Dielectric Assessment Kit

APPLICATION NOTE

DAK-TL Best Practices

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DAK-TL-P Best Practices

1 Introduction

The DAK-TL-P system is the first instrument that can be used to measure the dielectric properties of thin material layers and small quantities of liquids for a broad frequency range with high precision. 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 guidelines for users.

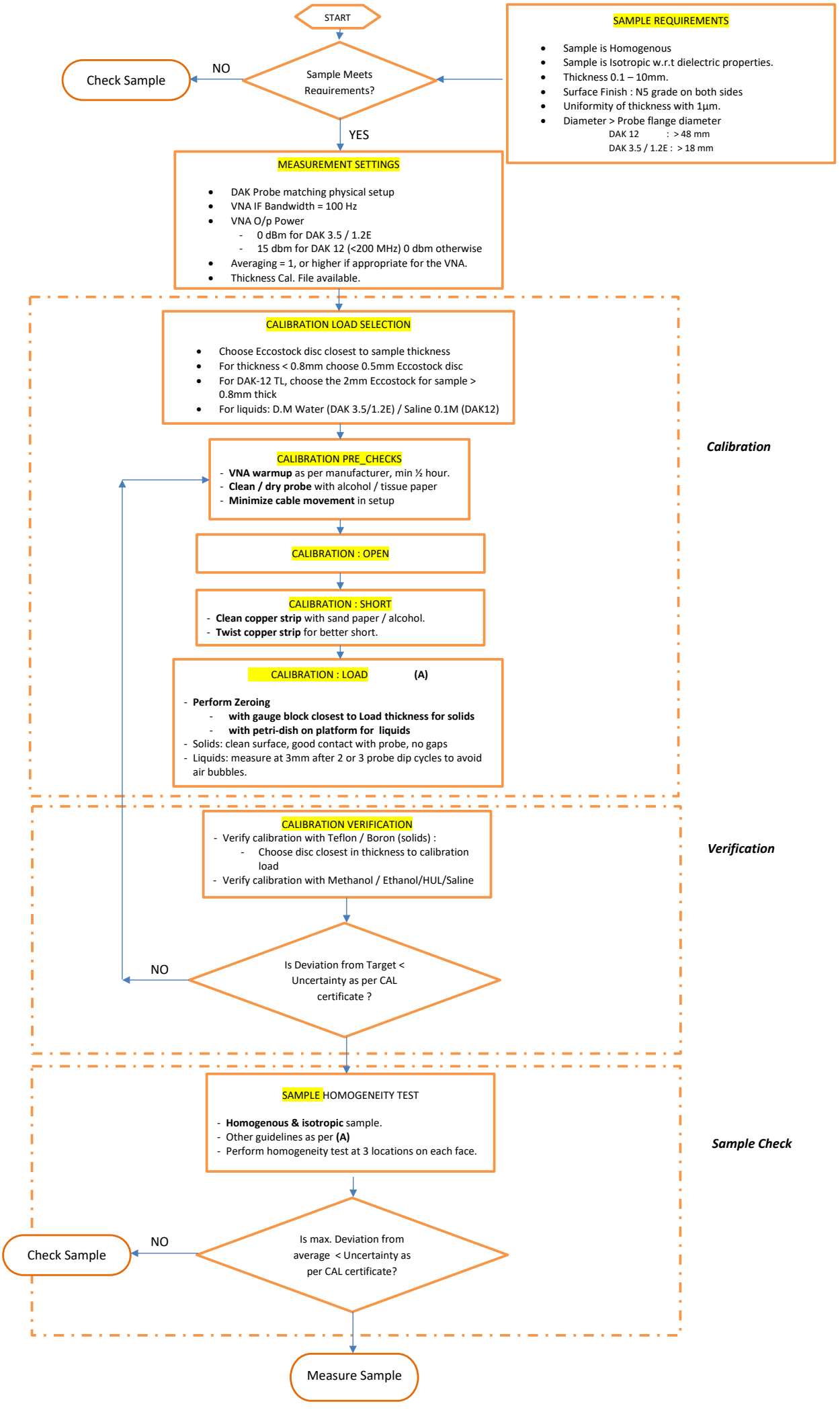
The typical measurement process comprises four steps:

- Selection of optimal measurement settings
- Calibration of the instrument
- 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, as outlined above, to ensure accurate results. Sample requirements for DAK-TL-P are discussed in Section 3, while the measurement practices are described in Section 4.

Flow Chart : Preparation for Sample Measurement with DAK TL-P



3 Sample Requirements

The DAK-TL-P 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 (applies to liquids).

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.

It is not possible to measure magnetic / metallic samples with DAK-TL-P.

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

Characteristic	**Recommended Value**
Thickness	0.1 – 10 mm
Surface Finish	N5 grade on both sides
Diameter	DAK 12: >48 mm* DAK 3.5 / 1.2E: >18mm* *measurements of smaller diameters are possible (see further sections)
Uniformity of Thickness	Within 1 μm

For lossy liquid or tissue samples, the provided metallic petri dish should be half-filled with the test liquid.

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.

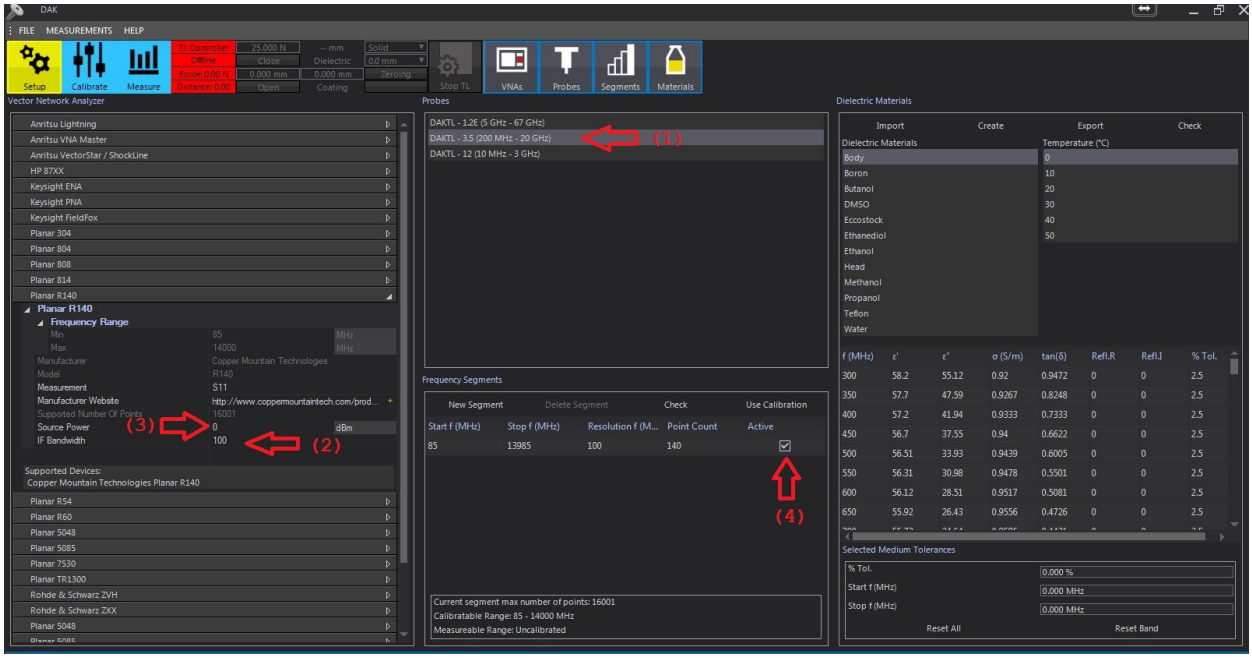


Figure 1.1: Measurement settings screen for DAK-TL-P

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 for DAK-3.5 TL-P and 1.2E TL-P. The maximum output power allowed by the VNA, +15 dBm, is recommended for measurements performed at frequencies <200 MHz with DAK 12 TL-P.

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 VNS 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, see (Figure 1.2).

Valid Thickness Calibration (6): A valid thickness calibration file for the DAK-TL base unit must be correctly loaded. The indication "Calibrated" in the status bar confirms that the file is loaded, see (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.

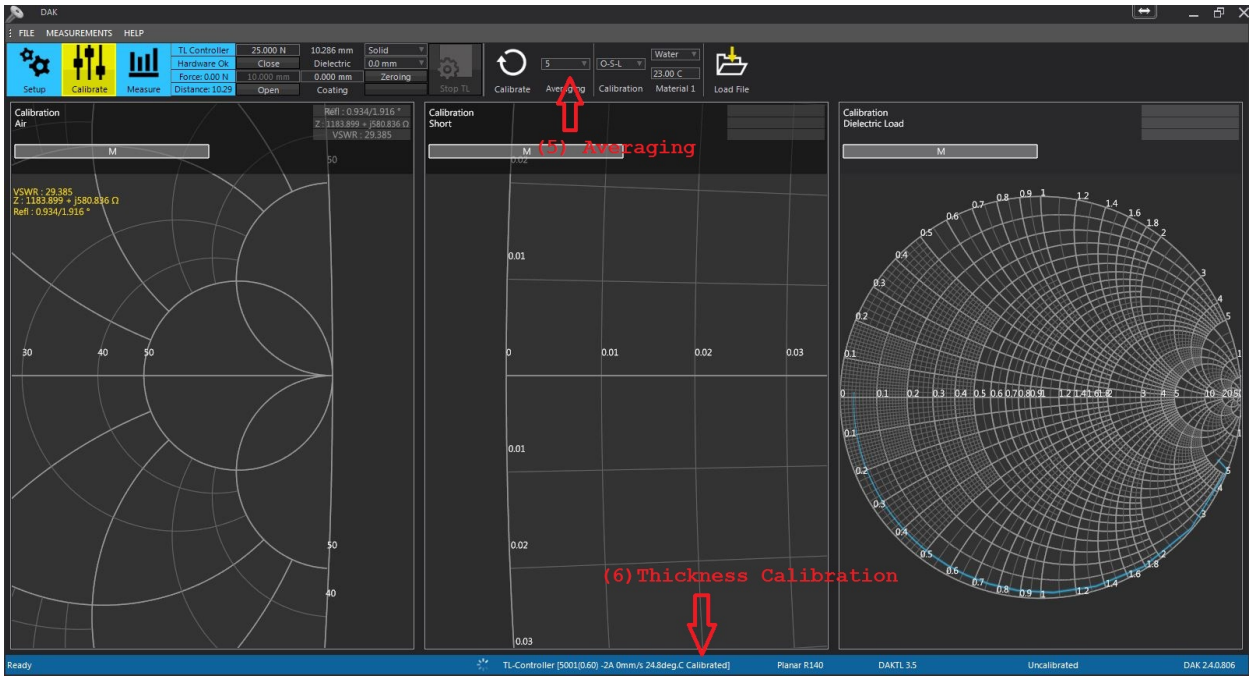


Figure 1.2: Trace Averaging, Valid thickness Calibration file for DAK-TL-P

4.2 Calibration

4.2.1 Calibration load selection

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 solid measurements with DAK-TL-P, the supplied Eccostock ($\epsilon' = 2.54$) discs of thickness 0.5, 2.0, and 5 mm are recommended to be used for most dielectric measurements. To select the disc most suitable for your calibration, please note the following:

- Choose a disc closest in thickness to the sample thickness.
- For sample thickness <0.8 mm, the 0.5 mm thick disc is recommended for all probe beam geometries.
- For the DAK12-TL beam, the 2 mm-thick disc is recommended for sample thickness >0.8 mm;
- If multiple samples with different thicknesses need to be measured, multiple calibrations should be created and saved with the suitable load discs for each thickness range.

For liquid measurement with the DAK3.5/1.2E-TL-P geometries, distilled water is recommended as the calibration load. For DAK12-TL P probe geometry, especially at frequencies less <500 MHz, 0.1 M saline (NaCl) solution is recommended as the calibration load.

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.

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.

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:

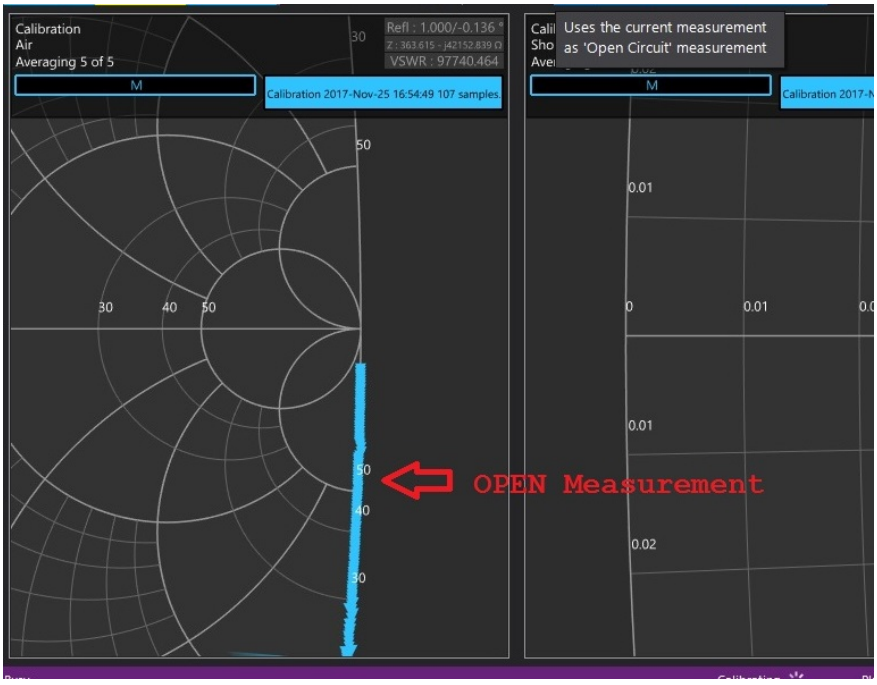


Figure 1.3: Smith chart output for an "Open" measurement

4.2.5 "Short" Measurement

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

Figure 1.4: Setup for "Short" Measurement

For the electrical contact with the probe to be good, the copper tape must be clean and not oxidized: use fine sandpaper to remove, e.g., contamination or oxides from the surface, then clean the tape with isopropyl alcohol or ethanol. Worn copper tape should be discarded and replaced with new tape. The "Short" measurement should be repeated by lightly twisting the copper tape, 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".

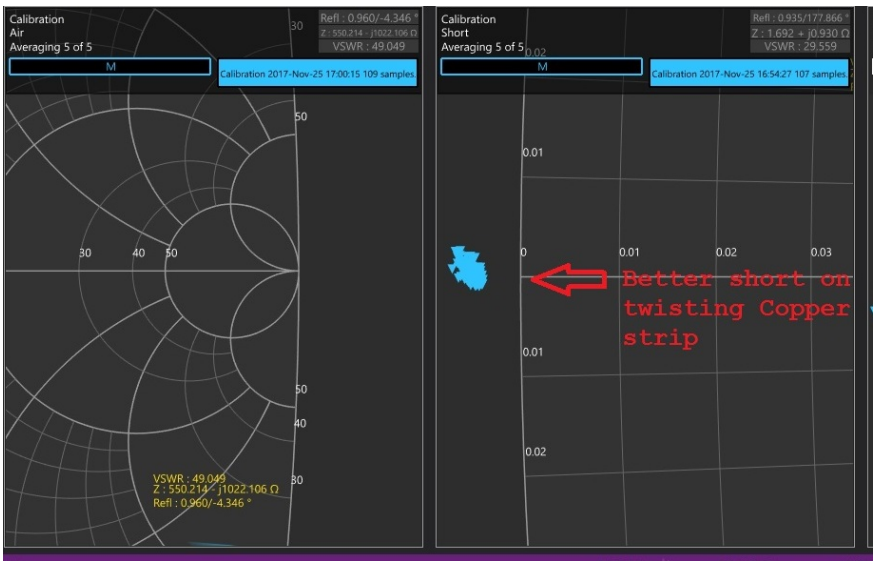


Figure 1.5: Smith chart showing a better short measurement

4.2.6 "Load" / Sample Measurement

Solids

With solid materials, the surface must be flat and clean for good electrical contact with the probe. The sample should meet the requirements described in Section 3 regarding homogeneity, and isotropicity. Before the actual measurement of the load, the instrument should be zeroed with a gauge block (available values are 0.5, 1, 2, and 8 mm) nearest in thickness to the sample being measured at the same force at which the load / sample is to be measured. A zeroing measurement performed with a 1 mm gauge block at 200 N force is shown in Figure 1.6:

Figure 1.6: Zeroing measurement performed prior to measurement of a solid

Zeroing increases the accuracy of the DAK-TL-P device for measurement thickness.

The load / sample can be measured after the zeroing operation.

Liquids

For liquids, zeroing must be performed with a clean and empty petri-dish placed on the platform as shown in Figure 1.8:

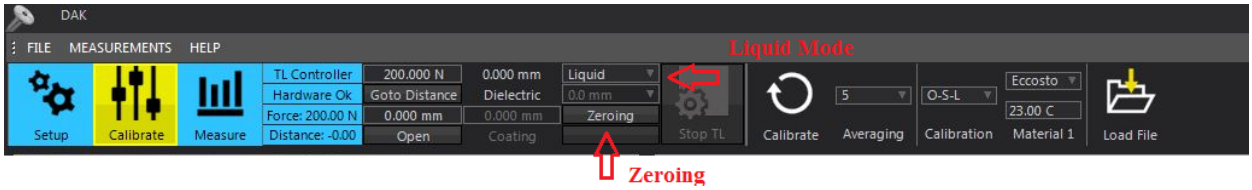


Figure 1.7: Zeroing measurement performed prior to measurement of a liquid

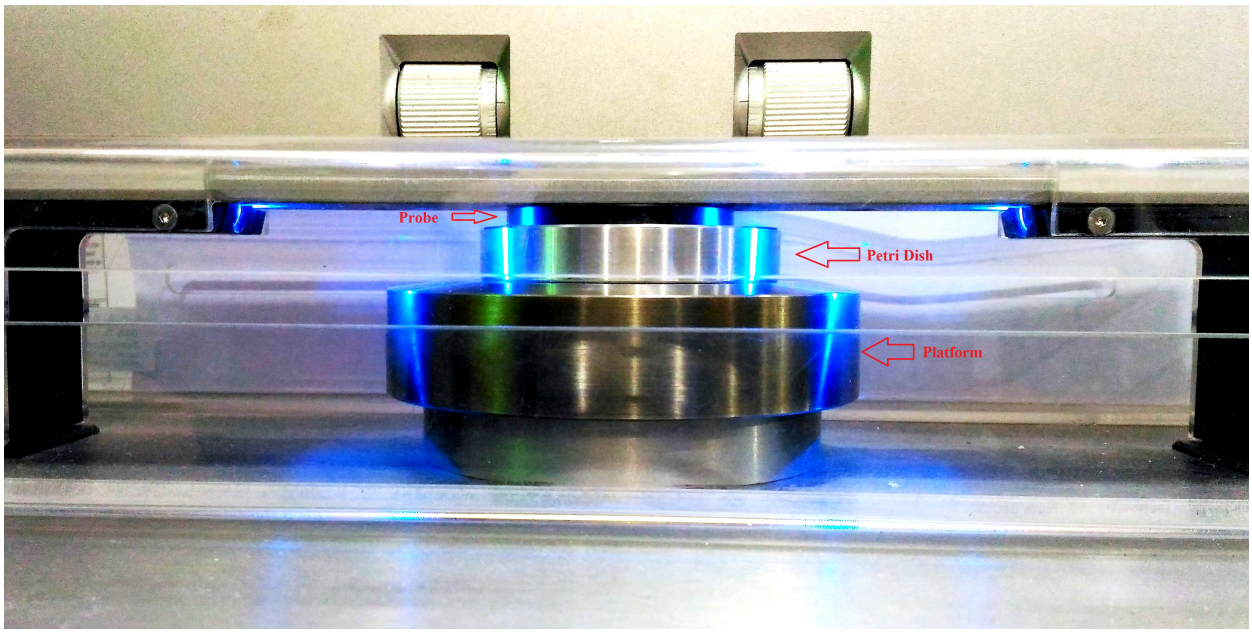


Figure 1.8: Zeroing with a clean and empty petri-dish placed on the platform

The petri-dish should be half-filled with the test liquid, and the measurements made at a depth of 3 mm. Before the measurement is made, it is recommended to move the platform to the "Open" position, then back to 3 mm depth two or three times to reduce the likelihood that air bubbles, which may be difficult to remove otherwise, become trapped under the probe .

The load / sample measurement can be made after zeroing as described above.

4.3 Calibration Verification

After calibration and according to good practice, the calibration should be verified with a different reference material.

For solid measurements with DAK-TL-P, the supplied Teflon or boron discs can be used as verification samples. It is recommended that a verification disc closest in thickness to the Eccostock load disc used for calibration (section 4.2.1) be chosen.

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.

The verification measurements should be performed according to the guidelines 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 assessment should meet the requirements described in Section 3. The temperature of the sample should be stabilized according to the guidelines 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.

The algorithms in the evaluation software are valid for homogeneous, isotropic materials[1]. The material closest to the center of the probe flange dominates the result. Therefore the system is well suited to verify that the sample under test is homogenous and isotropic. Homogeneity and isotropy can be assessed following the two tests:-

1. Homogeneity in x-y directions
2. Homogeneity in z-direction

4.4.1 Verification of Homogeneity in x-y Directions

To test surface homogeneity of solid samples, it is recommended that at least three measurements be performed on each face of the sample, for a total six or more measurements. The homogeneity test may be considered to pass if the measurements agree within the measurement uncertainty (k=2).

Table 1.1 shows an example of a set of sample recordings; each face is measured three times and the mean and standard deviation of the results are reported. Samples 1 – 4 are homogenous, while sample 5 exhibits a large deviation in permittivity between faces A and B, and is therefore considered inhomogeneous.

No.	Sample ID	Face A		Face B		Unc (K=2)		Homogeneity
		$\epsilon' \pm \sigma$	$tan(\delta) \pm \sigma$	$\epsilon' \pm \sigma$	$tan(\delta) \pm \sigma$	ϵ'	$tan(\delta)$	Test
1	Sample 1	3.55 ± 0.01	0.018 ± 0.001	3.53 ± 0.01	0.021 ± 0.001	0.07	0.02	PASS
2	Sample 2	3.58 ± 0.01	0.007 ± 0.002	3.58 ± 0.01	0.011 ± 0.002	0.07	0.02	PASS
3	Sample 3	4.25 ± 0.03	0.023 ± 0.002	4.26 ± 0.03	0.028 ± 0.002	0.17	0.02	PASS
4	Sample 4	4.33 ± 0.06	0.025 ± 0.003	4.33 ± 0.06	0.031 ± 0.003	0.17	0.02	PASS
5	Sample 5	19.91 ± 0.26	0.000 ± 0.001	23.40 ± 0.55	0.002 ± 0.002	0.82	0.02	FAIL

Table 1.1: Example showing sample homogeneity measurements at 2.45 GHz for a set of solid samples

[1]We are currently working on an extension of the system to determine the anisotropic properties.

4.4.2 Verification of Homogeneity in z Direction

Sample depth homogeneity can be verified by measuring the same sample with different DAK probes, as the fields penetrate different depths into the sample. A frequency which is covered by the ranges of two different DAK probes, which is as close as possible to the frequency of interest, should be chosen for the comparison. The homogeneity test may be considered to pass if the measurements agree within the measurement uncertainty (k=2).

Table 1.2 shows an example of a set of sample recordings; each sample is measured at the same frequency with two different probes. Samples 1, 2 and 3 pass the tests and are therefore homogeneous and isotropic. Samples 4 and 5 exhibit a larger deviation in the dielectric values than can be attributed to measurement uncertainty, and are therefore considered inhomogeneous or anisotropic with respect to depth.

No.	**Sample ID**	**DAK 3.5 TL**		**DAK 1.2E TL**		**Homogeneity**
		$\epsilon' \pm Unc.$	$tan(\delta) \pm Unc.$	$\epsilon' \pm Unc.$	$tan(\delta) \pm Unc.$	**Test**
1	Sample 1	3.52 ± 0.07	0.015 ± 0.03	3.50 ± 0.30	0.029 ± 0.03	PASS
2	Sample 2	3.56 ± 0.07	0.004 ± 0.03	3.47 ± 0.30	0.020 ± 0.03	PASS
3	Sample 3	4.69 ± 0.13	0.023 ± 0.02	4.74 ± 0.62	0.020 ± 0.03	PASS
4	Sample 4	4.76 ± 0.13	0.024 ± 0.02	3.90 ± 0.55	0.020 ± 0.03	FAIL
5	Sample 5	20.64 ± 0.58	0.001 ± 0.02	16.65 ± 2.36	0.000 ± 0.03	FAIL

Table 1.2: Example showing sample homogeneity measurements with different probes at 5GHz

From Table 1.1 & Table 1.2, it can be surmised that samples 1, 2 and 3 are homogenous and isotropic in x, y & z directions, and are fit for measurement with DAK-TL.

4.4.3 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.

When sample sets to be measured with DAK-TL-P comprise materials that range widely in thickness, it is recommended that calibrations be performed with load discs of thickness closest to that of each sample, as outlined in Section 4.2.6. Therefore, it may be convenient to measure samples in order of thickness, changing calibration files when appropriate.

5 Additional Information: Sample Diameter

The sensitivity of ϵ' measurement to sample diameter was evaluated by measuring Eccostock ($\epsilon' = 2.54$) discs of varying sizes (ϕ = 50, 45, 40, 35, 30, 25, 20, 15 & 10) mm with DAK 3.5 - TL and DAK 1.2E-TL beams. The deviation to target values, as a function of sample diameter for these measurements is shown in Figure 1.9

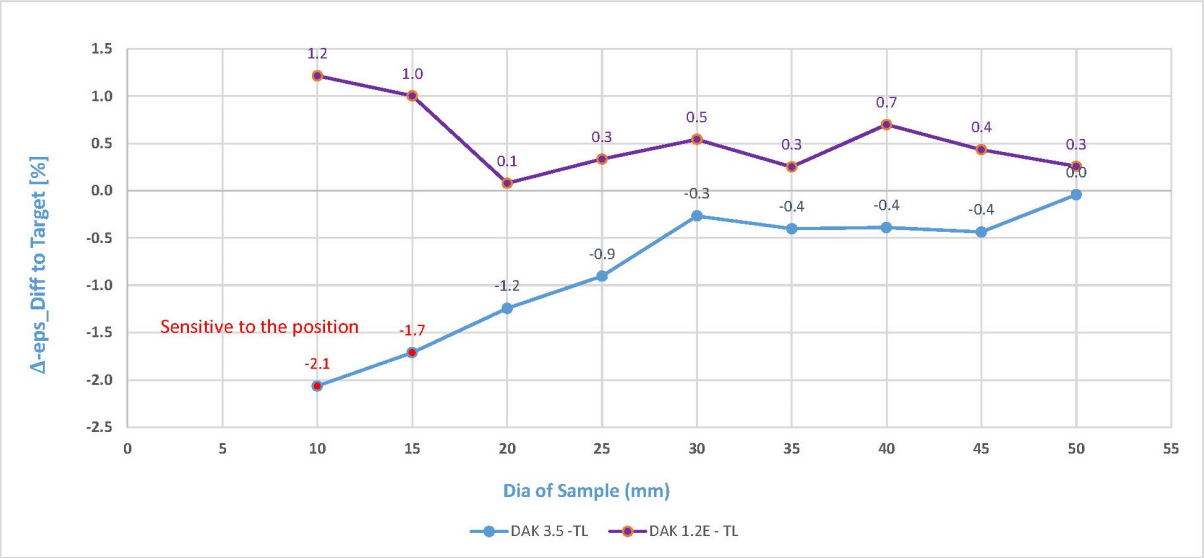


Figure 1.9: Sensitivity of relative permittivity measurements to sample (Eccostock) diameter

From Figure 1.9, it can be surmized that the results are not very sensitive to sample diameter as long the sample is wider than the coaxial probe flange diameter. For sample sizes larger than the flange diameter, measurements are none sensitive to probe diameter.

6 Conclusion

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