# Alpha-A, Alpha and Beta High Performance Dielectric, Conductivity and Electrochemical Impedance Analyzers

Concepts
Features
Principles of Operation
Application Examples

## Introduction

Novocontrol spectrometers for dielectric, conductivity and impedance material analysis have a world-wide reputation for highest quality and flexibility. Several systems for frequencies from µHz up to GHz are available. Sample cells are offered in standard parallel plate transmission and reflection geometry, four wire contact arrangement and as interdigit comb electrodes. This allows characterization of solid materials, liquids, powders and thin films. Several temperature control systems covering a range from -160°C .. 1300°C are available. Within a modular concept, several impedance analysis systems can be combined with each temperature control system and most of the sample cells. Automatic system and experiment control and sophisticated data evaluation is performed by WinDETA and WinFIT software packages which have established a world-wide standard in dielectric, conductivity and impedance material analysis.

Alpha-A System Set-Up Example

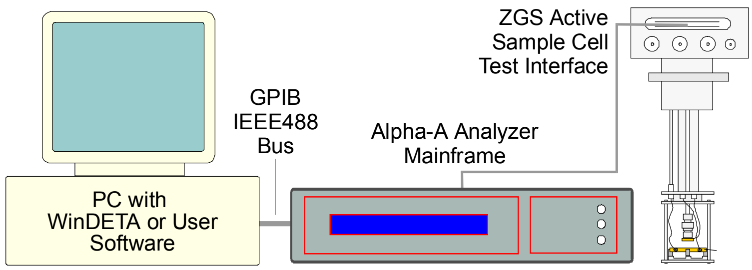


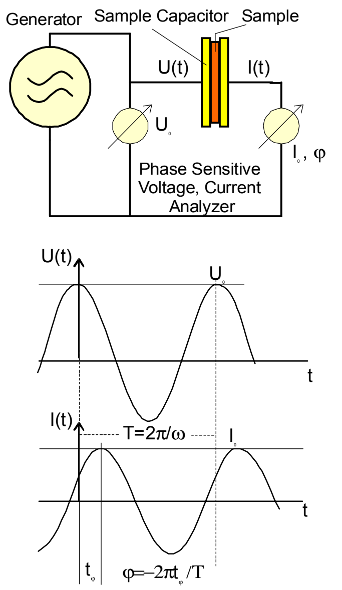

Fig.1. The sample current is converted by the ZGS active sample cell test interface for the Alpha-A mainframe into two voltages from which the sample impedance is evaluated and converted to complex permittivity or conductivity by internal firmware. All system functions are controlled by the Alpha-A mainframe via high level GPIB commands which can be easily integrated in own programs. Optional turn key operation is provided by Novocontrol WinDETA / WinFIT software including data evaluation, visualization and export.

## New approach to material analysis

In the frequency range from 3 µHz .. 20 MHz, the Novocontrol Alpha and Beta analyzer series are used as the base spectrometer component which measures complex impedance of a sample material prepared between two ore more electrodes. The instruments can be used as general purpose precision impedance analyzers too, but are especially designed for dielectric, conductivity, and electrochemical impedance material spectroscopy.

## Alpha and Beta series

The Alpha and Beta analyzers are available since 1998. They are single unit analyzers which combine a series of exceptional features like ultra wide impedance range, frequency range and high accuracy in a fully automated straightforward to handle instrument. By this they have defined a milestone in economical high performance instrumentation. The Beta analyzer has the same functionality as the Alpha analyzer, but has additional high impedance differential voltage inputs for 3 or 4 electrode




$$U(t) = U_0 \cos(\omega t)$$

$$I(t) = I_0 \cos(\omega t + \varphi) =: re(I^* \exp(j\omega t))$$

Fourier Transform over n Periods

$$I^*(\omega) = I' + iI'' = \frac{2}{nT}\int_0^{nT} I(t)\exp(i\omega t)\,dt$$

$$I_0 = \sqrt{I'^2 + I''^2}\,;\ \tan(\varphi) = \frac{I''}{I'}$$

Impedance $Z^*(\omega) = Z' + iZ'' = \frac{U_0}{I^*(\omega)}$

Permittivity

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

$C_0$ Empty Cell Capacity

Conductivity

$$\sigma^*(\omega) = \sigma' - i\sigma'' = \frac{1}{Z^*(\omega)}\frac{d}{A}$$

d Electrode Spacing; A Electrode Area

Fig.2. Principles of impedance and material measurement

measurements as indicated in fig. 3 and tab. 1.

## Alpha-A modular series

The Alpha and Beta analyzers exceptional performance is extended by the new Alpha-A modular series with additional special functionality like e.g., extended voltage and current range, fast measurement rates and DC measurement functionality including potentiostat and galvanostat control functions. As these features for technical and economical reasons are difficult to implement in a single instrument, the Alpha-A system consists of a mainframe unit which can be connected to a series of test interfaces operated by the mainframe.

## Alpha-A versus Alpha and Beta series

The Alpha-A is the successor of the Alpha and Beta analyzer series which are still available as economical alternatives. The Alpha-A system has all functions of the Alpha and Beta series, but supports several test interfaces in addition. With this, the Alpha-A series is our most powerful and flexible system and is recommended for new instrument designs. Basic functions of the three series are listed in tab. 1.

In the following only the common features and technical principles of the Alpha-A, Alpha and Beta series are described. The special functions of the

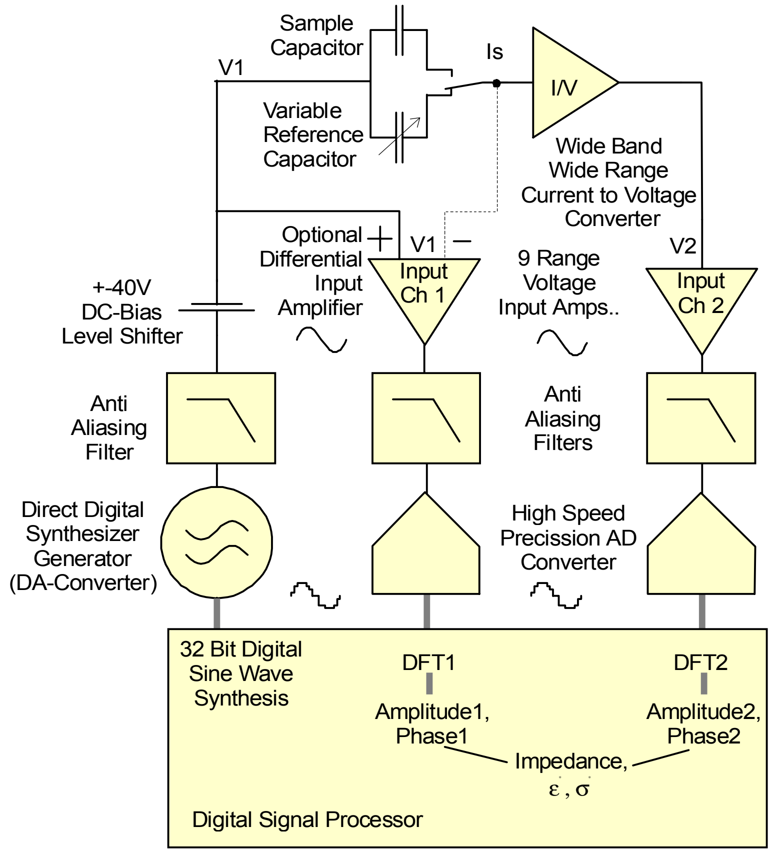



Fig.3. Basic system set-up for Alpha-A, Alpha and Beta analyzers. For details, see text.

Alpha-A test interfaces and the modular concept are described in the Novocontrol technical brochure "Test Interfaces for Alpha-A Modular Measurement System".

## Characterization of low loss dielectrics

Due to the extraordinary high upper impedance limit of > $10^{14}$ $\Omega$ nearly all kind of dielectrics and isolators can be measured even down to very low frequencies below the mHz range. The high accuracy in loss factor tan($\delta$) < $3 \cdot 10^{-5}$ (resolution < $10^{-5}$) provides access to material properties not available until now. Even lowest loss materials used in ceramic capacitors, isolators in power industry or weakest molecular relaxations processes can now be analyzed over a wide frequency range.

## No limitations : High and low conductive materials

In contrast to other systems for dielectric spectroscopy, the Alpha-A, Alpha and Beta analyzers are not limited to high impedance dielectric samples. The lower impedance limit < 0.01 $\Omega$ allows also to analyze conductive samples like semiconductors, electrolytes and electrochemical systems. As the complete impedance range of 16 orders of magnitude is accessible with one device,

| Function | Alpha-A | Alpha | Beta |
|---|---|---|---|
| Dielectric; conductivity, Impedance Sepctroscopy | With additional TI | X | X |
| Gain phase measurements | X | X | X |
| TI support | X | | |
| 3 and 4 electrdoe configurations | with ZG4, POT/GAL 15V10A or 30V2A TI | | X |
| Non linear spectroscopy | X | X | X |
| Active sample cell specifed at the electrodes | with ZGS TI | | |
| High voltage | with HVB1000 or HVB300 TI | | |
| High Current | with POT/GAL 15V10A or 30V2A TI | | |
| Potentiosta, galvanostat functions | with POT/GAL 15V10A or 30V2A TI | | |
| Fast measuremts up to 150 points per second | with mainframe option F | | |
| Dc bias | with option B | with option B | with option B |

Tab. 1. Functions of the Alpha-A, Alpha and Beta analyzer series. TI: Test interface

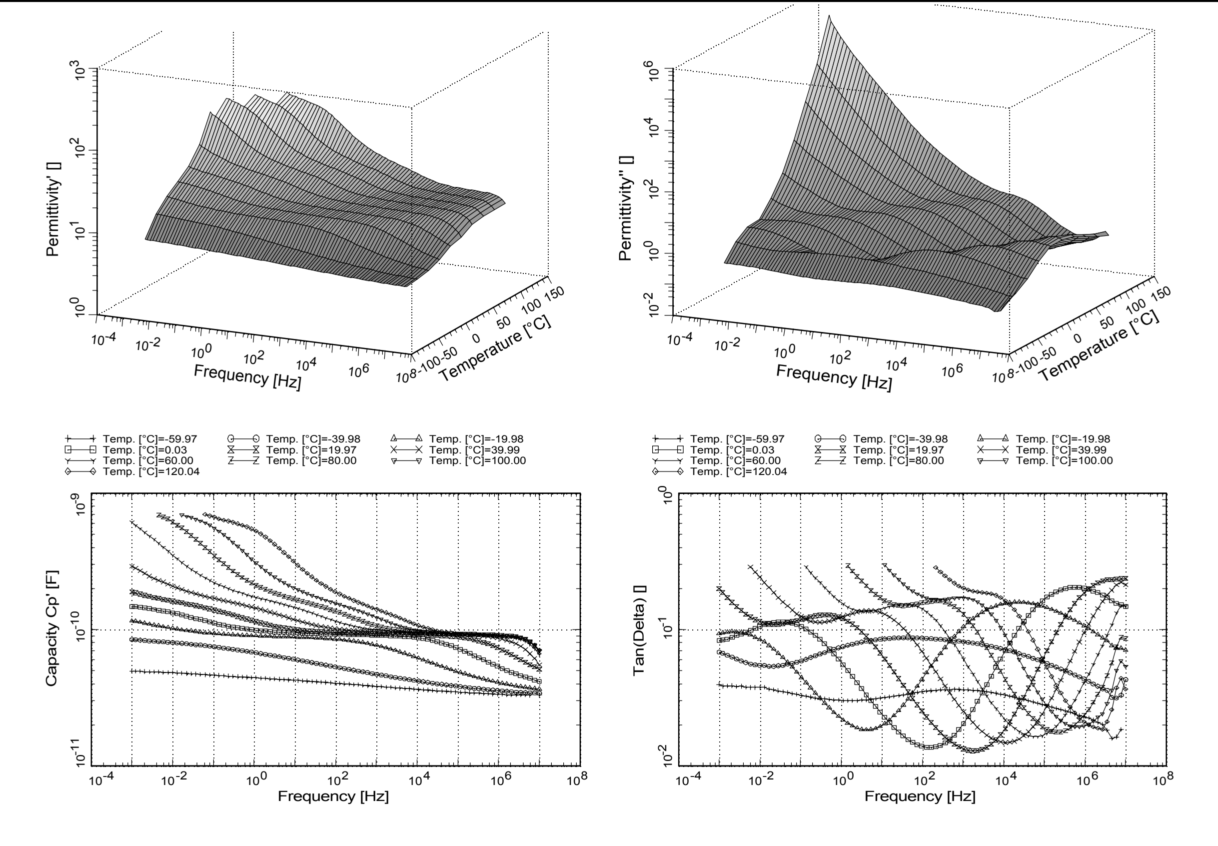



Fig.5. PVDF results, 3D representation of permittivity, 2D diagrams for measured capacity and loss factor tan($\delta$)

even samples with for instance temperature induced metal insulator transitions can be measured.

## Capacities down to 1 fF

The combination of ultra wide impedance and broadband high accuracy results in an exceptional capacity range, too. Especially small capacities in the pF range or below are usually difficult or not at all to measure with most impedance or dielectric analyzers.
The Alpha-A, Alpha or Beta analyzers are specified to measure capacities as low as 1 fF (0.001 pF) within 10 Hz .. 500 kHz. Applications are e.g. broadband characterization of small crystals, measurement of stray capacities or impedance of ill defined electrode geometries like e.g. two needle electrodes separated by 1 cm sample material.

## Specified at the sample position

In the Alpha-A in combination with a ZGS active sample cell test interface, the impedance converter is directly mounted on top of the sample cell connected by rigid lines. This set-up guarantees high accuracy up to 20 MHz and enables optional control of sample temperature. The accuracy specification for material measurements applies to the sample position, offering a turnkey solution without calibration errors due to cable inductance, contacts, stray capacities, grounding and shielding.

## Innovative technology

The Alpha-A, Alpha and Beta analyzers are based on state of the art digital signal processing techniques. Automatic device control including self calibration is provided by Novocontrol MS-Windows software WinDETA. As shown in fig. 2, the basic operation is to create a sine wave at the frequency of interest, apply it to the sample and measure the sample voltage U(t) and result current I(t). From this, the amplitude $I_0$ and phase angle $\varphi$ of the current harmonic base wave component $I^*(\omega)$ is calculated by complex Fourier transform (FT) of I(t). In addition to the phase detection, the FT suppresses all frequency components in I(t) except a narrow band centred around the generator frequency. This improves accuracy and reduces noise and DC drifts by several orders of magnitude. E.g. measuring a signal covered by a noise signal of 1000 times larger is possible. Finally, the impedance $Z(\omega)$ and material parameters $\varepsilon^*(\omega)$ and $\sigma^*(\omega)$ are calculated.

## Digital signal synthesis

Such kind of set-up is implemented in the Alpha series as shown in fig. 3. The optional high impedance differential input amplifier is only included in the Beta series. For the Alpha-A series, fig. 3 has to be separated in two parts. The lower part includes the frequency response analyzer portion from the two input channels down to the signal processor. The upper part with the impedance converter, reference capacitors , differential input amplifier and additional not shown components are realized within several test interfaces. For details refer to tab.1 and the Novocontrol technical brochure "Test Interfaces for Alpha-A Modular Measurement System". In fig. 3. a digital signal processing system is used both

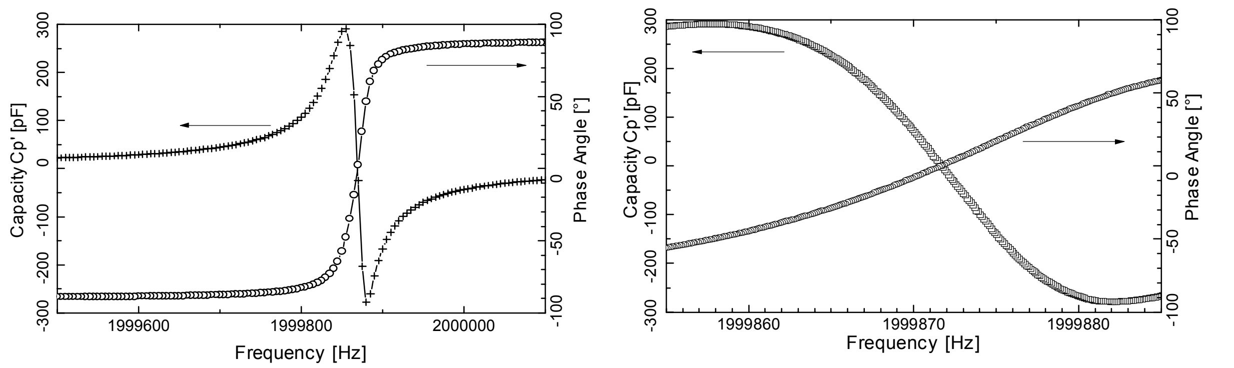



Fig.4 . Dielectric properties of a 2 MHz quartz crystal, half power bandwidth ≅ 30 Hz, 0.1 Hz frequency resolution

for frequency generation and analysis of the input signals. The generator signal is directly digitally synthesized over the entire frequency range. The signal processor continuously calculates with 50 MHz rate digital sine wave values which are transformed into a sine wave voltage. This is done by a high speed digital to analogue (DA) converter followed by a higher harmonic filter. This kind of signal generation guarantees highest stability and a frequency resolution of 32 bit corresponding to e.g. 10 mHz out of 20 MHz (optional 40 MHz).

## Digital signal analysis

The voltages from two independent input channels are amplified, filtered and converted into two digital data streams which are phase sensitive digitally on-line analyzed with respect to their harmonic base waves by discrete Fourier transform (DFT).
Channel 1 measures directly the voltage V1 applied to the sample. The resulting sample current Is is transformed by a wide current - wide frequency range precision impedance converter into the voltage V2 measured by channel 2.

## Reference technique

Most of the signal generation and analysis is performed in the digital system part. Therefore phase and stability errors in this part can be minimized to neglectible values. In order to reach similar high accuracy in the analogue systems components too, the Alpha-A, Alpha and Beta analyzers use a special reference technique. After each direct measured impedance point, the sample is replaced by a precision low loss reference capacitor. The reference measurement includes all linear systematical deviations of the system and therefore can be used in order to eliminate them. This technology in combination with straightforward digital design enables the high accuracy required for material analysis and especially spectroscopy of low loss dielectrics.

## Measurement examples

The performance for low loss materials is shown and discussed in a preceding article of this newsletter. The measurements in Fig. 4 and 5 were done with a Alpha-A analyzer with active sample cell test interface ZGS and a Quatro Cryosystem system for temperature control. In order to demonstrate the overall system performance PVDF /1/ was chosen as a material with not too low losses. Fig. 5 shows the dielectric spectrum for frequencies from 1 mHz .. 10 MHz at several temperatures. PVDF shows an $\alpha$ and $\beta$ relaxation with tan($\delta$) losses in the range 0.01 .. 0.2. At higher temperatures, DC conductivity creates low frequency increase of $\varepsilon''$. As shown in the 3 dimensional diagrams, the data could be measured over the entire frequency and temperature range nearly without artefacts. In order to show the data more detailed, the diagrams for capacity and tan($\delta$) were confined to the range of interest.
For demonstration of the frequency resolution and stability, a commercial high Q quartz crystal used as frequency standards e.g. in clocks and similar kind of electronic devices was measured. The crystal was mounted in the ZGS active sample cell and temperature stabilized to 20°C +-0.01°C. Fig. 4 shows the dielectric properties with the typical resonant behaviour with 5 Hz (left diagram) and 0.1 Hz (right diagram) resolution. The resonance frequency $f_0$ is at 1999871.8 Hz with a half power width of ≅ 30 Hz. Below $f_0$, the device behaves like a 22 pF capacitor with -90° phase shift. With increasing frequency the real part of capacity Cp' and the phase shift increases, too. At $f_0$ the impedance becomes purely resistive indicated by vanishing Cp' and phase shift. Above $f_0$, the impedance changes to inductive behaviour corresponding to 90° phase shift and a corresponding negative Cp'. As shown in fig. 4, even at 0.1 Hz resolution, the measurements are free from significant artefacts and noise. It should be emphasized, that each data point is independently measured without averaging or correlation to neighboured points and that there is no synchronization between the sample crystal and the Alpha internal oscillator.

I/1/ PVDF samples were supplied by Elf Atochem France, Materials Research Lab., Dr. B. Ernst, F-27470 Serquigny

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# Broadband Dielectric Measurement Techniques – Recent Progress

High resolution measurements in the frequency range from $10^{-4}$ Hz – $10^{7}$ Hz are based on frequency response analysis. Recently Novocontrol GmbH launched a new system "Alpha High Resolution Dielectric / Impedance Analyzer". Its performance – especially in comparison to the combination Broadband Dielectric Converter BDC + frequency response analyzer (FRA) - is examined and presented in the following measurements.
As a low loss test sample, an air capacitor mounted in the sample cell of each system was used. It was prepared from parallel gold plated electrodes with 30 mm diameter. The electrode distance was adjusted to 50 µm by two silica spacer rods resulting in about 125 pF capacity.
Due to the conductance of the rods, the capacitor shows especially at ambient temperature remaining losses. By cooling the capacitor to low temperatures, the conductivity of the spacer rods decreases and the losses are continuously reduced.
This kind of capacitor is well suited for testing the tan(□) accuracy and resolution of dielectric analysis systems. Results for three different temperatures are shown in Fig. 1. At -50°C and frequencies above 10 Hz, the capacitor losses are approx. tan(δ) ≈ $10^{-4}$. At lower frequencies tan(δ) increases to about $3 \cdot 10^{-4}$ at 0.1 Hz. This dependency can be measured without significant noise by the Alpha analyzer at frequencies below 100 kHz. The combination BDC + FRA shows the same dependency, but superimposed by about 100% noise due to the system resolution limit tan(δ) of $10^{-4}$ which is in the same order as the sample loss.
If the temperature is lowered to -100°C and -140°C, the capacitor losses decrease further and the better resolution limit of the Alpha analyzer is the limiting factor as shown in the lower two diagrams of Fig. 1. Compared to the BDC + FRA combination, the resolution is improved from $10^{-4}$ to $10^{-5}$. At the high frequency end above 100 kHz, the results show an increase in tan(δ) which is not intrinsic to the sample, but due to high frequency limitations for both systems. At 10 MHz, the accuracy is decreased to about tan(δ) ≈ 0.01 corresponding to about 1° phase accuracy.

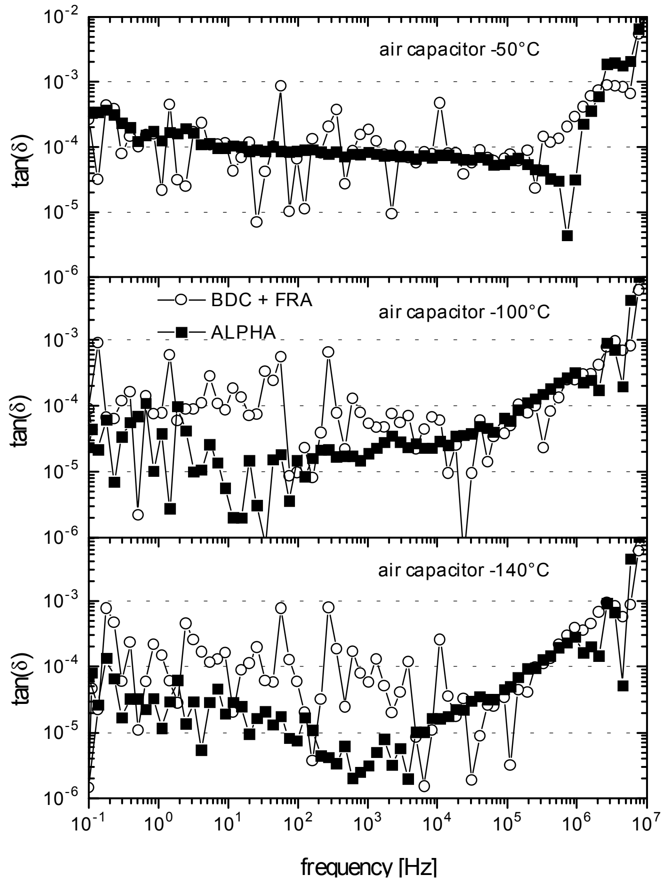


Fig.1. tan(δ) of an air capacitor (thickness: 50 µm, diameter: 30 mm) at different temperatures measured Alpha Dielectric Analyzer (solid squares) and the combination Broadband Dielectric Converter BDC + frequency response analyzer (open circles)

Fig. 2 shows similar measurements for a low loss Polyethylene sample at ambient temperature. The sample capacity is about 160 pF and in good agreement for both systems. The difference of about 1% in the absolute value is due to stray capacities and mounting deviations of the different sample cells. The tan(δ) of the sample is between $10^{-4}$ .. $3{\cdot}10^{-4}$. The results of the two measurement systems show the same behaviour as for the low loss capacitor. Whereas for the BDC + FRA combination the phase resolution limit is reached, the results can be resolved with only 10% scatter by the Alpha. The tan(δ) increase at high frequencies is again due to internal limitations of both systems. This limitation can be seen in the capacity too. The deviations at 10 MHz are about 1.5pF for the Alpha and about 8pF for the BDC + FRA combination.

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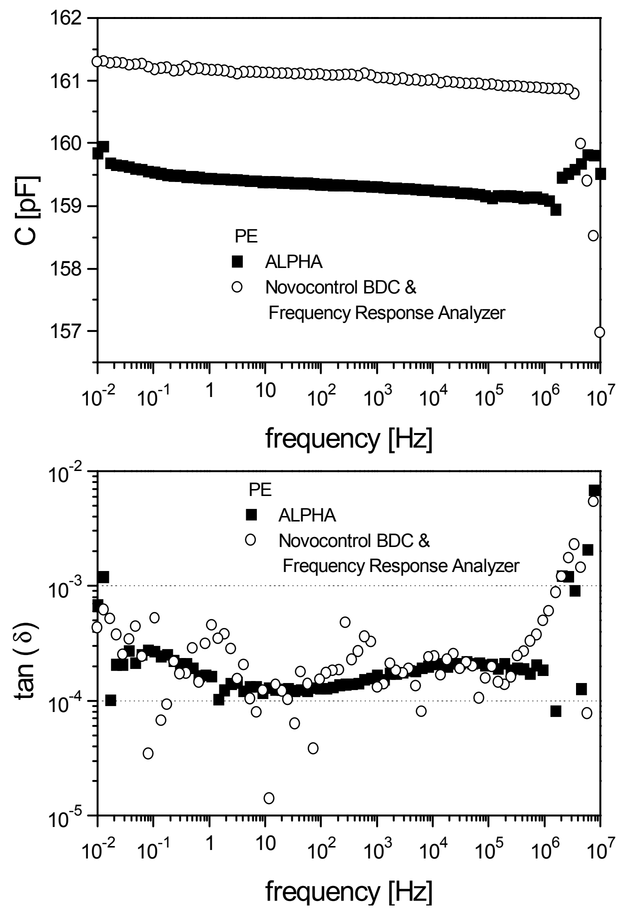


Fig.2. Capacitance C and tan(δ) of a Polyethylene (PE) sample (thickness: 40 µm, diameter: 20 mm) otherwise as Fig. 1.

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