Agilent
Basics of Measuring the Dielectric Properties of Materials

Application Note

Agilent Technologies

Contents

Introduction

Every material has a unique set of electrical characteristics that are dependent on its dielectric properties. Accurate measurements of these properties can provide scientists and engineers with valuable information to properly incorporate the material into its intended application for more solid designs or to monitor a manufacturing process for improved quality control.

A dielectric materials measurement can provide critical design parameter information for many electronics applications. For example, the loss of a cable insulator, the impedance of a substrate, or the frequency of a dielectric resonator can be related to its dielectric properties. The information is also useful for improving ferrite, absorber, and packaging designs. More recent applications in the area of industrial microwave processing of food, rubber, plastic and ceramics have also been found to benefit from knowledge of dielectric properties.

Agilent Technologies Inc. offers a variety of instruments, fixtures, and software to measure the dielectric properties of materials. Agilent measurement instruments, such as network analyzers, LCR meters, and impedance analyzers range in frequency up to 325 GHz. Fixtures to hold the material under test (MUT) are available that are based on coaxial probe, coaxial/waveguide transmission line techniques, and parallel plate.

Dielectric Theory

The dielectric properties that will be discussed here are permittivity and permeability. Resistivity is another material property which will not be discussed here. Information about resistivity and its measurement can be found in the Agilent Application Note 1369-1[1]. It is important to note that permittivity and permeability are not constant. They can change with frequency, temperature, orientation, mixture, pressure, and molecular structure of the material.

Dielectric constant

A material is classified as "dielectric" if it has the ability to store energy when an external electric field is applied. If a DC voltage source is placed across a parallel plate capacitor, more charge is stored when a dielectric material is between the plates than if no material (a vacuum) is between the plates. The dielectric material increases the storage capacity of the capacitor by neutralizing charges at the electrodes, which ordinarily would contribute to the external field. The capacitance with the dielectric material is related to dielectric constant. If a DC voltage source V is placed across a parallel plate capacitor (Figure 1), more charge is stored when a dielectric material is between the plates than if no material (a vacuum) is between the plates.
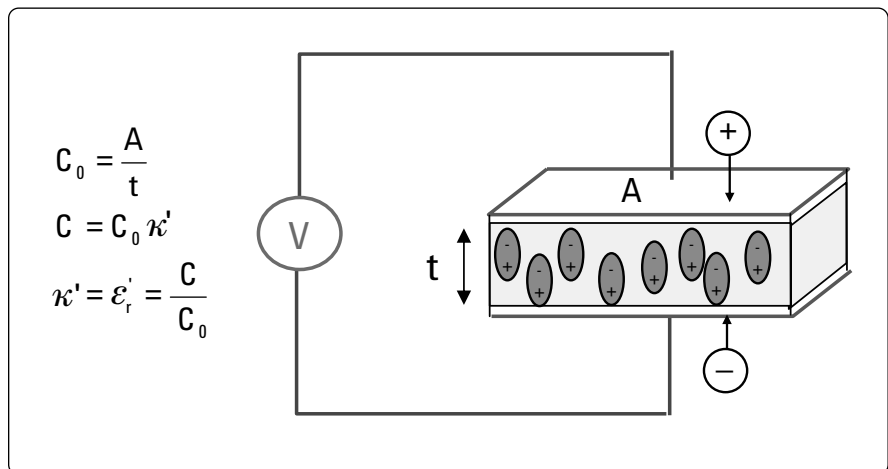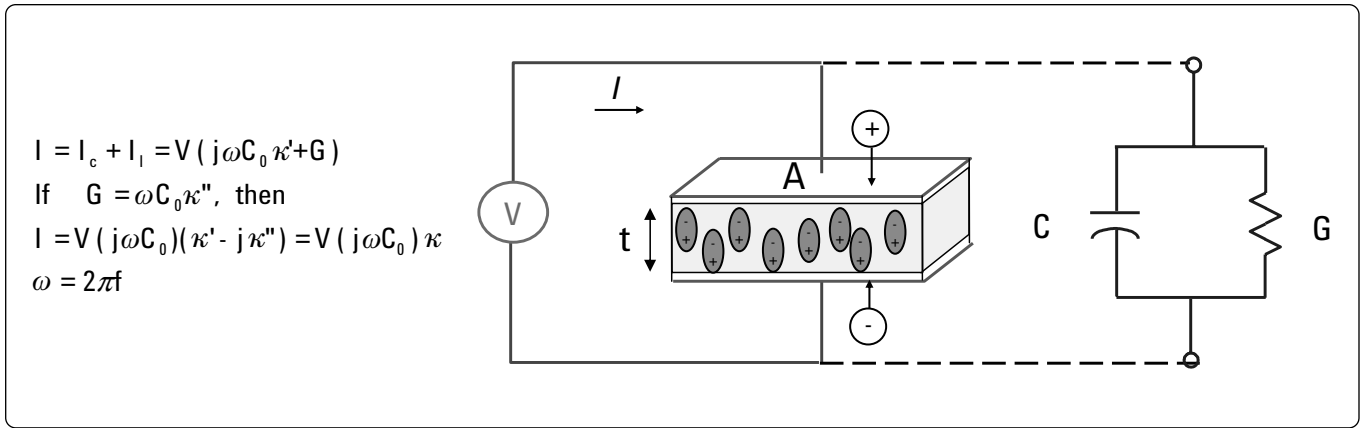

$$C_0 = \frac{A}{t}$$

$$C = C_0 \, \kappa'$$

$$\kappa' = \varepsilon_r' = \frac{C}{C_0}$$

Figure 1. Parallel plate capacitor, DC case

Where C and C_0 are capacitance with and without dielectric, $\kappa' = \varepsilon_r'$ is the real dielectric constant or permittivity, and A and t are the area of the capacitor plates and the distance between them (Figure 1). The dielectric material increases the storage capacity of the capacitor by neutralizing charges at the electrodes, which ordinarily would contribute to the external field. The capacitance of the dielectric material is related to the dielectric constant as indicated in the above equations. If an AC sinusoidal voltage source V is placed across the same capacitor (Figure 2), the resulting current will be made up of a charging current I_c and a loss current I_l that is related to the dielectric constant. The losses in the material can be represented as a conductance (G) in parallel with a capacitor (C).

$I = I_c + I_l = V(j\omega C_0 \kappa' + G)$

If $\quad G = \omega C_0 \kappa''$, then

$I = V(j\omega C_0)(\kappa' - j\kappa'') = V(j\omega C_0)\kappa$

$\omega = 2\pi f$

Figure 2. Parallel plate capacitor, AC case

The complex dielectric constant κ consists of a real part κ' which represents the storage and an imaginary part κ'' which represents the loss. The following notations are used for the complex dielectric constant interchangeably $\kappa = \kappa^* = \varepsilon_r = \varepsilon_r^*$.

From the point of view of electromagnetic theory, the definition of electric displacement (electric flux density) D_f is:

$D_f = \varepsilon E$

where $\varepsilon = \varepsilon^* = \varepsilon_0 \varepsilon_r$ is the absolute permittivity (or permittivity), ε_r is the relative permittivity, $\varepsilon_0 \approx \frac{1}{36\pi}$ x 10^{-9} F/m is the free space permittivity and E is the electric field.

Permittivity describes the interaction of a material with an electric field E and is a complex quantity.

$\kappa = \frac{\varepsilon}{\varepsilon_0} = \varepsilon_r = \varepsilon_r' - j\varepsilon_r''$

Dielectric constant (κ) is equivalent to relative permittivity (ε_r) or the absolute permittivity (ε) relative to the permittivity of free space (ε_0). The real part of permittivity (ε_r') is a measure of how much energy from an external electric field is stored in a material. The imaginary part of permittivity (ε_r'') is called the loss factor and is a measure of how dissipative or lossy a material is to an external electric field. The imaginary part of permittivity (ε_r'') is always greater than zero and is usually much smaller than (ε_r'). The loss factor includes the effects of both dielectric loss and conductivity.

When complex permittivity is drawn as a simple vector diagram (Figure 3), the real and imaginary components are 90° out of phase. The vector sum forms an angle δ with the real axis (ε_r'). The relative "lossiness" of a material is the ratio of the energy lost to the energy stored.

$$\tan \delta = \frac{\varepsilon_r''}{\varepsilon_r'} = D = \frac{1}{Q}$$

$$= \frac{\text{Energy Lost per Cycle}}{\text{Energy Stored per Cycle}}$$
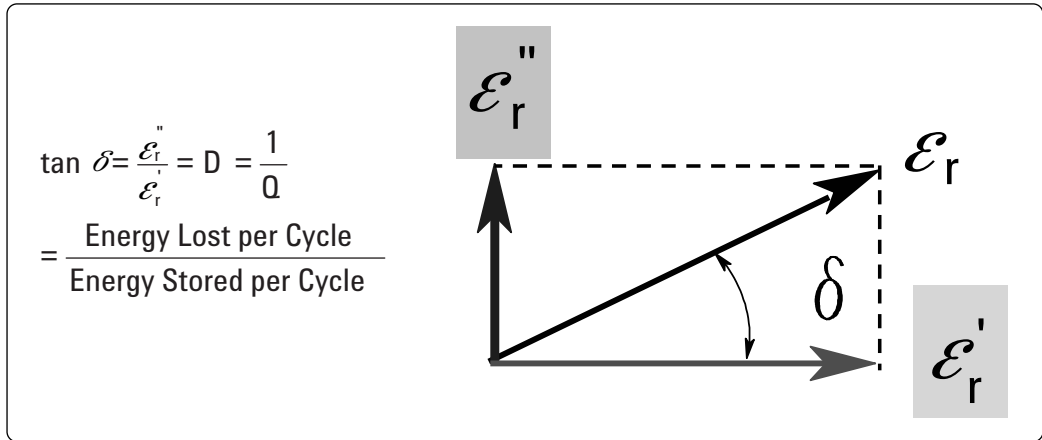
Figure 3. Loss tangent vector diagram

The loss tangent or tan δ is defined as the ratio of the imaginary part of the dielectric constant to the real part. D denotes dissipation factor and Q is quality factor. The loss tangent tan δ is called tan delta, tangent loss or dissipation factor. Sometimes the term "quality factor or Q-factor" is used with respect to an electronic microwave material, which is the reciprocal of the loss tangent. For very low loss materials, since tan $\delta \approx \delta$, the loss tangent can be expressed in angle units, milliradians or microradians.

Permeability

Permeability (µ) describes the interaction of a material with a magnetic field. A similar analysis can be performed for permeability using an inductor with resistance to represent core losses in a magnetic material (Figure 4). If a DC current source is placed across an inductor, the inductance with the core material can be related to permeability.

$$L = L_0 \mu'$$

$$\mu' = \frac{L}{L_0}$$
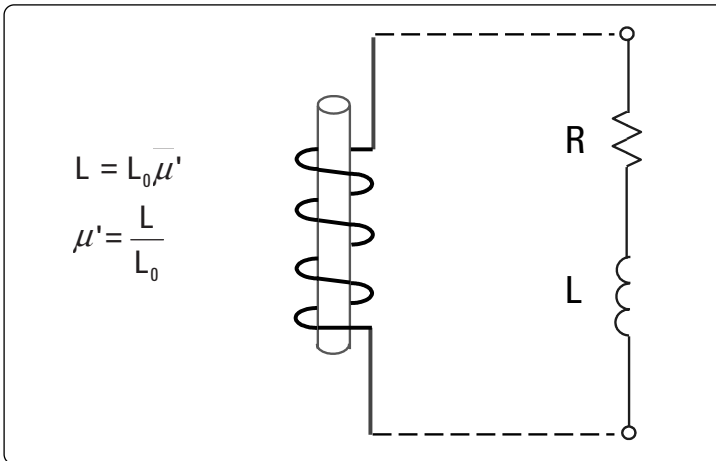
Figure 4. Inductor

In the equations L is the inductance with the material, L_0 is free space inductance of the coil and µ' is the real permeability. If an AC sinusoidal current source is placed across the same inductor, the resulting voltage will be made up of an induced voltage and a loss voltage that is related to permeability. The core loss can be represented by a resistance (R) in series with an inductor (L). The complex permeability (µ* or µ) consists of a real part (µ') that represents the energy storage term and an imaginary part (µ") that represents the energy loss term. Relative permittivity μ_r is the permittivity relative to free space:

$$\mu_r = \frac{\mu}{\mu_0} = \mu_r - j\mu_r{}''$$

$\mu_0 = 4\pi \times 10^{-7}$ *H/m* is the free space permeability

Some materials such as iron (ferrites), cobalt, nickel, and their alloys have appreciable magnetic properties; however, many materials are nonmagnetic, making the permeability very close to the permeability of free space ($\mu_r = 1$). All materials, on the other hand, have dielectric properties, so the focus of this discussion will mostly be on permittivity measurements.

Electromagnetic Wave Propagation

In the time-varying case (i.e., a sinusoid), electric fields and magnetic fields appear together. This electromagnetic wave can propagate through free space (at the speed of light, c = 3 x 10^8 m/s) or through materials at slower speed. Electromagnetic waves of various wavelengths exist. The wavelength λ of a signal is inversely proportional to its frequency f ($\lambda = c/f$), such that as the frequency increases, the wavelength decreases. For example, in free space a 10 MHz signal has a wavelength of 30 m, while at 10 GHz it is just 3 cm. Many aspects of wave propagation are dependent on the permittivity and permeability of a material. Let's use the "optical view" of dielectric behavior. Consider a flat slab of material (MUT) in space, with a TEM wave incident on its surface (Figure 5). There will be incident, reflected and transmitted waves. Since the impedance of the wave in the material Z is different (lower) from the free space impedance η (or Z_0) there will be impedance mismatch and this will create the reflected wave. Part of the energy will penetrate the sample. Once in the slab, the wave velocity v, is slower than the speed of light c. The wavelength λ_d is shorter than the wavelength λ_0 in free space according to the equations below. Since the material will always have some loss, there will be attenuation or insertion loss. For simplicity the mismatch on the second border is not considered.

$$Z = \frac{\eta}{\sqrt{\varepsilon_r'}} \qquad \eta = Z_0 = \sqrt{\frac{\mu_0}{\varepsilon_0}} = 120\pi$$

$$\lambda_d = \frac{\lambda_0}{\sqrt{\varepsilon_r'}} \qquad v = \frac{c}{\sqrt{\varepsilon_r'}}$$

$$\eta \text{ or } Z_0 \qquad Z = \eta / \sqrt{\varepsilon_r'}$$

TEM

Air ε_0'

MUT ε_r'

Impedance lower
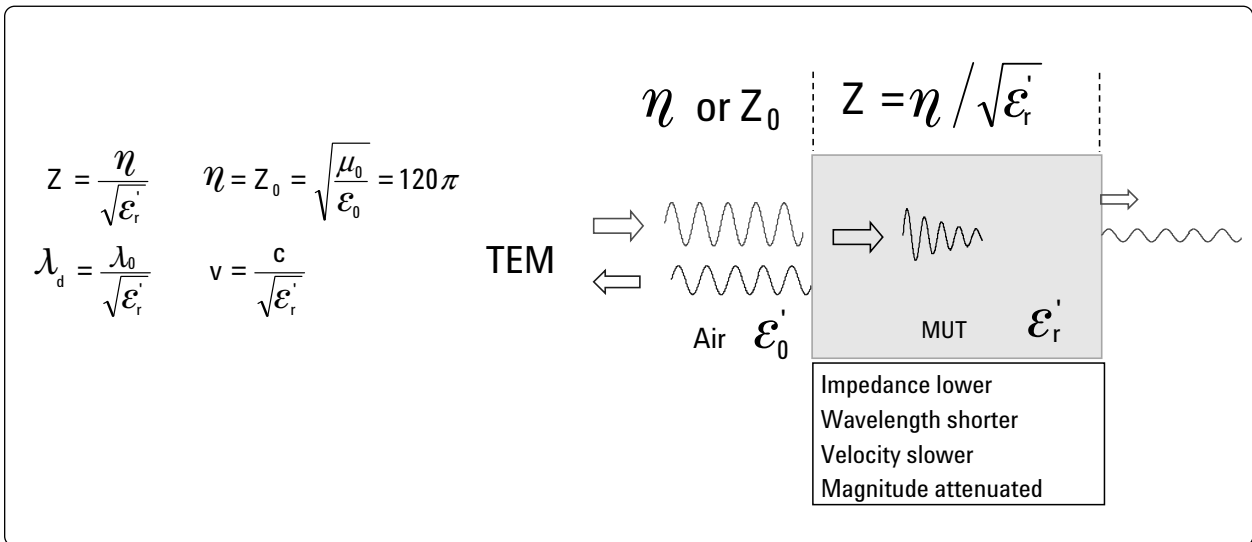Wavelength shorter
Velocity slower
Magnitude attenuated

Figure 5. Reflected and transmitted signals

Figure 6 depicts the relation between the dielectric constant of the Material Under Test (MUT) and the reflection coefficient $|\Gamma|$ for an infinitely long sample (no reflection from the back of the sample is considered). For small values of the dielectric constant (approximately less than 20), there is a lot of change of the reflection coefficient for a small change of the dielectric constant. In this range dielectric constant measurement using the reflection coefficient will be more sensitive and hence precise. Conversely, for high dielectric constants (for example between 70 and 90) there will be little change of the reflection coefficient and the measurement will have more uncertainty.
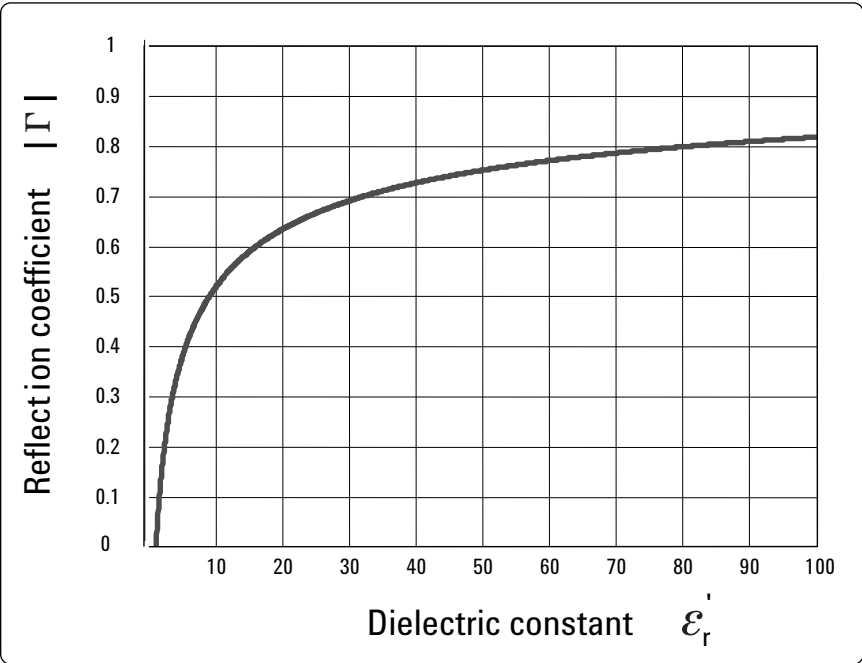

Figure 6. Reflection coefficient versus dielectric constant

Dielectric Mechanisms

A material may have several dielectric mechanisms or polarization effects that contribute to its overall permittivity (Figure 7). A dielectric material has an arrangement of electric charge carriers that can be displaced by an electric field. The charges become polarized to compensate for the electric field such that the positive and negative charges move in opposite directions.

At the microscopic level, several dielectric mechanisms can contribute to dielectric behavior. Dipole orientation and ionic conduction interact strongly at microwave frequencies. Water molecules, for example, are permanent dipoles, which rotate to follow an alternating electric field. These mechanisms are quite lossy – which explains why food heats in a microwave oven. Atomic and electronic mechanisms are relatively weak, and usually constant over the microwave region. Each dielectric mechanism has a characteristic "cutoff frequency." As frequency increases, the slow mechanisms drop out in turn, leaving the faster ones to contribute to ε'. The loss factor (ε_r'') will correspondingly peak at each critical frequency. The magnitude and "cutoff frequency" of each mechanism is unique for different materials.
Water has a strong dipolar effect at low frequencies – but its dielectric constant rolls off dramatically around 22 GHz. Teflon, on the other hand, has no dipolar mechanisms and its permittivity is remarkably constant well into the millimeter-wave region.
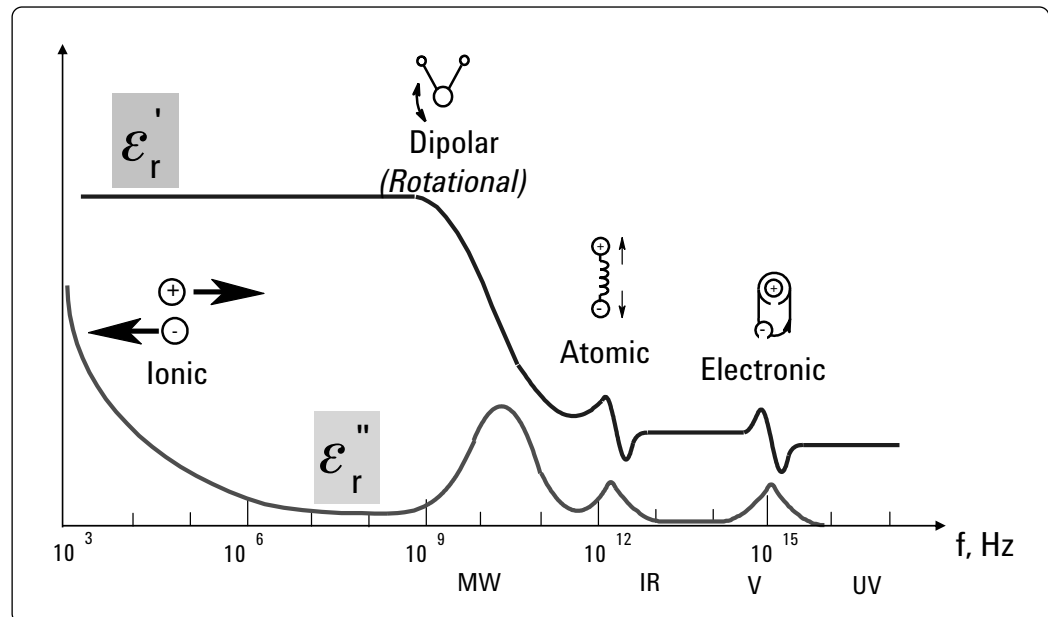

Figure 7. Frequency response of dielectric mechanisms

A resonant effect is usually associated with electronic or atomic polarization. A relaxation effect is usually associated with orientation polarization.

Orientation (dipolar) polarization

A molecule is formed when atoms combine to share one or more of theirs electrons. This rearrangement of electrons may cause an imbalance in charge distribution creating a permanent dipole moment. These moments are oriented in a random manner in the absence of an electric field so that no polarization exists. The electric field E will exercise torque T on the electric dipole, and the dipole will rotate to align with the electric field causing orientation polarization to occur (Figure 8). If the field changes the direction, the torque will also change.
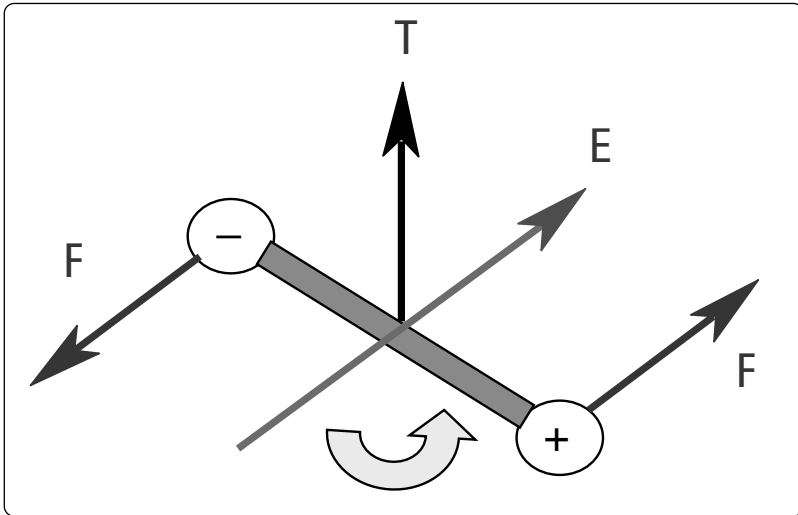

Figure 8. Dipole rotation in electric field

The friction accompanying the orientation of the dipole will contribute to the dielectric losses. The dipole rotation causes a variation in both ε_r' and ε_r'' at the relaxation frequency which usually occurs in the microwave region. As mentioned, water is an example of a substance that exhibits a strong orientation polarization.

Electronic and atomic polarization

Electronic polarization occurs in neutral atoms when an electric field displaces the nucleus with respect to the electrons that surround it. Atomic polarization occurs when adjacent positive and negative ions "stretch" under an applied electric field. For many dry solids, these are the dominant polarization mechanisms at microwave frequencies, although the actual resonance occurs at a much higher frequency. In the infrared and visible light regions the inertia of the orbiting electrons must be taken into account. Atoms can be modeled as oscillators with a damping effect similar to a mechanical spring and mass system (Figure 7). The amplitude of the oscillations will be small for any frequency other than the resonant frequency. Far below resonance, the electronic and atomic mechanisms contribute only a small constant amount to ε_r' and are almost lossless. The resonant frequency is identified by a resonant response in ε_r' and a peak of maximum absorption in ε_r''. Above the resonance, the contribution from these mechanisms disappears.

Relaxation time

Relaxation time τ is a measure of the mobility of the molecules (dipoles) that exist in a material. It is the time required for a displaced system aligned in an electric field to return to 1/e of its random equilibrium value (or the time required for dipoles to become oriented in an electric field). Liquid and solid materials have molecules that are in a condensed state with limited freedom to move when an electric field is applied. Constant collisions cause internal friction so that the molecules turn slowly and exponentially approach the final state of orientation polarization with relaxation time constant τ. When the field is switched off, the sequence is reversed and random distribution is restored with the same time constant.

The relaxation frequency f_c is inversely related to relaxation time:

$$\tau = \frac{1}{\omega_c} = \frac{1}{2\pi f_c}$$

At frequencies below relaxation the alternating electric field is slow enough that the dipoles are able to keep pace with the field variations. Because the polarization is able to develop fully, the loss (ε_r'') is directly proportional to the frequency (Figure 9). As the frequency increases, ε_r'' continues to increase but the storage (ε_r') begins to decrease due to the phase lag between the dipole alignment and the electric field. Above the relaxation frequency both ε_r'' and ε_r' drop off as the electric field is too fast to influence the dipole rotation and the orientation polarization disappears.

Debye relation

Materials that exhibit a single relaxation time constant can be modeled by the Debye relation, which appears as a characteristic response in permittivity as a function of frequency (Figure 9). ε_r' is constant above and below the relaxation with the transition occurring near the relaxation frequency (22 GHz). Additionally, ε_r'' is small above and below relaxation and peaks in the transition region at the relaxation frequency.
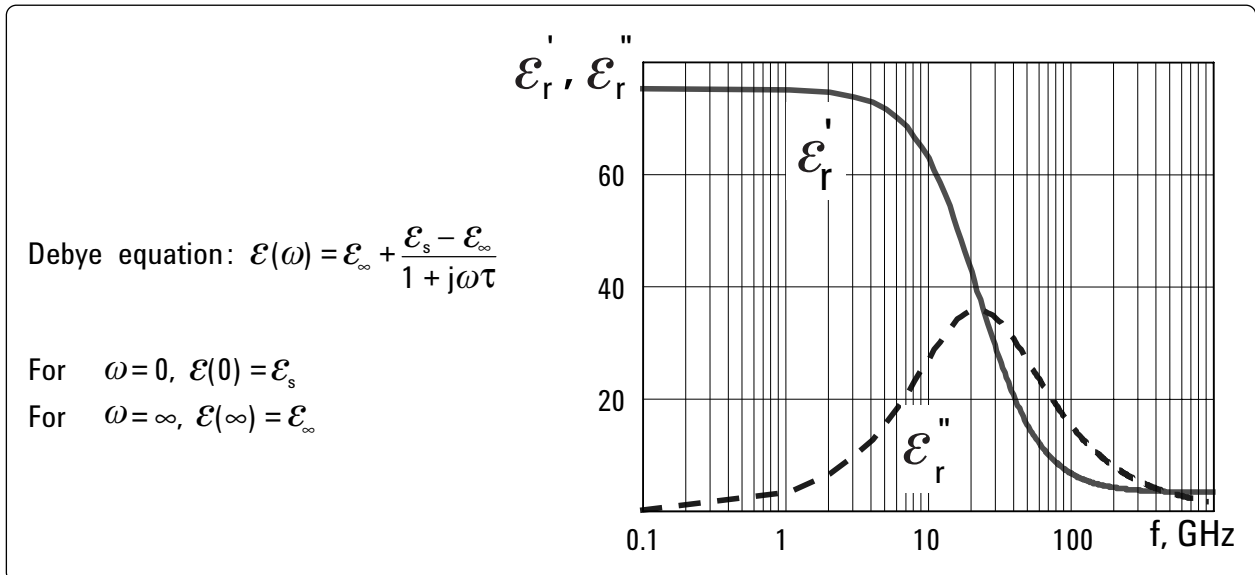

Debye equation: $\varepsilon(\omega) = \varepsilon_\infty + \dfrac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau}$

For $\omega = 0,\ \varepsilon(0) = \varepsilon_s$

For $\omega = \infty,\ \varepsilon(\infty) = \varepsilon_\infty$

Figure 9. Debye relaxation of water at 30° C

In calculating the above curves the static (DC) value of the dielectric constant is ε_s= 76.47, the optical (infinite frequency) value of the dielectric constant is ε_∞= 4.9 and the relaxation time τ = 7.2 ps.

Cole-Cole diagram

The complex permittivity may also be shown on a Cole-Cole diagram by plotting the imaginary part (ε_r'') on the vertical axis and the real part (ε_r') on the horizontal axis with frequency as the independent parameter (Figure 10). A Cole-Cole diagram is, to some extent, similar to the Smith chart. A material that has a single relaxation frequency as exhibited by the Debye relation will appear as a semicircle with its center lying on the horizontal $\varepsilon_r'' = 0$ axis and the peak of the loss factor occurring at $1/\tau$. A material with multiple relaxation frequencies will be a semicircle (symmetric distribution) or an arc (nonsymmetrical distribution) with its center lying below the horizontal $\varepsilon_r'' = 0$ axis.

The curve in Figure 10 is a half circle with its center on the x-axis and its radius $\frac{\varepsilon_s - \varepsilon_\infty}{2}$. The maximum imaginary part of the dielectric constant ε_{rmax}' will be equal to the radius. The frequency moves counter clockwise on the curve.
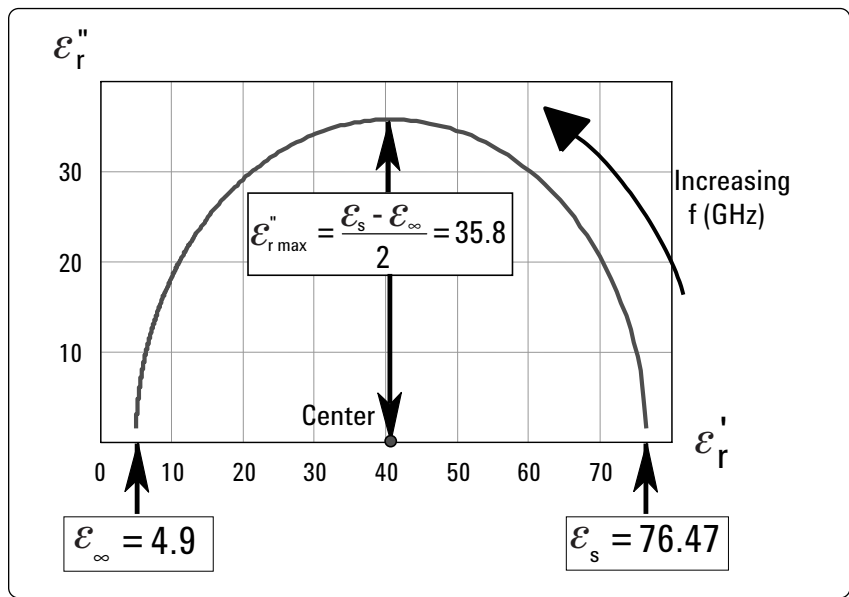

Figure 10. Cole-Cole diagram of Figure 9

Ionic conductivity

The measured loss of material can actually be expressed as a function of both dielectric loss (ε_{rd}'') and conductivity (σ).

$$\varepsilon_r'' = \varepsilon_{rd}'' + \frac{\sigma}{\omega \varepsilon_0}$$

At low frequencies, the overall conductivity can be made up of many different conduction mechanisms, but ionic conductivity is the most prevalent in moist materials. ε_r'' is dominated by the influence of electrolytic conduction caused by free ions which exist in the presence of a solvent (usually water). Ionic conductivity only introduces losses into a material. At low frequencies the effect of ionic conductivity is inversely proportional to frequency and appears as a 1/f slope of the ε_r'' curve.

Interfacial or space charge polarization

Electronic, atomic, and orientation polarization occur when charges are locally bound in atoms, molecules, or structures of solids or liquids. Charge carriers also exist that can migrate over a distance through the material when a low frequency electric field is applied. Interfacial or space charge polarization occurs when the motion of these migrating charges is impeded. The charges can become trapped within the interfaces of a material. Motion may also be impeded when charges cannot be freely discharged or replaced at the electrodes. The field distortion caused by the accumulation of these charges increases the overall capacitance of a material which appears as an increase in ε_r'.

Mixtures of materials with electrically conducting regions that are not in contact with each other (separated by non-conducting regions) exhibit the Maxwell-Wagner effect at low frequencies. If the charge layers are thin and much smaller than the particle dimensions, the charge responds independently of the charge on nearby particles. At low frequencies the charges have time to accumulate at the borders of the conducting regions causing ε_r' to increase. At higher frequencies the charges do not have time to accumulate and polarization does not occur since the charge displacement is small compared to the dimensions of the conducting region. As the frequency increases, ε_r' decreases and the losses exhibit the same 1/f slope as normal ionic conductivity.

Many other dielectric mechanisms can occur in this low frequency region causing a significant variation in permittivity. For example, colloidal suspension occurs if the charge layer is on the same order of thickness or larger than the particle dimensions. The Maxwell-Wagner effect is no longer applicable since the response is now affected by the charge distribution of adjacent particles.

Measuring Systems

Network analyzers

A measurement of the reflection from and/or transmission through a material along with knowledge of its physical dimensions provides the information to characterize the permittivity and permeability of the material. Vector network analyzers such as the PNA, PNA-L, ENA, and ENA-L make swept high frequency stimulus-response measurements from 300 kHz to 110 GHz or even 325 GHz (Figure 12). A vector network analyzer consists of a signal source, a receiver and a display (Figure 11). The source launches a signal at a single frequency to the material under test. The receiver is tuned to that frequency to detect the reflected and transmitted signals from the material. The measured response produces the magnitude and phase data at that frequency. The source is then stepped to the next frequency and the measurement is repeated to display the reflection and transmission measurement response as a function of frequency. More information on the network analyzer functioning and architecture is available in the Application Notes 1287-1[2] and 1287-2[3].

Simple components and connecting wires that perform well at low frequencies behave differently at high frequencies. At microwave frequencies wavelengths become small compared to the physical dimensions of the devices such that two closely spaced points can have a significant phase difference. Low frequency lumped-circuit element techniques must be replaced by transmission line theory to analyze the behavior of devices at higher frequencies. Additional high frequency effects such as radiation loss, dielectric loss and capacitive coupling make microwave circuits more complex and expensive. It is time consuming and costly to try to design a perfect microwave network analyzer.
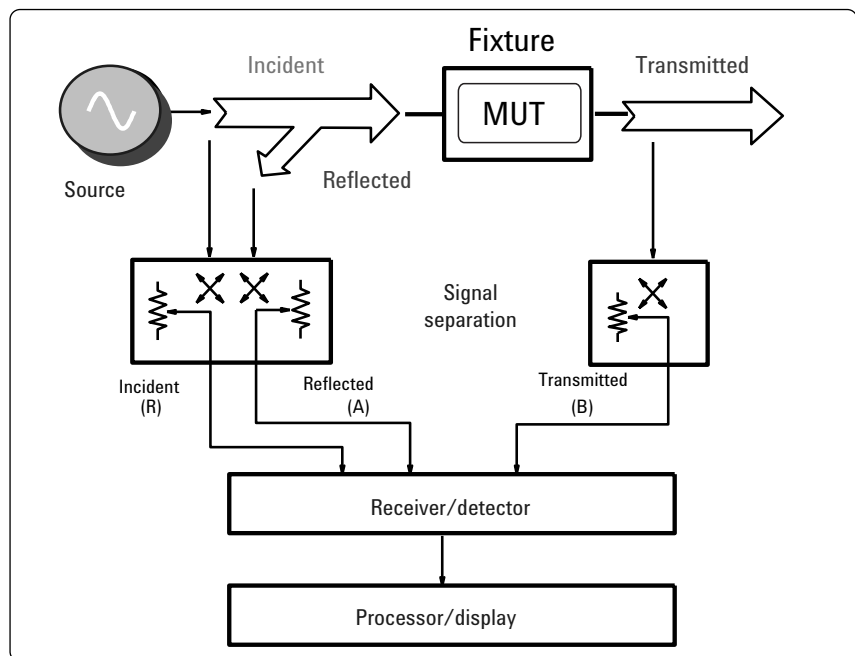

Figure 11. Network analyzer

Instead, a measurement calibration is used to eliminate the systematic (stable and repeatable) measurement errors caused by the imperfections of the system. Random errors due to noise, drift, or the environment (temperature, humidity, pressure) cannot be removed with a measurement calibration. This makes a microwave measurement susceptible to errors from small changes in the measurement system. These errors can be minimized by adopting good measurement practices, such as visually inspecting all connectors for dirt or damage and by minimizing any physical movement of the test port cables after a calibration. More information on the network analyzer calibration is available in the Application Note 1287-3[4].

Impedance analyzers and LCR meters

Impedance analyzers and LCR meters such as the ones listed in Figure 12 are used to measure the material properties at lower frequencies. The material is stimulated with an AC source and the actual voltage across the material is monitored. Material test parameters are derived by knowing the dimensions of the material and by measuring its capacitance and dissipation factor.
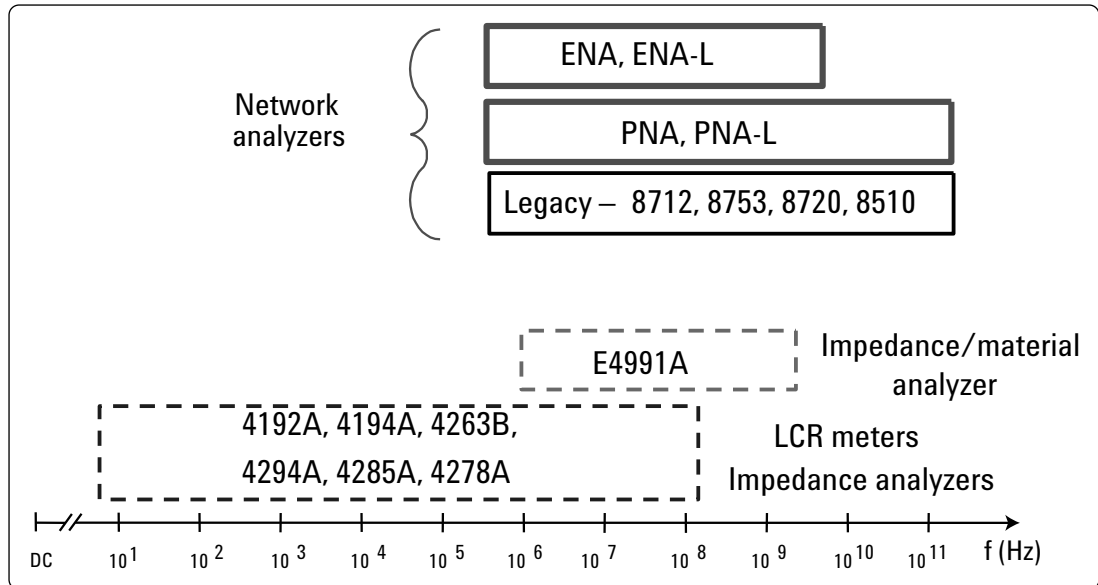

Figure 12. Frequency coverage of Agilent Technologies instruments used for dielectric measurements

Fixtures

Before the dielectric properties of a material can be measured with network analyzer, impedance analyzer, or LCR meter, a measurement fixture (or sample holder) is required to apply the electromagnetic fields in a predictable way and to allow connection to the measurement instrument. The type of fixture required will depend on the chosen measurement technique and the physical properties of the material (solid, liquid, powder, gas).

Software

The measured data from the instrument is not always presented in the most convenient terminology or format. In this case, software is required to convert the measured data to permittivity or permeability. Software may also be required to model any interaction between the fixture and MUT to allow the extraction of the bulk material properties.

Measurement Techniques

Coaxial probe

Method features
- Broadband
- Simple and convenient (non-destructive)
- Limited ε_r accuracy and tan δ low loss resolution
- Best for liquids or semi-solids

Material assumptions
- "Semi-infinite" thickness
- Non-magnetic
- Isotropic and homogeneous
- Flat surface
- No air gaps

The open-ended coaxial probe is a cut off section of transmission line. The material is measured by immersing the probe into a liquid or touching it to the flat face of a solid (or powder) material. The fields at the probe end "fringe" into the material and change as they come into contact with the MUT (Figure 13). The reflected signal[5] (S_{11}) can be measured and related to $\varepsilon_r{}^*$.

A typical measurement system using a coaxial probe method consists of a network or impedance analyzer, a coaxial probe and software. Both the software and the probe are included in the 85070E dielectric probe kit. An external computer is needed in many cases to control the network analyzer through GP-IB. The 82357A USB to GPIB interface provides a convenient and flexible way to realize this connection. For the PNA family of network analyzers the software can be installed directly in the analyzer and there is no need for an external computer.
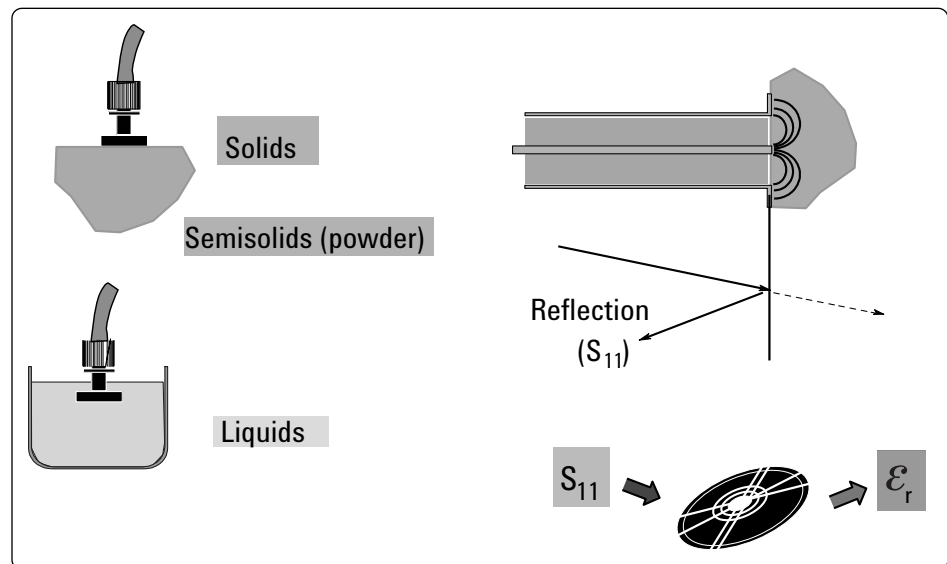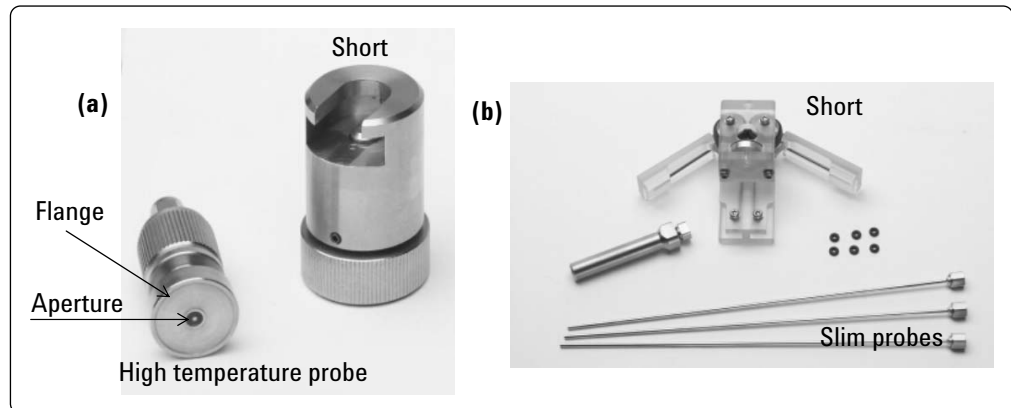

Figure 13. Coaxial probe method

Figure 14 shows the two probes that are available in the 85070E kit; the high temperature probe (a) and the slim form probe (b). The high temperature probe (a) is shown with the shorting block to the right. Three slim probes are shown at the bottom of (b) with the short on the top and a couple of other accessories.

Figure 14. Two dielectric probe configurations

Rugged in design, the high temperature probe features a hermetic glass-to-metal seal, which makes it resistant to corrosive or abrasive chemicals. The probe withstands a wide –40 to +200° C temperature range, which allows measurements versus frequency and temperature. The large flange allows measurements of flat surfaced solid materials, in addition to liquids and semi-solids. The slim form probe features a slim design, which allows it to fit easily in fermentation tanks, chemical reaction chambers, or other equip-ment with small apertures. The slim design also allows it to be used with smaller sample sizes. This probe is best used for liquids and soft semi-solids. For castable solids, the probe is economical enough to be cast into the material and left in place. Because of the consumable nature of this design, these probes are offered in sets of three. The Slim Form probe kit comes with a sealed Slim Form holder that adapts a 2.2 mm outer diameter to 10 mm inner diameter bracket included in the kit as well as commercially available "Midi" sized adapters and bushings. Additional detailed information is available in the Dielectric Probe Technical Overview[6] and Software Online Help[7].

The dielectric probes are compatible with the Agilent network analyzers and the E4991A impedance analyzer. With the impedance analyzer the high temperature probe is specified from 10 MHz.

Before measuring, calibration at the tip of the probe must be performed. A three-term calibration corrects for the directivity, tracking, and source match errors that can be present in a reflection measurement. In order to solve for these three error terms, three well-known standards are measured. The difference between the predicted and actual values is used to remove the systematic (repeatable) errors from the measurement. The three known standards are air, a short circuit, and distillate and de-ionized water. Even after calibrating the probe, there are additional sources of error that can affect the accuracy of a measurement. There are three main sources of errors:

- Cable stability
- Air Gaps
- Sample thickness

It is important to allow enough time for the cable (that connects the probe to the network analyzer) to stabilize before making a measurement and to be sure that the cable is not flexed between calibration and measurement. The automated Electronic Calibration Refresh feature recalibrates the system automatically, in seconds, just before each measurement is made. This virtually eliminates cable instability and system drift errors.

For solid materials, an air gap between the probe and sample can be a significant source of error unless the sample face is machined to be at least as flat as the probe face. For liquid samples air bubbles on the tip of the probe can act in the same way as an air gap on a solid sample.

The sample must also be thick enough to appear "infinite" to the probe. There is a simple equation[6] to calculate the approximate thickness of the sample for the high temperature probe sample and suggested thickness for the slim probe sample. A simple practical approach is to put a short behind the sample and check to see if it affects the measurement results.

Figure 15 shows a comparison of measurements of dielectric constant and loss factor of methanol at room temperature (25° C) using the high temperature probe, with theoretical calculations using the Cole-Cole model. The following parameters are used in the Cole-Cole calculations:

$\varepsilon_{s} = 33.7$, $\varepsilon_{\infty} = 4.45$, $\tau = 4.95 \times 10^{-11}$, $\alpha = 0.036$.

(a)

(b)
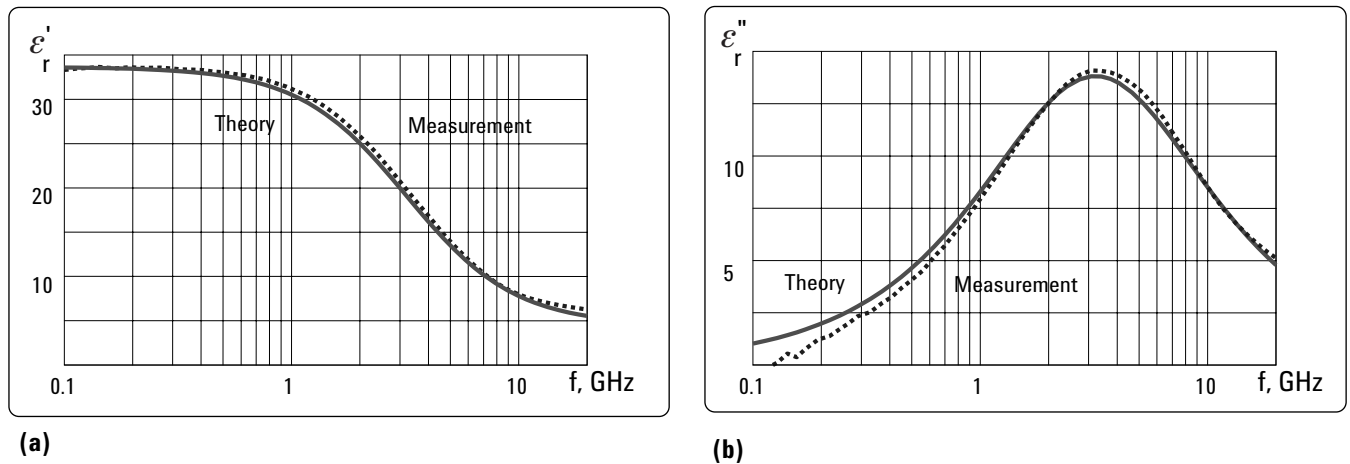
Figure 15. Measured dielectric constant (a) and loss factor (b) of methanol at 25° C compared with Cole-Cole model

A disadvantage of the dielectric probe method is the limited accuracy, under some conditions, when compared to other methods like the transmission line method using the 85071E and resonator method.

Transmission line

Transmission line methods involve placing the material inside a portion of an enclosed transmission line. The line is usually a section of rectangular waveguide or coaxial airline (Figure 16). ε_r^* and μ_r^* are computed from the measurement of the reflected signal (S_{11}) and transmitted signal (S_{21}).

Material assumptions
- Sample fills fixture cross section
- No air gaps at fixture walls
- Smooth, flat faces, perpendicular to long axis
- Homogeneous

Method features
- Broadband – low end limited by practical sample length
- Limited low loss resolution (depends on sample length)
- Measures magnetic materials
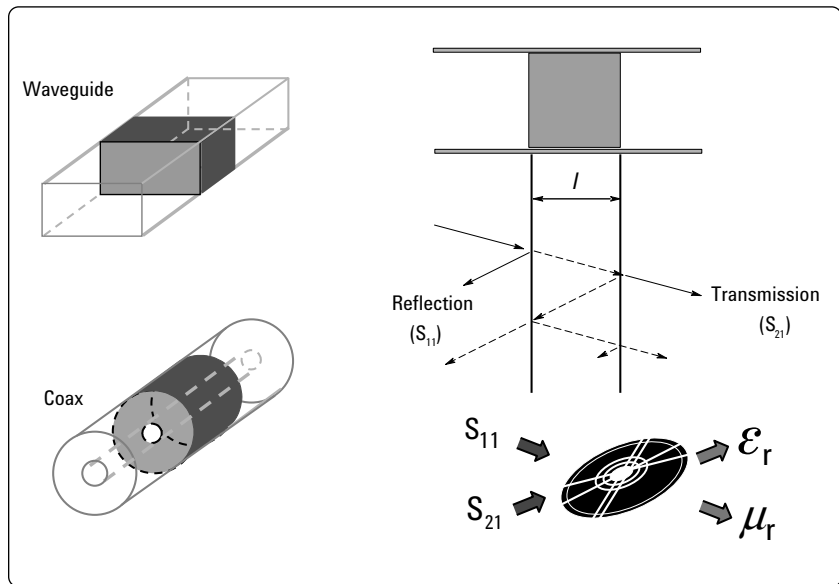- Anisotropic materials can be measured in waveguide

Figure 16. Transmission line method; waveguide and coaxial line case

Coaxial transmission lines cover a broad frequency range, but a toroid-shaped sample is more difficult to manufacture (Figure 17(a)). Waveguide fixtures extend to the mm-wave frequencies and the samples are simpler to machine, but their frequency coverage is banded (Figure 17(b)). A typical measurement system using a transmission line technique consists of a vector network analyzer, a coaxial airline or waveguide section, software such as the 85071E to perform the conversion to ε_r^* and μ_r^*, and an external computer. For the PNA family of network analyzers the software can be installed inside the network analyzer so there is no need for an external computer. Additional information about the 85071E software can be found in the Technical Overview[8] and Software Online Help[9].


(a)
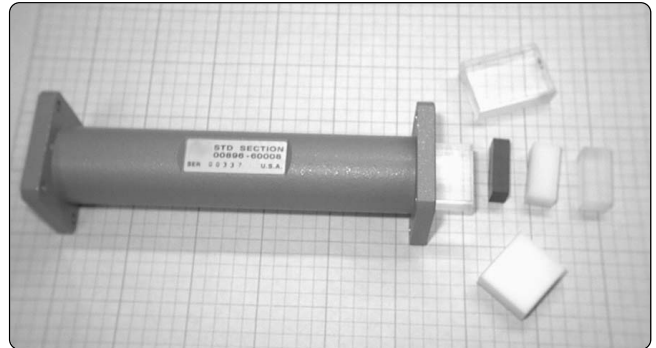
(b)

Figure 17. Coaxial 7 mm air line with samples (a) and X-band waveguide straight section with samples (b)

The 50 Ohm airline from Agilent verification kits (Figure 17(a)) is the recommended coaxial sample holder. Every waveguide calibration kit in the 11644A family contains a precision waveguide section (Figure 17(b)), recommended for a waveguide sample holder.

Figure 18 shows measurement results of permittivity (a) and loss tangent (b) of two Plexiglas samples with lengths of 25 mm and 31 mm respectively, in an X-band waveguide. The sample holder is the precise waveguide section of 140 mm length that is provided with the X11644A calibration kit (Figure 17(b)). The network analyzer is a PNA, the calibration type is TRL and the precision NIST algorithm[9] is used for calculation. In both graphs below there are two pairs of traces for two different measurements of the same samples. The top two measurements of each graph are performed for the case when the sample holder is not calibrated out.

(a)

(b)
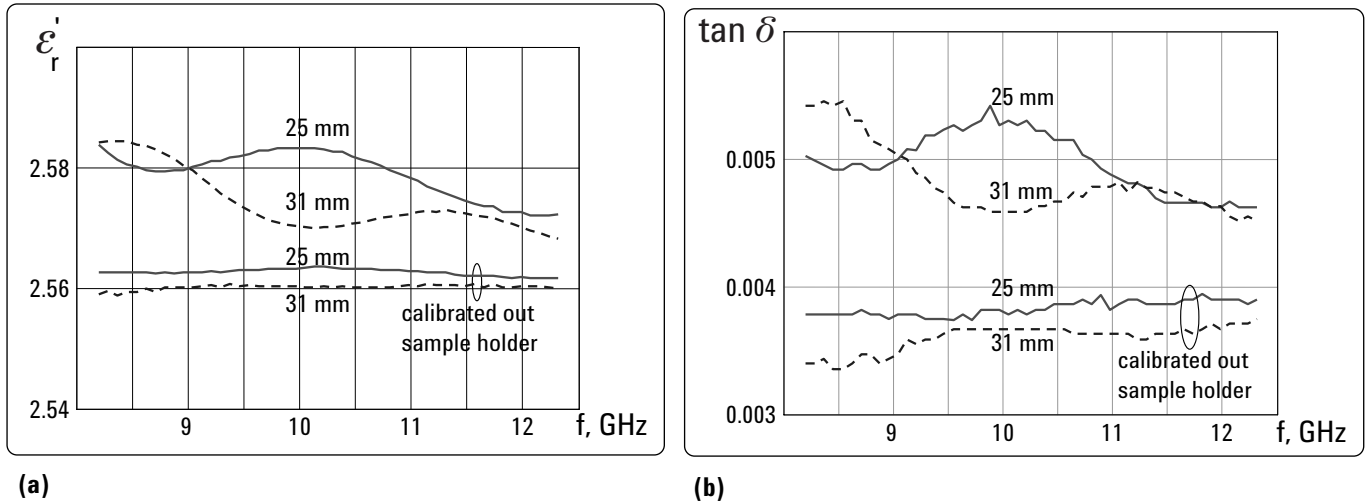
Figure 18. Measurement of two Plexiglas samples, 25 mm and 31 mm long in a X-band waveguide

In this case based on the sample length and sample holder length, the 85071E software will rotate the calibration plane correctly to the sample face, but will not compensate for the losses of the waveguide. The bottom two measurements of the same samples are performed for the case when the sample holder is part of the calibration and the waveguide losses and electrical length are calibrated out. As expected, the loss tangent curves (b) show lower values when the sample holder is calibrated out and they are more constant with respect to frequency. This is due to the fact that the waveguide losses are no longer added to the sample's losses. With the PNA network analyzer, besides calibrating out the sample holder, it is possible to perform fixture de-embedding, which will lead to the same results. This approach requires measuring the empty sample holder after the calibration.
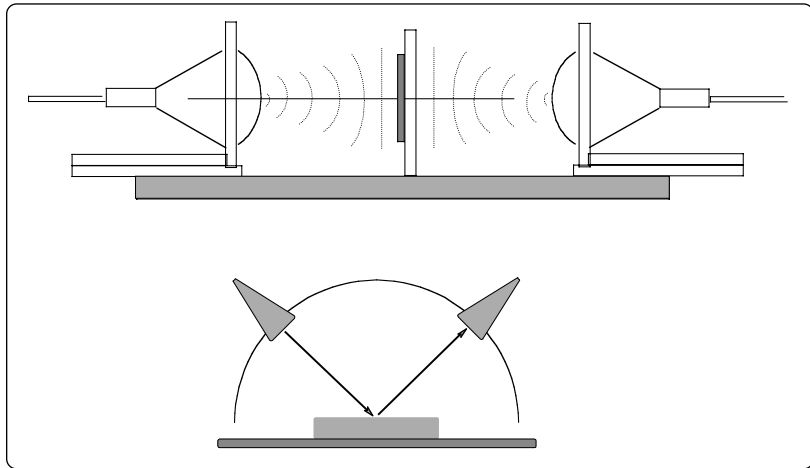
Free space

Material assumptions
- Large, flat, parallel-faced samples
- Homogeneous

Method features
- Non-contacting, non-destructive
- High frequency – low end limited by practical sample size
- Useful for high temperature
- Antenna polarization may be varied for anisotropic materials
- Measures magnetic materials

Free-space methods use antennas to focus microwave energy at or through a slab of material without the need for a test fixture (Figure 19). This method is non-contacting and can be applied to materials to be tested under high temperatures and hostile environments. Figure 19 shows two typical free-space measurement setups: an S-parameter configuration (upper) and the NRL arch (lower). An exemplary measurement system using a free-space method consists of a vector network analyzer, a "fixture" (antennas, tunnels, arches, etc.), software like the 85071, and a computer (not necessary if the PNA family is used). Calibrating the network analyzer for a free space measurement is challenging. Free space calibration standards present special problems since they are "connector-less". A calibration can be as simple as a response calibration or as complex as a full two-port calibration depending on the convenience and accuracy desired. A TRL (Thru-Reflect-Line) or TRM (Thru-Reflect-Match) calibration may actually be easier than other calibration techniques in free space.

Figure 19. Free space measurement setups

Time domain gating is often used to take the place of or supplement an existing calibration. The 85071E software offers an optional free space calibration routine called GRL (Gated Reflect Line). This calibration routine increases the ease of use and reduces the costs associated with TRM and TRL calibration methods. The Gated Reflect Line calibration technique converts a coaxial or waveguide 2-port calibration into a full 2-port free space calibration. Use of this option requires a PNA Series network analyzer or an 8510 with the time domain option, an appropriate free space fixture, and a metal calibration plate. This option also includes a gated isolation/ response calibration, which reduces errors from diffraction effects at the sample edges and multiple residual reflections between the antennas.

Accurate free space measurements are now possible without expensive spot focusing antennas, micro positioning fixtures, or direct receiver access. The 85070E software automatically sets up all the free space calibration definitions and network analyzer parameters, saving engineering time. With the PNA, additional ease and time savings is provided with ECal, electronic calibration. A guided calibration wizard steps the user through the easy calibration process. Figure 20 depicts the result of a GRL calibration measuring Rexolite material in U-band (40-60 GHz) with a PNA network analyzer and 85071E software. The fixture is made from a readily available, domestic use, shelving unit to demonstrate that when doing a GRL calibration, even with the simplest set up, it is still possible to perform precise measurements.
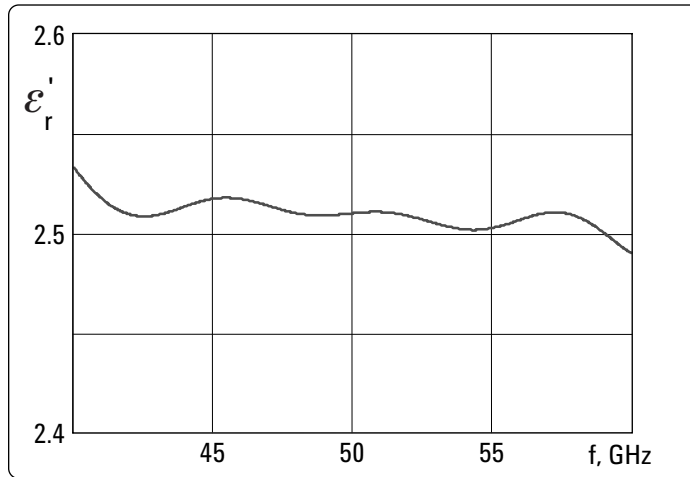
Figure 20. Measurement of Rexolite sample in a U-band (40 – 60 GHz)

High temperature measurements are easy to perform in free space since the sample is never touched or contacted (Figure 21). The sample can be heated by placing it within a furnace that has "windows" of insulation material that are transparent to microwaves. Agilent Technologies does not provide the furnace needed for such a type of measurement. Figure 21 illustrates the basic set up.

Figure 21. High temperature measurement in free space

Resonant cavity

Resonant versus broadband techniques

Resonant techniques
- High impedance environment
- Reasonable measurements possible with small samples
- Measurements at only one or a few frequencies
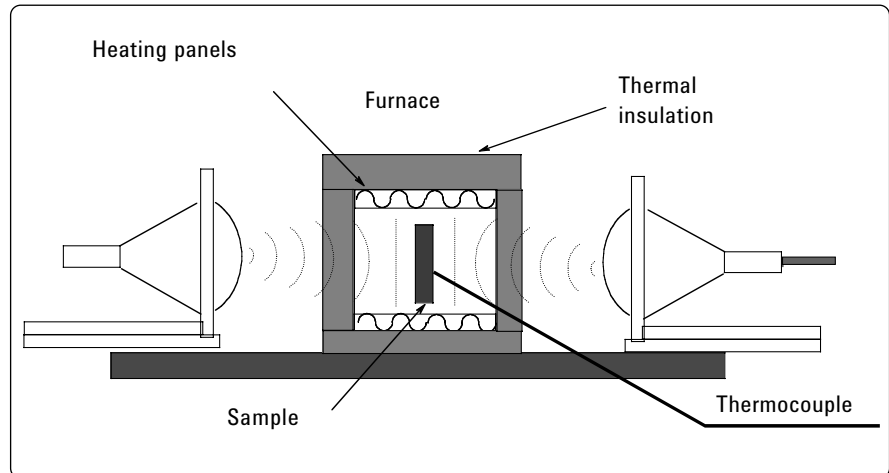- Well suited for low loss materials

Broadband techniques
- Low impedance environment
- Requires larger samples to obtain reasonable measurements
- Measurement at "any" frequency

Resonant cavities are high Q structures that resonate at certain frequencies. A piece of sample material affects the center frequency (f) and quality factor (Q) of the cavity. From these parameters, the complex permittivity (ε_r) or permeability (μ_r) of the material can be calculated at a single frequency. There are many different types of cavities and methods. Here, the most widely used cavity perturbation method, as described in ASTM 2520[10], is considered. This method uses a rectangular waveguide with iris-coupled end plates, operating in TE_{10n} mode (Figure 22). For a dielectric measurement the sample should be placed in a maximum electric field and for a magnetic measurement, in a maximum magnetic field. If the sample is inserted through a hole in the middle of the waveguide length, then an odd number of half wavelengths (n = 2k + 1) will bring the maximum electric field to the sample location, so that the dielectric properties of the sample can be measured. An even number of half wavelengths (n = 2k) will bring the maximum magnetic field to the sample location and the magnetic properties of the sample can be measured.

$$\varepsilon_r' = \frac{V_c\left(f_c - f_s\right)}{2V_s f_s} + 1$$

$$\varepsilon_r'' = \frac{V_c}{4V_s}\left(\frac{1}{Q_s} - \frac{1}{Q_c}\right)$$

V is the volume

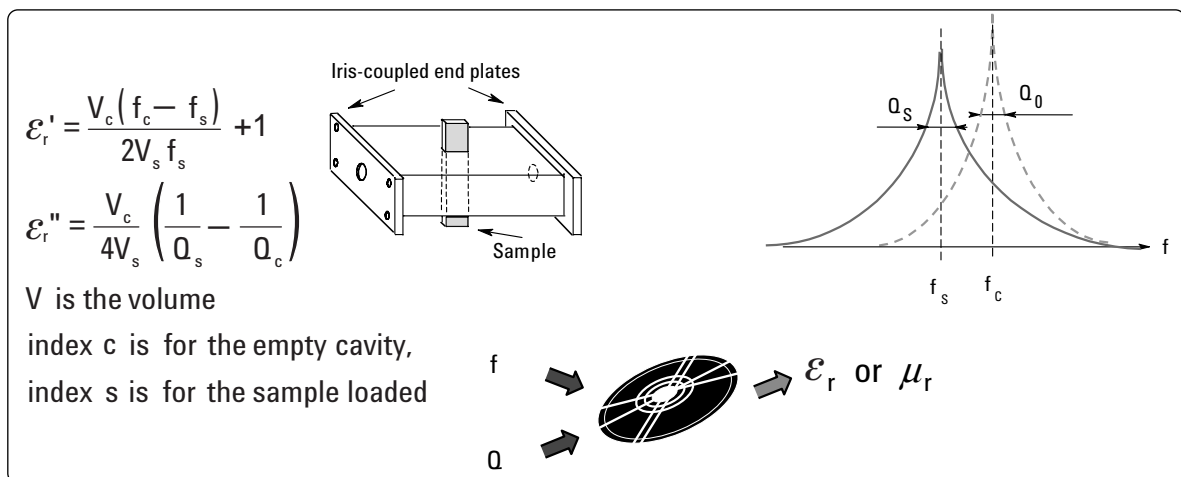index c is for the empty cavity,

index s is for the sample loaded

Figure 22. Resonant cavity measurement

The cavity perturbation method requires a very small sample such that the fields in the cavity are only slightly disturbed to shift the measured resonant frequency and cavity Q. This assumption allows simplifying the theory to use the equations above to calculate the dielectric properties of the material. Although Agilent Technologies does not provide a ready-made resonator (i.e. iris-coupled end plates), this method is part of the 85071E software, which allows for automatic measurement of all necessary parameters and calculation of the complex dielectric constant. It is easy to adapt the 140 mm long precision waveguide section available in the X11644A waveguide calibration kit for resonator measurements (Figure 23). A hole needs to be drilled exactly in the middle of the waveguide length and the two iris-coupled end plates need to be manufactured. The dimension of the iris hole is b/2.2, where b is the narrow dimension of the waveguide cross section[10].
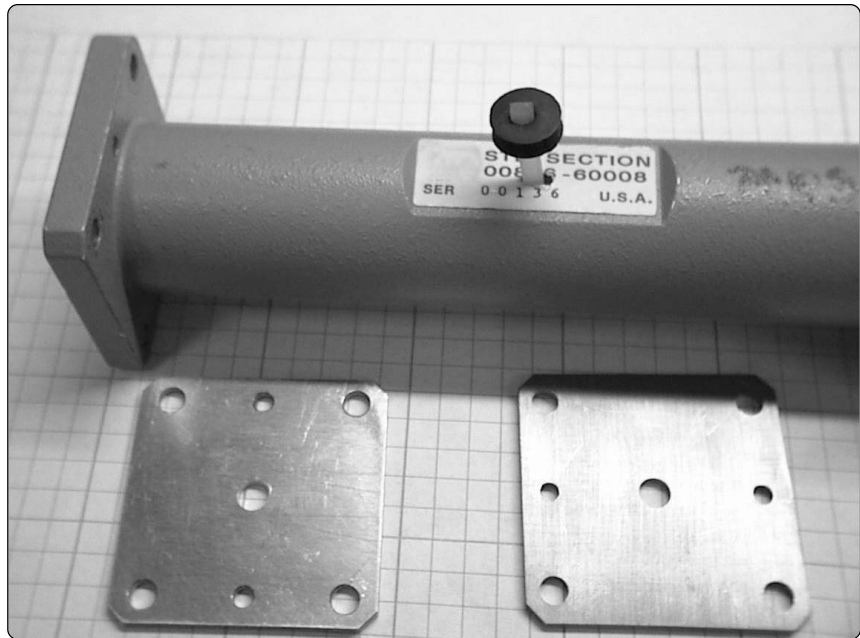
Figure 23. X-band cavity resonator parts and inserted sample

Figure 24 shows measurements of three different samples with this cavity. The three measurements are presented on the same graph for comparison purposes.
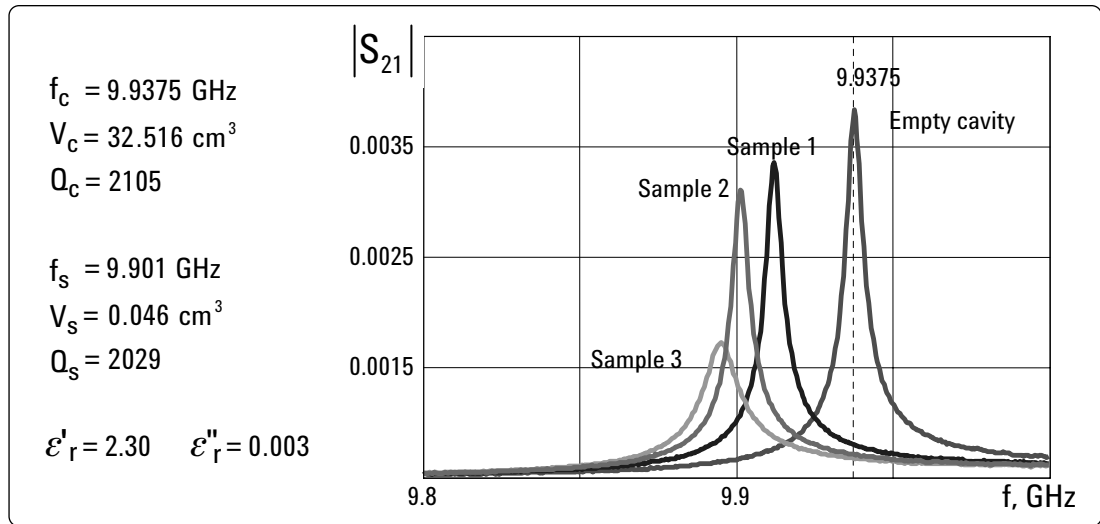

f_c = 9.9375 GHz

V_c = 32.516 cm^3

Q_c = 2105

f_s = 9.901 GHz

V_s = 0.046 cm^3

Q_s = 2029

ε'_r = 2.30 ε''_r = 0.003

Figure 24. Graphs of the empty cavity and three different samples

The resonant frequency of the empty cavity is f_c = 9.9375 GHz (for TE_{107} mode) and it shifts to a lower frequency when the sample is inserted in the cavity. When the resonator is loaded with a sample, the resonance curve broadens, which results in a lower quality factor Q. On the y-axis of Figure 23 is the magnitude of the linear transmission coefficient $|S_{21}|$. The 8720ES network analyzer is used for these measurements. On the left of the figure is a calculation for the Sample 2, which has a cross section of 0.29 by 0.157 cm.

A typical measurement system using the resonant cavity method consists of a network analyzer, a cavity, software, and an external computer (except when using a PNA Series). There is no need to calibrate the network analyzer for this type of measurement.

Although the resonant cavity technique is extremely accurate, it is still subject to errors. The network analyzer must have excellent frequency resolution (1 Hz) to measure very small changes in the Q factor. The sample cross-section dimensions must be known precisely. There is also additional error due to the approximation in the analysis (perturbation theory). This method has limitations for low-loss samples due to the comparatively low Q-factor of the empty rectangular waveguide cavity. A cylindrical type of cavity offers much higher Q-factors, but it has its own disadvantages, the biggest of which is the difficulty to manufacture it.

Parallel plate

The parallel plate capacitor method involves sandwiching a thin sheet of material between two electrodes to form a capacitor. A typical measurement system using the parallel plate method consists of an LCR meter or impedance analyzer and a fixture such as the 16451B dielectric test fixture, which operates up to 30 MHz. More information about other Agilent Technologies low frequency materials measurement solutions is available in Application Note 1369-1[1] and 380-1[11].

Comparison of Methods

Many factors such as accuracy, convenience, and the material shape and form are important in selecting the most appropriate measurement technique. Some of the significant factors to consider are summarized here:

- Frequency range
- Expected values of ε_r and μ_r
- Required measurement accuracy
- Material properties (i.e., homogeneous, isotropic)
- Form of material (i.e., liquid, powder, solid, sheet)
- Sample size restrictions
- Destructive or nondestructive
- Contacting or noncontacting
- Temperature
- Cost

Figure 25 provides a quick comparison between the measurement methods that have been discussed already and, in Figure 26, are summarized the available fixtures and instruments for materials measurements.

Coaxial probe ε_r	Broadband, convenient, non-destructive Best for lossy MUTs; liquids or semi-solids
Transmission line ε_r and μ_r	Broadband Best for lossy to low loss MUTs; machineable solids
Free space ε_r and μ_r	Non-contacting Best for high temperatures; large, flat samples
Resonant cavity ε_r and μ_r	Accurate Best for low loss MUTs; small samples
Parallel plate ε_r	Accurate Best for low frequencies; thin, flat sheets

Figure 25. Summary of the measurement techniques

Transmission line software — 85071E

Dielectric probe — 85070E

16453A — Dielectric material test fixture

16451B — Dielectric test fixture

16452A — Liquid test fixture

16454A — Magnetic test fixture

DC   10Hz   100Hz   1kHz   10kHz   100kHz   1MHz   10MHz   100MHz   1GHz   10GHz   100GHz

4263B, 4284A, 4285A, 4294A — LCR meters/impedance analyzers

E4991A — Impedance/material analyzer

Network analyzers {
PNA, PNA-L
ENA, ENA-L
Legacy – 8712, 8753, 8720, 8510
}

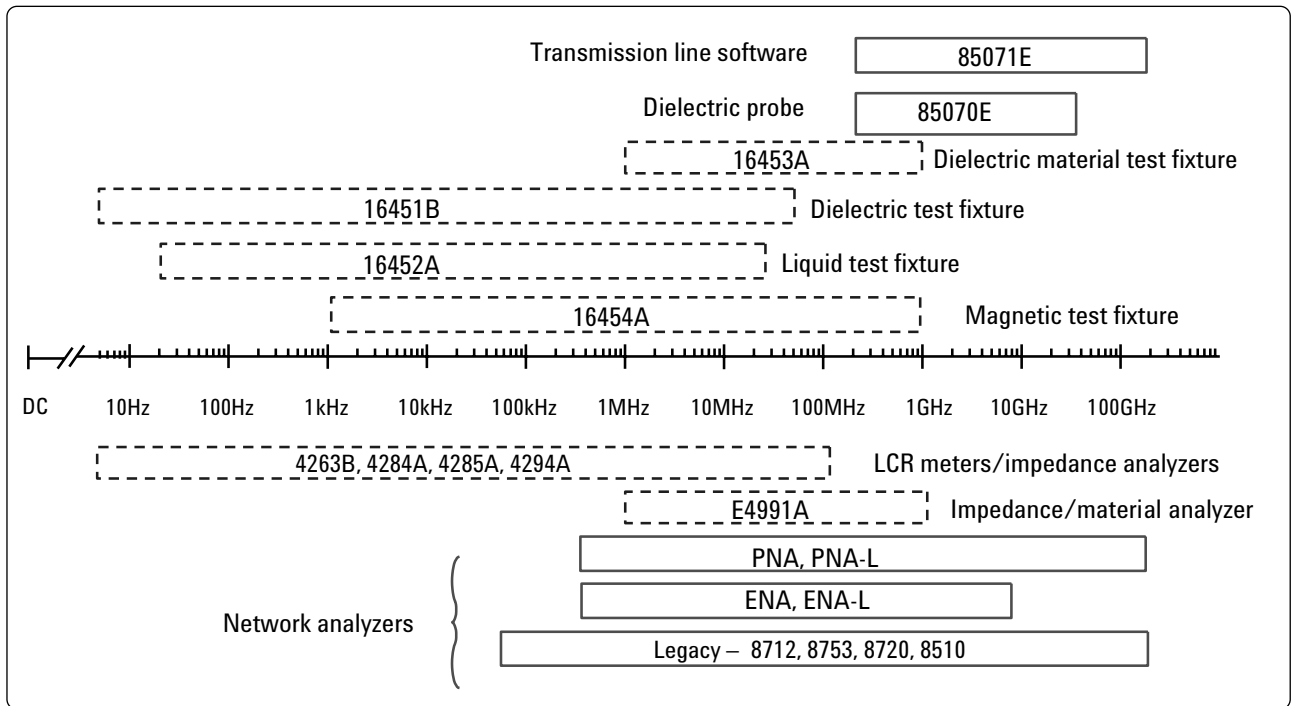**Figure 26. Agilent Technologies instruments and fixtures**

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