# Agilent AN 1300-8

# Dielectric and Magnetic Material Characterization with the Novocontrol Concept 60 System

Applicaton Note

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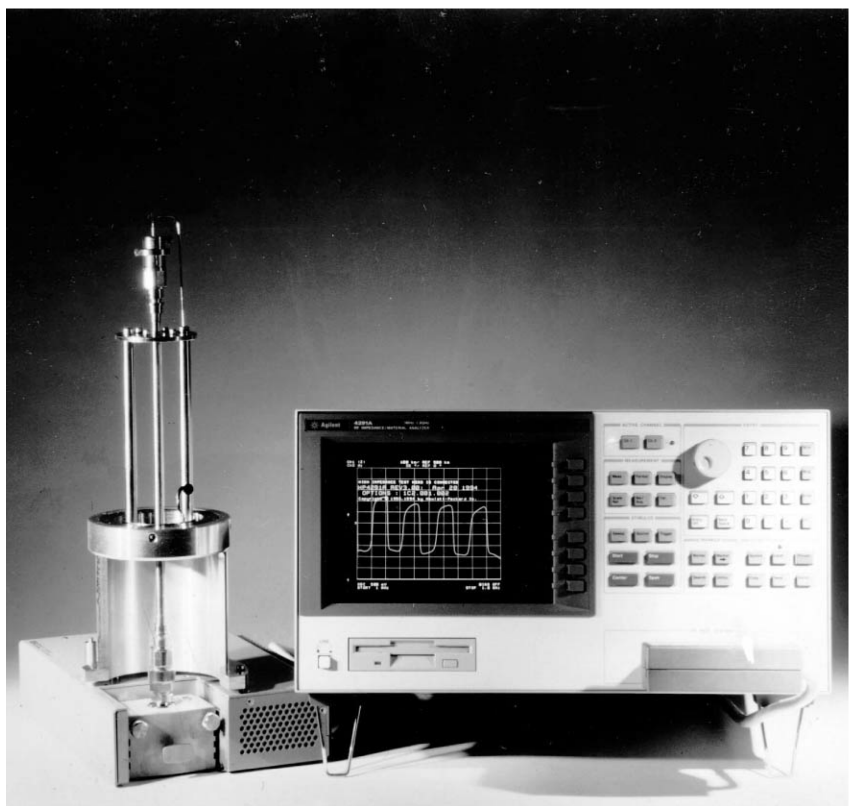
**Novocontrol GmbH**

**Agilent 4291B 1.8 GHz**
**Impedance/Material Analyzer**

## Introduction

Dielectric spectroscopy is a valuable tool for the characterization of materials properties. The complex dielectric function ε reflects the molecular relaxation and transport processes of the material. As ε = ε' - iε" depends on many different physical quantities, dielectric measurements often are done as a function of frequency, temperature, time, and DC bias (superimposed static electric field). The Novocontrol Concept 60 system based on the Agilent 4291B 1.8 GHz Impedance/Material Analyzer automatically performs these measurements. The 4291B was developed especially for impedance measurements and uses a new technique developed by Agilent Technologies. This technique offers more accurate characterization of devices and materials. Compared to solutions based on spectrum or network analyzers, it has a much higher resolution and a broader impedance range, which are key considerations. In addition, the magnetic properties of materials can be evaluated by measurements of the permeability, which are also supported by the Concept 60 system.



**Novocontrol Concept 60 System Based on the Agilent 4291B**

## Dielectric Behaviour and Material Properties

Temperature analysis is particularly important for the characterization of materials, as the electric response measured by the dielectric function strongly depends on temperature. From the temperature dependence, a lot of the information on the physical, chemical, and electrical material properties can be obtained. First, it allows separate isolators, polymers, plastics, semiconductors, metals and superconductors. Also the solid, glass, liquid, and gas state, and other structural phase transitions of the material can be distinguished and characterized. For each material class and state, the temperature dependence contains special information.

- For isolators, the magnitude and temperature dependence of the residual conductance gives information about the amount and nature of impurities.

- For polymers and plastics, the dielectric spectra are shifted by temperature. From the temperature dependence, information on the molecular dynamics like activation energy and glass temperature can be obtained.

- For semiconductors, the temperature dependence of the dielectric function allows you to determine the most important electric parameters like gap energy, charge carrier density, and mobility.

- In metals, the temperature dependence of the dielectric function (which here is traditionally evaluated in terms of the complex conductivity) gives information on the electron phonon interaction and the residual resistance.

- At low temperatures, the properties of superconductors can also be studied. This is of particular interest for high temperature superconductors with transition temperatures up to -140°C and above. In this case, the temperature dependence of the dielectric function can give information on the mechanism of high temperature superconductivity that is still not resolved.

The above examples mention only the most familiar topics related to dielectric temperature analysis. In addition, there are an unlimited number of special problems that can be evaluated by the temperature analysis of the complex dielectric function. Therefore, adding temperature control to a dielectric measurement system multiplies its performance.

## System Performance

In the Concept 60 system, the Agilent 4291B is extended with a high performance cryogenic temperature control system that was particularly designed for dielectric applications. The complete system is controlled by a PC with the MS-Windows software package WinDETA.

**System description:**

| Independent parameters | |
|---|---|
| Frequency | 1 MHz to 1.8 GHz |
| Temperature | –160°C to +450°C |
| DC-Bias | –40 V to +40 V |
| Time | 2 s to $10^7$ s |

Any multi-dimensional combination of the independent parameters is supported for both experiment set-up and graphical or numerical evaluation.

| Dependent parameters | Range |
|---|---|
| Dielectric function $\varepsilon$ | 1 to 1000 |
| Conductivity $\sigma$ | $10^3$ to $10^{-8}$ S/cm |
| Loss factor tan ($\delta$) | $2 \times 10^{-3}$ to 1000 |
| Impedance Z | 0.1 to $10^5$ $\Omega$ |

**Other dependent parameters supported by the WinDETA software are:**

Real, imaginary part and absolute value of dielectric function, conductivity, permeability, capacity, inductance and admittance in parallel and series circuit representation, loss angle, loss factor, phase angle, and power factor. Further information on accuracy and range of the dependent parameters is given in the Agilent 4291B Operation Manual.

## Applications of Dielectric Spectroscopy

**Key scientific applications include:**

- Relaxation processes on the molecular dynamics of liquid crystals, polymers and liquids

- Charge transport in semiconductors, organic crystals, ceramics, etc.

- Timing development of chemical reactions, polymerization and curing processes

- Structural material properties like phase compositions, phase transitions and crystallization processes

- Non-linear electrical and optical effects

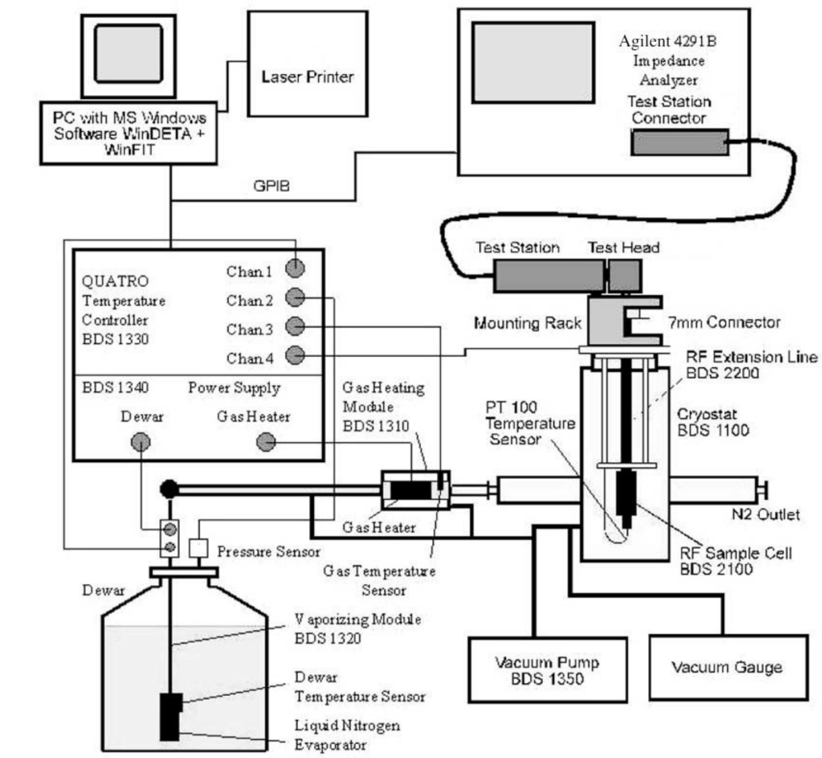



**Figure 1. Novocontrol Dielectric Spectrometer Concept 60 based on the Agilent 4291B**

Industrial applications include:

- Quality control and characterization of isolating and semiconductor materials.
- Materials that change their dielectric function if exposed to gases or liquids are used in sensor applications.

Another interesting application is the characterization of printed circuit boards. This is necessary, as the functional behaviour of boards, operated with high speed electronics up to the GHz range, is largely influenced by the dielectric properties of the board materials.

## System Components

The Concept 60 dielectric spectrometer consists of the following components shown in Figure 1:

- A special RF sample cell with the sample capacitor
- A microwave precision extension line connecting the sample cell to the impedance analyzer
- 4291B 1.8 GHz Impedance/ Material Analyzer
- Temperature control system allowing exposure of the sample capacitor to temperatures from −160°C to +40°C with 0.01°C stability and very short settling times
- PC with the MS-Windows software packages WinDETA and WinFIT that control the measurement flow, operate all devices, and evaluate the measured data

## Principle of Operation

The material under test generally is placed between two electrodes, creating a capacitor as shown in Figure 2.

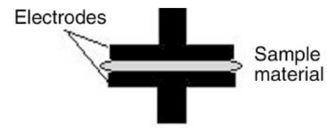



**Figure 2. Sample material placed into a measurement capacitor**

The capacitor plates are round with a diameter from 1 mm to 12 mm. The spacing between the plates may be selected between 2 mm and 10 µm. For special applications, other sample geometries may be used. The sample impedance $Z_S$ is connected to the dielectric function by

$$\varepsilon = \varepsilon' - j\varepsilon'' = \frac{-j}{2\pi f Z_s C_0} \quad (1)$$

where f denotes frequency and $C_0$ is the vacuum capacity of the empty sample capacitor. The sample material is placed between two electrodes in a special high frequency sample cell as shown in Figure 6. The sample cell is mounted into a cryostat and exposed to a heated gas stream being evaporated from a liquid nitrogen dewar (Figure 1). The test head of the 4291B is connected to the sample cell by a microwave precision extension line.

## Measurement Example

The dielectric function of glycerol for temperatures between 193 K and 333 K is shown in Figures 3 and 4. Figure 3 shows the real part ε', and Figure 4 shows the imaginary part ε" of the dielectric function. The curves represent the main dielectric relaxation (α-relaxation) due to orientation polarization of permanent dipoles in the applied electric field. ε' is correlated to the polarization strength. ε" measures the electric losses of the relaxation and the dc-conductivity.

For a fixed temperature and low frequency, the electric dipoles follow the external field without delay. The polarization is constant at a high level. As frequency increases, the dipoles follow the field more slowly and are only partly oriented. As can be seen from Figure 3, the polarization decreases. The electric losses show a maximum at the crossover frequency from high to low polarization (Figure 4).

At higher temperatures, the mobility of the molecular dipoles increases and the relaxation frequency is shifted to higher values. In addition, the dc-conductivity caused by thermal activated free charge carriers increases. This can be seen in Figure 4 in the increase of ε″ on the left, from the dielectric relaxation in the 1 MHz range. An analytical evaluation of the measured data is done by the Novocontrol WinFIT software, which is a part of the standard Concept 60 system. The solid lines in Figures 3 and 4 are calculated according to the relaxation function of Havriliak-Negami (including the Cole-Davidson and Debye functions) in combination with an ac-conductivity term and fitted to the data by WinFIT.

From this evaluation, information about the molecular dynamics of the material, such as the information below, can be obtained.

- Activation energy
- Glass transition temperature
- AC- and DC-conductivity
- Time domain behaviour

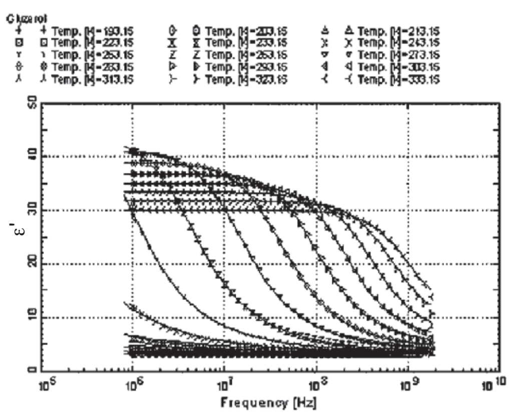



**Figure 3. Real part ε' of the dielectric function of Glycerol for several temperatures**

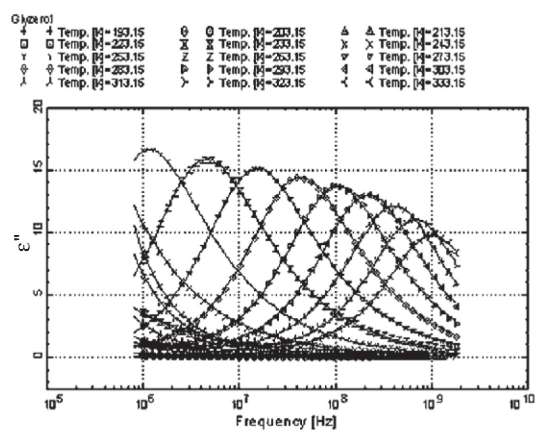



**Figure 4. Imaginary part ε″ loss index of the dielectric function of Glycerol for several temperatures**

## Impedance Measurement

In contrast to the low frequency techniques, coaxial connecting cables above 10 MHz always introduce errors to impedance measurements. Above approximately 30 MHz, additional standing waves arise at the line and a direct measurement of the sample impedance completely fails. This can be avoided by application of microwave techniques taking the measurement line as the main part of the measured impedance into account. Therefore, the connection from the analyzer test head to the sample cell is made by a coaxial precision line with defined, and nearly temperature independent, propagation constants.

The sample capacitor is used as the termination of the extension line. The complex reflection factor $r(l)$ at the analyzer end of the line is dependent on the sample impedance and is measured with the Agilent 4291B. The incoming and reflected waves are separated with two directional couplers and are phase sensitive measured. $r$ is defined as the ratio of the voltages (or electrical fields) of the reflected wave to the incoming wave on the line; it depends on the location of the measurement on the line. (See Figure 5.)

$$r(x) = \frac{U_{Ref}(x)}{U_{In}(x)} \qquad (2)$$

For an ideal line, $r(l)$, which is measured by the analyzer, can be transformed to the reflection factor $r(0)$ at the sample end of the line by

$$r(0) = r(l)\, e^{2l(\alpha+j\beta)} \qquad (3)$$

where $\alpha$ is the damping constant and $\beta = 2\pi l/\lambda$ ($\lambda$: wavelength) the propagation constant of the line. From (5) , the sample impedance is calculated by

$$Z_s = Z_o \frac{1 + r(0)}{1 - r(0)} \qquad (4)$$

where $Z_0$ is the wave resistance of the line (50 Ω).

In practice, lines are not ideal and sophisticated calibration procedures have to be applied. The line parameters $\alpha$, $\beta$ must be homogenous over the entire line and also independent of temperature, as the calibration generally can only be carried out at room temperature.

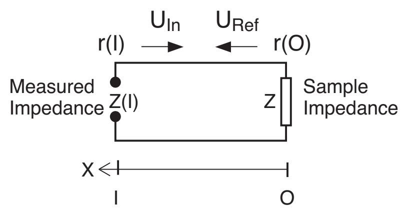



**Figure 5. Reflection factors and impedance on a line terminated by the sample**

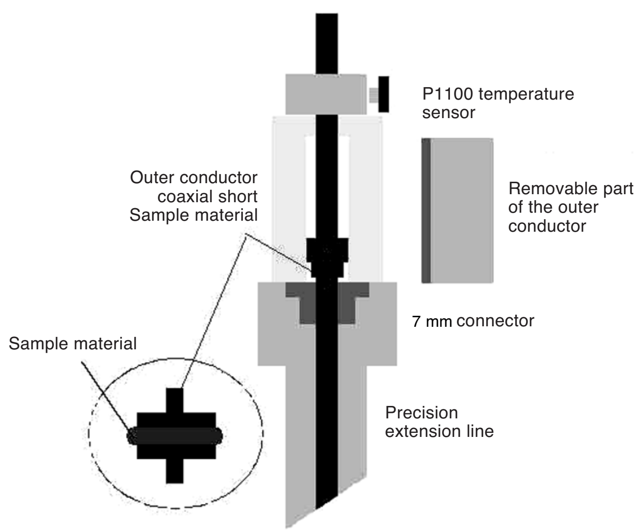



**Figure 6. RF sample cell with coaxial short and sample capacitor**

The same criteria mentioned for the line apply to the sample cell shown in Figure 6. Therefore, an additional calibration that eliminates the influence of internal impedances in the sample cell is applied. In order to keep the wavelength as short as possible, the test head of the 4291B is directly mounted at the top of the cryostat with the sample cell.

The frequency range is 1 MHz to 1.8 GHz with a resolution in tan (δ) < $10^{-3}$ and overlaps with other Novocontrol Dielectric Analysis Systems in the lower frequency range. This is important, as the accuracy of measurements at the limits of the range rapidly decreases.

## Magnetic Material Measurements

In addition to the dielectric measurement by a capacitor, magnetic permeability measurements are supported.

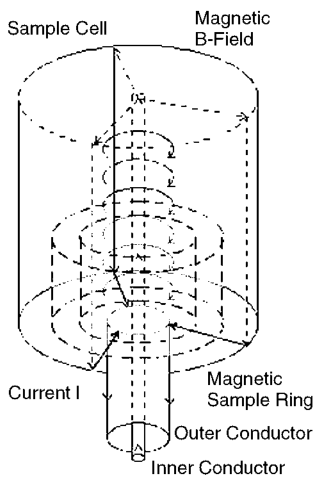



**Figure 7. Principle of the sample cell for magnetic material measurements**

The sample must have the geometry of a cylindrical ring. It is placed centered in the magnetic cell as shown in Figure 7. If the sample cell is completely filled by the sample material, the magnetic permeability

$$\mu^* = \mu' - j\mu'' \qquad (5)$$

of the sample material can be calculated from the complex sample inductivity by

$$\mu^* = \frac{L^*}{L_0}; \qquad L^* = j\omega Z^* \qquad (6)$$

($L_0$: empty cell inductivity, $\omega = 2\pi/$frequency, $j^2 = -1$, $Z^*$: measured sample cell impedance)

In practice, the sample material must not completely fill the cell. In this case, the inductivity of the unoccupied cell volume is subtracted from the measured impedance in Equation (6). Magnetic material properties are determined under computer control, with or without temperature control, by the Novocontrol winDETA software.

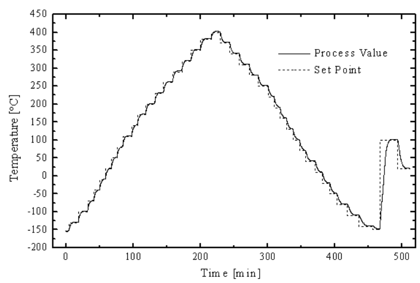



**Figure 8. Temperature setpoint and sample temperature in dependence of time for the Quatro Cryosystem**

## Quatro Cryosystem

The Quatro Cryosystem allows the user to automatically set any sample temperature required. The system is modular and may be combined with any Novocontrol Dielectric Analysis System. The main parts of the system shown in Figure 1 are the cryostat, the gas jet, and the microprocessor controlled Quatro system. The Quatro consists of four independent loops controlling the sample temperature, the gas temperature (at the gas heater), the temperature of the liquid nitrogen in the dewar, and the pressure in the dewar.

The liquid nitrogen evaporator heats the liquid nitrogen until a specified pressure in the dewar is reached. The actual pressure is controlled by channels 1 and 2 of the Quatro controller. The pressure is automatically adjusted for optimal performance depending on the setpoint of the sample temperature (high pressure at low temperatures, low pressure at high temperatures). Due to the pressure, the cold gas flows out of the dewar through the gas jet to the cryostat. The transfer line from the dewar to the gas jet is vacuum isolated. The gas jet is controlled by channels 3 and 4 of the Quatro controller in order to match the actual temperature at the sample to the desired temperature setpoint. The gas jet is mounted directly onto the vacuum-isolated cryostat in which the sample cell is located.

Due to the high stability of the gas pressure and the two-circuit arrangement of the gas heating, the temperature stability is better than 0.01°C. Moreover, as the gas flows directly along the sample cell, this design allows a very fast and safe operation, which is required for automatic operating systems. Even if the sensors are damaged or the liquid nitrogen dewar becomes empty, there will be no damage to the system or the sample.

A very important requirement of the cryosystem for dielectric measurements is a fast temperature settling time, as settling times are consuming the main part of the measurement time and are slowing down the system performance. On the other hand, the dielectric response may depend on a very sensitive temperature (e.g., in the region of phase transitions). Therefore, a cryosystem that is both fast and very accurate is required. Also, after a setpoint step, the sample temperature should approach the setpoint, but should not exceed it. This is important if samples near a phase transition are measured; but the transition temperature must not be exceeded as the sample will change its structure or even become damaged in this case (e.g., when it melts).

The Quatro Cryosystem meets these three requirements very well. A typical performance diagram of a temperature frequency measurement from –160°C to +400°C with twenty-two steps is shown in Figure 8. Typical stabilization times (for 0.1°C stability) are about 8 minutes per temperature step (20 minutes for 0.01°C). This means, that the demonstration measurement (Figures 3, 4) with fifteen temperature steps is performed in less than 2 hours. Moreover, Figure 8 shows that the sample temperature follows the setpoint with almost no oscillations and without overshooting.

## System Installation, Software and PC

Each Novocontrol system is a turnkey installation including software, PC, and printer. All system parameters are computer controlled by the MS-Windows software winDETA. Besides the 4291B, WinDETA supports many other Agilent impedance analyzers and several temperature controllers in one common graphical user interface. It automatically performs the calibration procedures for the dielectric sample cells and allows dielectric measurements up to four dimensions. The basic dielectric parameters, like complex dielectric function, impedance, etc., are evaluated and graphically displayed in two- and three-dimensional representations.

For further data evaluation, the program WinFIT is offered. It supports data manipulation and nonlinear curve fitting with the Havriliak-Negami, Cole-Davidson, Cole-Cole, Debye, Williams-Landel-Ferry, and Vogel-Fulcher functions. In addition, modelling of the measured data by RCL (resistor, capacitor, inductor) components and complex arbitrary functions is supported. WinFIT can also transform the data from the frequency domain into the time domain. This is performed either by numerical Fourier transformation of the measured data or by using the Debye relaxation time distribution of the fit functions in the frequency domain.

## Conclusion

The Concept 60 system has recently been developed by Novocontrol and is a top class turnkey solution for dielectric measurements of materials dependent on frequency, temperature, time, and DC-Bias. The system is particularly suited for scientific applications in polymer, ceramic, and semiconductor research. It can also be used in quality control applications. The Concept 60 is based on the Agilent 4291B, the Novocontrol Cryosystem, and the Novocontrol software packages WinDETA and WinFIT.

The system limits are:

frequency: 1 MHz to 1.8 GHz
temperature: -160°C to +400°C
dc-bias: -40 V to +40 V
time: 2 s to 107s

Moreover any multi-dimensional combination of these parameters is supported, which makes the Concept 60 superior for top class scientific and engineering applications.

Novocontrol Concept 60 System is a product of Novocontrol GmbH.

Please contact Novocontrol GmbH for any purchasing requests or technical/support questions.

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