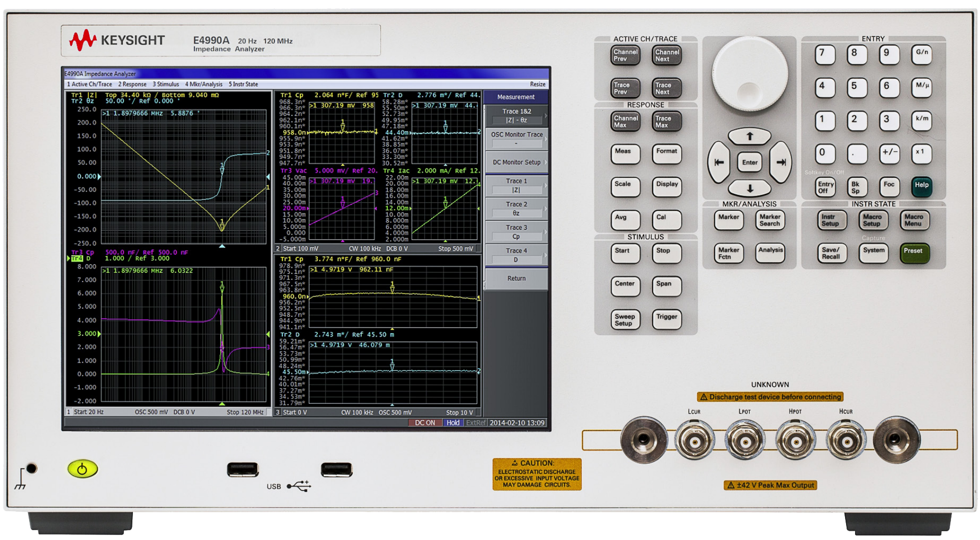
Keysight Technologies
Materials Measurement: Liquid Materials

Application Brief



Overview

There are increasing demands for the electrical measurement to investigate the properties of liquid materials that is characterized by permittivity and conductivity.

Permittivity, also called as dielectric constant is related to the electric polarization and conductivity is the measure of a material's ability to conduct electric current when applied an electrical field. Dielectric spectroscopy, also known as electrochemical impedance spectroscopy (EIS) measures permittivity and conductivity as a function of frequency. Complex permittivity measurement enables not only to evaluate electrical property but also to analyze the composition, the structure of the heterogeneous system and the interaction of the molecules in the liquid.

The electrical measurement is fast, non-destructive, non-invasive and cost effective method and also allows continuous monitoring of liquid material without any pretreatment. It can be applied to liquid, emulsion and colloidal suspension including chemical materials, crude oil, cosmetics, wine or whiskey brewing as well as blood and biological cell.

Complex permittivity can be derived from the electrical impedance of the liquid, which can be measured using impedance analyzers with appropriate fixtures.

Problem

For scientists who are not familiar with the electrical measurement, it is not easy to understand the electrical model of liquid materials.

Complex permittivity measurement is done by measuring the electrical impedance of the material using LCR meters or impedance analyzers. Although they are accepted as standard instruments in evaluating electronic components and materials, special devices will be required when applying to liquid materials.

Dielectric spectroscopy is suited for in-situ characterization of colloidal dispersion or molecular dynamics however it requires accurate measurement system over a wide frequency range.

Solution

Impedance analyzers are the best instruments for the evaluation of electronic components and materials. The Keysight Technologies, Inc. 16452A liquid test fixture is designed to be used with the Keysight E4990A impedance analyzer for the permittivity measurement of liquid materials. Their four-terminal-pair configuration effectively eliminates the errors due to the cables. The 16452A adopts parallel plate capacitance method covering the frequency range from 20 Hz to 30 MHz and has liquid container between two electrodes that form a capacitor filled with the liquid material. The distance of the electrodes is variable by changing spacers and it requires sample of less than 10 mL.

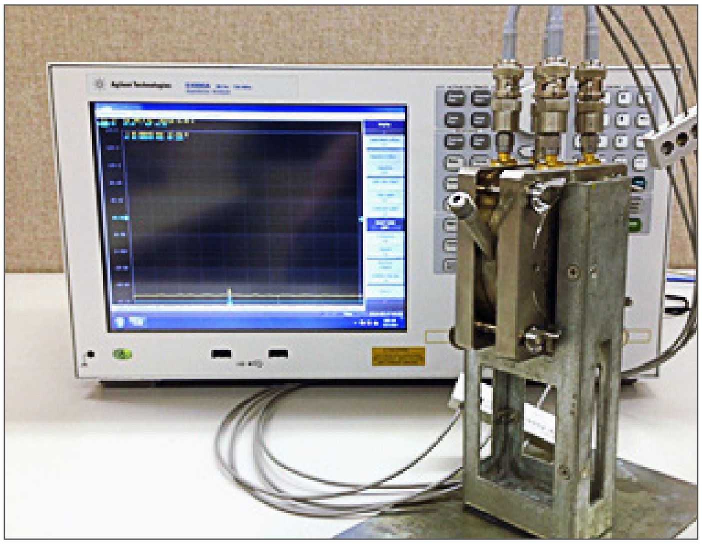


Figure 1. Impedance analyzer with liquid test fixture

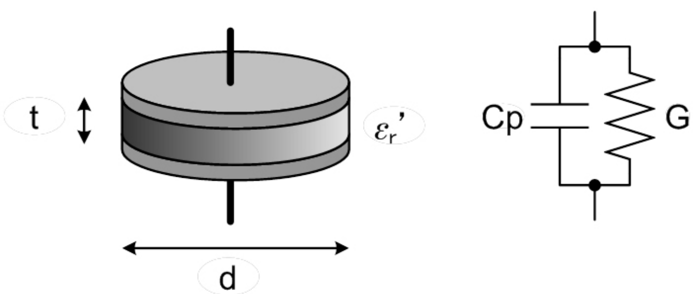


Figure 2. Parallel plate capacitance method

Complex relative permittivity is derived by comparing the capacitance of the 16452A fixture with and without the liquid material filled in it, which provides accurate measurement regardless the size of the fixture.

$$\varepsilon^* = \varepsilon' - j\varepsilon'' = \left(\frac{C_P}{C_0} - j\frac{G}{\omega C_0} \right)$$

where ε^* : complex permittivity

ε_0 : permittivity of free space

C_P : measured capacitance

G : measured conductance

C_0: air capacitance

ω: angular frequency

Shown below is an example in which mixtures of purified water and anhydrous ethanol with different mixing ratio were measured with the 16452A and the E4990A. The volume ratio of the water is 0/25/50/75/100%, respectively. The relative permittivity increases when increasing the mixing ratio of water. At 0% and 100%, the values are consistent with the permittivity of ethanol (24.3 at 25 °C) and water (78.3 at 25 °C) found in literatures, while the permittivity in-between well agree with those calculated from the mixing ratio.

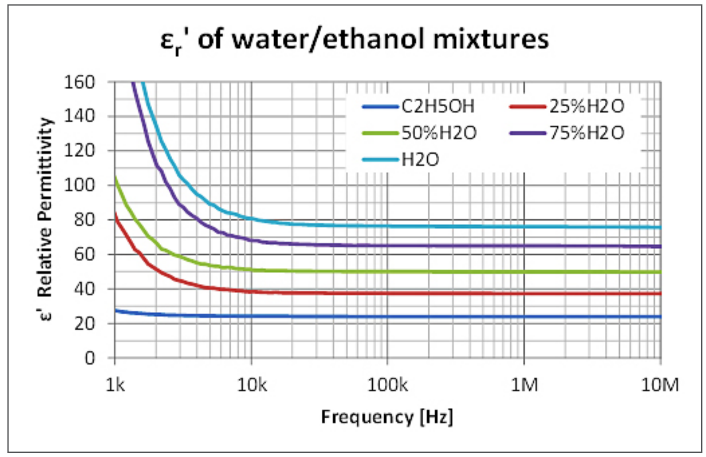


Figure 3. Relative permittivity of water/ethanol mixture

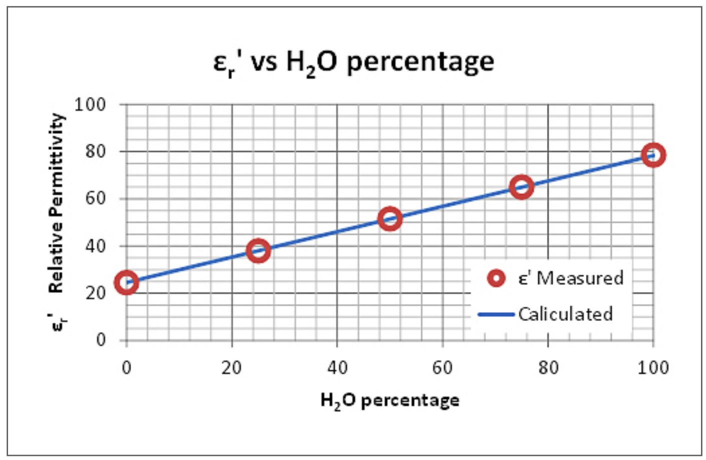


Figure 4. Permittivity change by volume fraction

The 16452A is suited for the measurement of liquid materials with low permittivity and low conductivity. It provides accurate measurement eliminating the errors due to the fringe capacitance by the guard electrode.

If sample contains electrolyte, problem occurs below 10 kHz because of the electrode polarization, which is shown in the graph above and is more serious when applying to bio materials. Although there is no established method to eliminate or to correct this effect, platinized platinum electrodes (platinum black coated platinum electrode) properly reduce it.

The following is an example of a hand-made measurement cell (fixture) with the platinized platinum electrodes. The system consists with the E4990A impedance analyzer, the 42942A terminal adapter, the 16092A spring clip fixture and the measurement cell.

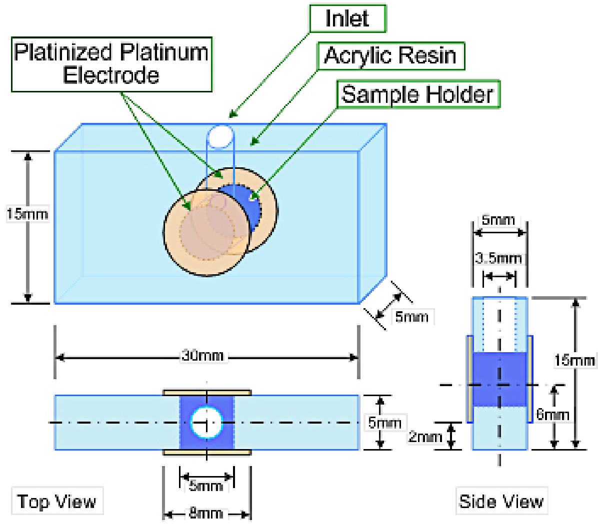


Figure 5. Schematic of a sample measurement cell

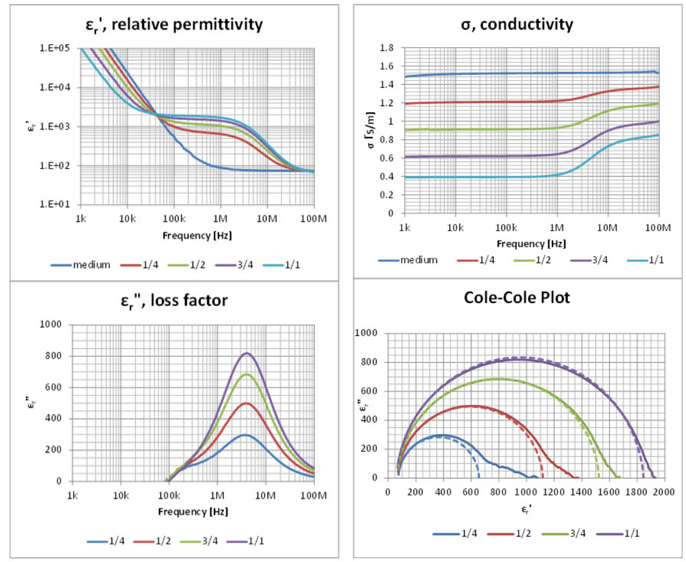


Figure 7. Dielectric spectra of red blood cell of different concentration

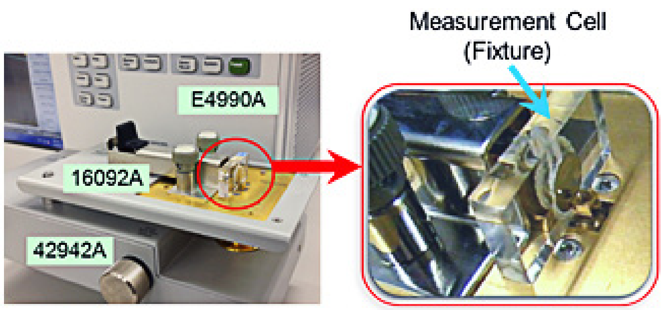


Figure 6. Measurement setup

The origin of the large permittivity is the interfacial polarization at the cell membrane where charged ions in the cell transferred by the electric field are accumulated. As the frequency increases, the movement of the charged ions cannot follow the change of the electrical field and the permittivity decreases consequently. It is called dielectric relaxation and the behavior provides information on the structure and the electrical properties of the constituent. In this way, complex permittivity is closely related to the physical property of the liquid material and can be applied to the investigation or to the monitor of bio-materials.

Measurement errors due to the analyzer and the adapter can be removed by the correction function built in the analyzer. Conversion from the measured capacitance to the permittivity is done using two known sample as references.

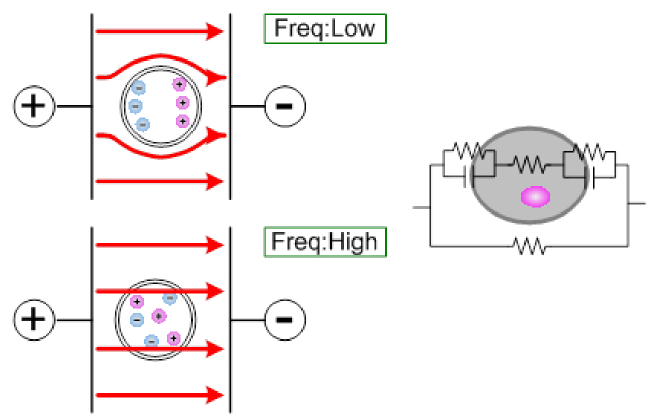


Figure 8. Interfacial polarization and equivalent circuit

The next example shows a dielectric spectroscopy measurement result done with the system described above. The measurement cell has been calibrated using air and water. The sample is suspension of red blood cell of horse which has been separated by centrifugation, washed by phosphor buffered saline (PBS) and diluted with PBS at different concentrations.

The relative permittivity of the medium (PBS only) is close to that of water, while for the cell suspension, huge permittivity is observed below 10 MHz and conductivity decreases accordingly. Permittivity also increases as the concentration of red blood cell increases. As can be seen in the graph, complex permittivity measurement up to 100MHz is possible with the system. It should be noted that the increase of the permittivity below several 10 kHz is due to the electrode polarization and does not reflect actual dielectric property of the sample. In the result of εr" and Cole-Cole plot, electrode polarization and DC conductivity have been curve fitted and then removed mathematically.

At higher frequencies, another relaxation is observed, that is caused by dipolar polarization of polar molecule. Water molecule, for example, has dipole and the dipolar polarization rolls off around 22 GHz. The Keysight dielectric probe kit combined with the Keysight network analyzers covers permittivity measurement up to microwave region and is suited for the research of liquid materials at the level of a molecule.

Conclusion

Complex permittivity of liquid material reflects the physical properties of constituent. Dielectric relaxation is observed due to various mechanisms, which gives information on liquid materials. Complex permittivity measurement provides simple, fast and non-destructive method that can be applied to physics, industrial processes, bio-chemical analysis, etc.

Parallel plate capacitance method is widely used for permittivity measurement. The Keysight E4990A impedance analyzer combined with appropriate test fixture with parallel electrodes is suitable for the application and the 16452A liquid test fixture is one designed for liquid measurement. At higher frequencies where the molecular dynamics are of interests, the dielectric probe kit used with Keysight network analyzers provides optimum solution. For more information, application notes and white papers are listed under references.

References

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- *Basics of Measuring the Dielectric Properties of Materials, Application note,* Literature number 5989-2589EN
- *Keysight 85070E Dielectric Probe Kit, Technical overview,* Literature number 5989-0222EN

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