## TR-1332

# D64665 METHOD FOR EXTENSION OF DIELECTRIC CONSTANT AND LOSS MEASUREMENTS OF LIQUIDS TO 100 MHz WITH A FIXED-GEOMETRY SAMPLE HOLDER

by

Z. G. Sztankay

November 1966





DIAMOND LABORATORIES

WASHINGTON, D.C. 20438

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## METHOD FOR EXTENSION OF DIELECTRIC CONSTANT AND LOSS MEASUREMENTS OF LIQUIDS TO 100 MHz WITH A FIXED-GEOMETRY SAMPLE HOLDER

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#### ABSTRACT

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A method for measuring the dielectric properties of liquid specimens on a Q-meter at frequencies up to 100 MHz was developed by (1) assuming an equivalent circuit for a cylindrically shaped liquid-sample holder, (2) determining relevant circuit parameters by experiment, and (3) deriving the necessary equations taking full account of the effects of sample-bolder inductance.

Measurements made on two liquid samples are detailed and show that good accuracy is obtained when the standard liquid employed is a lossless dielectric with dielectric constant (K) near the K of the sample under test.

#### 1. INTRODUCTION

The effect of sample-holder inductance on dielectric constant and loss measurements in the megahertz frequency range was discussed in a previous report (ref 1). It was concluded in that report that the modified Hartshorn-Ward method in use with variable capacitance sample holders circumvents the errors due to the inductance. However, it was pointed out that with nonvariable sample holders one must correct for the inductance effects.

The liquid-sample holder used in this laboratory has no provision for changing capacitance. Thus one must measure the capacitance change used in the calculation for dielectric constant, K, by means of the change in the measuring-meter capacitance. At frequencies below 20 MHz this creates no problems; but at higher frequencies, where a Boonton Model 190-A Q-meter is used to indicate resonance, the sample-holder inductance affects the results. The loss calculation is also affected, since one cannot retune to the same capacitance values with and without sample.

#### 2. THEORY

#### 2.1 Summary of Previous Results

The basic equations for the dielectric constant and loss tangent, derived earlier (ref 1), will be summarized in brief. The dielectric constant, K, is given by (ref 1, eq 13)

$$K = \frac{\Delta C_e}{\beta_v^2 C_A} + 1$$
 (1)

where  $C_A$  is the air capacitance of the space in the sample holder that is filled with sample. The measured change in capacitance

of the sample holder upon insertion of the sample,  $\Delta C$ , is related, for small capacitance changes, to the actual change,  $\Delta C_p$ , by (ref 1, eq 4)

$$\beta^{2} \equiv \frac{\partial C_{e}}{\partial C_{p}} \sim \frac{\Delta C_{e}}{\Delta C_{p}}$$
(2)

The factor  $\beta$ , of course, is a function of the capacitance. For large capacitance changes, such as are usually involved in equation 1, this must be taken into consideration. Therefore we define  $\beta_v^2$  as (ref 1 eq 12)

$$\beta_{v}^{2} \equiv \frac{\stackrel{c}{e_{1}} - \stackrel{c}{e_{0}}}{\stackrel{c}{c_{1}} - \stackrel{c}{c_{p}}}$$
(3)

where the subscripts o and i refer to the capacitance with the sample out and in, respectively. Once we know the sample-holder equations, we can equate  $\beta^3$  to  $\beta^3$  at a particular capacitance value. This value will be between C and C (and C and C pi po).

The effective capacitance  $C_g$  is influenced by the sample-holder inductance at high frequency. It also includes parts of the sample holder not accessible to the sample, while  $C_i$  is the actual capacitance of the part containing the sample.

Provided the effective conductance of the whole measuring circuit,  $G_R$ , may be written in the form of equation 15 of reference 1, the loss tangent may be calculated from (ref 1, eq 22)

$$\tan \delta_{\rm X} = \frac{1}{\beta_{\rm i}^3 \omega K C_{\rm A}} \left[ \frac{\omega C_{\rm R}}{Q_{\rm o}} \left( \frac{Q_{\rm o}}{Q_{\rm i}} - 1 \right) - \Delta \text{ (other terms in } G_{\rm R} \right]$$
(4)

where the subscripts i and o refer to sample in and out, respectively;  $C_R$  is the total equivalent parallel resonant capacitance;  $G_R$  is the total equivalent parallel conductance; and Q is the Q of the equivalent parallel circuit. The second term in equation 4 refers to changes in  $G_R$ , upon inserting the sample, other than that due to the sample conductance. The resonant capacitance  $C_R$ is given by (ref 1, eq 14)

$$C_{R} = C_{e} + \alpha C_{Q}$$
 (5)

where C is the Q-meter tunable capacitance and  $\alpha$  is its inductance correction factor ( $\alpha$  is discussed in ref 2, and also defined in ref 1, eq 17).

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The dependence of equation 4 on  $C_{\rm R}$  and the absolute value of Q (rather than just the ratio  $\bar{\rm Q}/\bar{\rm Q}_{\rm l}$ ) may be eliminated by relating them to the difference in capacitance of the two halfpower points, as measured by the change in  $C_{\rm Q}$  with sample out,  $\Delta C_{\rm Qol/2}$  (ref 1). The equation for the loss tangent then becomes (ref 1, eq 27).

$$\tan \delta_{x} = \frac{1}{\beta_{1}^{2} \varpi K C_{A}} \begin{bmatrix} \alpha_{0}^{2} \varpi \left| \Delta C_{Q_{0} \frac{1}{2}} \right| \\ -\frac{2}{2} & \left( \frac{Q_{0}}{Q_{1}} - 1 \right) - \Delta \text{ (other terms in } G_{R} \end{bmatrix}$$
(6)

where the subscripts i and o on the parameters  $\beta$  and  $\alpha$  indicate the capacitance values (with sample in and out, respectively) at which the parameters  $\beta$  (eq 2) and  $\alpha$  (ref 1, eq 17) are to be taken.

#### 2.2 Sample Holder Equivalent Circuit

To use the equations for the dielectric constant and loss, it is necessary to know the equivalent circuit of the sample holder. A drawing of the holder is shown in figure 1. The equivalent circuit chosen to represent the holder was one in which the inductance, conductance, resistance, and capacitance are evenly distributed throughout, as shown in figure 2. In the case of the capacitance, the total capacitance, C, is  $C = C_{p} + C_{p}$ , where C is the very small constant capacitance not influenced by the sample.

The circuit shown in figure 2 is also the equivalent circuit of a transmission line. The input impedance may thus be obtained from transmission line theory (ref 3). In our case it is more convenient to use its reciprocal, the admittance

$$Y_{e} = \frac{1}{Z_{o}} \left[ \frac{Z_{o} \cosh \Gamma + Z_{R} \sinh \Gamma}{Z_{R} \cosh \Gamma + Z_{o} \sinh \Gamma} \right]$$
(7)

where Z , the characteristic impedance, equals  $\sqrt{Z/Y}$ , and  $\Gamma$ , related to the propagation constant of transmission line theory, is given by  $\sqrt{ZY}$ . Z is the total series impedance, Z = R+jwL, and Y is the total parallel admittance, Y=G+jwC. The parameters R, L, G, and C, like Z and Y, are defined here to be the total resistance, inductance, conductance, and capacitance for the sample holder rather than the per unit length quantities used in transmission line theory. The load impedance Z<sub>R</sub> should be taken to be an open circuit, as this is most consistent with the physical situation. Thus equation 7 reduces to



conductor





insulator

Figure 1. Cross-sectional view of liquid sample holder.



Figure 2. Assumed equivalent circuit of sample holder. There are n repeating sections, each having the circuit parameters C/n, G/n, L/n and R/n with  $n \rightarrow \infty$ . The total capacitance, conductance, inductance and resistance equals C, G, L and R, respectively.

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$$Y_{e} = \frac{1}{2_{o}} \tanh \Gamma$$
 (8)

We may assume that the series resistance is small,  $mL \gg R$ , and that the parallel conductance is also small,  $mC \gg G$ . The former assumption is always good in the frequency range of interest ( $\geq 10$  MHz), and the latter would come into question only for very lossy samples, as it is equivalent to the condition that tan  $\delta \ll 1$ . With the above assumptions we obtain

$$\frac{1}{\overline{z}} \sim \sqrt{\frac{C}{L}} - j \frac{1}{2\pi} \sqrt{\frac{C}{L}} \left(\frac{G}{C} - \frac{R}{L}\right)$$
(9)

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$$\Gamma \sim j = \sqrt{LC} + \frac{\sqrt{LC}}{2} \left( \frac{G}{C} + \frac{R}{L} \right)$$
(10)

The substitution of equations 9 and 10 into equation 8 yields

$$\mathbf{F}_{e} = \mathbf{j} \int \overline{\mathbf{L}} \tan w \sqrt{\mathbf{I}} \overline{\mathbf{L}} - \frac{\mathbf{j} \mathbf{T} \mathbf{C}}{4 \cos^{2} w \sqrt{\mathbf{I}} \overline{\mathbf{C}}} \left[ \left( \frac{\mathbf{G}}{\mathbf{w}} \right)^{2} - \left( \frac{\mathbf{R}}{\mathbf{w}} \right)^{2} \right] \\ + \frac{\mathbf{G} + \mathbf{R} \mathbf{C} / \mathbf{L}}{2 \cos^{2} w \sqrt{\mathbf{I}} \overline{\mathbf{C}}} + \frac{1}{2w} \int \overline{\mathbf{C}} \left( \frac{\mathbf{G}}{\mathbf{C}} - \frac{\mathbf{R}}{\mathbf{L}} \right) \tan w \sqrt{\mathbf{I}} \overline{\mathbf{C}}$$
(11)

Under the assumptions mentioned earlier, the second imaginary term is negligible compared with the first, at least at frequencies of interest. Changing the form of the first imaginary term slightly, and extanding the trigonometric functions in the real terms, keeping three terms in the expansions, and combining, we obtain

$$Y_{e} \sim j = C \frac{tan = \sqrt{JC}}{= \sqrt{LC}} + G \left[ 1 + \frac{2}{3} = 2LC + \frac{6}{15} (=2LC)^{2} + \cdots \right] + \frac{R}{3} (=C)^{2} \left[ 1 + \frac{4}{5} = 2LC + \cdots \right]$$
(12)

The equivalent parallel capacitance of the liquid sample holder is, therefore

$$C_{c} = C \frac{t z_{2} \oplus \sqrt{1C}}{\pi \sqrt{1C}}$$
(13)

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$$C_{a} \sim C \left[ 1 + \frac{1}{3} \omega^2 LC + \frac{2}{15} (\omega^2 LC)^2 + \cdots \right]$$
 (14)

and the equivalent parallel conductance is

$$G_{e^{\sim \omega}} \left[ 1 + \frac{2}{3} \omega^2 LC + \frac{6}{15} (\omega^2 LC)^2 + \cdots \right] + \frac{R}{3} (\omega C)^2 \left[ 1 + \frac{4}{5} \omega^2 LC + \cdots \right] (15)$$

The factor  $\beta^2$ , defined by equation 2, is obtained by differentiating equation 14 with respect to  $C_p$ , with  $C = C_p + C_n$ :

$$\beta^2 \sim 1 + \frac{2}{3} \omega^2 LC + \frac{6}{15} (\omega^2 LC)^2 + \cdots$$
 (16)

This is precisely the term multiplying G in equation 15. Further, from equation 14, we may write

$$\left(\frac{\frac{C}{e}}{C}\right)^{2} \sim 1 + \frac{2}{3} \omega^{2} LC + \frac{17}{45} (\omega^{2} LC)^{2} + \cdots$$
(17)

This is almost exactly equal to  $\beta^2$ , and at least approximately equal to the term multiplying R/3( $\omega$ C)<sup>2</sup> in equation 15.

The equivalent parallel conductance of the sample holder may thus be written as

$$G_{e} \sim \beta^{2} G + \frac{R}{3} (\omega C_{e})^{2}$$
(18)

The total effective conductance of the measuring circuit is therefore in the form of equation 15 of reference 1, with  $G_p \rightarrow G$ ,  $G_n = R_p = 0$ , and  $R_p \rightarrow R/3$ . (The last four terms in that equation are the Q-meter conductance terms.) Thus the loss tangent may be calculated from equation 4 or 6. Next, to find  $\beta$  (equation 3), we take the difference of equation 14 between  $C_p = C_{pi}$  and  $C_p = C_{po}$ , remembering that  $C = C_p + C_n$ , and obtain

$$\frac{C_{ei} - C_{eo}^{2}}{C_{pi} - C_{po}^{2}} = 1 + \frac{1}{3} \omega^{2} L \left[ \frac{C_{pi}^{2} - C_{po}^{2}}{C_{pi} - C_{po}} + 2C_{n} \right] + \frac{2}{15} (\omega^{2} L)^{2} \left[ \frac{C_{pi}^{3} - C_{po}^{3}}{C_{pi} - C_{po}} + \frac{3C_{n}^{2} (C_{pi}^{2} - C_{po}^{2})}{C_{pi} - C_{po}} + 3C_{n}^{2} \right] (19)$$

If we define  $C_{AV}$  by

4

$$C_{Av} \equiv \frac{C_{i} + C_{o}}{2} = \frac{C_{pi} + C_{po}}{2} + \tilde{c}_{n}$$
 (20)

we find that equation 19 may be written exactly through the first two terms, and to a good approximation in the third term, as

$$\beta_v^2 \sim 1 + \frac{2}{3} \omega^2 LC_{Av} + \frac{6}{15} (\omega^2 LC_{Av})^2 + \cdots$$
 (21)

Comparing equation 21 with equation 16, we see that  $\beta_v^2 = \beta_{Av}^2$ , the value of  $\beta^2$  at  $C = C_{Av}^2$ .

To sum up the results of this section, we write down the following rpproximate relationships:

$$C_{e} \sim \beta C = \beta (C_{p} + C_{n})$$
(22)

$$G_{e} \sim \beta^{2}G + \frac{R}{3} (\omega C_{e})^{2}$$
(18)

$$K = \frac{\Delta C_{e}}{\beta_{Av} C_{A}} + 1$$
 (23)

and

$$\sim \frac{\tan \omega \sqrt{LC}}{\omega \sqrt{LC}}$$

$$\sim 1 + \frac{1}{3} \omega^{2} LC + \frac{2}{15} (\omega^{2} LC)^{2} + \cdots$$

$$(24)$$

#### 2.3 Discussion of Sample-Holder Equations

Theoretically, we should be able to use the equations obtained in the provious section to correct for the inductance error once we have found the value of L. However, we could still not find the correction factor to  $\Delta C_e$ ,  $\beta_{Av}^2$ , without first knowing C. Furthermore, the equivalent circuit is, of course, only approximate, so it is doubtful that the equation for  $\beta_{Av}$  is usable in such a direct way.

If very many measurements are to be made with the same liquid sample holder, the best procedure would be to find  $\beta$  as a function of C from equation 22, using standard liquids whose dielectric constants are known. We could then make plots of  $\beta$ 

versus C at the frequencies of interest, and refer to these plots when making corrections. It may well prove worthwhile to do this, but for the time being, an alternate method, outlined in the next section, was chosen.

Whatever method of finally correcting for the inductance errors is used, one very significant reduction in error may be made by using a standard liquid with dielectric constant known and close to that of the sample for the sample-out measurement. If K is the dielectric constant of the standard, the dielectric constant of the unknown will be given by

$$K = \frac{\Delta C_{e}}{\beta_{Av} c_{A}} + K$$
(25)

instead of equation 23. If K is close to K, the same percentage error in  $\beta_{Ay}^{2}$  will have much less significance than if equation 23, which is really the special case of equation 25 with K = 1, is used.

#### 2.4 Procedure for Calculating K

The dielectric constant is to be calculated from equation 25. In that equation,  $C_A$  and K are known, and  $\Delta C_c$  can be readily measured. Since  $C_R$  is constant, equation 5 yields

$$\Delta C_{e} = C_{ei} - C_{eo} = \alpha_{o} C_{Qo} - \alpha_{i} C_{Qi}$$
(26)

where the subscripts i and o refer to sample in and out (standard in), respectively. The function  $\alpha(C_{0})$  may be obtained from the Q-meter manual (rcf 2). A plot of  $\alpha$  as a function of frequency and C<sub>0</sub>, taken from the manual , is shown in figure 3 of this report, however, for convenience. The equation for  $\alpha$  is also given in figure 3. The function  $\beta_{Av}$ , however, is not known. If the change in capacitance  $\Delta C_{0}$  is very small, we can use the  $\beta$  appropriate to the standard,  $\beta_{0}$ , as an approximation to  $\beta_{Av}$ . The factor  $\beta$  can be found from equation 22 with C = C and  $C_{0} = C_{0}$ . C is known and equation 5 gives C once C has been determined (discussed later).

When  $\Delta C$  is large, we cannot equate  $\beta_{AV}$  to  $\beta_{O}$ . With some approximations, however, we find  $\beta_{AV}$  from  $\beta_{O}$ . First, to find  $C_{AV}$ , for use in the correction term,

$$C_{AV} \sim \frac{C_{eAV}}{\beta_{o}} = \frac{1}{2} \left( \frac{C_{e1}}{\beta_{o}} + C_{o} \right)$$
(27)



Figure 3.  $\alpha$  as a function of frequency (from ref 6 p 16).

All the terms on the right in equation 27 are known or measurable. Second, we note that  $\beta^2$  and  $\beta^2_{Av}$  are given by equation 16 with C = C and C = C, respectively. We see from that equation that, approximately,

$$\beta_{A_{\chi}}^{2} \sim \left[1 + (\beta_{O} - 1) \frac{C_{AV}}{C_{O}}\right]^{2}$$
(28)

Equation 28 is incorrect only in the third term of  $\beta_{A_{yy}}$ , as

1+ 
$$(\beta_0^{-1}) \frac{C_{Av}}{C_0} = 1 + \frac{1}{3} \omega^2 LC_{Av} + \frac{2}{15} (\omega^2 L)^2 C_{Av} C_0^{+\cdots}$$
 (29)

By examining equations 27 and 29, we see that if  $C_{i} > C$  the first approximation causes an overcorrection as  $C_{AV}^{i}$  comes out too large (since in this case  $\beta_{i} < \beta_{i}$ ), while the second one caused an under correction, as in the third term of  $\beta_{AV}^{2}$ ,  $C_{o}$ , which is in this case less than  $C_{AV}$ , appears in place of  $C_{AV}^{2}$ . If  $C_{i} < C$ , the reverse takes place. Thus the two errors have a partially cancelling effect.

For calculation purposes, we may eliminate some of the steps by substituting C /C for  $\beta$  in equation 27, and the result into equation 28. After rearranging, we find that

$$\beta_{Av}^{2} = \frac{1}{4} \left[ 1 + \frac{C_{eo}^{+C}e_{i}}{C_{o}} - \frac{C_{ei}^{-1}}{C_{eo}^{-1}} \right]^{2}$$
(30)

Equation 30 may then be used directly in equation 25 for calculation of the dielectric constant

#### 2.5 Procedure for Calculating Tan $\delta$

The loss tangent may be calculated from either equation 4 or 6. For either equation, we need to know  $\beta_i$ , but that is now easily calculated. First, we obtain  $C_i$  from

$$C_{i} = C_{o} + \frac{\Delta C_{e}}{\beta_{Av}^{2}}$$
(31)

Thus  $\beta_i$  is given by

$$\beta_{i} = \frac{C_{ei}}{C_{i}} = \frac{C_{ei}}{C_{o} + \Delta C_{e} / \beta_{Av}^{2}}$$
(32)

All quantities on the right in equation 32 are known.

Next we must consider the subtractive term in the tan  $\delta_{\rm c}$  equations. We must, therefore, write down the total parallel conductance, including the measuring meter conductances. With the Boonton Model 190-A Q-meter, or a meter with a similar equivalent circuit, the total conductance is

$$G_{R} = \beta^{2}G + \frac{R}{3}(\omega C_{e})^{2} + \alpha^{2}G_{v} + \alpha^{2}(\omega C_{Q})^{2}R_{c} + G_{d} + (\omega C_{R})^{2}R_{m}$$
(33)

The first two terms are from equation 18, and the last four are the Q-meter conductance terms. The latter are given in reference 2, and their method of derivation is discussed in reference 1. The terminology differs only in that the combination of  $R+R_1$ in the manual is here (and in ref 1) labeled  $R_m$ , and the C of the manual is here (and in ref 1) called  $C_0$ .

If we assume that the sample-holder conductance is zero with the sample out, and that we may neglect the small change due to the difference between  $\alpha$  and  $\alpha$ , in the already small G, term, and if we remember that  $C_R$  does not change, we see that

$$\Delta \text{ (other terms in } G_{R}) = \frac{R}{3} \omega^{2} (C_{ei}^{3} - C_{eo}^{2}) + R_{c} \omega^{2} (\alpha_{i}^{2} C_{Qi}^{2} - \alpha_{o}^{2} C_{Qo}^{2})$$
(34)

All terms on the right-hand side are known except for R, which must be determined by experiment. This is best done by making measurements, first with the sample holder removed (denoted by subscript o) and subsequently with the empty sample holder, or sample holder with a lossless dielectric, in the circuit (subscript i). In this case tan  $\delta_x = C_{eo} = 0$ ; therefore equations 4 and 34 yield

$$\frac{R}{3} - \frac{1}{C_{ei}^{2}} \left[ \frac{C_{R}}{\omega Q_{o}} \left( \frac{Q_{o}}{Q_{i}} - 1 \right) + R_{c} \left( \alpha_{o}^{2} C_{Qo}^{2} - \alpha_{i}^{2} C_{Qi}^{2} \right) \right]$$
(35)

The meter contact resistance R is obtained from the manual (ref 2) as

$$R_{c} \sim 0.0004 \sqrt{F_{MHz}} \Omega \qquad (36)$$

when  $\mathbf{F}_{\underline{MHz}}$  is the frequency in megahertz. Once equation 35 has been used to obtain R as a function of frequency for a sample holder, this value of R may be used in equation 34 and equation 4 or 6 to find tan  $\delta_{\underline{v}}$  for a sample.

As with the K measurement, we may reduce the error by filling the sample holder with a standard dielectric for the sample-out measurement. If the standard has a K similar to the unknown,  $C_{ei}$  will not be too different from  $C_{eo}$  (and  $C_{o}$  from  $C_{o}$ ) and, hence, the magnitude of the correction terms will be reduced. Presumably one would use the same standard for this calculation as for the K calculation, as then no additional measurement need be performed. This imposes one further requirement for the standard—that it be lossless.

#### 2.6 Determination of $C_R$

The total resonant capacitance of the circuit,  $C_R$ , is needed in both the dielectric constant and loss tangent calculation. In the K calculation, however,  $C_R$  is needed only in the calculation for the inductance correction term  $\beta_{A_V}$ . Specifically, equation 5 is used to calculate the effective capacitances, and it involves  $C_R$ . Equation 5 itself affords the best way of calculating  $C_R$  for this purpose, by determining  $C_Q$ with the sample holder not attached (i.e., with  $C_e = 0$ ). With  $C_R$  determined in this manner, the effective capacitance,  $C_e$ , is really measured as the difference between two  $C_Q$  readings, suitably corrected by  $\alpha$ .

However, because of the limited range of the C<sub>Q</sub> dial, and the large sample and standard effective capacitances, especially at high frequencies, it is not always possible to resonate with the coil only at the same frequency as with the sample and standard. In this case, one should resonate with coil only at a frequency,  $\omega_{c}$ , as close as possible to the measurement frequency, and find the equivalent-circuit resonant inductance in parallel with C<sub>R</sub> and G<sub>R</sub>, L<sub>R</sub> (mostly due to the particular work coil being used, but including L<sub>i</sub>, defined in the manual) from

$$L_{R} = \frac{1}{\omega_{c}^{2}C_{Q}} - L_{c}$$
(37)

The internal inductance correction is taken care of by the subtraction of L, which is given in the manual ( $L \sim 0.0014 \mu h$ ) (ref 2). The resonant capacitance at the measurement frequency is then obtained from

$$C_{R} = \frac{1}{\omega^{2} L_{R}}$$
(38)

In the loss calculation,  $C_R$  is an integral p. of the calculation. However, as discussed earlier, it may be related to the width of the resonance curve, and thus eliminated from the equation, along with Q. It is mainly on the basis of eliminating Q, rather than the absolute value of  $C_R$ , that it is recommended that the loss be calculated using equation 6 instead of equation 4. However, it should be noted that this equation gives tan  $\delta_R$  mainly as a function of capacitance differences, rather than absolute values, as well as voltage ratios. The absolute values do appear in the correction term, given by equation 34, in which the C 's are already known, as they were determ ned for the dielectric constant calculation.

#### 3. EXPERIMENTAL PROCEDURE AND RESULTS

#### 3.1 Validity of Equations

Table I gives low-frequency and effective capacitances measured to check the validity of the proposed sample-holder equations. Data were taken at various frequencies for the empty sample holder and holder filled with benzene. Benzene is essentially lossless and has a constant K for frequencies up to and beyond those used in these measurements.

Capacitance bridge measurements were made at 100 kHz to determine the true capacitances C and C of the empty and benzene-filled sample holder, respectively.<sup>1</sup> The Q-meter was used for high-frequency determinations, at 22, 50, 78, 85, and 92 MHz, of C and C. These quantities were calculated using equation 5, with C being determined by resonating the meter with the sample holder not attached. For the 92-MHz point, equations 37 and 38 had to be used. For that reason the results at that frequency are less precise.

Quantities calculated from table I are given in table II. The ratio C /C gave  $\beta$  and allowed the calculation of L from equation 24. The expanded form of that equation was found convenient in this case, as it converges rapidly for small C. The results for L differ somewhat for the different frequencies, with an average value of L = 0.050  $\mu$ K. The differences may well be explained by the inaccuracies of the absolute capacitance measurements with the Q-meter. The value of L obtained for a given frequency was then used, along with C<sub>i</sub>, to calculate  $\beta_i$ from equation 24, this time using the form of the equation involving the tangent. This gave a calculated C that could be compared with the experimentally determined C<sub>ei</sub>. In all cases, the experimental C was higher than the predicted one. This suggests that L, as determined from data for the empty sample holder, does not apply exactly to the benzene-filled sample holder.

TABLE I	-MEASURED	CAPACITANCES	(pF)	IN	CHECK	OF	EQUIVALENT	CIRCUIT
---------	-----------	--------------	------	----	-------	----	------------	---------

f, MHz	22	50	78	85	92
c	26.4	26.4	26.4	26.4	26.4
C,	56.2	56.2	56.2	56.2	56.2
Ċ	26.4	27.3	29.7	31.2	31
C ei	57.4	63.7	82.5		98

TABLE II.-CALCULATED QUANTITIES FROM EQUIVALENT CIRCUIT AND TABLE I

f, MHz	22	50	78	85	92
βο	-	1.034	1,125	1.182	1.17
L,µH	-	0.039	0.052	0.060	0.05
₿ <sub>i</sub>	-	1.08	1,33	-	1.53
C <sub>ei</sub> , pF	-	60.7	74.9	-	85.7

TABLE III. --- EFFECTIVE CAPACITANCES (pF) CALCULATED FROM LOW-FRE-QUENCY DATA OF TABLE I AND CIRCUIT EQUATIONS WITH  $L = 0.060 \mu H$ 

f, MHz	22	50	78	85	92
C <sub>ei</sub>	57.4	53.4	78.7	85.6	94.7
Ceo	26.7	27.9	30.4	31.3	32.3

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If a value of  $L = 0.060 \ \mu H$  is chosen, the predicted  $C_{ei}$ 's, table III, are quite close to the experimental values. The value of 0.060  $\mu H$  is not very far from the actual average value of 0.050  $\mu H$ . In fact, the experimental value at 85 MHz was 0.060  $\mu H$  (unfortunately, no benzene data were taken at that frequency). The higher value of L, however, when used in equation 14 with C to calculate the predicted C 's, results in a little too high a value of the C 's.

It should be remembered in assessing the results that these comparisons are between capacitances that differ by more than a factor of two. The inexactness of the corrections are probably due to the approximate applicability of the equivalent circuit, which led to the circuit equations. The proposed correction method for samples is based on the same equations, but comparisons are to be made on capacitances that are relatively close to each other. The results confirm that the method should not be used except with a standard that does not differ widely in dielectric constant from the sample.

#### 3.2 Determination of R

The sample-holder series resistance R was determined using equation 35. Measurements were made of the circuit Q's and meter capacitances needed to resonate with the sample holder not attached, with it attached but empty, and with it attached and filled with benzene. The Q-meter readings were corrected for internal effects as described in the manual (ref 2) and the precursor of this report (ref 1, equation 16). Frequencies used were 22, 50, 78, and 85 MHz.

R was calculated at each frequency, first with the empty-sample-holder Q's and C<sub>Q</sub>'s (subscript i) being compared with the sample-holder-removed Q's and C<sub>Q</sub>'s (subscript o). The results are shown in figure 4.

The resistances are plotted against the square root of the frequency because of the expected linear relationship, due to the skin effect. The large scatter of the points is caused by subtraction of two similar numbers in both terms of equation 35. This is especially true in the first term, as  $Q_0/Q_1$  is very close to unity. especially at the lower-frequency points, which are therefore quite unreliable.

Putting benzene, which is essentially lossless in this frequency range, into the sample holder alleviated this problem. With benzene,  $Q_i$  and  $C_{Qi}$  were considerably reduced, so that the subtractive terms in equation 35 became relatively smaller compared with the additive terms. It should be mentioned parenthetically, however, that the reduction in  $Q_i$ , which is the circuit  $Q_i$  is not due to loss in the benzene, but rather to the increase in the capacitance  $C_{ei}$ .



The results obtained with benzene are also plotted against the square root of the frequency in figure 4. The points fall very nicely on a straight line through the origin, showing the skin effect. If  $F_{\rm WHZ}$  represents the frequency in megahertz, then R may be written as

$$\frac{R}{3} = 0.007 \sqrt{F_{MHZ}} \Omega$$
(39)

Figure 4 would seem to justify having one more significant figure in this expression (if we consider only the points with benzene in the holder), but if we remember the approximate nature of the equation used in calculating the R's, we see that one significant figure is all that is justified. By comparing with equation 36, it is seen that the sample-holder resistance is considerably higher than the Q-meter resistance, so that the R term in equation 34 is usually negligible. This, however, would not be true if the conditions of measurement were such that the C's were considerably higher than the C's.

Equations 39 and 36 may be used in equation 34, and the latter substituted into equation 6 (or equation 4). The final form of t tan  $\delta$  equation using the capacitance variation method for  $C_p/Q_o$  (i.e., equation 6) then becomes

$$\tan \delta_{\mathbf{x}} = \frac{1}{\beta_{i}^{2} K C_{A}} \left[ \frac{\alpha_{o}^{2} | \mathcal{L} C_{Q_{0}}|_{\mathcal{L}}}{2} \left( \frac{Q_{0}}{Q_{i}} - 1 \right) - 4 \times 10^{-8} F_{MEZ}^{\frac{3}{2}} (C_{ei}^{2} - C_{eo}^{2}) - 2 \times 10^{-9} F_{MEZ}^{\frac{3}{2}} (\alpha_{ei}^{2} C_{Q_{i}}^{2} - \alpha_{o}^{2} C_{Q_{i}}^{2}) \right]$$

$$(40)$$

The capacitances in this equation must be in picofarads, and  $F_{\text{WHz}}$  is the frequency in megabertz. The last term, as mentioned, is usually negligible.

#### 3.3 Results with Two Samples

Dielectric constant and loss calculations using these methods have been made on several liquids. The calculation of two samples, C-methylstyrene and poly-C-methylstyrene, at 50 and 78 MHz, will be discussed, and the results compared with low-frequency measurements. Data with very similar samples were taken up to 90 MHz, with similar results, but those data were not taken by the author of this report and will therefore not be discussed here.

The data and calculated qualcities are given in table The first section gives the Q-meter capacitance measure-IV. ments, corrected for the internal inductance; the effective capacitances calculated from them; the effective capacitance change,  $\Delta C_{i}$ ; and the low-frequency capacitance of the sample holder with the standard (benzene) measured on a capacitance bridge at 100 The next section gives the necessary quantities in the K kHz . determination, calculated as outlined earlier. The long method was used, instead of equation 30, to more clearly indicate the magnitudes of the terns involved. Using a value of 23.5 pF for  $C_{k}$ , also determined earlier at low frequency, and K = 2.28 for the dielectric constant of benzene at 25°C, the results for K are 2.58 and 2.54 for the monomer and polymer, respectively, at both frequencies.

The parameters of the loss calculations are given in the third section of table IV. The value of  $\beta_1$  was calculated from equation 32. The Q's listed in the table have been corrected for the internal errors (ref 2). The next entry of the table gives the total loss term; and this is followed by the two correction terms. The total loss term in this case was calculated using  $C_R/Q_0$  directly (i.e., equation 4 instead of 6 was used). The second correction term is negligible in all these cases, but the first is substantial, especially for the lower loss monomer. The tan  $\delta_1$ 's for the two frequencies are substantially the same, but differ considerably for the two samples.

The results are plotted as a function of frequency in figure 5, along with results obtained at 22 MHz with the Q-meter (these were not outlined in table IV as the corrections at that frequency are quite small), and lower-frequency points using a capacitance bridge. The results show, as was expected, that the monomer has a constant K and tan  $\delta$  up to  $10^8$  Hz, while the K of the polymer shows a drop, with a corresponding peak in the loss. The high-frequency results are quite consistent with the lowfrequency measurements. This is true despite the inductance corrections which are quite large, especially at 78 MHz.

#### 4, CONCLUSIONS

It is concluded that the procedures for calculating the dielectric constant and loss that have been outlined give reasonably good results up to ~100 MHz. Of course, the accuracy of K will depend on how close the dielectric constants of the standard and sample are. We can always reduce the error by finding, or mi\_ng, a standard whose dielectric constant is closer to that of the sample. For the loss, results will be reasonable for  $0.1 > \tan \delta$ > 0.001. The upper limit originates from assumptions made in

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Relevant equation	Parameter	Applicable specimen	Monomer	Polymer	Monomer	Polymer
	F, MHz	sample & standard	50	50	78	78
	C <sub>R</sub> , pF	no holder	87.1	87.1	110.5	110.5
	$\alpha_{o}^{C}_{OO}, pF$	standard	23.4	23.4	27.9	27.9
	$\alpha_{1}C_{Q1}$ pF	sample	14.2	15.5	11.4	13.9
:	C <sub>o</sub> , pF	standard	56.3	56.3	56.3	<b>56</b> .3
(5)	C <sub>eo</sub> , pF		63.7	63.7	82.6	82.6
(5)	C <sub>ei</sub> , p <del>y</del>		72.9	71.6	99.1	96.6
(26)	∆C <sub>e</sub> , pF		9.2	7.9	16.5	14.0
(22)	β		1.13	1.13	1.47	1.47
(27)	C <sub>Av</sub> , pF		60.4	59.8	61.9	61.0
(28)	β <sub>Av</sub> <sup>2</sup>		1.30	1.30	2,30	2.28
	∆C <sub>o</sub> , pF		7.08	6.08	7.17	6.14
	C <sub>A</sub> , pF		23.5	23.5	23.5	23.5
	∆c∕c <sub>A</sub>		0.30	0,26	0.30	0 ,26
	к	standard	2.28	2.28	2.28	2.28
(25)	К	sample	2.58	2.54	2.58	2.54
(31)	C <sub>i</sub> , pF		63.4	62.4	63.5	62.4
(32)	β <sub>i</sub>		1.15	1.15	1.56	1.55
	β <sub>i</sub> ²		1.32	1.32	2.44	2.39
	۹ <sub>0</sub>	standard	226	226	194	194
	Q <sub>1</sub>	sample	185	145	138	114
	$\frac{C_{R}}{Q_{o}}\left(\frac{Q_{o}}{Q_{i}}-1\right)$ , pF		0.085	0.22	0.23	0.40
	$w_{\overline{3}}^{R}$ (C <sup>2</sup> -C <sup>2</sup> ), pF		0.02	0.02	0.08	0.07
	$\omega R_{c} (\alpha_{1}^{3} C_{O1}^{3} - \alpha_{0}^{2} C_{O0}^{2}), pF$		-0.0003	-0.0003	-0.001	-0.001
	$\tan \delta_{\mathbf{x}}(\beta_{1}^{2}KC_{A}), pF$		0.065	0.20	0.15	0.33
	$\beta_1^2 KC_A$ , pF		80.2	78.7	147.8	142.9
(40)	tan ô <sub>x</sub>	sample	0.0008	0.0025	0.0010	0.602
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TABLE	IVOUTLINE	FOR	DIELECTRIC	CONSTANT	AND	LOSS	TANGENT	CALCULATIONS	FOR	Q-METHYLSTYP	<b>GENE</b>

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. • 3 • • deriving the equivalent circuit, and the lower limit comes from the size of the sample holder correction term. The latter limit is very approximate, as it depends on the circuit capacitances. Whether one can assign an actual number to the loss tangent, or only make the statement that it is smaller than a certain number, will depend on the individual circumstances. The loss calculation also includes the large multiplicative  $\beta_1^2$  correction. It is, therefore, considerably less accurate than the dielectric constant determination.

It should be possible to make measurements at somewhat higher frequencies, perhaps in the 100-to-200-MHz range, by using smaller capacitance sample holders, as the correction term goes as powers of  $1/3\omega^2 LC$ . Some experimentation on this has been done. It would, however, be difficult to go to still higher frequencies with this technique. An obvious upper frequency limit is, of course, reached when the expansions used no longer converge, but the method should certainly be abandoned before that limit is reached. One should judge, in each instance, from the size of  $\beta$ , how much, if any, significance should be attached to the results.

#### 5. PROCEDURE FOR HIGH-FREQUENCY MEASUREMENTS

To make a measurement with the liquid sample holder at fraquencies greater than 20 MHz, use Q-meter 190-A and follow the procedure outlined below.

First, choose a standard liquid with negligible loss compared with that of the sample and a known, constant-with-frequency, dielectric constant as close to that of the sample as is available. (For low K samples this standard dielectric may be air.) If K is not known even approximately, various standards may have to be tried. Then measure the following quantities:

C<sub>QR</sub> - the resonant C with the sample holder not attached (in rare cases, this may be obtained only at a frequency different from the measuring frequencythen see discussion by equations 37 and 38).

 $C_{00}$  - The resonant  $C_{0}$  with the standard in the holder.

Q - the resonant circuit Q with the standard (unless extreme accuracy is desired, and seems justified, the Q's may be taken directly as the meter readings for frequencies up to 100 MHz, as only their ratio is used; if deemed advisable, correct as described in manual.)

$$C_{Q0+}$$
 - the  $C_Q$  above  $C_{Q0}$  where  $Q = Q_0 \sqrt{2}$   
 $C_{Q0-}$  - the  $C_Q$  below  $C_{Q0}$  where  $Q = Q_0 \sqrt{2}$   
 $C_{Q1}$  - the resonant  $C_Q$  with the sample in the holder  
 $Q_1$  - the resonant circuit Q with the sample  
Next, obtain from the manual, or figure 3 of this report:

 $\alpha_{\rm R}$  - the internal correction factor (for L<sub>c</sub>) with  $C_{\rm Q} = C_{\rm QR}$  $\alpha_{\rm o}$  - the internal correction factor with  $C_{\rm Q} = C_{\rm Qc}$  $\alpha_{\rm i}$  - the internal correction factor with  $C_{\rm Q} = C_{\rm Qi}$ 

Now calculate:

$$C_{R} = \alpha_{R}C_{QR}$$

$$C_{eo} = C_{R} - \alpha_{o}C_{Qo}$$

$$C_{ei} = C_{R} - \alpha_{i}C_{Qi}$$

$$\Delta C_{e} = C_{ei} - C_{eo}$$

$$\Delta C_{Qo2} = C_{Qo+} - C_{Qo-}$$

Further, obtain from notebook, references, or low-frequency measurement:

 $C_{0}$  - low-frequency capacitance of sample holdsr with standard  $C_{A}$  - air capacitance of portion of holder that is accessi-

A ble to the liquids

🤞 - dielectric constant of the standard

Now calculate:

$$\beta^{2}_{Av} = \frac{1}{4} \left[ 1 + \frac{C_{eo} + C_{ei}}{C_{o}} - \frac{C_{ei}}{C_{eo}} \right]^{2}$$

$$K = \frac{\Delta C_{e}}{\beta^{2}_{Av}C_{A}} + K$$

$$\beta_{i} = \frac{C_{ei}}{C_{o} + \Delta C_{e}/\beta^{2}_{Av}}$$

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$$\tan \delta_{x} = \frac{1}{\beta_{1}^{2}KC_{A}} \left[ \frac{\alpha_{c}^{2} | \Delta C_{Q0} | \gamma_{3}|}{2} \left( \frac{Q_{0}}{Q_{1}} - 1 \right) - 4x 10^{-8} F_{MHz}^{\frac{3}{2}} (C_{e1}^{2} - C_{e0}^{-3}) - 2x 10^{-9} F_{MHz}^{\frac{3}{2}} (\alpha_{1}^{2} | C_{Q1}^{2} - \alpha_{0}^{2} | C_{Q0}^{2}) \right]$$

In the last equation, note that:

 $F_{\rm Max}$  - frequency of measurement in megahertz

All capacitances in picofarads

The quantity  $\frac{\alpha^{2}|i\lambda}{2}$  may be replaced by  $\frac{c_{R}}{Q}$ , if desired (see page 7).

The following simplifications can sometimes be made:

(1) The last term in the tan 5 calculation should always be dropped unless the  $C_Q$ 's are considerably larger than the  $C_Q$ 's.

(2) At the lower frequencies the internal inductance corrections, described in the manual, may be omitted (i.e.,  $\alpha_{\rm R} = \alpha_{\rm I} = \alpha_{\rm I} = 1$ ).

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