig. 1 is a diagram of the system.

The detector signals were amplified using Tektronix AM502 amplifiers with the bandwidth set from dc to 1 kHz and digitized using a Macintosh II equipped with a 12 b analog to digital converter (GW Instruments).

Sample holders were made by modifying female–female coaxial connectors of either type N or type SMA. The type-N connectors were used at lower frequencies (where the standing

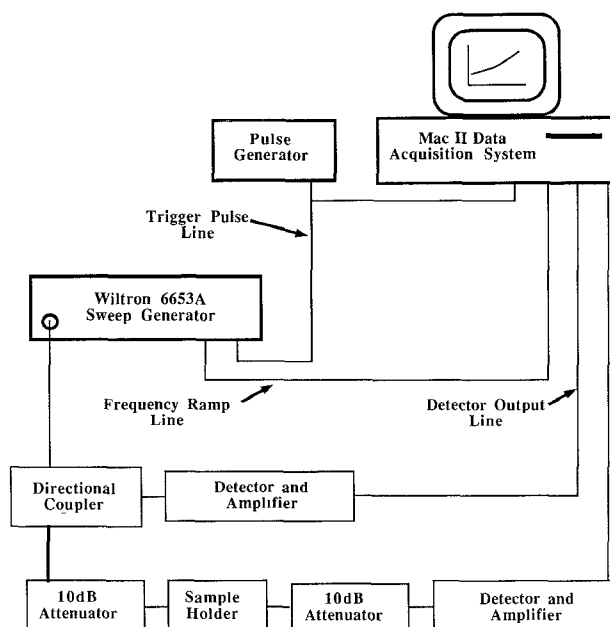


Fig. 1. Schematic diagram of the microwave system layout and the data acquisition system.

wave ratio was low) and where larger sample volumes were required. Transitions to type-N connectors from SMA connectors have a higher SWR and therefore reduced accuracy. The most satisfactory results across the entire 2 to 26.5 GHz band were obtained using components with SMA connectors only. A precision hole of 0.864 mm centered on a diameter to within 0.076 mm was drilled through the SMA (Narda Microwave Corp.) connector. The diameter of the hole was kept small in order to tightly fit glass or quartz capillaries (0.5 mm ID maximum, 0.85 mm OD) which hold the sample and to avoid distortion of the microwave electric field. A diagram of this component is given in Fig. 2 and a photo of the sample holder is shown in Fig. 3. After modification this component was compared with a duplicate to ensure that changes in performance (SWR and loss) were small (less than 5% effect). A machined brass clamp designed to fit snugly to the SMA sample holder was mated to a temperature stabilizer which can monitor and maintain the sample temperature to within 1°C over the range of -27°C to 100°C .

The size of the sample in the volume could be varied from 0.003 to 2.6 μL using capillaries of different inside diameters. Both quartz and glass capillaries were used with equal success. Quartz capillaries were used when subsequent UV spectroscopic measurements were required. It was important to keep the size of the sample as small as possible to minimize the reflections caused by the discontinuity in the real part of the dielectric constant. For the data in this paper, a sample volume of 0.16 μL was used. At 26.5 GHz, the maximum frequency measured, the wavelength in the TFE (Teflon) filed coax ($\epsilon' = 2.04$) was 8 mm,

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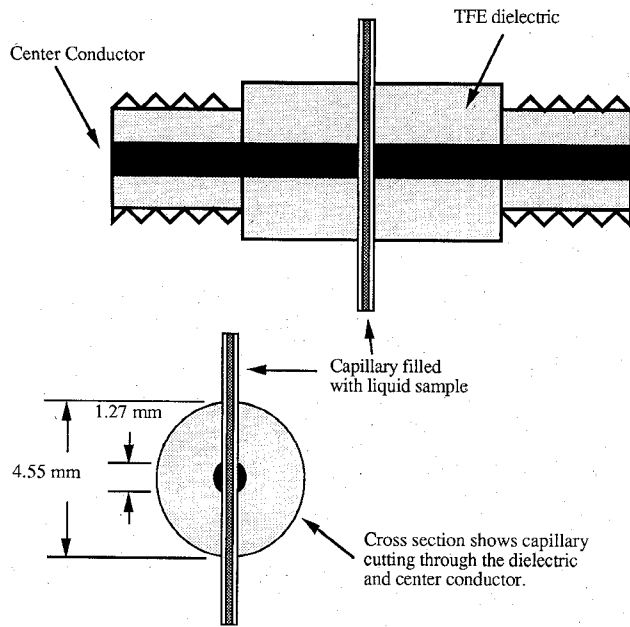


Fig. 2. A hole is drilled across the diameter of an SMA connector, and a sample contained in a capillary is inserted as shown.

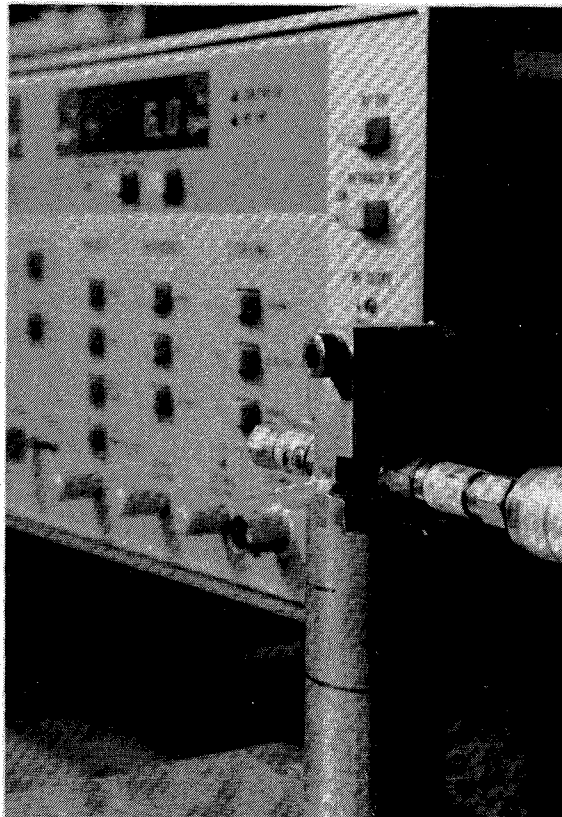


Fig. 3. The SMA sample holder is sandwiched between two 10 dB attenuators. The sample holder is held in a brass fitting to stabilize and control the temperature of the holder and sample. A sample within a glass capillary is shown inserted into the hole drilled across the SMA sample holder. In the background is the sweep generator.

far larger than the maximum sample diameter, 0.5 mm. The reflected power caused by this discontinuity can be calculated [6] as a function of sample size. For the maximum sample size at the maximum frequency (maximum reflection) the reflected power is less than 3% of the total microwave power on the sample. In this estimate we averaged the sample and TFE dielectric constants over the volume of a disk across the coax with a thickness equal to the maximum diameter of the sample (0.5 mm), and water ($\epsilon' \sim 75$) was assumed as the sample. The measured reflected power is less than 5%, justifying the above assumptions. Most samples of interest (organics, hydrocarbons, etc.) have real dielectric constants less than that of water, which accordingly reduce the mismatch and the reflected power. The accuracy of this approach, however, is reduced as the ratio of the loss tangent drops far below that of water ($\tan \delta \approx 0.16$ at 3 GHz) because larger samples will be required. Although this technique was specifically designed to monitor small changes in the properties of biopolymers in water, measurements of polyethylene glycol, ethyl alcohol, and methyl alcohol at low frequencies (3 GHz), where the loss tangent is low (~ 0.04 to 0.06), were within 20% of published values. This technique is not recommended for samples with $\tan \delta < 0.04$. This technique is applicable up to approximately 50 GHz. Below that frequency higher order modes in the coax arising from the two-dimensional nature of the sample geometry are cut off.

III. THEORY OF ABSORPTION IN COAXIAL GEOMETRY

An expression can be derived which links the absorbed power per unit sample volume to the imaginary part of the complex dielectric constant of the sample and the volume-averaged microwave electric field:

$$\frac{P_{\text{absorbed}}}{V_{\text{sample}}} = \frac{\frac{1}{2} \int \omega \epsilon'' E^* E dV}{\int dV} \quad (1)$$

where ω is the microwave radial frequency, ϵ'' is the imaginary part of the dielectric constant, E is the microwave electric field, dV is the differential volume, and V_{sample} is the total volume of the sample exposed to microwaves. Assuming that the sample is uniform over the volume and that the electric field in the coax is inversely proportional to the radius, the integrals can be evaluated:

$$\frac{P_{\text{absorbed}}}{V_{\text{sample}}} = \frac{\frac{1}{2} \omega \epsilon'' E_0^2 a^2 \left(\frac{1}{a} - \frac{1}{b} \right)}{b - a} \quad (2)$$

where a is the outer radius of the inner conductor and b is the inner radius of the outer conductor.

The electric field at the surface of the inner conductor, E_0 , can be determined from the solution of the following equation:

$$P_{\text{in}} = \frac{V_{\text{p-p}}^2}{2R} = \frac{\left(E_0 \int_a^b \frac{a}{r} dr \right)^2}{2R} = \frac{E_0^2 a^2 \left(\ln \left(\frac{b}{a} \right) \right)^2}{2R} \quad (3)$$

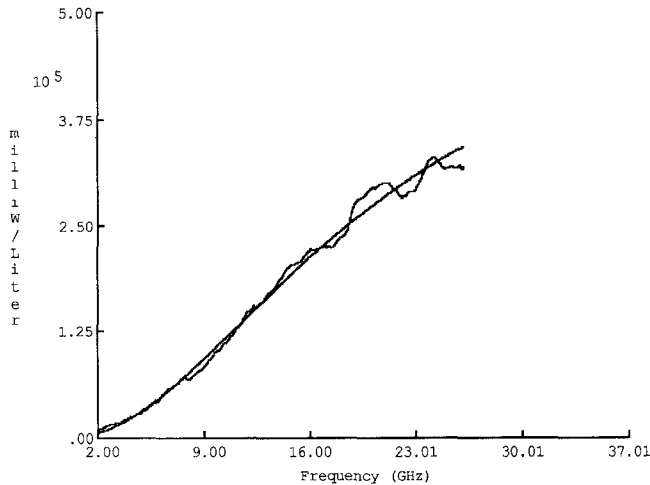


Fig. 4. The microwave absorption of light water (0.16 μ L) is shown. The microwave power level at the sample was 0.5 mW. The curve generated using the Debye relaxation model for the dielectric constant is superposed on this graph.

where P_m is the input microwave power, V_{p-p} is the peak to peak voltage, and R is the impedance of the coax. Continuity of the electric field is assumed since the long, thin sample has boundaries that are approximately parallel to the radial electric field; therefore the quasi-static approximation [7] is made. The angle between the electric field and the sample boundaries varies between 6° at the outer conductor and 23° at the inner conductor with a volume average of less than 12° . E_0 from (3) is inserted into (2) to form the final expression for the power absorbed per volume:

$$\frac{P_{\text{absorbed}}}{V_{\text{sample}}} = \frac{\omega \epsilon'' P_m R}{\left(\ln \left(\frac{b}{a} \right) \right)^2 ab} \quad (4)$$

Using this expression, the imaginary component of the dielectric constant can be determined from the measurements of the absorbed power and the input power level.

IV. SWEEP MEASUREMENTS IN LIGHT AND HEAVY WATER

In order to verify the correct calibration of the system, measurements of the microwave absorption of known dielectric samples were made. For example, the microwave absorption of water in the coax line was measured and compared with the absorption calculated from published values [4], [5] of the complex dielectric constant and (4). The absorption caused by the capillary has been subtracted from the sample plus capillary data by measuring the absorption of the capillary alone. The absorption was less than 1% and was therefore a minor component of the data.

The microwave absorption of light water is shown in Fig. 4. The largest deviation of the measured absorption and the calculated absorption was 10%, which occurred at the highest frequencies measured (~ 26 GHz). The deviation in excess of 5% occurs because of the residual standing waves in the coax, again from the mismatch caused by the sample itself. The reproducibility of this measurement was 2%, set by the amplifier noise and sweep generator output flatness reproducibility.

The absorption was compared with that which can be calculated using the Debye relaxation theory [4], [5]. The Debye relaxation time for light water was measured and found to be 0.0079 ns by fitting the curve in Fig. 3 using (4) and a dielectric constant of the form

$$\epsilon''(\omega) = (\epsilon_{dc} - \epsilon_{\infty}) \left(\frac{\omega \tau}{1 + \omega^2 \tau^2} \right) \quad (5)$$

where the first multiplicative term is the difference between the zero-frequency and the infinite-frequency dielectric constant and τ is the relaxation time. The published value [4], [5] for the relaxation time is 0.009 ns as calculated from complex dielectric constant measurements using discrete frequency cavities. Our computer fit using (5) and their data gives a value of 0.0083 ns, which is very close to the value of 0.0079 ns which we measured. A recently published value [8] for light water was 0.008 ns. The relaxation time for heavy water was also measured and the ratio of the relaxation times of heavy to light water was found to be 1.19 at 23°C , which is within 5% of the value (1.24) which can be calculated from the ratio of viscosities [4], [5].

V. CONCLUSIONS

A simple technique to measure the broad-band microwave absorption of small capillary encapsulated liquid samples (or solid samples that do not need to be held in capillary tubes) which are inserted into the dielectric region of a coax connector has been developed. The measured value of the microwave absorption of heavy and light water is within 10% of the value calculated from published values of the dielectric constant at all frequencies up to the highest frequency measured and within 5% at frequencies (< 20 GHz) where the effects of the impedance mismatch caused by the sample are minimal. The measured differences in the Debye relaxation times of light and heavy water agrees with published values to even better accuracies owing to the similarities in the dielectric constants and thus similarities in the mismatch in the coax caused by the sample. This technique is being employed to investigate microwave absorption in the hydration layer which surrounds polymers in solution.

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