

## STANDARD OPERATING PROCEDURE

Dielectric Instrument HP Precision LCR Meter 4284A (20 Hz – 1 MHz) Page 1 of 7	Version: 1.0	Date: 08/16/12	Written by: Rosa Graczyk Reviewed by: Whitney Schmidt Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University

**Chemistry Department Safety Office: Gilbert Hall Room 153**  
**Subramanian Research Group: 7-6750**  
**Emergency Medical Services: 911**  
**Campus Student Health Center: 7-9355**  
**Poison Control: 9-1-800-222-1222**  
**OSU Environmental Health and Safety: 7-2273**  
**Campus Security: 7-7000**

### USE OF DIELECTRIC INSTRUMENT

#### 1. Introduction

This SOP discusses the procedure and safety guidelines for the use of the dielectric instrument (HP Precision LCR Meter 4284A (20 Hz – 1 MHz)) located in Gilbert Hall Room 214.

#### 2. Scope

This procedure applies to all staff, students and visitors of the Subramanian Research Group in the Department of Chemistry at Oregon State University that work in the laboratory and have the potential to use the dielectric instrument.

#### 3. Safety

- Personnel who operate the dielectric instrument must be trained to understand proper use, maintenance and must never operate alone. Improper handling or use can result in severe body injuries.

##### **General precautions:**

- Personal protective equipment (PPE) including safety glasses, protective laboratory gloves, closed-toed footwear and laboratory coat must be worn during operation of the dielectric instrument.
- Only trained laboratory personnel can operate the apparatus.
- If someone is not available from the Subramanian group for weekend use, the dielectric instrument should not be used.
- **Improper use may lead to severe instrument damage and bodily harm.**

##### **Risk of injury:**

- High temperature furnace.
- High frequency meter (20 Hz – 1 MHz).

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### **Incident Response:**

- If any injury occurs seek immediate first aid or, if necessary, seek medical assistance by calling 911.
- A colleague should be on hand for assistance; never operate dielectric instrument alone.
- Notify Subramanian group lab contact of any incident after appropriate aid has been received.

### 4. **Training and Competency**

The trainee must have already mastered an understanding of and have been given the instruction in the use of the dielectric instrument by an approved trainer (the instrument supervisor or any trained member of the Subramanian Research Group). Competency will be assessed by close observation of the trainee by the instrument supervisor or an approved trainer. The training records are attached at the end of this SOP, pg5.

### 5. **Equipment and Maintenance / Handling**

- Turn on the HP LCR meter and allow to stabilize for at least one hour before use.
- Ensure that the platinum plates of the sample holder are clean of residue before loading a sample and after sample removal. Clean with isopropyl alcohol and a Kim Wipe if necessary.
- All materials pertaining to the dielectric instrument are located in the Dielectric drawer under the instrument.
- The sample must be securely held in the center of the platinum plates with no space between the sample and the plate.
- Ensure that the internal and external sample holder bolts are secure and place insulation around the sample holder in the furnace before starting a measurement.
- Corrections with air (open) and a copper plate (closed) are necessary before running a measurement.
- Carefully clean the surrounding area after each measurement; make sure to not inhale the dust particles of the insulation.



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### 6. Operating Procedures

#### Pellet Preparation:

1. Sand the faces of the pellet to produce an even thickness.
2. Measure pellet thickness, diameter, and weight to ensure that the percent theoretical density is above 75%.
3. If the sample is less than 75% theoretical density, it may be necessary to ball mill the sample and add a binder to increase the pellet density.
4. Paint faces of the pellet with silver coating and allow to dry, preferably overnight.
5. As a precaution, measure the resistivity of the sample after the silver coating is dry, the samples must be completely insulating in order for an accurate measurement to take place.

#### LCR Setup:



1. Press MEAS SETUP, which is a soft key above the 4 directional cursors in the center of the meter.



2. Press the soft key for Correction, located to the side of the display screen.



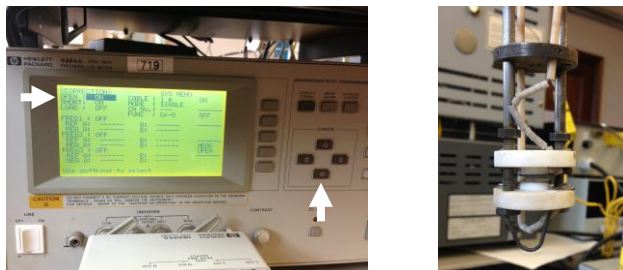
3. Press the soft key “down” cursor to highlight the word OPEN. Make sure that the platinum plates are approximately the same distance apart as the thickness of your pellet.  
\*\*This will allow for the free space correction to be completed, so the distance

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between plates of the sample holder must be approximately the same thickness as the sample.\*\*



4. Press the soft key for MEAS OPEN, located in the display screen.

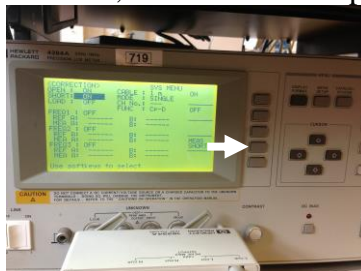


5. Wait for the correction to be completed and saved.
6. Press the soft key “down” cursor to highlight the word SHORT. Place a copper plate, approximately the same size as the sample, between the platinum plates. Make sure there is good contact between the surface of the copper pellet and the platinum plates, no space should be between them.

\*\*This will allow for the system to recognize conductivity between the plates, so a pellet of copper approximately the same size of the sample must be placed between plates of the sample holder.\*\*



7. Press the soft key for MEAS SHORT, located in the display screen.



8. Wait for the correction to be completed and saved.
9. Remove copper pellet and secure sample between the plates, load the sample holder into metal sleeve and slide the sleeve into the tube furnace if high temperature measurements are to be completed.

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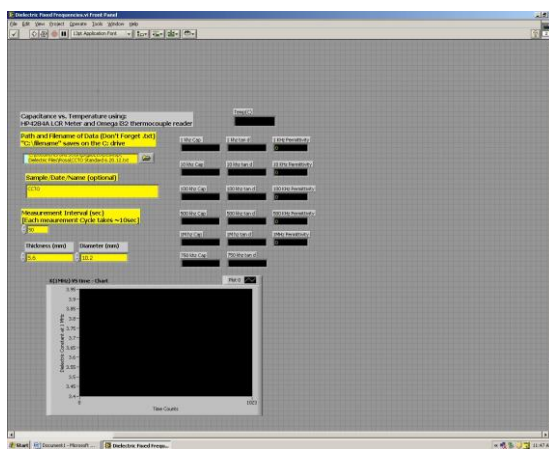
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10. The LCR meter should be ready for use; the display will revert to the home screen after some time.

**\*\*If an error message appears during the SHORT correction, the system is accurate with only the OPEN correction at room temperatures, and generally below 100°C. There is an additional tong attachment in the top Dielectric drawer, which allows for both the OPEN and SHORT corrections to be completed and room temperature data to be collected. \*\***

### LabView Setup:



1. Open LabView from the desktop.
2. In the main screen, click to open the “Dielectric Fixed Frequency” file.
3. Click on the folder icon to create a new sample/run file, press OK  
**\*\*Remember to save/name the file as a .txt\*\*\***
4. Input sample info in the available box.
  - a. Sample number and composition, weight, etc.
5. Input Measurement Interval (in seconds)
  - a. 50 is appropriate for 1 measurement every minute.
6. Input sample thickness and diameter (in mm).
7. Press the **⇒** icon at the top of the screen to start the run.
8. When the run is completed, which is dependent on the temperature sequence of the furnace press the **●** icon to stop the run, the program will continue to run until this icon is pressed.
9. The data is located in a Data folder on the desktop as a delimited .txt, which can be opened in Excel.
10. A column of  $\kappa$ ,  $\tan \delta$ , and capacitance will be supplied for each frequency

### Furnace Setup:

1. Set the temperature sequence for the measurement with the furnace interface. Instructions for operating the furnace should be located next to the instrument.
2. After the run has been stopped in the LabView Program, turn the furnace off and remove the sample holder from the furnace.

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3. Ensure that the surrounding area and sample holder is clean after finishing a measurement.

### 7. Controls and Calibrations

Follow the protocol for the LCR Setup to ensure that the proper corrections are completed before a sample is measured.

### 8. Waste Disposal

Carefully remove sample from holder and store appropriately.

### 9. Relevant Documents / References

N/A

### 10. Signage / Summaries / Templates

Competency Training Records Form – Attached, see page 5.

- Copies of this form are to be stored and filed and in the Dielectric drawer, Gilbert Hall 214.

