Dielectric Assessment Kit

APPLICATION NOTE

DAK/DAKS Best Practices

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DAK/DAKS Best Practices

1 Introduction

The DAK family of products can be used to obtain precise measurements of dielectric parameters of a variety of samples in solid, semi-solid, or liquid form over a broad frequency range. The system is fully automated and software controlled. Nevertheless, the measurements must be performed with care and attention to details to obtain reliable results. This application note provides additional guidance for users.

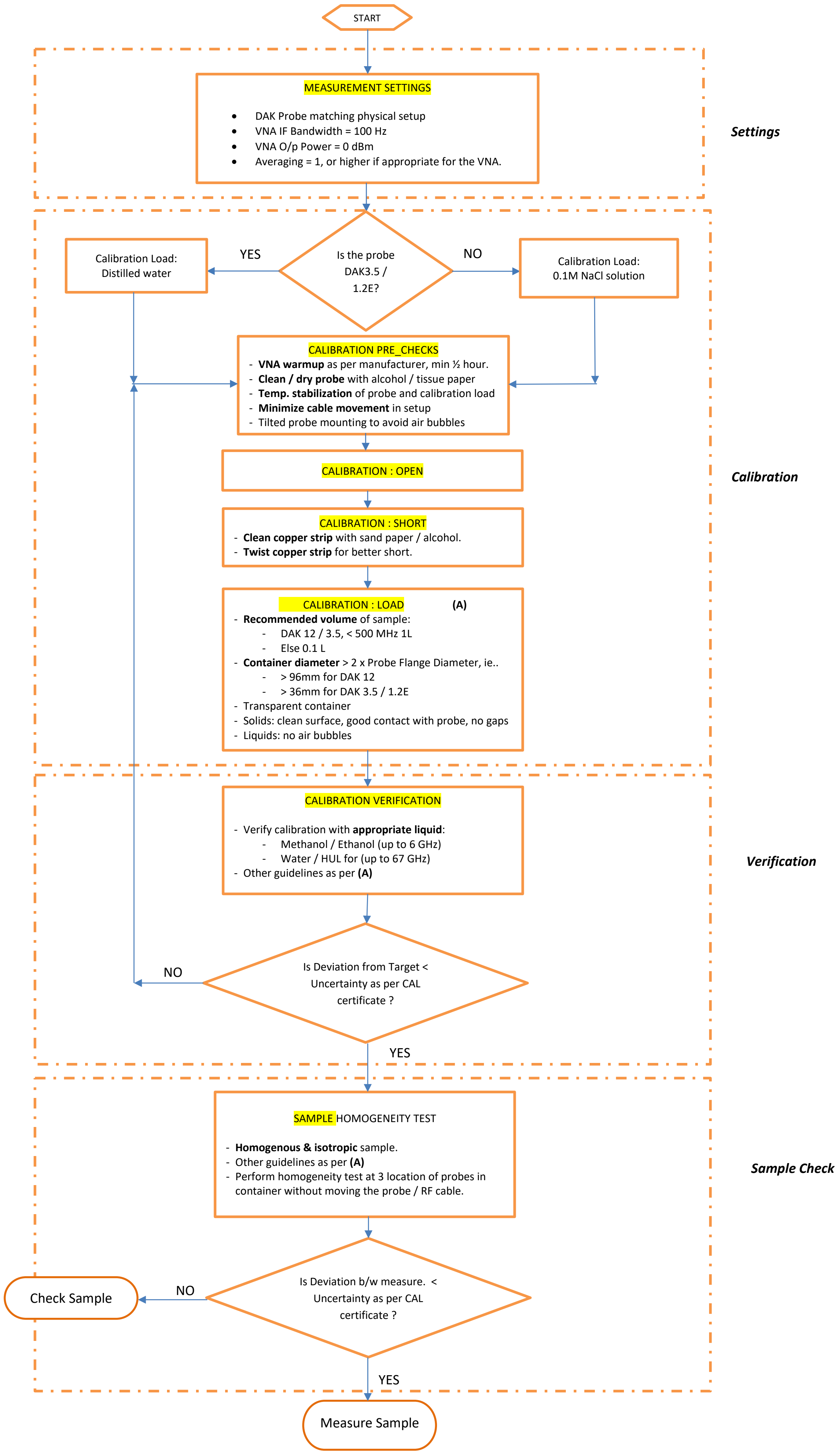
The typical measurement process comprises four steps:

- Selection of optimal measurement settings
- Calibration of the instrumentation
- Verification of the calibration
- Measurement of the samples

2 Objective

The objective of this document is to provide guidance on sample preparation and best practices for the measurement process, outlined above, to ensure accurate measurements. Sample requirements for DAK/DAKS are discussed in Section 3, while the measurement practices are described in Section 4.

Flow Chart : Preparation for Sample Measurement with DAK / DAKS



3 Sample Requirements

The DAK (DAK / DAKS) software algorithm is based on the following assumptions about the sample:

1. The sample is homogenous.
2. The sample is isotropic.
3. The sample is in good contact with the coaxial probe and within the metallic mirror, i.e., the contact is uniform and free of any air gaps or bubbles.

The solver is based on the assumption that the sample size is infinite, i.e., the reflection from sample boundaries is assumed to be negligible.

Note: It is not possible to measure magnetic / metallic samples with DAK / DAKS.

The volume of liquid sample recommended to avoid reflection from sample boundaries is summarized in the following table. Please note that the reflections are a strong function of the electromagnetic losses. It can be easily verified if the sample is large enough for accurate measurement by placing a metal foil under or on the sides of the container/sample and check if there is no change in the reflection coefficient (DAK software screen).

Probe Geometry	Frequency Band	Sample Volume	Container Diameter
DAK 12 / DAK 3.5	≤5GHz	>1 L	>100 x 100 x 100 mm
DAK 3.5 / DAK 1.2E	>5 GHz	>0.1 L	>30 x 30 x 30 mm

For solid samples, the sample must have a smooth surface finish to avoid air gaps when in contact with the DAK probe. An N5-grade surface finish is recommended for best results. The sample diameter should large enough that at least the flange area is covered.

To verify whether the sample boundaries extend far enough, i.e., do not cause any significant reflections, a metal foil can be placed under or on the sides of the container. Any resulting changes in the reflection coefficient can be observed on the DAK software screen.

4 Measurement Process

4.1 Selection of optimal measurement settings

The settings that have the greatest influence on measurements are shown in Figures 1.1 and 1.2.

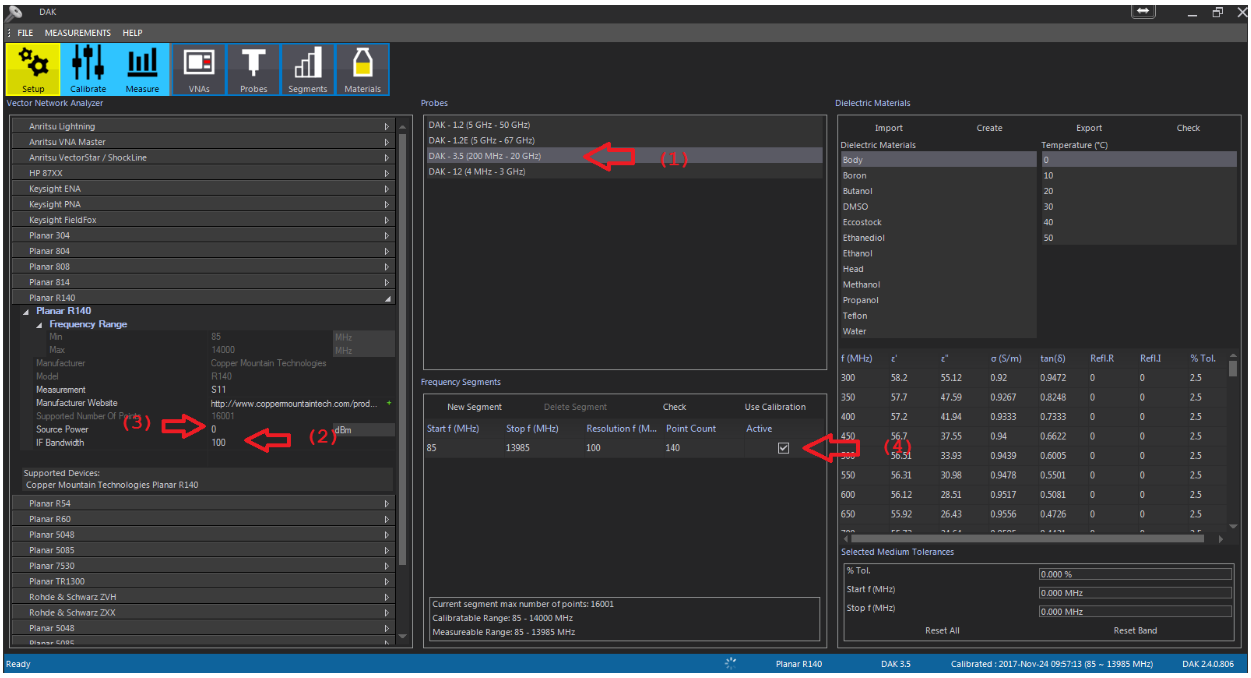


Figure 1.1: Measurement settings screen for DAK / DAKS

The highlighted settings in Figures 1.1 and 1.2 are described below.

DAK Probe (1): Under probe selection, ensure that the correct probe is selected.

VNA IF Bandwidth (2): A higher bandwidth increases measurement speed, but the traces become noisier. A lower bandwidth reduces noise, but is slow. A VNA bandwidth setting of 100 Hz is suited for most dielectric measurements.

VNA Output Power (3): The default VNA output power of 0 dBm is recommended.

Frequency Segments (4): The active frequency segments for the trace should be a subset of the measurement frequency range of the VNA.

Averaging (5): Averaging several VNA traces reduces noise in and the error of the reported result. A VNA that is properly warmed up and exhibiting stable operation, trace averaging should not be necessary, thus Averaging can be set to 1 but can be increased if required to reduce noise, however with a trade-off in measurement speed. This setting is accessible during both the calibration and the measurement processes. (Figure 1.2)

Once calibration has been completed, the settings in Figure 1.1 should not be changed further. If a change is required, the instrument should be freshly calibrated.

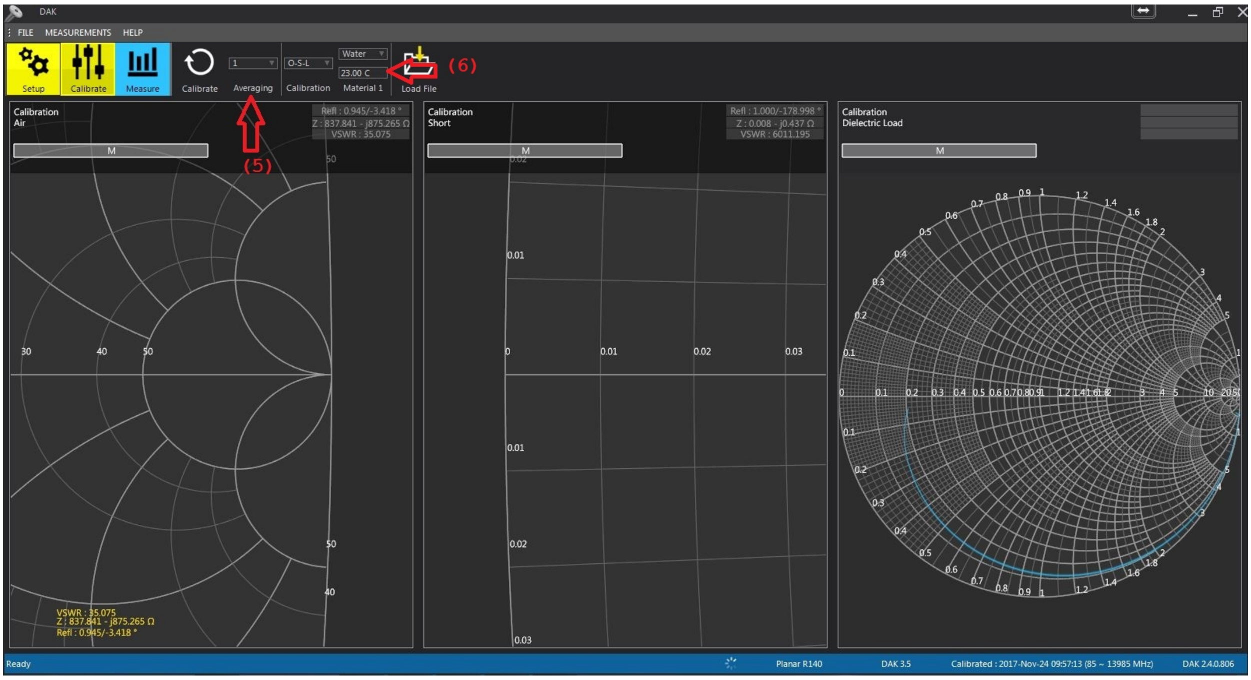


Figure 1.2: VNA averaging settings and calibration load temperature

4.2 Calibration

4.2.1 Selection of the Calibration Load

Ideally, the behavior of the calibration load should be similar to that of the target material, i.e., with a relatively flat response for the desired frequency range.

For liquid measurements made with the DAK 3.5 / 1.2 E probe geometries, the recommended calibration load is distilled water. For the DAK-12 probe geometry, especially those performed at frequencies <500 MHz, the recommended calibration load is a 0.1 M saline (NaCl) solution.

The calibration load should meet the requirements described in Section 3 regarding sample volume, homogeneity, and isotropicity.

4.2.2 Load / Sample Temperature (For Liquids Only)

Dielectric parameters of liquids are temperature sensitive (up to 2% variation in dielectric properties per 1°C temperature change), thus, a precise record of the temperature of the sample at the time of the measurement is crucial to achieve reproducible results. The load temperature must be precisely measured (±0.05 °C) and entered into the software (see Figure 1.2). Temperature gradients between the probe and the sample should be avoided.

Note that the evaporation of volatile liquids, e.g., alcohol, can cool the probe. To avoid influencing the temperature of the next material placed in contact with the probe, the temperature of the probe must be allowed to equilibrate after any exposure to volatile liquids.

It is recommended that gels be stabilized by storing (in a watertight container) in a water-bath kept at the desired measurement temperature for a sufficiently long time, e.g., overnight. The water-bath temperature should be measured immediately before dielectric measurement cycle is started and entered in the software as the sample temperature.

4.2.3 Cable Movement

During the calibration process, the VNA calibration plane is transferred to the probe flange-sample interface, and the transfer function therefore includes all four "S" parameters of the connecting radiofrequency (RF) cable. For the calibration to remain valid, cable movement must be minimized, as it can change the "S" parameters of the cable and hence invalidate the calibration results. It is recommended that a fresh calibration be undertaken whenever there is any cable movement.

For DAKS, where the probe is connected directly to the VNA, cable movement is not relevant.

4.2.4 "Open" Measurement

"Open" measurements are performed with the probe in contact with air. The probe must be clean and dry: the probe surface should be cleaned with both water and alcohol, especially after measuring oily materials, and then allowed to equilibrate as described in Section 4.2.2.

Figure 1.3 shows an "Open" measurement on the Smith chart after the "O" button is pressed:

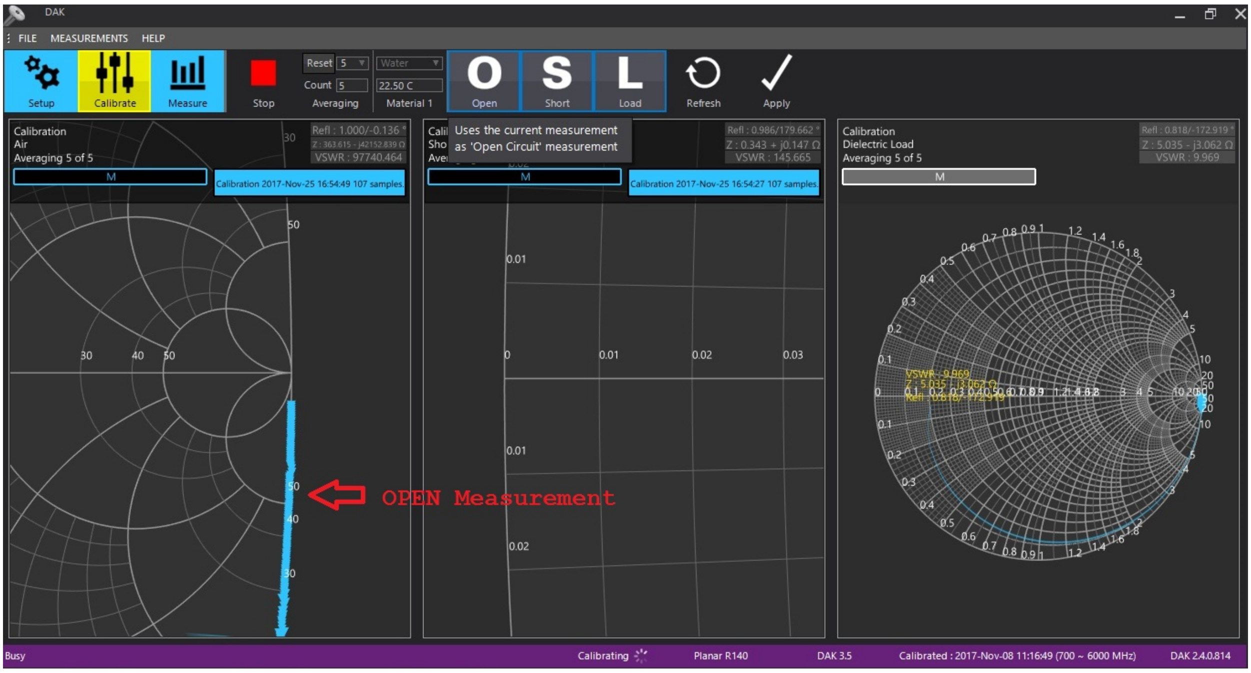


Figure 1.3: Smith chart output for an "Open" measurement

4.2.5 "Short" Measurement

The accuracy of the "Short" measurements is important for measurement precision. In the short measurement, a copper strip mounted on the shorting block is pressed against the probe, as shown in Figure 1.4.

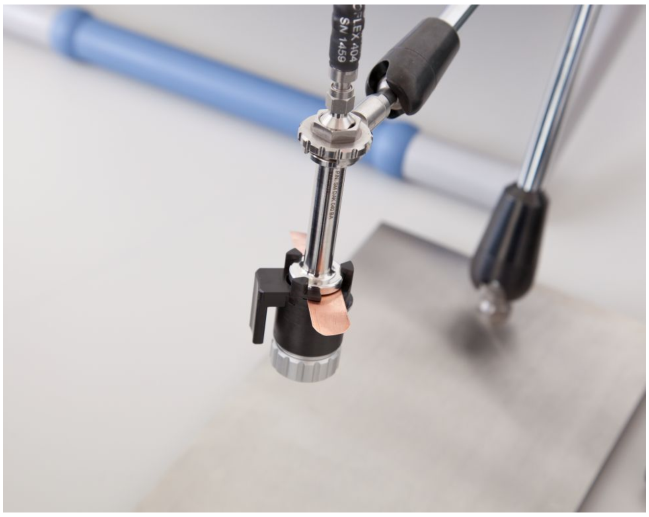


Figure 1.4: Setup for a "Short" measurement

For the electrical contact with the probe to be good, the copper strip must be clean and not oxidized: use fine sandpaper to remove, e.g., contamination or oxides from the surface, then clean the strip with isopropyl alcohol or ethanol. Worn copper strips should be discarded and replaced with new ones. The "Short" measurement should be repeated by lightly twisting the copper strip, and, if a better short measurement – i.e., located on the left hand side of the Smith chart (Figure 1.5) – is obtained, the better measurement should be used for "Short".

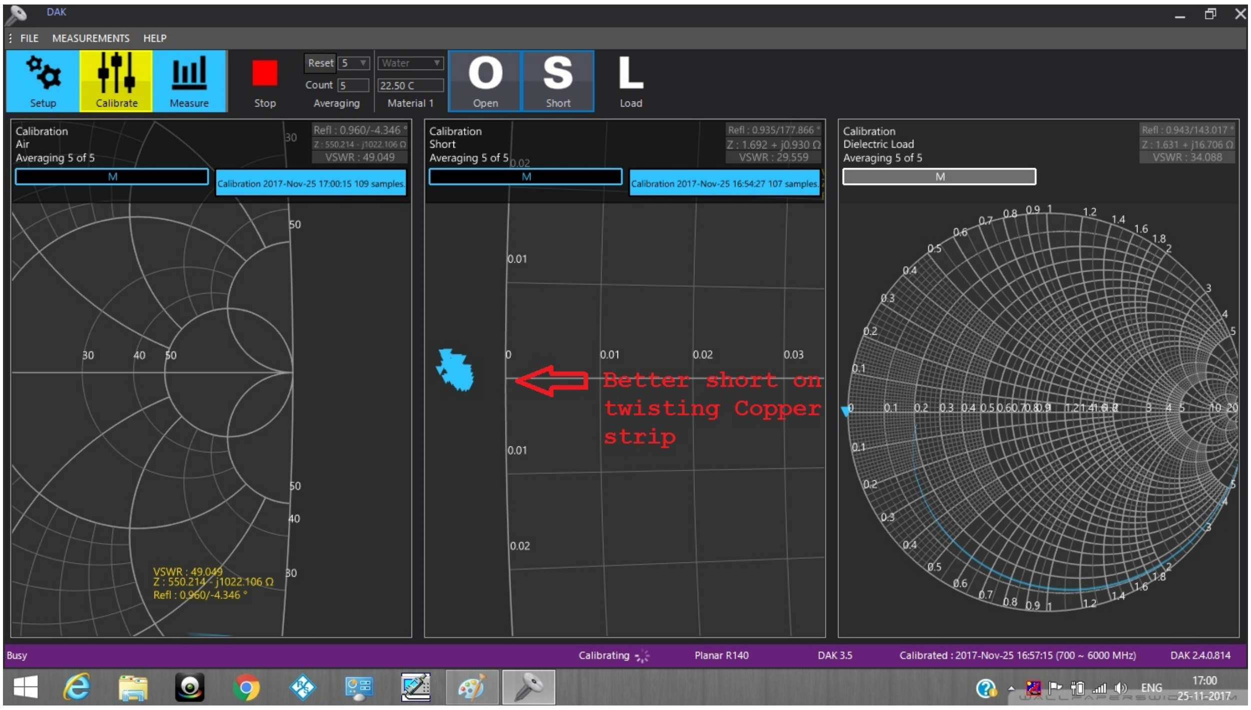


Figure 1.5: Smith chart showing a better short measurement

4.2.6 "Load" Measurement

The load must be homogeneous, isotropic, and free of impurities that could affect the dielectric measurement. Pure grade liquids, e.g., distilled water, 0.1 M saline solutions, alcohols, can be purchased. Proper storage of liquids will keep the materials free of dust and other impurities. Liquids that contain impurities should be discarded.

Good electrical contact must be maintained between the probe and the load. For liquids, this means removing any bubbles that form under the probe. The probe should be mounted face down, slightly offset from vertical. This orientation prevents formation of air bubbles. The probe should be visually inspected for air bubbles, and any incidental bubbles should be removed with a cotton swab, as shown in Figure 1.6).

Figure 1.6: Removal of air bubbles from DAK probe immersed in liquid

For solid materials, the surface must be flat and clean to make good electrical contact with the probe.

4.3 Calibration Verification

According to good practice, calibration should be verified with more than one reference material.

For liquid measurements, when distilled water is used as the calibration load, verification can be performed with alternative reference liquids such as methanol, ethanol, or equivalent. Dielectric values for these reference liquids are available in the software for frequencies up to 6 GHz. When a liquid other than distilled water is used as the calibration load, distilled water or saline can be used as an additional verification liquid.

Verification measurements should be performed according to the guidelines detailed in Section 4.2.6.

The measurement results should be within the uncertainty specified in the calibration certificate.

4.4 Sample Measurements

Samples for dielectric measurement should should meet the requirements described in Section 3 regarding sample volume, homogeneity, and isotropicity. The temperature of liquid samples should be stabilized according to the guidelines detailed in Section 4.2.2.

The probe should be clean and dry. If required, the probe should be cleaned with both water and alcohol and dried with lint-free tissue before measurements are made.

Sample measurements should be performed according to the guidelines detailed in Section 4.2.6.

4.4.1 Checking Sample Homogeneity

The algorithms in the evaluation software are valid for homogeneous, isotropic materials. The material closest to the center of the probe flange dominates the result. It is good practice to test material homogeneity before any sample measurements are performed.

To test the homogenity of liquid samples, it is recommended that at least three measurements be performed with the probe at different locations in the container, without touching the probe or the connecting RF cable.

For solid materials, the three measurements can be performed on two faces of the sample, for a total six measurements.

The computed deviation from the average of each measured value at any representative frequency should be within the uncertainty specified in the calibration certificate at that frequency.

4.4.2 Calibration Verification between Measurements

When testing groups of samples that require significant cleaning of the probe, e.g., when water-based samples are measured after oil-based materials, the probe should be cleaned to avoid cross-contamination of samples; it is further recommended that the temperature be allowed to equilibrate (section 4.2.2) and the calibration should be verified (section 4.3) between these measurements.

5 Conclusion

This document provides guidelines for sample preparation and best measurement practices for dielectric measurements performed with DAK / DAKS. Users are encouraged to follow these recommendations carefully to ensure the most precise and repeatable measurements of dielectric properties of samples.