Agilent Technologies

Microwave Dielectric Spectroscopy Workshop

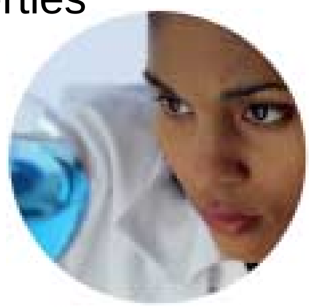
“Measure the Difference”

Agilent Technologies
& Innovative Measurement Solutions

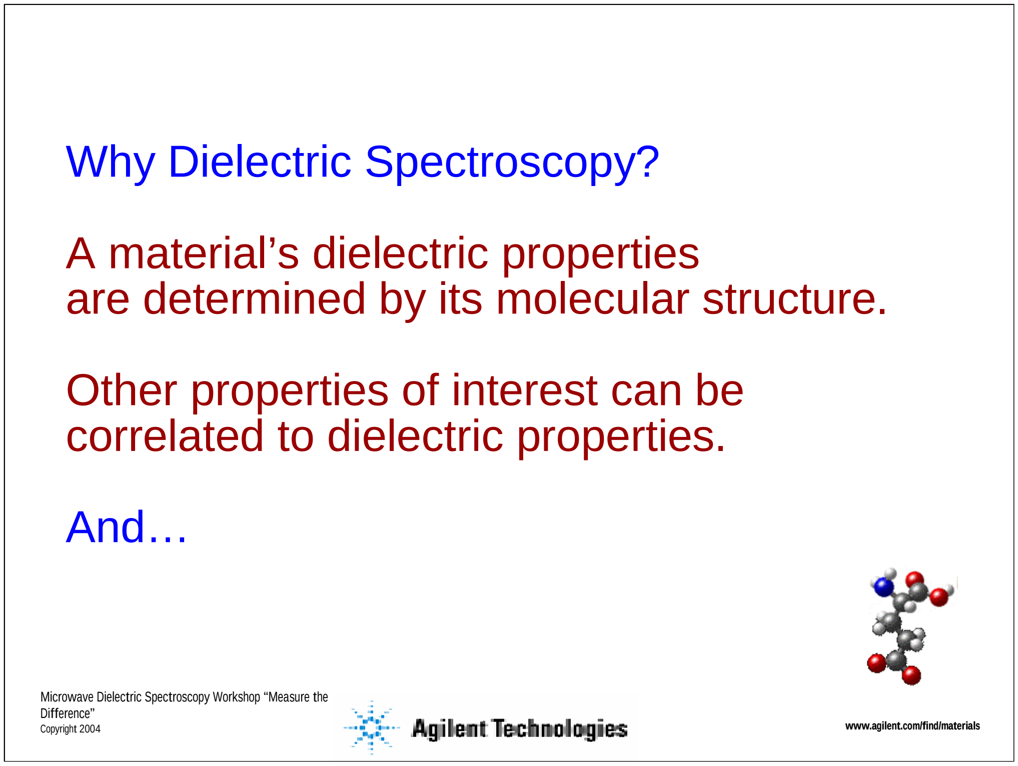
In the mid 1980s, Agilent Technologies developed several products that measure the electromagnetic properties of material samples. At that time Agilent Technologies was a part of the Hewlett Packard Company. These products were developed to meet the needs our customers involved in the development of electronic components, circuits and systems. These products were also used by Department of Defense contractors involved in the design and development of stealth vehicles. Since there introductions these products have found many other applications. The aim of this presentation is to give a survey of these and other potential applications .

“Measure the Difference”

- ➢ Why Dielectric Spectroscopy?
- • Definition of dielectric properties
- • Relationship to other properties
- • Measurement techniques



The presentation consists of four parts. First we’ll answer the question, why dielectric spectroscopy. Then will define dielectric properties and explains why one material’s dielectric properties is different from another material. Then well discuss several measurement techniques and show demonstrations.



So, why dielectric spectroscopy?

An alternative title for this presentation could have been, *What are dielectric properties and why would I want to measure them?.* The answer to this question is obvious to electrical engineers and physicists that are involved in the design and development of electrical/electronic components, circuits and systems. The answer is not as obvious to other scientists involved in other technical endeavors. The general answer is that a materials dielectric properties are determined by its molecular structure. If the molecular structure changes it dielectric properties changes.

Measurements are:

Fast

Setup in minutes, measure in seconds.

Non-Destructive

Many materials can be measured “as is”.
No sample preparation required

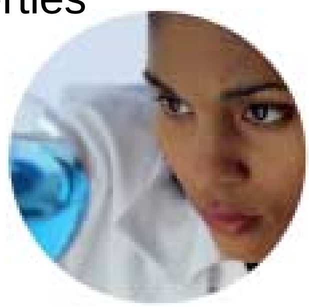
Non-Contacting

With Free-Space techniques, your material is not even touched!

.Measurements are setup in minutes and made in seconds providing real time data. Depending on measurement technique, measurements can be non-destructive, and even non-contacting. Later in this presentation, we will show you examples of these techniques.

"Measure the Difference"

- Why Dielectric Spectroscopy?
- ➢ Definition of dielectric properties
- Relationship to other properties
- Measurement techniques



Permittivity Definition

- One of the factors that determine how the material interacts with an electromagnetic field
- Its dimensions are farad per meter (capacitance per distance)

The dielectric properties, or permittivity, is one of the factors that determines how a material interacts with an applied electromagnetic field.

Its fundamental dimensions are $T^2Q^2M^{-1}L^{-3}$ where T, Q, M and L are time, charge, mass and length respectively. Normally this is expressed as farad per meter (capacitance per distance).

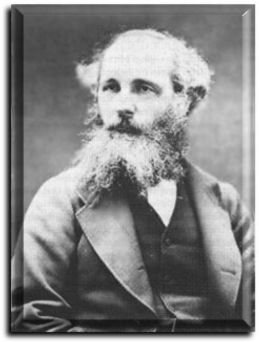
Maxwell's Equations

$$\nabla \times \mathbf{E} = -\frac{\partial \mathbf{B}}{\partial t}$$

$$\nabla \times \mathbf{H} = \frac{\partial \mathbf{D}}{\partial t} + \mathbf{J}$$

$$\nabla \bullet \mathbf{D} = \rho$$

$$\nabla \bullet \mathbf{B} = 0$$



James Clerk Maxwell

The solution of Maxwell's equations determines how microwave energy propagates through a material. Maxwell's equations can be expressed in many equivalent forms. Its most general form is listed here.

Where:

J is the current density

E is the electric field intensity

D is the electric flux density

H is the magnetic intensity field

B is the magnetic flux density

p is the charge density

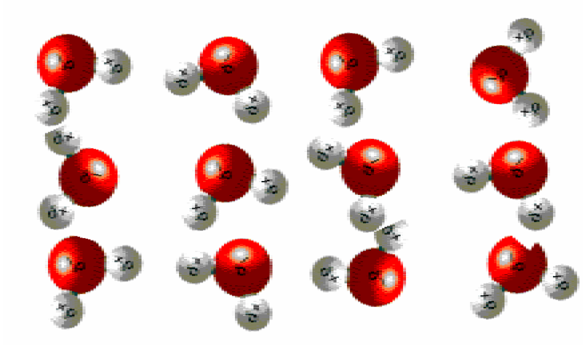
These equations are based on Faraday's law and Ampere's law. These equations are always satisfied.

Constitutive Relations

$$\mathbf{D} = \varepsilon \mathbf{E}$$
$$\mathbf{B} = \mu \mathbf{H}$$
$$\mathbf{J} = \sigma \mathbf{E}$$

Other factors that make the solution unique to a particular situation are the boundary conditions and the satisfaction of the following constitutive relations. **e**, **u**, and **s** are, respectively, the permittivity, permeability and conductivity of the media. These are commonly referred to as the electromagnetic properties of the material. Most materials are non-magnetic. The consequence of this is that **m** is known. At high frequencies, the effect of **s** can be ignored. This is because its effect varies inversely with frequency. At lower frequencies **s** is one of the factors that determine the loss factor.

Molecular Structure of Water



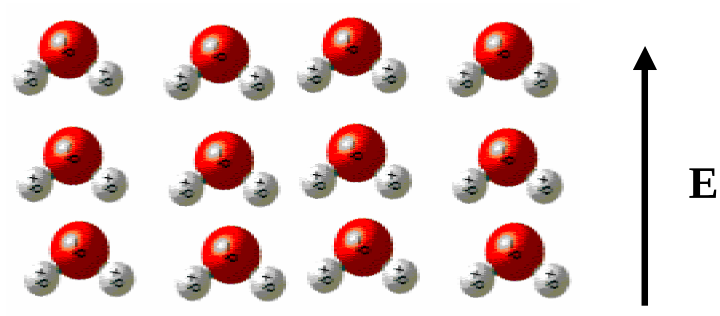
Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies

www.agilent.com/find/materials

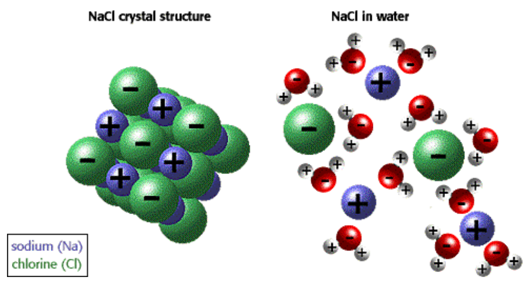
Consider the molecular structure of water. I apologize to all chemists for the cartoon depiction of this simple molecule. It does, however, serve to illustrate the concept of a material's dielectric properties. The water molecule consists of two hydrogen atoms and one oxygen atom. While the molecule is electrically neutral it's charge distribution is not constant over its volume. Because of its bonding mechanism, the hydrogen side of the molecule is more positive that the oxygen side. In the absence of any forces other than thermal energy, a group of water molecules will take on a random orientation as depicted.

Constant Electric Field

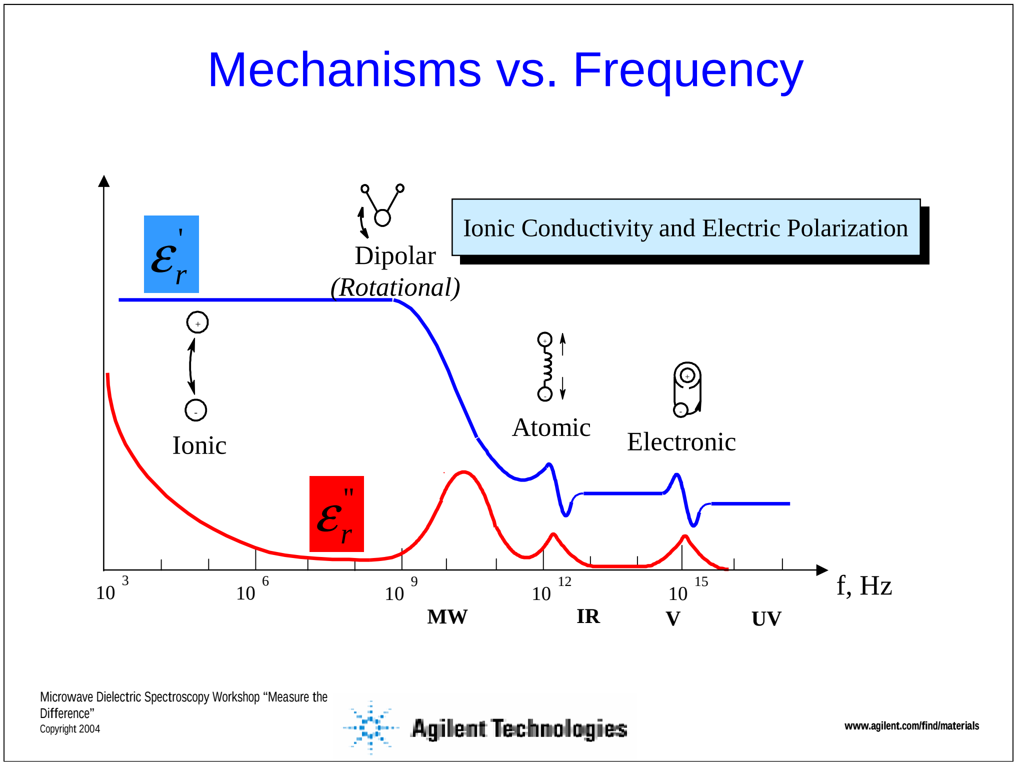


If a constant electric field is applied to this group of water molecules they will tend to orient with the applied field. This orientation ,or polarization, causes the water to have a particular capacitance per meter.

Water and NaCl



Now consider adding salt to the water molecules. The bonds of the salt crystals will break when added to the water creating positive and negative ions. The ions will tend to align themselves with the positive or negative regions of the liquid water. Thermal forces infers with this alignment.



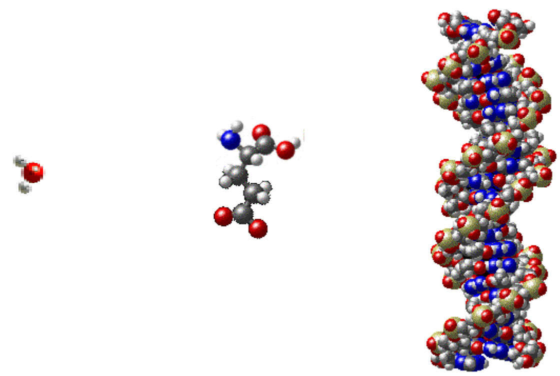
This plot illustrates how the dielectric properties of the salt/water mixture varies with the frequency of the applied electric field. The dielectric properties of a material is represented by a complex quantity known as permittivity. A material's permittivity is usually normalized to the permittivity of a vacuum. The real part of permittivity, e_r', is a measure of the energy stored and is called the dielectric constant. The imaginary part of permittivity, e_r'', is a measure of the energy loss and called the loss factor.

At low frequencies, the water molecules can follow the applied electric field resulting in maximum values of e_r'. This polarization is a form of energy storage. At these same frequencies, the positive and negative ions move in accordance to the electric field. This electric current corresponds to an energy loss.

As the frequency increases the water molecules can no longer keep up with the changing electric field. This results in less energy storage and higher rotational losses. At these frequencies the mass of the ions prevents them from responding the the changing electric field.

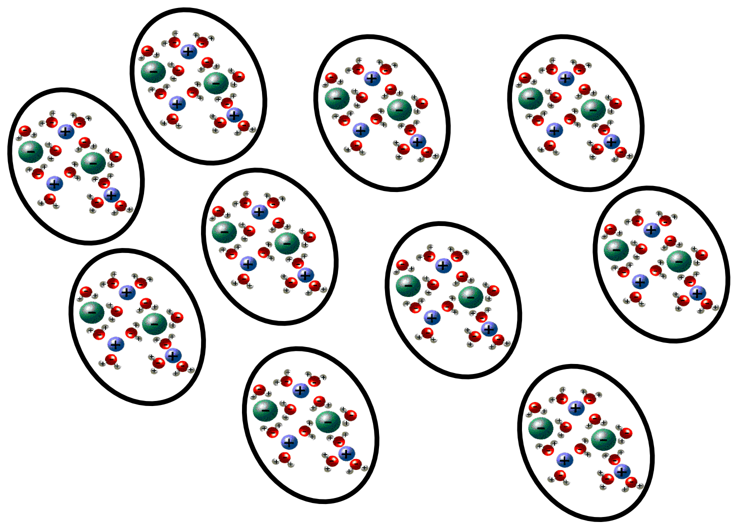
At even higher frequencies the water molecules no longer respond to the electric field . Permittivity usually is measured at frequencies below 10^{11} Hz. At frequencies above this the water molecules are stretched. At even higher frequencies the water molecules are pulled apart.

Small and Large Molecules



Many materials are a mixture of different sized molecules. The permittivity of these mixtures of materials will depend on the interaction of these molecules, their mass, charge and charge distributions.

Emulsions and Encapsulation



Microwave Dielectric Spectroscopy Workshop “Measure the Difference”

Agilent Technologies

www.agilent.com/find/materials

The many structure features and characteristics of colloids can be related to permittivity measurements.

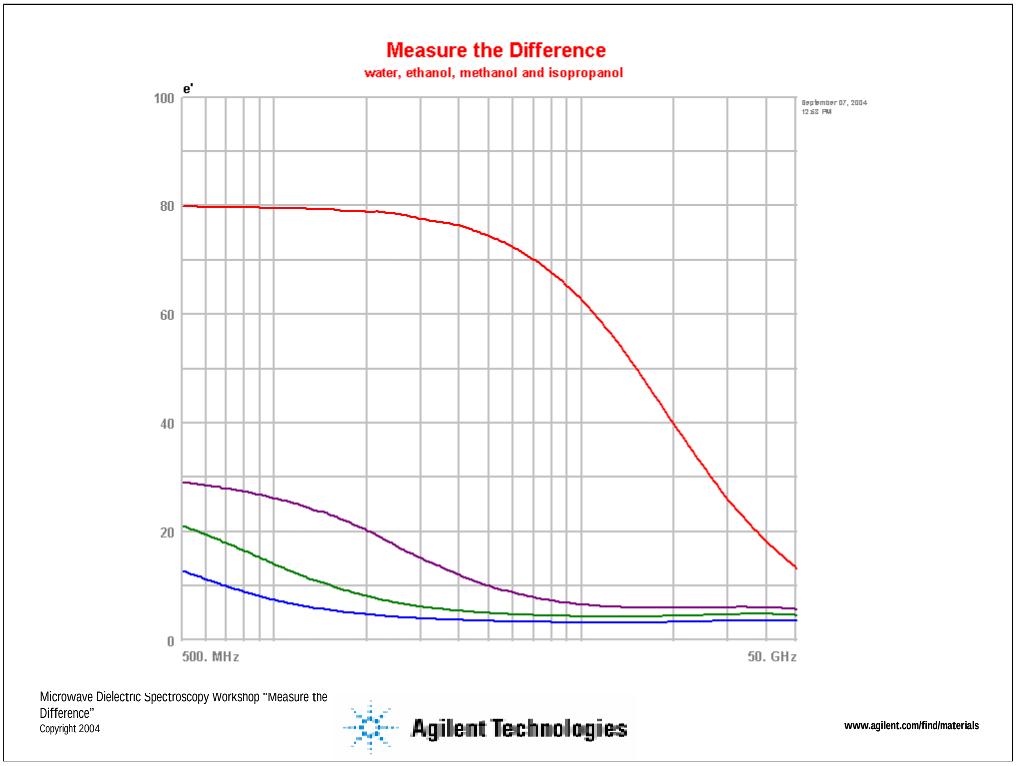
"Measure the Difference"

- Why Dielectric Spectroscopy?
- Definition of dielectric properties
- ➢ Relationship to other properties
- Measurement techniques

Dielectric Properties can be related to:

Moisture content
Biomass
Bulk density
Bacterial content
Cancer detection
Chemical reaction
Mechanical stress
Structure
Consistency
Concentration
Fruit ripeness
Most other physical properties …

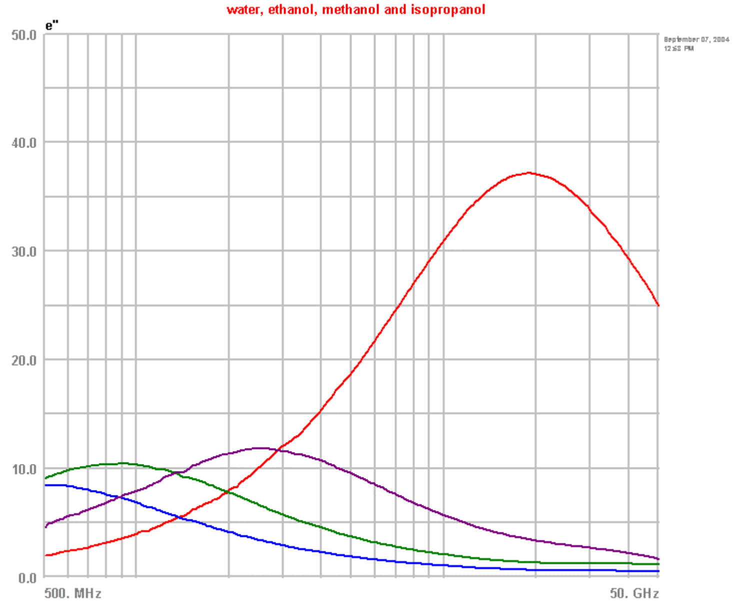
Here is a partial list of material properties that have been related to dielectric properties. The amount of water in a solid or liquid can be determined by measuring the material’s permittivity. The advantage of using this technique over the traditional oven dried methods is that permittivity can be measured in near real time. Cancer cells have a different permittivity than healthy cells. As chemicals react the permittivity of the mixture changes. A list of references to these and other applications is located at the end of this presentation.



Here's a look at water and three different alcohols. The X axis of the graph is frequency and the Y axis is the real part of permittivity, which represents a materials ability to store energy. The red trace is water.

Measure the Difference

water, ethanol, methanol and isopropanol



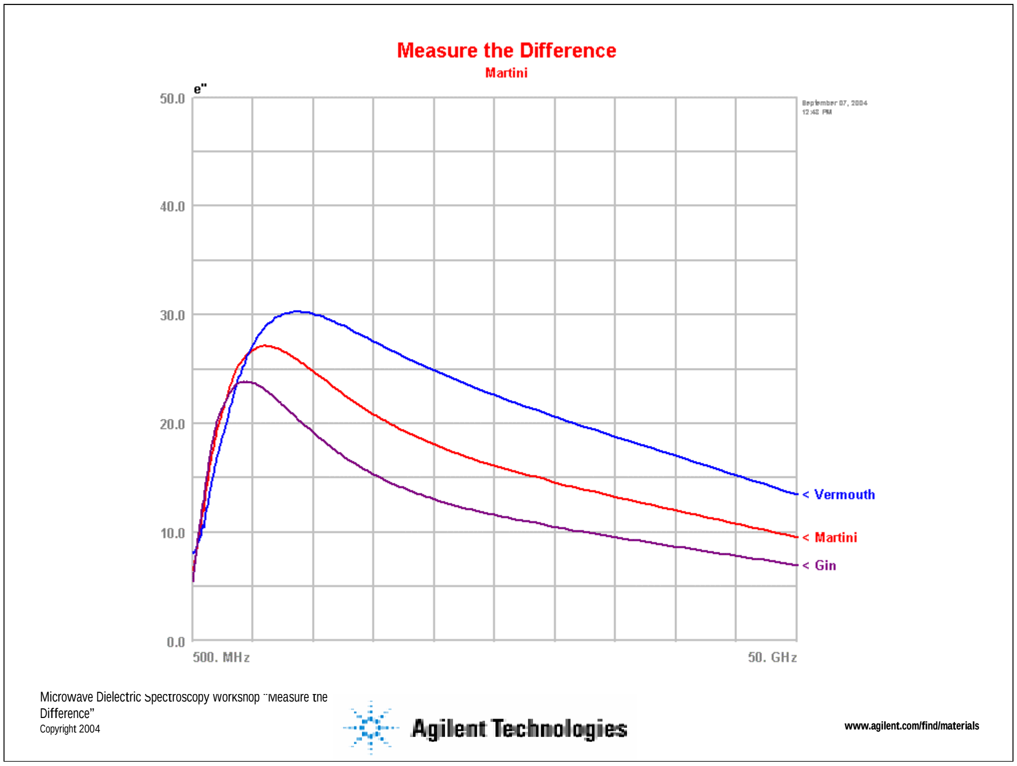
Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies

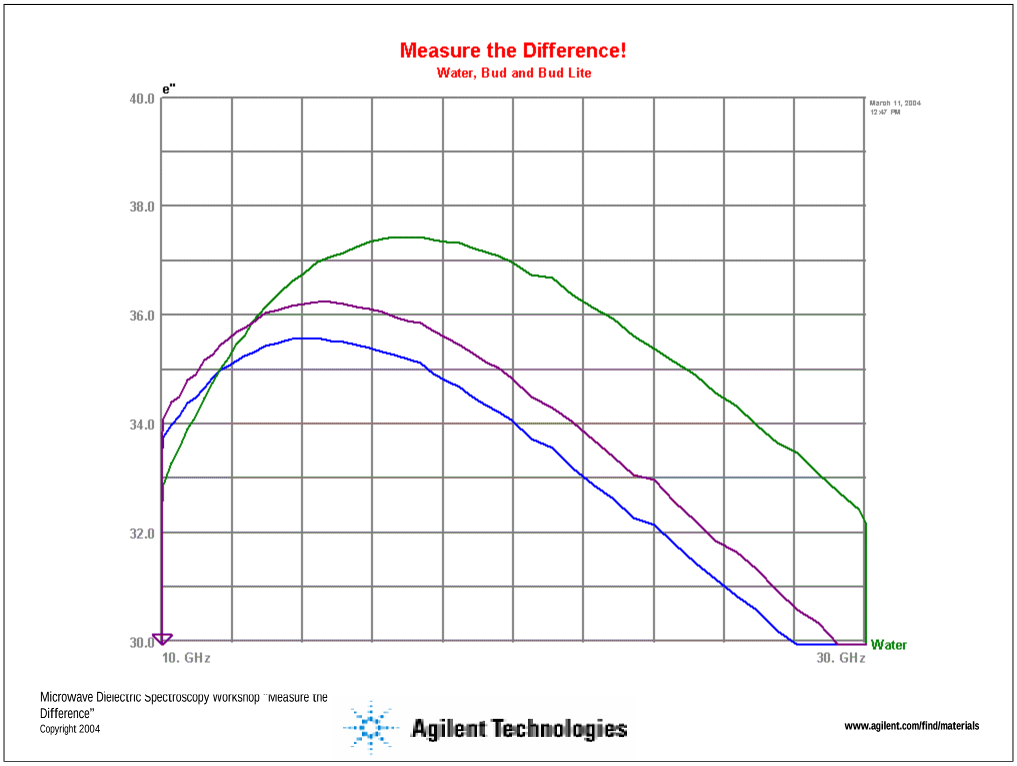
www.agilent.com/find/materials

Here we are looking at the same measurement as the last slide, but the x axis is now the imaginary part of permittivity. So, we are looking at the lossiness of our material. You can see that each alcohol and the water have their peak loss at different frequencies.

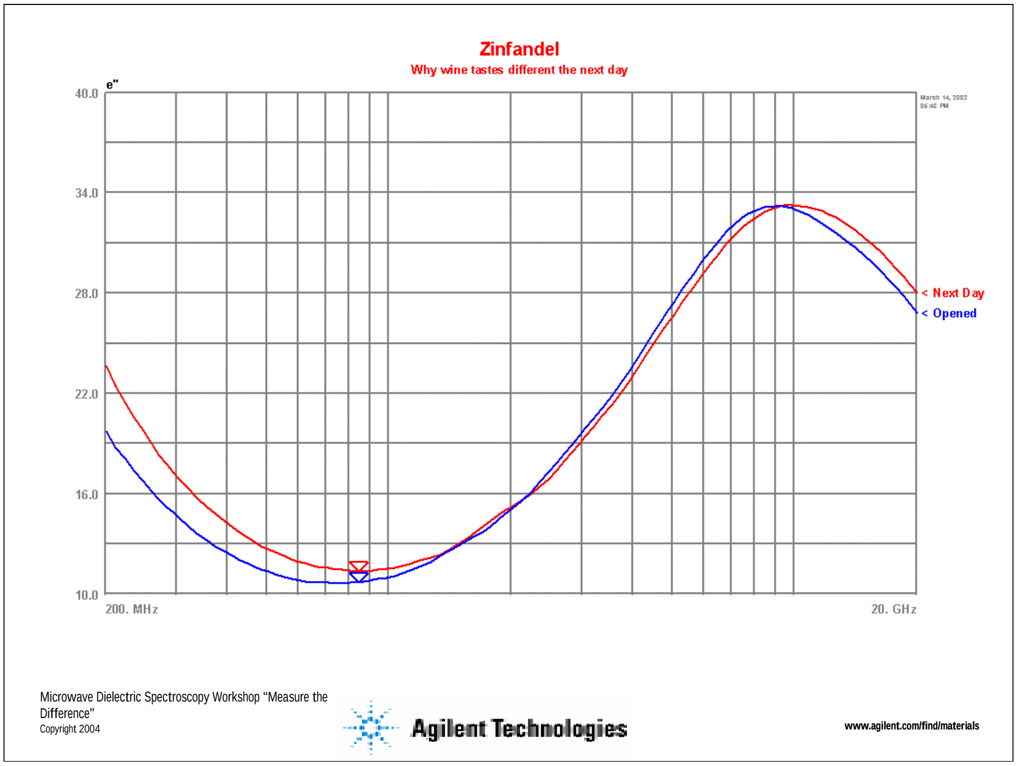
Two other common ways to view permittivity are loss tangent, which is the imaginary part divided by the real part, vs. frequency, and Cole-Cole which is the imaginary part vs the real part (imaginary part on the y-axis, real part on the x-axis).



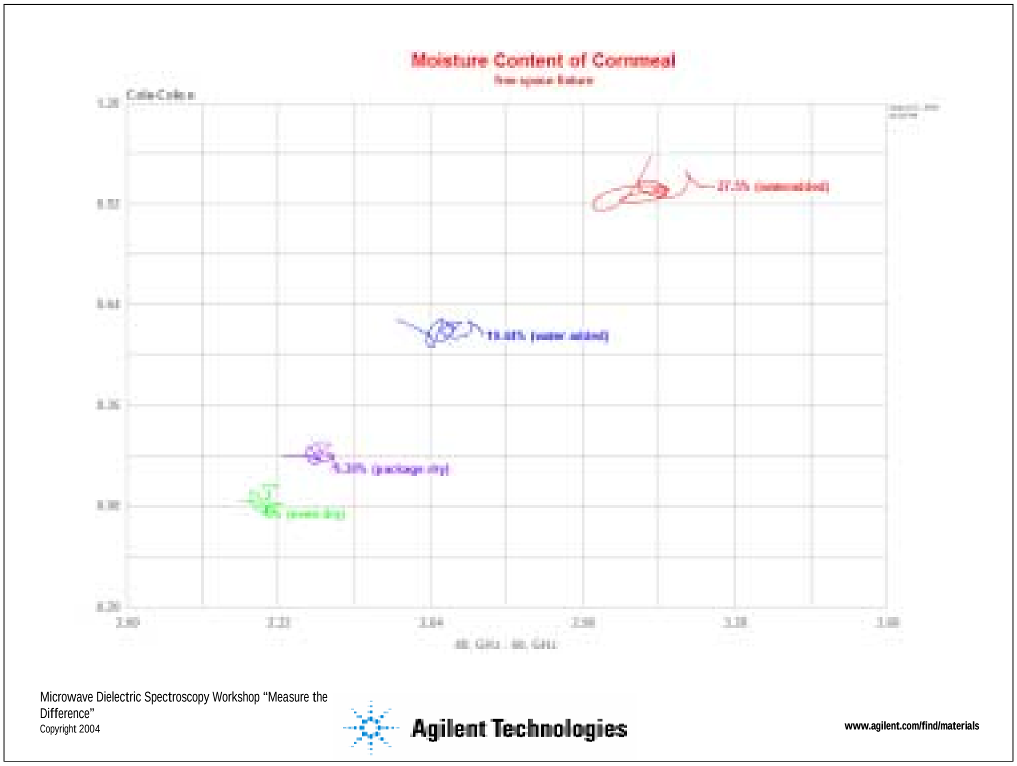
A martini is a mixture of gin and vermouth. Measuring the permittivity of this and other mixtures would allow you to determine the concentration of the mixture.



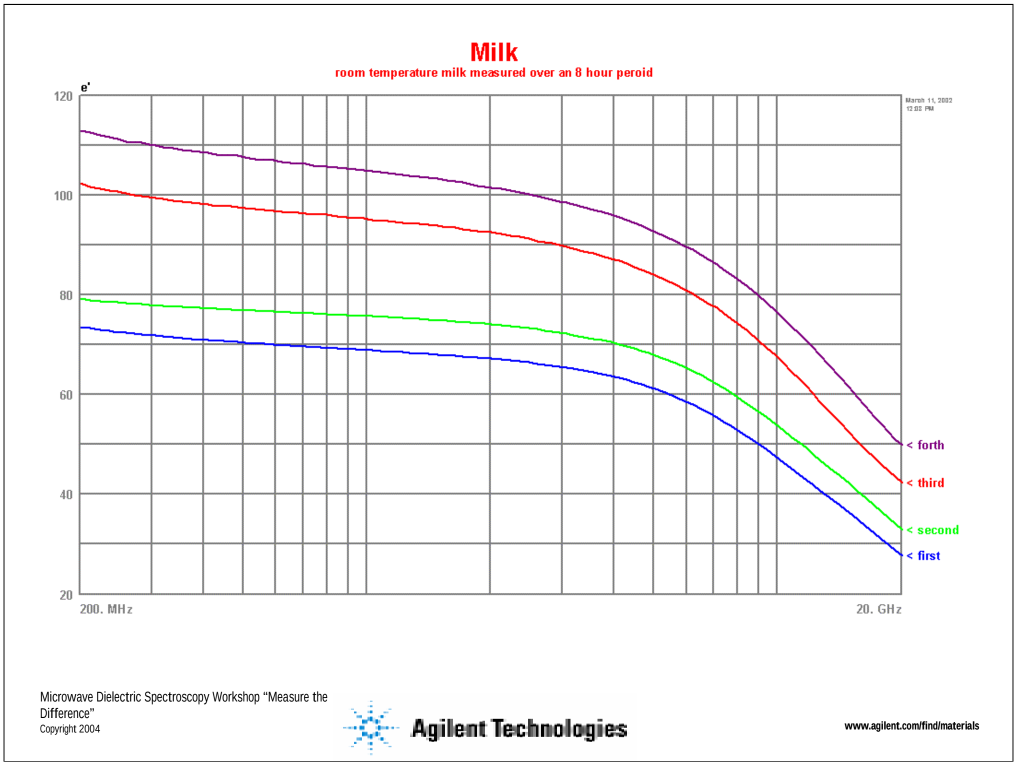
Here's a look at the differences between beer, lite beer and water. The green trace is water, the purple trace is lite beer, and the blue trace is regular beer. On first glance, one may think that lite beer is just beer with water added. But looking closer, you can see that the maximum value of the two beers are lower frequency of the water. Adding water to beer will not give the same result as lite beer.



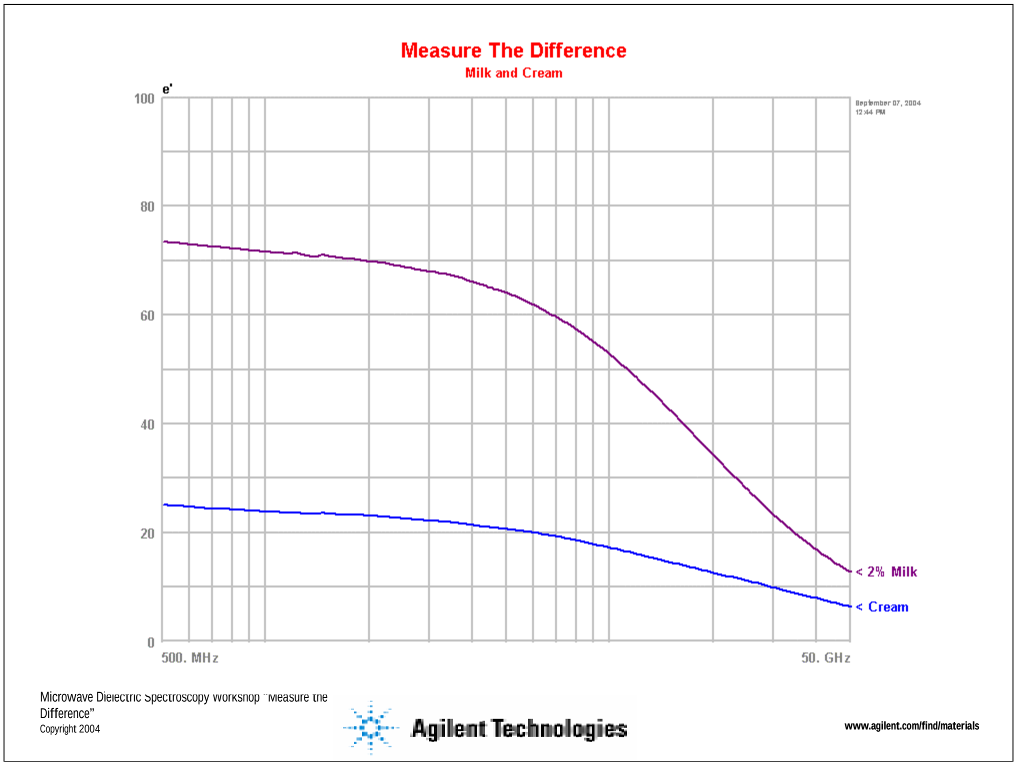
Wine never tastes the same the day after it was opened despite re-corking it. This change is measurable.



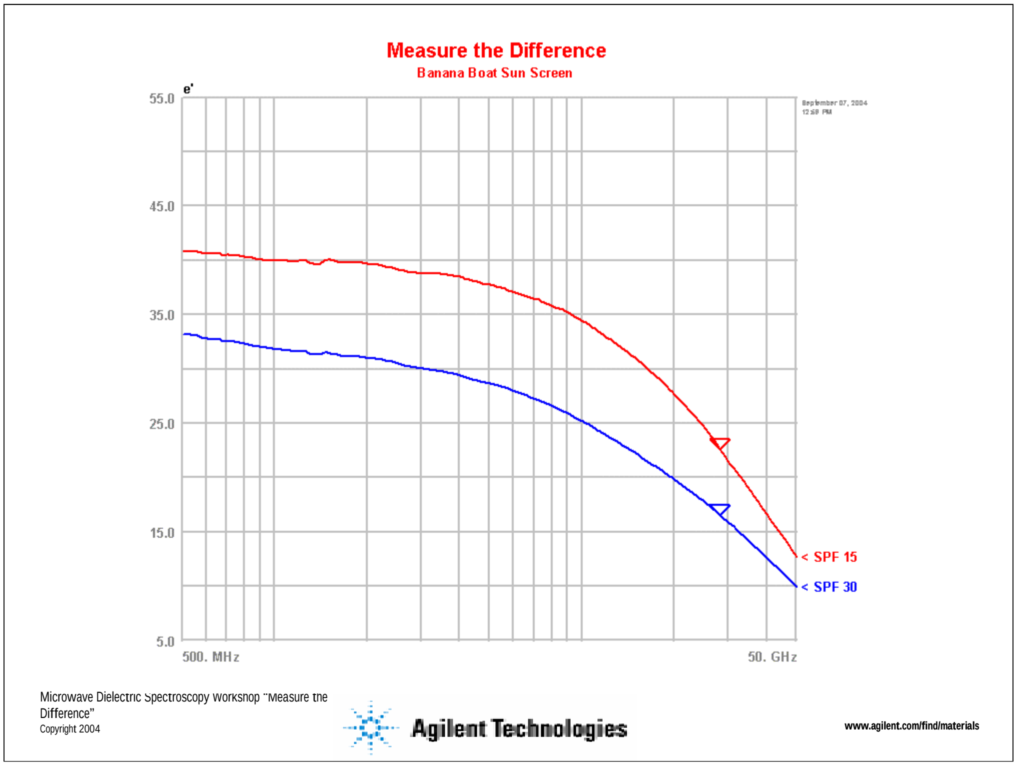
This is a measurement of Cornmeal samples with different levels of moisture content measured with a freespace system. The graph shows a Cole-Cole display. You can see that both the real part, on the x axis, and the imaginary part, on the y axis, increase as the moisture content increases. The University of Georgia has done some in depth work correlating moisture content to dielectric properties.



A glass of room temperature milk was measured over an eight hour period. The milk changed from being drinkable to spoiled. This suggests that bio-content can be correlated to dielectric properties.



The permittivity of skim milk is different from cream. This measurement suggests that fat content could be determined from permittivity.



And just so you don't think that this measurement technique is only good for food products, here is a measurement of sun screen. The red trace is SPF15 and the blue trace is SPF30. Since microwave dielectric spectroscopy is not an optical technique it does not have problems measuring opaque or white materials.

Applications:

Food Science

Chemistry

Biology

Medicine

Drug Discovery

Agriculture

Non-destructive testing

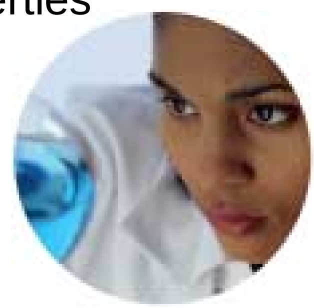
Electrical/Electronic Devices

Stealth Vehicles

Because a material's permittivity changes with molecular changes its measurement has found application in many industries. It can be used to determine the suitability of materials used to package microwaveable foods. Chemical reactions can be monitored. Bio-mass can be measured in fermentation. Measurements can detect the presence of cancerous tumors. Drug/protein interaction can be observed. Moisture content can be monitored in real time.

“Measure the Difference”

- Why Dielectric Spectroscopy?
- Definition of dielectric properties
- Relationship to other properties
- ➢ Measurement techniques

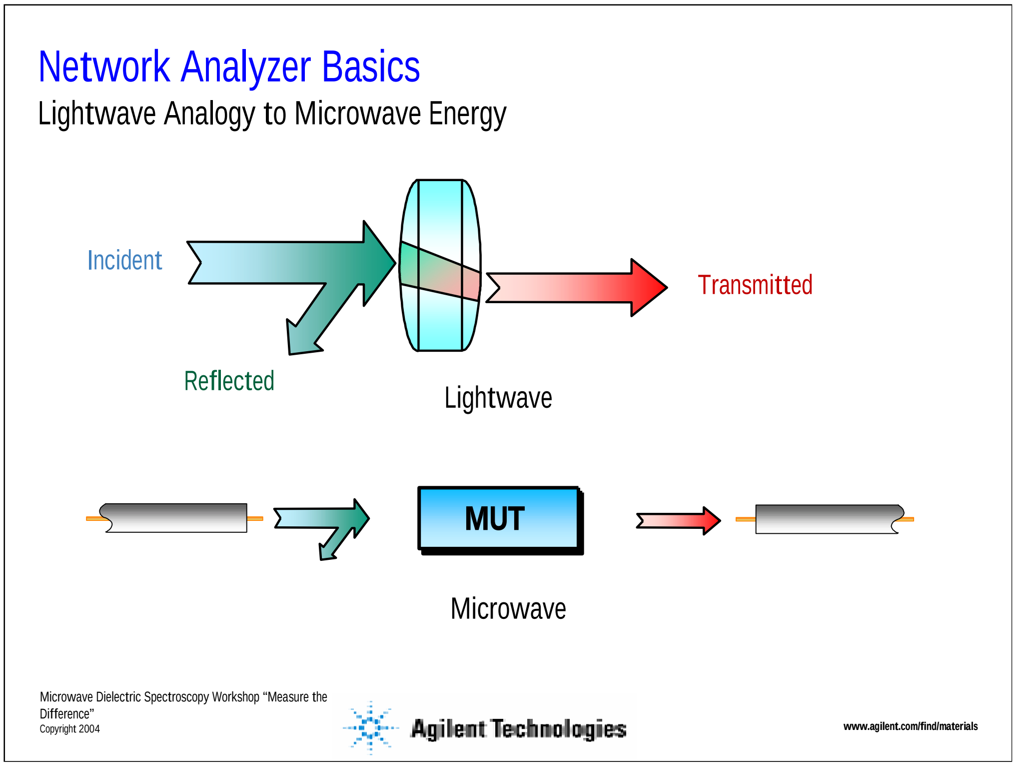


.

Measurement Techniques

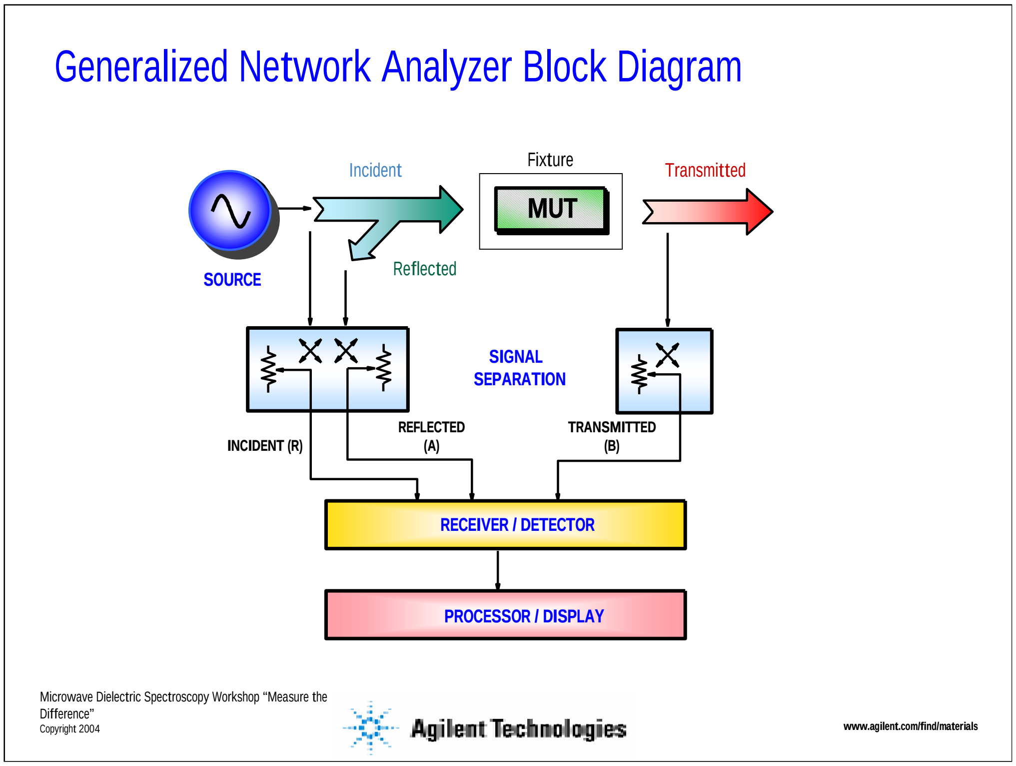
- Coaxial Probe
- Transmission Line
- Freespace
- Resonant Cavity

Today, we will be demonstrating techniques that use a microwave Network Analyzer. The Network Analyzer is useful because it is broadband, 300KHz to 325Ghz, and it’s general purpose capabilities make it useful for a variety of techniques. The Impedance Analyzer offers additional techniques for lower frequency applications.



First a brief look at how a network analyzer works. One of the most fundamental concepts of network analysis involves incident, reflected and transmitted waves traveling along transmission lines. It is helpful to think of traveling waves along a transmission line in terms of a lightwave analogy. We can imagine incident light striking some optical component like a clear lens. Some of the light is reflected off the surface of the lens, but most of the light continues on through the lens. If the lens were made of some lossy material, then a portion of the light could be absorbed within the lens. If the lens had mirrored surfaces, then most of the light would be reflected and little or none would be transmitted through the lens. This concept is valid for RF signals as well, except the electromagnetic energy is in the RF range instead of the optical range, and our components and circuits can be electrical devices, networks and even materials, instead of lenses and mirrors.

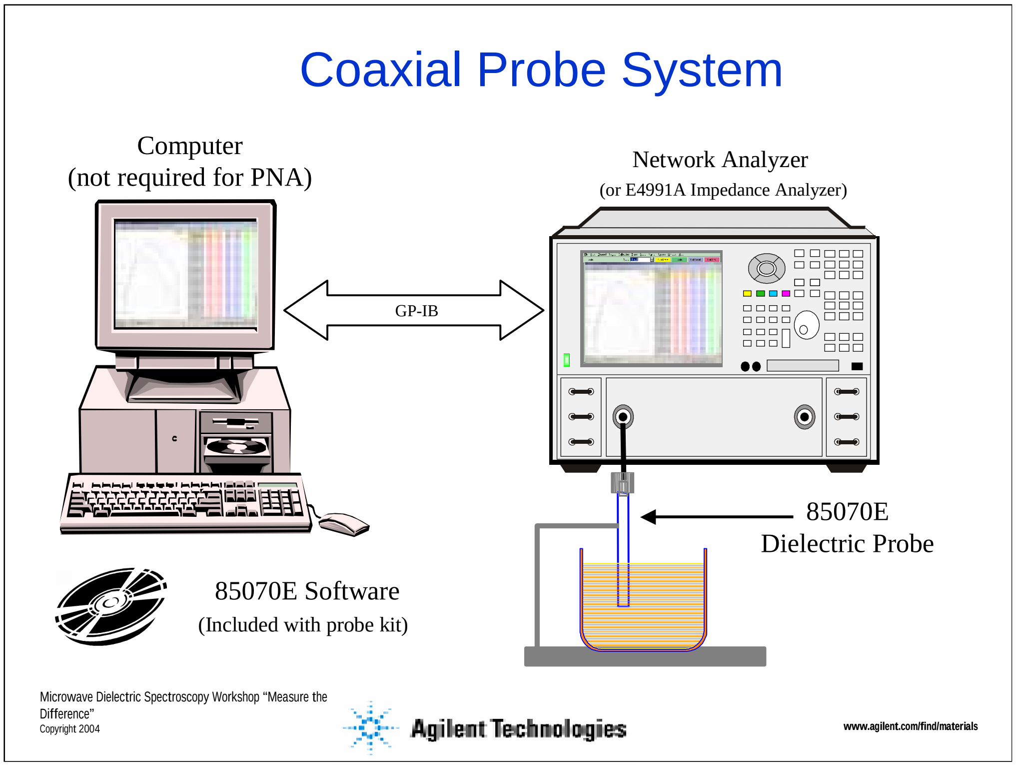
Network analysis is concerned with the accurate measurement of the *ratios* of the reflected signal to the incident signal, and the transmitted signal to the incident signal.



Here is a generalized block diagram of a network analyzer, showing the major signal-processing sections. In order to measure the incident, reflected and transmitted signal, four sections are required:

- Microwave signal source for stimulus
- Signal-separation devices
- Receivers that down convert and detect the signals
- Processor/display for calculating and reviewing the results

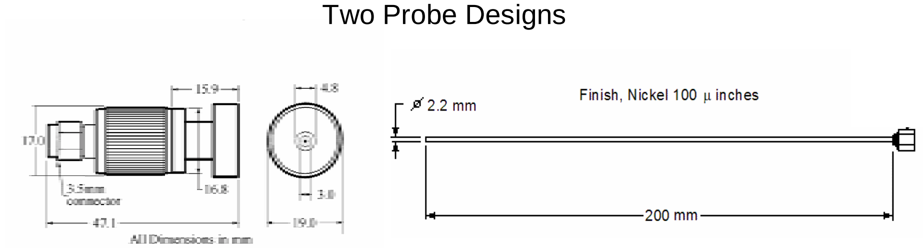
A reflection measurement is the ratio of the Reflected signal detected at A, over the Incident signal detected at R. A transmission measurement is the ratio of the Transmitted signal detected at B, over the Incident signal detected at R. Ratioed measurements reduce errors caused by imperfections in the source. Errors caused by differences in these signal paths or any leakage signals within the network analyzer can be calibrated out by the user before measurements are made.. Calibration is typically a simple procedure where three known standards are measured.



A typical coaxial probe system consists of a vector network analyzer, a coaxial probe, an external computer with HP-IB card and software. For the PNA series network analyzers, a computer is not required because the software can be run directly on the instrument.

Coaxial Probe Technique

Two Probe Designs



High Temperature Probe	Slim Form Probe
•0.200 – 20GHz	•0.500 – 50GHz
•Withstands -40 to 200 degrees C	•Fits in tight spaces, smaller sample sizes
•Survives corrosive chemicals	•Low cost consumable design
•Flanged design allows measuring flat surfaced solids in addition to liquids and semi-solids	•For liquids and soft semi-solids only

Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies

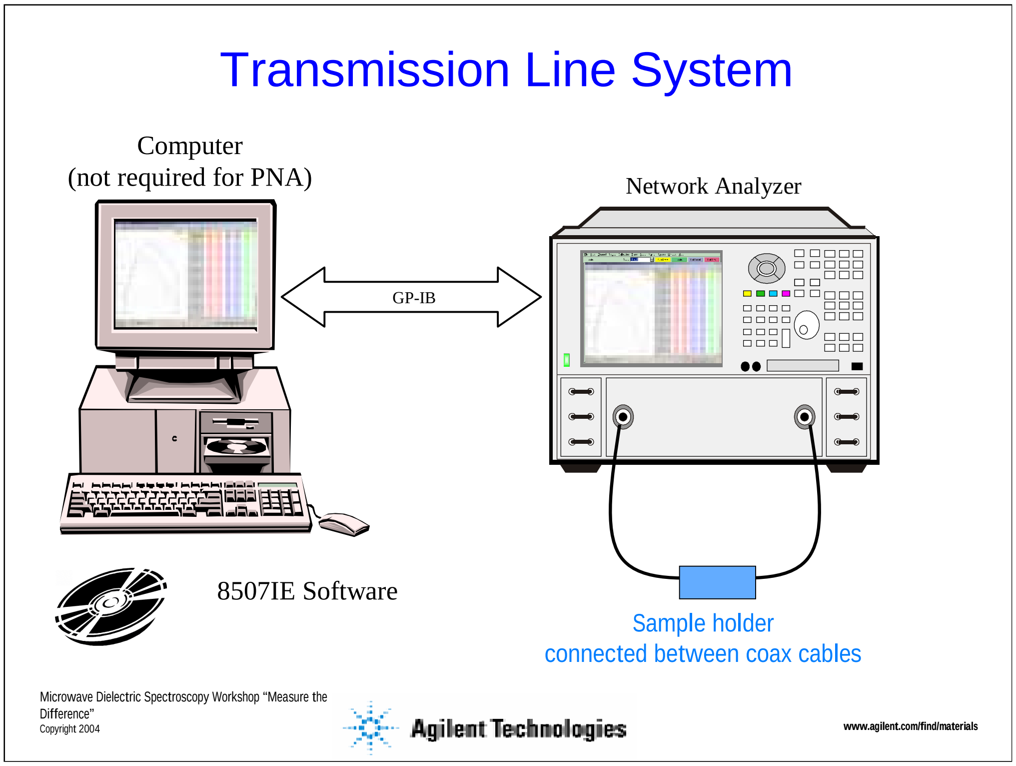
www.agilent.com/find/materials

The coaxial probe technique is best for liquids and semi-solid materials. For hard solid materials one flat surface is required. Special precautions should be taken to avoid air gaps between the sample and the probe (this may be air bubble in the case of liquid). Since only the s_{11} parameter is measured, only the dielectric properties can be calculated, and the MUT should be non magnetic. The underlying theory presumes infinite sample. In reality the sample should be "thick enough". The method is simple, convenient, nondestructive (no special sample is needed in most of the cases) and with one measurement we can sweep up to 50 GHz. The disadvantages of the method are the limited accuracy compared with other methods (transmission method 85071E, resonator methods) and the limitation of the thickness of the sample.

Coaxial Probe Technique

Strengths	Limitations
Non-destructive for many materials	Requires sample thickness of > 1 cm (typical)
Broad frequency range, 0.200 – 50GHz.	Sample must be homogenous and isotropic.
Ideal for liquids or semisolids	Solids must have a flat surface
Convenient, easy to use.	Resonate cavity technique better for extremely low loss measurements.

The coaxial probe method is convenient and operates over a wide, 200 MHz to 50 GHz, frequency range. It is not well suited to low loss materials, magnetic materials or where high accuracy is desired.



A typical transmission line system consists of a vector network analyzer and a sample holder connected between the two network analzyer ports.. Agilent provides software that converts the reflection and transmission coefficients to dielectric properties.   For the PNA series network analyzers, a computer is not required as the software can be run directly on the instrument.. For all other network analyzers, an external computer with HP-IB card is required.

Transmission Line Technique

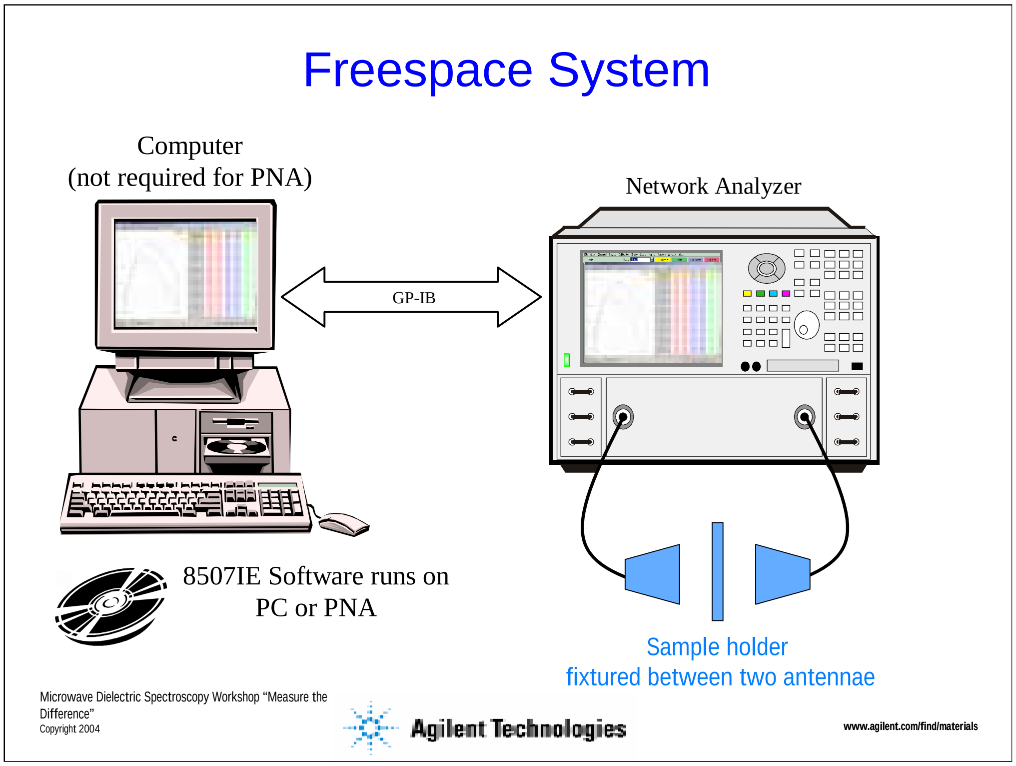
Strengths	Limitations
Widely available coax or waveguide fixtures.	Precise sample shape required (usually destructive)
Broad frequency range, 0.100 to 110GHz)	Needs large samples for low frequencies.
Good solution for hard solid materials.	Liquids, powders and gases must be contained
Can measure magnetic materials.	Resonate cavity technique better for extremely low loss measurements.

Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies

www.agilent.com/find/materials

The transmission line method is best for solid materials that can be precisely machined to fit inside a coaxial or waveguide airline. Although it is more accurate that the coaxial probe technique, it is still somewhat limited in resolution for low loss materials.



A typical freespace system consists of a vector network analyzer, two antennae facing each other with a sample holder between them. Agilent provides software that converts the reflection and transmission coefficients to dielectric properties. For the PNA series network analyzers, a computer is not required as the software can be run directly on the instrument.. For all other network analyzers, an external computer with HP-IB card is required.

Freespace Technique

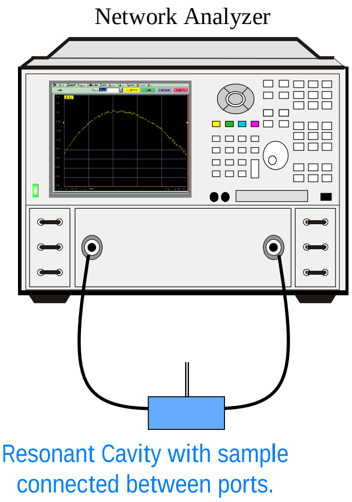
Strengths	Limitations
Non-contacting and non-destructive for many materials. Ideal for remote sensing.	Samples need flat parallel faces.
Broad frequency range, to 325Ghz (range set by antennae and network analyzer).	Very large samples needed at low frequencies.
Ideal for high temperature applications.	Resonate cavity technique better for low loss measurements.
Agilent GRL calibration technique eliminates need for expensive fixturing.	

Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies
www.agilent.com/find/materials

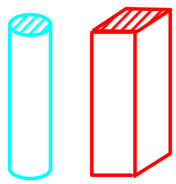
The freespace technique works well for sheet materials, powders, or liquids. Since it is a non-contacting technique, it is ideal for remote sensing and high temperature applications. Special ovens can be purchased with microwave "windows". The sample is placed inside and the test equipment can remain safe outside. Agilent's GRL (Gated Reflect Line) calibration technique eliminates the need for expensive fixturing needed with other calibration techniques.

Resonant Cavity System



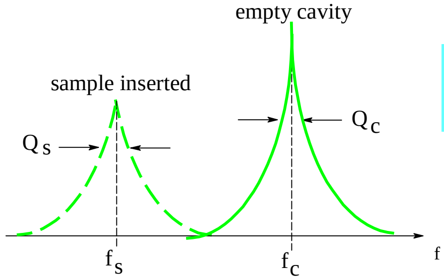
The resonant cavity system consists of a resonant cavity connected between two ports of a network analyzer with coax cables. The dielectric properties can be calculated from the transmission response of the cavity, measured empty and then with the sample. The calculation can be performed manually, or from user written software controlling the network analyzer over LAN or GPIB.

Cavity Perturbation Algorithm



ASTM 2520 Method

A vertical rod or bar sample is inserted in a TE_{10n} rectangular waveguide resonant cavity. There is no need to calibrate the analyzer since only frequency is measured. Scalar analyzer can be used.



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

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

V_s is the volume of the sample

V_c is the volume of the empty cavity

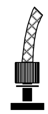
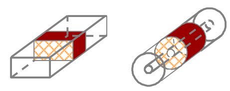
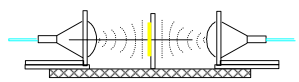
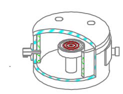
The ASTM 2520 is a fairly simple and commonly use method. This algorithm is dependent on the center frequency and Q measure with and without the sample inserted. The volume of the empty cavity and sample are also required. Other more accurate and complex methods have been developed at NIST.

Resonant Cavity Technique

Strengths	Limitations
More accurate than broadband techniques.	Results at one frequency.
Sensitive to low loss materials	High loss materials need very small sample size.
Small samples size.	Analysis can be complex.
Tubing can be routed through cavity for real time fluid measurements.	Solid materials must be precisely machined to fit in cavity (usually destructive)

The cavity technique is the most accurate one, especially for low loss materials. But measurement are made at single frequencies only and the analysis can be complex

Summary of Techniques

Coaxial Probe		Broadband Non-destructive Best for liquids and semi-solids Software available from Agilent
Transmission Line		Broadband Best for machineable solids Software available from Agilent
Freespace		Broadband Non-Contacting Best for flat sheets, powders, high temperatures Software available from Agilent
Resonant Cavity		Single Frequency Accurate Small samples Best for low loss materials

Microwave Dielectric Spectroscopy Workshop "Measure the Difference"

Agilent Technologies

www.agilent.com/find/materials

Here's a summary of the techniques we talked about.

For More Information

Check out our web site at:
www/agilent.com/find/materials

Call us at:
+1 (800) 829-4444

Email us at:
materials@agilent.com

Agilent technologies is interested in working with you to determine if microwave/dielectric spectroscopy can be used to solve your measurement problem. If you have any questions or would like to discuss your measurement problems with us please drop us an email or contact your local Agilent representative. If practical we would like to make arrangements to measure your material to see if microwave/dielectric spectroscopy would be useful.