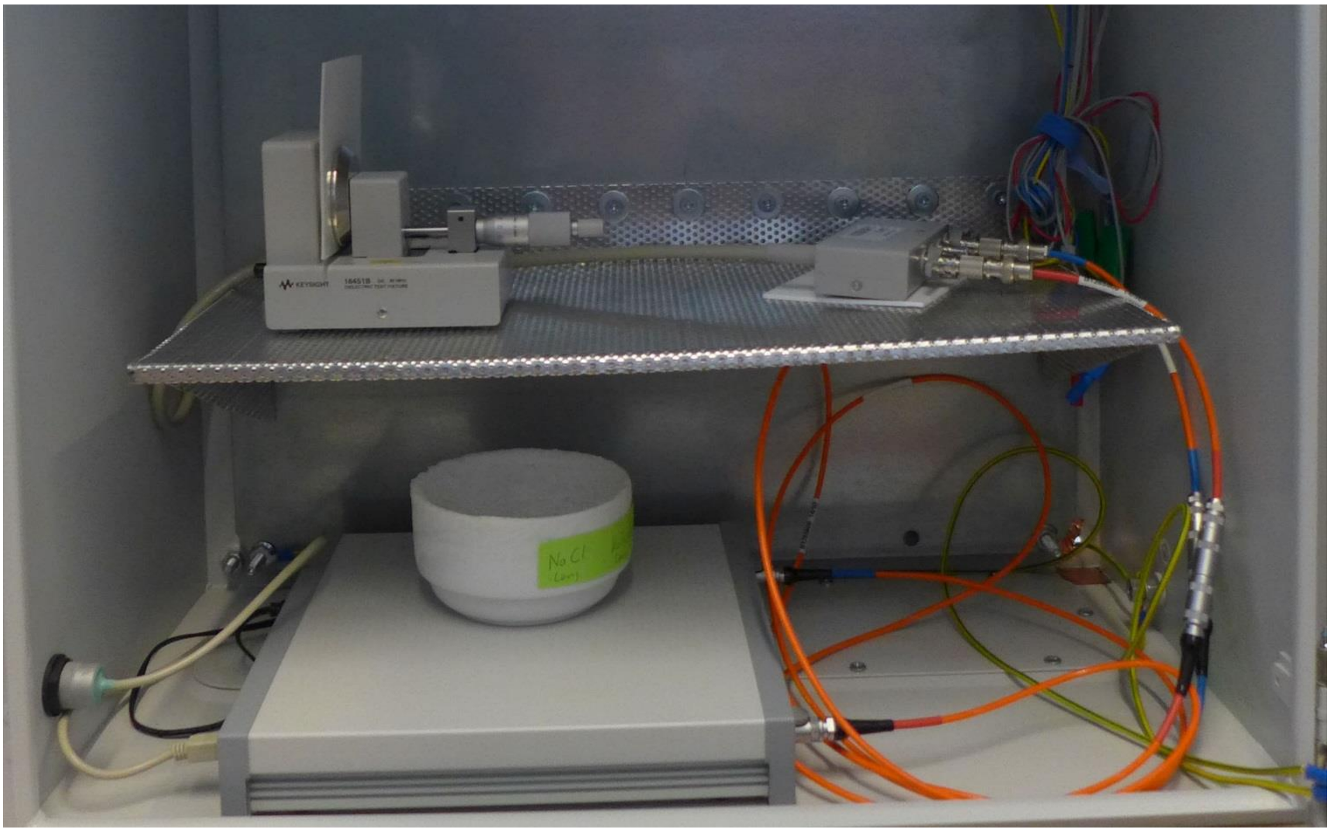
SPECTANO 100 - Application Note

Dielectric Analysis of Solid Insulations

using Dielectric Test Fixture 16451B from Keysight



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Visit www.omicron-lab.com for more information.

Contact support@omicron-lab.com for technical support.

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Note: Basic procedures such as setting-up and adjusting SPECTANO 100 are described in the SPECTANO 100 user manual. You can download the SPECTANO 100 user manual at the SPECTANO 100 download area located at https://www.omicron-lab.com/

Note: All measurements in this application note have been performed with the SPECTANO Analyzer Suite V1.00. Use this version or a higher version to perform the measurements shown in this document. You can download the latest version at the SPECTANO 100 download area located at https://www.omicron-lab.com/

1 Measurement Task

In this document, we show how to measure the dielectric response of different solid insulation materials using SPECTANO 100 the Dielectric Test Fixture 16451B from Keysight.

Various solid standard and composite insulation materials are measured and compared with literature results.

This application note provides only information on how to use SPECTANO 100 together with the Dielectric Test Fixture 16451B to measure the dielectric properties of solid insulation materials. For more information on the analysis of dielectric material curves see our SPECTANO 100 videos on the SPECTANO 100 Knowledge and Applications area located at https://www.omicron-lab.com/

2 Safety Instructions

WARNING

Death or serve injury caused by high voltage or current

Before starting a measurement, read the safety rules and operation and connection instructions in the SPECTANO User Manual and observe the application specific safety instructions in this Application Note when performing measurements to protect yourself from high-voltage hazards.

3 Requirements to perform accurate dielectric material analysis

The measurement error of dielectric properties is not only caused by capacitance measurement errors, but also by

- errors in the solid test material dimensions and structure
- the presence of conductive contaminations or moisture on the test cell or the material sample surface,
- the test cell construction,
- and the measurement setup itself.

Therefore, we recommend to comply with the requirements to perform accurate dielectric material analysis outlined in the application note **Requirements to perform accurate dielectric material analysis**[1].

[1] All SPECTANO 100 application notes and further video tutorials can be found on the SPECTANO 100 *Knowledge and Applications* area located at https://www.omicron-lab.com/.

4 Dielectric Test fixture 16451B from Keysight

Dielectric properties are very important to understand the relationship between the structure and the characteristics of materials. Various test cell types are available on the market that help to analyze important dielectric material parameters such as relative permittivity ε_r, dielectric losses tan(δ), capacitance and impedance.

The Dielectric Test Fixture 16451B from Keysight includes different electrode types which can be replaced depending on the measurement requirements or test sample dimension. The following electrodes are available:

Table 1: Electrode dimensions of Dielectric Test Fixture 16451B from Keysight

	Electrode A	Electrode B	Electrode Unguarded
Electrode type	Disk electrodes	Disk electrodes	Disk electrodes
Guard ring check box	Checked	Checked	Unchecked
Electrode diameter (d)	38 mm	20 mm	56 mm
Guard gap (g)	200 µm	150 µm	

NOTICE

When measuring the dielectric properties of solid materials without guarding, the measurement is influenced by stray capacitances at the edge of the solid test material. Thus, we recommend to use the Dielectric Test Fixture 16451B electrode types

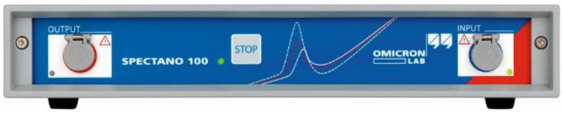
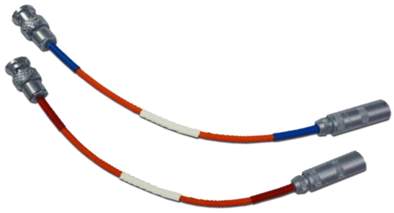
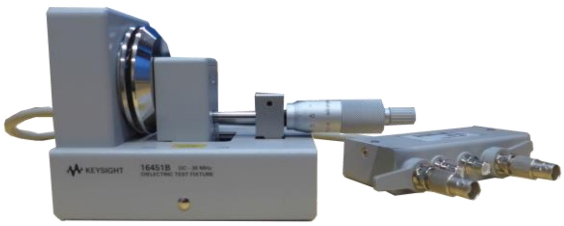
- Electrode A or
- Electrode B

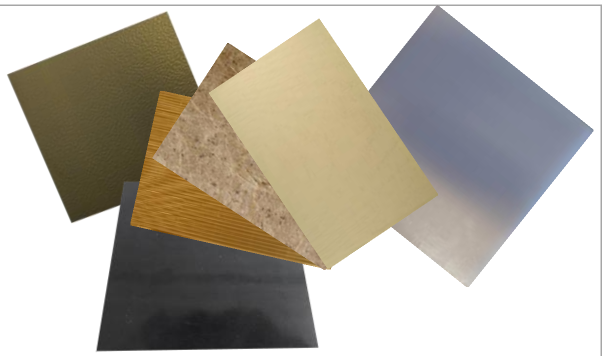
instead of the unguarded electrodes.

The maximum measurement voltage for the Dielectric Test Fixture is
± 42 V peak (AC + DC).

5 Measurement Equipment

The measurement setup consists of the following items:

SPECTANO 100 Dielectric Material Analyzer	
Standard accessories: • Triaxial cable • Grounded DRA supply • USB cable	
Triaxial to BNC plug adapter cable set	
Dielectric Test Fixture 16451B from Keysight used with guarded electrode type **A** **Note**: If disk electrodes without guard ring are used it is recommended to perform an air reference measurement to eliminate influences of unknown capacitances and to improve the measurement accuracy.	

Solid insulation samples with different specifications and dimensions like polymer, rubber, pressboard, aramid paper, oil impregnated paper, resin/epoxy, ESD pad or material components	
Temperature and humidity USB data logger: EasyLog EL-GFX-2+	
EMC chamber or another grounded steel chamber or box **Note:** Using a temperature or climate chamber to shield the test system and to control the humidity and/or temperature is also possible.	
NaCl to control the humidity	

6 Solid Material Analysis with Dielectric Test Fixture 16451B

6.1 Measurement Setup

NOTICE

- Always ground SPECTANO 100 according to the safety rules by using the grounding connector at the rear panel
- Contaminations strongly influence the measurement. For cleaning requirements read the test cell user manual. Always clean dielectric test cells properly prior measurement.

6.1.1 Preparation for Safe Operation

- Before operating SPECTANO 100, ground it as described in the SPECTANO 100 user manual.
- Ensure that the ground is in good condition, clean, and free of oxidation.
- Before handling SPECTANO 100 in any way, ground any parts of the test object that must be connected to ground.
- Always turn off SPECTANO 100 with the power switch before connecting or disconnecting any cables.
- Never remove any cables from SPECTANO 100 or the test object during a test.
- Open a new SPECTANO 100 Analyzer Suite test. Read and follow the instructions.

6.1.2 Hardware Setup

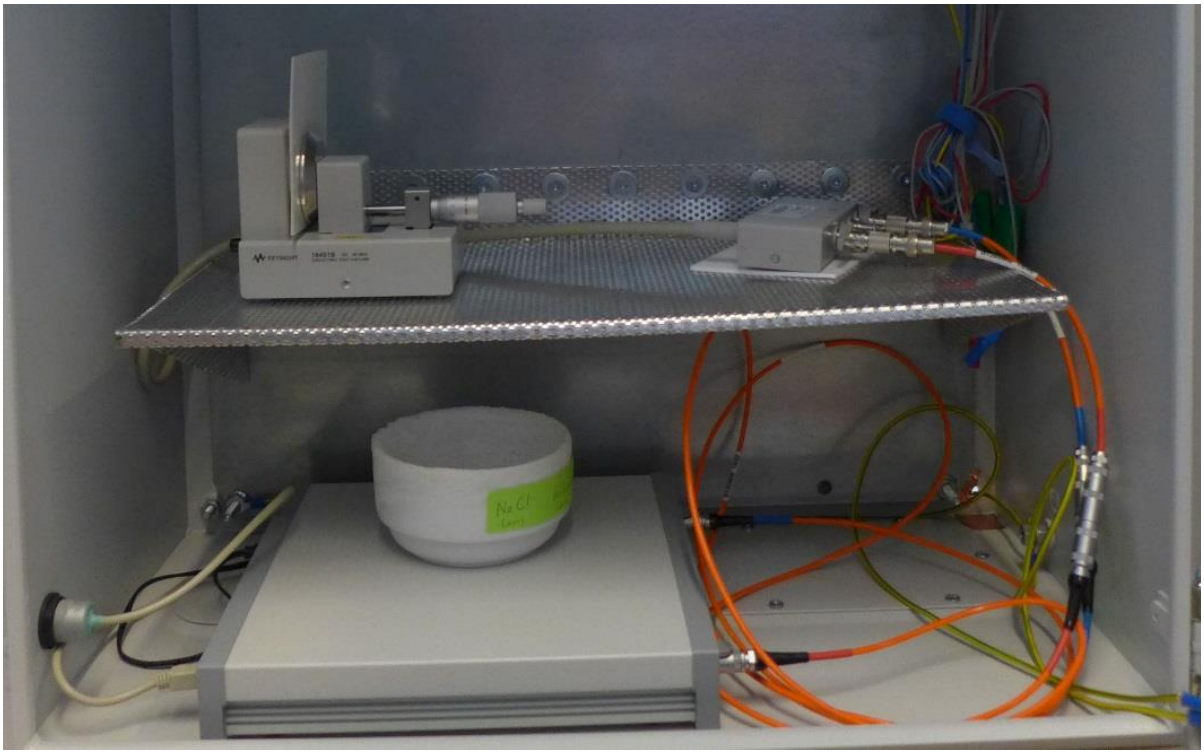


Figure 1: Dielectric material analysis using Dielectric Test Fixture 16451B from Keysight - Setup

To conduct the measurement, perform the following steps:

1. Fix the solid sample probe between the two electrodes
   Ensure that the electrodes are parallel and the sample probe surface has a good contact to the electrodes. For more information about the test cell adjustment and the sample probe preparation read the application note **Requirements to perform accurate dielectric material analysis** [2]

2. Insert the Keysight 16451B Dielectric Test Fixture into the shielding device
   The integration of SPECTANO 100 into the shielding device in addition can improve the measurement accuracy. This is very important for low signal measurements. For more information on small signal measurements and shielding refer to the application note **Requirements to perform accurate dielectric material analysis** [2]

3. Connect the shielding device to ground.

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[2] All SPECTANO 100 application notes and further video tutorials can be found on the SPECTANO 100 *Knowledge and Applications* area located at https://www.omicron-lab.com/.

4 If your shielding device has no temperature and humidity control system, insert the following additional equipment into the shielding device:
   - USB data logger to detect the environmental conditions during the measurement
   - NaCl solution or any other material or equipment to stabilize the humidity in the shielding device
5 Connect the blue and red BNC plug to triaxial plug adapters to the dielectric test fixture
6 Connect the BNC plug adapter cables with the blue and red standard triaxial cable
7 Connect the standard triaxial cables to the SPECTANO 100 OUTPUT and INPUT

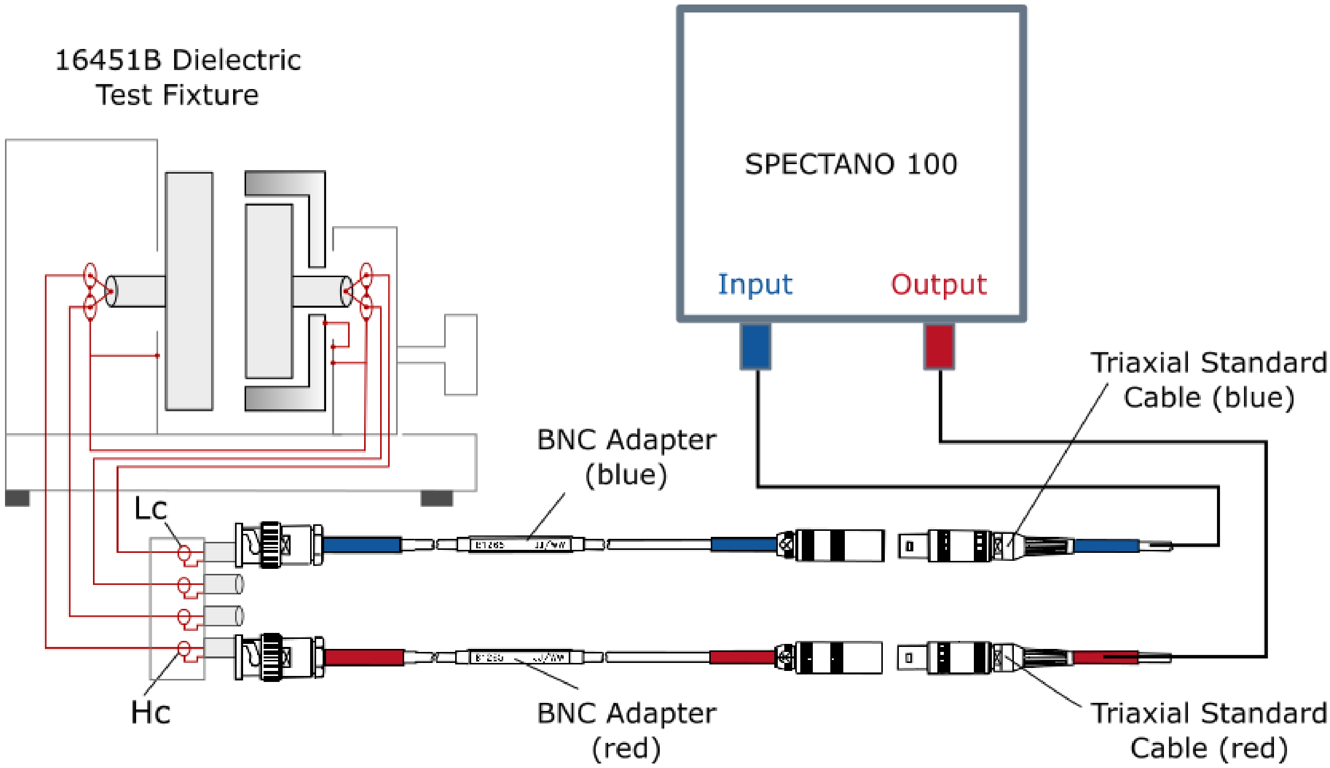


Figure 2: Setup schema using SPECTANO 100 and Dielectric Test Fixture 16451B from Keysight

8 Connect the power supply connector of the grounded DRA power supply to SPECTANO 100.
9 Connect SPECTANO 100 to a PC using the USB cable.
10 Start and setup the software (see chapter 6.1.3)
11 Start the temperature and humidity data logger or temperature/climate chamber

NOTICE
Before starting the measurement, ensure that the temperature in the chamber, test cell and sample itself is stable. According to international standards like ASTM D924-08 the measurement of dielectric properties may be made when the test sample is within ±2.0 °C of the desired temperature.

12 Start the measurement

6.1.3 Software Setup

Open a new SPECTANO 100 Analyzer Suite test.

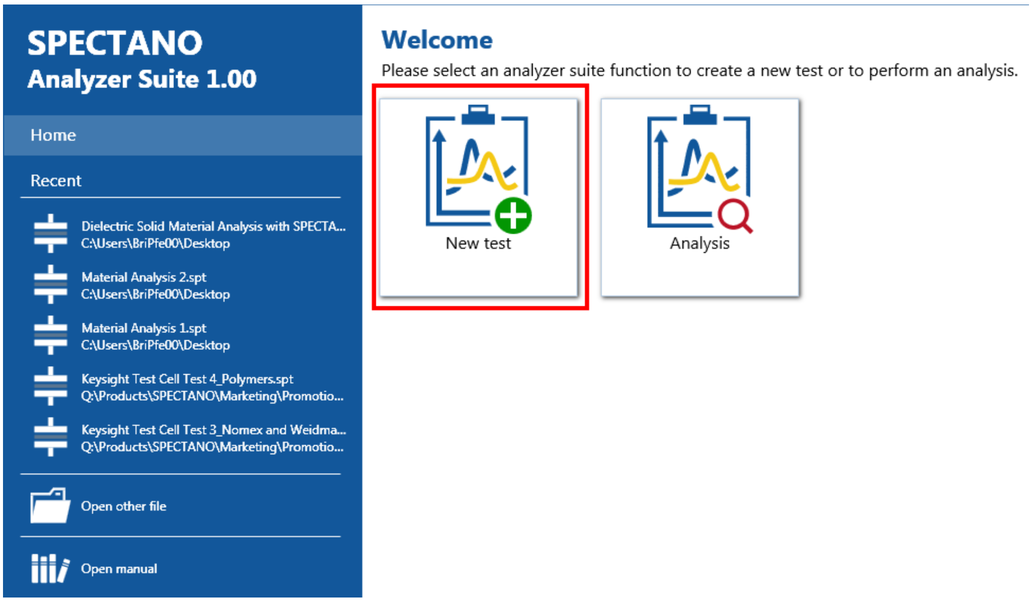


Figure 3: Open new SPECTANO 100 test

To measure the dielectric properties, choose the following setup:

1. Add a measurement for each material, temperature, humidity or measurement voltage you want to analyze.
2. Open the **General test settings** view
3. If you want to perform measurements automatically one after another you can define a waiting time between the measurements.

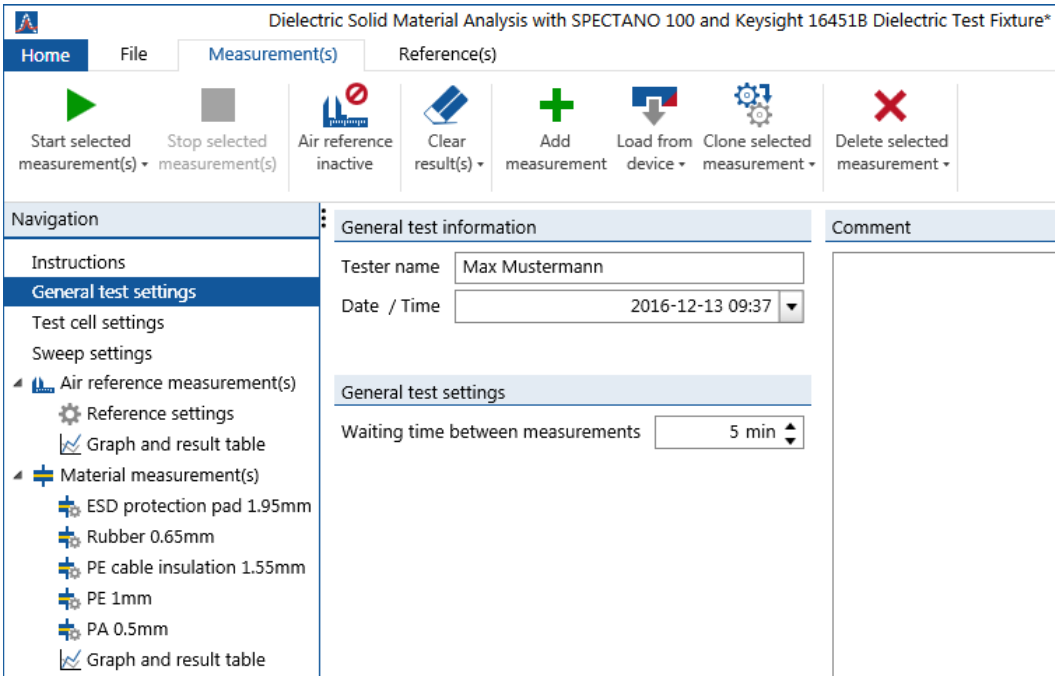


Figure 4: General test settings example for measurement of different polymers and plastics

4 Open the ***Test cell settings*** view.

5 Select the suitable test cell configuration.

The test cell and sample dimensions are needed to calculate the vacuum capacitance c_0 which is further on needed to get the relative permittivity of the dielectric material: $\varepsilon_r = \frac{C_r}{c_0}$

The measurements described in this application note are performed with the guarded electrode type **Electrode A**. Set the following parameter for this electrode type:

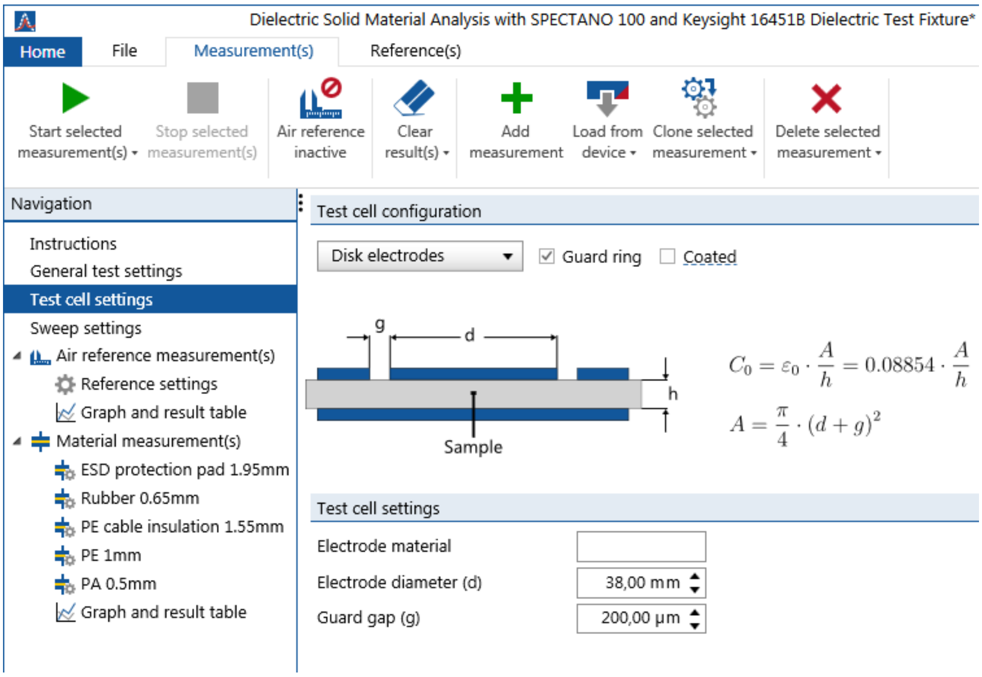


Figure 5: Test cell settings to be selected for Dielectric Test Fixture 16451B - Electrode A

NOTICE

- Select **Coated** if the material surface is coated or an additional thin film electrode is used.
- The Keysight 16451B Dielectric Test Fixture package includes special test fixtures for solid materials with thin film electrodes next to the classical disk. Also, this test fixtures can be modeled with the SPECTANO 100 Analyzer Suite.

6 Open the **Sweep settings** view

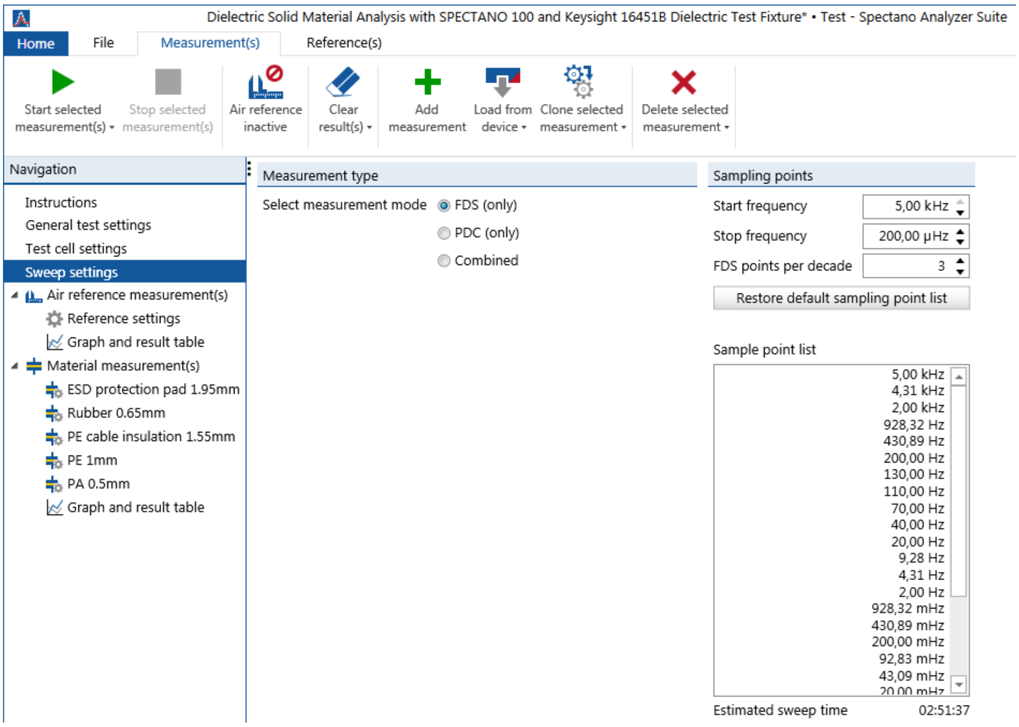


Figure 6: Sweep settings example for measurement of different polymers and plastics

7 Select a measurement mode depending on the material kind and test requirements.

NOTICE

- The examples described in this document are performed with the **FDS only** (Frequency Domain Spectroscopy) mode. See the SPECTANO 100 user manual or the **Software Functionality** section on our webpage https://www.omicron-lab.com/ for more information on this mode and other available measurement modes.
- Independent from the selected measurement mode always **ensure** that the material is **not pre-polarized before starting** a new measurement.

8 Enter your customized frequency sweep depending on the material type and test requirements

NOTICE

The examples described in this application note are performed with a frequency sweep from 5 kHz to 200 µHz with 3 FDS points per decade.

9 We recommend to perform a reference measurement to eliminate influences of unknown capacitances and to improve the measurement accuracy, especially if the used test cell does not have a guard ring. Please follow the reference measurement instructions in the SPECTANO 100 user manual. Afterwards follow the next step.

10 Open the first **Measurement** view

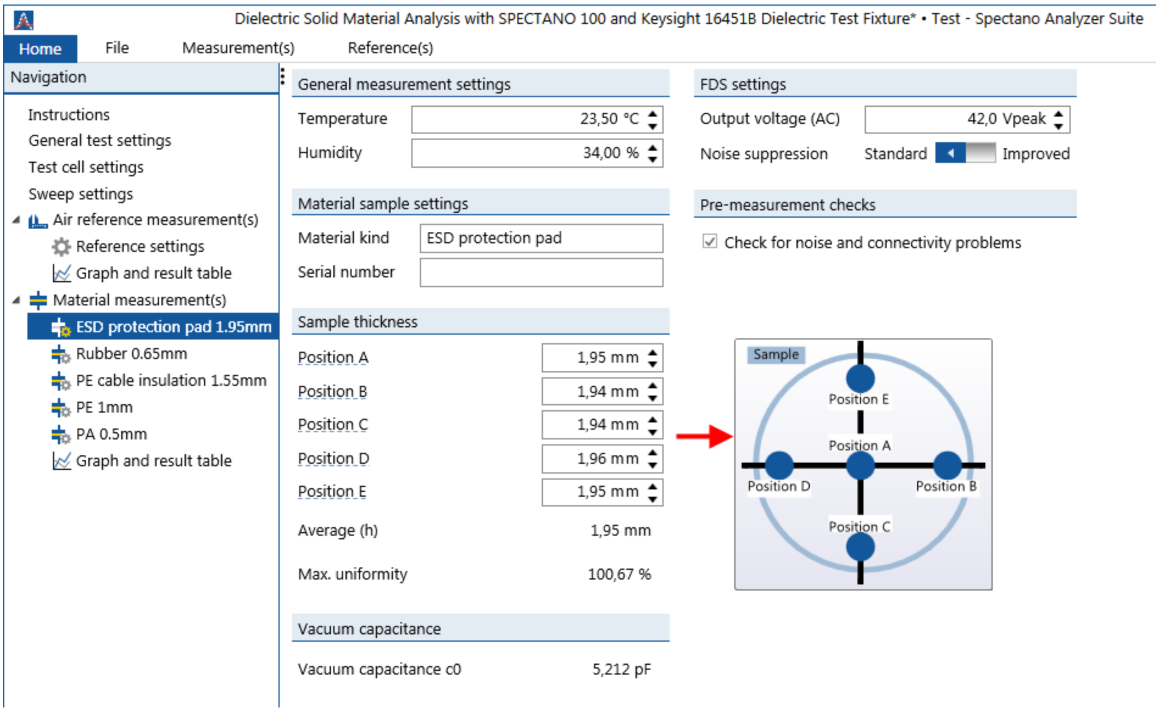


Figure 7: Measurement settings (FDS only mode) example for measurement of an ESD protection pad with 1,95mm thickness

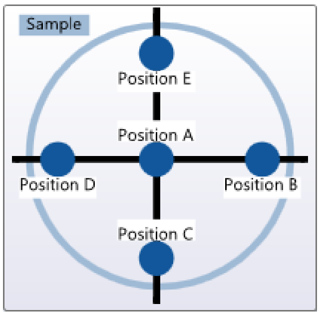
11 Apply the following settings for all measurements:

a. Actual temperature equal to the environmental temperature measured at the beginning of the measurement
b. Actual humidity at the beginning of the measurement
c. Output voltage depending on the material to test and test requirements

> **NOTICE**
> The maximum applicable voltage range for the Dielectric Test Fixture 16451B from Keysight is ± 42 V peak (AC + DC). Higher voltages can damage the test fixture.
>
> The examples described in this application note are performed with 42 V_{peak} (AC).

d. Material kind
e. Sample thickness (h) to the thickness of your sample if available. The vacuum capacitance c_0 will be calculated by the software.

The SPECTANO 100 Analyzer Suite offers entering more than 1 sample thickness if the disk electrode test cell is selected.



With this values the software will inform you if the uniformity of your material is less than ±1% of the average thickness.

According to international standards, the uniformity should be within ±1% of the average thickness to guarantee proper contact between material surface and electrodes.

> **NOTICE**
> Ensure that the sample surface is even and cleaned to guarantee a proper contact to the electrodes.

f. Check the pre-measurement checkbox to detect connectivity problems, quality of the measurement signal, overload or pre-polarization of the sample. Depending on the selected measurement mode we recommend to select the following pre-measurement checks:

Table 2: Pre-measurement checks depending on selected measurement mode

FDS only	**PDC only or Combined**
Pre-measurement checks ☑ Check for noise and connectivity problems	Pre-measurement checks ☑ Check for noise and connectivity problems Delay the start of the measurement until... ☑ The depolarization current falls below 20,00 pA ☐ The time elapsed 120 s

12 Repeat entering the measurement settings for all other measurements in the list.
You can use the **Clone selected measurement** function to create multiple measurements with the same settings.

13 Depending on the test requirements start a single measurement or measurement sequence after the setup for the hardware and software has been finished and checked:

a. For starting a single measurement select a measurement from the **Start single measurement** list

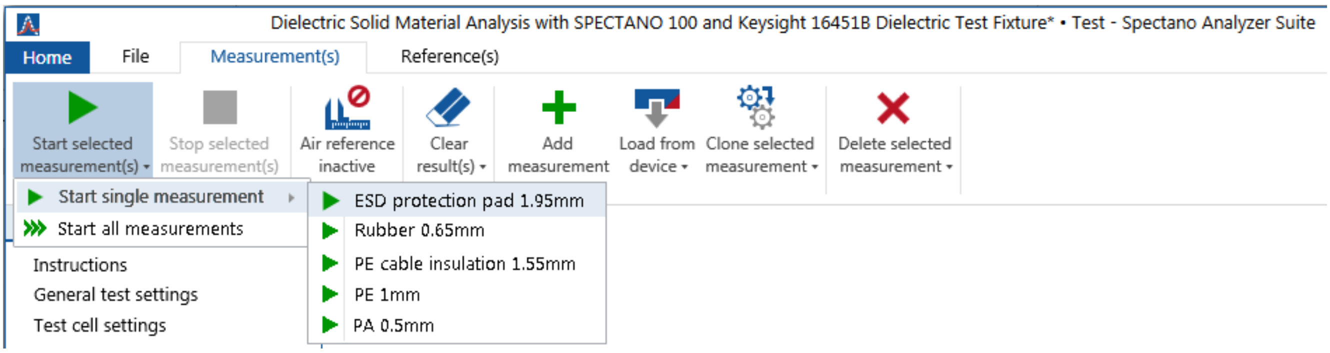


Figure 8: Start single measurement

b. For starting a measurement sequence press the **Start all measurements** button

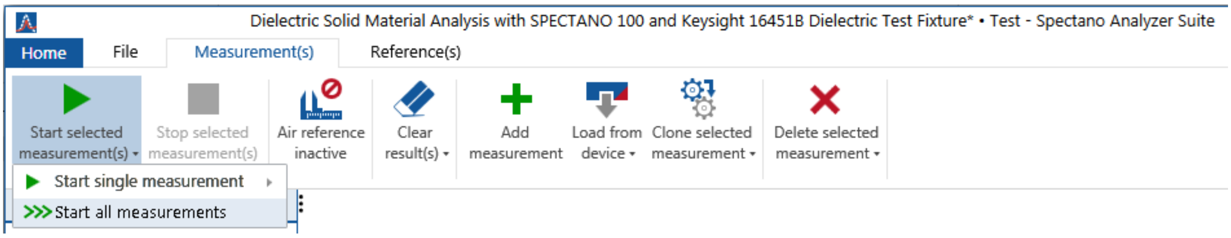


Figure 9: Start measurement sequence

6.2 Measurement Results

6.2.1 Result views

After the measurement is finished the software will display the tan(δ) curve of the sample. If you are interested in other results, please use open the ***Chart result view*** combo box to switch to switch to another result display mode.

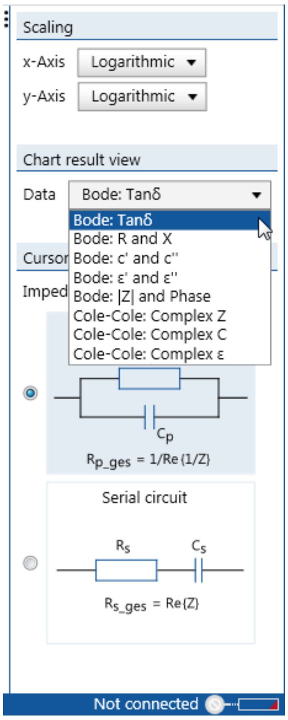


Figure 10: Changing display mode

NOTICE
When the **PDC only** or **Combined** measurement mode is selected the polarization and depolarization current results over time can be displayed in addition.

Figure 11 shows the measurement results table. Here you can enter the desired frequency and the according results will be displayed. Alternatively, you can move the cursors in the result diagram.

	Frequency	Tanδ	ε'	-ε''	|Z|	Phase	Cp	Rp	c'	-c''	R	-X
Insulation oil @25°C	2 Hz	0,0002185	2,11E+00	4,6E-04	581,507 MΩ	-89,987 °	136,8471 pF	2,661 TΩ	136,8471 pF	0,0299 pF	127,054 kΩ	581,507 MΩ
Insulation oil @50°C	2 Hz	0,0003393	2,09E+00	7,1E-04	585,322 MΩ	-89,981 °	135,955 pF	1,725 TΩ	135,955 pF	0,0461 pF	198,602 kΩ	585,322 MΩ
Insulation oil @75°C	2 Hz	0,0004507	2,08E+00	9,36E-04	589,592 MΩ	-89,974 °	134,9704 pF	1,308 TΩ	134,9704 pF	0,0608 pF	265,729 kΩ	589,592 MΩ

Figure 11: Result table view

The screenshots in the following chapters show results for different solid insulation material samples measured with SPECTANO 100 and the Dielectric Test Fixture 16451B from Keysight.

6.2.2 FDS measurement results of different polymers

Error! Reference source not found. shows the measured tan(δ) curves over frequency. Tan(δ) represents the relative loss of a dielectric material and is equal to the ratio of lost energy to stored energy.

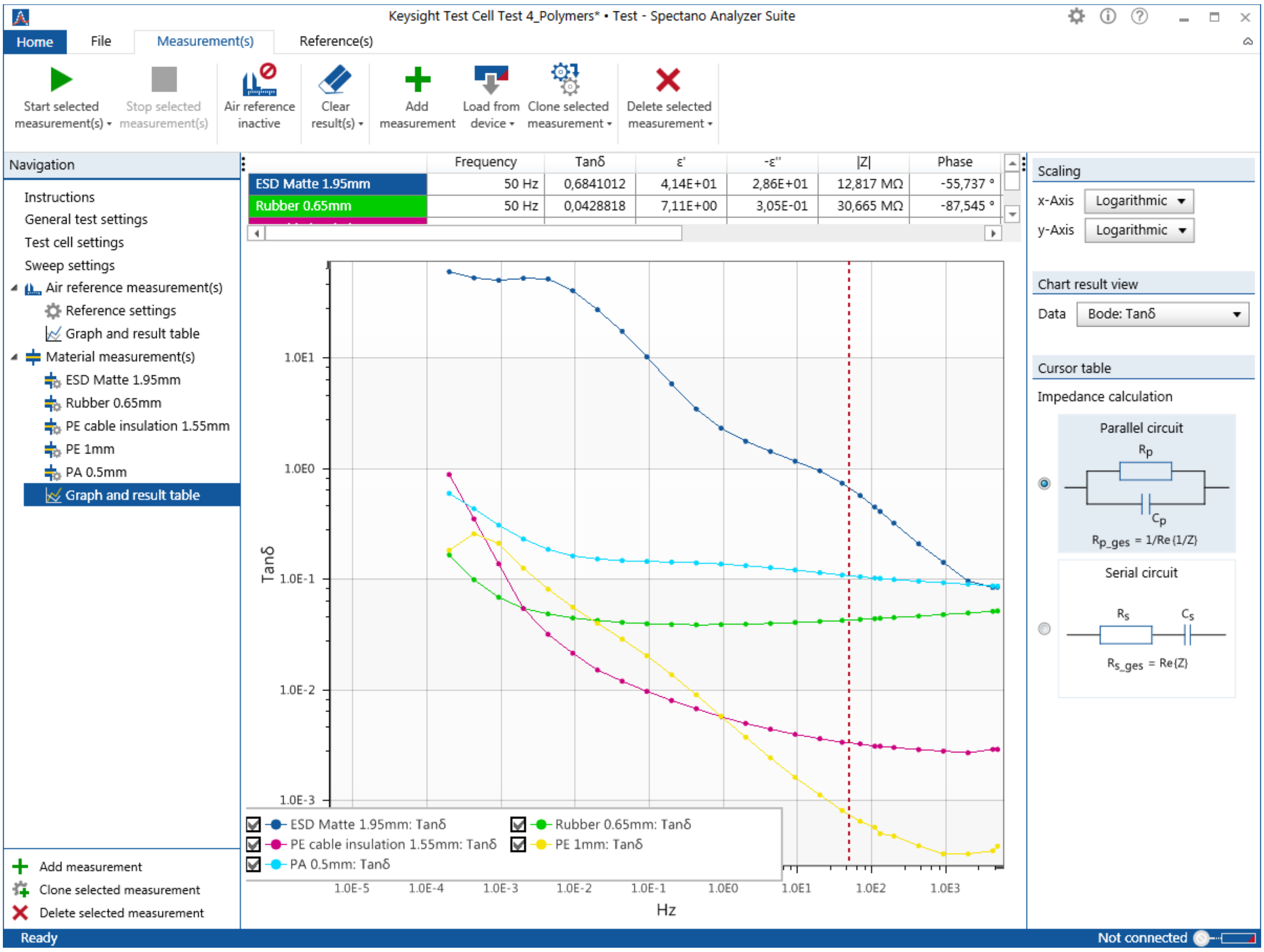


Figure 12: Tan(δ) example curves of different polymers

The curves shown in **Error! Reference source not found.** the relative permittivity ε_r over frequency. ε_r' is displayed on the primary and ε_r'' on the secondary y-axis.

NOTICE

- ε_r' indicates how much energy from an external electric field is stored in a dielectric material
- ε_r'' indicates the losses within the dielectric material when an external electric field is applied.

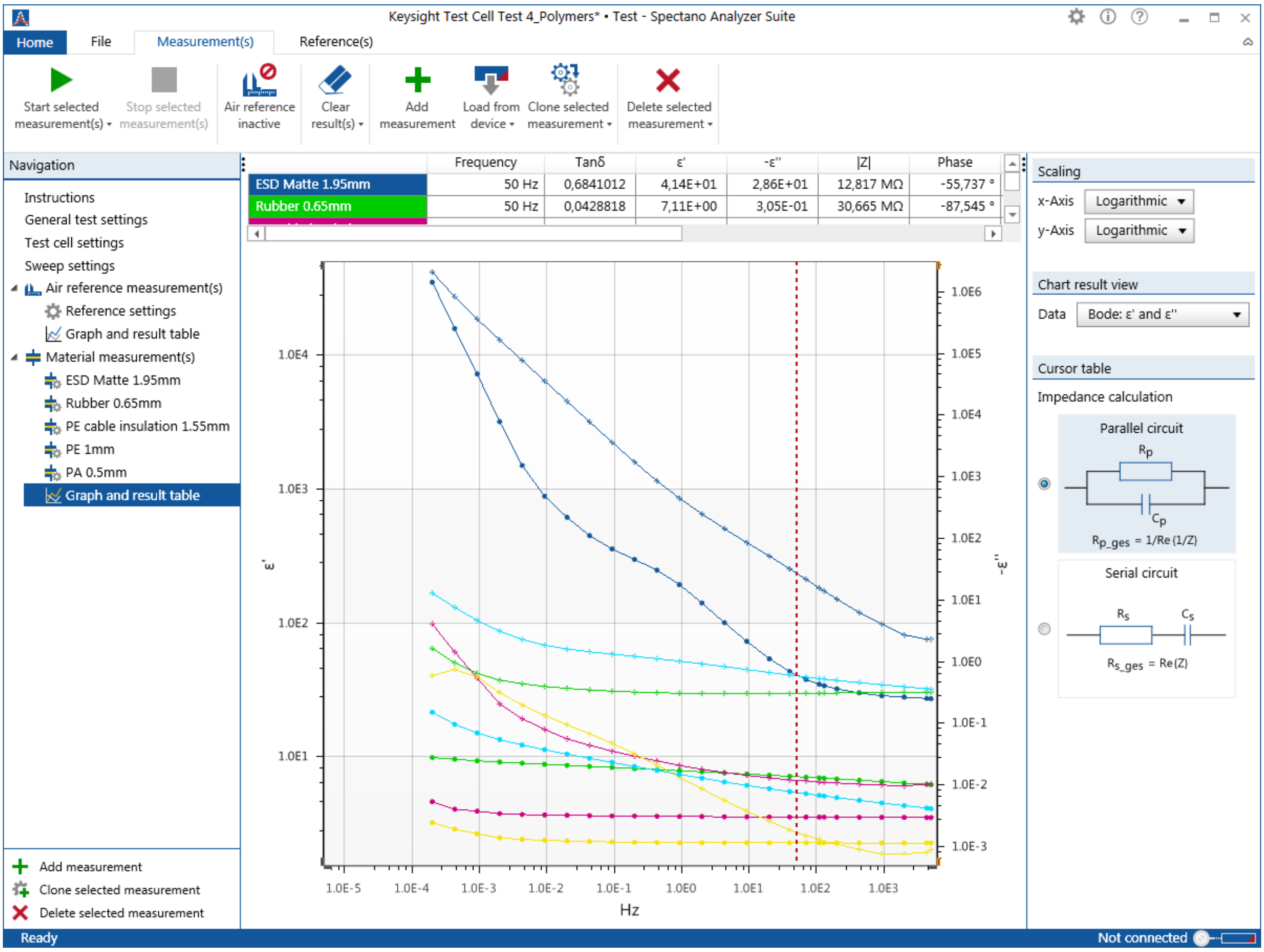


Figure 13: Relative permittivity example curves of different polymers

The measured values of tan(δ) and the permittivity meet the expected measurement results.

6.2.3 FDS measurement results of different aramid and insulation papers

Figure 14 shows the measured tan(δ) curves over frequency. Tan(δ) represents the relative loss of a dielectric material and is equal to the ratio of lost energy to stored energy.

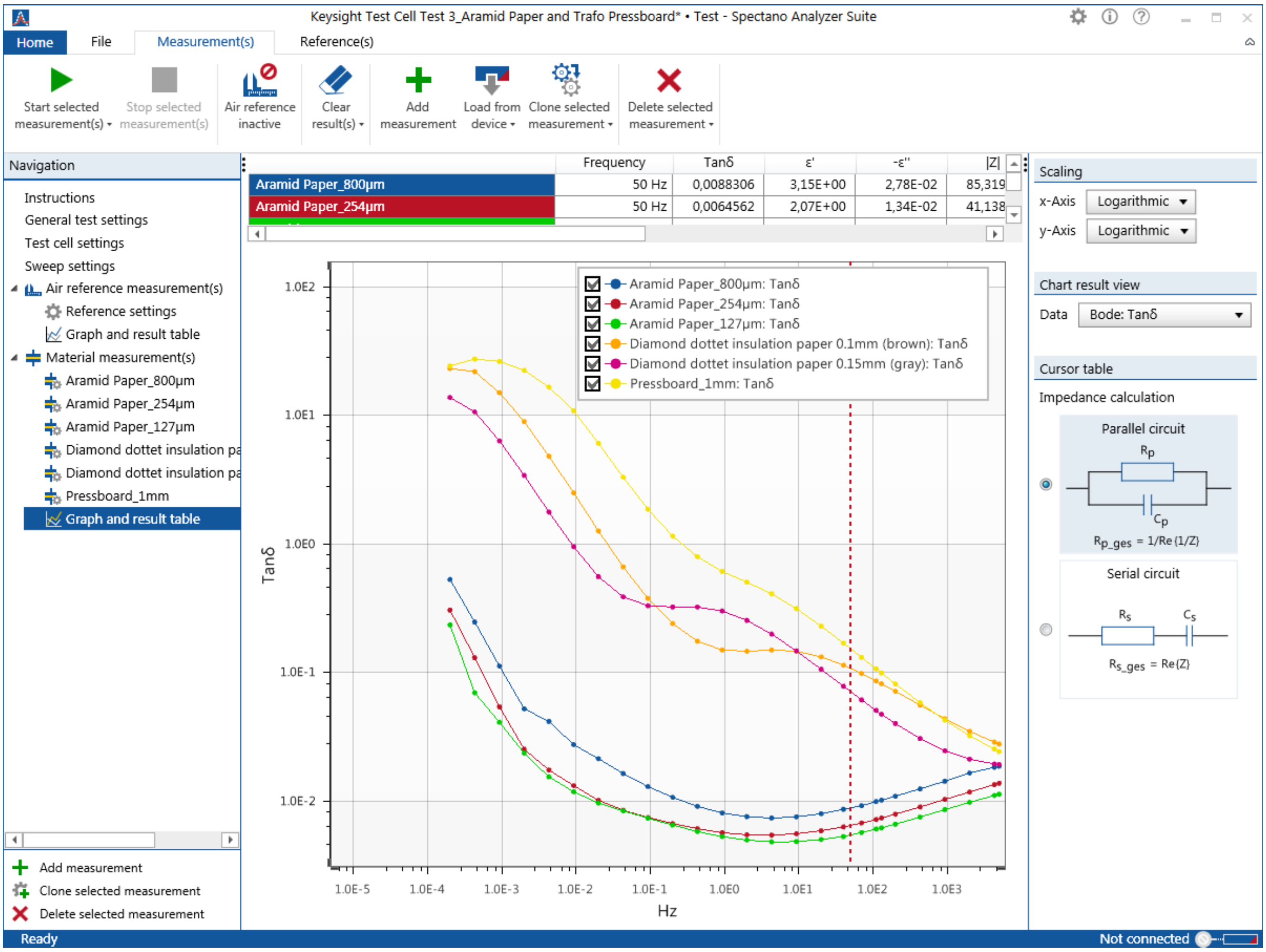


Figure 14: Tan(δ) example curves of different aramid and insulation papers

The red, green and blue graphs (aramid paper results) are typical curves for cellulose.
The pink, orange and yellow curves are typical for diamond dotted insulation paper. Diamond dotted paper is not consisting of pure cellulose. It is a composite material consisting of paper coated with epoxy resin and thus will not show a typical cellulose curve.

6.2.4 FDS measurement results of oil impregnated pressboards

Figure 15 shows the measured tan(δ) curves over frequency. Tan(δ) represents the relative loss of a dielectric material and is equal to the ratio of lost energy to stored energy.

When a dielectric material is placed in an electric field, charges slightly shift from their stable positions causing dielectric polarization. Dielectric loss result from polarization processes within molecules and results a hump in the result curve. Different dielectric layers with different permittivity and conductivity in one material (like for example in oil impregnated press boards) can lead to accumulations of the charge carriers at the surfaces of the dielectric layers. This polarization is called interfacial/space charge polarization. The field distortion caused by the accumulation of these charges increases the overall capacitance of a material which appears as an increase in ε_r'. This phenomenon is also visible in the tan(δ) chart as hump as shown in
Figure 15 for example for the blue curve between 95 µHz and 1mHz.

The curves indicate that interfacial polarization occurs between the two material components oil and pressboard.

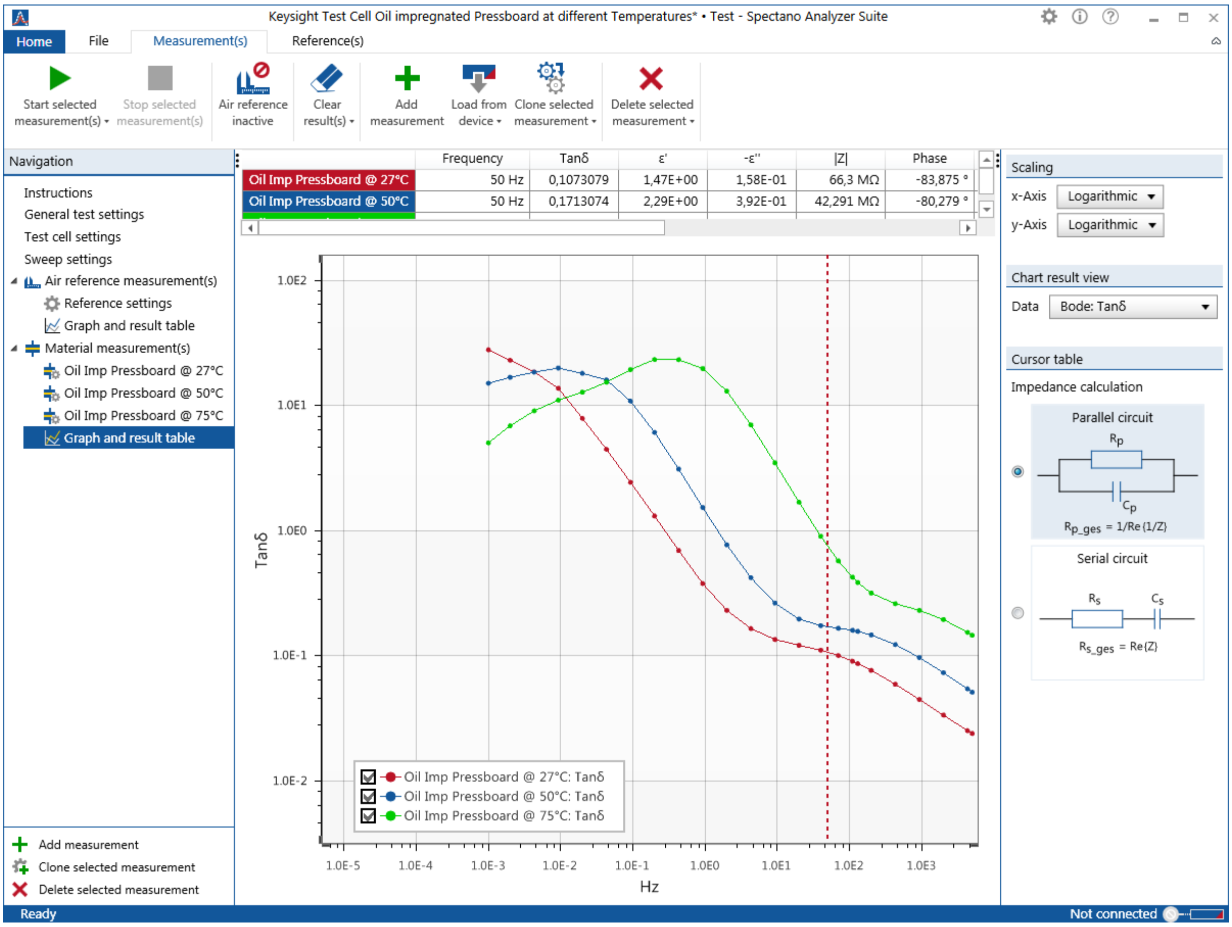


Figure 15: Tan(δ) example curves of oil impregnated paper

NOTICE

The impregnation of the pressboard with oil and the test itself is not made under vacuum. Thus, the test result can be influenced by air molecules like oxygen. However, this example shows that in principle oil impregnated paper can be analyzed using SPECTANO 100 together with the Keysight 16451B Dielectric Test Fixture.

7 Summary

Some test cells on the market like the solid test fixture of Keysight are made for a frequency range from 20 Hz to 120 MHz but this application note illustrates that the Dielectric Test Fixture 16451B can also be used at much lower frequencies with the OMICRON Lab's Dielectric Material Analyzer SPECTANO 100.

The results obtained with SPECTANO 100 and the Dielectric Test Fixture16451B were reproducible and consistent under the same environmental and test conditions. The measured results for the different materials are within the expected value range measured at 50 Hz and room temperature:

Table 3: Examples: Polymer literature relative permittivity values in comparison to measured values

Material	Permittivity literature value at 50 Hz [3]	Measured permittivity value at 50 Hz [4]
Polyethylene	2.2 – 2.4	2.28
Paper (Dry)	2.0	Diamond dotted paper: 2.47 [5]

NOTICE

To compare the measured results with results from literature always ensure that your material under test is a standard material or has the same material composite and structure. The dielectric parameters depend on the material structure and composite. Further on, the environmental conditions must be the same. The results of some materials are highly influenced by temperature, pressure or humidity.

SPECTANO 100 and its accessories and tools offers all features and functionalities needed to measure liquid dielectric materials. By using appropriate material samples the SPECTANO 100 and its accessories offers a powerful platform for the dielectric material analysis of a high variety of liquid insulation materials.

[3] Published: http://www.clippercontrols.com/pages/Dielectric-Constant-Values.html

[4] Measured with SPECTANO 100 and Dielectric Test Fixture 16451B from Keysight

[5] The humidity during the measurement was 34% therefore the paper was not 100% dry which leads into the small deviation of 0.47 in comparison to the permittivity value provided by the literature.

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