DHR Dielectric Accessory Getting Started Guide

Revision D Issued January 2016

Notice

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Introduction

Important: TA Instruments Manual Supplement

Please click the TA Manual Supplement link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2004 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 1 - Basic immunity test requirements.

For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

Safety

WARNING: The operator of this accessory is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

AVERTISSEMENT: L'utilisateur de cet accessoire est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

The DHR Dielectric Accessory works in conjunction with an LCR meter and the DHR Rheometer. Refer to your instrument's appropriate Getting Started Guide and your LCR meter documentation for important information regarding safety and operation.

Required Equipment

While operating this accessory, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

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Chapter 1:

Introducing the Dielectric Accessory

Overview

The DHR Dielectric Accessory uses a material characterization technique similar to dynamic mechanical analysis, except that an oscillating electrical field (AC field) is used (as opposed to a mechanical force [stress]), and the oscillating strain is a stored charge (Q) in the sample. Dielectric analysis is a very powerful technique for characterizing polar materials such as PVC, PVDF, PMMA, and PVA. It is also very powerful for monitoring curing kinetics of materials such as epoxy and urethane systems. The wide range of frequencies available in dielectric analysis (20 Hz to 1 MHz and higher depending on the LCR Meter also extends the measurement range over traditional dynamic mechanical analysis (typically below 100 Hz).

In dielectric analysis, the sample basically behaves like a capacitor. Q is measured as its derivative dQ/dt = AC current. When the oscillating electric field is applied to a material, random dipoles in the material orient with the field (as shown in Figure 1). The Dielectric Accessory measures the degree to which the sample is storing a charge (capacitance) or passing the charge (conductance) through its bulk.

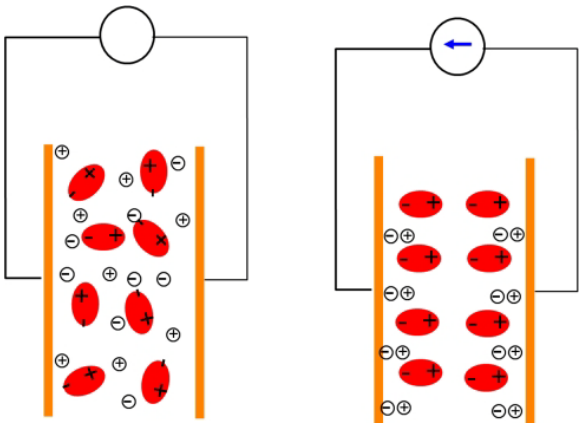

Figure 1 Sample behavior during dielectric analysis.

The combination of the Dielectric Accessory with a high-precision DHR rheometer allows for the simultaneous collection of rheological and dielectric information. In addition, dielectric measurements can be run independently of rheological measurements and still benefit from the instrument test platform. This is because dielectric measurements are conducted on a material sandwiched between parallel plates (with some axial force applied to ensure good surface contact) and surrounded by some type of temperature control. Gap temperature compensation keeps the gap constant, allowing for consistent capacitive measurements.The ETC oven temperature control, axial force control with gap temperature compensation capability, and software for interface, all lead to more accurate dielectric measurements.

For additional information related to theory of dielectric analysis, refer to *Introducing the Dielectric Accessory* in TRIOS Help.

Dielectric Accessory Components

The following section describes the components of the Dielectric Accessory.

LCR Meter

The LCR Meter measures capacitance between the dielectric parallel plate test geometries. The Dielectric Accessory enables the DHR and TRIOS Instrument Control Software to control the LCR Meter; thus, operation requires no user intervention.

Only the Agilent E4980A model LCR Meter is compatible with the DHR. Refer to Table 1 for specifications.

Dielectric Parallel Plate Geometries

The Dielectric Accessory includes a special set of 25-mm parallel plates (shown in Figure 2) which are connected directly to the LCR Meter. The wires from inside the electrodes connect back to the LCR Meter, allowing capacitance and DE loss to be measured across the sample.

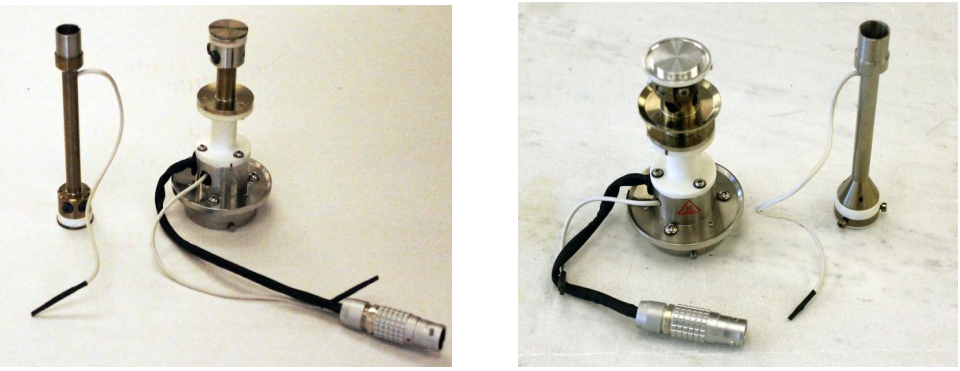

Figure 2 Dielectric parallel plate (left) and disposable dielectric plate (right) geometry.

These special user-installable upper and lower test geometries have been designed for the DHR to measure the dielectric properties of a sample. When you install the upper and lower geometries, the special dielectric connections within the geometries are automatically engaged.

Dielectric Accessory Specifications

Table 1: Dielectric Accessory Specifications

Item/Area	Specification
Geometry	25-mm parallel plate
Temperature system compatibility	ETC
Controller to LCR Meter interface	USB
Temperature range	-150 to 350°C
LCR Meter compatibility	Agilent model E4980A
Dielectric frequency range with LCR Meter Agilent model E4980A	20 Hz to 2 MHz
Dielectric potential for LCR Meter Agilent model E4980A	0.005 to 20 Volts*

* The Agilent E4980A LCR Meter requires the installation of OPT001 on them in order to reach the maximum of 20 Volts. Without OPT001, the maximum is 2 Volts. Note that LCR Meters purchased from TA Instruments already have OPT001 installed.

Chapter 2:

Setting Up the Dielectric Accessory

This chapter briefly describes the setup of the Dielectric Accessory on the DHR.

The Dielectric Accessory is used in conjunction with Agilent LCR Meter model E4980A. For safety and other important information related to the LCR Meter, refer to the manufacturer's documentation.

Preparing the Instrument

NOTE: Refer to your instrument documentation for detailed procedures on removing and reassembling components.

1 Raise the stage to maximum height.

2 Remove all upper and lower test geometries (if installed).

3 Thoroughly inspect the geometry mounting surfaces (that is, the Smart Swap™ base and the motor shaft) and clean off any material that may interfere with the mounting of the dielectric lower geometry.

Installing the Dielectric Accessory on the Instrument

The Dielectric Accessory BNC junction boxes can be installed on the left or right side of the instrument. Also, ensure that there is ample room on your benchtop for the LCR Meter.

Follow the instructions below to install the Dielectric Accessory on the instrument.

Installing the BNC Junction Boxes

The BNC junction boxes are preferably installed on the left of the instrument.

1 Locate the accessory threaded holes on the accessory mounting ring below the stress head (see the image below). This will be the location for the upper BNC junction box installation.

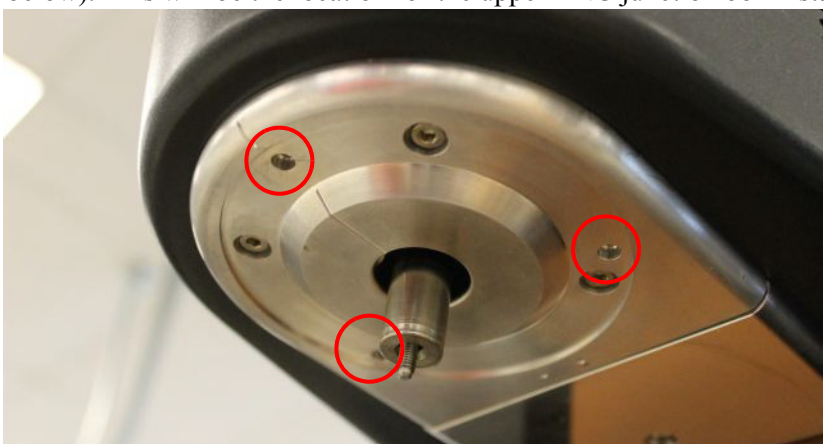

Figure 3 Mounting ring for upper BNC junction box.

2 Install the upper BNC junction box by aligning the holes in the support ring with the threads in the mounting ring. The BNC box is oriented to the left. Tighten the Phillips head screws to properly secure the box in place.

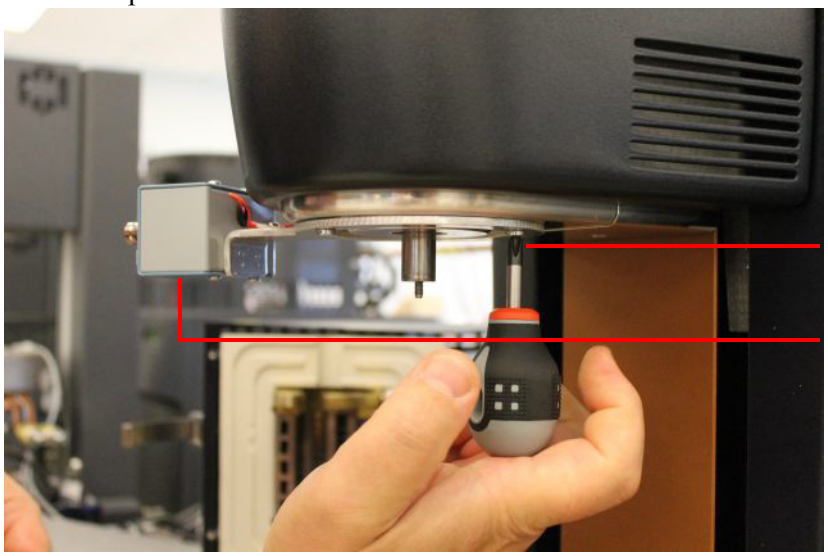

Figure 4 Upper BNC junction box installed on the mounting ring. The ring with BNC junction box is oriented to the left.

3 Install the lower BNC junction box onto the instrument base as follows:

a Unscrew the bottom cover to expose the location surface for the lower BNC junction box support ring.

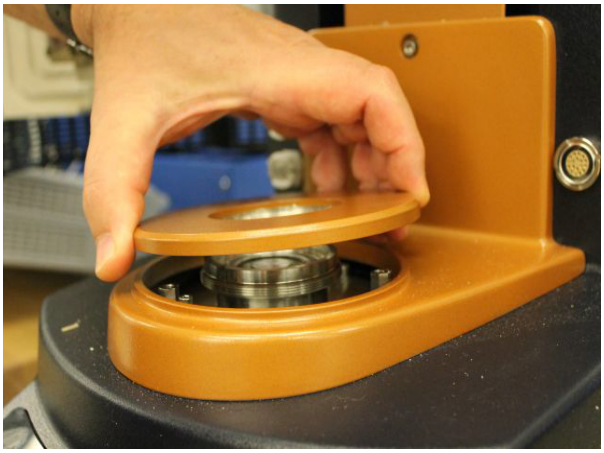

Figure 5 Base cover unscrewed to expose the mounting surface for the lower BNC junction box support ring.

b Remove the three hex screws using the hex key provided in the kit, and insert the 3 support stands instead.

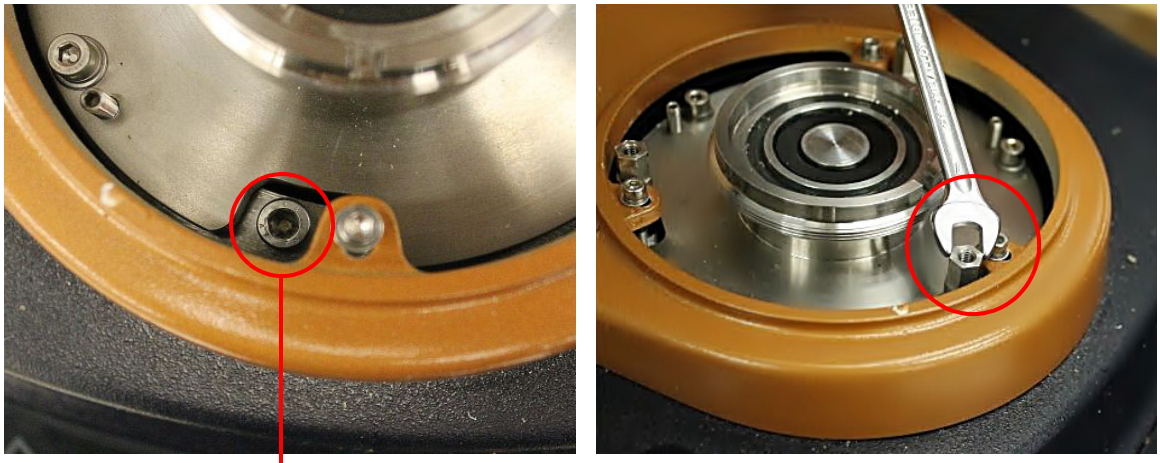

Figure 6 Inserting support stand.

c Install the lower BNC junction box by aligning the holes in the BNC junction box support ring with the threaded holes on the support stands. The BNC junction box is on the left side (shown in Figure 7).

d Secure the lower BNC junction box to the mounting base by tightening the Phillips head screws.

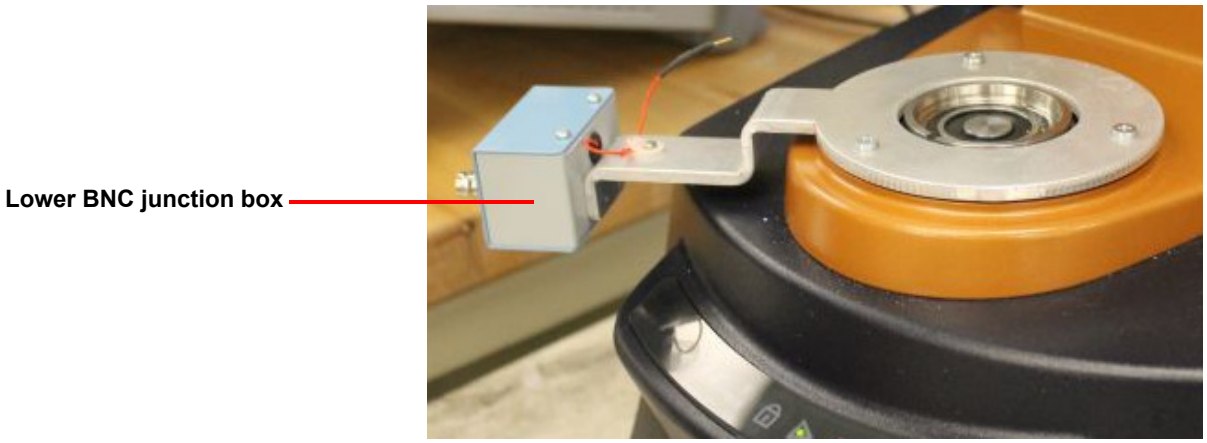

Figure 7 Lower BNC junction box installed on the mounting base.

4 Proceed to the next section, "Installing the Geometries".

Installing the Geometries

The dielectric lower (standard and disposable) geometries are integrated in the Smart Swap™ base for the ETC. The upper geometry mounts to the motor shaft. Refer to Figure 8 for reference. A temperature sensor is integrated into the bottom plate.

Standard Geometry

1 Install the lower geometry as you would any other Smart Swap base. Ensure that the geometry cable does not interfere with the ETC doors.

NOTE: When the Smart Swap base is attached the displacement is zeroed and only a +/- 8rad rotation of the upper geometry is allowed. This is to protect the cable connection. Therefore, it is good practice to use the key pad lock prior to attaching the base so that when the upper geometry is attached it is in the middle of the allowable displacement range.

2 Connect the lower geometry cable to the lower BNC junction box cable.

3 Install the upper geometry as you would any other upper geometry. Ensure that the geometry cable does not get caught in the ETC door.

4 Connect the upper geometry cable to the upper BNC junction box cable.

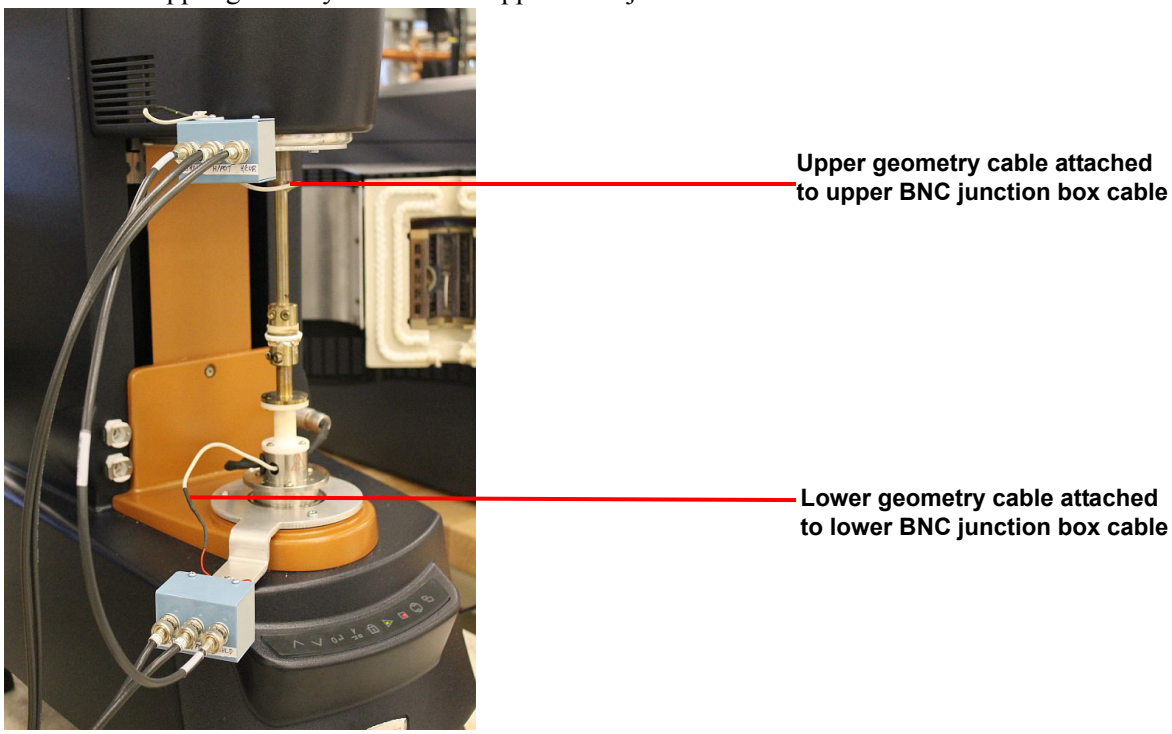

Figure 8 Upper and lower geometries installed and connected to BNC junction boxes.

5 Proceed to the next section, "Installing the LCR Meter".

Disposable Dielectric Parallel Plate Geometry

On the disposable dielectric plate lower and upper geometry, the plates can be easily installed and removed. This is important when the sample cures and the upper and lower plates cannot be separated after the experiment.

Installing the disposable plates

In order to install the disposable plates proceed as follows:

1 Loosen the two clamping screws offset by 90°, shown in Figure 9

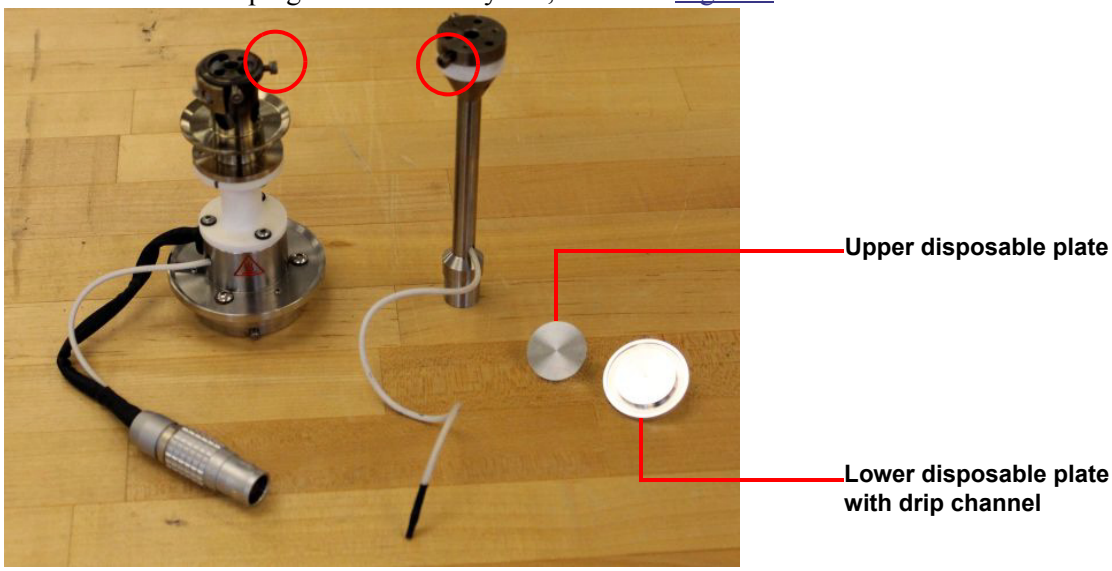

Figure 9 Two 90° offset clamping screws on the upper and lower geometry. It is recommended that you use a plate with drip channel on the bottom geometry.

2 Insert the disposable plates with the clamping shaft into the disposable plate holder and tighten the two screws. Always zero the gap after loading new disposable plates.

Removing the disposable plates

In order to remove the disposable plates after the experiment is completed:

1 Loosen the two clamping screws to release the disposable plates on the lower and upper geometry.

2 Raise the motor head until the disposable plates disengage from the geometry holder.

3 Pull out the plates with the sample.

Installing the LCR Meter

TA Instruments recommends reading the manufacturer's documentation before installing the LCR Meter.

Figure 10 The Agilent E4980A LCR Meter.

1 Place the LCR meter on the bench in close proximity to the instrument. It is recommended that the LCR Meter is placed to the left of the instrument, as the BNC junction boxes are also located to the left.

2 Connect the BNC shield cable from the **SHLD** port on the upper BNC junction box to the **SHLD** port on the lower BNC junction box.

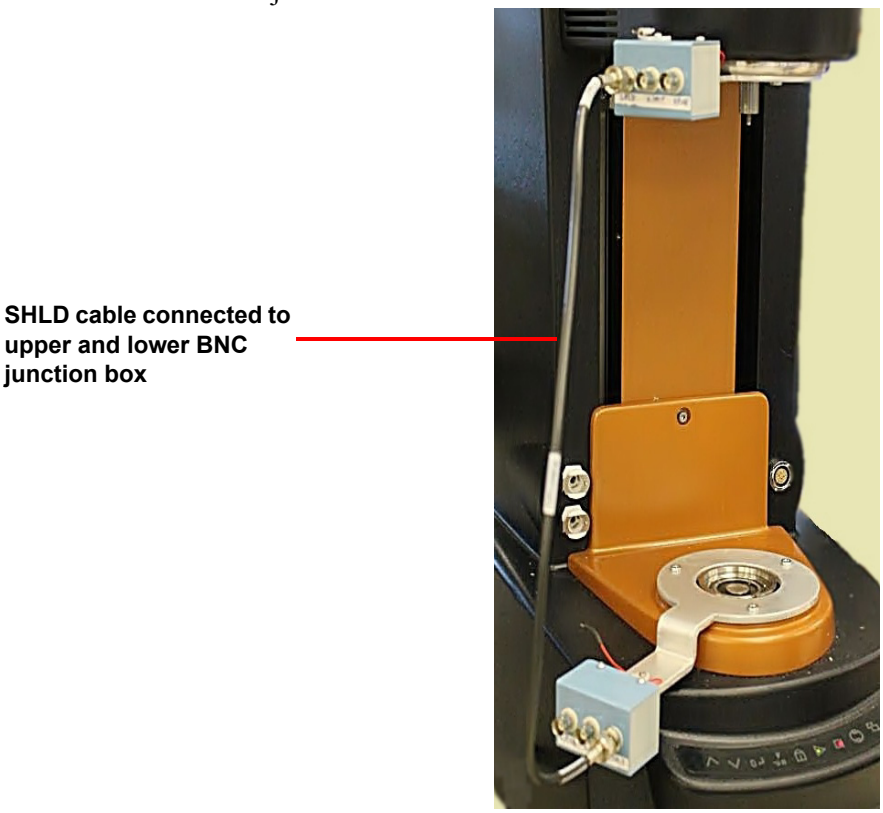

Figure 11 Shield cable connections.

3 Obtain the main BNC wire harness (contains four black coaxial cables).

4 Connect the main BNC wire harness from the upper BNC junction box to the LCR Meter front panel. Refer to Figure 12 for reference.

   a Connect the **HCUR** cable on the main BNC wire harness from the **HCUR** port on the upper BNC junction box to the **HCUR** port on the LCR Meter.

   b Connect the **HPOT** cable on the main BNC wire harness from the **HPOT** port on the upper BNC junction box to the **HPOT** port on the LCR Meter.

5 Connect the main BNC wire harness from the lower BNC junction box to the LCR Meter front panel. Refer to Figure 12 for reference.

   a Connect the **LCUR** cable on the main BNC wire harness from the **LCUR** port on the lower BNC junction box to the **LCUR** port on the LCR Meter.

   b Connect the **LPOT** cable on the main BNC wire harness from the **LPOT** port on the lower BNC junction box to the **LPOT** port on the LCR Meter.

6 AFTER the LCR Meter drivers have been installed on the PC running TRIOS, connect the **USB** cable from the back of the PC to the **USB** port on the back of the LCR Meter. Do not make this connection before the drivers have been installed.

7 Connect the power cable from the rear of the LCR Meter to a power source.

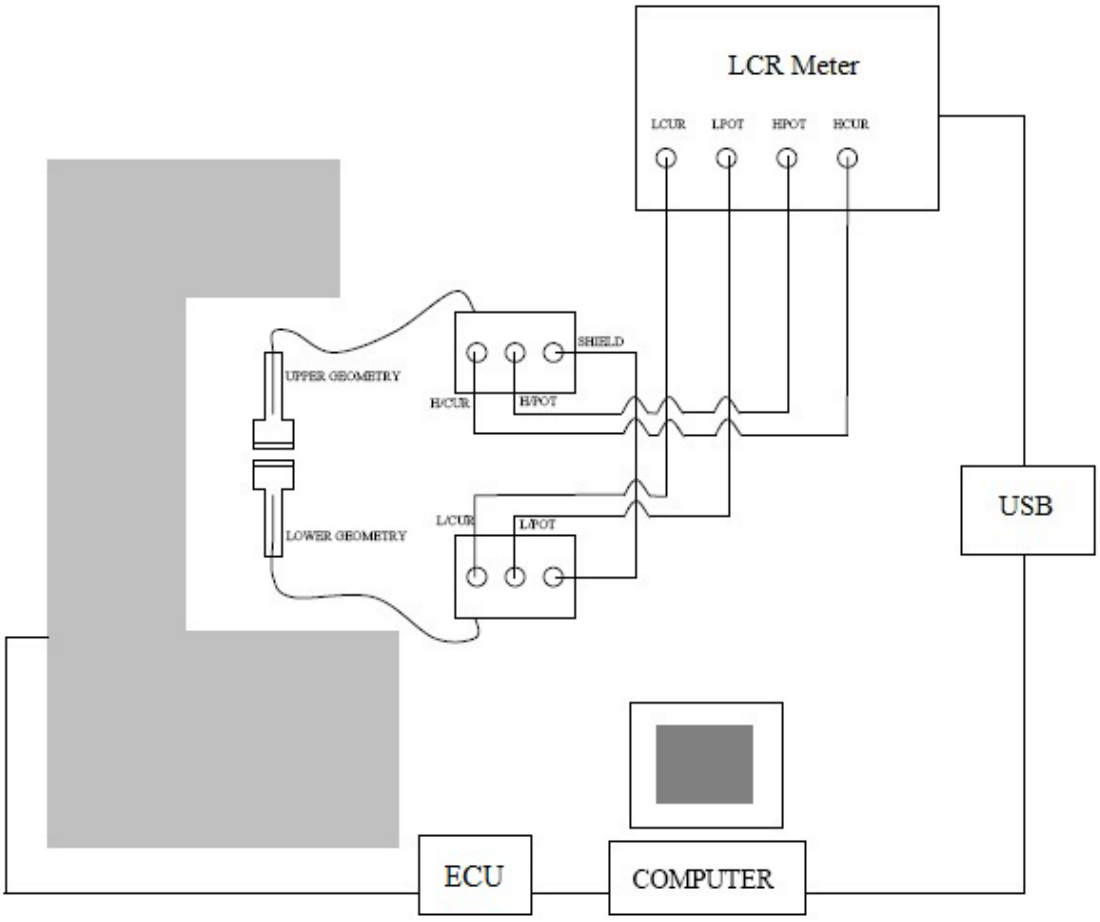

Figure 12 Main BNC wire harness connection block diagram.

This completes installation of the dielectric accessory. Proceed to the next chapter for operational and maintenance information.

Chapter 3:

Operating and Maintaining the Accessory

This chapter briefly describes the operation and maintenance of the Dielectric Accessory.

Establishing Connection with TRIOS

In order to operate the Dielectric Accessory, TRIOS must be able to communicate with the LCR Meter. Since the communication is done via the USB port, the drivers for the LCR meter must be installed on the TRIOS computer before connecting the USB cable. Please refer to the manufacturer's support information to install the drivers. Note that TRIOS V2.5 or higher and the associated instrument firmware version 9.13 or higher are required for control of the LCR Meter.

1. Connect to the instrument through the TRIOS software.
2. Connect the LCR Meter via the USB port and then turn the power on. When the LCR is operational, the message “LCR Meter: Enabled” displays in the TRIOS status bar.
3. Once a dielectric measurement is initiated, the LCR Meter displays the message **TRIOS Controlled**.

Calibrating the LCR Meter

Open and short calibration ensure that the LCR Meter reads the proper impedance at two extremes: during a virtually zero capacitance (very large gap between two parallel plates [*open calibration*]) and during a near zero resistance (two parallel plates in contact [*short calibration*]). Note that cable length is another calibration entry that should be made before doing open and short calibrations.

Perform the open and short calibrations once a day when using the LCR Meter.

For calibration procedures, refer to *Calibrating the LCR Meter* in TRIOS Help.

Operating the Dielectric Accessory

Operating the Dielectric Accessory on the DHR requires the following steps:

1 Selecting and preparing the sample.

2 Selecting and installing the geometry.

3 Setting up your experiment through TRIOS Software.

4 Zeroing the gap at the subsequent, initial experimental temperature.

5 Loading and trimming the sample, as required.

6 Starting the test.

If necessary, calibrate the LCR meter before running any experiments. Refer to "Calibrating the LCR Meter" on page 18 for additional information.

Dielectric measurements can be performed on the instrument in three modes:

Standalone dielectric measurements. In this mode, a dielectric thermal analysis measurement is performed according to a specific temperature and frequency profile. No mechanical excitation and measurements are possible during this mode. Only the axial force control is active to compensate for gap changes when sample dimension changes occur during the measurement. Only dielectric variables are reported in the result file.

Simultaneous dielectric and mechanical measurements. In this mode, a dielectric and a mechanical measurement are performed simultaneously. The dielectric measurement uses a fixed frequency and voltage. The dielectric measurement can be done in conjunction with all available test modes. Mechanical and dielectric variables are reported in the results file.

Hybrid Dielectric DMA measurements. In this mode, oscillatory mechanical and dielectric frequency scans can be combined using various temperature profiles. Mechanical and dielectric variables are reported in the results file.

WARNING: Since the electrodes are connected via a cable to the connector box, the electrodes cannot undergo continuous rotation. Do not attempt to run the Dielectric Accessory simultaneously with large strain (more than one revolution) transient and flow test modes!

AVERTISSEMENT: Vu que les électrodes sont connectés via un câble à la boîte de raccordement, les électrodes ne peuvent pas subir une rotation continue. N'essayez pas d'utiliser l'accessoire diélectrique simultanément avec les modes d'essai transitoire et de détermination d'écoulement de grande tension (plus d'un tour)!

CAUTION: To avoid breaking the electrode cable during the motor calibration, connect the electrodes to the connector box ONLY AFTER the instrument is powered up.

MISE EN GARDE: Pour éviter de rompre le câble de l'électrode pendant l'étalonnage du moteur, raccordez les électrodes au boîtier du connecteur SEULEMENT APRÈS la mise sous tension de l'instrument.

CAUTION: To avoid possible damage to the Dielectric Accessory, do not run experiments above 350°C.

MISE EN GARDE: Pour éviter de possibles dégâts à l'accessoire diélectrique, n'effectuez pas d'expérience au-delà de 350°C.

For more detailed instructions on operating the Dielectric Accessory and setting up dielectric experiments, refer to *Operating the Dielectric Accessory* in TRIOS Help.

Maintaining the Dielectric Accessory

The maintenance required for the Dielectric Accessory consists of the following tasks:

- Thoroughly clean the plate geometries if they become visibly soiled.
- Perform open and short calibrations on the LCR Meter once a day when performing dielectric measurements (refer to "Calibrating the LCR Meter" on page 18 for additional information).

Replacement Parts

The table below lists the replacement parts available for the Dielectric Accessory.

Table 2: Replacement Parts for Dielectric Accessory

Part Number	Description
546602.901	DHR 25 mm Upper Dielectric Geometry
546603.901	DHR 25 mm Lower Dielectric Geometry
546601.901	DHR Dielectric Accessory Kit
523080.937	Aluminum Disposable Plates 8 mm (package of 20)
523250.927	Aluminum Disposable Plates 25 mm (package of 100)
523250.928	Aluminum Disposable Plates 25 mm with drip channel (package of 100)
520400.927	Aluminum Disposable Plates 40 mm (package of 100)

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