ARES-G2 / RSA-G2

Dielectric Accessory

Getting Started Guide

Revision E Issued November 2013

Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without 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
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- TA Instruments End-User License Agreement
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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 ARES-G2 / RSA-G2 DETA Accessory works in conjunction with an LCR meter (hereafter referred to as the dielectric bridge) and the ARES-G2 Rheometer or RSA-G2 Dynamic Mechanical Analyzer. Refer to your instrument's appropriate Getting Started Guide and your dielectric bridge 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.

Table of Contents

Chapter 1:

Introducing the Dielectric Accessory

Overview

The ARES-G2 / RSA-G2 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 bridge) 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.

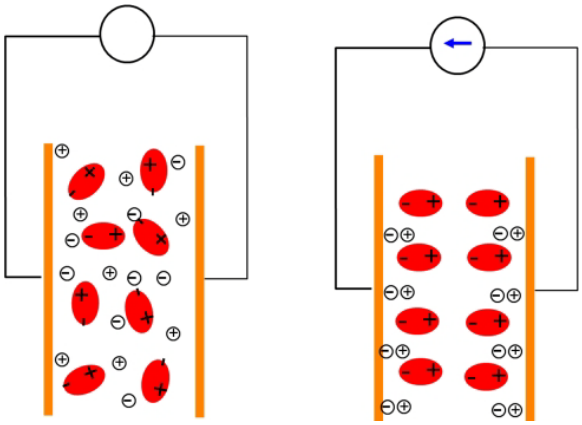

Figure 1 Sample behavior during dielectric analysis.

The combination of the dielectric option with a high-precision ARES-G2 Rheometer or RSA-G2 Dynamic Mechanical Analyzer (DMA) 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 Forced Convection Oven (FCO) 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 included with the dielectric accessory.

LCR Meter

The LCR Meter measures capacitance between the dielectric parallel plate test geometries. The Dielectric Accessory option enables the ARES-G2 or RSA-G2 and TRIOS Instrument Control Software to control the LCR Meter; thus, operation requires no user intervention.

Table 1 below lists the LCR Meters that are compatible with the ARES-G2 and RSA-G2.

Table 1: ARES-G2 / RSA-G2 and LCR Meter Compatibility*

LCR Meter	Frequency	AC Test Signal (potential)
Agilent 4284A (Discontinued)	20 Hz to 1 MHz	0.005 to 20 Volts**
Agilent 4285A	75 kHz to 30 MHz	0.005 to 2 Volts*
Agilent E4980A	20 Hz to 2 MHz	0.005 to 20 Volts**

* The manufacturer's model- and range-specific operating constraints apply to all LCR Meter output specifications. Please see the manufacturer's published specification for detailed performance limits that apply to the specific LCR Meter and output settings being used.

** The Agilent 4284A and E4980A meters require the installation of OPT001 on them in order to reach the maximum of 20 Volts. Without OPT001, the maximum is 2 Volts. Note that Meters purchased from TA Instruments already have OPT001 installed.

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.

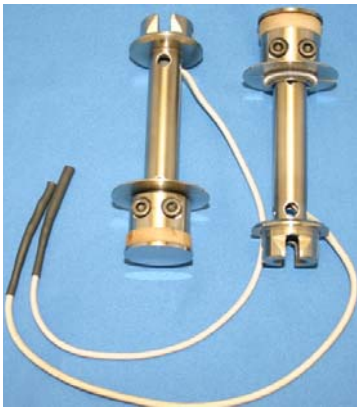

Figure 2 Dielectric parallel plates.

These special user-installable upper and lower test geometries have been designed for the ARES-G2 and RSA-G2 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 2: Dielectric Accessory Specifications

Item/Area	Specification
Geometry	25-mm parallel plate
Temperature system compatibility	FCO
Instrument to LCR Meter interface	IEEE internal to instrument
Temperature range	–150 to 350°C
LCR Meter compatibility	Agilent models 4284A, 4285A, E4980A
Dielectric frequency range with Meter	Depends on LCR Meter selection. See Table 1.
Dielectric potential	Depends on LCR Meter selection. See Table 1.

Chapter 2:

Setting Up the Dielectric Accessory

This chapter briefly describes the setup of the dielectric accessory on the ARES-G2 Rheometer or RSA-G2 DMA.

The Dielectric Accessory is used in conjunction with an Agilent LCR Meter; see Table 2 for Meter options. For safety and other important information related to your particular LCR Meter, refer to the manufacturer's documentation.

NOTE: Proper installation of the Dielectric Accessory requires a TA Instruments Service Representative to install a communication board into the instrument. Contact TA Instruments for more information.

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 as well as upper and lower PRTs (if installed).

3 Thoroughly inspect the geometry mounting surfaces (that is, the transducer anvil and the motor anvil) 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 and LCR Meter can be installed on either the right or left side of the instrument, but should be installed on the side opposite the FCO. Regardless of the position you choose for installation, note that the junction boxes and LCR Meter should be installed on the same 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. For this manual, the installation will be performed on the left side of the ARES-G2 Rheometer.

Installing the BNC Junction Boxes

The BNC junction boxes can be installed on either the left or right side of the instrument, but on the opposed side of the FCO (if installed).

1 Locate the 1/4-inch accessory holes on either side of the transducer bracket (see the image below). This will be the location for the upper BNC junction box installation.

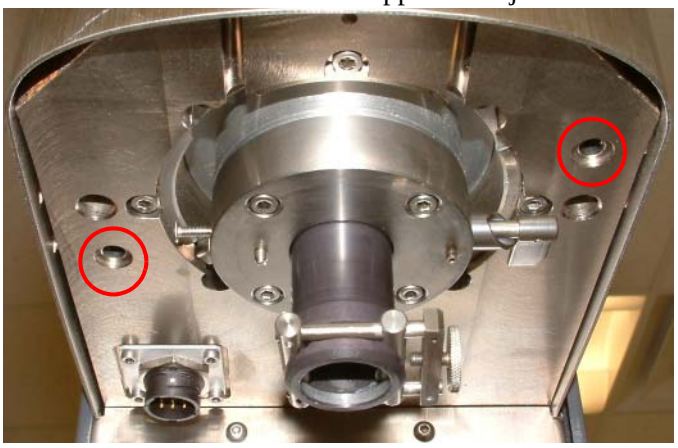

Figure 3 Installation locations for upper BNC junction box.

2 Install the upper BNC junction box into the 1/4-inch accessory hole located on the transducer bracket, and use a 1.5-mm Allen wrench to tighten the cone point set screw on the side of the stage. Properly orient and secure the box into place.

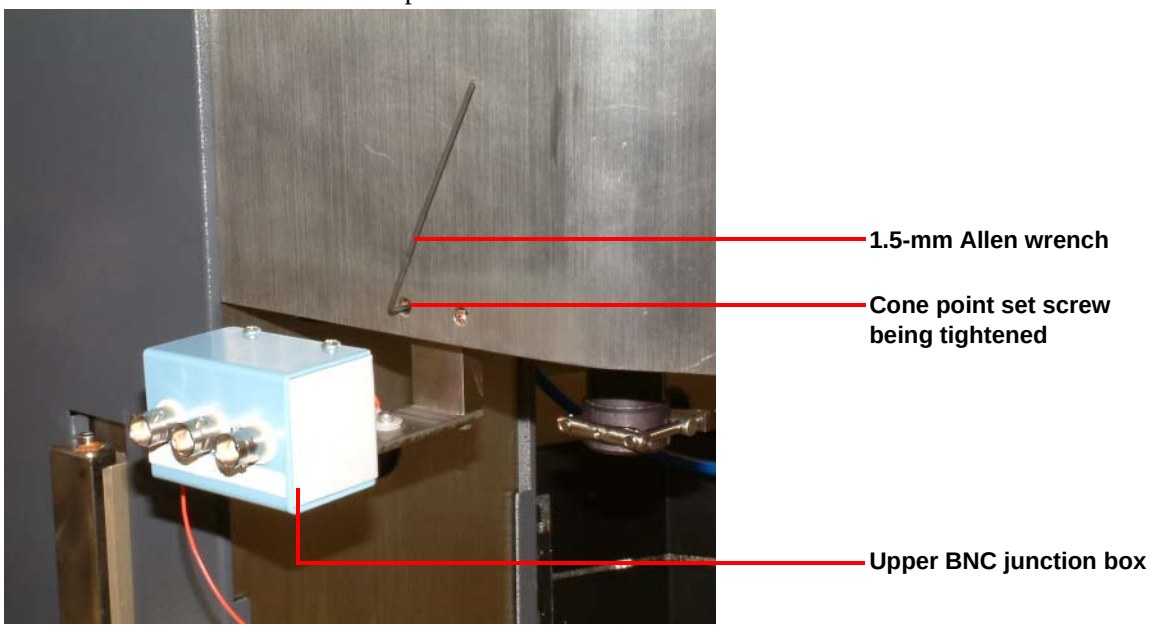

Figure 4 Upper BNC junction box installed on left side of transducer bracket.

3 Install the lower BNC junction box below the upper BNC junction box.

a Use a 3-mm Allen wrench (on the ARES-G2) or a 4-mm Allen wrench (on the RSA-G2) to remove the left- or right-side motor cover screw (remove the screw that is directly below the upper BNC junction box).

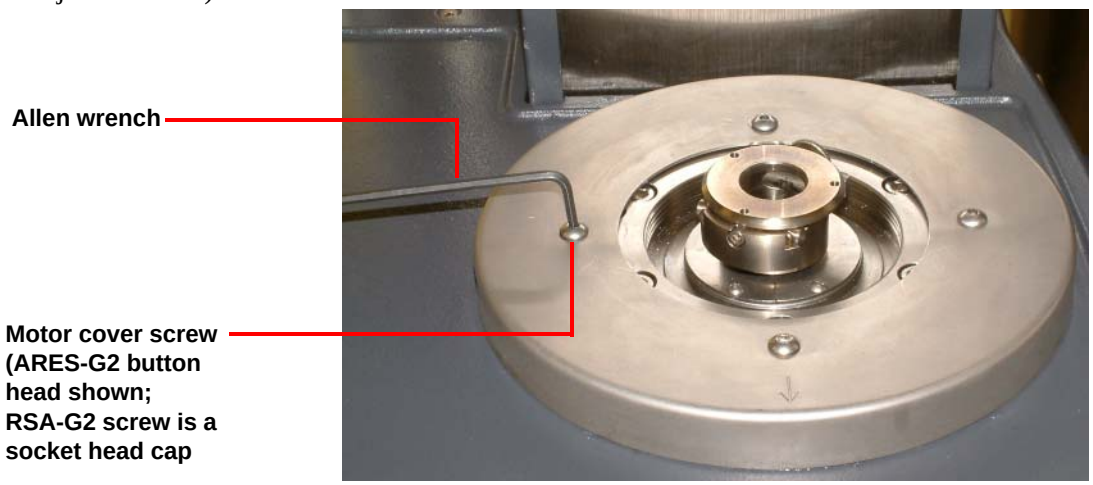

Figure 5 Motor cover screw being removed with 3-mm Allen wrench on the ARES-G2.

b Install the lower BNC junction box over the hole, making sure to align the hole on the bracket with the hole on the motor cover. Ensure that the alignment pins at the end of the bracket (shown in Figure 6) are facing downwards.

c Secure the lower BNC junction box to the motor cover.

- If installing the dielectric accessory on the ARES-G2, use the motor cover screw removed in step a above.
- If installing the dielectric accessory on the RSA-G2, use the M5 x 30-mm long socket head cap screw supplied in the kit.

Note that two pins on the end of the lower BNC junction box bracket ensure proper alignment with the motor cover.

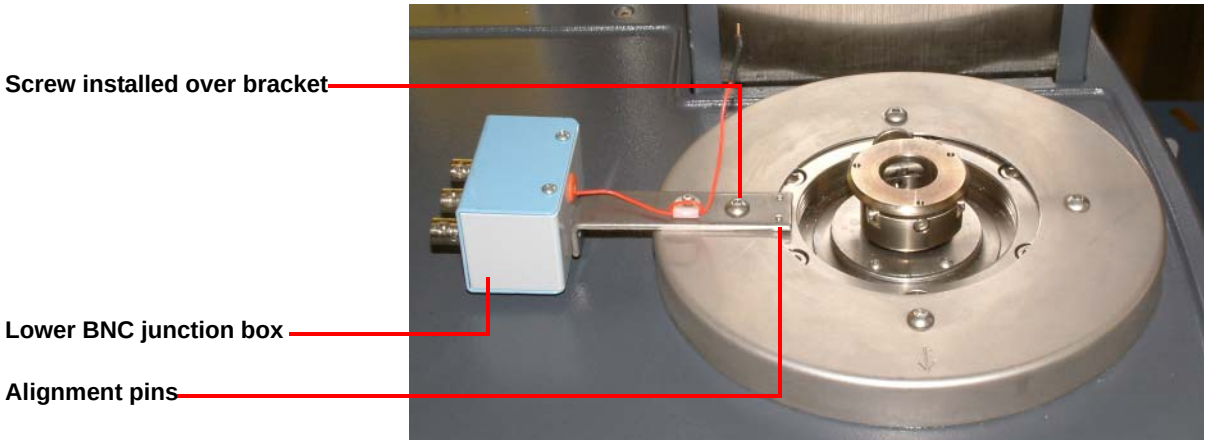

Figure 6 Lower BNC junction box installed on motor cover.

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

Installing the Geometries

The dielectric upper and lower geometries are identical; you can install either geometry into the upper and lower geometry position. Refer to Figure 7 for reference. Note that the motor and transducer geometry PRTs are NOT used with these geometries. Temperature is read from the sample (middle) PRT located inside the oven.

1 Install the upper geometry as you would any other upper geometry. Ensure that the geometry cable faces towards the back of the instrument.

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

3 Install the lower geometry as you would any other lower geometry. Ensure that the geometry cable faces towards the back of the instrument.

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

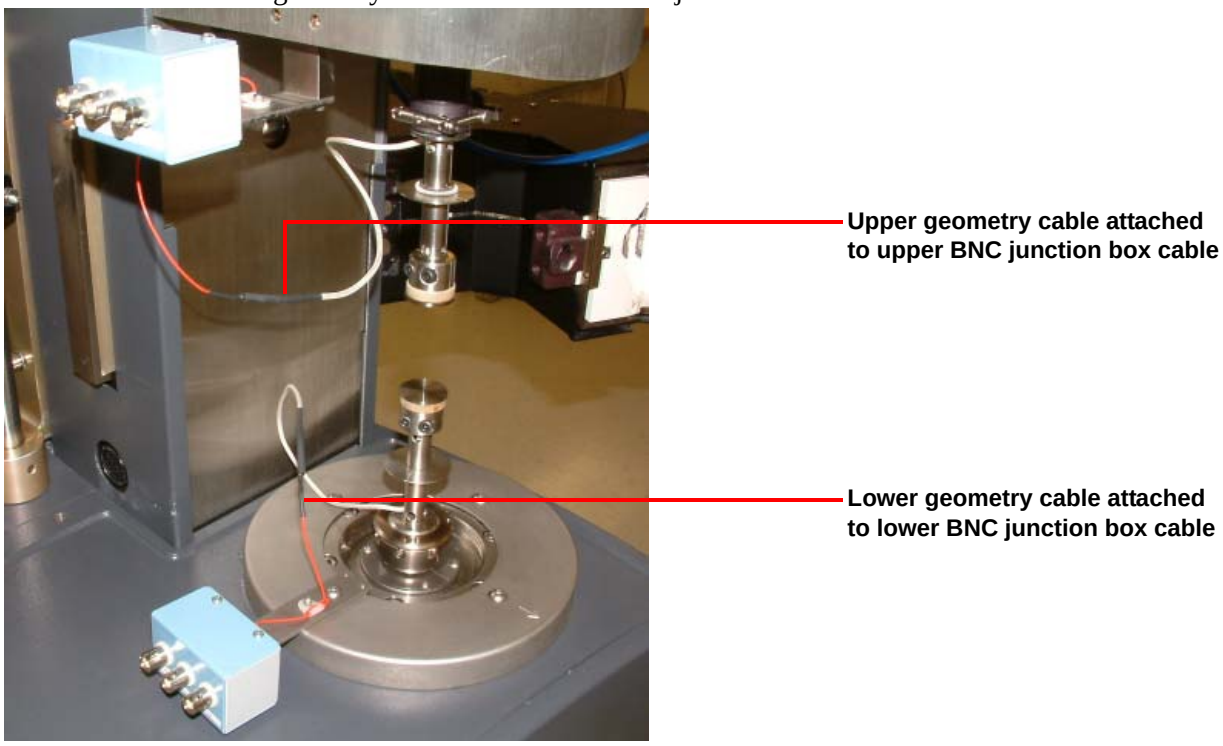

Figure 7 Upper and lower geometries installed and connected to BNC junction boxes.

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

Installing the LCR Meter

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

Figure 8 The Agilent E4980A LCR Meter.

1. Place the LCR Meter on the bench in close proximity to the instrument. Ensure that the LCR Meter is on the same side of the instrument as the BNC junction boxes.
2. Connect the shield cable from the **SHLD** port on the upper BNC junction box to the **SHLD** port on the lower BNC junction box.

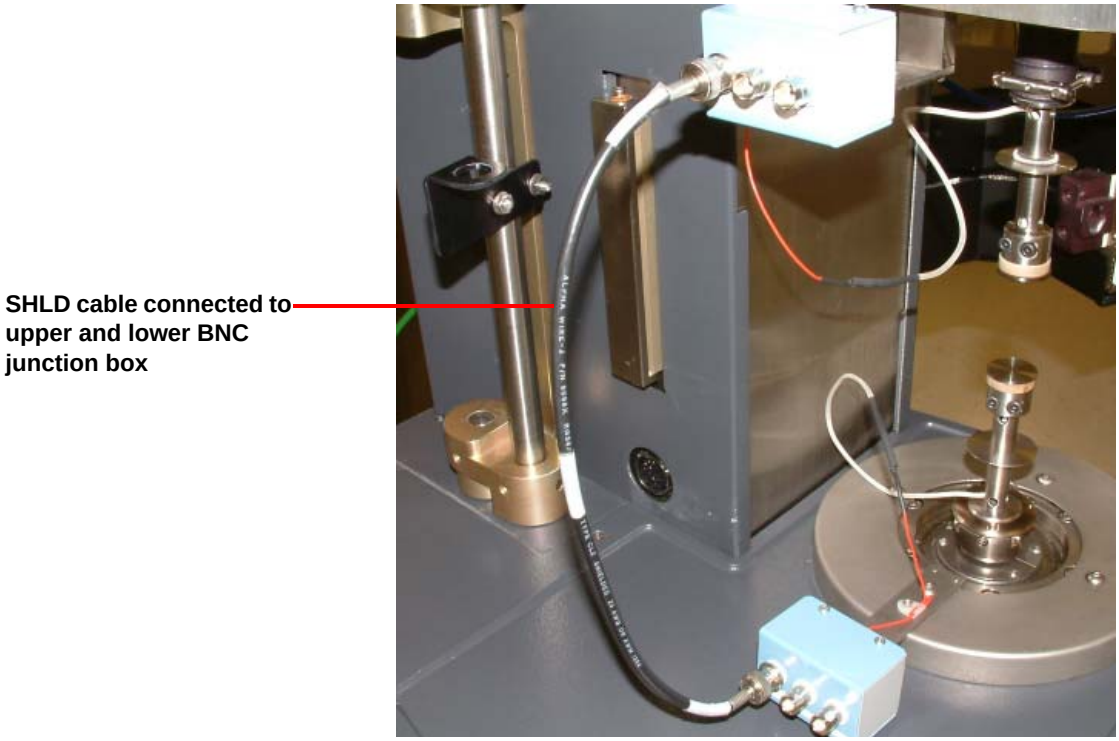

Figure 9 Shield cable connections.

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 10 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 10 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 Connect the GPIB cable from the back of the instrument to the **GPIB** port on the back of the LCR Meter.

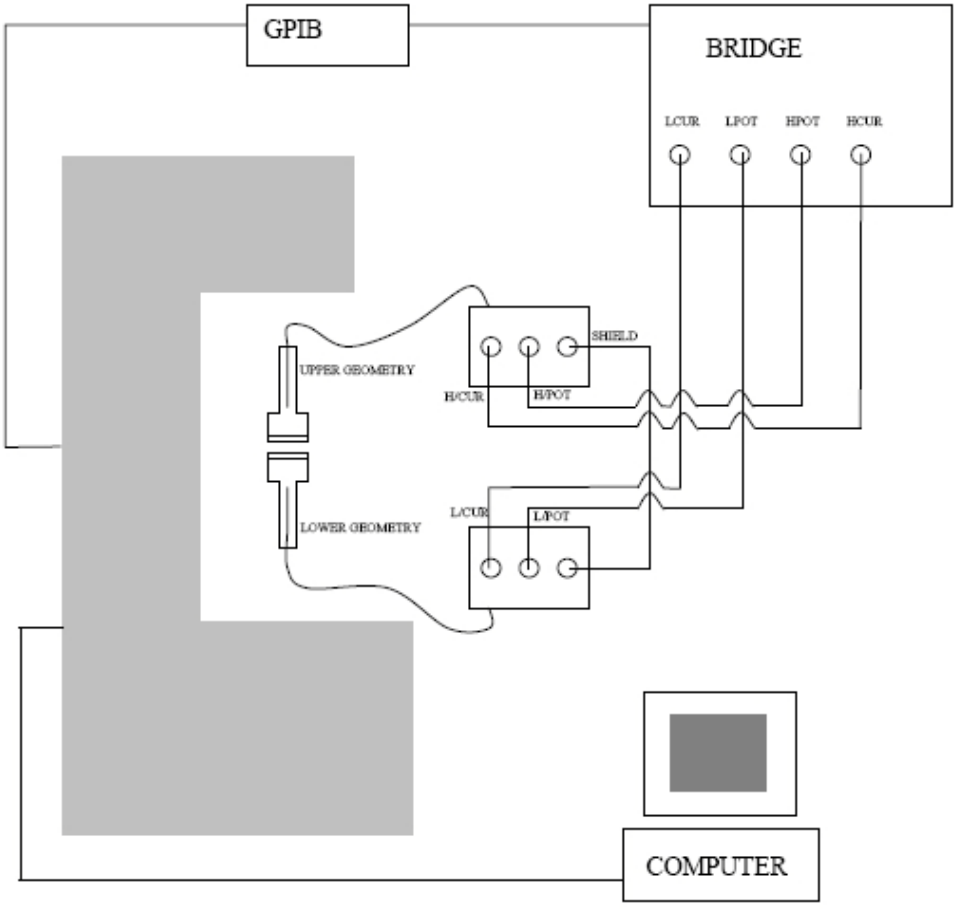

Figure 10 Main BNC wire harness connection block diagram.

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

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, you must first establish a connection through the instrument control software (TRIOS Software). Note that TRIOS V1.8 or higher and the associated instrument firmware are required for Dielectric Accessory control.

1 Power on the LCR Meter and allow it to initialize.

2 Connect to the instrument through the TRIOS Software.

3 Click the TRIOS icon at the top-left corner of the application.

4 Select **Options** from the drop-down menu.

5 Select **ARES G2** or **RSA G2** > **Accessory** to display a list of hardware configuration options.

6 In the **Active Hardware Accessory** section, select **Dielectric Analysis**.

7 In the **Dielectric Analysis** section, select the applicable **LCR Meter**. TRIOS automatically enters the allowable **Voltage Range** and **Frequency Range** for the selected LCR Meter.

If desired, click **Detect** to confirm communication between the instrument and the LCR Meter.

8 Select **OK** to save the configuration and close the TRIOS Options window.

9 Once the dielectric measurement starts, the LCR Meter displays the message **AresG2 Controlled** or **RSAG2 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 ARES-G2 or RSA-G2 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 16 for additional information.

Dielectric measurements can be performed on the instrument in two 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. Instrument 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 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!

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 16 for additional information).

Replacement Parts

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

Table 3: Replacement Parts for Dielectric Accessory

Part Number	Description
402553.901*	25-mm dielectric geometry for **ARES-G2**
402614.901*	25-mm dielectric geometry for **RSA-G2**

*This part number orders ONE dielectric geometry. For a pair of dielectric geometries, order two of the desired item.

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