

Errata

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HP References in this Application Note

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APPLICATION NOTE 153

PERMEABILITY, PERMITTIVITY AND CONDUCTIVITY
MEASUREMENTS WITH
TIME DOMAIN REFLECTOMETRY

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FOREWORD

This application note has been prepared for chemists, physicists, and electronic engineers. The quite different background of these readers requires detailed treatment of the subject to make it understandable to everyone. On the other hand, going into too much detail would impair the ease of reading. We have compromised by adding two appendices, one on Laplace transforms which are less familiar to the chemist, and one on the theory of dielectrics which may be of interest to electronic engineers and physicists.

This application note replaces Application Note 118.

TABLE OF CONTENTS

Section	Page	Section	Page
1. Introduction	1	8. Practical Aspects of TDR	
2. Principle of Operation	1	Measurements	11
3. Hewlett-Packard Time Domain		Sample Cells for Single	
Reflectometers	1	Reflection Method	11
4. Single Reflection Time Domain		Sample Cells for Thin Sample	
Reflectometry	3	Method	13
5. Thin Sample Time Domain		Measurement Techniques	13
Spectroscopy	5	Computerized Sampling Systems	14
6. Measurement Accuracy	10	Applications	15
7. Effects of System Risetime	11	Appendix I	
		The Correlation Between Time and	
		Frequency Domain	17
		Appendix II	
		The Principle of Dielectric	
		Measurements	20

NOTATION

C	Capacitance per unit length of coaxial line
c	Light velocity
D	Diameter of outer conductor of coaxial line
d	Diameter of inner conductor of coaxial line
G	Parallel conduction per unit length
L	Inductance per unit length of coaxial line
l	Length (thickness) of sample
R	Series resistance per unit length of coaxial line
R_n	Signal due to the n-th reflection
S_{ik}	Scattering coefficient in the frequency domain
S(1)	Unit step pulse
s_{ik}	Scattering coefficient in the time domain
V_0	Voltage of the applied step pulse
Z	Input impedance of coaxial line with sample
Z_1	Characteristic impedance of coaxial line with sample
Z_0	Characteristic impedance of empty coaxial line
Δ	Relative error of κ due to thin sample approximation
γ	Propagation constant of coaxial line with sample
γ_0	Propagation constant of empty coaxial line
κ	Relative permittivity
κ_0	Static relative permittivity
κ_1	High frequency relative permittivity
κ_σ	See equation 5.4
ρ	Single reflection coefficient
ρ_0	Low frequency single reflection coefficient
ρ_1	High frequency single reflection coefficient
σ	Conductivity of sample
τ	Relaxation time constant
ω	Circular frequency

1. INTRODUCTION

The measurement of the dielectric behavior of solids and liquids is of practical and theoretical interest to engineers, chemists, and physicists. Electronic engineers are interested in the dielectric constant and loss of materials at certain frequencies for RF and microwave applications. Chemists and physicists obtain important information from such measurements about molecular structure, particularly in polymers and solvent-solute systems.

Likewise, the magnetic properties of many materials, i.e., ferrite cores and yttrium garnets, need to be measured over a wide frequency range, particularly in the UHF - and microwave region.

Generally the measurements are made by placing the substance between two plates of a capacitor (at low frequencies) or in a coaxial line and measuring the complex impedance in the frequency domain. A number of measurements over a wide frequency range is required for complete characterization, which is time consuming and demands a considerable investment in instrumentation, particularly for the microwave region. Therefore, in spite of its usefulness, this method has found rather limited applications. However, one can obtain the same information over a wide frequency range in only a fraction of a second by making the measurement not in the frequency domain but in the time domain, using a pulse that simultaneously contains all the frequencies of interest. This pulse method has been used for low frequency investigations on dielectrics. Modern tunnel diode pulse generators and wide band sampling oscilloscopes extend this method into the microwave region where savings in time and equipment are most pronounced.

2. PRINCIPLE OF OPERATION

The time domain reflectometer consists of a pulse generator which produces a fast rise time step, a sampler which transforms a high frequency signal into a lower frequency output, and an oscilloscope or any other display or recording device (see Figure 2.1).

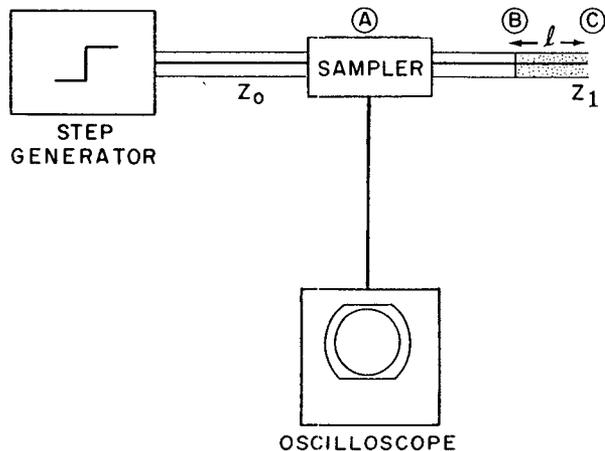


Figure 2.1 Time domain reflectometer setup.

Let us first consider the effect of a coaxial line, filled with a dielectric over a length l .

The pulse from the step generator travels along the coaxial line until it reaches the point A. The sampler detects and the oscilloscope displays the voltage step (A) as it travels past point A (see Figure 2.1 and 2.2). The coaxial line which transmits the pulse has a characteristic impedance Z_0 of 50 ohms. Whenever there is a discontinuity in this line, a fraction of the traveling wave is reflected back to the generator. Therefore, at the interface of the 50 ohm line with any other impedance Z_1 (point B) part of the step pulse is reflected and passes point A again, producing an additional signal (B) which is displayed on the oscilloscope. The time elapsed between the first and second step is equal to the transit time of the traveling wave from A to B and back to A again.

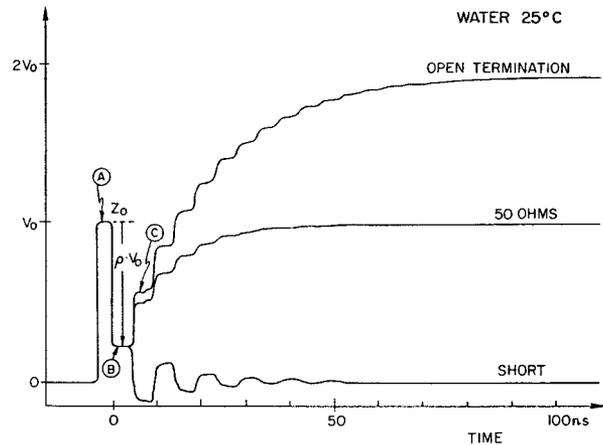


Figure 2.2 Typical TDR reflection from dielectric sample. (See text)

The remainder of the wave, which was not reflected at B, travels to C. If we terminate the line at C with an open end then all of the wave is reflected back in phase (assuming no losses due to radiation). Part of this pulse is reflected again at B and part of it goes through past A, giving rise to another step (C). The time between step (B) and (C) is the transit time from B to C and back to B again.

The multiple reflections between B and C continue until all of the pulse energy has been reflected back into the generator and is absorbed there. If we use a short at C as a termination, then an incident wave is totally reflected again, but 180° out of phase, and the multiple reflections reduce the signal at A until it reaches zero. The first reflection at B, however, is completely independent of the termination at C.

Similarly, a 50 ohm termination produces a reflection pattern, which converges towards the amplitude V_0 of the incident step.

3. HEWLETT-PACKARD TIME DOMAIN REFLECTOMETERS

There are different types of Hewlett-Packard time

domain reflectometers with risetimes from 35 to 160 ps to fit every need and budget. They are built for the 140 series or the 180 series plug-in oscilloscopes. The presently available combinations and some of their operating characteristics are shown in Figs. 3.2 and 3.3. Representative set-ups for the

140A and 180A oscilloscope, both with 35 ps risetime are shown in Figures 3.1a and 3.1b. The 140 system may be programmed by a computer for data acquisition using a digital-analog converter, connected to the external horizontal sweep, and an analog-digital converter connected to the y-output for the recorder.

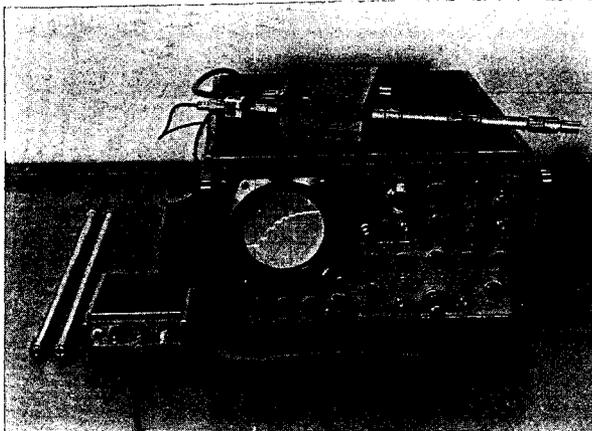


Figure 3.1a HP 140 System with 35 ps risetime.

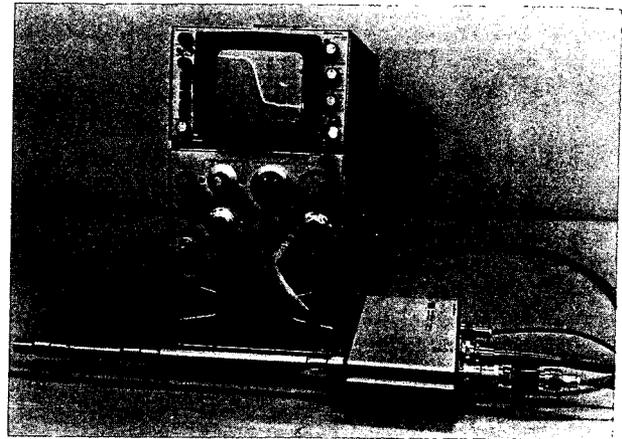


Figure 3.1b HP 180 System with calibrated 35 ps risetime.

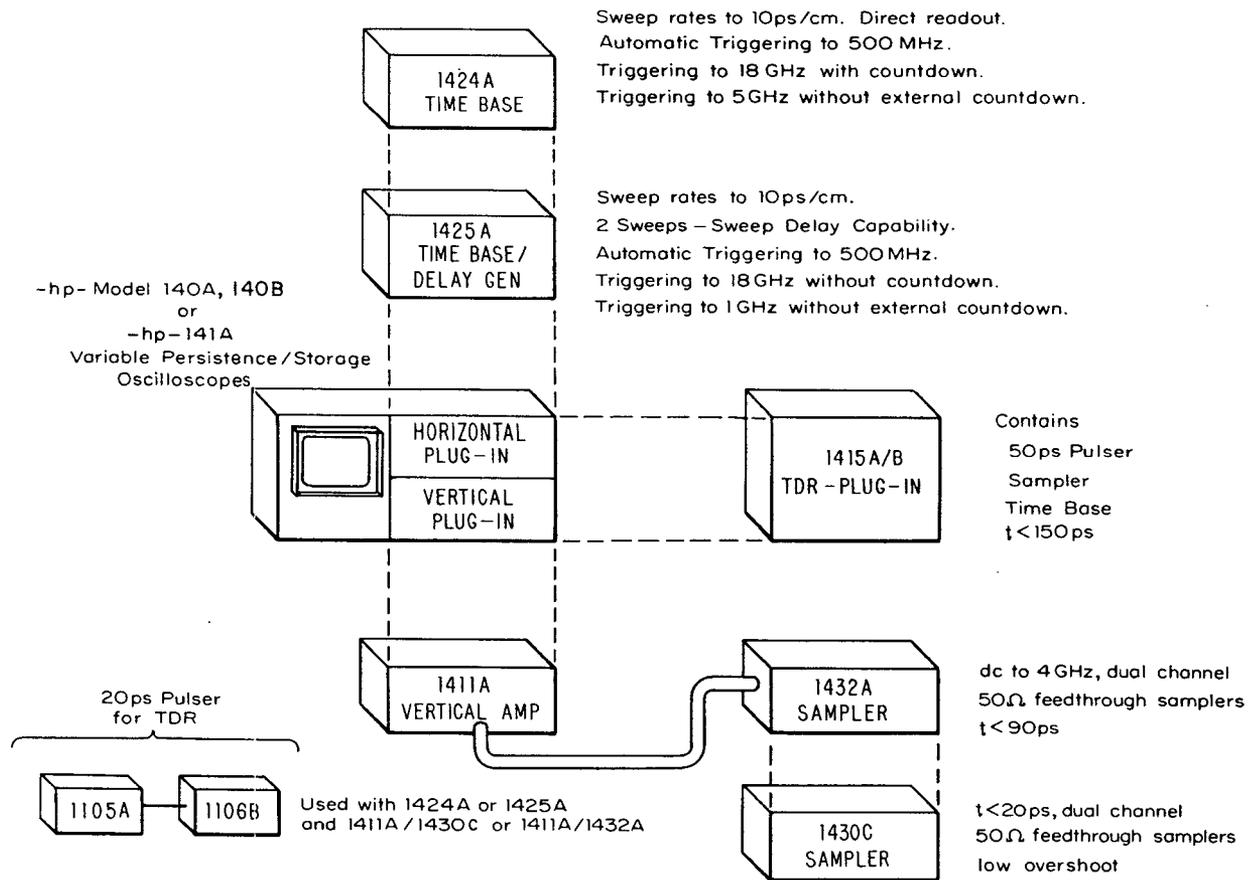


Figure 3.2 HP 140 System with either 1415A 130 ps risetime TDR plug-in or with 35 ps risetime TDR plug-ins and samplers.

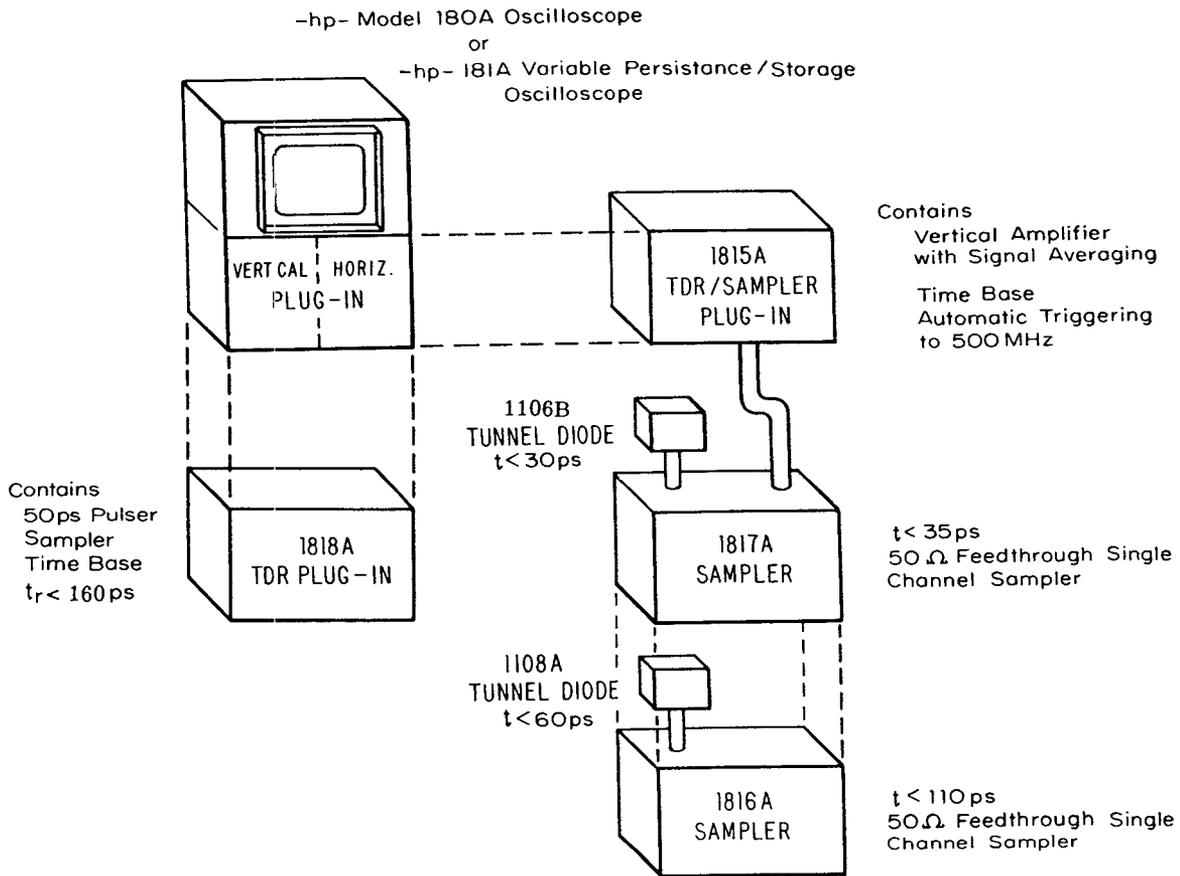


Figure 3.3 HP 180 System with either 1818A 160 ps risetime plug-in or with 35 ps risetime calibrated TDR.

4. SINGLE REFLECTION TIME DOMAIN REFLECTOMETRY

The magnitude of the first step, $V_o \cdot \rho$ of a line with the impedance Z_1 is

$$V_o \cdot \rho = V_o \frac{Z_1 - Z_o}{Z_o + Z_1} \quad \rho : \text{reflection coefficient of a line with the impedance } Z_o, \text{ terminated with } Z_1.$$

$$V_o : \text{pulse height} \quad (4.1)$$

A coaxial line filled with a dielectric of the permittivity κ has the characteristic impedance

$$Z_1 = \frac{60}{\sqrt{\kappa}} \ln \frac{D}{d} = \frac{Z_o}{\sqrt{\kappa}} \quad \begin{array}{l} D : \text{diameter of outer conductor} \\ d : \text{diameter of inner conductor} \end{array} \quad (4.2)$$

Thus, the reflection coefficient of the first reflection is a function of κ :

$$\rho = \frac{1 - \sqrt{\kappa}}{1 + \sqrt{\kappa}} \quad (4.3)$$

and

$$\kappa = \left(\frac{1 - \rho}{1 + \rho} \right)^2 \quad (4.4)$$

Any time dependence of the permittivity of a material due to dielectric relaxation results in a time dependence of the reflection coefficient and can, therefore, be determined.⁽¹⁾

(1) H. Fellner-Feldegg: J. Phys. Chem. 73,616 (1969)

The complex permittivity in the frequency domain

$$\kappa^*(\omega) = \kappa' - j\kappa''$$

can be transformed into the time domain by Laplace transformation

$$\kappa(t) = \frac{1}{2\pi} \int_{a-j\infty}^{a+j\infty} \kappa^*(\omega) e^{i\omega t} d\omega$$

$\kappa(t)$: time function of the permittivity

(see Appendix I and II).

We now see that the impedance Z_1 and therefore the reflection coefficient ρ are functions of the permittivity κ , both in the frequency and time domain. A time dependence of the permittivity is reflected in a time dependence of the reflection coefficient. The step at (B) in Figure 2.2 is modified depending on the relaxation properties of the dielectric. The measurement is made only at the interface between air and dielectric and is undisturbed as long as there are no additional reflections present from any point beyond point B in Figure 2.1. Therefore, the time for the return trip of the wave in the line filled with the dielectric limits the maximum time available for the measurement and is:

$$t[\text{ns}] = \frac{2l\sqrt{\kappa'}}{c} = \frac{\sqrt{\kappa'}}{15} l \quad [\text{cm}] \quad (4.5)$$

t : maximum available measurement time
 l : length of sample

For the application of single reflection TDR, the transformation of the reflection coefficient has to be made. Thus

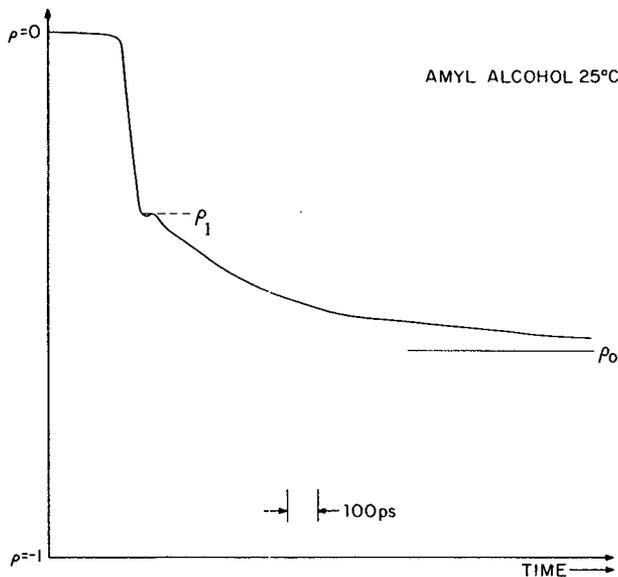


Figure 4.1 Oscilloscope trace of the reflection from the interface amyl alcohol-air.

$$\rho(t) = \frac{1}{2\pi} \int_{a-j\infty}^{a+j\infty} \frac{1 - \sqrt{\kappa^*(\omega)}}{1 + \sqrt{\kappa^*(\omega)}} \cdot e^{j\omega t} d\omega \quad (4.6)$$

This integral cannot be solved in closed form, even for ideal dielectrics, but we have derived expressions for the reflection coefficient as a function of time in the form of infinite series for Debye dielectrics.⁽²⁾ With the aid of a computer, these series have been used to compute this function for a wide range of cases of practical interest. These results are presented in graphical form in such a way as to facilitate determining the true relaxation time from observed reflected waveforms. Furthermore, several independent determinations of the relaxation time can be made from different features of the reflected waveform. The consistency of the values obtained for a given dielectric serves as a powerful check of the correctness of the assumptions underlying the method. The most important of these assumptions is that the dielectric is of the Debye type, having a single relaxation time.

Figure 4.1 gives an actual plot of the time dependence of the reflection coefficient $\rho(t)$ of amyl alcohol, recorded from the oscilloscope output with a Moseley X-Y recorder. ρ_1 and ρ_0 can be read directly.

The relaxation time τ can conveniently be determined from the measured time τ' for $\rho_0 - \rho(t)$ to decrease to some specified fraction of its initial value $\rho_0 - \rho_1$.

(2) H. Fellner-Feldegg, F. Barnett, J. Phys. Chem. 74, 1962 (1970)

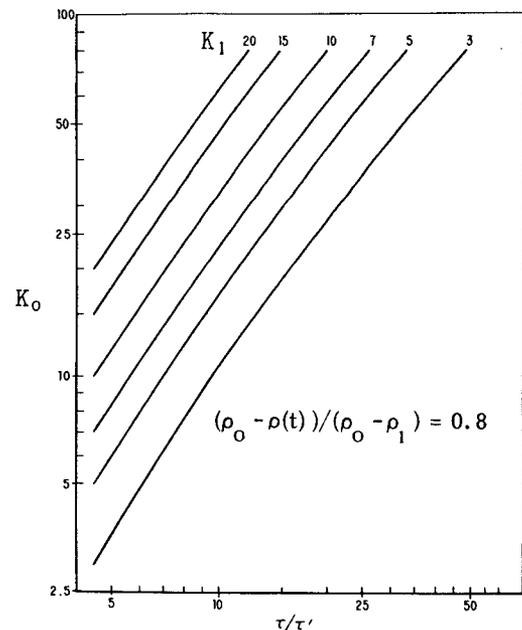


Figure 4.2 Ratio of the true relaxation time τ to the measured time τ' at the 80% point of the reflection coefficient.

Figs. 4.5, 4.6 and 4.7 show τ/τ' as abscissa with κ_0 , the static permittivity, as ordinate for a range of values of κ_1 , the high frequency permittivity, of practical interest. τ' is the time necessary for $(\rho_0 - \rho(t))/(\rho_0 - \rho_1)$ to reach the values 0.8, $1/e$, and 0.2, respectively, in the three figures. Both coordinates are plotted logarithmically. The resulting curves have relatively little curvature and are nearly parallel to each other over the range plotted.

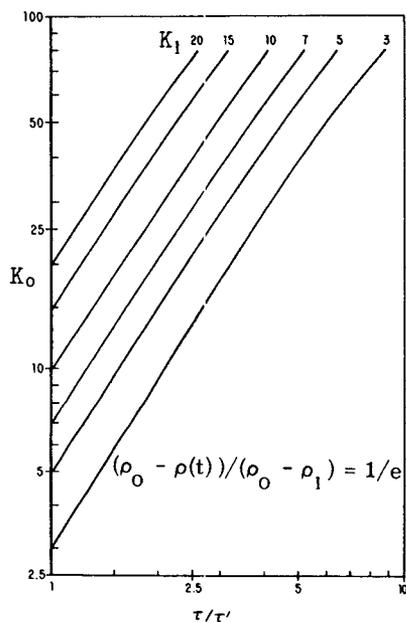


Figure 4.3 Ratio of the true relaxation time τ to the measured time τ' at $1/e$ point of the reflection coefficient.

5. THIN SAMPLE TIME DOMAIN SPECTROSCOPY

The above mentioned method is simple to carry out and easy to interpret. It requires, on the other hand, rather long coaxial sample cells for the measurement of long relaxation times (see equation 4.5) and makes the Laplace transformation difficult.

It turned out, going to the other extreme of using very thin samples, terminated by a 50 ohm load, a linear relation between signal and permittivity or permeability can be obtained. One measures directly the impulse response of the dielectric or magnetic sample and the step response of its conductivity. This makes the interpretation of data and the transformation between time and frequency domain very simple. The method is called THIN SAMPLE TIME DOMAIN SPECTROSCOPY.⁽⁵⁾

Recently, Suggett and co-workers⁽³⁾⁽⁴⁾ have described single reflection and transmission time domain techniques and combinations thereof. Their results on aliphatic alcohols and aqueous solutions show a very significant improvement in measurement accuracy and make time domain spectroscopy a precision measurement method. We like to refer the reader to these publications for further information.

(3) A. Suggett, P.A. Macness, M.J. Tait, H.W. Loeb, G.M. Young, *Nature* **228**, 456 (1970).

(4) H.W. Loeb, P.A. Quickenden, A. Suggett, G. M. Young, *Ber. Bunsenges. Phys. Chem* **75**, 1155 (1971).

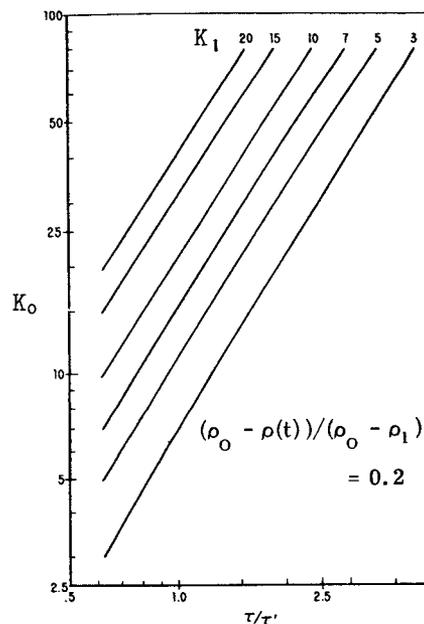


Figure 4.4 Ratio of the true relaxation time τ to the measured time τ' at the 20% point of the reflection coefficient.

We will first derive the equations for the frequency domain, using a well known transmission line equation, and then transform the result into the time domain.

The impedance Z of a transmission line of an impedance Z_0 , with a section of the impedance Z_1 and length l , terminated with Z_0 is given by

$$Z = Z_1 \frac{1 - \rho \exp(-2\gamma l)}{1 + \rho \exp(-2\gamma l)} \quad (5.1)$$

where γ is the propagation constant and ρ is the reflection coefficient of a line of the impedance Z_0 , terminated with Z_1 , as defined in the preceding section.

(5) H. Fellner-Feldegg, *J. Phys. Chem.*, to be published.

The line with the impedance Z_1 is obtained by filling a coaxial line of the characteristic impedance Z_0 with the sample of a length ℓ . The sample shall have a relative permittivity $\kappa^* = \kappa' - j\kappa''$, a conductivity σ , and a relative permeability $\mu^* = \mu' - j\mu''$. We are neglecting any series resistance in this line. Then the impedance is

$$Z_1 = \sqrt{\frac{j\omega L_1 \mu^*}{j\omega C \kappa^* + G}} = Z_0 \sqrt{\frac{\mu^*}{\kappa^* \sigma}} \quad (5.2)$$

and the propagation constant is

$$\gamma = \sqrt{j\omega L_1 \mu^* (j\omega C \kappa^* + G)} = \gamma_0 \sqrt{\mu^* \kappa^* \sigma} \quad (5.3)$$

with

$$Z_0 = \sqrt{\frac{L}{C}}, \quad \frac{G}{C} = 4\pi\sigma, \quad \gamma_0 = j\omega\sqrt{LC} = \frac{j\omega}{c}$$

and

$$\kappa^*_\sigma = \kappa^* - j\frac{G}{\omega C} = \kappa^* - j\frac{4\pi\sigma}{\omega} \quad (5.4)$$

We are using the cgs-system and are, therefore, including the factor 4π in the conductivity term. Also

$$\rho = \frac{\sqrt{\frac{\mu^*}{\kappa^*_\sigma} - 1}}{\sqrt{\frac{\mu^*}{\kappa^*_\sigma} + 1}} \quad \text{and} \quad 1 - \rho^2 = \frac{4\sqrt{\frac{\mu^*}{\kappa^*_\sigma}}}{\left(\sqrt{\frac{\mu^*}{\kappa^*_\sigma} + 1}\right)^2}$$

The reflection coefficient from the sample, expressed as a scattering coefficient S is then

$$S_{11} = \frac{[1 - \exp(-2\gamma\ell)] \rho}{1 - \rho^2 \exp(-2\gamma\ell)} \quad (5.5)$$

and the change in the transmission coefficient due to the sample, referenced to the output port of the sample, is

$$\begin{aligned} [S_{21} - \exp(-\gamma_0\ell)] = \\ \frac{[\exp(-\gamma\ell) - 1] [1 + \rho^2 \exp(-\gamma\ell)]}{1 - \rho^2 \exp(-2\gamma\ell)} + [1 - \exp(-\gamma_0\ell)] \end{aligned} \quad (5.6)$$

Expanding the exponential term into a Taylor series and using the linear term only gives the reflection coefficient

$$S_{11} = 2\gamma\ell \frac{\rho}{1 - \rho^2} = \frac{\gamma_0\ell}{2} \left(\mu^* - \kappa^* - \frac{4\pi\sigma}{j\omega} \right) \quad (5.7)$$

and the difference in the transmission coefficient

$$[S_{21} - \exp(-\gamma_0\ell)] = \quad (5.8)$$

$$\gamma_0\ell - \gamma\ell \frac{1 + \rho^2}{1 - \rho^2} = -\frac{\gamma_0\ell}{2} \left(\mu^* + \kappa^* + \frac{4\pi\sigma}{j\omega} - 2 \right)$$

Equations 5.7 and 5.8 can be used for the measurement in the frequency domain.

The response to a step pulse is

$$(S_{11})_{\text{step}} = \frac{S_{11}}{j\omega} = \frac{\ell}{2c} \left(\mu^* - \kappa^* - \frac{4\pi\sigma}{j\omega} \right)$$

and likewise

$$\begin{aligned} (S_{21})_{\text{step}} - \frac{\exp(\gamma_0\ell)}{j\omega} = \frac{S_{21} - \exp(-\gamma_0\ell)}{j\omega} = \\ -\frac{\ell}{2c} \left(\mu^* + \kappa^* + \frac{4\pi\sigma}{j\omega} - 2 \right) \end{aligned}$$

It is the advantage of this approach that one obtains a linear superposition of the frequency response of permeability, permittivity and conductivity (the latter divided by $j\omega$). This not only makes it easy to interpret the results in the frequency domain, but also allows to make the transformation into the time domain separately for each term.

The Laplace transformation into the time domain gives then

$$\begin{aligned} (s_{11})_{\text{step}} = \frac{\ell}{2c} \left[\mathcal{L}(\mu^*) - \mathcal{L}(\kappa^*) - \mathcal{L}\left(\frac{4\pi\sigma}{j\omega}\right) \right] \\ = \frac{\ell}{2c} \left[\mu(t) - \kappa(t) - 4\pi \int_0^t \sigma(t) dt \right] \end{aligned} \quad (5.9)$$

and

$$\begin{aligned} (s_{21})_{\text{step}} - [S(1)]_t = \\ -\frac{\ell}{2c} \left[\mu(t) - 1 + \kappa(t) - 1 + 4\pi \int_0^t \sigma(t) dt \right] \end{aligned} \quad (5.10)$$

where $[S(1)]_t$ is a unit step function at the time $t = \ell/c$ and $\mu(t)$, $\kappa(t)$, $\sigma(t)$ are the impulse response of the permeability, permittivity and conductivity, respectively. The integral of the impulse response $\sigma(t)$ is the step response of the conductivity.

The only difference between the reflected and transmitted waves, both in the frequency and time domain, is the sign of the permeability term. This allows to separate it from the permittivity and conductivity term by measuring the transmission and the reflection. However, the reflection measurement will, in general, be more accurate, since the signal is directly proportional to the material constants and the sample length, and can be well separated from the incident step pulse whereas in the transmission measurement one has to take the difference between $\exp(-\gamma_0\ell)$ or $S(1)$ and the relatively small value of $(S_{21})_{\text{step}}$ or $(s_{21})_{\text{step}}$, respectively.

$1/c$ has the value of 30 ohms which can be used advantageously when expressing the conductivity σ in $\text{ohms}^{-1}\text{cm}^{-1}$. Figures 5.1, 5.2 and 5.3 give some typical time responses obtained with Thin Sample Time Domain Spectroscopy, for magnetic, dielectric and conductive materials.

The results obtained in the time domain can either be transformed into the frequency domain by numerical transformation, or can be evaluated directly. We shall explain this on the example of an ideal and a lossy dielectric, but will keep in mind that it is equally applicable for any dielectric or magnetic sample.

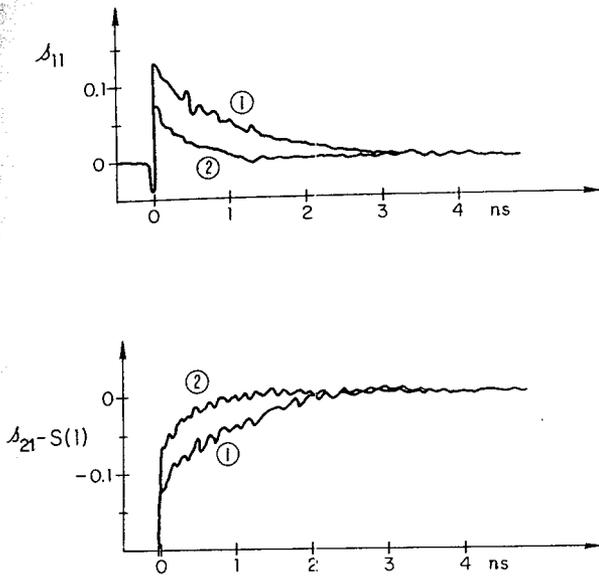


Figure 5.1 Reflection and transmission of a thin sample of ferromagnetic material. Ferrite toroid core: (1) Ferramic Q₂, (2) Ferramic Q₃.

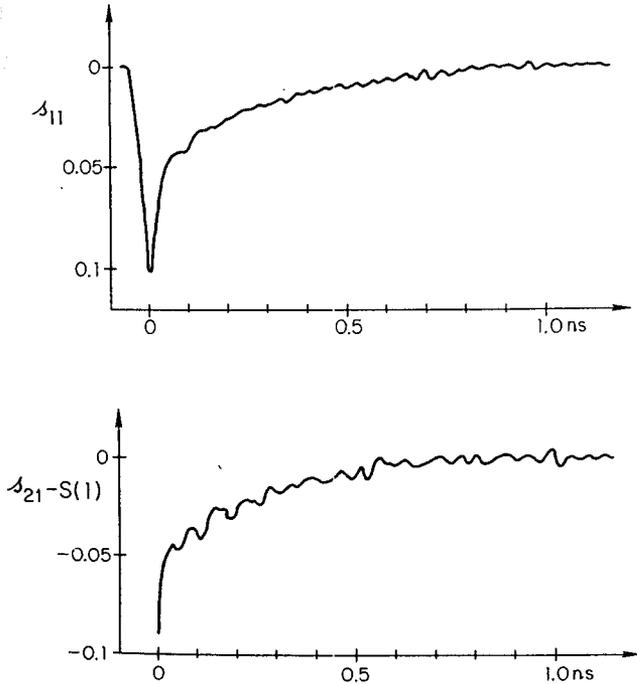


Figure 5.2 Reflection and transmission of a thin sample of a dielectric: Glycerin 25°C.

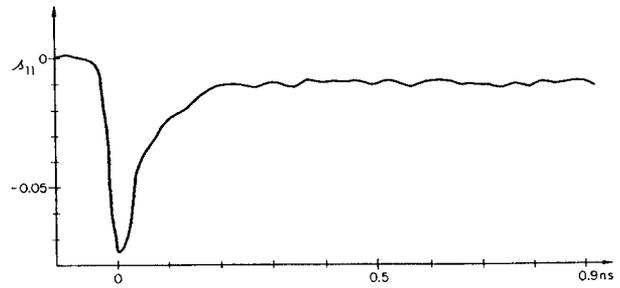


Figure 5.3 Reflection from a dielectric with conductivity. Saturated solution of potassium chloride in methanol at 25°C.

Consider a thin sample of a non magnetic ($\mu = 1$) Debye dielectric with no conductivity. The frequency and time response of the reflection coefficient to a step pulse is then

$$\begin{aligned} (S_{11})_{\text{step}} &= \frac{l}{2c} (1 - \chi^*) \\ &= \frac{l}{2c} \left(1 - \chi_1 - \frac{\chi_0 - \chi_1}{1 + j\omega\tau} \right) \end{aligned}$$

$$(s_{11})_{\text{step}} = \frac{l}{2c} (1 - \chi_1) \delta(t) - \frac{l}{2c\tau} (\chi_0 - \chi_1) e^{-t/\tau} \quad (5.11)$$

Figure 5.4 shows the time response schematically. The area of the δ -function is proportional to $1 - \chi_1$, (it has a negative value since it is a negative impulse) and the area under the curve is

$$\int_0^{\infty} s_{11}(t) dt = \frac{l}{2c} (1 - \chi_0) \quad (5.12)$$

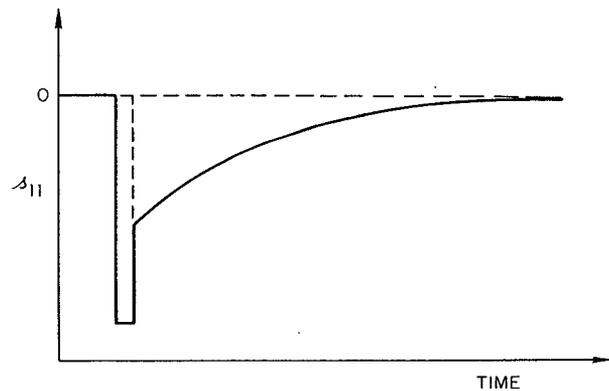


Figure 5.4 Time response of a thin sample of an ideal dielectric.

Figure 5.5 shows the close match between theoretical and experimental values, obtained on a number of dielectrics. The low value of s_{11} for water may be due to incomplete filling of the sample cell because of the strongly hydrophobic Teflon beads.

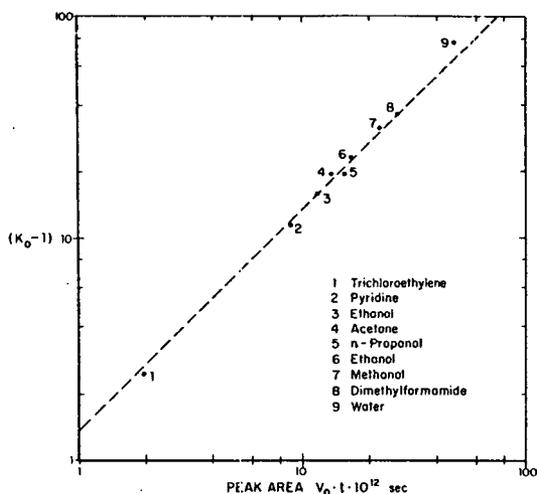


Figure 5.5 Static permittivity versus peak area for different dielectrics. Cell length: 0.46mm. Dashed line: Theoretical dependence, using equation 5.12.

The exponential part of the time response curve gives the relaxation time, which can be obtained simply from the slope of the log s_{11} versus time plot. For non ideal dielectrics one obtains κ_1 and κ_0 in the same way; however, the log s_{11} plot will deviate from a straight line depending on the relaxation properties of the dielectric.

The finite response of the measuring system degrades the δ -function in equation 5.11 to a peak of finite width whose area is proportional to $l(1 - \kappa_1)/2c$.

Electrical conductivity, present in the sample, is added to the frequency or time dependence of the reflection coefficient. If the conductivity is independent of frequency, it will produce an offset of the base line of

$$(s_{11})_{\text{cond}} = 2\pi\sigma l/c = 188.5 \sigma l \quad (5.13)$$

Finally, we have to specify the error introduced by using only the first term of the Taylor expansion of equation 5.5 and 5.6. We will solve the problem for the frequency domain first.

Figure 5.6 shows a computer plot of the exact value of S_{11} or $[S_{21} - \exp(-\gamma_0 l)]$ vs. κ or μ for different values of $\gamma_0 l$ according to equations 5.5 and 5.6. The dashed lines give approximate values, derived from equations 5.7 or 5.8. The difference between equation 5.5 and 5.6 (reflection and transmission, respectively) contains only the third and higher order terms of the Taylor expansion and is about 10^{-6} for the range of values plotted.

The relative difference Δ of κ , derived from the exact and the approximate equations, $\Delta = (\kappa - \kappa_{\text{approx}})/\kappa$, is very closely equal to the value of S_{11} . The difference $S_{11} - \Delta$ versus κ is plotted in Figure 5.7. For small values of $\gamma_0 l$ and for $\kappa \gg 1$, this difference approaches $\gamma_0 l/4$. It is therefore, quite convenient to use this relation between Δ and S_{11} not only to estimate the error made in using the thin sample approx-

imation, but also to correct for it, which in turn permits to employ relatively thick samples, producing sufficiently strong signals for exact measurements in the frequency domain.

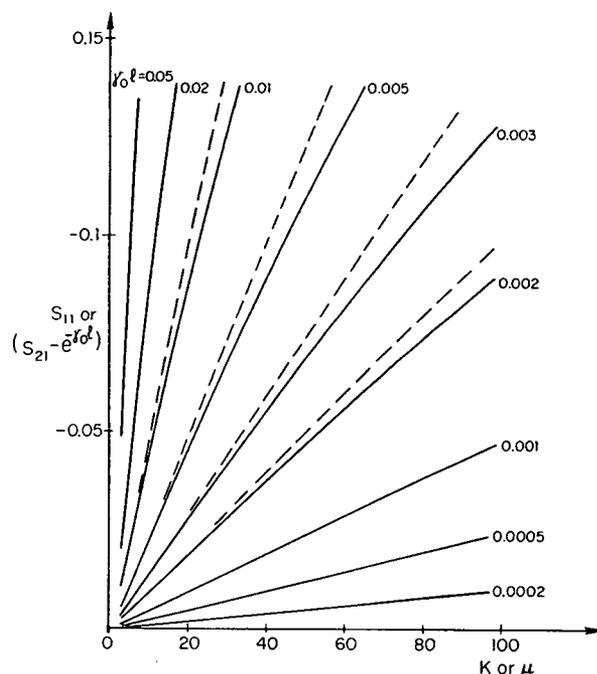


Figure 5.6 Reflection or transmission signal from a thin sample versus permeability or permittivity for different values of $\gamma_0 l$. Solid curves: exact solution; dashed lines: thin sample approximation.

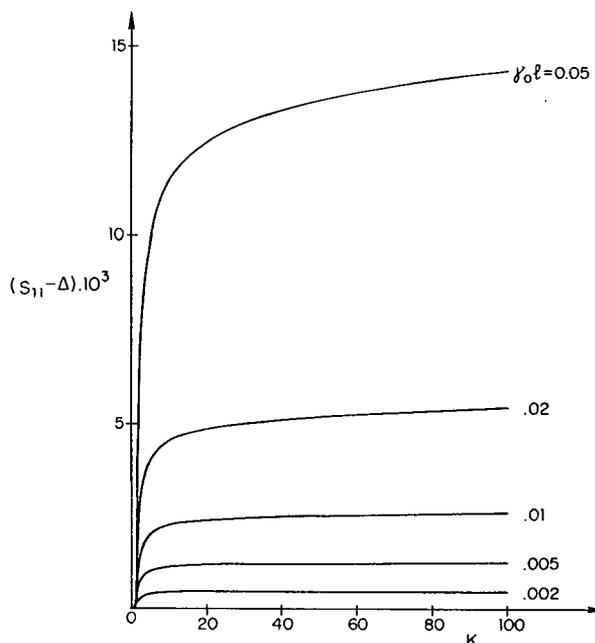


Figure 5.7 Difference between reflection signal S_{11} and relative measurement error, resulting from the thin sample approximation in the frequency domain.

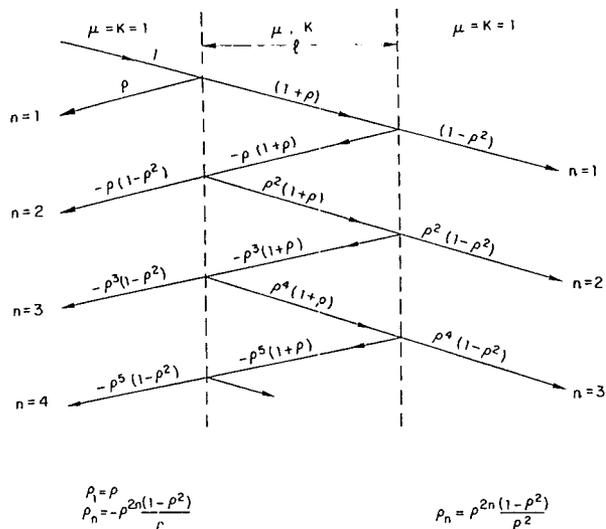


Figure 5.8 Multiple reflections from a section of transmission line with permeability μ and permittivity κ and length ℓ .

It is difficult to transform these relations into the time domain. Therefore, we will derive equation 5.9 (without conductivity term) in the time domain to obtain Δ .

Consider again a section of a transmission line, of the length ℓ filled with a material with the permeability μ and the permittivity κ , inserted into a line of the impedance Z_0 and terminated with the same impedance. We further stipulate that μ and κ do not vary during the time interval t_r considered in the following. Then the reflection coefficient for the first reflection will be

$$\rho = \frac{\sqrt{\mu/\kappa} - 1}{\sqrt{\mu/\kappa} + 1} \quad (5.14)$$

The return time within the sample is

$$t_0 = \frac{2\ell\sqrt{\mu \cdot \kappa}}{c} \quad (5.15)$$

Consecutive multiple reflections can easily be analyzed⁽⁶⁾ and are shown in Figure 5.8. They produce a reflected signal which is shown schematically in Figure 5.9 and experimentally in Figure 2.2.

The step height, referenced to V_0 , is

$$R_n = \rho + \sum_{i=2}^n \rho_i = \rho^{2n-1}$$

(6) B. M. Oliver, Hewlett-Packard Journal Vol. 15, No. 6 (1964).

and the area of step 1 to n is

$$A_n = t_0 \sum_{i=1}^n R_i = t_0 \rho \frac{\rho^{2n} - 1}{\rho^2 - 1}$$

Consequently, the total area is given by

$$A_\infty = t_0 \sum_{i=1}^{\infty} R_i = t_0 \frac{\rho}{1-\rho^2}$$

and, using equations 5.14 and 5.15, gives

$$A_\infty = \frac{\ell}{2c} (\mu - \kappa)$$

which is the same as equation 5.9 without conductivity term.

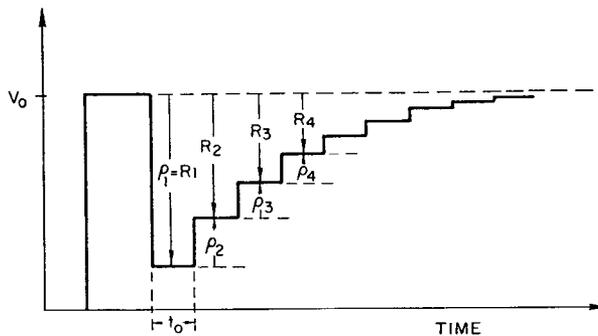


Figure 5.9 Schematic time response of a section of transmission line as in Figure 5.8.

We now specify a time interval $t_r = n \cdot t_0$ to which is defined either by the rise time of the measuring system or by the condition that μ or κ do not vary during this time. We will make an error $\Delta = (A_\infty - A_n) / A_\infty$ of the measurement of μ or κ which is

$$\Delta = \rho^{2n}$$

or

$$\Delta = \left(\frac{|\sqrt{\mu/\kappa} - 1|}{|\sqrt{\mu/\kappa} + 1|} \right)^{t_r c / \ell \sqrt{\mu \kappa}} \quad (5.16)$$

This function is shown in Figure 5.10. For $t_r = 33$ ps, a typical rise time of the -hp- 140 or 180 system, $t_r c = 1$ and equation 5.16 can be approximated by

$$\ell = 1/2(\kappa - 1) \text{ for } \Delta = 2\%$$

$$\ell = 3/4(\kappa - 1) \text{ for } \Delta = 5\%$$

which is convenient for the choice of the maximum permissible sample length.

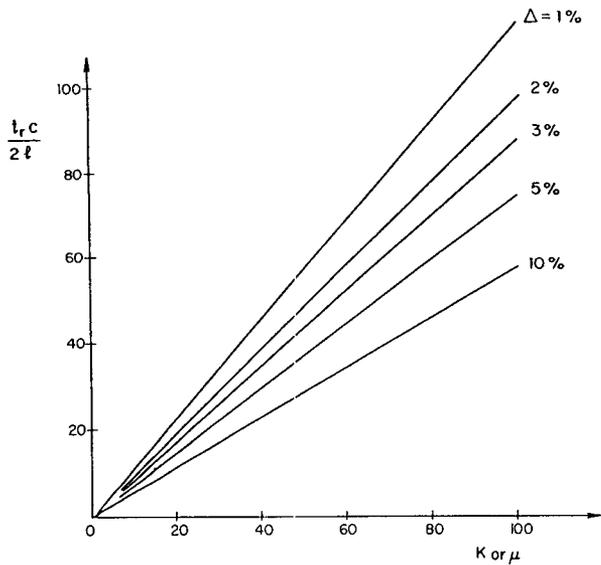


Figure 5.10 $t_r c / 2l$ versus permittivity or permeability for different values of relative measurement error $\Delta = \Delta\kappa / \kappa$ in time domain.

6. MEASUREMENT ACCURACY

A remark about the accuracy of dielectric measurements with TDR. First, it depends on the mechanical accuracy of the coaxial line used. The standard 7mm precision coaxial air lines have an impedance of 50 ± 0.1 ohms or a maximum deviation of 0.2%. Since

$$\kappa = \left(\frac{Z}{Z_0}\right)^2$$

the relative error in κ can be $\pm 0.4\%$.

A second source of error is any inaccuracy in the measurement of the reflection coefficient ρ . It may be due to nonlinearity of the amplifier in the oscilloscope or the recorder, which is less than 1%, or due to unwanted reflections from poorly matched transmission lines, including the sampler. Arrangements shown in Section 8 can suppress the latter and allow an over-all measurement accuracy of the reflection coefficient $\frac{\Delta\rho}{\rho} \leq 0.01$ or $\Delta\rho \leq 1 \cdot 10^{-3}$ whichever is greater. The transformation of ρ into κ , however, multiplies this error by a factor

$$\frac{\Delta\kappa}{\Delta\rho} = -\sqrt{\kappa} (1 + \sqrt{\kappa})^2 \text{ and } \frac{\Delta\kappa}{\Delta\rho/\rho} = -(1 - \kappa)\sqrt{\kappa} \tag{6.1}$$

for the single reflection method.

$\Delta\kappa$ is shown in Figure 6.1 for the different sources of error mentioned. The error due to the tolerance in the coaxial line can be reduced to an insignificant amount by measuring the physical dimensions of

inner and outer conductors with an accuracy of 0.5 and $1.0 \mu\text{m}$ respectively. If this is done, the absolute accuracy of the dielectric constant using the single reflection method, is determined mainly by amplifier nonlinearities, except for the lowest values where noise and unwanted reflections become the limiting factor. Obviously, relative measurements, using a reference sample, can be made with much higher accuracy.

The accuracy of the thin sample method is limited by the error introduced by the thin sample approximation and by the noise level of the measuring system. It also depends on the mechanical accuracy of the measuring cells. One attempts to use the maximum permissible sample length, consistent with the desired accuracy, and can apply the correction term in the frequency domain if necessary. Time averaging by multiple scanning improves noise and base line drift considerably if a longer measurement time can be tolerated.

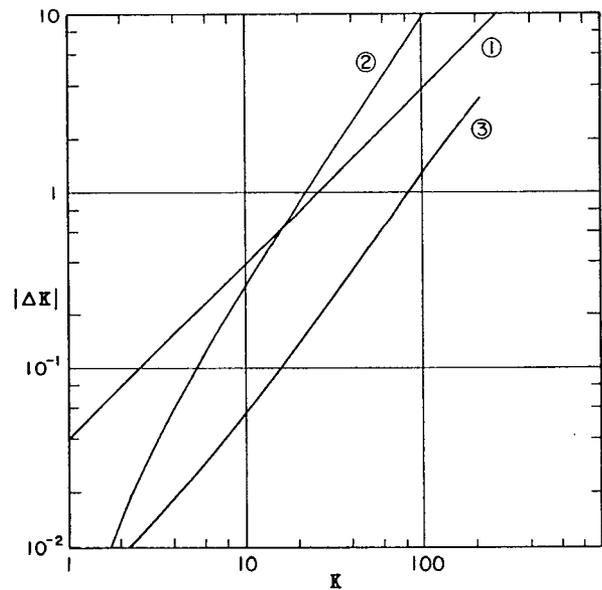


Figure 6.1 Different contributions to the absolute measurement of permittivity $\Delta\kappa$.

- 1) Error due to dimensional tolerance of 7mm precision air line.
- 2) Error due to the relative measurement error of reflection coefficient, $\Delta\rho/\rho = 0.01$ for single reflection method.
- 3) Error due to noise and unwanted reflections, $\Delta\rho = 1 \cdot 10^{-3}$ for single reflection method.

A poorly defined interface between air and dielectric e.g., a liquid meniscus, produces another error that reduces the system risetime or high-frequency

cutoff. Imagine a dielectric with no relaxation reflecting a δ -function impulse, which contains all frequencies of equal amplitude. The reflected pulse will be a δ -function again, if the interface air dielectric is parallel to the electric field. If the interface extends over a certain distance in the direction of the traveling wave, then the reflected pulse will be spread out, corresponding to the return trip time of the wave through the interface. This reflected pulse has a limited high-frequency spectrum. The convolution in the time domain of this pulse with the reflection from any real dielectric (with relaxation) having a perfect interface gives the actual reflection obtained from a real dielectric with an extended interface. Errors introduced in the measurement of high-frequency permittivity and relaxation time can be eliminated by terminating the sample cell with plastic or ceramic beads similar to the ones shown in Figure 8.1 and 8.4. These beads have inner and outer conductor dimensions that produce a characteristic impedance of 50 ohms. They are, therefore, electrically in no way different from the remainder of the empty coaxial line but form a perfect physical interface with any liquid in contact with them.

7. EFFECTS OF SYSTEM RISE TIME

Finally, we shall consider the influence of the finite risetime of a step pulse on the measurement of κ_1 , the high frequency permittivity by the single reflection method. The pulse produced by the tunnel diode generator approximates a linear voltage ramp that rises from zero to V_0 within the time t_r . This ramp is the integral of a step pulse and the response of the dielectric becomes the integral of the step pulse response divided by t_r .

$$f(t)_{\text{ramp}} = \frac{1}{t_r} \int i(t)_{\text{step}} dt$$

Thus, the derivative of the measured time response of the reflection coefficient during the risetime of the pulse times t_r gives the response to a step pulse. In most cases investigated to date, the slope is practically constant, as shown in Fig. 7.1. The linear extrapolation of ρ to time t_r gives ρ_1 and, from equation(4.4), κ_1 .

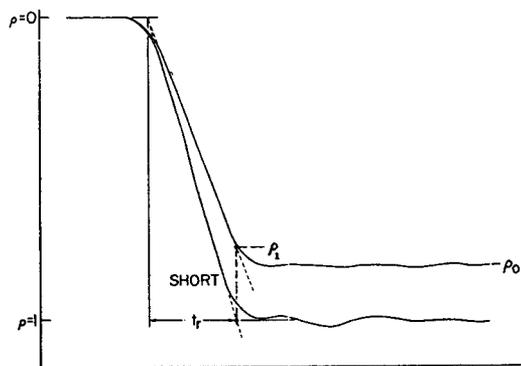


Figure 7.1 Determination of ρ_1 for a pulse with finite risetime. It is obtained by extrapolation of the linear part of the curve to time t_r .

Using the thin sample technique, one needs to determine the area of the initial peak to measure the high frequency permittivity or permeability. One obtains quite consistent results by measuring only the first half of the area up to the maximum peak height and multiplying by two, since the second half will often be distorted due to relaxation effects.

It should be born in mind, however, that the measurement of the high frequency permittivity or permeability is limited for both methods by the rise time of the measuring system. Therefore, substances with a short relaxation time will not yield the true high frequency value but an intermediate one.

8. PRACTICAL ASPECTS OF THE TDR MEASUREMENTS.

SAMPLE CELLS FOR SINGLE REFLECTION METHOD.

A simple and versatile cell for measurements of liquids and granular solids is the standard 7 mm diameter precision coaxial line of 10 and 20 cm length with Amphenol APC-7 connectors (see Figure 8.1).

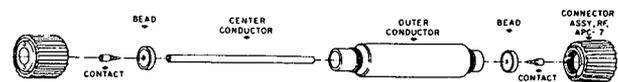


Figure 8.1 7mm precision air line. It is simple, accurate, and useful for many applications. (HP 11566A and 11567A)

As previously mentioned, the maximum deviation from the 50 ohm impedance is $\pm 0.2\%$. The plastic bead on each end is machined accurately enough to provide a liquid-tight seal for a few hours. The plastic may be replaced with any ceramic material, such as alumina, for operation at temperatures above 80°C or to provide resistance against attack by organic solvents. The assembly, including the center conductor, is held in place on one side by the APC-7 connector, to allow filling the line from the other end. The hybrid connector is of high quality and does not introduce any significant reflections up to 18 GHz.

A number of solid brass beads, of the same dimension as the plastic beads were machined and silver plated to act as a short on one end of the sample line. For the measurement of the dielectric material the line is connected with the short on the far end from the pulse generator. The line is then turned around to measure the reference pulse from a short in about the same location as the interface air-dielectric was before.

For measurements at controlled temperatures, the sample cell may be connected on each side with another empty coaxial line, wrapped tightly in plastic or aluminum foil and surrounded by a water jacket that is connected to a temperature bath. The water jacket should extend at least 10 cm over each end of the empty line to insure uniform temperature in the sample. For measurements with liquids at low temperatures, the coaxial line should be mounted

vertically and the signal supplied from the bottom to preserve a well defined interface between dielectric and air when the sample contracts during cooling.

Solids can be measured in a coaxial cell by filling the line with molten material, if possible, or by machining the substance to the dimension of the line, or by wrapping it tightly around the center conductor when available as foil, or by shredding or granulating it. TDR measures an average value of the permittivity if a line is not completely filled with a dielectric. Therefore, for absolute measurements the fill factor has to be known. Relative measurements of dielectric constant and exact determinations of relaxation time can be made, however, if the fill factor is unknown but constant. A change in packing density of the material over the length of the line will cause unwanted reflections from any point where a change in impedance occurs. This effect can be minimized by immersing the material to be tested in a liquid of the same dielectric constant. Different silicon oils and mixtures thereof have been used successfully for the measurement of a number of plastics.

The dielectric properties of large batches of liquids may be measured with a vertical coaxial line immersed in the substance. Since the characteristic impedance, Z_0 , depends only on the ratio of the diameters of inner and outer conductor, the line can be made any size for mechanical stability, as long as no higher modes than the TEM mode are excited. A tapered 50 ohm section should be used to connect it to the standard coaxial line to avoid reflections. Small holes may be drilled at the upper end to allow the liquid in the measuring line to communicate with the outside. Still better exchange between inside and outside can be achieved by using a wire mesh as an outer conductor. The mesh acts as a homogeneous conductor if the holes in the mesh are small compared with the spatial resolution of the TDR. A continuous monitoring of chemical reactions by measuring the permittivity in a time interval where it is most sensitive to changes in the chemical composition is entirely feasible. In addition, the surface position of the liquid can be measured to provide a simple liquid level control. This application was suggested long ago⁽⁷⁾ and has already found some practical use.

The only limitation of the straight coaxial line is its length. A standard 7 mm line becomes quite unstable if more than 30 cm long due to vibrations and sagging of the center conductor. The line can be supported with one or two thin (0.5 mm) washers of mica without introducing serious reflections but such lines become unwieldy if they are longer than 50 cm. On the other hand, one great advantage of dielectric measurements with TDR is the freedom from multiple reflections if we measure only within the time for the return trip of the wave in the sample cell (see Sec. 4). A 40 cm long line, filled with a dielectric of $\epsilon = 20$ limits the range to < 12 ns.

(7) J. Brockmeier, Hewlett-Packard Journal Vol. 17, No. 5 (1966).

One way to make long compact coaxial lines is shown in Figure 8.2. Spiral grooves have been cut into a copper tube of 8 cm diameter. An extruded polypropylene spacer fits tightly into the grooves and supports a copper wire used as a center conductor. An outer tube fits closely over the inner one. Both tubes are sealed to a bottom plate, forming a rectangular coaxial line of 7 m length which can be filled from the top and immersed in a temperature bath. The empty line has 50 ohms impedance and a return trip time of about 50 ns. Because of the polypropylene spacer, the line has to be calibrated with known dielectrics. Relaxation times up to 200 ns have been measured which corresponds to 1 MHz in the frequency domain.

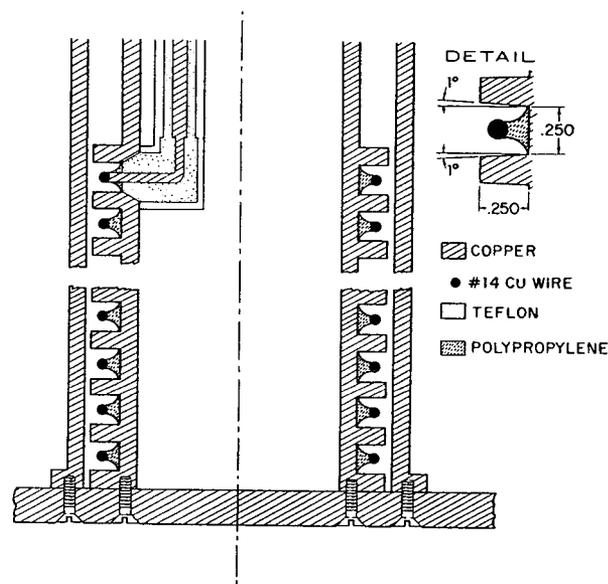


Figure 8.2 Helical coaxial line of 7m length. It is used mainly for the measurement of long relaxation times.

Another kind of transmission line well suited for many applications is the strip line. One of many possible configurations of such a line is shown in Figure 8.3. Two parallel, conductive strips were prepared by etching a printed circuit board. This line can be connected to a coaxial line with a balun transformer. Placing a piece of dielectric material over the two strips reduces the characteristic impedance of the line and results in a dip of the oscilloscope trace. Since the test material fills only part of the field region, a calibration curve with known dielectrics has to be made. Once this is done, the relaxation properties of solids can be measured without much sample preparation. This method may be used as a simple and expedient check of dielectrics for quality control and similar applications. Also, multilayers of plastic foil, pressed against the two conductors, give rather reproducible readings.

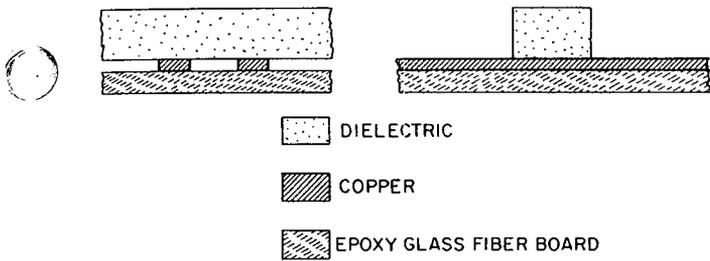


Figure 3.3 Sample testing with the strip line.

SAMPLE CELLS FOR THIN SAMPLE METHOD

The required sample thickness is typically 0.1 to 1mm. With solid samples it is often easy to machine, punch or press a disc of appropriate dimensions. Investigations on thin films can be made by punching a number of discs and pressing them together until the desired thickness is obtained. These samples are then inserted into a 10 cm precision coaxial line, similar to the one shown in Figure 8.1.

Two cells for liquid samples are shown in Figure 8.4 and 8.5. Both use Teflon beads which make a tight seal between inner and outer conductor, enclosing a sample cell between them. In the cell shown in Figure 8.4, the inner conductor has a slightly smaller diameter at the position of the beads to maintain a 50 ohm impedance. The spacer at the center has the nominal diameter of the inner conductor and defines the cell thickness very accurately. Thus, there is no reflection due to the empty cell. The cell of Figure 8.5 has a straight center conductor and two Teflon beads, made such that they have a tight press fit at the inner conductor and a slip fit at the outer conductor. The beads are pressed onto the inner conductor with a removable spacer between them. There is a well defined gap between the beads after the spacer has been removed. Since there is a slight impedance mismatch due to the beads, the empty cell will produce a reflection which has to be subtracted from the measured value.

The cells are inserted into the outer conductor until the cell gap is just flush with the end face of the outer conductor. Holding the assembly vertically and applying the sample with a syringe to this end face produces a meniscus around the Teflon bead and fills the cell completely. The cell is then pushed further into the outer conductor until the second bead just disappears. The sample is now sealed between the beads and excess liquid is wiped off. The cell is then pushed into the final position within the outer conductor, and the connectors are fastened. The assembly is ready for measurement.

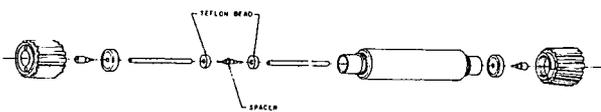


Figure 8.4 Sample cell for thin sample technique with diameter of inner conductor adjusted for 50 ohm impedance at the beads.



Figure 8.5 Sample cell for thin sample technique with standard inner conductor.

MEASUREMENT TECHNIQUES

Some precautions are necessary to use the high sensitivity of TDR to its fullest advantage. One is the suppression of unwanted reflections, the other is the correction of slight pulse shape deviations from an ideal step pulse.

Interfering reflections may appear superimposed on the oscilloscope trace during the time interval in which the reflection coefficient ρ drops to its final value. They may be small bumps when originating from local discontinuities or small steps when caused by extended impedance mismatches between pulse generator and sample. They are produced by the reflected pulse from the interface air-dielectric traveling towards the generator. Any finite impedance mismatch in the sampler can produce such reflections. They can be spaced farther apart or suppressed by inserting about 40 cm of a precision air line between sampler and measuring cell. Placing a short in the place of the air-dielectric interface allows one to clearly examine these interferences. Figure 8.6 shows the trace of a short, 50 ohm-line, and a dielectric with a deliberately produced poor connection at the sampler.

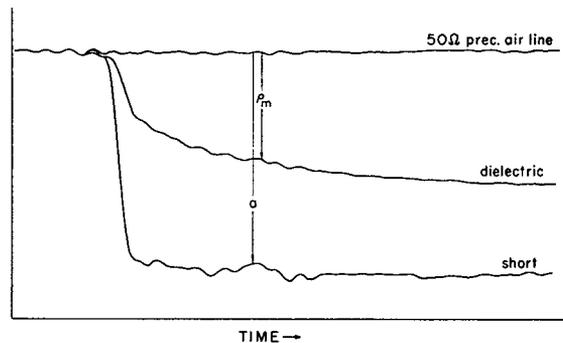


Figure 8.6 Trace of the reflection coefficient from a 50ohm line, a dielectric and a short. Pulse shape imperfections and unwanted reflections can be corrected by measuring ρ_m and a at any point in time. The true value of the reflection coefficient $\rho = \rho_m/a$.

With the single reflection method, one often observes a slight pulse overshoot at the bottom of the step due to the sampler frequency response, and a certain slow droop to a final value. Both may cause measurement errors of the high frequency permittivity ϵ_1 and of the relaxation time τ . This interference can be eliminated by the following procedure: First, connect the measuring cell, with a short at the far end (as seen from the instrument), and record the trace. Then, turn it around to have the short in front of the cell and record its trace on the same graph.

Finally, substitute the cell by a precision air line and superimpose the traces (Figure 8.6). The true value of reflection coefficient ρ at any point in time is ρ_m/a .

The measurement accuracy may be improved further for repeated measurements with materials of fairly constant permittivity by making the ratio of inner to outer diameter in a coaxial line smaller to give an impedance of about 50 ohms when filled with the dielectric. In Figure 6.1 we have shown for a standard 50 ohm coaxial line that the absolute measurement accuracy improves considerably for $\kappa \rightarrow 1$, or $Z \rightarrow 50$ ohm.

Relative measurements between a sample and a reference material can be made with the HP 1430C sampler with two sampling inputs which can be used simultaneously as shown in Figure 8.7 and 8.8. The two inputs from the sampler to the HP 1411A sampling amplifier can be displayed separately or one may be subtracted from the other. This feature is of particular interest since it allows extraction of small differences in the dielectric behavior between the sample and the reference due to impurities, reaction products, temperature, or other influences. Most errors or interferences cancel, and the ultimate measurement accuracy is determined by the noise level of the instrument.

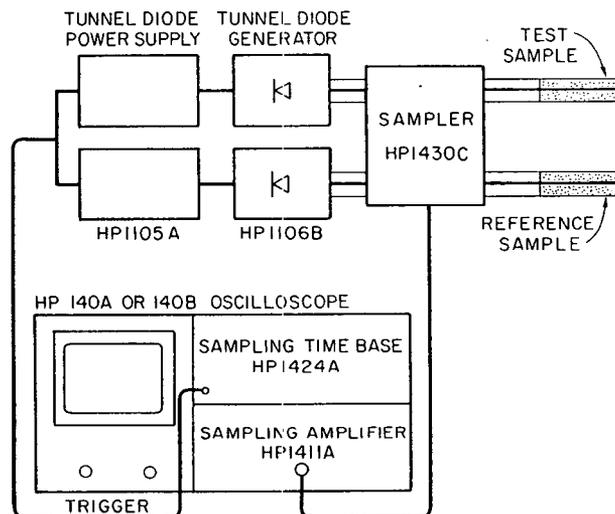


Figure 8.7 Differential TDR measurement with the HP 1430C sampler.

COMPUTERIZED SAMPLING SYSTEMS

Data acquisition and data handling by computer is not only quite convenient but allows also to improve considerably the measurement accuracy. This improvement can be achieved by multiple scanning, which reduces base line drift and enhances the signal to noise ratio, and by referencing to a time marker, which suppresses time jitter and drift. Multiple scanning and time averaging are well known and widely used. The need for a time marker, however, requires some explanation: When making the transfor-

mation into the frequency domain, it is advantageous to divide the resulting amplitude spectrum by the input spectrum of the measuring set-up in order to get the undistorted response due to the dielectric. The input spectrum may be obtained by inserting a small discontinuity in place of the sample or by using a reference sample with no relaxation, and transforming the time response. However, any time difference between the onset of sample and reference pulse produces a phase shift between the two frequency spectra and gives erroneous results. As Suggett⁽³⁾⁽⁴⁾ and Nicolson and Ross⁽⁸⁾ pointed out, time differences of only one picosecond can cause significant measurement errors. It is advisable to use the three point scanning method of Nicolson⁽⁹⁾ whereby each computer controlled scan starts at a point (1) (Fig. 8.9) prior to the onset of the initial pulse and establishes the base line. This can be done for reflected or transmitted pulses with the single reflection or thin sample method. The computer then seeks a second point on the leading edge of the initial pulse at a given signal value ΔV . This point (2), being on a steep slope, defines the time reference accurately. The data collecting scan then starts at any given point (3), which is referenced to (2) in time and to (1) in amplitude. The time and base line reference can be carried out prior to each data point or prior to each scan.

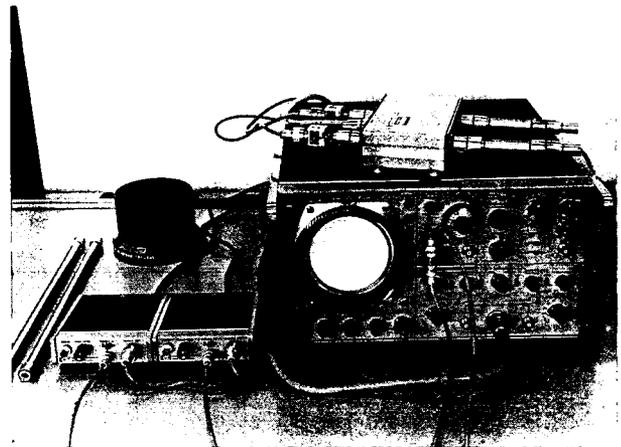


Figure 8.8 Differential TDR measurement with the HP 1430C sampler.

There are a number of Hewlett-Packard computerized sampling systems available. The simplest approach is to use an existing 140-system, to drive the external time base from the computer (for example -hp- 2100A) via a digital-analog converter (-hp- 12555A) and to acquire the data from the Y-recorder output via an analog converter (-hp-12564A). This system is simple,

(8) A.M. Nicolson, G.F. Ross, IEEE Trans. Instr. and Meas. **IM-19**; No. 4, 377 (1970).

(9) A.M. Nicolson, WESCON Techn. Papers. **13**; 4, 22/3 (1969)

inexpensive and easy to install. The Cole-Cole plot in Figure 8.10 has been obtained with this set-up. The speed of data acquisition is about 10 ms per data point. A more elaborate system, which is extremely convenient to operate, is the -hp- 5450 Fourier Analyzer. It features a keyboard with function keys for data acquisition, Fourier transformation, inverse transform, convolution, correlation, etc. The user is free to concentrate on the measurement problem and does not need to be concerned about software and system configuration.

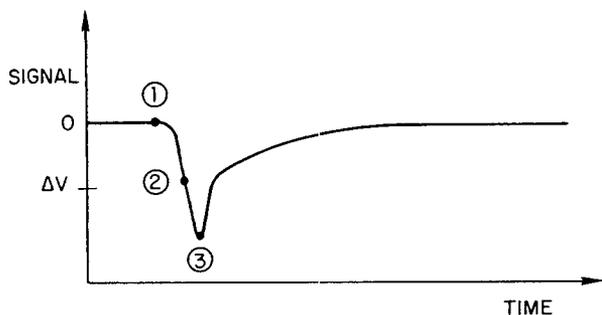


Figure 8.9 Three point method for reducing base line and time drift Reference (8) and (9).

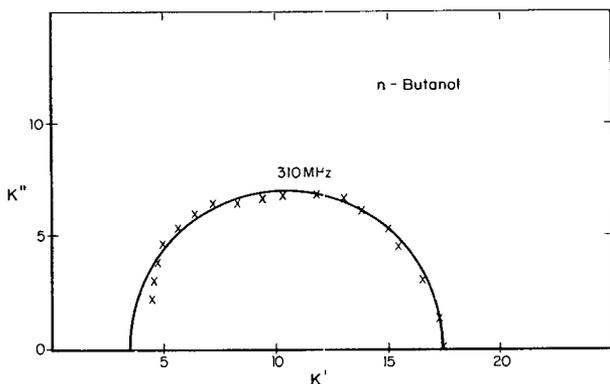


Figure 8.10 Cole-Cole plot of n-Butanol 25°C.

APPLICATIONS

The greatest assets of this method are speed, simplicity, and ease of data evaluation. In the past, the ability to characterize dielectrics over a wide frequency range was limited to a few laboratories and the number of substances investigated was restricted by the time and effort required. Multiple reflections, which are ever present in measurements in the frequency domain, require careful attention and make data evaluation tedious. In contrast, the display on the oscilloscope screen of the time domain reflectometer is easy to interpret, displays immediate information about the behavior of the material investigated, and shows if the instrument is operating properly and if any unwanted reflections are present. The measurement range for relaxation times of about 50 ps to 200 ns (corresponding to a frequency range of about 1 MHz

to 3 GHz) covers a region of great interest. The accuracy will be sufficient for many applications and can be improved with special techniques mentioned earlier in this section. The methods described by Suggestt et al (3) show that time domain measurements can be made as accurate as frequency domain measurements.

A number of applications have been tried or were suggested by different people. The characterization of dielectric and magnetic materials for quality control and incoming inspection can be done in a very short time and with a minimum of sample preparation. The time scale and vertical sensitivity of the instrument can be adjusted to get an optimum display for each substance tested, and an overlay for the oscilloscope screen can be used to define the range of acceptance. The information content is high since three parameters are obtainable from one measurement: κ_0 , κ_1 , τ or σ .

In many materials, the high frequency permittivity is quite sensitive to impurities of small free molecules, such as water, monomers, etc. These impurities have very short relaxation times...sometimes below the time resolution of the time domain reflectometer... and relatively high specific permittivities. On the other hand, host materials such as polymers have high frequency dielectric constants in the range of $\kappa_1 = 2$ to 8. The high accuracy of TDR in this range (see Figure 6.1) allows the determination of less than 1000 ppm of water, which is of interest for the plastics industry. Adjusting the ratio of inner to outer diameter of the line to give 50 ohms impedance when filled with the dielectric and immersing the material in a silicon oil of the same dielectric constant makes this measurement quite precise. To our knowledge, there is no other simple and nondestructive method available for this test and a continuous monitoring system for impurities is possible.

Since the measurement speed is essentially limited by the scan density and the pulse generator repetition rate, which is roughly 100 kHz when triggered from the sampling time base, very fast reactions can be monitored. Using the HP 141 or 181 variable persistence and storage oscilloscope and adjusting the repetition rate of the pulse generator according to the reaction rate, allows storing a number of traces of the reflection coefficient vs. time on the oscilloscope screen and gives immediate information about changes taking place during the reaction. The stored traces can then be photographed and evaluated in detail.

A relatively slow reaction which has been observed with TDR is the polymerization of epoxy resins inside a coaxial line. Figures 8.11 and 8.12 show the time dependence of the dielectric constant and relaxation time for different polymerization times. The strong change of the static permittivity κ_0 with time at short polymerization times is due to the free rotation of the monomer of the epoxide (glycerin diepoxide) and the catalyst (aliphatic diamine). This change becomes less as the polymerization progresses. Also, the high frequency dielectric constant changes with time in-

dicating that the amino- and oxy-groups of the monomers, which have very short relaxation times, are reacting during the polymerization. The difference between the dielectric constant in the infrared region

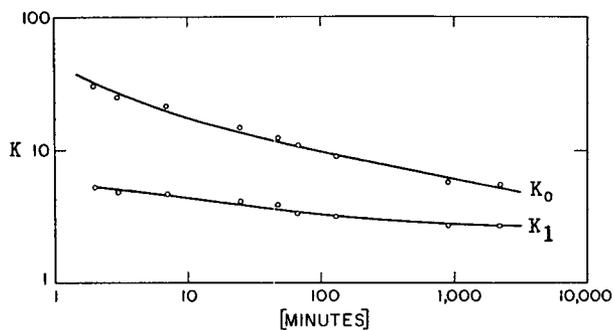


Figure 8.11 Polymerization of glycerin diepoxide with an aliphatic diamin: High frequency and static permittivity vs. polymerization time. This resin appeared hard after 1 hour, yet some reaction is still going on after 15 hours.

and the measured κ_1 of the fully polymerized material gives further information about the structure of this resin. Obviously, TDR can be applied in a similar way for monitoring chemical reactions on a production scale.

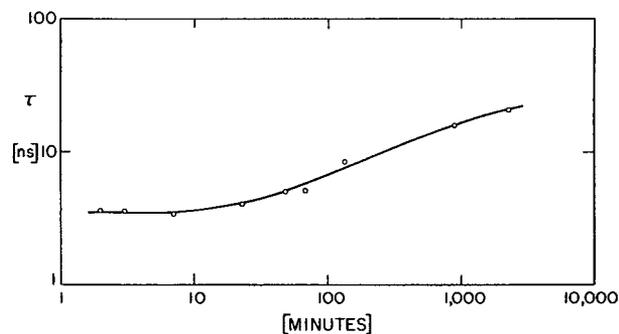


Figure 8.12 Polymerization of glycerin diepoxide with an aliphatic diamin: The relaxation time goes up as the monomers polymerize and form, on the average, dimers, trimers, and so on.

APPENDIX I

THE CORRELATION BETWEEN TIME AND FREQUENCY DOMAIN

It is well known that distortion of a sinusoidal signal, with its signal power concentrated at one frequency ω_0 , produces harmonic frequencies $m\omega_0$, thus distributing the power over a certain frequency range. In a more general way, we can state that any time dependent signal can be expressed in terms of its frequency spectrum. The quantitative correlation between the time domain and the frequency domain is given by Fourier or Laplace transform. We will use the Laplace transform, which is more appropriate for time domain reflectometry, and will give a few basic facts which may help to understand the dielectric measurements in the time domain. The reader may consult any one of a number of books on transform calculus for a deeper understanding.

The transform from the time domain into the frequency spectrum is given by

$$F(\omega) = \int_0^{+\infty} f(t) \cdot e^{-i\omega t} dt \quad (1)$$

As an example, we will make the Laplace transform of a square pulse train with pulse length t_1 , repetition time t_0 , and unity amplitude. The integral then becomes

$$F(\omega) = \sum_{n=0}^{+\infty} \int_{nt_0}^{nt_0+t_1} e^{-i\omega t} dt \quad (2)$$

since $f(t) = 1$ for every time interval t_1 , starting at $n \cdot t_0$, and $f(t) = 0$ for the remainder of the time. Thus

$$\begin{aligned} F(\omega) &= \frac{i}{\omega} \sum_{n=0}^{+\infty} e^{-i\omega t} \left[\begin{array}{l} nt_0 + t_1 \\ nt_0 \end{array} \right] \\ &= \frac{i}{\omega} (e^{-i\omega t_1} - 1) \sum_{n=0}^{+\infty} e^{-in\omega t_0} \end{aligned}$$

Since

$$\begin{aligned} &i (\exp(-i\omega t_1) - 1) \\ &= 2 \frac{\left(\exp\left(\frac{i\omega t_1}{2}\right) - \exp\left(\frac{-i\omega t_1}{2}\right) \right) \exp\left(\frac{-i\omega t_1}{2}\right)}{2i} \\ &= 2 \sin \frac{\omega t_1}{2} \cdot \exp\left(\frac{-i\omega t_1}{2}\right) \end{aligned}$$

we get

$$|F(\omega)| = \frac{2}{\omega} \sin \frac{\omega t_1}{2} \sum_{n=0}^{n=+\infty} e^{-in\omega t_0} \quad (3)$$

The summation term in equation (3) is different from zero only for $\omega t_0 = 2\pi m$ ($m = 1, 2, 3, \dots$) or

$$f_{m-1} = \frac{\omega}{2\pi} = \frac{m}{t_0} \quad (4)$$

Equation (4) represents the fundamental frequency $f_0 = \frac{1}{t_0}$ and its harmonics. Their amplitude distribution is given by the absolute value of the complex term in equation (3). Thus

$$|F(\omega)| = t_1 \cdot \frac{\sin \frac{\omega t_1}{2}}{\frac{\omega t_1}{2}} \quad (5)$$

The frequency spectrum envelope corresponding to a single step of unit height at $t = 0$ is

$$F(\omega) = \int_0^{\infty} e^{-i\omega t} dt = \frac{1}{i\omega} \quad (6)$$

$$|F(\omega)| = \frac{1}{\omega}$$

The frequency spectrum of the square pulse train, according to equation (3), is given in Figure AI-1d. We can draw a number of important conclusions:

1. The spectrum contains only the fundamental frequency and its harmonics. Pulse spacing depends only on the repetition time t_0 of the pulse train. The lower the repetition rate the closer the spacing (see Figure AI-1e). Therefore, the spectrum of a single pulse becomes a continuum.
2. The nodes in the amplitude distribution depend only on the pulse length t_1 and move closer together for longer pulses.

3. A fast risetime pulse contains higher harmonics than a slow risetime pulse and can be seen by comparing the spectrum of a rectangular and of a triangular pulse in Figure AI-1c. It becomes even clearer in Figure AI-2 where we have plotted the frequency spectrum of single pulses of finite and infinite length with different risetimes.

4. The high frequency part of the spectrum depends on the shape of the pulse in the vicinity of $t = 0$ and vice versa. In Figure AI-2, the high frequency spectrum of pulses is identical if the risetime at the start of the pulse is the same. Therefore, approximations for certain spectral ranges can be made if it is difficult to obtain the Laplace transform for the entire pulse.

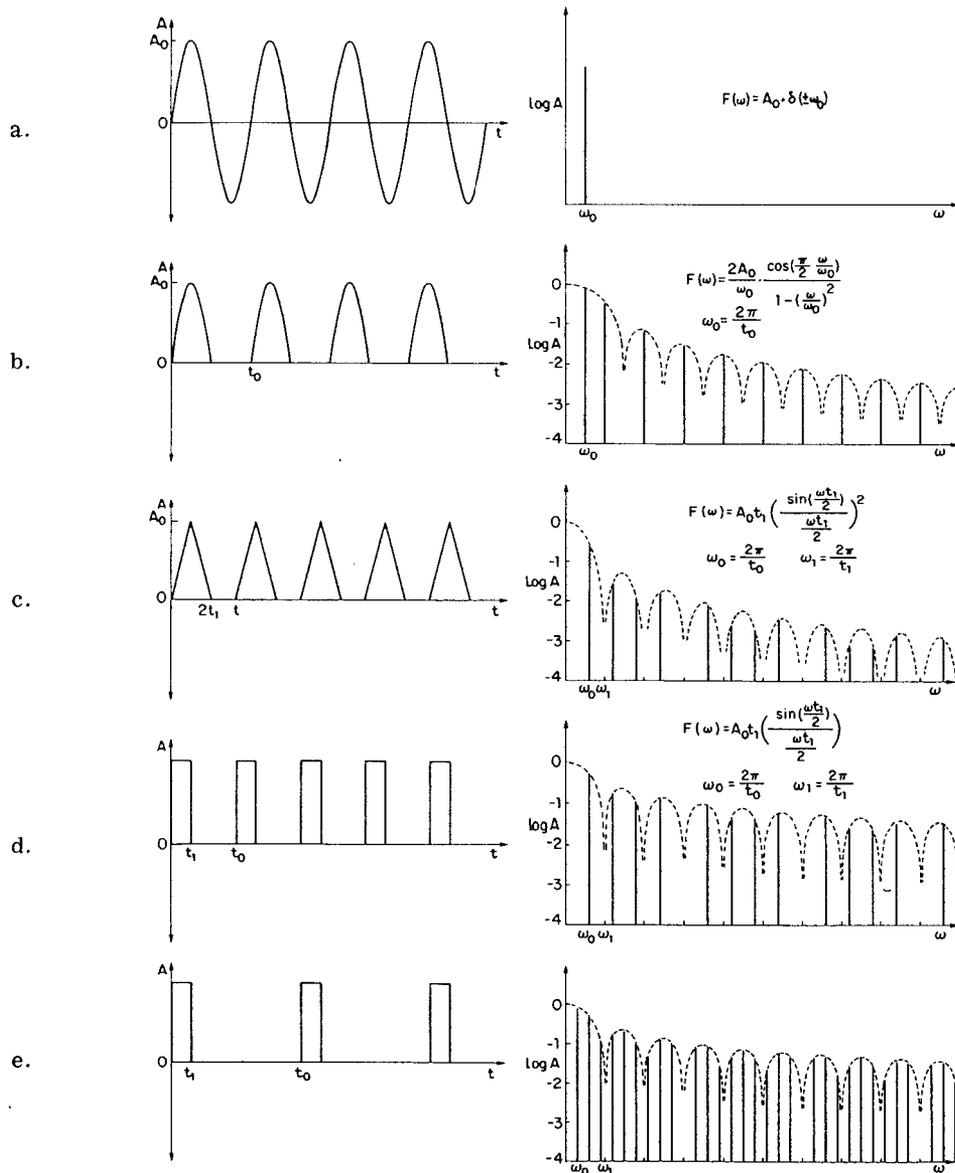


Figure AI-1 Frequency spectrum of various pulse trains

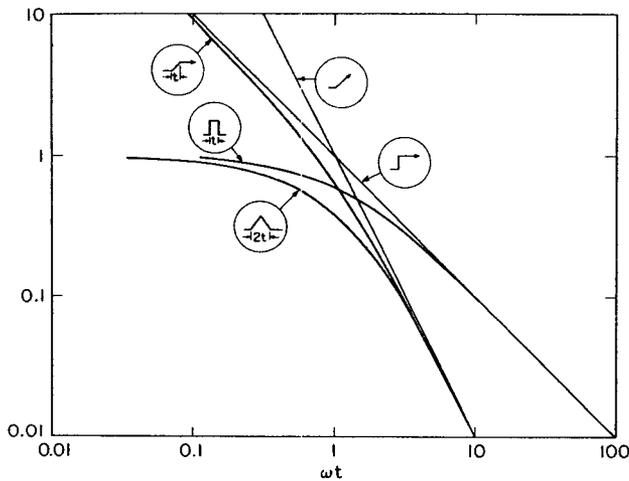


Figure AI-2 Frequency spectrum of single pulses of unit height and/or unit slope with $t = 1$.

We have used one other theorem in the text which is explained briefly: The frequency response $H(\omega)$ of any system to an input signal $G(\omega)$ can be represented by its transfer function $F(\omega)$ which is

$$H(\omega) = G(\omega) \cdot F(\omega)$$

Laplace transform into the time domain gives

$$h(t) = g(t) * f(t) = \int_{\tau=0}^t f(\tau) \cdot g(\tau - t) d\tau. \quad (7)$$

The simple product of input signal with the transfer function in the frequency domain becomes a convolution in the time domain. However, for an infinitely short pulse of unit area (δ -function) the convolution integral in equation (7) becomes

$$h(t) = \int_{\tau=0}^t f(\tau) \cdot \delta(\tau - t) d\tau = f(t)$$

since

$$\int \delta(\tau - t) = 0 \text{ for } \tau = t \text{ and} \\ 1 \text{ for } \tau = t.$$

The frequency spectrum of the δ -function is constant and unity, giving

$$H(\omega) = F(\omega) \cdot \delta(\omega) = F(\omega).$$

The integral of $\delta(t)$ is a step pulse, $u(t)$, with infinitely short risetime and unit height. $\int u(t) dt$ in turn is a ramp, $s(t)$, with unit slope. Also the transform of an integral

$$f(t) = \int f'(t) dt \quad \text{is}$$

$$F(\omega) = \frac{1}{\omega} F'(\omega),$$

the transform of the integrand divided by ω . Since $\delta(\omega) = 1$, we get $u(\omega) = \frac{1}{\omega}$ and $s(\omega) = \frac{1}{\omega^2}$. This means,

however, that the response of a system to a step pulse

$$H(\omega)_{\text{step}} = \frac{1}{\omega} \cdot F(\omega) \quad \text{is} \quad (8)$$

$$h(t)_{\text{step}} = \int f(t)_{\delta} dt,$$

the integral of the system response. And, consequently,

$$h(t)_{\text{ramp}} = \iint f(t)_{\delta} dt^2. \quad (9)$$

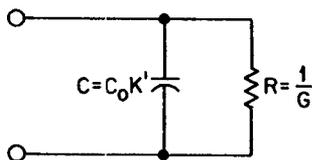
We used equations (8) and (9) in computing the response of a dielectric to a step pulse and to a pulse with linear risetime.

APPENDIX II

THE PRINCIPLE OF DIELECTRIC MEASUREMENTS

Many measurements of dielectric properties, particularly at low frequencies, are made by measuring the impedance of a capacitor with and without the test material between the two plates. A number of measurements at different frequencies over a wide range are required for the characterization of dielectrics.

The equivalent circuit for a capacitor filled with a dielectric is



The admittance Y ($= 1/Z$, Z : impedance) of this circuit is

$$Y = j2\pi fC + G = j\omega C + G = jB + G \quad \begin{array}{l} B : \text{Susceptance} \\ G : \text{Conductance} \end{array}$$

The admittance of the same capacitor without dielectric would be

$$Y_0 = j\omega C_0$$

The dielectric changes Y_0 to Y . Therefore, we multiply Y_0 with a complex factor, the permittivity $\kappa^* = \kappa' + j\kappa''$, to get Y :

$$\begin{aligned} Y &= \kappa^* \cdot Y_0 = (\kappa' - j\kappa'') Y_0 \\ &= j\omega C_0 (\kappa' - j\kappa'') \quad (1) \\ &= j\omega \kappa' C_0 + \omega \kappa'' C_0 \\ &= j\omega C + G \end{aligned}$$

and

$$\begin{aligned} C = \kappa' C_0 \quad \text{or} \quad \kappa' &= \frac{C}{C_0} \\ G = \omega \kappa'' C_0 \quad \cdot \quad \kappa'' &= \frac{G}{\omega C_0} \end{aligned} \quad (2)$$

$$G [\Omega^{-1}]$$

$$C, C_0 [\text{Farad}]$$

The dielectric constant κ' is a measure of the degree of electrical polarization in the material, due either to formation of dipoles by induced polarization or the orientation of permanent dipoles. This polarization in an electric field produces free charges at the surface of the material which, in turn, tends to reduce the electric field inside the material. The surface charge density P is given by

$$P = E(\kappa' - 1) \quad (3)$$

The dielectric loss may be due either to conductivity (electronic or ionic) in material or to energy dissipation of rotating permanent dipoles in an alternating field. For the former, the conductance can be simply related to the conductivity (σ) of the material by

$$\frac{G}{C_0} = 4\pi\sigma = 4\pi/\rho = \omega\kappa''$$

since (for a plate capacitor)

$$G = \sigma \cdot \frac{A}{d} [\Omega^{-1}] \quad C_0 = \frac{A}{4\pi d} [\text{cm}].$$

ρ : resistivity

A : area of plates

d : spacing between plates

The theory for the behavior of permanent dipoles has been developed by Debye^(1,2) and Onsager⁽³⁾, using in part Langevin's⁽⁴⁾ theory of permanent magnetic dipoles. We can give the results only and have to refer the reader to the literature for further details:

At low frequencies the dipoles will have time enough to orient themselves in the electric field, giving a high dielectric constant. At high frequencies, the dipoles cannot follow the fast changing field; they remain randomly oriented and do not contribute to the dielectric constant. The transition occurs in the vicinity of a certain frequency $2\pi f_0 = \omega_0$ and at this

(1) P. Debye, Phys. Z. B, 97 (1912).

(2) P. Debye, Polar Molecules, Dover Publ., N.Y.

(3) L. Onsager, J. Am. Chem. Soc. 58, 1486 (1936).

(4) M.P. Langevin, J. Physique 4, 678 (1905).

frequency, the dielectric loss has a maximum value. The solution of the differential equation

$$\mu E e^{j\omega t} \cos \theta - \eta \frac{d\theta}{dt} = 0 \tag{4}$$

θ : angle between dipole and field E
 μ : dipole moment
 η : friction constant

with appropriate boundary conditions and for an electric field $E < \frac{\mu}{KT}$ [corresponding to $< 10^7$ V/cm at room temperature and $\mu = 1$ (Debye)] gives

$$\chi^*(\omega) = \chi'(\omega) + j\chi''(\omega) = \chi_1 + \frac{(\chi_0 - \chi_1)}{(1 + j\omega\tau)} \tag{5}$$

χ_0 : low frequency dielectric constant
 χ_1 : high frequency dielectric constant

After separation into real and imaginary parts one gets

$$\chi'(\omega) = \chi_1 + \frac{\chi_0 - \chi_1}{1 + \omega^2 \tau^2} \tag{6}$$

$$\chi''(\omega) = \frac{(\chi_0 - \chi_1) \cdot \omega\tau}{1 + \omega^2 \tau^2} \tag{7}$$

τ : relaxation time

This function is shown in Figure AII-1.

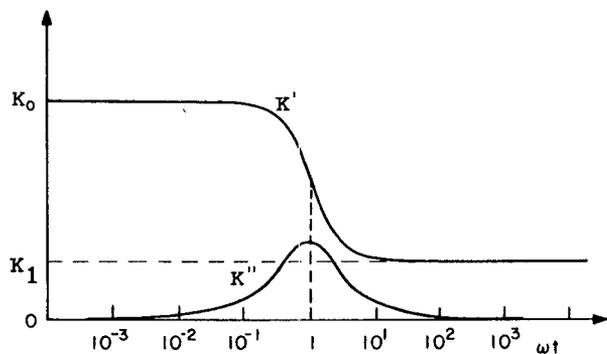
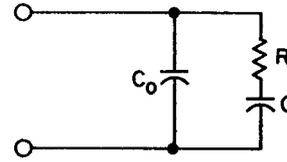


Figure AII-1 Real and imaginary part of the permittivity, χ' and χ'' respectively, vs. frequency.

The equivalent circuit for a capacitor filled with such a dielectric is



The Laplace transform of equation (5) gives

$$\chi(t) = \delta(\chi_1) + \frac{(\chi_0 - \chi_1)}{\tau} e^{-\frac{t}{\tau}} \tag{8}$$

which is the response of the permittivity to an infinitely short voltage impulse whose time integral is unity (δ -function) and which produces the permittivity χ_0 . The contribution to capacitance due to the permittivity χ_1 decays immediately, while $(\chi_0 - \chi_1)$ decays according to (8).

It comes closer to a practical approach to consider the response of the system to a voltage step pulse $u(t)$ which is the integral of a δ -function ($u(t) = 0$ for $t < 0$, $u(t) = 1$ for $t > 0$). Therefore, the time response of permittivity is the integral of equation (8). We get

$$\chi(t) = \chi_1 + (\chi_0 - \chi_1) (1 - e^{-\frac{t}{\tau}}) \text{ with } \chi(0) = \chi_1 \tag{9}$$

see Figure AII-2.

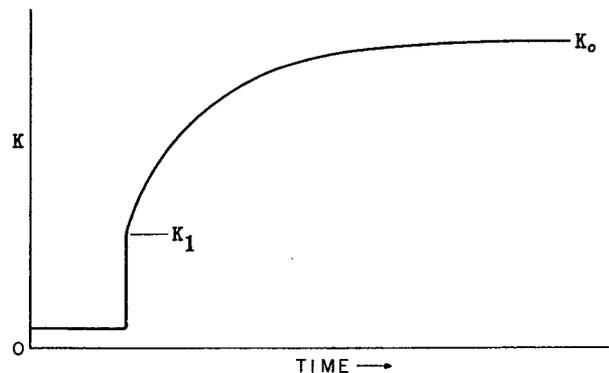


Figure AII-2 Step response of the permittivity of an ideal dielectric.

The curve starts at κ_+ , since we assume that the time constant of this contribution to permittivity is very small compared with the time scale used. The permittivity then rises to κ_0 with the time constant τ .

There may be other dispersion regions of the permittivity. Particularly at short relaxation times there may be fast orientation polarization of small dipoles, induced or dipole orientation of atoms, and induced electron polarization. All these contribute to κ_1 , the high frequency permittivity. The following table gives some information about relaxation time, frequency, and wavelength range where these processes occur (for Debye behavior).

Whenever there is an interaction between the oriented group or molecule and the surrounding medium, the viscosity will have an influence on the relaxation time. Since the viscosity of solids and liquids is

strongly temperature dependent, the relaxation time of the first three groups also shows strong temperature dependence. Functional groups are generally small enough to move within the sphere in influence of their own molecule and the viscosity loses its meaning.

It may be pointed out now that the Hewlett-Packard Time Domain Reflectometer covers a range of $3 \cdot 10^{11}$ to 10^{-6} seconds which allows measuring the orientation of molecules and some functional groups in solids and liquids. This is a region of great theoretical and practical interest. Longer relaxation times can be measured with rather simple bridge and pulse circuits. It would be desirable to be able to measure relaxation times of a few picoseconds with TDR for identification of specific functional groups. At the moment, there is no indication that this will be possible soon and such measurements have to be made in the frequency domain.

TABLE AII-1

Relaxation Mechanism	Relaxation Time [sec]	Frequency [sec ⁻¹]	Wavelength [cm]	Temp. Depend. of Relax. Time
1) Orientation of molecular (?) groups in solid polymers	$10^4 - 10^{-4}$	$1.6 \cdot 10^{-5} - 1.6 \cdot 10^3$	$2 \cdot 10^{15} - 2 \cdot 10^7$	Strong
2) Orientation of functional groups in solids	$10^{-5} - 10^{-11}$	$1.6 \cdot 10^4 - 1.6 \cdot 10^{10}$	$2 \cdot 10^6 - 2$	Medium
3) Orientation of molecules in liquids	$10^{-6} - 10^{11}$	$1.6 \cdot 10^5 - 1.6 \cdot 10^{10}$	$2 \cdot 10^5 - 2$	Strong
4) Orientation of functional groups in liquids	$10^{-9} - 10^{-12}$	$1.6 \cdot 10^8 - 1.6 \cdot 10^{11}$	$2 \cdot 10^2 - 0.2$	Small
5) Orientation of molecules in gases	$10^{-8} - 10^{-12}$	$1.6 \cdot 10^7 - 1.6 \cdot 10^{11}$	$2 \cdot 10^3 - 0.2$	Small
6) Atom polarization (rotational and vibrational)	$5 \cdot 10^{-16} - 10^{-14}$	$3 \cdot 10^{14} - 1.5 \cdot 10^{13}$	$10^{-4} - 2 \cdot 10^{-3}$	None
7) Electron polarization	$1.6 \cdot 10^{-16} - 1.6^{-15}$	$10^{15} - 10^{14}$	$3 \cdot 10^{-5} - 3 \cdot 10^{-4}$	None

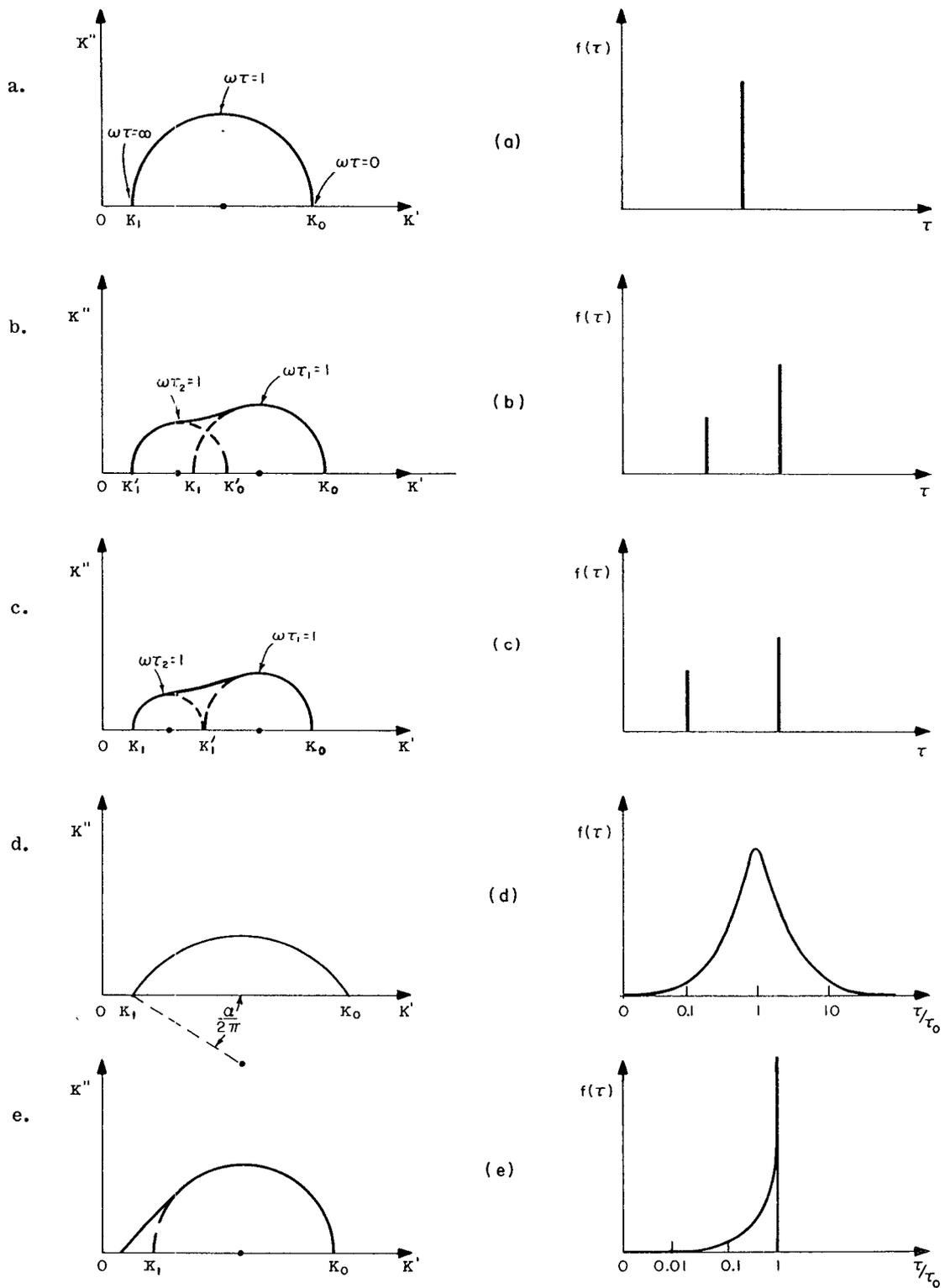


Figure AII-3 Cole & Cole diagram of relaxation spectrum for different types of dielectrics.

In the case of time domain reflectometry the Laplace transform of the reflection coefficient has to be made, as mentioned on Page 4. The mathematical treatment for Debye dielectrics is given by H. Fellner-Feldegg and F. Barnett.⁽⁵⁾

The differential equation (4) assumes a single and constant dipole moment and friction constant, resulting in one single relaxation time. If there are two independent relaxation times of about the same magnitude in one substance or in a mixture of two species, then the permittivity is given as a linear addition of two terms with relaxation times τ_1 and τ_2 in equation (10). This addition is preserved when making the transform into the frequency domain. Thus

$$\kappa^*(\omega) = \kappa_1 + \frac{\kappa_0 - \kappa_1}{1 + j\omega\tau_1} + \frac{\kappa'_0 - \kappa'_1}{1 + j\omega\tau_2} \quad (10)$$

$$\begin{aligned} \kappa(t) = & \kappa_1 + (\kappa_0 - \kappa_1) \left(1 - e^{-\frac{t}{\tau_1}}\right) \\ & + (\kappa'_1 - \kappa'_0) \left(1 - e^{-\frac{t}{\tau_2}}\right) \end{aligned} \quad (11)$$

In the frequency domain, Cole & Cole⁽⁶⁾ have shown that the real and imaginary part of the permittivity, plotted in the complex plane for different frequencies, gives a semicircle for ideal Debye relaxation (see Figure AII-3a). Consequently, for the case of two independent relaxation times, one gets a plot shown in Figure 3b. Figure 3c shows the Cole & Cole plot for a substance with two well separated relaxation times, for example, an aliphatic alcohol. In the time domain, the logarithmic plot results in two straight lines with entirely different slopes.

If inter- and intramolecular interactions become important, then single relaxation times broaden into relaxation spectra. Fuoss and Kirkwood⁽⁷⁾ and Cole⁽⁸⁾ assume a relaxation spectrum which is symmetrical on a logarithmic scale about a central relaxation time τ_0 . The empirical equation of Cole fits the data to an arc of a circle in the Cole & Cole diagram and is of the form

$$\kappa^*(\omega) = \kappa_1 + \frac{\kappa_0 - \kappa_1}{1 + (j\omega\tau_0)^{1-\alpha}} \quad (12)$$

Finally, glycerol and other substances show a highly asymmetric Cole & Cole diagram, described by Davidson & Cole⁽⁹⁾ (Figure 3e) with

$$\kappa^*(\omega) = \kappa_1 + \frac{\kappa_0 - \kappa_1}{(1 + j\omega\tau_0)^\beta} \quad (13)$$

(5) H. Fellner-Feldegg, F. Barnett
J. Phys. Chem. 74, 1962 (1970).

(6) K. S. Cole, R. H. Cole, J. Chem.
Phys. 9, 341 (1941).

(7) R. M. Fuoss, J. G. Kirkwood, J. Am. Chem.
Soc. 63, 385 (1941).

(8) R. H. Cole, J. Chem. Phys. 23, 493 (1955).

(9) D. W. Davidson, R. H. Cole, J. Chem. Phys.
19, 1484 (1951).



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