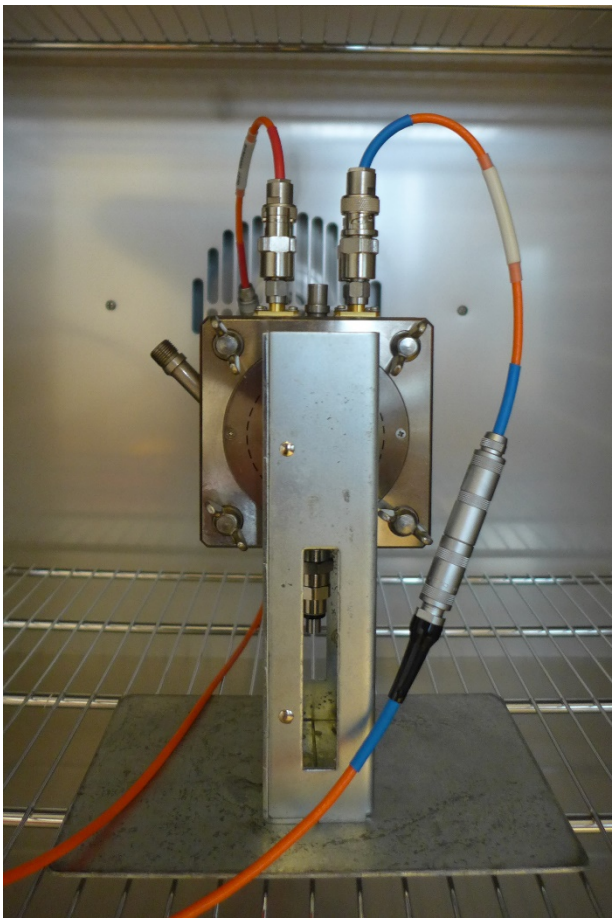
SPECTANO 100 - Application Note

Dielectric Analysis of Insulation Liquids

using Liquid Test Fixture 16452A from Keysight



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

Table of Contents

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 from 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 from 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 liquid insulation materials using SPECTANO 100 and the Liquid Test Fixture 16452A from Keysight.

This application note provides only information on how to use SPECTANO 100 together with the Liquid Test Fixture 16452A to measure the dielectric properties of insulation liquids. 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 document 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

- 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 Liquid Test Fixture 16452A 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 Liquid Test Fixture 16452A from Keysight includes spacers of different thicknesses to cover different filling volumes. The spacers can be replaced depending on the measurement requirements or test volume. The following spacers are available:

Table 1: Spacer of Liquid Test Fixture 16452A from Keysight

Spacer thickness	1.3 mm	1.5 mm	2.0 mm	3.0 mm
Necessary liquid volume in ml	34 ml	38 ml	48 ml	68 ml
Electrode gap[2]	0.3 mm	0.5 mm	1.0 mm	2.0 mm

The measurements described in this application note are performed with the **spacer of 1.3mm thickness**.

NOTICE

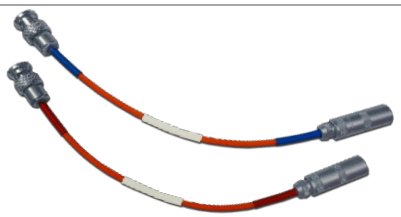
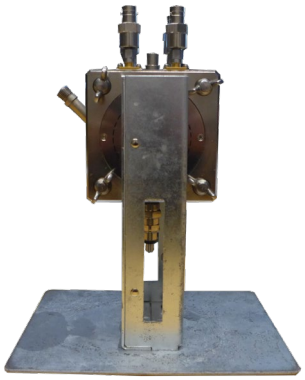
The optimum spacer thickness depends on the conductivity of the liquid under test. For highly insulating liquids use a smaller spacer or a higher measurement voltage.

The maximum measurement voltage for the Liquid Test Fixture 16452A from Keysight is 30 Vrms.

[2] The liquid test fixture 16452A from Keysight is a test cell with disk electrodes without GUARD ring. The defined electrode gap equates to the sample thickness value to be entered in the SPECTANO Analyzer Suite.

5 Measurement Equipment

The measurement setup used in this application note 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	
Liquid Test Fixture 16452A from Keysight with 1.3mm spacer **Note**: We recommend to perform an air reference measurement to eliminate influences of unknown capacitances and to improve the measurement accuracy, because this test fixture with disk electrodes does not have a guard ring.	

Insulation liquid samples **Note:** For this application note we used silicone oils from Wolfgang Spielberger e.K. (polydimethylsiloxane)	
Temperature chamber FD115 from Binder **Note:** For the analysis of insulation fluids it is always important to stabilize the environmental and measurement temperature using a temperature or climate chamber	

6 Silicone Oil Measurement with Liquid Test Fixture 16452A

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.
- Bubbles (e.g. by improper probe handling) in liquid insulation materials should be avoided. Entrapped air or bubbles influence the measurement significantly.

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 which have to 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 first. Read and follow the instructions.

6.1.2 Hardware Setup

Figure 1: Dielectric material analysis using the Liquid Test Fixture 16452A from Keysight - Setup

Figure 2: Dielectric material analysis using the Liquid Test Fixture 16452A from Keysight - Connection

To conduct the measurement, perform the following steps:

1 Fill the insulation liquid sample into the Liquid Test Fixture 16452A from Keysight.

NOTICE

Ensure that the test cell is cleaned and the sample is prepared before filling. For more information about cleaning, preparation and filling a test cell for liquid analysis and sample see

- International standards for dielectric analysis of liquids like ASTM standard D924-08
- Application note **Requirements to perform accurate dielectric material analysis** [3]

2 Connect the SMA-BNC Adapters to the Liquid Test Fixture 16452A from Keysight like described in the test cell user manual
3 Insert the Liquid Test Fixture into the temperature or climate chamber
4 Connect the blue and red BNC plug to triaxial plug adapters to the Liquid Test Fixture 16452A from Keysight
5 Connect the BNC plug adapter cables with the blue and red standard triaxial cable
6 Connect the standard triaxial cables to the SPECTANO 100 OUTPUT and INPUT

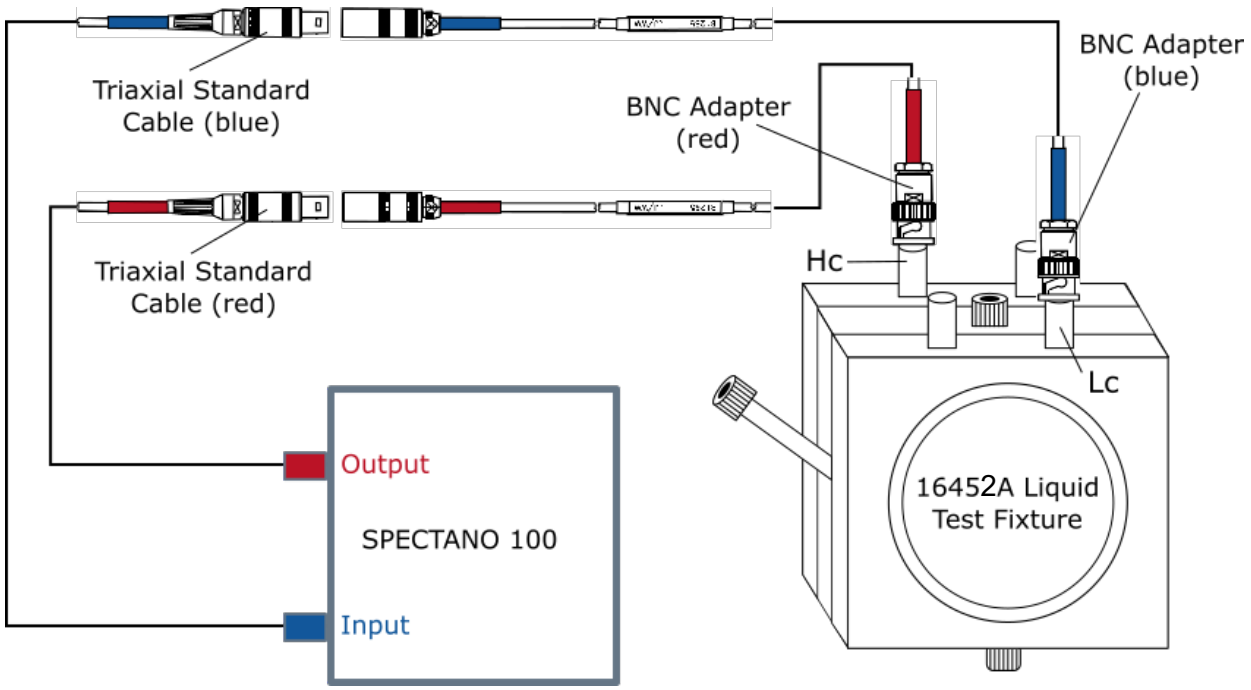


Figure 3: Setup schema using SPECTANO 100 and Liquid Test Fixture 16452A from Keysight

[3] 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/.

7 Connect the power supply connector of the grounded DRA power supply to SPECTANO 100.
8 Connect SPECTANO 100 to a PC using the USB cable.
9 Start and setup the software (see chapter 6.1.3)
10 Start the temperature or 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.

11 Start the measurement when the temperature of the test sample is stable

6.1.3 Software Setup

Open a new SPECTANO 100 Analyzer Suite test.

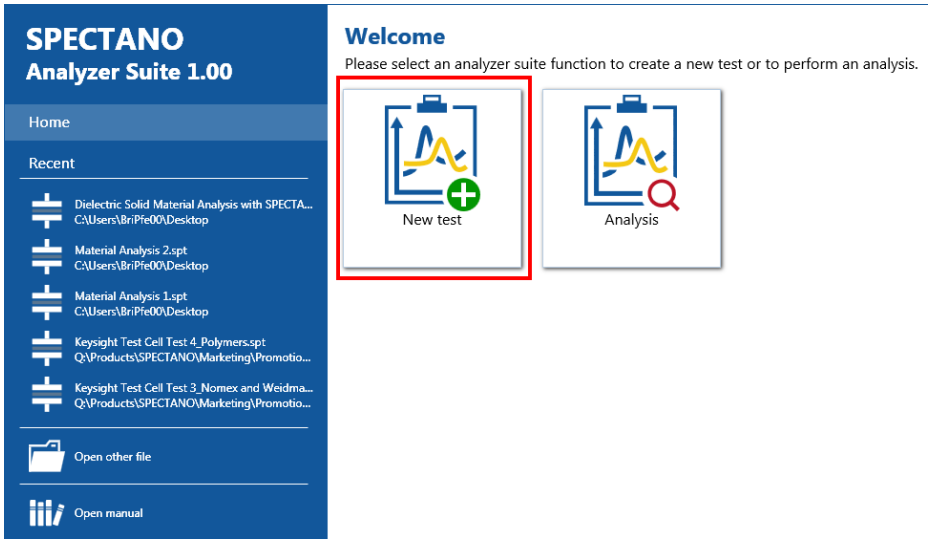


Figure 4: Open new SPECTANO 100 test

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.

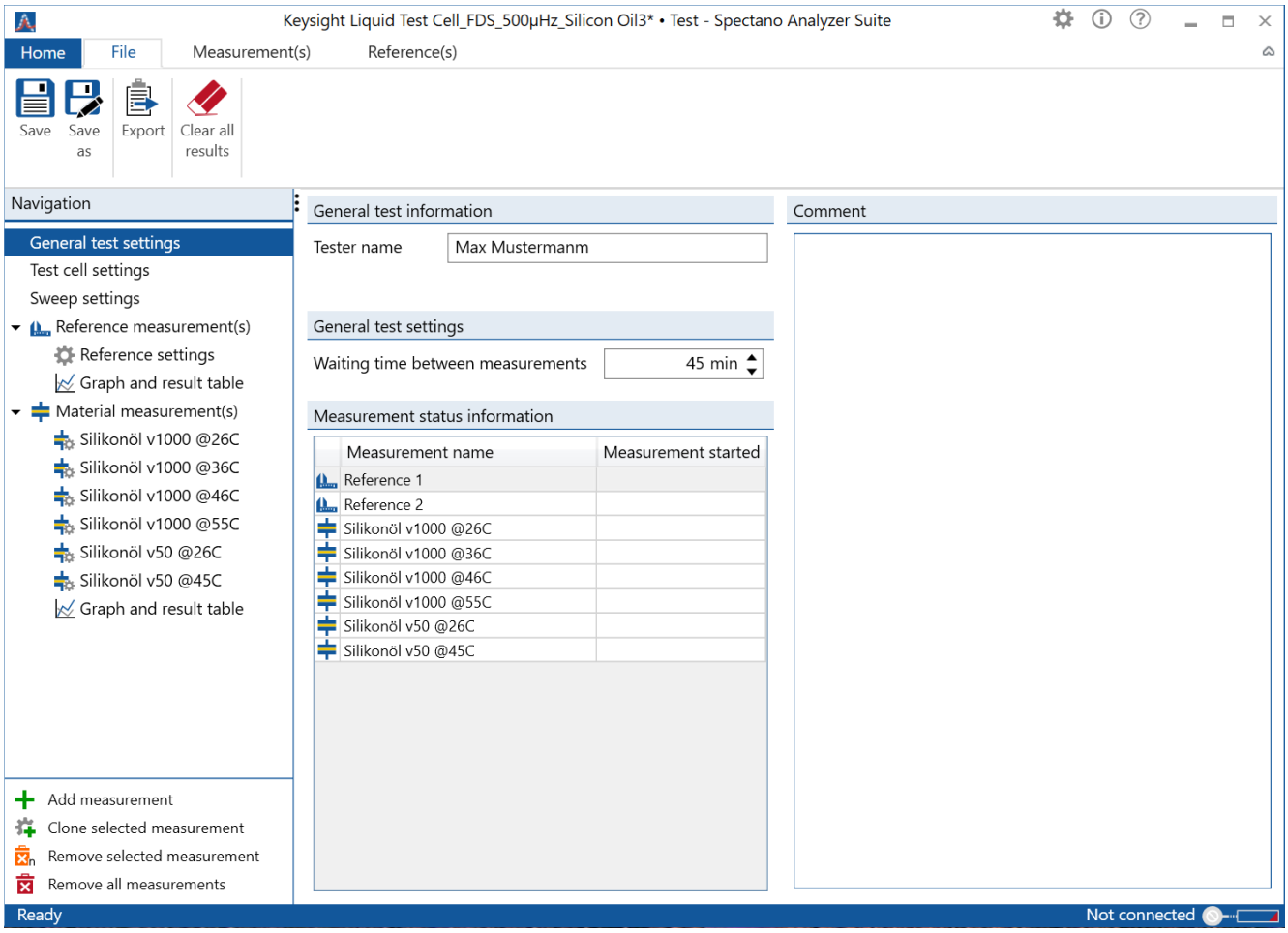


Figure 5: General settings example for measurement of silicone oil at different temperatures

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: $\varepsilon_r = \frac{C_r}{c_0}$
Set the following parameter for the Liquid Test Fixture 16452A from Keysight:

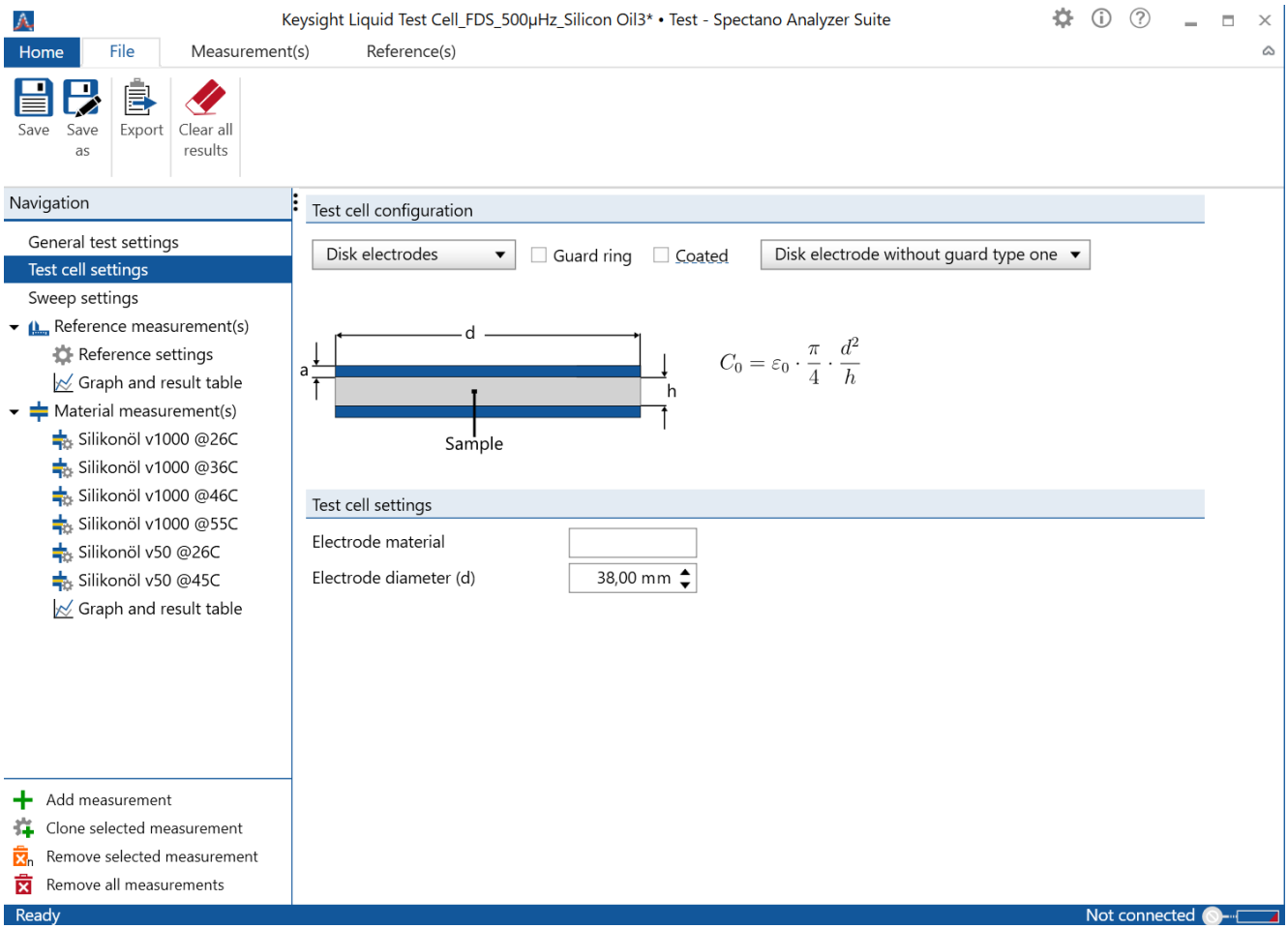


Figure 6: Test cell settings to be selected for Liquid Test Fixture 16452A from Keysight

6 Open the **Sweep settings** view

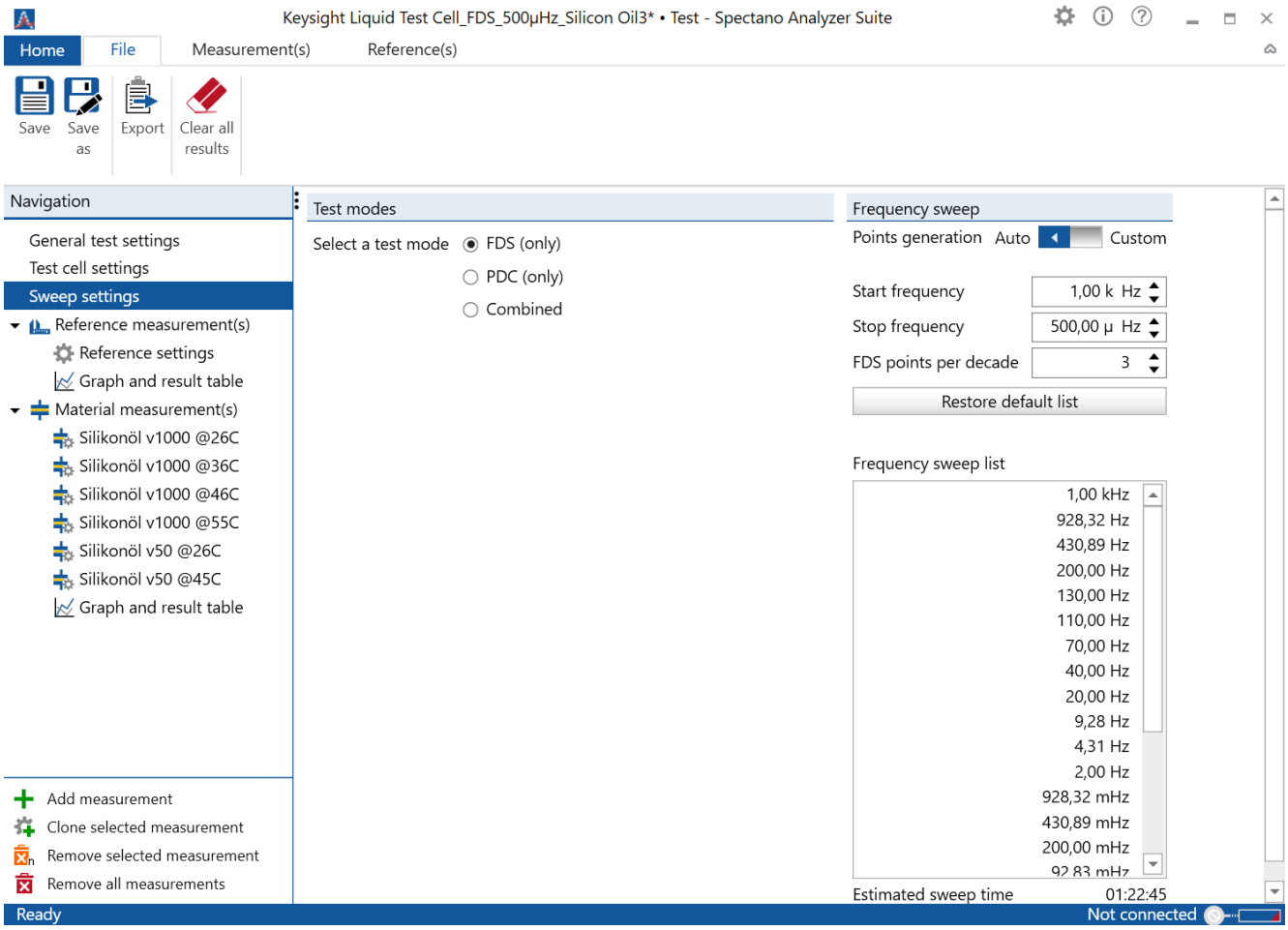


Figure 7: Sweep settings example for measurement of silicon oil at different temperatures

7 Select a measurement mode depending on the material type 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 1 kHz to 500 µHz with 3 FDS points per decade.

Note: The defined operating frequency range the Liquid Test Fixture 16452A from Keysight is 20 Hz to 30 MHz.

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

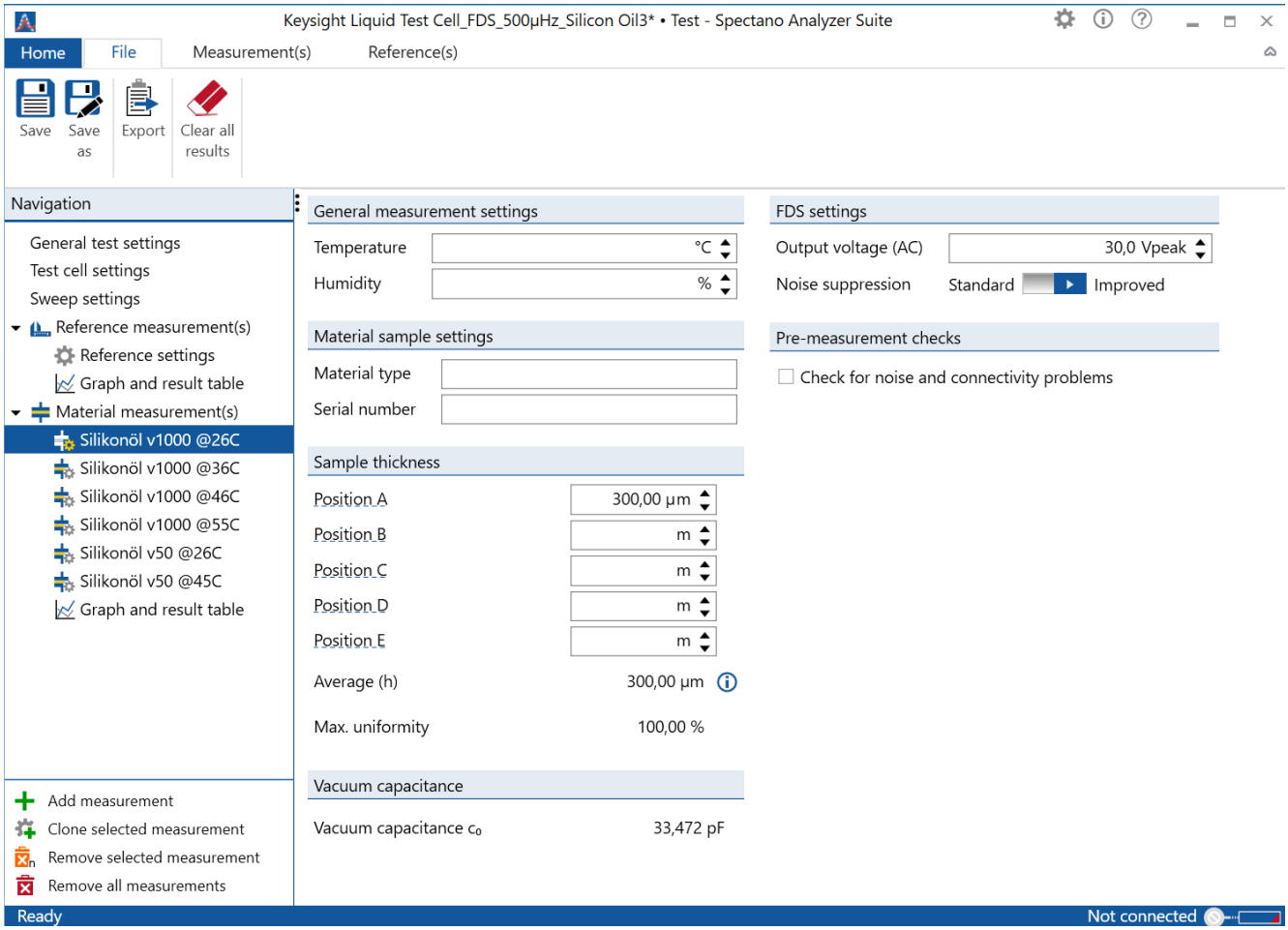


Figure 8: FDS settings for measurement of silicon oil with a sample thickness of 1.3mm

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

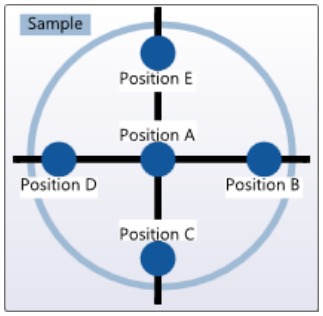
NOTICE

The maximum applicable voltage range for the Liquid Test Fixture 16452A from Keysight is 30 Vrms. Higher voltages can damage the test fixture.

The examples described in this application note are performed with 30 V_{peak} (AC).

d. Material type
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. For the Liquid Test Fixture 16452A from Keysight it is enough to enter one sample thickness because a pre-defined spacer ring is used to generate the thickness.

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	Pre-measurement checks
☑ Check for noise and connectivity problems	☑ 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

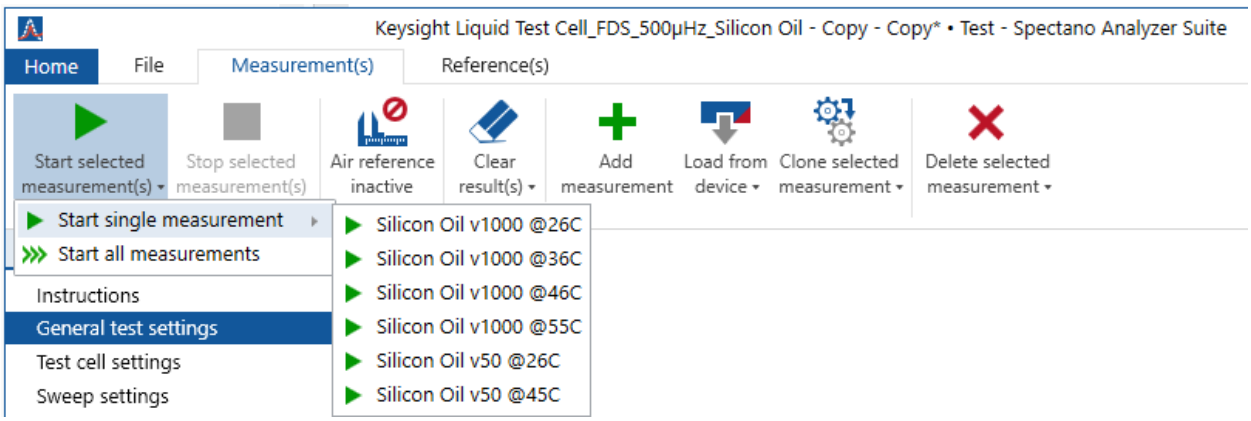


Figure 9: Start single measurement

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

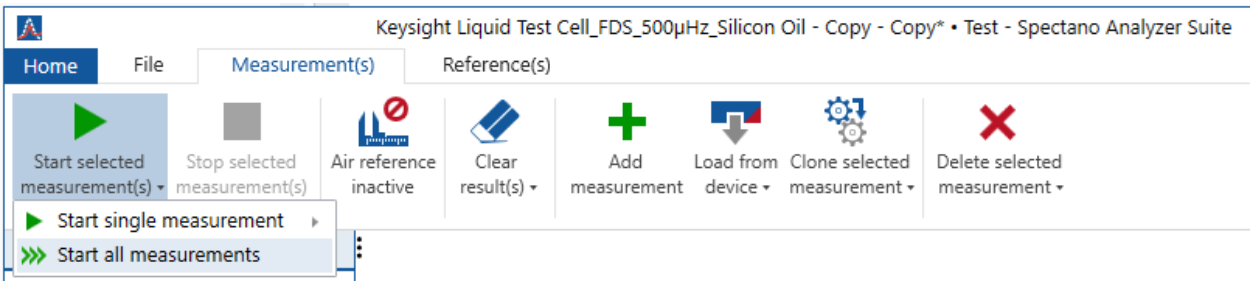


Figure 10: Start measurement sequence

6.2 Measurement Results

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.

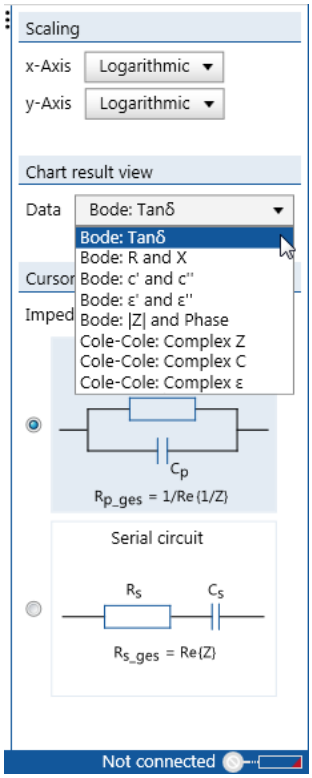


Figure 11: Changing display mode

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 12 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 of Z	|Y|	Phase of Y	Cp	Rp	c'	-c''	R	-X
Silicone oil v1000 @26°C	50 Hz	0,000672	2,59E+00	1,74E-03	39,293 MΩ	-89,961 °	27,267 nS	89,961 °	86,7941 pF	57,574 GΩ	86,7941 pF	58,3246 fF	27,137 kΩ	39,293 MΩ
Silicone oil v1000 @36°C	50 Hz	0,0007554	2,56E+00	1,94E-03	39,741 MΩ	-89,957 °	26,959 nS	89,957 °	85,8151 pF	51,831 GΩ	85,8151 pF	64,8294 fF	31,083 kΩ	39,741 MΩ
Silicone oil v1000 @46°C	50 Hz	0,0009767	2,53E+00	2,47E-03	40,346 MΩ	-89,944 °	26,554 nS	89,944 °	84,5277 pF	40,681 GΩ	84,5277 pF	82,5615 fF	40,709 kΩ	40,346 MΩ
Silicone oil v1000 @55°C	50 Hz	0,00087	2,5E+00	2,17E-03	40,766 MΩ	-89,95 °	26,281 nS	89,95 °	83,6577 pF	46,135 GΩ	83,6577 pF	72,7815 fF	36,514 kΩ	40,766 MΩ

Figure 12: Result table view

The screenshots in the following chapters show results for two different silicon oil samples measured at different temperatures using SPECTANO 100 and the Liquid Test Fixture 16452A from Keysight.

6.2.1 FDS measurement results

Figure 13 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.

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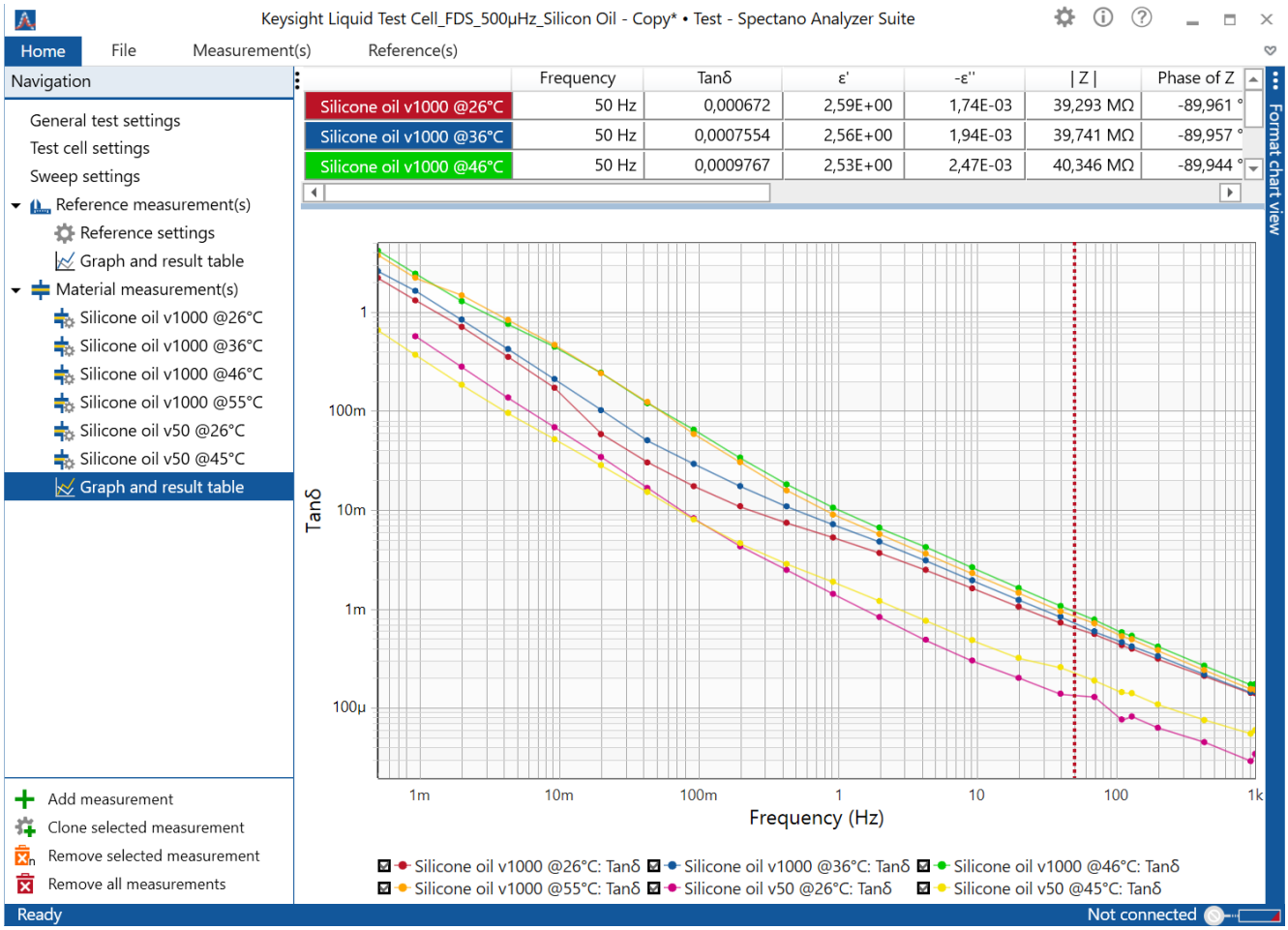

Figure 13: Tan(δ) example curves of two different silicon oil samples at different temperatures

The resulting curves of the two different silicon oils are typical curves for insulation liquids. As can be seen, the dielectric losses tan(δ) increase with the temperature and result into a change of the material structure and specific conductivity σ(ω).

The dielectric response of an ideal insulation liquid is a straight line with a slope of -20 dB/dec because of the conductive behavior having nearly no polarization effects.

The curves shown in Figure 14 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.

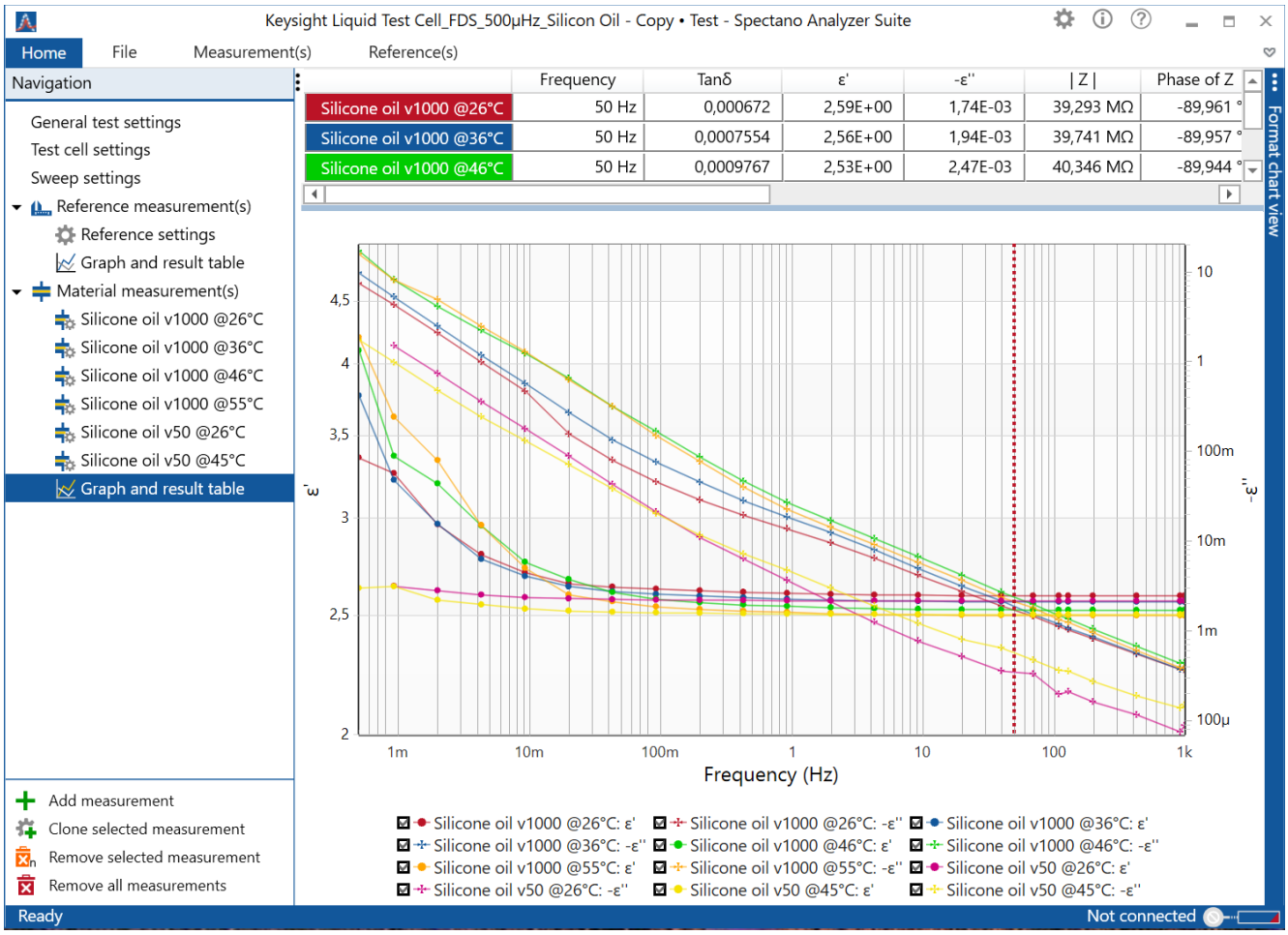

Figure 14: Relative permittivity example curves of different polymer and plastic samples

The measured values of tan(δ) and the permittivity meet the expected typical measurement results for the analyzed insulation oil.

7 Determination of Specific Conductivity σ(ω) and Resistivity R(ω)

For more information on the

- Determination of the specific conductivity σ(ω) and resistivity R(ω) of insulation liquids read the Application Note **Dielectric Material Analysis of Insulation Liquids** [4]
- 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/.

8 Summary

Despite the fact that the examined Liquid Test Fixture 16452A from Keysight was designed for a frequency range from 20 Hz to 30 MHz this application note illustrates that it can be used at much lower frequencies with OMICRON Lab's Dielectric Material Analyzer SPECTANO 100.

The results obtained with SPECTANO 100 and the Liquid Test Fixture 16452A from Keysight 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 [5]	Measured permittivity [6]
Silicone oil (polydimethylsiloxane)	2.3 – 2.8	2.5 – 2.6

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 composition and structure Since dielectric parameters depend strongly depend on these material properties. Further on, the environmental conditions must be the same. The results for some materials are highly influenced by temperature, pressure or humidity.

SPECTANO 100 and its accessories and tools offer all features and functionalities required 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.

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

[5] Published e.g. under http://www.mit.edu/~6.777/matprops/pdms.htm or http://www.clippercontrols.com/pages/Dielectric-Constant-Values.html

[6] Measured with SPECTANO 100 and Liquid Test Fixture 16451A from Keysight

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