THE KAZAN FEDERAL UNIVERSITY
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The Methodology of the Experiment on the Dielectric Spectrometer Novocontrol BDS Concept 80

Part 1. Dielectric Measurements from 10^{-5} Hz to $3 \cdot 10^{9}$ Hz on the Dielectric spectrometer Novocontrol BDS Concept 80

Study guide

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The study guide discusses the "Alpha-A High Resolution Dielectric, Conductivity and Impedance Measurement System", "Radio Frequency Extension for Electric Material Measurements" and "Quatro Cryosystem" that are required to perform the experiments from 10^{-5} Hz to $3\cdot10^{9}$ Hz on the dielectric spectrometer BDS Concept80 (Novocontrol Technologies GmbH & Co. KG). The aim of this work is to describe in detail the experimental procedure on this dielectric spectrometer: preparing the system for measurement, calibration procedure, different types of sample cells, performing a measurement, safety precautions.

This study guide will be useful for students, graduate students, postgraduate students as well as for the researchers in the field of the dielectric spectroscopy of complex systems.

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Introduction

Dielectric spectroscopy measures the dielectric permittivity as a function of frequency and temperature. It can be applied to all non-conducting materials. The frequency range extends over nearly 15 orders in magnitude: from μHz to GHz range. Dielectric spectroscopy is sensitive to dipolar species as well as localized charges in a material, it determines their strength, their kinetics and their interactions. Thus, dielectric spectroscopy is a powerful tool for the electrical characterisation of nonconducting or semiconducting materials in relation to their structure.

Dielectric and impedance spectroscopy measures electrical properties of a material as it is subjected to a periodic electrical field to characterise its molecular kinetics and transport properties. In order to measure as much as quantitative data Novocontrol's hard and software is covering a broad range both frequency and temperature.

Many key aspects of materials properties can be derived from the frequency and temperature dependent measured complex permittivity, conductivity, and impedance spectra. For example, time, strength and shape parameters of molecular relaxations, characterization of structural phase transitions over temperature like glass transition, activation energy, charge mobility and diffusion coefficients.

Time dependent measurements of chemical time variant materials provide rates of blending or cure, crystallization and chemical reactions.

Dielectric and impedance spectroscopy are valuable characterization tools for ceramics, glasses, porous materials, polymers, liquid crystals, semiconductors, ionic conductors, batteries, fuel cells, corrosion analysis, biomedical and biological systems, etc.

This study guide assumes that readers are familiar with the basic concepts of impedance measurements and dielectric spectroscopy. You may get further information about these topics from [1-5].

Alpha-A High Resolution Dielectric, Conductivity and Impedance Measurement System

1. Introduction

The Alpha Analyzer measures the complex dielectric, conductivity and impedance function of materials as function of frequency with high precision. It is especially optimized for dielectric materials with high impedance and low loss factors over a broad frequency range, but can accurately measure high conductive material with low impedance too. In addition, if it used as a frequency response analyzer in gain phase mode high precision measurement of two AC voltages and their phase relation is supported.

The Alpha analyzer is realized as a modular measurement system with one base unit called the Alpha analyzer mainframe and several test interfaces which can be connected to the mainframe (see Fig. 1). Usually the mainframe is supplied with at least one additional test interface. Additional test interfaces may be added later to an existing mainframe.

The Alpha Analyzer Mainframe provides the basic functionality to operate all test interfaces. The mainframe contains a frequency response analyzer with a sine wave and DC bias generator and two AC voltage input channels (see Fig. 1).

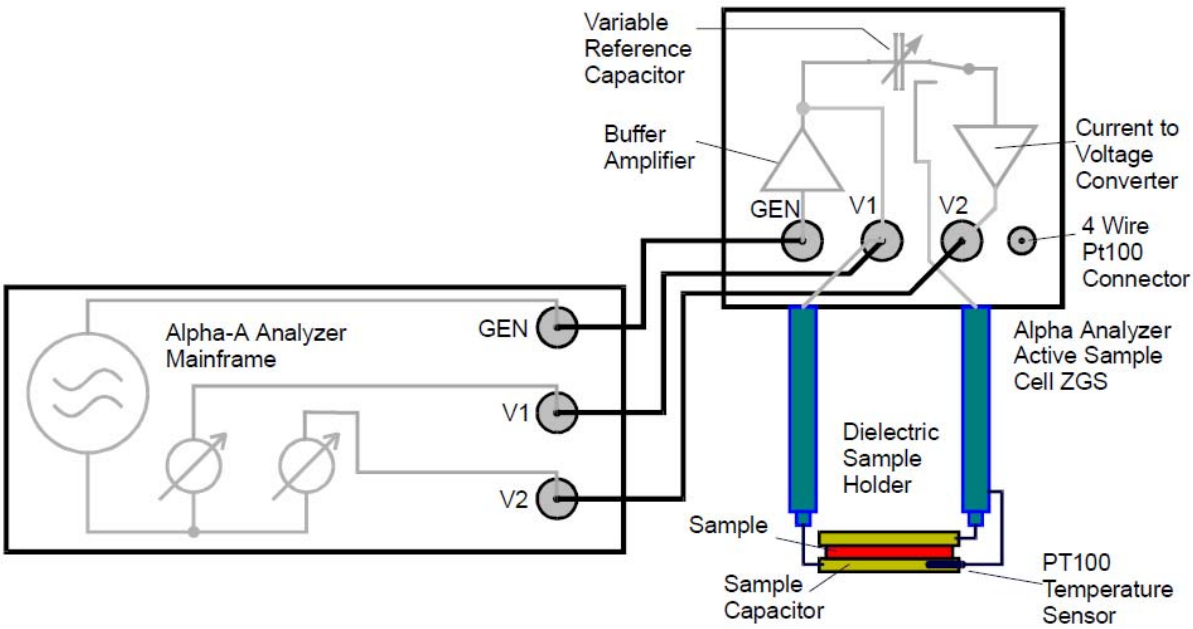


Figure 1. Setup of an electric material measurement with the active sample cell interface.

Each input channel measures the AC voltage amplitude of an applied sine wave. In addition, the phase shift between the sine waves applied to the both inputs is detected. More detailed, each channel measures the amplitude and phase angle of the harmonic base wave component of a signal applied to the input. The harmonic base wave component is measured at the frequency of the AC sine wave generator. Most other signal components are suppressed. In addition, higher harmonics may be measured [6].

Further, the mainframe contains the power supplies, analog and digital control lines and the firmware for operation of the test interfaces. The following test interfaces can be combined with the mainframe.

Test interfaces for dielectric, conductivity, impedance and gain phase measurements

Active Sample Cell ZGS includes a sample cell with two parallel electrode configuration for dielectric, conductivity and impedance measurements on materials (see Fig. 1). The range and accuracy specifications apply at the electrodes, cable and cell effects are minimized. The active cell design provides best accuracy and ranges for two electrode material measurements in terms at low loss, high frequency and high impedance performance. The cell can be temperature controlled by the Novocontrol temperature control systems between -160 ÷ 400 °C.

Ranges: Frequency 3 μHz ÷ 20 MHz, Impedance 0.01 ÷ 10^{14} Ω, Capacity 1 fF (0.001 pF) ÷ 1F, loss factor tan(δ) 10^{-5} ÷ 10^{4}, AC voltage 100 μV ÷ 3V, DC bias -40 ÷ +40V, 70 mA.

Accuracy: Relative impedance or capacity, absolute loss factor tan(δ) < $3*10^{-5}$, absolute phase angle < 2 m°.

Resolution: loss factor tan(δ), impedance, capacity < 10", phase angle < 0.6 m°.

2. Principle of Dielectric, Conductivity and Impedance Measurement

The principle of an electric material measurement is shown in Figure 2 [6].

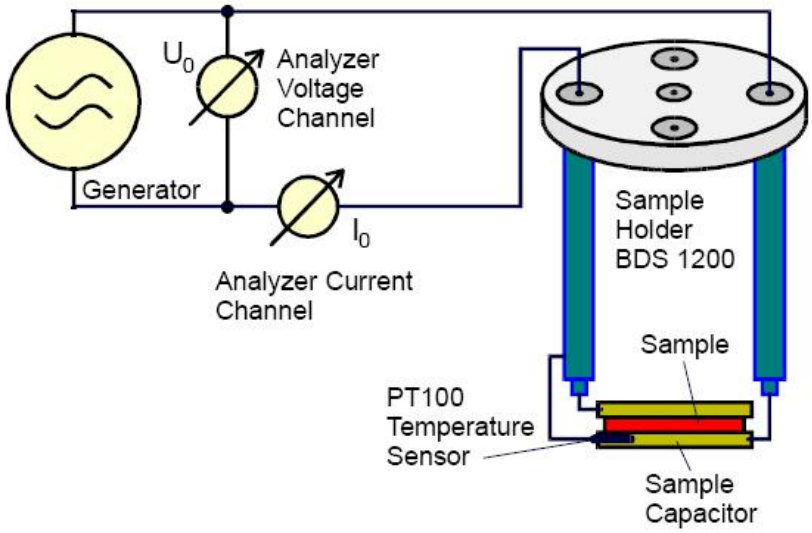


Figure 2. Principle of a dielectric or impedance measurement.

The sample material is usually mounted in a sample cell between two electrodes forming a sample capacitor. For details, refer to the "Sample Preparation and Measurement Accuracy" chapter. A voltage U_0 with a fixed frequency $\omega/2\pi$ is applied to the sample capacitor. U_0 causes a current I_0 at the same frequency inside the sample. In addition, there will be a phase shift between current and voltage described by the phase angle φ (see Fig. 3).

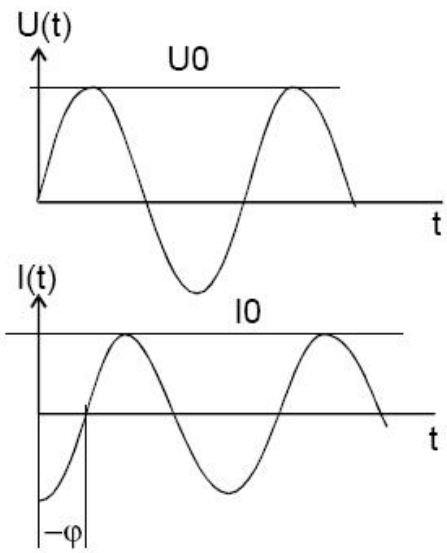


Figure 3. Amplitude and phase relations between voltage and current of a sample capacitor.

The ratio between U_0 and I_0 and the phase angle φ are determined by the sample material electric properties (permittivity and conductivity) and the sample geometry. For easy calculation and representation of the formulas [6], it is convenient to use complex notation.

$$U(t) = U_0 \cos(\omega t) = \mathrm{Re}(U^* \exp(i\omega t))$$
$$I(t) = I_0 \cos(\omega t + \varphi) = \mathrm{Re}(I^* \exp(i\omega t))$$

with $$U^* = U_0$$

and $$I^* = I' + iI''; I_0 = \sqrt{I'^2 + I''^2}; \tan\varphi = \frac{I''}{I'}.$$

For a sample with linear electromagnetic response, the measured impedance of the sample capacitor

$$Z^* = Z' + iZ'' = \frac{U^*}{I^*} \tag{1}$$

is connected with the dielectric function of the sample material by

$$\varepsilon^*(\omega) = \varepsilon' - i\varepsilon'' = \frac{-i}{\omega Z^*(\omega)} \frac{1}{C_0} \tag{2}$$

where C_0 is the capacity of the empty sample capacitor.

The specific conductivity σ^* is related to the dielectric function by

$$\sigma^* = \sigma' - i\sigma'' = i2\pi f \varepsilon_0 (\varepsilon^* - 1).$$

3. Principle of Gain Phase Measurement

As described in the previous chapter, in impedance mode the response voltage and current of a sample to an applied sine wave signal are measured. In gain phase mode, a second voltage is measured instead of the current. This allows to measure two response voltages of an applied sine wave driver signal at two arbitrary points of a system under test (SUT). The two voltages are measured with the Alpha frequency response analyzer (FRA) channels CH1 and CH2. The applied sine wave is created by the Alpha sine wave generator [6].

A typical application would be to measure the transfer function of an amplifier or

transformer. In this case, the Alpha generator output and input CH1 have to be connected to amplifier input and the Alpha input CH2 to the amplifier output. The amplifier complex transfer function defined by Vout/Vin is determined by measuring the voltages at CH2 and CH1 and dividing them.

It should be noted, that in gain phase mode the Alpha can be used as a digital lock in amplifier with two input channels, too. The main function of a lock in amplifier is to apply a signal with a defined frequency to a system under test and to measure a response signal. As a further lock in amplifier feature, only the frequency component of the applied signal in the response signal is detected. As usually most of the response signals noise and DC errors are at other frequencies, they will therefore be suppressed.

4. Special Alpha Analyzer Features

If the setup is used for measuring electric material properties, the following problems may arise.

Wide Impedance Range

Most dielectric samples have low conductivity ($\varepsilon' << \varepsilon''$) and behave in a first order approximation as a capacitor. Therefore, the impedance Z of these samples at low frequencies generally becomes very high (e.g. 10^{14} Ω at 0.01 Hz). On the other hand, as the impedance of a capacitor decreases with 1/frequency, the sample impedance is generally low at the high frequency end (e.g. 10 Ω at 10 MHz). In addition, if the sample has conductivity, it may have low impedance (e.g. 1 Ω) over the entire frequency range.

This feature is implemented in the Alpha test interfaces for impedance measurement which can measure impedance from 0.01 Ω to 10^{14} Ω allowing to measure dielectric samples down to lowest frequencies in the μHz range.

The wide impedance range results in an exceptional capacity range too, which is from 1 fF (0.001 pF) to 1 F. This is in contrast to (so called) general impedance analyzers typically covering an impedance range from 0.01 Ω to 10^8 Ω. For these analyzers, the high impedance limit (10^8 Ω) confines the measurement of dielectric samples to frequencies > 10 Hz typically.

Low Loss Capability

Many dielectric samples have very low loss factors $\tan(\delta) = \varepsilon''/\varepsilon'$ down to 10^{-4} or even 10^{-5}. $\tan(\delta)$ is determined by the phase angle φ of the measured voltage to the measured current (see Fig. 3). Therefore, if $\tan(\delta)$ is very small, the absolute value of the phase angle φ has to be measured with very high precision (e.g. for $\tan(\delta) = 10^{-5}$ δ and $\varphi=90°-\delta$ have to be measured with a absolute precision of 0.001°).

This can be done by most of the Alpha test interfaces for impedance measurement due to the following two features [6]. First, the Alpha mainframe frequency response analyzer part works with a digitally design optimized for high phase and amplitude resolution. Second, remaining phase deviations caused by analog components in the signal path like amplifiers or the current to voltage converter are cancelled out by a special reference technique used in the test interfaces for impedance measurements. This is in contrast to general impedance analyzers which have a phase angle accuracy of 0.1° ÷ 1° corresponding to a $\tan(\delta)$ limit of $10^{-3} \div 10^{-2}$ typically.

Cable and Cell Effects Minimised, Accuracy Specified at the Electrodes or Close to the Sample

For frequencies > 1 MHz, the sample impedance may become in the same order as the inductive impedance of the BNC cables connecting the sample holder with the analyzer. This limits the measurement to frequencies below approximately 3 MHz. For high impedance samples, long cables cause additional problems due to electrical noise created by mechanical vibrations. Therefore, the cable length should be kept as short as possible for optimum high frequency and high impedance performance.

This is realized for the active sample cell ZGS as shown in Figure 1. The sensitive part of the input amplifiers is directly incorporated on top of the dielectric sample cell or in the active interface. The connection to the sample cell is done which customized air insulated lines optimized both for high frequency performance and isolation at high impedance. This setup can measure electric material samples (with not too low loss factors) up to 20 MHz. As the interface is separated from the mainframe it can be located close to the sample and sample connections cables can be kept short [6].

5. Test Interfaces for Dielectric, Conductivity and Impedance Measurements

5.1. Active Sample Cell ZGS

The active sample cell ZGS is optimized for measurements of dielectric, conductivity and impedance of materials. It includes a sample cell in which the sample material is mounted between two parallel plate electrodes. The cell includes all components which are crucial for wide band, wide impedance range and high accurate measurements like output-, input amplifiers, reference capacitors, connection lines, electrode mounting and insulation. The range and accuracy specifications apply therefore at the electrodes. Due to the active cell design with the critical electronic components in the cell head, cable and cell effects can be minimised.

The active cell design provides best accuracy and ranges for two electrode material measurements in terms at low loss, high frequency and high impedance performance. The cell can be controlled by on the Novocontrol temperature control systems between -160 ÷ 400 °C.

5.1.1. Instrument Description

The active sample cell ZGS contains the following elements (see Fig. 4) [6]:

Alpha Mainframe Connector. It should be connected to the Alpha Active Sample Cell Connector on the Alpha mainframe rear, providing power and control lines for the active sample cell. **Caution**: Do not connect any other devices to this terminal. Connecting other devices (like serial or parallel ports) may result the damage of devices. Use only the original cable supplied by Novocontrol.

GEN Terminal. This is the input socket for the active sample cell voltage buffer amplifier. It should be connected to the GEN socket of the Alpha mainframe. This input should not be connected to other terminals. Maximum voltage limit: +- 3 Vrms.

V1 Terminal. In impedance mode, it should be connected to the V1 socket of the Alpha mainframe for sensing the voltage applied to the sample by the Upper Electrode. V1 is internally connected to the voltage signal generator (buffer amplifier) output and the Lower Electrode. V1 should used in gain phase mode as the driver signal for the sample system under test. This output is short protected to system ground. Do not apply any external voltages or currents.

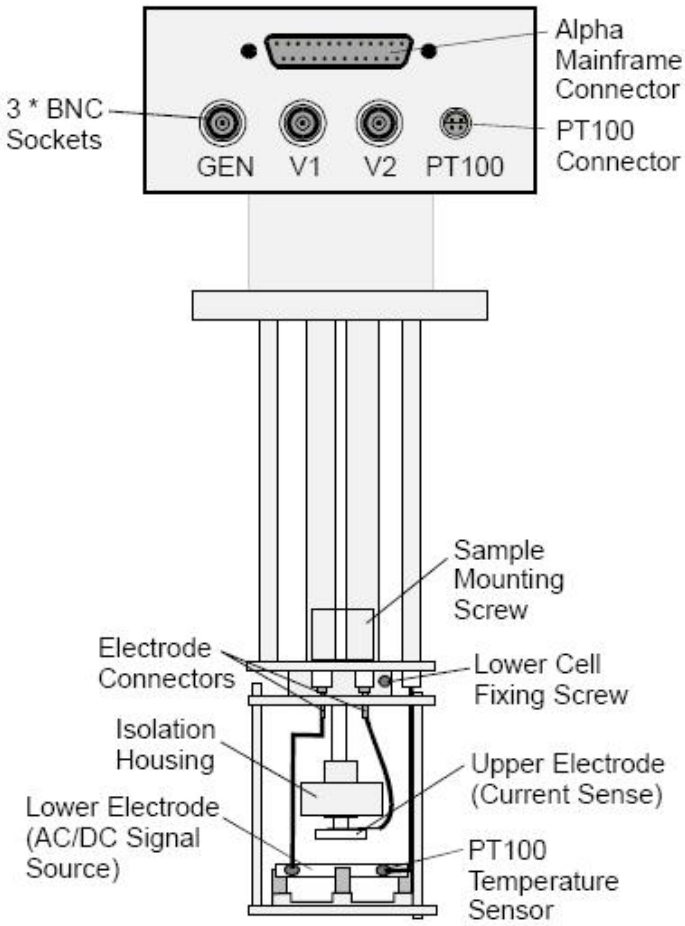


Figure 4. Active Sample Cell ZGS.

V2 Terminal. Provides the voltage output of the cell internal current to voltage converter. For dielectric or impedance measurements it should be connected to the V2 socket of the Alpha mainframe. This output is short protected to system ground. Do not apply any external voltages or currents.

PT100 Connector. This four wire connector can be used for sensing the sample temperature. This is done with a PT100 temperature sensor mounted in the Lower Electrode plate. The PT100 is connected as shown in Figure 5.

Upper Electrode. The sample should be mounted between the Upper and Lower Electrode. For details of sample preparation and mounting refer to the "Sample Preparation and Measurement Accuracy" chapter. The Upper Electrode senses the sample current. Current limits: Protected to +-200 mA. DC Voltages up to +-50 V may be applied as long the current limits are not exceeded.

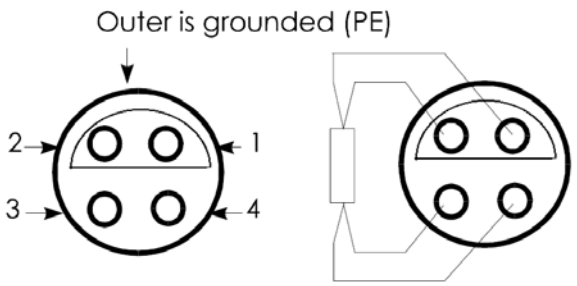


Figure 5. PT100 temperature connector of the active sample cell.

Lower Electrode. Applies the signal generator output voltage to the sample material. The Lower Electrode is internally connected to the V1 output of the active sample cell. The output voltage is 0 V ÷ 3 V AC superimposed by optional +-40 V DC (bias). The output impedance is 50 Ω. The maximum DC current bias is limited to about 70 mA. This output is short protected to system ground. Do not apply any external voltages or currents.

The active sample cell lower part containing the electrodes can be separated as shown in Figure 6. This enables easier handling during the sample preparation and allows the active head to be used with various lower samples cells.

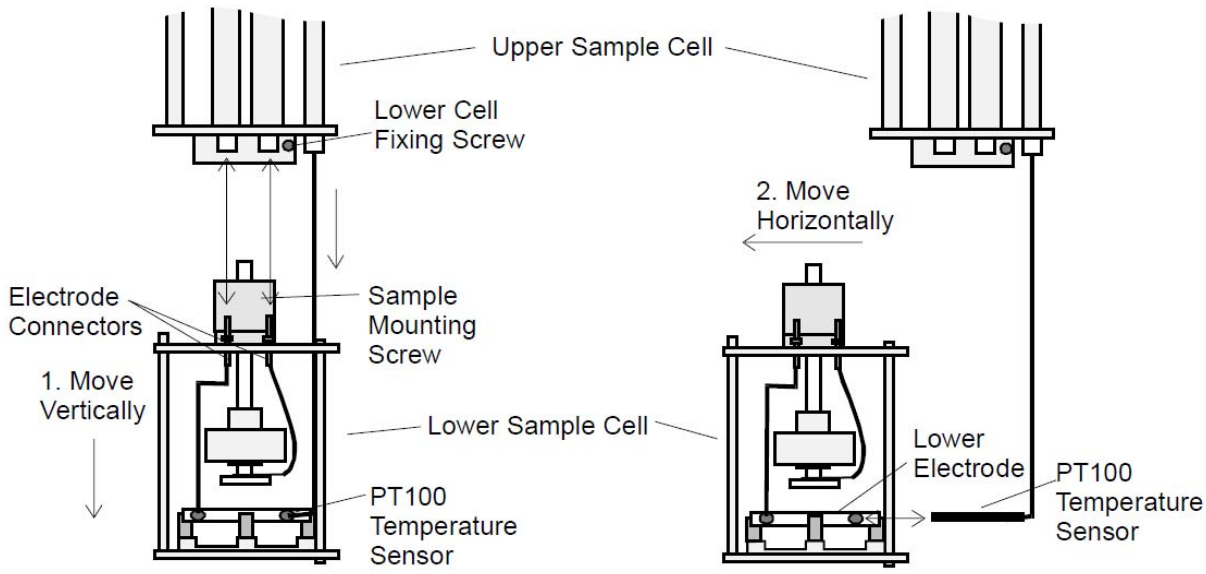


Figure 6. Procedure of separating the lower sample cell from the upper cell.

To separate the Lower Sample Cell from the Upper Sample Cell:

• Loosen the Lower Cell Fixing Screw.

• In a first step, slowly move the lower sample cell vertically to the upper sample cell as shown in Figure 6.

While moving stepwise, pull the both Electrode Connectors out of the connection lines. The PT100 Sensor remains in the lower electrode at this time and shifts itself out of the upper sample cell. The PT100 can be shifted vertically by about 5 cm to the upper sample cell, but can not be disconnected from the upper sample cell. **Warning**: Do not pull too strong. Otherwise the PT100 connections may be damaged.

• In a second step move the lower sample cell horizontally to pull the PT100 sensor out of the Lower Electrode.

To measure a sample, it must be build into the sample capacitor. For this purpose, the sample should not directly be placed between the electrodes of the active sample cell. Instead, it should be placed between two disposable electrodes as shown in Figure 7. Disposable electrodes are available from Novocontrol in various diameters [6].

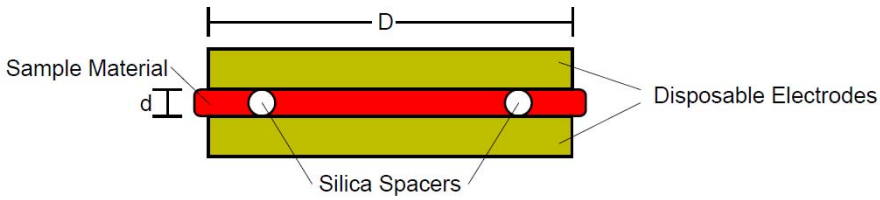


Figure 7. Sample capacitor prepared with two round disposable plate electrodes.

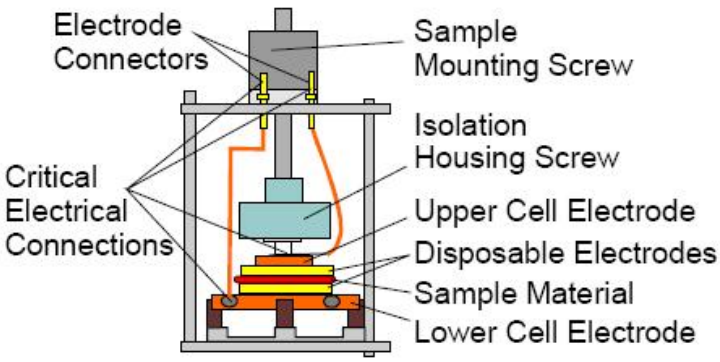


Figure 8. Lower sample cell part with sample mounted between disposable electrodes.

The two electrodes with the sample between can be seen as a separate sample capacitor which can be placed between the active sample cell electrodes as shown in Figure 8 [6]. This procedure simplifies the sample preparation, protects the electrodes from contamination and allows to remove the sample easily.

Important

- Use the Sample Mounting Screw to fix the Disposable Electrodes by the Upper Cell Electrode.
- Do not use the Insulation Housing Screw to fix the sample. This may result in improper contact and damage of the upper electrode isolation.
- Before starting a measurement check the Critical Electrical Connections for proper contact.
- Tighten to the Upper Cell Electrode by rotating it clockwise against the Isolation Housing Screw until a fix connection is established.
- Tighten the screw of the Lower Cell Electrode connection wire with screw driver until a fix connection is established.
- Check the two Electrode Connectors for proper contact with the upper sample cell connection lines.
- From time to time, all four Critical Electrical Connections contacts should be cleaned and polished to remove insulating oxide layers. This applies especially if the cell was exposed to high temperatures before.

For this purpose:

- remove the Upper Cell Electrode by rotating it counter-clockwise,
- remove the Lower Cell Electrode connection wire by unscrewing it from the Lower Cell Electrode,
- pull the two electrode connectors out of the upper sample cell connections lines.

5.1.2. System Setup for Impedance Mode

In order to setup the Active Sample Cell ZGS for the measurement of dielectric, conductivity or impedance, the following connections have to be established (see Fig. 9) [6]. **Caution**: Before setup any connections switch off the Alpha mainframe and all devices connected to it.

Note: For proper operation, the active sample cell has to be shielded. This is generally done by placing it into a metal cryostat or a metal shielding tube.

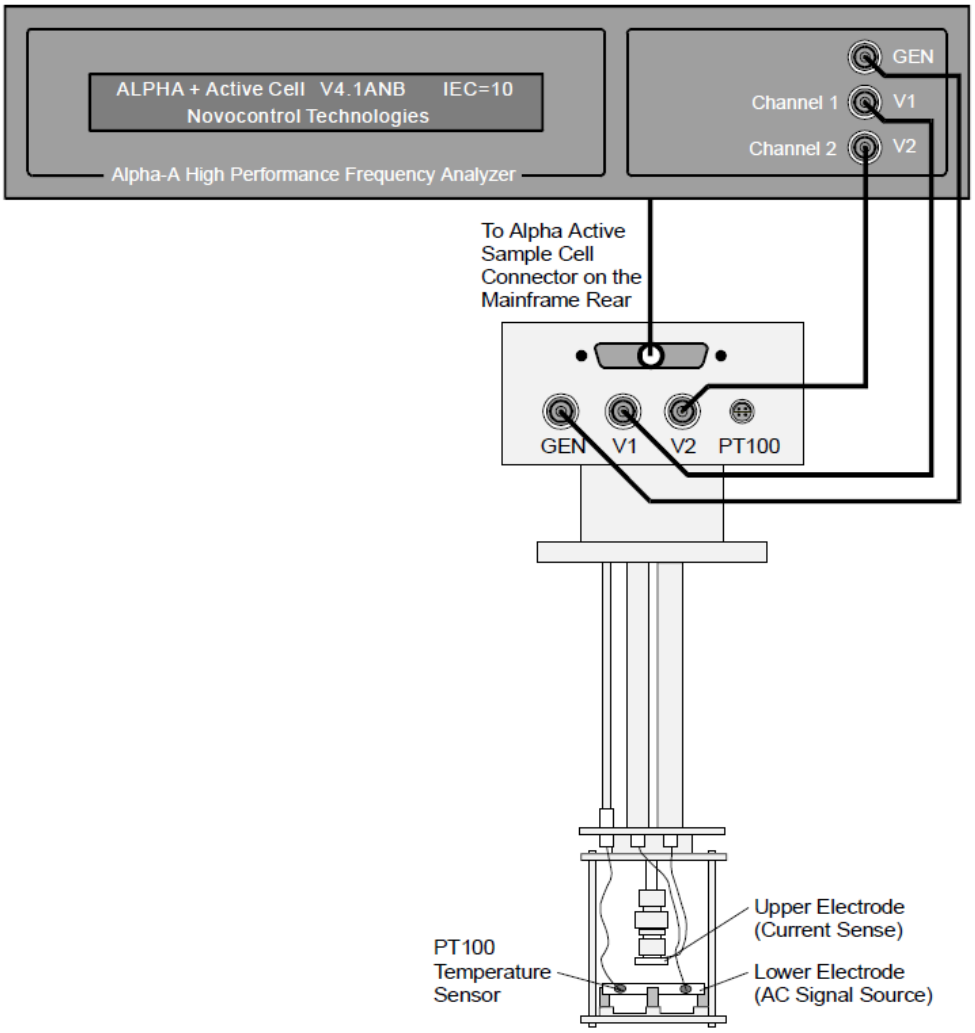


Figure 9. Connections of the Alpha mainframe with the active sample cell ZGS in impedance mode.

6. Sample Preparation and Measurement Accuracy

This paragraph gives some basic hints for sample preparation and sources of inaccuracy in electric material measurements.

6.1. Optimal Sample Capacity

For optimal measurement results, the sample geometry should be chosen according the following rules [6]:

Best performance will be reached with sample capacities **from 50 pF to 200 pF (optimum around 100 pF)** in the frequency range 100 kHz ÷ 10 MHz. At lower frequencies, higher capacities up to 2 nF will give good results, too. **Note**: For most test interfaces, the Alpha

will be able to measure also capacities in a wide range from 0.001 pF up to 1F depending on frequency, but the accuracy decreases outside the capacity ranges specified above.

If nevertheless, small capacities < 50 pF have to be measured, the stray capacity of the active sample cell ZGS which is about 1 pF has to be taken into account. The stray capacity is caused by non ideal shielding of the electrode connection wires. It has to be subtracted from the measured data. If WinDETA software is used [7], the stray capacity should be entered in the Cell Stray + Spacer Capacity field of the Sample Specification dialogue.

If the dielectric constant ε' of the sample is approximately known, the sample capacity can be obtained from

$$C = \varepsilon' \varepsilon_0 \frac{\pi D^2}{4d} \; or \; \frac{C}{pF} \approx 6{,}95 \cdot \varepsilon' \frac{(D/mm)^2}{(d/\mu m)}$$

where D is the diameter of the capacitor electrodes and d is the distance between them.

Electrode Stray Capacity

In order to minimise the errors due to edge effects at the sample capacitor borders, the ratio d/D should be kept as small as possible. Choose large diameters and small thickness. If WinDETA software is used, the edge effects are considered. Refer to Edge Compensation and Sample Specification in the WinDETA manual [7] for more information on edge effects.

6.2. Sample Surface and Contacts

The surface of solid samples should be as flat as possible to make good contact with the external electrodes. In addition it is highly recommended to metallize the sample surface in the contact regions. This can be done by evaporation or sputtering a metal film on the sample. Another easier and cheaper way is to cover the sample surface with conductive liquid silver paint. In this case one has to make sure, that the sample material is not damaged by the solvent of the liquid silver paint.

6.3. Measurement of Liquids

If liquid samples have to be measured, additional spacers can be used in order to adjust the electrode spacing as shown in Figure 10. The spacer material should have a low and frequency independent dielectric constant and low losses. Convenient spacer materials are thin

silica needles (available from Novocontrol) or small stripes of Teflon or Polyethylene. The contribution of the spacers to the measurement may be considered. Refer to Sample Specification and Evaluation of Data in the WinDETA manual [7] for details on spacer compensation. For liquids with ion conductivity, the low frequency side of the spectra may be affected by electrode polarization as described in item **B5** below.

6.4. Measurement of Liquids with the Novocontrol Vacuum Sealed Sample Cell BDS 1308

For some low viscosity liquids with low partial pressure, sample material may evaporate out of the open sandwich capacitor especially at higher temperatures and the measurement accuracy may decrease. In order to prevent evaporation, the Novocontrol liquid sample cell BDS 1308 can be used (see Fig. 10) [6].

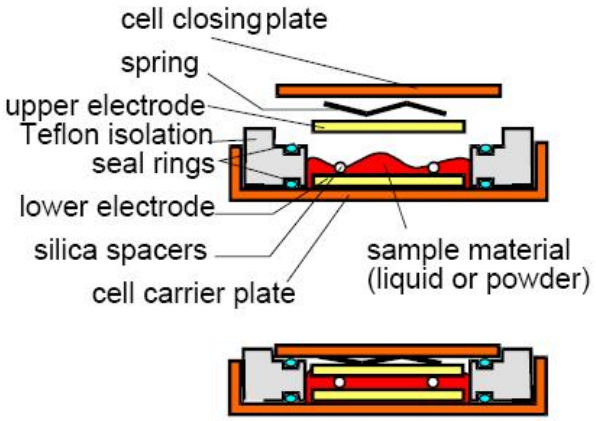


Figure 10. Novocontrol liquid sample cell BDS 1308.

In the open state, the upper electrode is removed from the cell, and the sample material covers the lower electrode mounted at the cell bottom. In the closed state, liquid sample material which does not fit between the electrodes can flow around the upper electrode. The distance between the electrodes is adjusted by separate spacers made of Silica rods or Teflon rings, stripes or squares. The upper electrode is pressed by a spring and the cell closing plate to the spacers. The two seal rings attached to the Teflon isolation prevent evaporation of sample material out of the cell.

The BDS 1308 is mounted in the same way as the standard sandwich capacitor between the electrodes of the active sample cell ZGS or the Novocontrol BDS 1200 sample cell.

Important: For the BDS 1308 cell an additional stray capacity of about 5.5 pF from the Teflon isolation between the cell closing plate and the cell carrier plate has to be taken into account. If WinDETA is used, the capacity has to be entered in the WinDETA Cell Stray + Spacer Capacity field of the Sample Specification dialogue.

In addition it should be considered, that the cell Teflon isolation and the spacers may create additional losses which can be modelled by an additional resistor in parallel to the sample electrodes. Especially for high insulating low loss liquids, this may create additional non intrinsic losses in the measured results.

The isolation parallel resistance value may be determined from the value of the parallel impedance Zp1 of an empty cell measurement. This value can be compared with the Zp1 of the measured liquid. The measured losses are only intrinsic if Zp'(empty cell) >> Zp'(cell with liquid). In contrast to the stray capacity, Zp'(empty cell) will be strongly temperature-dependent. In addition Zp'(empty cell) strongly decreases if the cell isolation or spacers are not properly cleaned or contaminated.

6.5. Mounting of External Electrodes in the active sample cell ZGS

Finally, the sample capacitor (or liquid sample cell) should be mounted between the upper and lower electrodes of the sample cell as shown in Figure 8 [6].

6.6. Typical Sources of Errors

Accuracy of the Measured Impedance of the Sample Capacitor

This includes the accuracy of the Alpha impedance measurement and the influences of cables and sample holders. This accuracy depends on the used test interface type, frequency of measurement and the actual sample impedance.

The actual sample impedance can be adjusted by selecting a proper sample geometry according to the rules described at the beginning of this paragraph. This ensures the sample impedance to be in a good Alpha accuracy range.

Accuracy in the Dielectric Function Evaluation to Preparation of the Sample Capacitor

It means that the Alpha measures the correct complex capacity C_s^* of the sample capacitor, but the used equation [6]

$$C_s^* = \varepsilon^* \varepsilon_0 A / d \tag{3}$$

does not hold (ε* - complex sample permittivity, ε_0 - vacuum permittivity, A - sample area, d - sample thickness).

<u>The following sources of error are the most important ones:</u>

B1. Stray- and Edge Capacities

These are additional capacities in parallel to the capacity of the sample capacitor. They add to the real part of the measured capacity Cp' and permittivity eps'. Stray capacities are caused by non ideal isolation from the sample cell and optional spacers between the electrodes.

B2. Edge Capacities

Equation (3) presumes that the electrical field between the sample capacitor plates is homogenous between the plates and zero outside. This is true only for samples with infinite plates. For real samples, there will be always an inhomogeneous field at the sample borders and in addition the field outside the sample capacitor will not be zero. This results in an additional edge capacity parallel to the sample capacity. Refer to Edge Compensation and Sample Specification in the WinDETA manual [7] for more information on edge effects.

B3. Uncertainties in the Sample Geometry

This is mostly due to not well known samples thickness. It should be noted, that for a sample with a thickness of 0.1 mm, the error in the thickness has to be less than 1μm for a 1% accurate result. The thickness of most samples will not be homogenous within this range. In addition, if the sample is mounted between the electrodes of the sample cell, pressure will be applied to the sample which may reduce the sample thickness.

B4. Bad Electrical Contact between Sample Material and Electrodes

As dielectric materials are most often high insulating, the contacts to the electrode are very often the dominant source of error. Bad contacts are often caused by not flat sample surfaces. In this case only a part of the sample material will be in contact with the electrodes. In addition, bad contacts may be caused by microscopic layers covering either the metal electrodes or the sample material (e.g. oxide layers). Bad contacts generally reduce the measured ε values and or introduce additional losses (tan(δ) increases).

The contact problem can be solved in most cases if a metal film (e.g. gold) is evaporated on both sides of the sample surface. At least the sample should be coated with liquid silver. In this case one has to make sure that the sample material is not damaged by the liquid silver paint solvent. The measured results may depend on the metal used for evaporation, especially in the case of electrode polarisation. Sample metalization is especially important if materials with high dielectric functions are measured [6].

B5. Electrode Polarisation

For samples with ionic conductivity, free ions in the sample material can migrate through the sample material. If they reach the border to the electrode, the ions often can not enter the metal electrode. In this case the ions will accumulate an additional charge layer at the electrodes. This leads to a strong increase of the real part permittivity ε' to values of $100 \div 10^7$ typically. In addition, the real part conductivity σ' decreases at low frequencies. Similar effects may be observed if insulating layers due to contact problems exist at the electrode borders. The following procedures may be applied to keep the electrode polarization as low as possible.

- **Usage of a four wire test interface (ZG4 or POT/GAL) in four wire mode with a four wire liquid sample cell**

In addition to the two electrodes which apply the electrical signal (connected to I high) and measure the sample current (connected to I low) the sample cell should have two further electrodes (connected to V high and V low) to sense the potential in the liquid. These electrodes should be positioned in volume fraction containing no polarisation charge. The measured voltage between the V high and V low electrodes will not be affected by electrode polarisation in this case and the system will measure the true liquid intrinsic impedance. This technique is limited by input impedance of V high and V low voltage channels [6].

- **Wide Electrode Spacing**

The spacing (sample thickness) between the electrodes should be selected as wide as possible. This is because the thickness of the charge layers due to electrode polarization is independent from the sample thickness and the ratio of charge layer impedance to the sample impedance improves for thick samples.

- **Clean Liquids and Electrodes**

As the formation of the polarization charge layers directly depends on the ion concentration, the amount of ions has to be kept as low as possible. Any impurities either from the preparation procedures, electrodes or the sample cell may drastically increase the ion content and shift the onset of electrode polarization to higher frequencies. Therefore all volumes and surfaces which may have contact with the sample liquid should be kept as clean as possible. If possible, the entire sample preparation should be done under vacuum.

- **Data Subtraction from two Measurements**

The electrode polarization can be partly corrected, if the same liquid is measured once with an electrode spacing *d* and for a second time with the double spacing 2*d*. In first order approximation, the measured impedance Zm1 of the first measurement is the sum of the charge layer impedance Zc and the intrinsic sample impedance Zs [6]

$$Zm1 = Zc + Zs + Zc.$$

For the second measurement, the sample impedance doubles due to the doubled spacing, but the charge layer impedance remains the same:

$$Zm2 = Zc + 2*Zs + Zc.$$

If the results of the two measurements are subtracted, the charge impedance cancels out

$$Zm2 - Zm1 = Zs.$$

It should be emphasised, that this will work only approximately.

7. Cylindrical Liquid Sample Cell BDS 1307

7.1. General

The three electrode cylindrical sample cell avoids the errors related to the thermal expansion of a measured liquid, protects against sample leakage and prevents evaporation (see Fig. 11) [8]. It also increases the accuracy of the measurement by decreasing the influence of fringing fields. The sample cell is mounted between the parallel plate electrodes of the NOVOCONTROL sample cell for dielectric measurements.

7.2. Schematic View of the Liquid Sample Cell

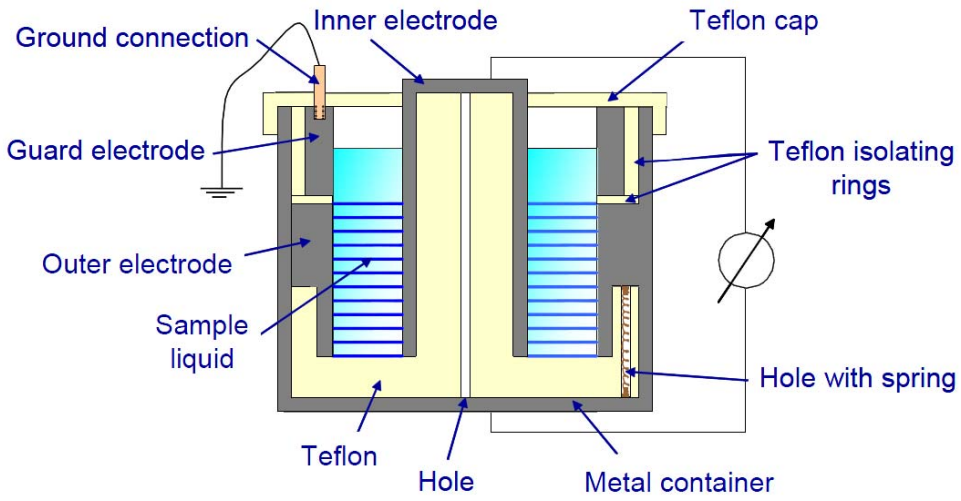


Figure 11. The schematic of the assembled sample cell. The sample is represented by the blue shading and the blue lines represent the effective measuring field.

In Figure 11 grey indicates metal parts and yellow indicates the Teflon isolations. Before each measurement all parts (see Fig. 12) should be cleaned and assembled (see Fig. 13).

Figure 12. The components of the cell.

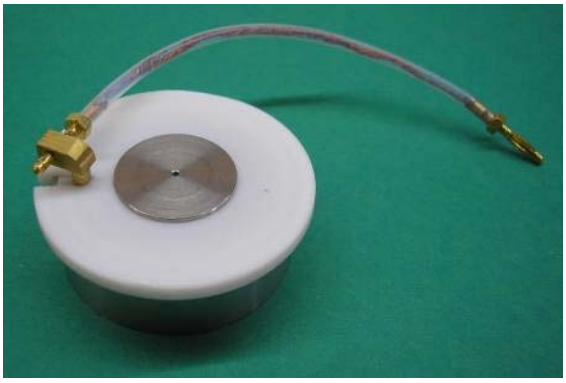


Figure 13. The assembled sample cell.

The big Teflon part should be placed inside the metal container. A special small hole in the Teflon allows air to escape from the empty volume smoothly without any warping during the installation.

The inner electrode looks like a cap and covers the central column of the Teflon part in order to reduce any stray capacitance between the inner electrode and the metal container. In the middle of the inner cap electrode there is a small hole for easier reassembling of the sample cell. **Note**: the best way to dissemble the cell is to use compressed air via this hole.

The outer electrode and the metal container are connected by a spring to maintain a good electric contact through a wide temperature interval.

The outer electrode and the guard electrode are isolated one from another by a thin Teflon isolating ring. The guard electrode is isolated from the metal container by means of an additional Teflon isolating ring and grounded using the gold plated un-insulated plug connected to another plug via connecting wire covered by Teflon. A special device is mounted on the original NOVOCONTROL sample cell for inserting the plug, thus completing the ground for the liquid cell.

A measured liquid should fill up the sample cell to approximately the middle of the guard electrode, depending on a thermal expansion of the liquid. An effective measuring region is marked by blue lines in the schematic drawing (see Fig. 11). The Teflon top prevents pollution of the liquid sample from the environment. The assembled sample cell is presented in Figure 13.

Then the liquid sample can be mounted into the standard sample cell of the NOVOCONTROL setup (see Fig. 14).

Figure 14. The liquid sample cell implemented into the standard NOVOCONTROL sample cell.

7.3. Calibration Procedure and Dielectric Measurements of Liquid Properties

The default cell parameters are [8]:

Empty cell capacity C_{0_real} = 1.0275 pF

Stray Capacity C_{add} = 1.65 pF.

These parameters are discussed in detail below. In order to use the cell with WinDETA the above default values correspond to the following setting in the WinDETA "Sample Specification" dialog of the "Measurement menu".

Sample Diameter	Sample Thickness	Cell Stray + Spacer Capacity	Spacer Area	Use Edge Compensation
20 mm	2.774 mm	1.65 pF	0	Off

The default parameters as stated above may slightly differ from cell to cell. If higher accuracy in the absolute values is required, the cell parameters should be established for each cell as described below. The geometric capacitance of the cylindrical liquid sample cell C_{0_geom} can be calculated using the formula [8]:

$$C_{0_geom} = \frac{2\pi\varepsilon_0 L}{\ln\frac{b}{a}},$$

where ε_0=8.85·10^{-12} F/m is the dielectric permittivity of vacuum, L is the height of the outer electrode, b and a are diameters of the outer and inner electrodes, correspondingly.

In order to carry out the dielectric measurements, the parameters of a plate capacitor of the standard NOVOCONTROL sample cell should be estimated to give the same value of C_{0_geom}. Fixing a diameter of the capacitor electrodes D equal to the diameter of the inner electrode, a thickness d can be calculated as [8]

$$d = \frac{D^2 \ln\frac{b}{a}}{8L}.$$

In order to improve the accuracy, we recommend calibrating the sample cell by using standard samples (see Table 1). Since C_{0_geom} can be smaller than the real value of the empty capacitance C_{0_real}, and also there is an additional capacity C_{add} created by the design of the sample cell. It can be subtracted from the measured capacity C'_p by the WinDETA program [7]. The C_{add} capacity is in a parallel with the sample capacity and it remains constant for any

measurements contributing to only real part of the complex permittivity. Therefore, a calibration procedure based on a differential method and it consists of test measurements as follows:

Pure water or any other standard pure liquid like ethanol, methanol, 1-propanol, etc. The regular frequency scans at relatively high frequencies (≥100 kHz) at specified temperature should be performed.

An open liquid cell without any liquid inside.

The measured capacity C'_p can be calculated as

$$C'_p = \varepsilon_{meas} C_{0_geom} = \varepsilon_{real} C_{0_real} + C_{add},$$

where ε_{meas} and ε_{real} are the static dielectric permittivity measured and the static dielectric permittivity of the tested liquid at this particular temperature point, correspondingly. Taking into account that $\varepsilon_{real_air} = 1$ one can obtain C_{add} in the form

$$C_{add} = \varepsilon_{meas_air} C_{0_geom} - C_{0_real}, \text{ where } C_{0_real} = \frac{\left(\varepsilon_{meas_liquid} - \varepsilon_{meas_air}\right) C_{0_geom}}{\varepsilon_{real_liquid} - 1}.$$

The calculated value of C_{add} should be placed under the "Cell Stray + Spacer Capacity [pF]" description in the Sample Specification window of the Novocontrol WinDETA software [7]. Furthermore, C_{0_real} should be used instead of C_{0_geom}. For this purpose, d can be calculated as

$$d = \varepsilon_0 \frac{\pi D^2}{4 C_{0_real}}$$

and used in WinDETA. The dielectric permittivity values of several pure liquids at 25°C is presented in Table 1 [8].

Table 1. Standard samples.

Liquid	**ε at 25 °C**	**Reference**
Water, H_2O	78.36	U. Kaatze, *J. Chem. Eng. Data*, **34**, 371 (1989).
Methanol, CH_4O	32.66	Marsh K.N., IUPAC Recommended Reference Materials for Realization of Physico-Chemical Properties, section permittivity, *Pure Applied Chemistry*, **53**, 10, 1847 (1981).
Ethanol, C_2H_6O	24.55	Dannhauser W. and Bane W., *J. Chem. Phys.*, **40**, 10, 3058 (1964).
1-Propanol, C_3H_8O	20.45	Evans D.F. and Thomas J., *J. Chem. Phys.*, **75**, 1714 (1971).

An example of the application of the liquid sample cell for the measurement of glycerol/water mixture (60 mol.% of glycerol) is presented in Figures 15 and 16 [8].

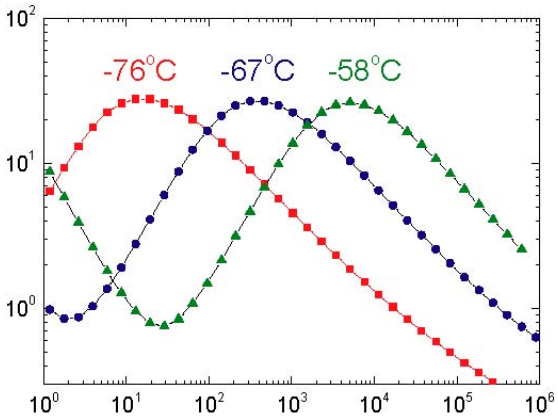


Figure 15. Three-dimensional plot of the frequency and temperature dependence of the dielectric losses for the glycerol/water mixture (60 mol.% of glycerol).

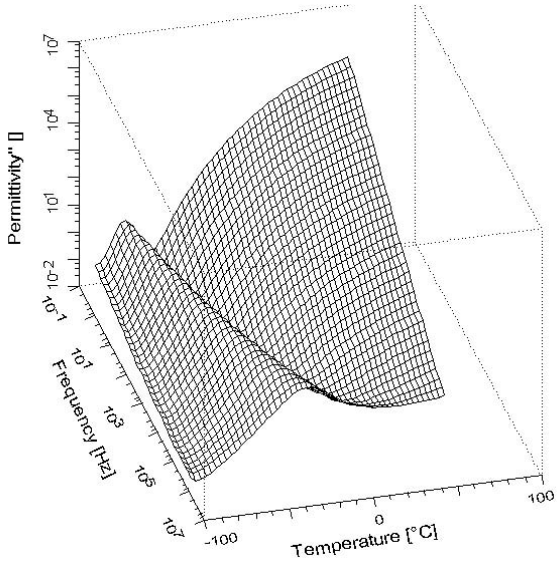


Figure 16. The frequency behaviour of the dielectric losses of the glycerol/water mixture (60 mol.% of glycerol) at different temperature points.

8. Calibrations

The following kinds of calibrations should be done for all test interfaces described in this study guide. All calibrations may be either started from the Novocontrol WinDETA software [7]. This means that each test interface has its own calibration data set. Performing a calibration for one test interface will improve result quality only for this test interface and will have no effect on other test interfaces connected later to the mainframe. If a test interface is connected to the mainframe, it automatically loads the proper test interface calibration data set. It is therefore not necessary to recalibrate the system anytime a test interface is changed.

8.1. Internal Interface All Calibration

The All Calibration includes the Reference Calibration described below. **Note**: Before starting a calibration, disconnect the sample or cables from test interface terminals according to the Table 2 [6].

Table 2. Test interface setups for the All and Reference Calibrations.

Test Interface	Action
Active Sample Cell ZGS	Remove the sample and leave the electrodes open with at least 3 mm spacing.
4-Wire Impedance Interface ZG4	Disconnect the cables from I high and I low terminals.
2-Wire Impedance Interface ZG2	Disconnect the cables from V out and I in terminals.

The calibration should be done in intervals of approximately 4 weeks to compensate for long term drift effects. **Note**: As low frequencies are involved, the calibration will run with the default settings for about 25 minutes.

8.2. Internal Interface Reference Calibration

This is a time saving version of the All Calibration. It calibrates the current-to-voltage converter internal reference capacitors relative to each other and additional internal components used in the reference measurement mode.

The calibration should be done from time to time. Highest accuracy will be reached, if the calibration is done immediately before a measurement. **Note**: Before starting a calibration, disconnect the sample or cables from test interface terminals as according to Table 2.

8.3. External Interface Low Impedance Load Short Calibration

Calibrates the current-to-voltage converter ranges of a test interface in impedance mode for sample impedance < 1 kΩ. In addition the properties of the sample connection lines and the sample cell are included. This calibration is only used for low impedance samples with impedance < 1 kΩ.

It should be done in intervals of approximately 4 weeks to compensate for long term drift effects. The calibration requires external 100 Ω resistor and a short calibration standards supplied with the test interface [6].

Important:

- The load short calibration measures very low impedance below 0.01 Ω. In 2- and 3- wire mode it is therefore required, that the contact resistance of the used cables and electrodes is reproducible within 0.01 Ω to a later measurement. It is recommended to check the calibration,

by measuring the 100 Ω standard and the short standard after the calibration and compare the results with the specified accuracy of the used test interface.

- After the calibration was done make sure that it is activated by the "Use Low Impedance Load Short Calibration" switch of the WinDETA dialog "Analyzers, Options Alpha, Configuration". Otherwise, the calibration data will be saved but not applied during measurements.
- Before performing a Load Short Calibration, an All Calibration is recommended.

8.3.1. Active Sample Cell ZGS

Only the 100 Ω normal with cylindrical shape is supplied. The cylinder should be mounted directly between the electrodes of the active sample cell. The short standard is realized by shorting the electrodes without a sample mounted between (see Fig. 17) [6].

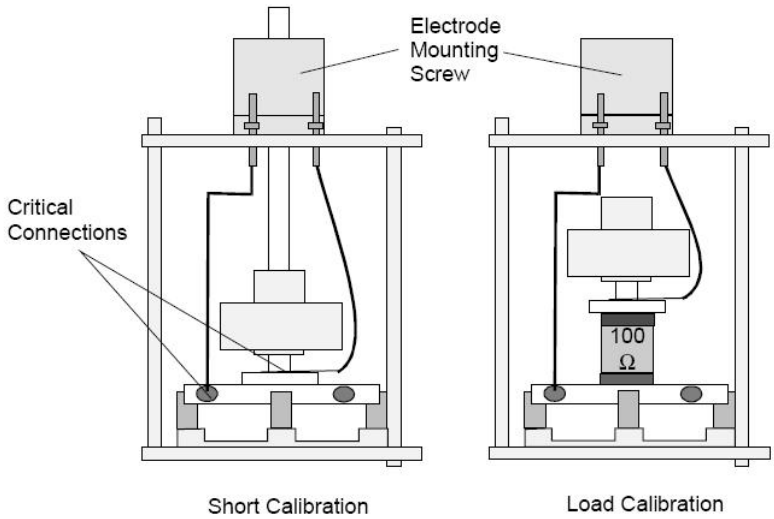


Figure 17. Short / Load calibration configurations for active sample cell ZGS.

Important: Both for the Load- and the Sort calibration it essential to have low contact resistance < 0.01 Ω between the electrodes and the 100 Ω standard, and between the shorted electrodes. For this purpose:

- make sure that the contact areas of the 100 Ω standard and the electrodes are clean and polished,
- make sure that the electrode wires are tight and properly mounted at the critical connections,
- prepare the 100 Ω standard and the shorted electrodes by tightening the electrode mounting screw with maximum finger torque.

8.3.2. Test Sample Results

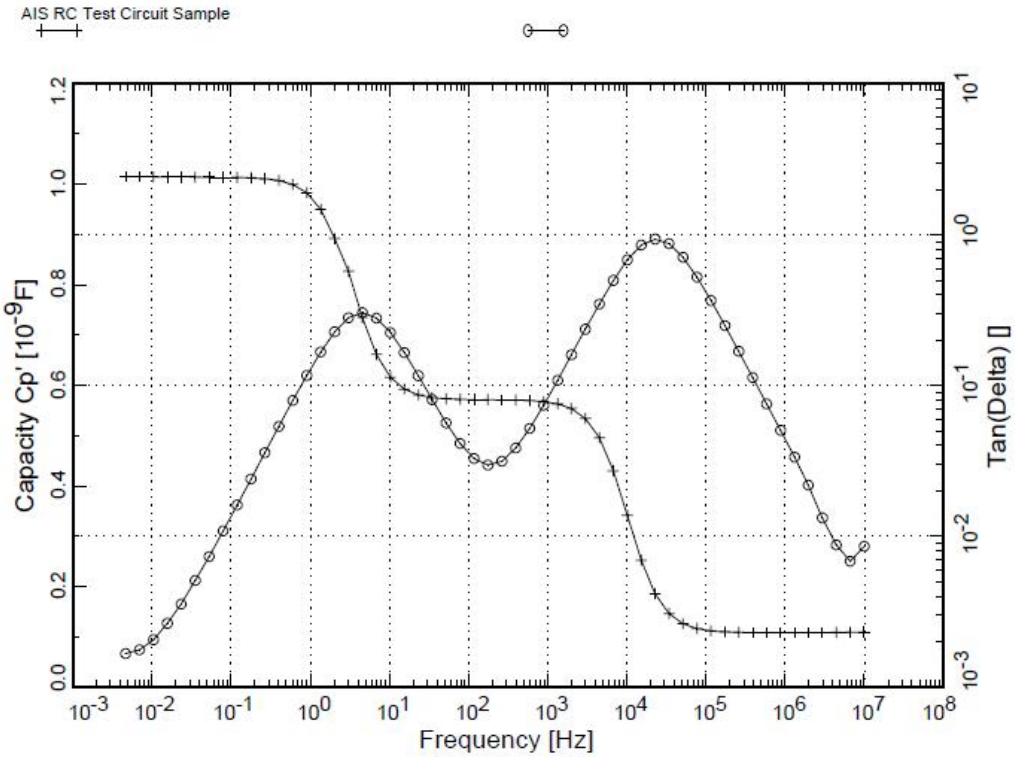


Figure 18. Typical results of the RC test sample network.

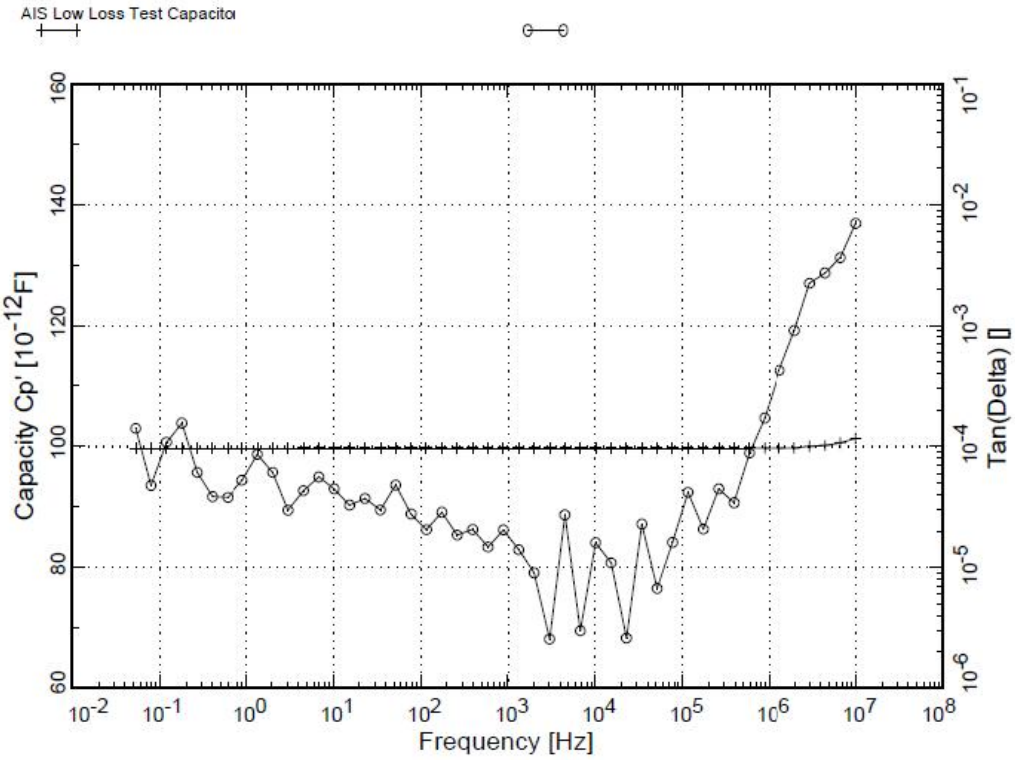


Figure 19. Typical results of the low loss test sample.

Radio Frequency Extension for Electric Material Measurements from 1 MHz to 3 GHz

9. Introduction

9.1. Basic System Features

The Radio Frequency (RF) extension provides a computer controlled solution for dielectric, conductivity and impedance measurements of materials as functions of frequency and temperature. The RF Extension is usually combined with an Agilent E4991 providing a frequency range from 1 MHz to 3 GHz. This study guide describes the RF extension in combination with the Novocontrol Quatro Cryosystem for the temperature range 160 ÷ 400 °C.

9.2. Basic Principles of System Operation

The basic system setup is shown in Figure 20 [9]:

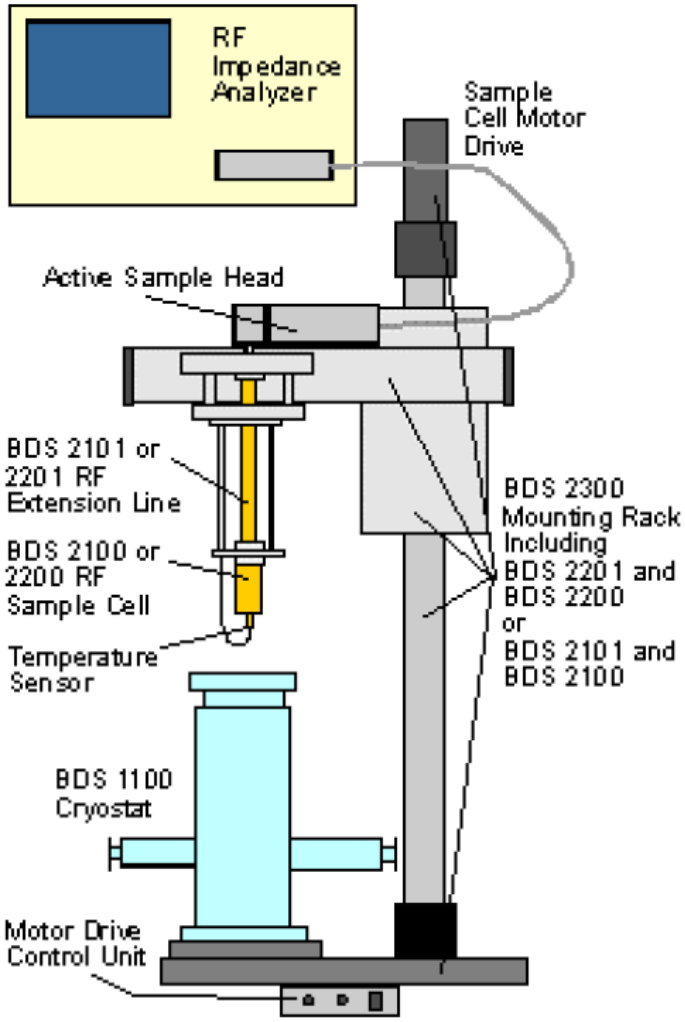


Figure 20. The basic system setup RF Extension.

The sample is mounted between two RF external electrodes which are mounted in the RF sample cell (see Fig. 21).

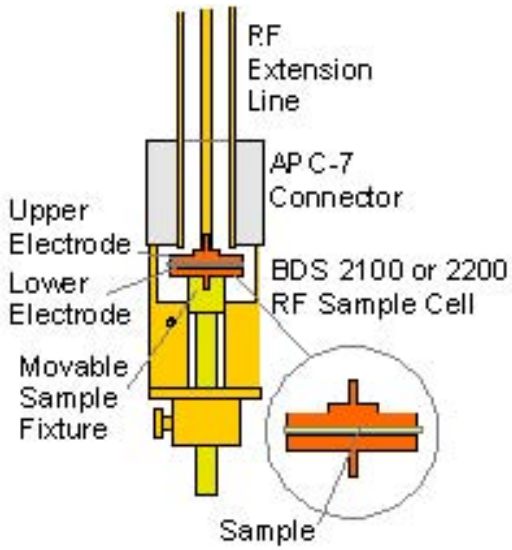


Figure 21. RF sample cell.

The RF cell is thermally isolated by the RF extension line which connects the RF cell to the impedance port located at the test station of the RF analyzer.

As this setup is very sensitive to mechanical stress, it is supported by the motor driven BDS 2300 mounting rack which allows to move the sample cell in and out of the cryostat by special mechanics avoiding mechanical forces on the extension line and APC-7 port connections.

9.2.1. RF Sample Cells and Electrodes

There are two versions of the RF sample cell which differ by the available inner diameter. BDS 2200 has 14 mm inner diameter and can be used with RF electrodes up to 12 mm. BDS 2100 has 7 mm inner diameter and can be used with RF electrodes up to 6 mm. As it supports larger electrodes, BDS 2200 is more flexible, the sample preparation is easier and due to the better known sample dimensions the result accuracy for absolute values is typically better as for BDS 2100. On the other hand, the upper usable frequency range for BDS 2200 is typically limited between 1 ÷ 2 GHz and 2 ÷ 3 GHz for BDS 2100 [9].

The sample cell components in open configuration are shown below.

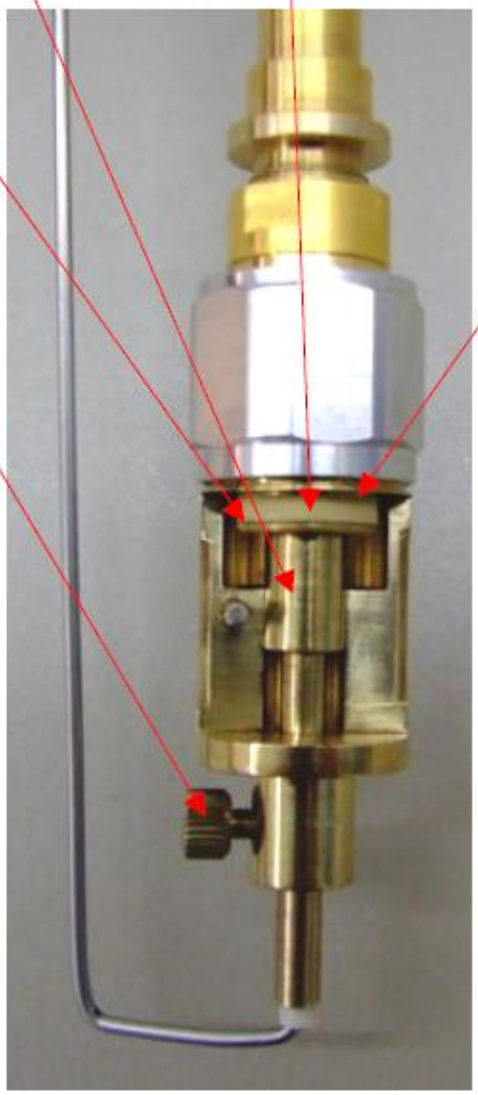
A set of *12 mm RF electrodes* with a *test sample* are shown below.

Note that the *upper electrode* has an additional *spacer ring* around its central pin and with this is not identical to the *lower electrode*.

While assembling the electrodes into the cell make sure that the upper and lower electrodes are not exchanged: the upper electrode with the spacer ring has to be located at the RF extension line side of the cell, the lower electrode has to be located at the cell moveable fixture side.

In order to mount the *electrodes* with the *sample* in between into the cell, attach the *lower electrode* to the *moveable fixture*, locate the *sample* on the *lower electrode* and locate the *upper electrode* on the *sample*.

Finally move up the *moveable fixture* so that the *upper electrode central pin* fits into *the central contact opening of the RF extension line APC-7 connector* and fix the moveable fixture by its *fixing screw*.



Note: For accurate measurement results, it is essential to create proper contacts between the sample, the electrodes and the cell contacts. For this purpose:

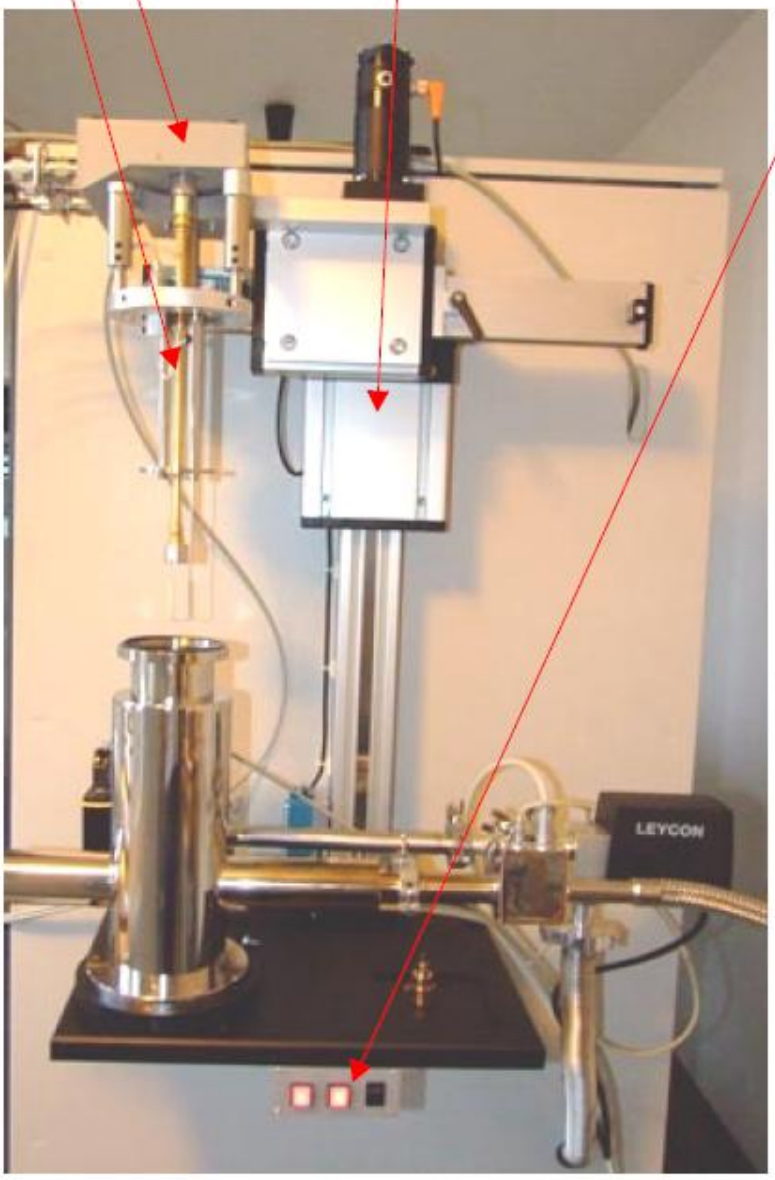
- Apply metal contacts to both sample sides by evaporation or sputtering.
- Always keep both electrode sides and the corresponding cell contacts clean and polished.
- While tightening the fixture fixing screw, push the fixture by the finger upwards in order to create pressure on the electrode contacts. It is essential to maintain the pressure until the fixture screw is completely tightened.

In order to close the cell, mount the cell *closing plate* and fix it with the *closing ring* by tightening the *fixing screw*.

9.2.2. Motor Drive and Control Unit

The *RF extension line* with the *RF sample cell* at its lower end is mounted at the *sample holder fixture* to which the *test station* of the RF impedance analyzer is attached too in order to create a mechanically stable connection.

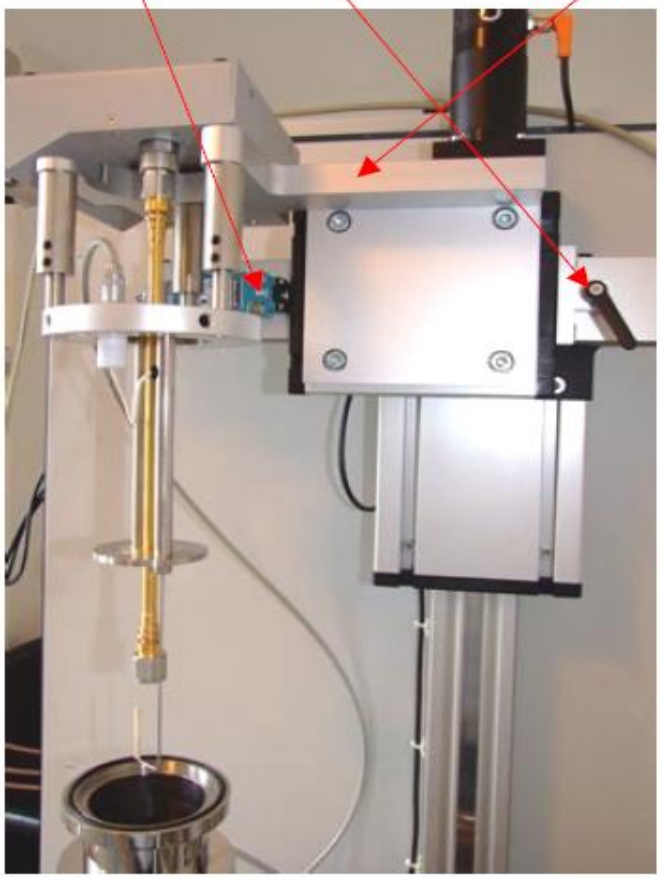
As this arrangement is sensitive to mechanical stress and has considerable weight, it is moved in - and out of the *cryostat* by the *electrical motor drive* which is operated by the *control unit*.



In order to activate the control unit, press the power switch at the control unit front panel right side. In order to move the sample cell by the motor drive into - or out of the cryostat, hold down the control unit Dn or Up button.

The motor drive can only be activated if the sample cell fixture is moved to its most left position so that the left end switch is activated.

For this purpose loosen the *horizontal shift fixing screw*, move the *sample cell fixture* to its most left position so that the *left end switch* is completely pushed and fix *the sample cell fixture* with the *horizontal shift fixing screw*.



Note: Before activating the motor drive and while the motor drive is moving:

- Make sure that no body parts are located within the motor drive operation range. Otherwise, body parts may be jammed or injured.
- If the sample cell has been moved out of the cryostat, consider that the sample cell and parts of the RF extension line may be very hot or cold. Touching these parts may result in injuries by burning or frostbite.
- Make sure that the motor drive is properly adjusted.
- While moving the sample cell into the cryostat, make sure that the sample cell and RF extension line fits into the cryostat without touching the cryostat walls or colliding with the cryostat top flange. In this case, stop moving down the motor drive and readjust the system.
- Make sure that the RF sample cell is properly mounted at the RF extension line end and the PT100 temperature sensor is completely inserted in its support within the RF sample cell as shown below.

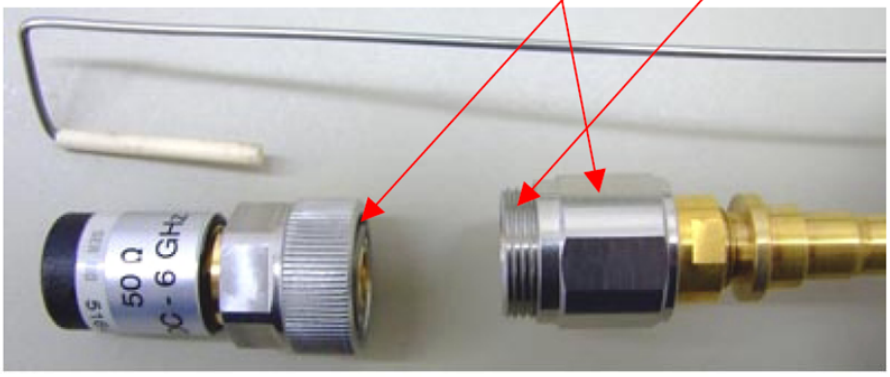
- Make sure that motor drive operating area is free and not blocked by any objects which may collide with drive components or may hinder it from moving.
- Make sure that no cables in the motor drive operating area will be damaged while the drive is moving.

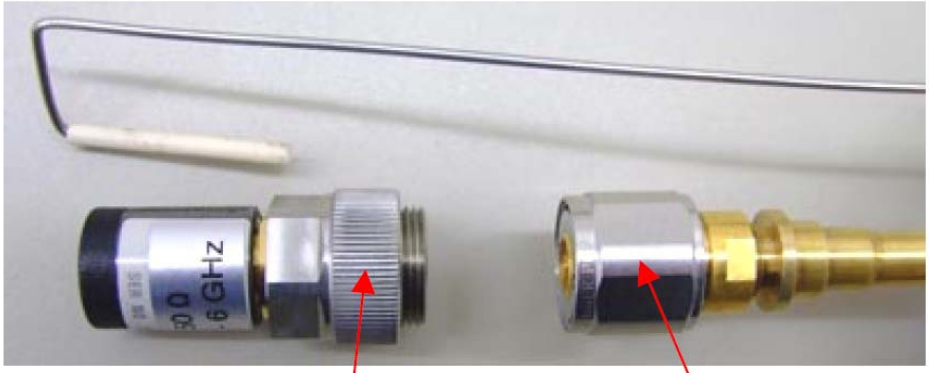
9.2.3. APC-7 Connectors

Both the *RF extension line* and the *calibration standards* supplied with the RF impedance analyzer have *APC-7 RF connectors*. These connectors can be configured *female* or *male* by performing a couple of rotations of its *outer mounting screw* in the not connected state.

In order to realize a proper electrical connection, make sure to have completely configured the both connectors to opposite sex.

The pictures below show the 50 Ohm calibration normal and the RF extension line lower end in the two possible valid configurations.





Note:

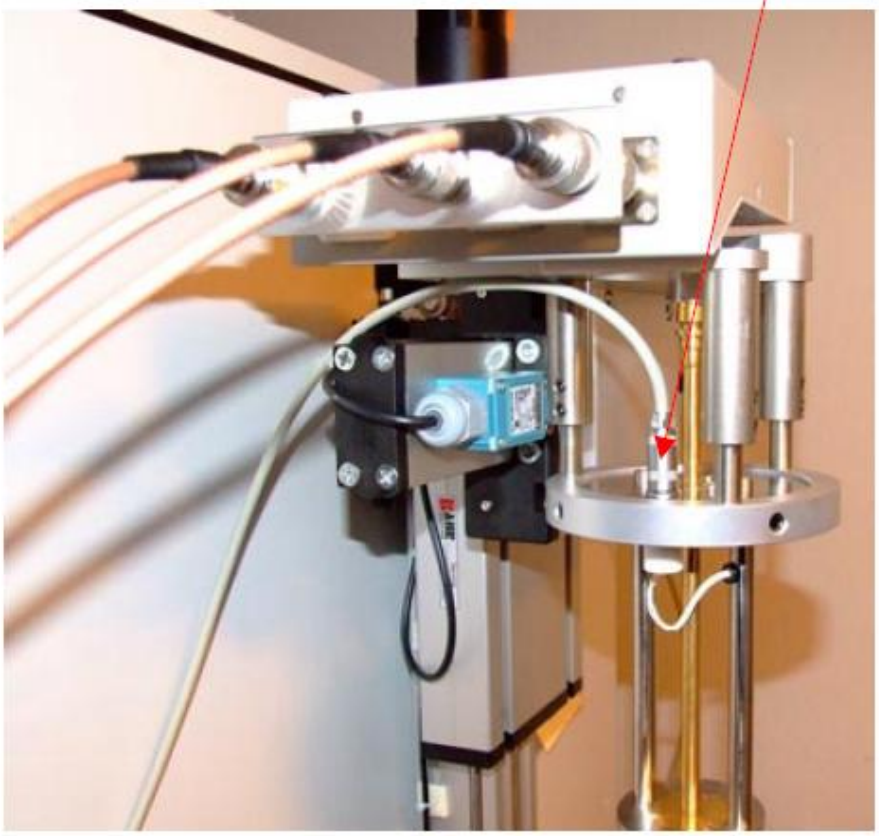
- While connecting the two connectors, only to tighten the *mounting screw* of the female connector and leave the *mounting screw* of the male connector loose.

10. Setting up the System for Measurement

10.1. Preparing the System for Measurement

- Make sure that the system is properly setup.
- Switch on the RF impedance analyzer at least 30 minutes before the first measurement or calibration to warm it up.
- Move the sample cell fixture to its most left position and fix it. Move the motor drive to its upper position. For details refer to the "Motor Drive and Control Unit" chapter.
- Make sure that the system is properly calibrated as described in the "Calibrations" chapter before a measurement is performed.

- Connect the Quatro controller *sample temperature channel (Channel 4) cable* to the *RF* extension line *temperature sensor connector*.



10.2. Calibrations

Before a measurement can be done, it is required to have performed the following two kinds of calibrations [9].

10.2.1. Line Calibration

It calibrates the system up to the lower end of the RF extension line. For this calibration, the Short, Open, 50 Ohm and Low Loss Capacitor calibrations supplied with the RF impedance analyzer are required which have to be connected in sequence to the RF extension line end instead of the sample cell during the calibration.

The calibration is performed by the WinDETA software and started from the menu "Analyzers, HP4991, Options, Calibrate Line" or "Analyzers, HP4291, Options, Calibrate Line" [7].

Note:

- Due to thermal drift it is recommended to perform the line calibration at least anytime after switching the RF analyzer on.
- Highest accuracy will be achieved if the calibration is performed directly before a

- It is recommended to set "Number of Averages per frequency" to 20 in the WinDETA dialog "Analyzers, HP4991, Options, Configuration" or "Analyzers, HP4291, Options, Configuration". This will reduce the calibration noise level.

10.2.2. RF Sample Cell Calibration

It calibrates stray capacities, serial resistance and inductance of the RF sample cell. During the calibration it is required to prepare a shorted and open electrode arrangement within the RF sample cell. The calibration is performed by the WinDETA software [7] and started from the menu "Analyzers, HP4991, Options, Calibrate Dielectric Sample Cell" or "Analyzers, HP4291, Options, Calibrate Dielectric Sample Cell".

Note: This calibration should be performed for each set of electrodes.

10.3. Performing a Measurement

Before a starting measurement:

- Make sure to have considered all previous items within this chapter.
- Make sure to have prepared a proper sample which has a capacity or impedance matching the RF impedance analyzer measurement range. Details can be found in the WinDETA program manual [7], chapter "General Description, Sample Preparation and Measurement Accuracy" and in the analyzer manual [6, 9]. A measurement is performed by the WinDETA software which can be configured to many modes and has many options. For details, refer to the WinDETA program manual [7], chapter "General Description, Setting Up a Measurement" and "Running a Measurement".

Note: It is recommended in to set "Averages over points" to 20 in the WinDETA dialog "Measurement, Averaging". This will reduce the result noise level.

11. Performance Test and Calibration Verification

11.1. Line Calibration Verification

The line calibration can be verified by measuring the four calibration standards in sequence with disconnected sample cell and comparing the results by the typical results shown in Figures 22-25.

For this purpose:

- Do a line calibration as described in the "Calibrations" chapter.
- In the WinDETA dialog "Analyzers, HP4991, Options, Configuration" or "Analyzers, HP4291, Options, Configuration" disable "Use Dielectric Sample Cell Calibration". This will

prevent the system from applying the correction for the not connected sample cell to the following measurements of the calibration standards.

- Connect the 50 Ohm calibration standard to the Novocontrol RF extension line end (instead of the sample cell) and start a frequency sweep. The result should be a constant real impedance of 50 Ohms. Compare the measured results with the typical results below.
- Repeat the above step for the other calibration standards LOW LOSS CAP., OPEN and SHORT. There should be no significant deviations between your results and the one shown below. Otherwise there may be a problem with your analyzer or the extension line. If the test is passed, the system is properly working and properly calibrated to the extension line end.

The typical results for the four calibration standards are shown in Figures 22-25 [9].

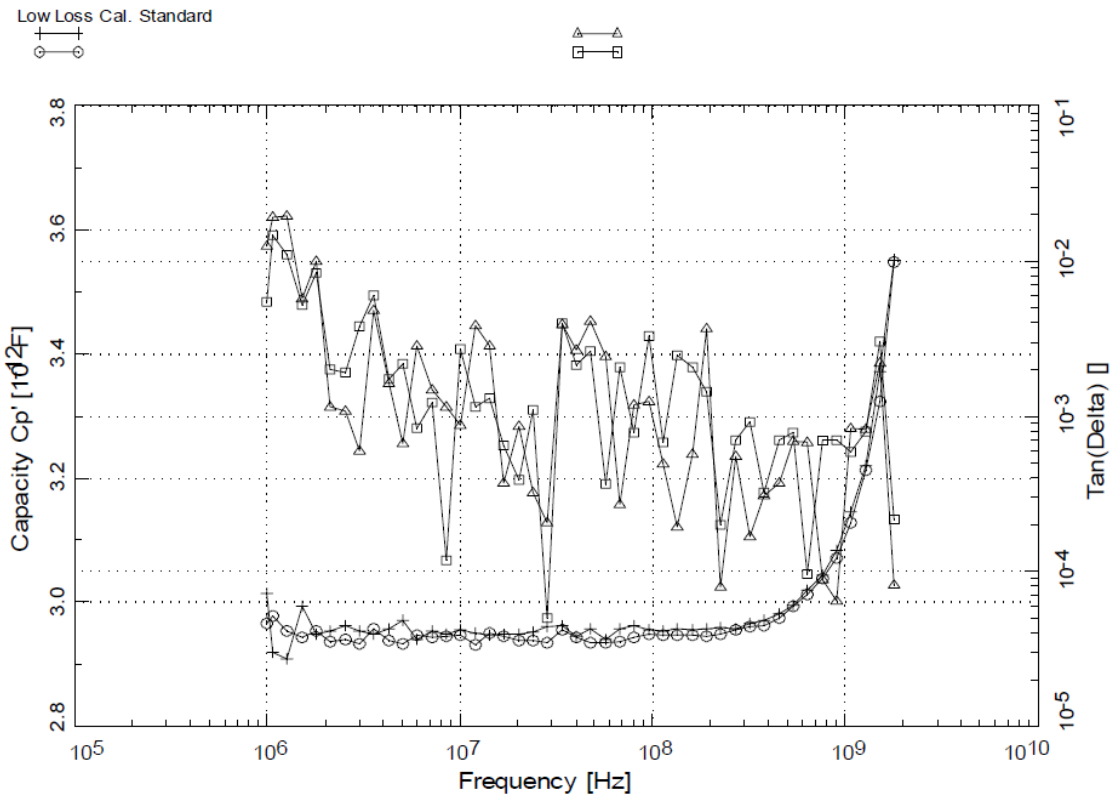


Figure 22. Typical results of the low loss calibration standard.

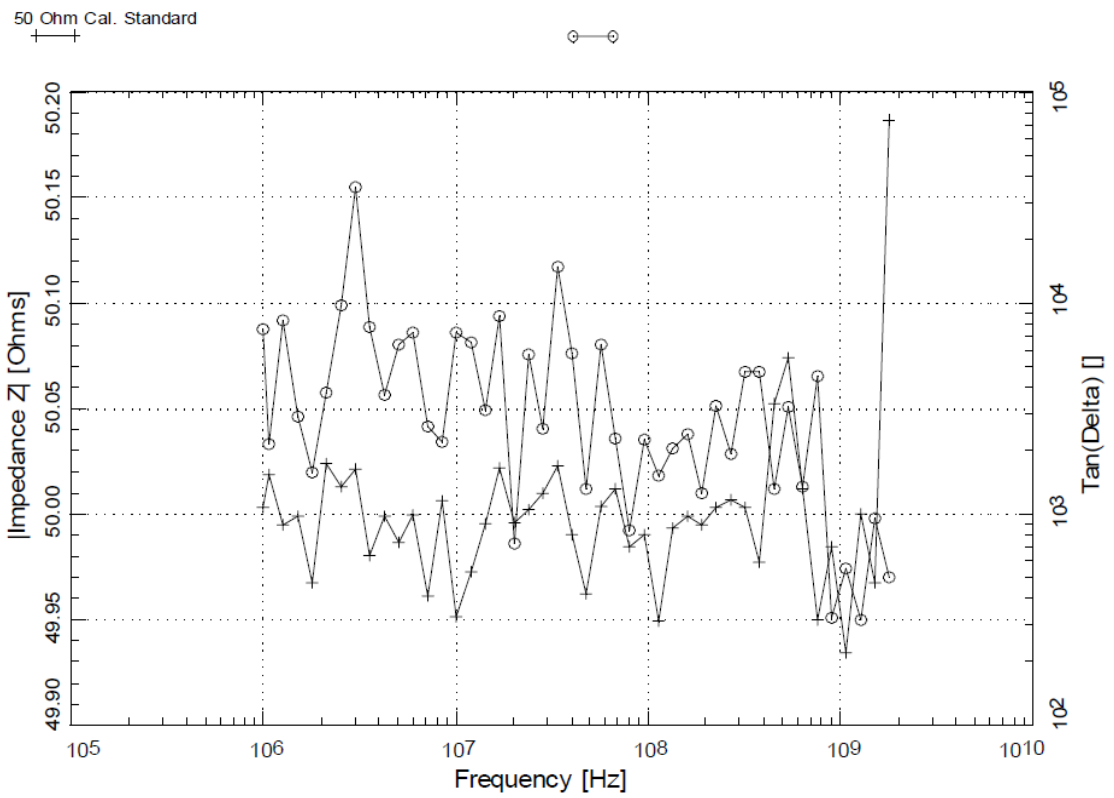


Figure 23. Typical results of the 50 Ohm calibration standard.

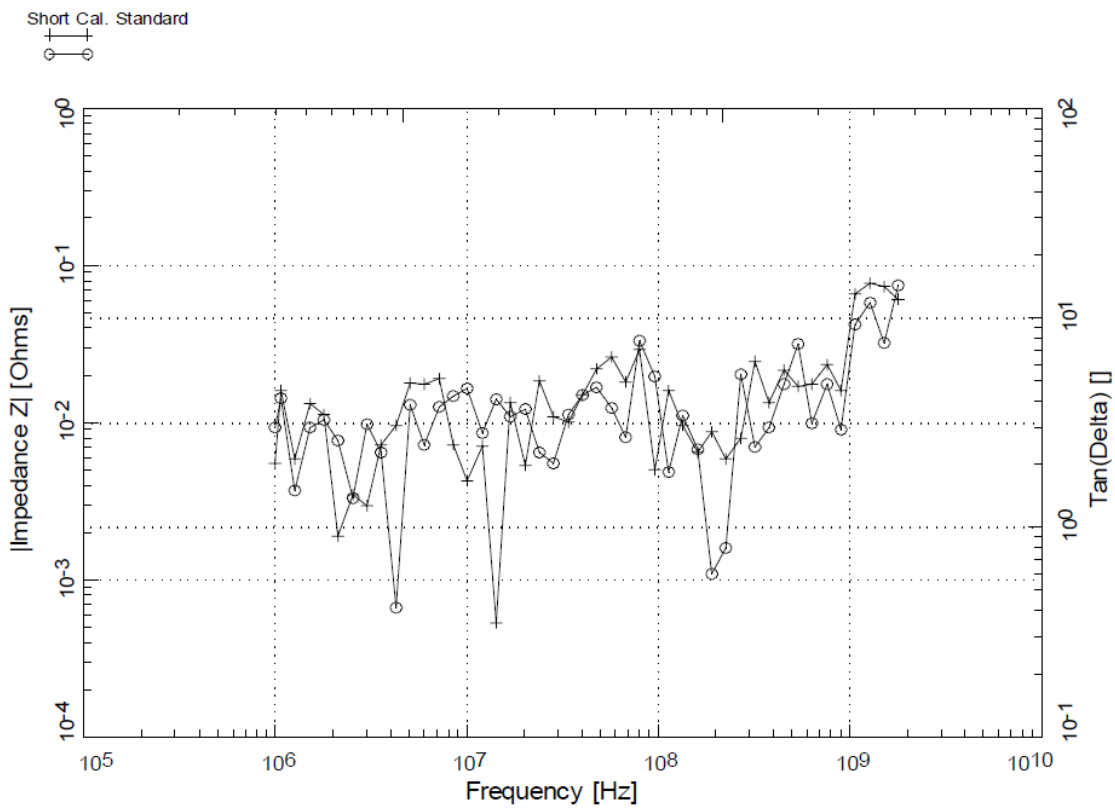


Figure 24. Typical results of the short calibration standard.

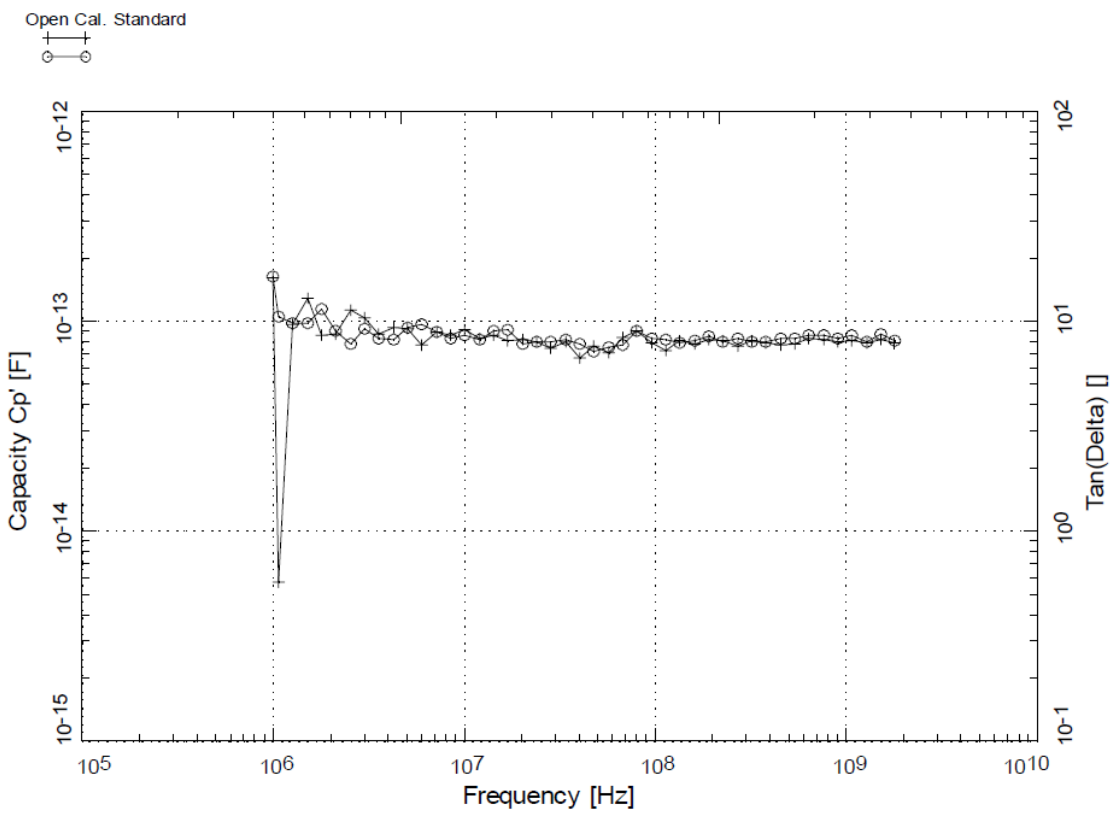


Figure 25. Typical results of the open calibration standard.

11.2. Sample Cell Calibration Verification

The sample cell calibration can be verified by measuring a known material and compare the results with typical results shown in Figure 26 [9].

For this purpose:

- In "Analyzers, HP4991, Options, Configuration" or "Analyzers, HP4291, Options, Configuration" enable "Use Dielectric Sample Cell Calibration" to activate the sample cell calibration in the following measurements. Perform a sample cell calibration as described in the "Calibration" chapter.
- The system should now be properly calibrated for the used electrode set. Only this electrode set should be used in following measurements.
- Before measuring a sample, a test with a low loss material like Teflon or air should be performed in order to verify the sample cell calibration and compared with the typical results (see Fig. 26).

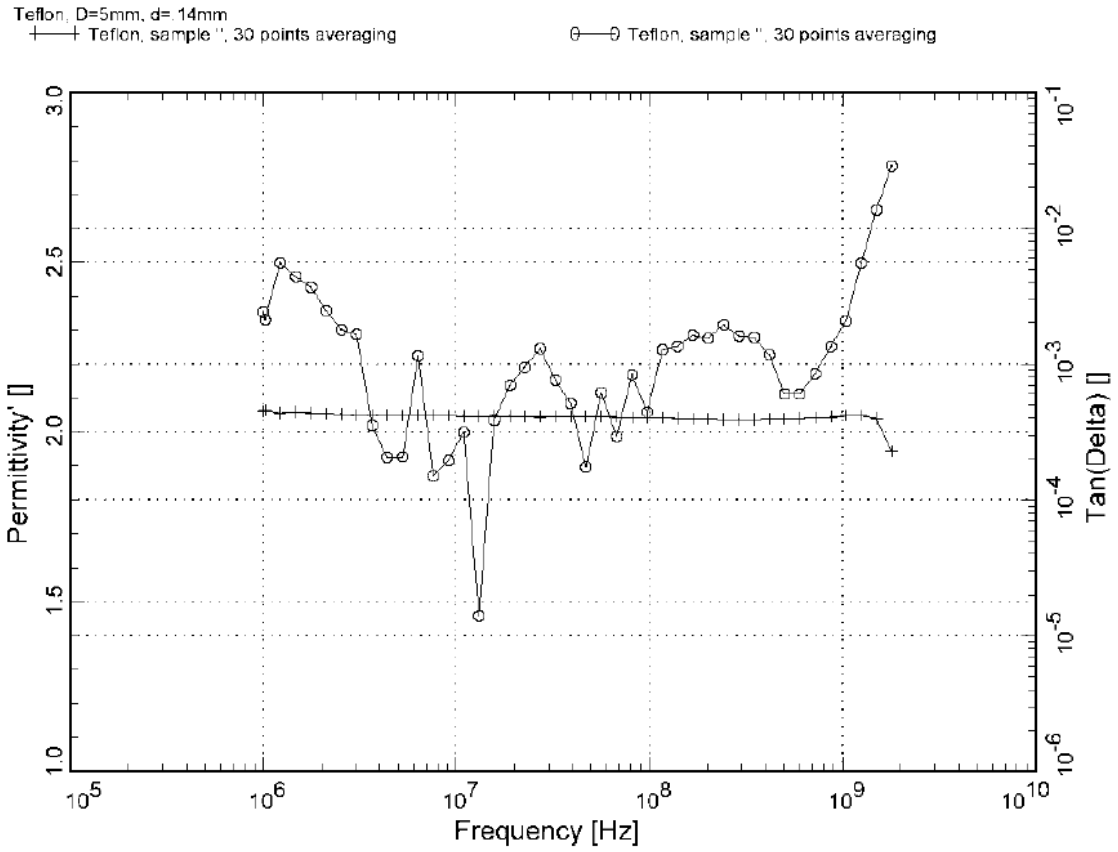


Figure 26. Typical results of the Teflon sample.

Quatro Cryosystem

12. Basic System Features

The QUATRO Cryosystem is a high quality turn key temperature control system for applications in materials research. It can be used with all Novocontrol sample cells for dielectric and impedance spectroscopy except for the High Temperature Sample Cell. The system was developed to set or ramp the temperature of the sample under test with high accuracy and reproducibility within a range from -160°C to 400 °C [10]. The QUATRO Cryosystem was designed for easy, safe and full automatic operation enabling computer controlled experiments over several days without supervision as required for low frequency measurements.

The QUATRO Controller has five sensor channels to measure the sample temperature, the gas temperature, the temperature of the liquid nitrogen in the dewar, the pressure in the dewar and the isolation vacuum pressure (see Fig. 27). These signals are used as input for the sample or gas temperature controller circuit which powers the gas heater. A second independent controller for the dewar pressure applies power to the dewar heater [10]. This design allows a very fast, precise and safe operation which is required for automatically driven systems. Even if sensors fail or the system runs out of liquid nitrogen, the system will automatically shut down to protect itself and the sample.

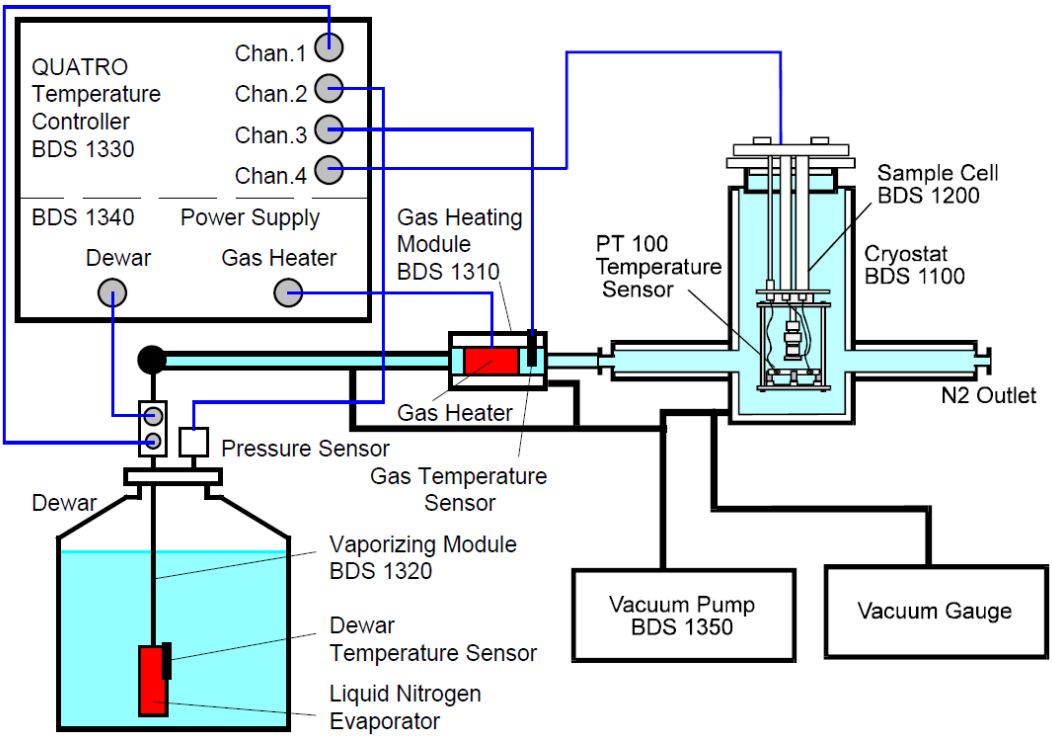


Figure 27. Setup of the QUATRO Cryosystem.

Features of QUATRO Cryosystem:

- high precision turn key temperature control system
- temperate range from -160°C to 400°C
- temperature ramps from 0.01°C/min to 20°C/min
- 0.01°C temperature stability
- temperature overshooting after set point step typically < 0.2°C
- stabilization times typically below 8 minutes for 0.1°C stability (see Fig. 28)
- low nitrogen consumption due to automatic pressure - temperature adaptation
- automatic adaptation of controller parameters (selftune)
- 4 channel microprocessor controller with 24 bit ADC and IEC communication port
- vacuum-isolated cryostat and nitrogen lines

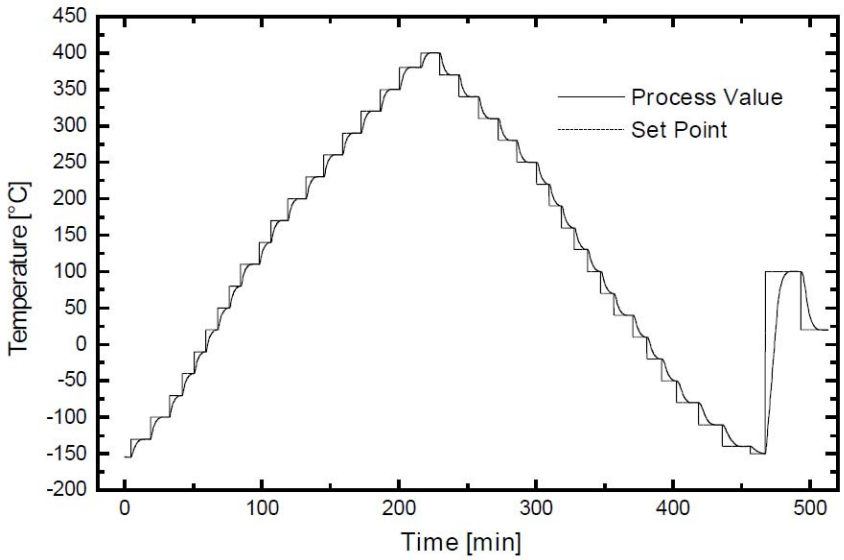


Figure 28. Stabilization characteristics of the sample temperature (process value) in dependence of the temperature setpoint. Setpoint step after sample temperature stabilization to 0.1°C accuracy.

12.1. Basic Principles of System Operation

The QUATRO Cryosystem can be configured for operation in either Liquid Nitrogen Mode or Dry Air / Nitrogen Mode as described below.

12.1.1. Liquid Nitrogen Mode

This is the standard operation mode. The sample temperature range is from -160°C to 400°C. Cold nitrogen gas is evaporated by heating inside a liquid nitrogen dewar to create a cold gas stream which is heated by an additional gas heater to adjust the sample temperature.

Depending on the sample temperature setpoint, the heating element of the evaporator module builds up a specified pressure in the liquid nitrogen dewar to create a constant flow of cold nitrogen gas within the gas heater module [10]. The pressure and temperature in the dewar are measured by two channels of the QUATRO Controller. The dewar pressure controller applies a variable amount of power to the dewar heater module to the dewar pressure to the pressure setpoint which is the intended pressure value.

With increasing dewar pressure:

- the flow rate of cold nitrogen gas within the gas heater module and the sample cell increases,
- the lowest temperature which can be reached by the system at the sample decreases,
- the maximum available cooling rate of the sample increases,
- the maximum available heating rate within the upper temperature range decreases,
- the nitrogen consumption increases.

The dewar pressure can be either adjusted to a fixed value or automatically adjusted depending on the sample or gas temperature setpoint.

12.1.2. Dry Air / Nitrogen Mode

In this mode the sample temperature range is from ambient to 400°C and no liquid nitrogen is required. It is useful for measurements which do not require temperatures below ambient temperature to save liquid nitrogen.

In the Dry Air/Nitrogen Mode, the dewar heater and dewar temperature sensor are not used by the system. The QUATRO Controller is configured by default to Liquid Nitrogen Mode. In order to switch it to Dry Air/Nitrogen Mode, refer to the chapter QUATRO Controller Front Panel Menus Description, N2/Air Menu (System Configuration) [10].

12.1.3. Sample and Gas Temperature Controller

Both for the Liquid Nitrogen and Dry Air/Nitrogen modes, the gas temperature controller circuit applies a variable amount of power to the gas heater module to match the gas temperature to the intended setpoint. After heated by the gas heater module, the nitrogen gas flows directly through the sample cell which is mounted in the cryostat. The system can be configured to stabilise the temperature either at the gas temperature sensor (Gas Temperature Mode) or at the temperature sensor of a sample cell built into the cryostat (Cryo Temperature Mode) [10].

12.2. Safety Functions

The QUATRO Controller provides an internal safety system which disables the gas and dewar heaters at the following events:

- sensor break or malfunction
- sensor cable damage
- dewar temperature beyond high limit (signals lack of liquid nitrogen)
- dewar underpressure
- dewar overpressure

Before enabling the gas heater, the nitrogen gas flow is verified by waiting for a certain start temperature (usually 0°C).

12.3. Remote Control Interface

The QUATRO Controller provides full remote control through a GPIB IEEE 488 computer interface. This allows the QUATRO Cryosystem to be included in automatic measurement setups. With the appropriate software, like Novocontrol WinDETA [7] and WinCHEM [11], fully automated experiments can be performed at several preselected temperatures. The host computer controls the QUATRO Cryosystem, the analysers and other devices. It collects, evaluates and displays the measured data and stores them for further processing with other programs.

13. QUATRO Controller

Figure 29. QUATRO controller front panel.

This unit includes the signal channels for the five system sensors, two digital controllers for the dewar pressure and sample temperature, the user display and control keys. The display indicates process values, setpoints and status information (see Fig. 29). Together with the 6 software controlled menu keys it is used for a menu system, too. The user can change setpoints, control the system or set configuration parameters. The function of the menu keys varies in different menus. The actual function is indicated above each key in the 4th display row [10].

13.1. Sensor Input Channels

There are four front panel sensor input connectors. The three Pt100 sensors are connected by Lemo plugs, the pressure sensor by a five pin DIN plug [10]. Connect the sensors follows:

Channel 1: Dewar evaporator temperature Pt100
Channel 2: Dewar pressure bridge sensor
Channel 3: Gas heater temperature Pt100
Channel 4: Cryostat (sample) temperature Pt100
Channel 5: Thermal isolation vacuum (sensor connected to QUATRO Controller rear panel)

13.2. Alarm Indicators

Several safety functions are provided which are displayed at the front panel by the alarm indicators A1 ... A8. Permanent activation of an alarm indicator has the following meaning [10].

A1: dewar evaporator temperature high limit exceeded, i.e., dewar empty or the liquid nitrogen level too low
A3: dewar overpressure
A4: dewar pressure too low to operate the gas heater
A5: gas heater temperature above Start Temperature Limit (generally 0°C). This feature prevents gas heater operation if the cold nitrogen gas stream is insufficient. If the gas heater is already running, this indicator may become active, but this has no further effect.

In addition, the alarm indicators may show a sensor break condition. If any of the sensors is not connected or the sensor itself or a cable is defect, two of the indicators are blinking.
A1 A2 blink: Invalid signal from dewar temperature Pt100 sensor
A3 A4 blink: Invalid signal from dewar pressure sensor
A5 A6 blink: Invalid signal from gas heater temperature Pt100 sensor
A7 A8 blink: Invalid signal from sample cell temperature Pt100 sensor

In any case of sensor break detection, the according controllers will be switched off and the heater power disabled.

14. QUATRO Power Supply

The power supply section contains the dewar evaporator and gas heater power supplies. In addition, it contains line power switches and terminals for the Vacuum system, QUATRO Controller and Analyzer units (see Fig. 30).

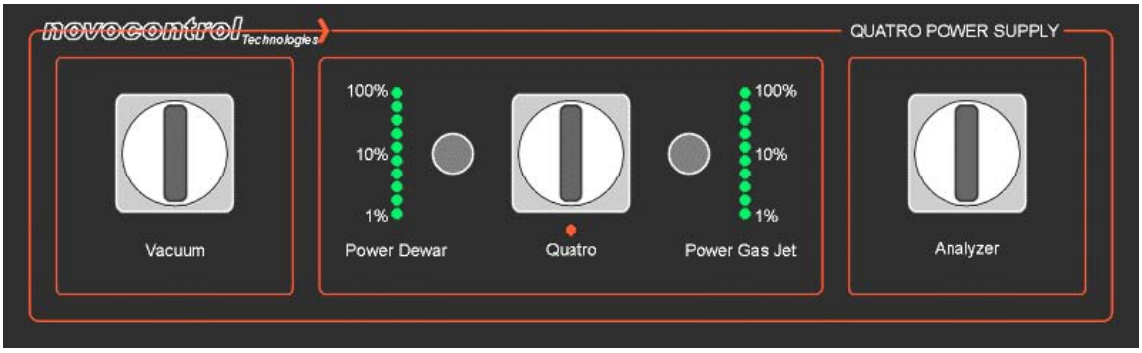


Figure 30. Power supply section.

14.1. Vacuum Switch

This switch is used to activate the vacuum pump and the electrical vacuum isolation valve. To activate the vacuum system, turn the switch clockwise out of the vertical position. The switch applies power to the "Vacuum" terminals at the QUATRO Power Supply rear side.

14.2. Quatro Switch

This switch is used to activate the line power for the QUATRO Controller unit and the two internal variable power supplies for the gas and dewar heater. To activate the switch, turn it clockwise out of the vertical position. The switch applies power to the QUATRO, "Dewar" and "Gas" heater terminals at the QUATRO Power Supply rear side [10]. After activation, the "Quatro" light should become active and after a few seconds, the display of the controller section should show the actual sensor values.

14.3. Analyzer Switch

This switch is used to activate the analyzers which do not have an own line power switch at their front panel like the Novocontrol Alpha or Beta analyzers. For activation, turn the switch

clockwise out of the vertical position. The switch applies power to the "Analyzer" terminals at the QUATRO Power Supply rear side.

14.4. Dewar and Gas Heater Power Supplies

These are two internal pulsed power supplies with 42V 15A peak power for the gas and dewar heaters which have to be connected to the corresponding terminals at the QUATRO Power Supply rear panel. Above the "Power Dewar" and "Power Gas Jet" labels two display bars indicate the power amount applied to the heaters, if connected. The amount of applied power is controlled by the QUATRO Controller.

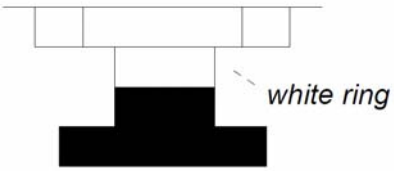
Note: If the gas or dewar heaters are not connected to QUATRO Power Supply or disconnected by the automatic fuse switches as described below or an heater element or connection cable is damaged, the corresponding display will show 100% power irrespective of the QUATRO Controller power output status.

This feature is useful to indicate proper operating conditions of the two heater elements. After switching on the system by the "Quatro" switch, the gas and dewar heaters are not powered by the QUATRO Controller unless activated by the user. Thus a display constantly showing 100% power indicates a problem with the corresponding heater channel.

The two automatic fuse switches at the right and left side of the display bars can be used to manually interrupt the connection to the dewar and gas heaters [10]. These switches also feature automatic fuses which prevent damage of the power supply circuitry, in case of external short circuit of the dewar or gas heaters.

The fuse switches have two positions:

Gas or dewar heater connected, fuse not blown



Gas or dewar heater disconnected, fuse blown

In case of a 'blown' fuse, check your equipment for short circuit. Repair, if necessary, and push the button until it snaps in. The normal resistance of the dewar and gas heater elements is 2 ÷ 3 Ω.

14.4.1. Preparing the Liquid Nitrogen Dewar for Operation

Warning: During all activities performed on the dewar with liquid nitrogen or cold nitrogen gas as described in the following chapters cold liquid nitrogen or cold nitrogen gas may spill out of the dewar.

In addition, parts of the dewar, evaporator unit and gas heater unit may be very cold or hot.

Getting in contact with liquid nitrogen, cold nitrogen gas or cold system parts may result in injury due to frostbite.

This applies especially to the eyes where even contact with only small amounts of liquid nitrogen may result in permanent injuries.

It is therefore necessary to put on the safety glasses and gloves supplied with the dewar before performing any activities as described in the following chapters.

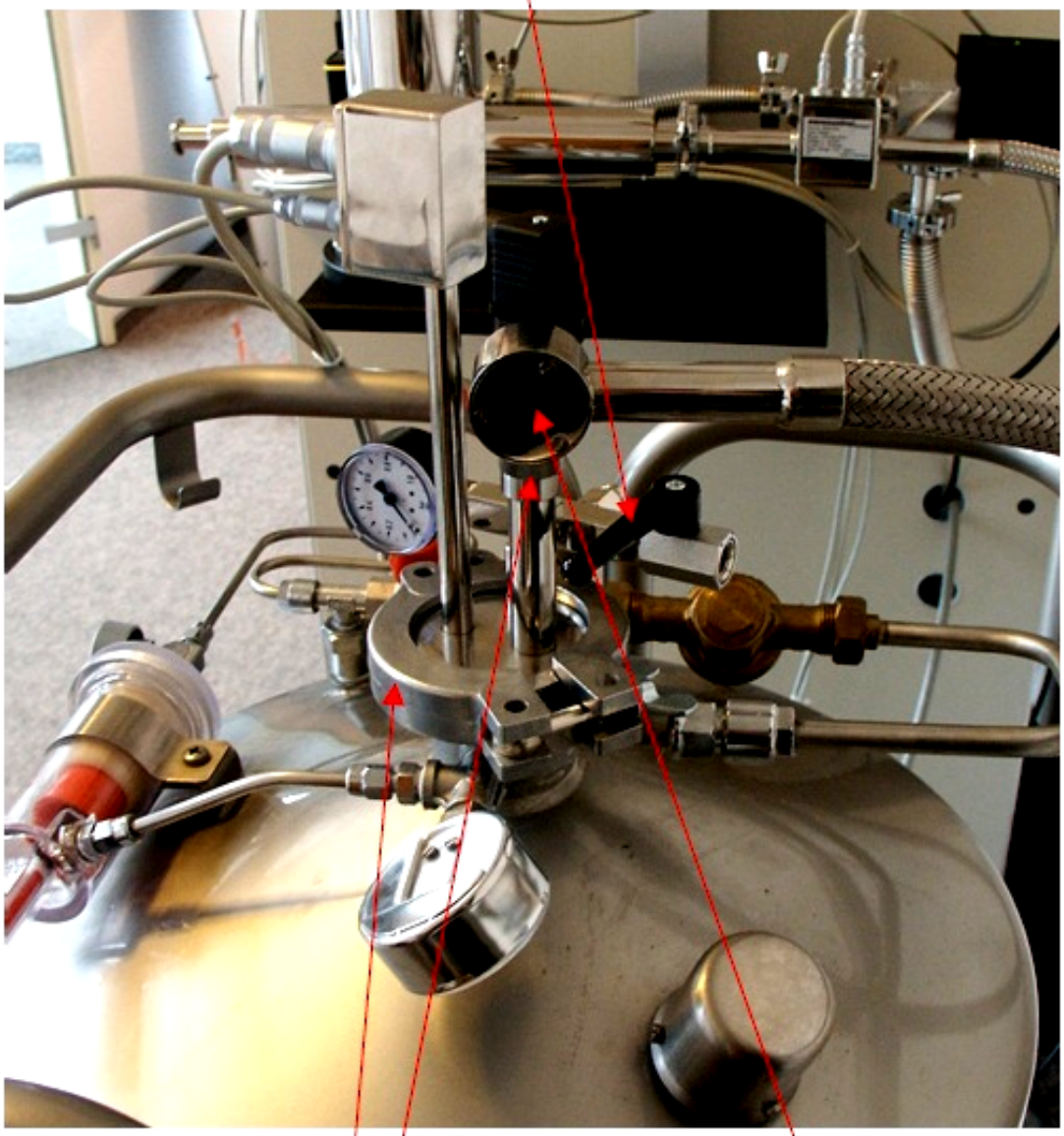
If the dewar is empty or the liquid nitrogen level is low, the dewar has to be filled. For this purpose proceed as follows.

14.4.2. Removing the Evaporator Unit out of the Dewar

Warning: Respect the safety warnings at the beginning of the "Preparing the Liquid Nitrogen Dewar for Operation" chapter.

1. Deactivate the dewar heater as described in the chapter QUATRO Controller Front Panel Menus Description, Power Menu.
2. Wait until the dewar pressure has dropped to a value < 3 mbar. The dewar pressure is shown in the QUATRO Controller display.
3. In order to release any remaining pressure in the dewar open the black pressure release valve at the evaporator unit as shown below. Wait until the pressure has completely released from the dewar. The picture shows the pressure release tab in the closed state.

Note: Do not open the pressure release valve as long as a considerable amount of pressure remains in the dewar. Release of a huge amount of cold gas might freeze, and, as a consequence, damage the sensor.



4. Loosen the gas heater tube clamp screw and pull the gas heater tube out of the evaporator unit. Note: The clamp screw should no be completely unscrewed from its counterpart tube but only loosened by rotating it approximately a half turning.

5. Remove the evaporator fixing clamp and pull the complete gas heater module out of the dewar.

14.4.3. Filling the Dewar with Liquid Nitrogen

Warning: Respect the safety warnings at the beginning of the "Preparing the Liquid Nitrogen Dewar for Operation" chapter.

The dewar should only be filled through its main top opening after removal of the evaporator unit.

Do not try to fill the dewar with the evaporator unit mounted through the gas heater mounting tube. This will result in freezing and permanent damage of the pressure sensor.

1. In order to fill the dewar insert a flexible filling tube into the dewar and slowly start filling with liquid nitrogen. Consider that at the beginning of the filling process the inner dewar parts may still be at ambient temperature and major amounts of cold gas may evaporate out of the dewar.
2. The dewar should be completely filled, but the dewar inlet opening tube should remain free from liquid nitrogen. If the dewar is only partly filled, it may last several days before the inner dewar parts not covered by liquid nitrogen get cold and the system may not reach its lowest temperature within this time.

14.4.4. Remounting the Evaporator Unit into the Filled Dewar

Warning: Respect the safety warnings at the beginning of the "Preparing the Liquid Nitrogen Dewar for Operation" chapter.

1. Before inserting the evaporator unit into the dewar, make sure that the metal-rubber seal ring for the evaporator fixing clamp is located on the dewar inlet opening tube.
2. Slowly move the evaporator unit into the dewar inlet tube until it gets in contact with the liquid nitrogen. Immerse the evaporator unit about 5 cm into the liquid nitrogen to cool it down. Never immerse the evaporator unit completely in a short time into the dewar. While cooling down major amounts of cold gas or liquid may flow out of the dewar.
3. While cooling down, the evaporator unit will be thermally isolated from the liquid nitrogen by a gas bubble which is created around the evaporator unit due to nitrogen boiling. At the end of the cooling process, the gas bubble will collapse and the liquid nitrogen will get in direct contact with the evaporator metal. This will result in strongly increased boiling and liquid nitrogen may spill out of the dewar. This effect can be avoided by pulling the evaporator out of the liquid nitrogen and slowly reinserting it until the boiling disappears. After this the evaporator can be slowly completely inserted into the dewar.
4. For the remaining evaporator and gas heater remounting procedure apply the "Removing the Evaporator Unit out of the Dewar" in reverse order.

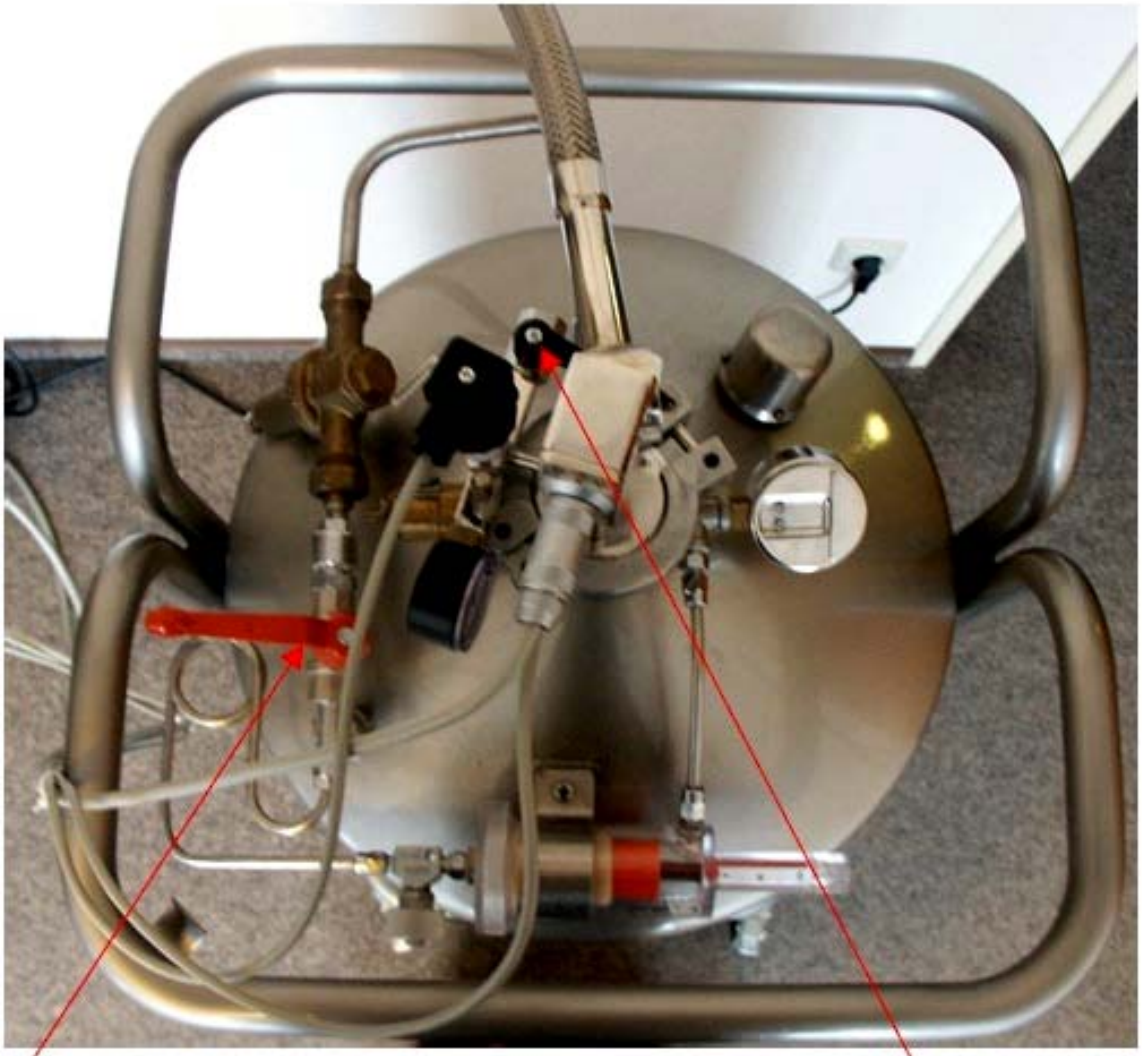
Note:

- Before inserting the gas heater tube into the evaporator tube make sure that the gas inlet opening at the lower end side of the gas heater tube is not blocked by ice. Otherwise use a hair dryer in order to completely remove the ice. If the inlet is completely or partly blocked, the system will not operate at all or will not reach the lowest temperature.
- While inserting the gas heater tube into the evaporator tube, do not apply any force. The gas heater tube has to move without force. If this is not the case make sure that the gas heater tube clamp screw is loosened and apply a small amount of vacuum oil on the gas heater tube for greasing. If the gas heater tube still does not easily move in, unscrew the heater tube clamp screw completely from the evaporator and check whether the clamp rubber seal ring is properly located in its position.
- If inserted, the gas heater tube lower end must not enter the liquid nitrogen but be located above the liquid level. This is usually not a problem unless the dewar has been filled to the top end (overfilled). In the usual situation (non-overfilled dewar), completely insert the gas heater tube into the evaporator tube opening.

5. Finally fix the heater tube clamp screw to seal the gas heater tube with respect to the evaporator tube. During operation, no gas must flow out at any system points except at the cryostat gas outlet.

6. Finally the dewar top should look like shown below.

The red tab should never be opened if the dewar is used in combination with the QUATRO Cryosystem. Opening this tab will lead to self pressure build up within the dewar even if the dewar heater is not powered which will lead to system malfunction.



Before operating the system, make sure that both the black pressure release tab and the red tab on the dewar are closed as shown above. The red tab is not available at the 120 liter dewar.

14.5. Activating the System

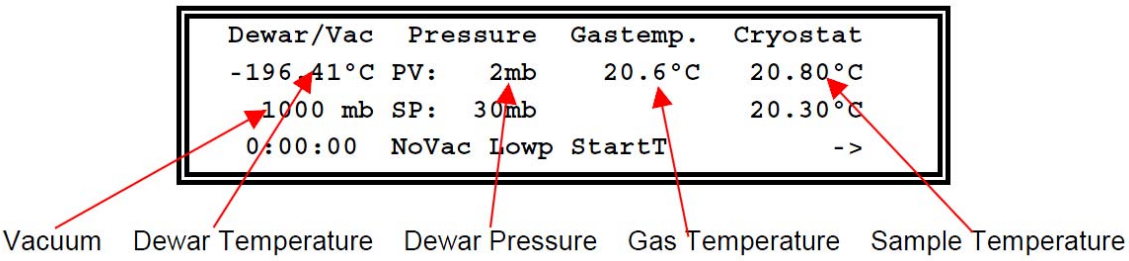
Before activating the system, carefully read the chapters Safety, Introduction, QUATRO Controller, Quatro Power Supply and Setting up the System for Operation. According to these chapters, make sure that your system is safe and is in an operational state.

In the following it is assumed that you are operating the system in the Liquid Nitrogen mode with the dewar connected.

1. In order to activate the QUATRO Controller and Power Supply, switch on the "Quatro" switch at the QUATRO Power Supply. This switch applies power to all system

components except for the vacuum pump. After this, the QUATRO Power Supply "Quatro" lights and the QUATRO Controller display should become active within about 20 seconds.

2. Check the QUATRO Controller display for reasonable sensor values in the Main menu:



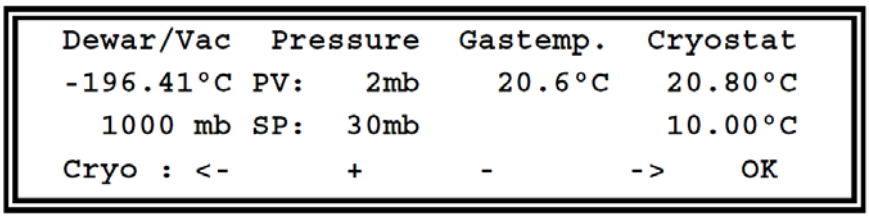
In addition, check the alarm indicators A1 ... A8 at the QUATRO Controller front panel. A4 (Low Pressure) and A5 (Start Temperature) should be permanently on and all others should be off [10].

3. Switch on the "Vacuum" switch at the QUATRO Power Supply. This switch applies power to the vacuum pump and the electric vacuum valve. After some minutes a vacuum should decrease. After about 30 minutes a vacuum of at least 10 µbar should be reached.

If the vacuum does not reach values below or equal 10 µbar:

- Check whether the pump is operational and the vacuum system properly connected to the vacuum inlet of the pump.
- Check whether the electrical vacuum valve is open. This should be indicated by a green light at the valve.
- Check whether all vacuum connections are properly mounted and tightly fixed and sealed.

4. Adjust the system for either control (regulation) of gas or sample temperature. For this purpose, press the -> menu key and than the Temp. menu key. After this the display should be indicated «Press the Cryo key» to toggle between Cryo and Gas.



Cryo: The system will stabilize the temperature at the sample cell temperature sensor. This is the most accurate and normal operation mode. In the Cryo mode, make sure to have mounted the sample cell in the cryostat.

Gas: The system will stabilize the temperature at the gas temperature sensor located inside the gas heater module. In this case it is optional to have the sample cell mounted into the cryostat. The gas temperature mode is therefore useful to keep the gas temperature at a constant value without shutting down the dewar and gas heaters in situations when the sample cell has to be removed from the cryostat for a short period of time.

5. Adjust the intended temperature setpoint (either gas or cryo according to your previous selection). For this purpose use the -> and <- menu keys to navigate on the setpoint and change the value by the + and - menu keys. Finally leave the Temp. menu with the OK key and press the <- menu key to return to the Main menu.

6. Activate the dewar and gas heaters. For this purpose, press the -> menu key and than the Power menu key. After this the display should look like

```
          Power of Heaters
     Cryostat              Dewar
       Off                  Off
    On     Off           On     Off    Ok
```

Press the On menu key below Cryostat to activate both heaters and leave the menu with the Ok key and press the <- menu key to return to the Main menu.

Immediately after activating the heaters, the dewar heater should be powered as indicated by the display power display of the QUATRO Power Supply. In addition, the pressure value in the Quatro Controller display should start rising and settle within about 5 minutes to the setpoint of 30 mbar. After this, the display should look like

```
Dewar/Vac  Pressure  Gastemp.  Cryostat
  -191.41°C PV:  30mb    19.6°C   20.80°C
   0.005 mb SP:  30mb             20.20°C
   0:00:00  DE CT      StartT          ->
```

The gas heater will not be powered immediately after activating the heaters, but will be delayed by the Start Temperature safety feature until the actual gas temperature has dropped for one time below the Start Temperature Limit which is 0 °C by factory preset.

After having activated the heaters, the system will need some time to build up pressure inside the dewar, thus creating a nitrogen flow inside the gas heater module hose. As the gas heater hose is at the beginning still at ambient temperature, an additional time of about 12 minutes from activating the heaters will be required until the inner gas heater hose gets cold and the temperature at the gas sensor will drop below 0 °C.

The Start Temperature delay feature has two functions:

- to make sure that there is a flow of cold gas through the gas heater to prevent overheating of the gas heater in the case of insufficient gas flow;
- to establish a flow of sufficient cold gas before the heater is activated to prevent strong overshooting of the temperature over the temperature setpoint. This may happen during the start phase if the heater would be activated too early in a state where sufficient cooling power may not be available.

Note:

- The gas heater will only be activated
  - if the Start Temperature feature is passed (gas temperature < Start Temperature Limit for one time)
  - if the gas or sample cell temperature is too low and requires heating to be stabilized or increased with respect to the temperature setpoint.
- Both heaters will not be activated or disabled if one of the Safety Limits is exceeded or a sensor break is detected. In this case the corresponding lights on the QUATRO Controller will flash and a message key word will be shown in the lowest display line.

7. After both heaters have been activated, the temperature should stabilize within a time of about 10 minutes to the temperature setpoint within ±0.5 °C. If the actual temperature differs more than 10 °C from the setpoint or the temperature is close to the system limits, extended stabilization times will be required.

8. In order to shut down the system, select the Power menu as described in item **6** and press the Off menu key below Dewar to disable both the dewar and gas heater. After this, the

QUATRO system may be switched off either at the "Quatro" switch at the QUATRO Power Supply. Finally the vacuum pump should be stopped by the "Vacuum" switch at the QUATRO Power Supply.

Note:

- It should be avoided to remove the sample cell out of the cryostat at temperatures significantly different from ambient. This causes the risk of injury if parts of the cell are touched in a hot or cold state. In addition at low temperatures icing will occur at the lower cell parts which may permanently affect the sensitive electrical electrode isolators.
- It is therefore recommended to adjust the temperature near ambient at the sample cell before removing it out of the cryostat.
- As an alternative, the sample cell may be left in the cryostat for several hours after disabling the heaters until the temperature has relaxed back to ambient.

14.6. Safety Precautions

The following safety precautions should be read carefully before using QUATRO Cryosystem [10].

1. Before connecting the QUATRO Cryosystem, the QUATRO Power Supply or the QUATRO Controller units to line voltage:

• Ensure that the line power voltage indicated on the back of QUATRO Controller and QUATRO Power Supply units and at the vacuum pump matches the actual voltage of your power line.

Caution: Operating instruments at incorrect line voltage may result in permanent device damage, possibly voiding warranty.

• Ensure that all line power cords are connected only to power lines with a protective earth contact. Grounding of any system parts must not be broken.

Warning: The QUATRO Cryosystem, QUATRO Controller and QUATRO Power Supply units and the vacuum pump power cords feature a separate ground wire for exclusive use with grounded power line outlets. If properly installed, the instrument chassis will be connected to protective earth by the power cord ground wire. Failure of this connection may result personal injury or death due to an electric shock.

• Make sure that the "Vacuum" and "Quatro" and "Analyzer" switches at the QUATRO POWER SUPPLY are set to OFF (vertical position).

2. Before activating the dewar or gas heaters, make sure that the system is in a safe state of operation. This means specifically:

• Make sure that an overpressure safety valve is mounted to your dewar and this valve is in a state of proper operation. **A dewar without safety valve is not allowed to be operated by the QUATRO. If a dewar without a safety valve is used, there will be the risk of an explosion with disastrous effects to humans and surroundings.**

• Make sure, that the nitrogen outlet of the cryostat is connected to a tube, conveying the nitrogen out of your lab into open air. **Nitrogen flow through the lab may be dangerous to health and lives of humans or animals due to oxygen deficiency. If the sample mounted in the sample cell contains environment polluting or poisonous materials, the nitrogen outlet has to be filtered appropriately before conveyed into open air.**

• Make sure, that the setpoints for temperature and pressure are adjusted to reasonable values. After activating the heaters, the QUATRO will try to settle to the actual setpoints for temperature and pressure. **Be sure, that your sample and sample cell will not be damaged by the applied temperature. Also consider that the actual temperature may overshoot the selected setpoint.**

3. If the Cryosystem is in operation or has been in operation:

• **Consider that parts of the system may be very hot or cold.** This may especially happen at the **outlet of the cryostat,** the **cryostat** itself, the **sample cell,** the **gas heating module** and the **dewar evaporator.**

• **Touching any of these parts may lead to injuries due to burn or frostbite**.

4. General

• There are no parts inside any system components which require maintenance or adjustments. Therefore no system components should be opened. In any case remove the power cords and any test cables before opening instrument cases or removing any parts from cases.

Warning: Opening of cases or system components or removing parts may expose conductors at high lethal voltages if the system is connected to line voltage.

• Whenever it is likely that the safety of the instrument has been impaired, it should be made inoperative and secured against any unintended operation. Safety could be impaired if

- an instrument case or cable are showing visible damage,
- the instrument fails to perform the intended operation.

Grounding

For safety, a ground connection is essential whenever measurement and control circuits are connected, even if the instrument is switched off. The instrument is grounded if connected to a line power plug equipped with a protective earth contact. The ground connection should be capable of carrying 25 A.

For reproducible measurement results, and for safety of operating personnel, **a proper connection to a protective earth ground** is essential. All sensor and power cables contain shields which are connected to protective earth by the QUATRO Power supply or QUATRO controller units. These shields are essential both for stable measurements and safety of all devices. Do not break any shields and replace cables if any damage is visible.

Ventilation

Some instruments inside the main rack have fan-assisted cooling. Ensure a free flow of air by allowing at least 10 cm clearance between the rear side of the case and the wall. Do not cover the fan outlet or air inlet with any material, since this might cause overheating of internal components.

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