

THE USE OF COAXIAL PROBES FOR PRECISE DIELECTRIC MEASUREMENTS: A REEVALUATION

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$$Y = j\omega \left\{ C_0(\epsilon' - j\epsilon'') + C_f \right\} \quad (1)$$

with

$$\Gamma = \frac{1-50.0Y}{1+50.0Y} \quad (2)$$

SUMMARY

A convenient method has been developed during the past few years for studying the dielectric properties of materials. The method consists of placing the open end of a semirigid coaxial line against the sample and measuring the probe reflection coefficient with an automated network analyzer (ANA). As part of an ongoing attempt to confirm previous reports of resonant-type absorption in DNA solutions at 1-10 GHz, we have analyzed the technique to estimate the magnitude of resonance-type artifacts expected from instrumental factors. The reflection coefficients of the probe were measured using the HP-8510 ANA with time domain gating to exclude connector artifact. We propose an improved probe technique that greatly diminishes artifacts arising from variation of the probe from nominal dimensions, and radiation conductance above 1 GHz.

and where C_0 and C_f are constants that depend on the line⁵. The ANA in these previous studies had been calibrated from measurements on the probe, when terminated by a short circuit, an open circuit, and a fluid (water or methanol) whose dielectric properties are known. For two of these standards (air and the liquid), the reflection coefficient used in the calibration was calculated from Eqs. 1 and 2. The earlier work used values of C_0 and C_f obtained by numerical solution of the field equations for the probe, assuming the probe had ideal geometry and dimensions equal to manufacturers' nominal values for the line⁵.

Two problems are apparent with such an approach: The actual line might deviate from "nominal" dimensions. Moreover, the admittance of the probe will deviate, at high frequencies, from Eq. 1 because of radiation effects, which can be represented by:

$$Y = j\omega \left\{ C_0(\epsilon' - j\epsilon'') + C_f \right\} + G(\epsilon^*, f) \quad (3)$$

where G is a strongly increasing function of permittivity and frequency. A detailed numerical solution for G was obtained by Mosig *et al.*⁶. In the current study, we empirically determine G and the probe constants C_0 and C_f .

INTRODUCTION

Fast and simple dielectric techniques have been developed for noninvasive measurement of the dielectric properties of materials¹. In these techniques a probe is constructed from an open-circuited segment of semi-rigid coaxial cable, whose end is placed against the sample under study. The reflection coefficient from the probe, measured with an ANA, is a function of the dielectric properties of the sample. This method was originally developed to study the dielectric properties of tissues *in vivo* from ca. 10 MHz to 10 GHz². It was also employed to study the dielectric properties of aqueous DNA solutions, which were reported to exhibit microwave-frequency "resonances" in their absorption properties^{3,4}. These resonant effects correspond to variations of the amplitude of the measured reflection coefficient of ca. 0.03 about a baseline value of about 0.8. To reliably interpret such measurements, careful analysis of the potential measurement artifacts (which typically appear resonant-like in appearance) is needed.

We are attempting to confirm the existence of these resonances using methods similar to those originally employed. The results from the measurements on the DNA will be presented elsewhere; here we consider the adequacy of the probe technique for such measurements, using improved instrumentation and avoiding the use of assumed properties of the probe to calibrate the instrumentation. We discuss two major potential sources of error in the technique, as previously employed, and suggest improvements in the method.

The above-cited studies¹⁻⁴ assumed that the admittance of the probe is a simple function of the complex dielectric permittivity of the medium:

PROCEDURE

Probes were constructed from lengths of semi-rigid coaxial line (3.6 mm o.d. with a type SMA connector, or precision 50 ohm 2.99 mm semirigid cable with a precision type K connector⁷. The end distal to the connector was machined flat and polished with fine crocus cloth. The lengths of the probes ranged from 9.5 to 14.0 cm.

The reflection coefficients of the probes were measured with the Hewlett-Packard model 8510 ANA. For these measurements, the ANA was calibrated using normal factory-standard calibration loads (short circuit, open circuit, 50 ohm sliding and fixed load) at the end of a length of precision flexible test cable. A probe was then connected to the test cable. The reference plane of the measurement was defined by shorting the end of the probe with aluminum foil and adjusting the electrical delay in the ANA until a constant 180 degree phase angle was observed.

To adjust for other artifacts, additional small corrections were performed. To remove artifact due to reflections from the probe's connection to the ANA test cable, the time domain gating feature of the HP-8510 ANA was employed: the data were transformed into the time domain, the echoes from the connector were gated out, then the resulting data were transformed back to the frequency domain. All measurements were performed from 0.045 to 18 GHz, in intervals of 0.045 GHz,

using the "step" mode of the ANA. Ten measurements were executed and averaged for each step.

Finally, the small residual loss in the probe and residual error in setting the reference plane were removed on the basis of measurements of the reflection coefficients of the open-ended probe. In the frequency range of the present measurements, it is known that the reflection coefficient of the probe in air should have essentially unit magnitude, and a phase angle that corresponds to a constant capacitance, for all frequencies below ca. 10 GHz⁵. The reflection coefficient was measured for open-circuited probes, and used to obtain small corrections (that increased linearly with frequency for the phase, and as the square root of the frequency for the magnitude); which were then applied to all of the subsequently obtained data. These corrections were less than 3 degrees in phase and less than 0.05 in magnitude at 10 GHz.

Measurements were then performed on a variety of fluids of known dielectric properties: 0-0.5 N KCl solutions, methanol, distilled water, and water-dioxane solutions⁸. All measurements were performed at 23-25° C. The KCl solutions were used for conductance standards, and the water-dioxane solutions for permittivity standards.

RESULTS

1. Calibration of the probe, 0.5 GHz. Figures 1A,B show the termination capacitance and conductance of one probe (constructed from 3.6 mm semirigid line) at 0.5 GHz, vs. the permittivity ϵ and conductivity σ of the solutions. The probe constant C_0 , which corresponds to the slope of these curves, differed by 4% when calculated from the permittivity or conductance standards (the quantity C_f , which is related to the y-intercept of the curves, was negligible.) This error is, however, perhaps a consequence of inadequate temperature regulation of the calibration fluids, which show variations in permittivity of ca. 0.5 %/°C, and in conductivity of ca. 2-3 %/°C. For the subsequent analysis, we assume the value 0.0195 pF for C_0 , obtained from the water-dioxane solutions. This compares well with the value 0.0220 pF theoretically obtained for this line⁶. This difference might arise from slight deviation of the line from "nominal" dimensions assumed in the calculations. It corresponds to a 10% difference in the phase angle of the reflection coefficient, which is likely to be significant in measurements on high permittivity fluids.

2. Electrical properties of the probe above 1 GHz. Figure 2 shows the reflection coefficient of the same probe, immersed in distilled water, at 1 GHz steps from 0-10 GHz. Also shown are reflection coefficients calculated from Eq. 2, using literature values for the complex permittivity of water. A significant deviation is observed, whose magnitude is as large as 0.16 at 10 GHz, that arises from radiation resistance⁶.

Figures 3A,B show the conductance and susceptance of the same probe vs. frequency, using a sample of distilled water. Also shown is the admittance calculated from Eq. 2. The difference, arising from radiation effects, is ca. 100% at 10 GHz (see Fig. 3A). Radiation effects are much smaller for the susceptance of the probes (Fig. 3B).

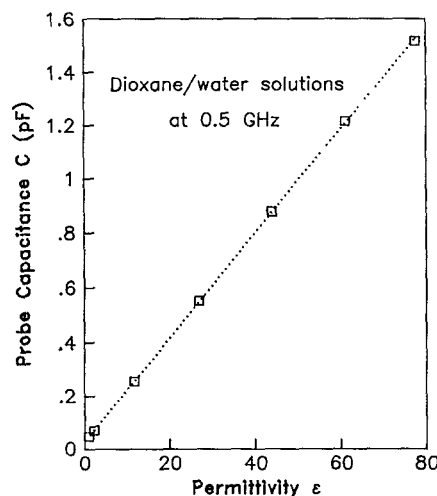
DISCUSSION

The probes we measured deviated from nominal properties for two reasons: small variations in their geometry, and radiation effects. The former are essentially nonreproducible, and vary with each probe. We estimate that a 10% uncertainty exists in the constant C_0 that will correspond, in measurements on high permittivity fluids, to variations in the reflection coefficient Γ of ca. 0.1 to 0.2 in magnitude above 1 GHz.

Radiation effects are more reproducible, and we propose an empirical method to take them into account.

1. Effect of Radiation on the Probe Impedance. Radiation effects are significant above 1 GHz with the 3.6 mm probe, when the probe is immersed in high dielectric constant fluids. The corresponding errors in Γ that would be introduced into the calibration of the ANA are as high as 0.16, for a 3.6 mm line at 10 GHz.

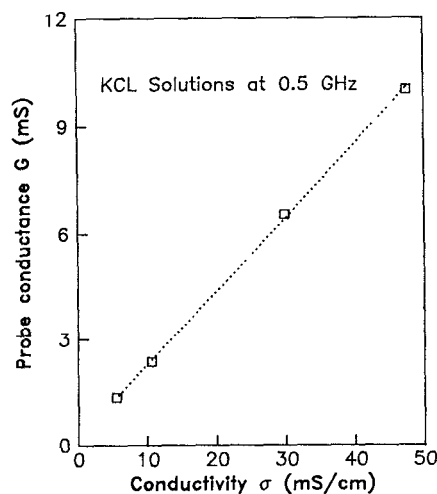
Fig. 1A Probe Calibration — Permittivity Standard



FIGURES

Fig. 1 A) Calibration of a 3.6 mm probe using dioxane-water mixtures of known permittivity ϵ^8 at 0.5 GHz. The best-fit slope, which is the probe's cell constant C_0 (see eq. 1), was found to be 0.0195. B) Plot of literature values of conductivity σ vs. measured conductance using same probe. The best-fit slope was 0.0203, which is sufficiently close to the capacitive cell constant.

Fig. 1B Probe Calibration — Conductance Standards



2. *Correction for Radiation Admittance.* Measurements were performed on fluids with a wide range of dielectric properties (distilled water, methanol, dioxane-water solutions). A simple empirical representation of the function G in Eq. 3 was found that adequately describes the radiation effects:

$$G(\epsilon^*, f) = 8.9 \times 10^{-9} f^3 (\text{REAL}[\epsilon])^2 \quad (4)$$

The admittance of one 3.6 mm probe, calculated from Eqs. 2, 3, and 4 with empirically obtained probe constants C_0 and C_f , is shown in Figs 3A. A more exact numerical correction, based on the approach of Mosig *et. al.*⁶, would be computationally inconvenient for such measurements.

3. Correction for Connector Artifact.

The time domain gating feature of the HP-8510 allows reduction in artifact from reflections from the connection between the probe and test cable of the ANA. The time-domain step response of the probe with its connector resembles that of an open circuit shunted by a small capacitance at the connector. In the frequency-domain, this leads to resonance-like artifacts in the reflection coefficient. Such artifacts can be largely removed by the time-domain gating method (Fig. 4), greatly reducing resonant-like effects in the data that can be mistaken for a property of the sample.

CONCLUSIONS

We conclude that the probe technique can introduce errors in the calibration of the ANA as large as 0.1-0.2 in the magnitude of the reflection coefficient. These errors are due to radiation effects and to variations in the electrical properties of the probe as it differs from the nominal dimensions and perfect geometry assumed in the calculations. These errors are 3-5 times larger than the variations in the probe reflection coefficient corresponding to the resonance of DNA solutions.

The effect of such errors is difficult to assess, since the two remaining standards (short and open circuit) are likely to be more accurate. Undoubtedly, however, it will lead to imperfect cancellation of stray reflections in the system, and be most disruptive when small variations in the properties of the sample must be observed, as with the DNA resonances. For measurements of relatively low precision, for which the probe technique was originally developed, such errors might be negligible. The methods we describe can avoid such errors and extend the usefulness of the probe technique to measurements of high precision.

ACKNOWLEDGEMENTS

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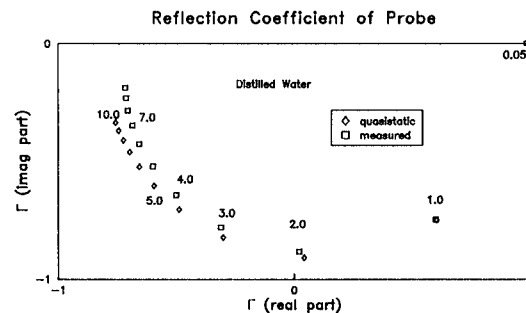


Fig. 2. Comparison of measured and theoretical reflection coefficients (calculated using Eqs. 1 and 2) over the range of measurement frequencies. At higher frequencies, the difference increases due to radiation effects. The frequency of each point is indicated in GHz.

Fig. 3A Admittance (3.6 mm probe, H₂O)

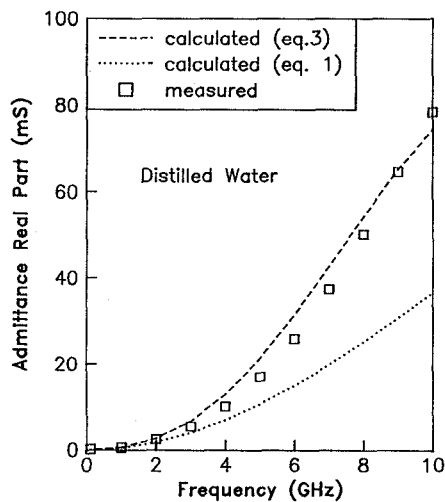
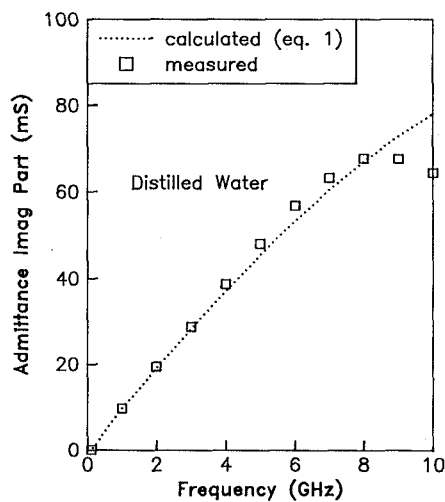


Fig. 3. Comparison of measured and calculated admittances (3A real part, 3B imaginary part) vs. frequency. The theoretical data were corrected to account for radiation effects using Eqs. 3 and 4.

Fig. 3B Probe Admittance (3.6 mm probe, H₂O)



Effect of Time-Domain Gating

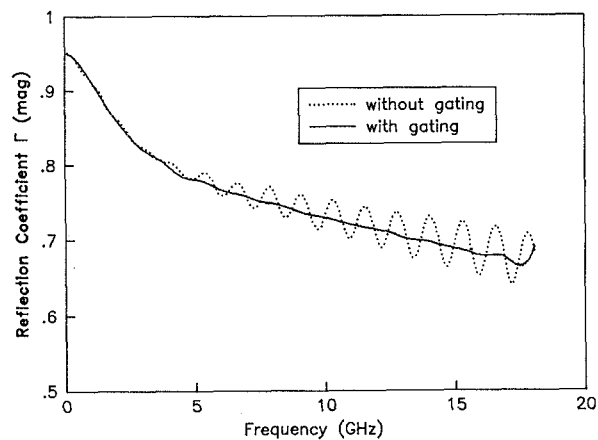


Fig. 4. Reflection coefficient response (magnitude) with and without gating, for probe immersed in dilute electrolyte solution (0.01 N NaCl). This shows resonant artifacts arising from reflections from the probe's connector, and the effect of time domain gating in removing them.