solartron analytical

# 1296A

## DIELECTRIC INTERFACE

## USER GUIDE

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# USER GUIDE

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*Solartron pursues a policy of continuous development and product improvement.*
*The specification in this document may therefore be changed without notice.*

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# DECLARATION OF CONFORMITY

CE

**The directives covered by this declaration**

73/23/EEC Low voltage Equipment Directive, amended by 93/68/EEC

89/336/EEC Electromagnetic Compatibility Directive, amended by 92/31/EEC & 93/68/EEC

**Product(s)**

## 1296A Dielectric Interface

**Basis on which conformity is being declared**

The product(s) identified above comply with the requirements of the EU directives by meeting the following standards:

| | |
|---|---|
| EN50081-1:1992 | Electromagnetic Compatibility – Generic Emission Standard Part 1:Residential, commercial and light industry. |
| EN50082-1:1992 | Electromagnetic Compatibility – Generic Immunity Standard Part 1:Residential, commercial and light industry. |
| EN61010-1:1993 | Safety requirements for electrical equipment for measurement, control and laboratory use, including amendment A2:1995. |

Accordingly the CE mark has been applied to this product.

**Signed**

**For and on behalf of Solartron Analytical, a division of Solartron Group Limited**

**Authority:** **Engineering Manager**

**Date:** **August 1997**

# GENERAL SAFETY PRECAUTIONS

The equipment described in this manual has been designed in accordance with EN61010 "Safety requirements for electrical equipment for measurement, control and laboratory use", and has been supplied in a safe condition. The equipment is intended for electrical measurements only. It should be used for no other purpose.

To avoid injury to an operator or service technician the safety precautions given below, and throughout the manual, must be strictly adhered to, whenever the equipment is operated, serviced or repaired. For specific safety details, please refer to the relevant sections within the manual.

Solartron accept no responsibility for accidents or damage resulting from any failure to comply with these precautions.

**GROUNDING**
To minimise the hazard of electrical shock, it is essential that the equipment be connected to a protective ground through the ac supply cable. The continuity of the ground connection should be checked periodically.

**AC SUPPLY VOLTAGE**
Never operate the equipment from a line voltage or frequency in excess of that specified. Otherwise, the insulation of internal components may break down and cause excessive leakage currents.

**FUSES**
Before switching on the equipment, you should check that fuses of the correct rating are fitted. The rating of the ac line fuse must accord with the voltage of the ac supply.

The fuses are accessible from the exterior of the equipment. Should any fuse continually blow, do not insert a fuse of a higher rating. Switch the equipment off, clearly label it "unserviceable" and inform a service technician.

**EXPLOSIVE ATMOSPHERES**
YOU MUST NEVER operate the equipment, or any sensors connected to the equipment, in a potentially explosive atmosphere. The equipment is NOT intrinsically safe and you could possibly cause an explosion.

**SAFETY SYMBOL**
For the guidance and protection of the user, the following safety symbol appears on the equipment:

| ***Symbol*** | ***Meaning*** |
|---|---|
|  | Refer to operating manual for detailed instructions of use. In particular, note the maximum voltage permissible at the input sockets as detailed in the Specification. |

*Continued overleaf.*

## General Safety Precautions (*continued from previous page*)

### NOTES, CAUTIONS AND WARNINGS

For the guidance and protection of the user, **Notes**, **Cautions** and **Warnings** appear throughout the manual. The significance of these is as follows:

| | |
|---|---|
| **NOTES** | highlight important information for the reader's special attention. |
| **CAUTIONS** | guide the reader in avoiding damage to the equipment. |
| **WARNINGS** | guide the reader in avoiding a hazard that could cause injury or death. |

### AVOID UNSAFE EQUIPMENT

The equipment may be unsafe if any of the following statements apply:

- Equipment shows visible damage.
- Equipment has failed to perform an intended operation.
- Equipment has been stored in unfavourable conditions.
- Equipment has been subjected to severe physical stress.

If in any doubt as to the serviceability of the equipment, don't use it. Get it properly checked out by a qualified service technician.

### LIVE CONDUCTORS

When the equipment is connected to its measurement inputs or supply, the opening of covers or removal of parts could expose live conductors. The equipment must be disconnected from all power and signal sources, before it is opened for any adjustment, replacement, maintenance or repair. Only qualified personnel, who should refer to the relevant maintenance documentation, must do adjustments, maintenance or repair.

### EQUIPMENT MODIFICATION

To avoid introducing safety hazards, never install non-standard parts in the equipment, or make any unauthorised modification. To maintain safety, always return the equipment to Solartron for service and repair.

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# 1. Introducing the 1296

## 1.1 WHAT THE 1296 DOES

The 1296 Dielectric Interface is a development of the Chelsea Dielectric Interface. It extends the capability of a Solartron FRA (1260, 1255, 1253, or 1250) and enables the FRA to be used for a wide range of materials testing. The 1296 can also be used with the Hewlett Packard HP4192 FRA –see Section 7 for details.

### 1.1.1 1296 HARDWARE FUNCTIONS

The 1296 unit is controlled by a PC. The PC communicates with the 1296, the FRA, and any other instruments in the system, via the GPIB. The system can also include an ac amplifier, a dc bias generator and a temperature controller.

The basic function of the 1296 hardware is to extend the current measurement range of the FRA downwards, from 6mA to 100fA, thus enabling the measurement of very high impedance. In combination with the software, it also gives additional accuracy to capacitance measurement by enabling the sample capacitance to be compared with high quality internal or external reference capacitors.

### 1.1.2 1296 SOFTWARE FUNCTIONS

The PC software for the 1296 is SMaRT, designed to run with Windows '95, '98, 2000, XP and NT4.

SMaRT offers a simple and effective way of configuring the 1296 system for sample measurement. Once the sample data has been obtained, other windows entries enable you to display the data in graphical form, or to export the data, in its original form or as a graph image, to other windows-based display facilities. Data is also saved to disk, for later recall and analysis.

Sample measurement configurations can be saved on disk, and reloaded for later use.

## 1.2 PC SYSTEM REQUIREMENTS

To achieve a 100% performance with your Dielectric Interface system, your PC must have the following specification.

| | |
|---|---|
| Recommended: | Pentium IV, 2GHz, 512MB RAM, 1GB disk space, 1600×1200 screen, Windows XP |
| Minimum: | Pentium II, 400MHz, 128MB RAM, 100MB disk space, 800×600 screen, Windows 95 |

## 1.3 THE 1296 ACCESSORIES

The following accessories are provided as part of the 1296 product:

- The SMaRT software, on a single CD,
- A 12961 Dielectric Reference Module,
- Four 0.5 metre BNC leads,
- An ac power lead

### 1.3.1 SAMPLE HOLDERS

The 1296 2A sample holder and associated electrode kits (1296 3A and 1296 4A) are available for use with both solid and liquids. More details are given in Section 4.

## 1.4 INSTALLING THE 1296

The installation of the 1296 hardware and software is described in Chapter 2 of this manual.

The hardware installation consists of setting up, where necessary, the GPIB addresses of the instruments used in the 1296 system and cabling the instruments together.

The software installation consists of loading the single CD and following the on-screen instructions.

## 1.5 USING THE 1296

The use of the 1296 is described in the SMaRT User Guide, which gives details of the SMaRT user interface. In essence, the procedure is:

1. Define the instrument set-up. (See SMaRT User Guide, Sect. 3.) This tells SMaRT which instruments are to be used, and their communication interface addresses. For the FRA you also define the input coupling. The procedure for defining the instrument set-up is also described in Section 2.3 of the 1296 User Guide. This is to enable you to set the system up prior to running the initial checkout.

2. Define the project. (See SMaRT User Guide, Section 4.) The project is simply a container or 'shell' for the various files relating to experiments with a common aim.

3. Define the experiment. (See SMaRT User Guide, Sect. 5.) Here you define the experiment parameters, for example: the frequency and level of the ac source, the dc bias, and the measurement parameters. You can set up a single sweep, of the frequency value for example, or you if you wish can set up several nested sweeps.

   Included in the experiment, is the sample definition. (See SMaRT User Guide, Sect. 5.1.1.) Here you enter the information that enables the 1296 to compute the relative permittivity of the sample (or "cell") to be measured. The information you enter can be either the cell dimensions or the empty cell capacitance.

4. Tell the 1296 to start. (See SMaRT User Guide, Sect. 6.)

5. When data has been measured you can:

   a. Display the data as a graph, and print it (see SMaRT User Guide, Sect. 7),

   b. Display the data as a table,

   c. Export the data in .CSV format, as a graph image, to other windows facilities (see SMaRT User Guide, Sect. 7.4).

## 1.6 1296 INFORMATION AVAILABLE

The following sources of information are readily available for the 1296:

- The 1296 User Manual, comprising the 1296 User Guide and the SMaRT User Guide

- The SMaRT Help Text

## 1.7 TECHNICAL SUPPORT

Should you have a problem with your 1296 system that cannot be resolved by referring to the information listed above, then please contact the Technical Support staff at our Farnborough, UK office:

Solartron Analytical

Unit B1 Armstrong Mall,

Farnborough Hampshire,

GU14 0NR England

Telephone: +44 (0) 1252 556800

Fax: +44 (0) 1252 556899

Email: support@solartronanalytical.com

When you contact us, please supply the following information:

a. The make and model of your PC, its operating system and version,

b. The issue of SMaRT you are using,

c. The nature of the problem. (Can it be easily reproduced?)

# 2. Installing the 1296

## 2.1 HARDWARE INSTALLATION

The procedure for installing the 1296 hardware is:

**1.** Check the GPIB address of each instrument to be included in the 1296 system and ensure that each address is unique and valid. The default GPIB addresses assumed by SMaRT are:

| | |
|---|---|
| Dielectric Interface: | 2 |
| FRA: | 12 (and 13) |
| Temperature Controller: | 6 |

The 1296 is supplied with the GPIB address set to 2, and it is recommended that you leave this as it is. However, if a change in the GPIB address of the 1296 is unavoidable then you can remove the top cover and set up the required address as shown in Figure 2-1.

**CAUTION:** Should you remove the top cover of the 1296 to change the GPIB address, you must:

a. Replace the cover promptly, to ensure that desiccant bag remains dry,

b. Before refitting the cover, ensure that the grounding lead is reconnected,

c. When refitting the cover, ensure that the grounding clips are in place, and will make good contact with the cover when this is in place.

Items b. and c. are needed to maintain the emc performance of the 1296.

**2.** For normal operation, connect the 1296 hardware as shown in Figure 2-2.

Note that the FRA input connections vary according to the type of FRA used, as shown in the following table. This shows the correspondence between the connectors on the 1296 (first column) and the connectors on the various types of FRA. *(Details for connecting to the HP4192 are given in Appendix E)*

| **1296** (rear) | **1250** (rear) | **1253** (rear) | **1255** (front) | **1260** (front) |
|---|---|---|---|---|
| Gen. | Gen, Hi* | Gen. | Gen. | Gen. |
| V1 | Chan. 1, Hi* | Chan. 1 | V1, Hi | V1, Hi |
| V2 | Chan. 1, Hi* | Chan. 2 | V2, Hi | V2, Hi |

*Short-circuit the Lo connection to ground, through a shorted BNC connector.

3. For operation with high voltage dc bias or high voltage ac, use the connections detailed in Sections 1.1.1 and 1.1.2.

4. When taking measurements, ensure that the sample and reference cables are not moved, to minimize measurement disturbance due to cable noise.

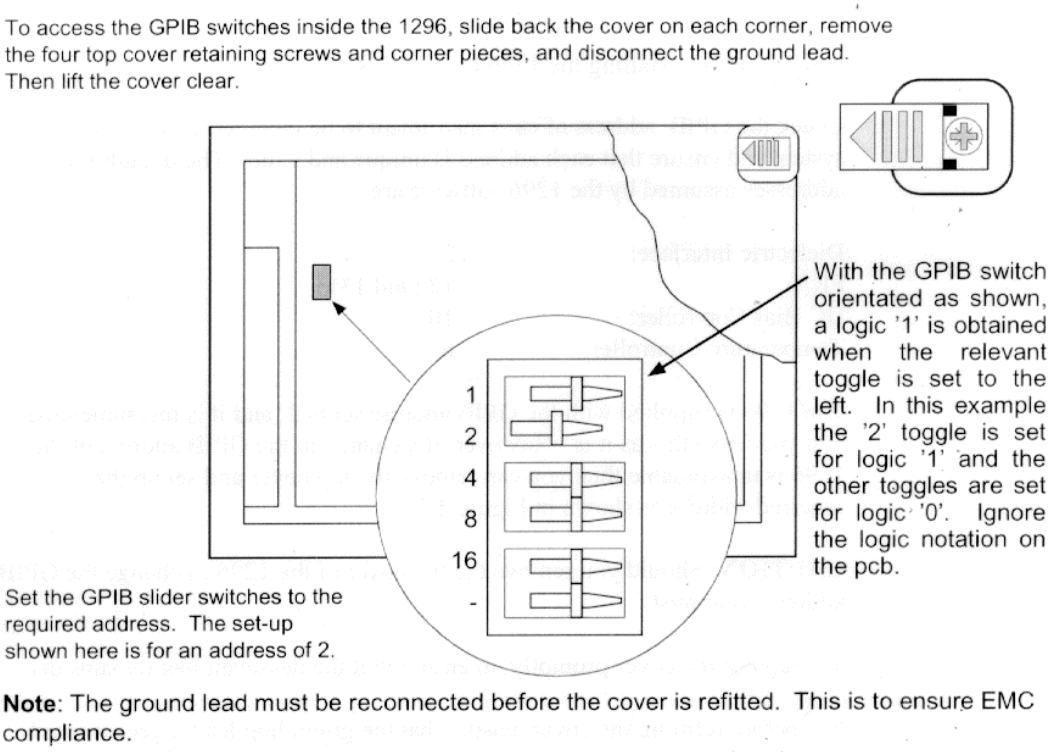



***Figure 2-1 Setting up the GPIB address switches in the 1296.***

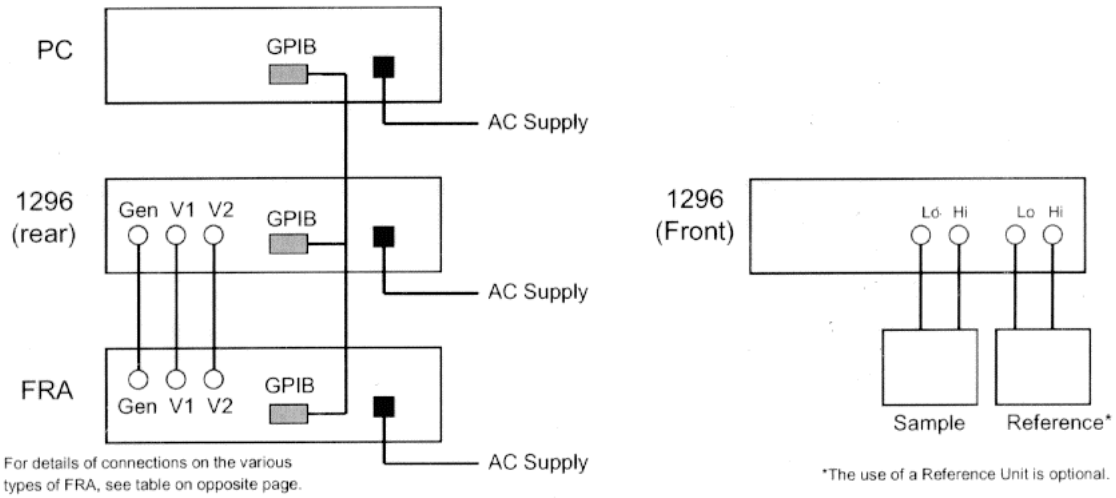



***Figure 2-2 Connecting the 1296 system for normal operation.***

### 2.1.1 HIGH VOLTAGE MEASUREMENTS

Methods are available for testing samples at higher voltages than those offered by the FRA generator output alone. There are two types of application:

a. Testing with a high ac voltage,

b. Testing with a low ac voltage, but with a high dc bias

Both types of testing benefit from the use of the external reference mode, but neither can be used with the internal reference mode.

**CAUTION:** The 1296 may be damaged if the test sample breaks down during high voltage testing. Therefore, use a protection resistor to limit the fault current to ±20mA. Correctly sited, this resistor should not affect the measurement.

**WARNING:** Where high voltage amplifiers or supplies are used, the appropriate safety precautions must be taken. Consult the manufacturers operating instructions.

#### 2.1.1.1 Using a High Voltage AC Source

High ac voltage testing can be done using the circuit shown in Figure 2-3.

When an experiment is run the ac voltage applied to the sample is the programmed value times the amplifier gain. However, the amplifier gain is not taken into account by the software. This has the following effect:

- Impedance result = actual impedance ÷ amplifier gain.
- Capacitance result = actual capacitance × amplifier gain.

For example, a 100pF capacitor, measured using an amplifier gain of ×100, appears to be a 100nF capacitor.

The voltage and frequency ranges covered depend on the performance of the amplifier and attenuator. Results obtained in the Normal measurement mode will have additional errors at high frequencies due to the attenuator characteristics. These errors can be minimised by using the Reference mode, with an external reference capacitor.

**NOTE:** The reference capacitance should be entered as it appears to the software. In the example above, a 100pF reference capacitance would be entered as 100nF.

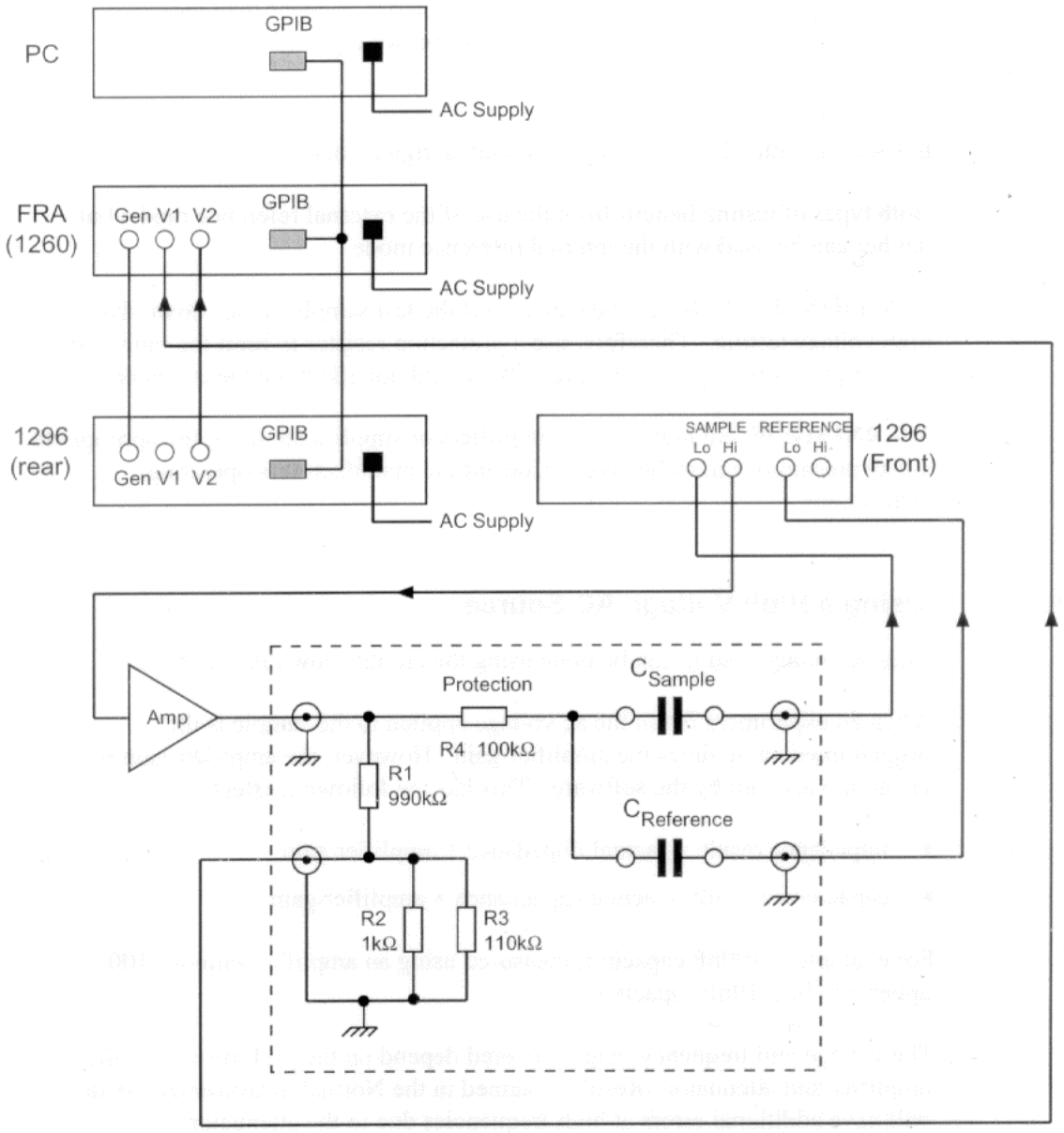



***Figure 2-3 Test circuit for a high voltage ac source.***

### 2.1.1.2 Using a High Voltage DC Bias

Additional dc bias (up to 1kV, approximately) can be applied to the sample from an external power supply. There are two methods of doing this:

a. Use a floating power supply, connected as shown in Figure 2-4.

b. Use a grounded bias circuit, with the ac source capacitively coupled as shown in Figure 2-5.

Method a. is the preferred method, but it is limited by the effects of floating-common to ground capacitance. Typically, this capacitance is in the region of 500nF, which reduces the ac test voltage to about 70% of its low frequency value at 6kHz.

Method b. is useful for higher frequencies, but the results at the lower frequencies should be treated with caution. This is due to the following:

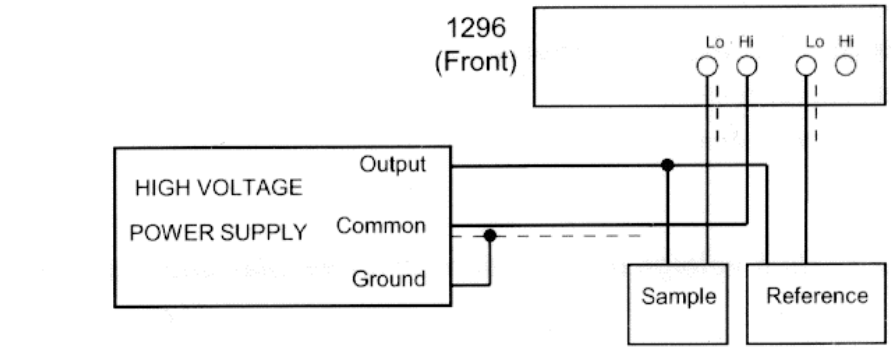
1. The test voltage falls off at low frequencies, due to the loading effect of R2 with C2. Measurement results are not affected until the test voltage becomes very small. The low frequency performance can be improved by increasing R2, but, if any dc bias current is present, the bias voltage is reduced.

2. The time constant of C3 and the 1MΩ input impedance of the FRA combined must be sufficiently large to minimise measurement errors at low frequencies. The accuracy of tan delta, in particular, is badly affected by phase shift. For this reason it is much better to use the external reference method to cancel these errors out.

**NOTES**

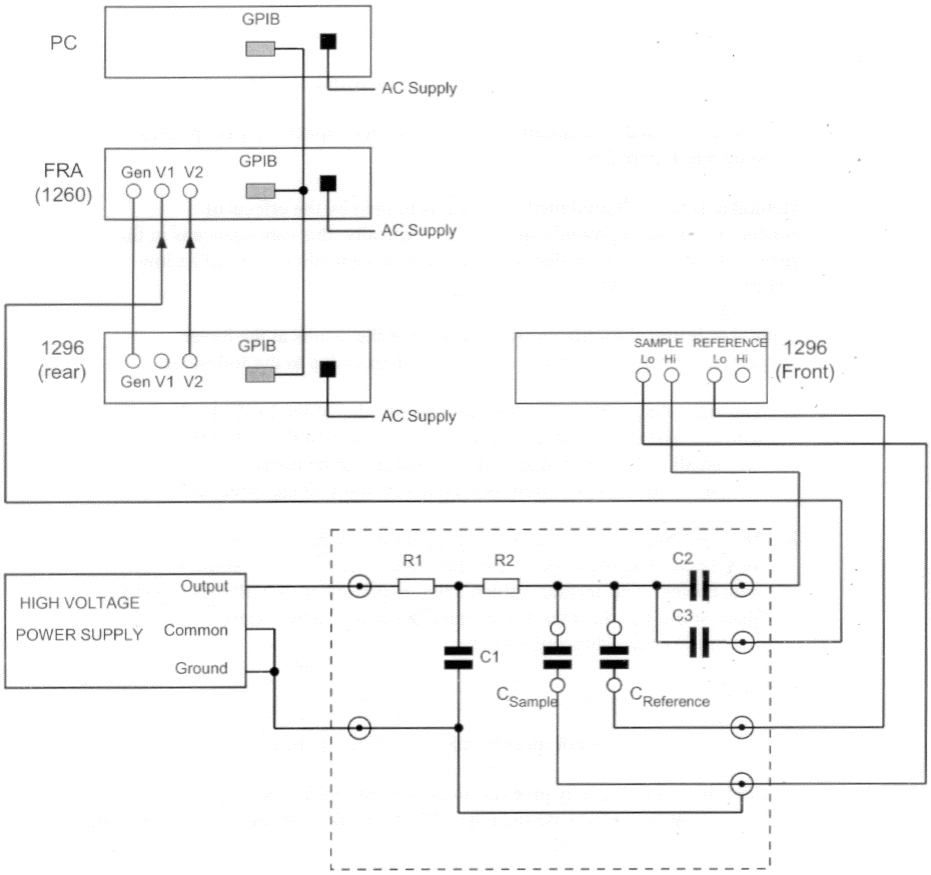
1. The reference capacitor must be rated to withstand the test/bias voltage.

2. High bias voltages may give rise to dc bias currents larger than the ac current. If this is the case then the 1296 is unable to range correctly and the FRA indicates overload.




The other connections are the same as those shown in Figure 2.2.

***Figure 2-4 High voltage dc bias, using a floating power supply.***




| | | |
|---|---|---|
| R1 | 10MΩ | Optional filter |
| C1 | 1nF, 4kΩ | |
| R2 | 10MΩ, 100MΩ, or 1GΩ, whichever gives the best results | |
| C2 | 10nF, 1.5kV polypropylene | |
| C3 | 100nF, 1.5kV polypropylene | |

***Figure 2-5 High voltage dc bias, with grounded bias and capacitively coupled ac.***

## 2.2 SOFTWARE INSTALLATION

We supply all the software you need to run the 1296 with SMaRT, on a single CD. To install the software:

1. On your PC, close down all tasks.

2. Insert the CD into the relevant drive on your PC.

3. If the CD does not run automatically, select ***Start***, ***Run...*** and enter the location of your CD ROM, e.g. ***d:\setup.exe***

Follow the on-screen instructions.

## 2.3 DEFINING THE HARDWARE SETTINGS

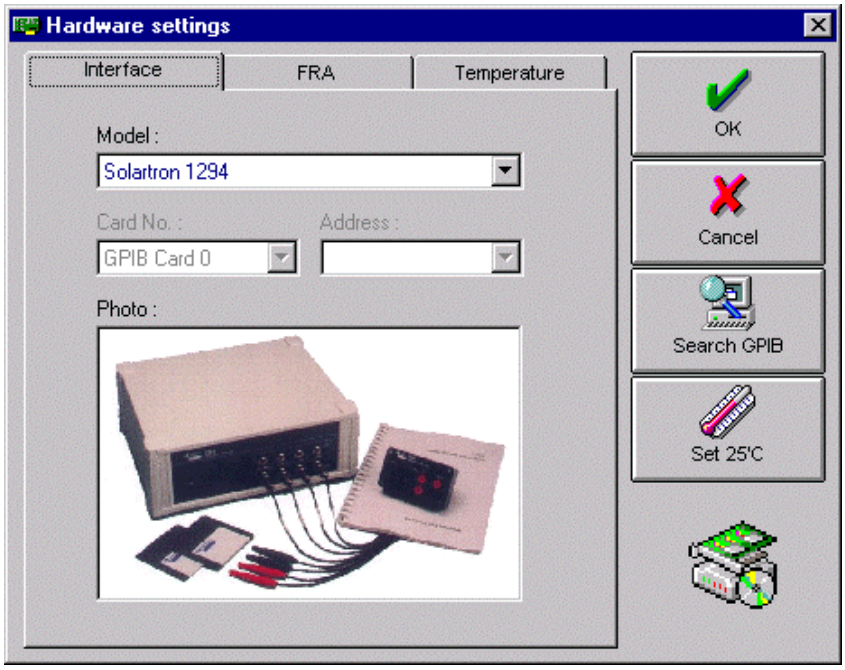
For your PC to be able to control the 1296 system, it must know the hardware settings. These are listed below. The default GPIB addresses of the 1296 Dielectric Interface and the FRA agree with those set on the hardware, as supplied. You can if you wish select different addresses from these, but all GPIB addresses entered must agree with the addresses that you set up on the instrument hardware.

You enter the hardware settings from the ***Hardware settings*** window of SMaRT. You can access this window either by clicking on the  icon in the toolbar or by selecting 'Hardware settings' in the ***Tools*** menu. The window contains four tabs, which relate to:

- The Dielectric Interface
- The FRA
- Temperature

These aspects of the hardware settings are described in Sections 2.3.1 through 2.3.4 below.

### 2.3.1 DIELECTRIC INTERFACE

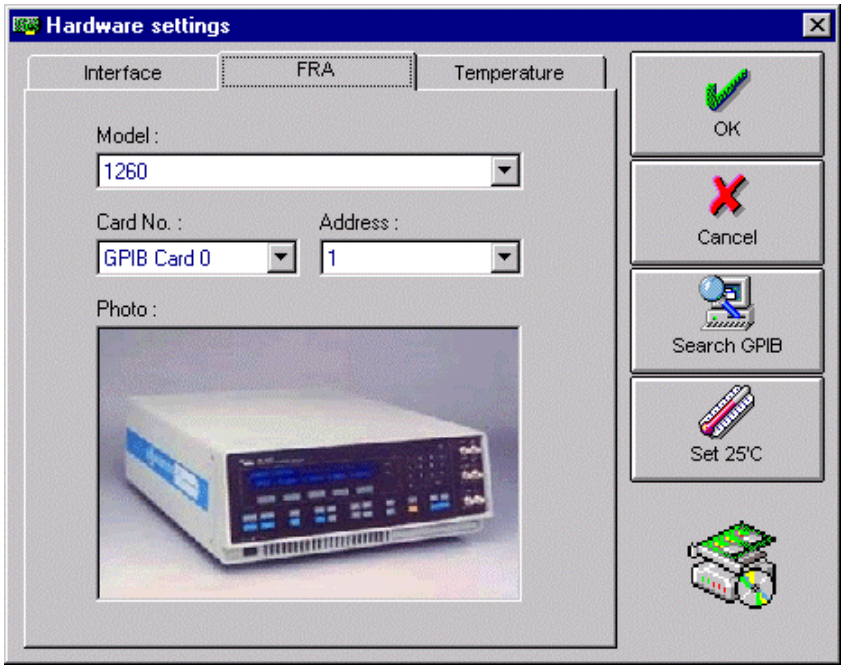



Model: 1296, 1294 or none (1260 stand alone mode)

Card No. Identifies the system controller GPIB card in the PC

Address: GPIB address of the Dielectric Interface (1 through 30)

## 2.3.2 FREQUENCY RESPONSE ANALYSER (FRA)



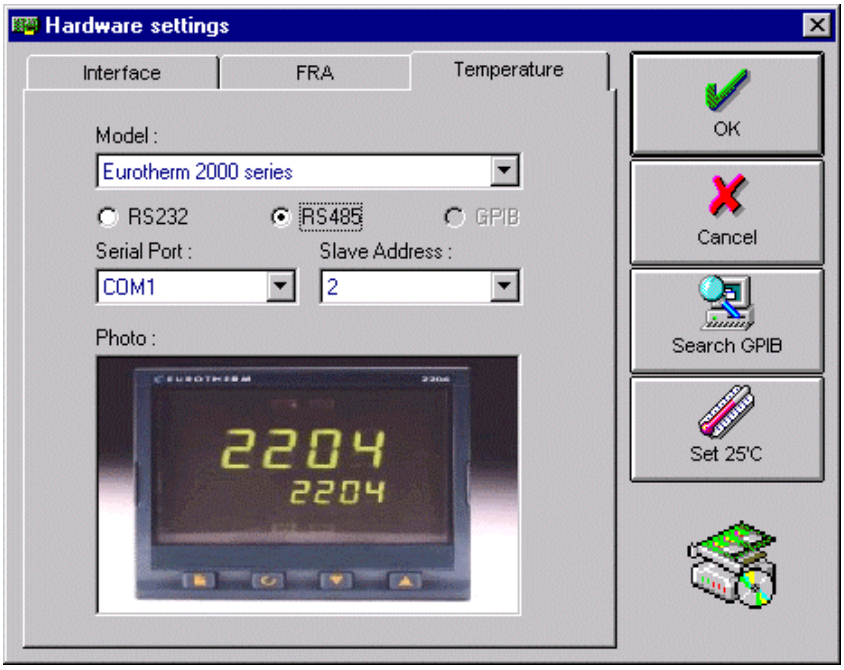

Model: Solartron 1260, 1250, 1253, or 1255

Card No. Identifies the system controller GPIB card in the PC

Address: GPIB address of the FRA (1 through 30)

**Note:** The FRA uses two addresses the even one shown in the Address box and the address that follows it. A warning is displayed if you attempt to enter the second FRA address for any of the other instruments in your 1296 system.

### 2.3.3 TEMPERATURE CONTROLLER




Model: 'None' –an instrument controlled from outside the system, as selected above– 'Lakeshore 340', 'Oxford Instruments ITC503', or 'Eurotherm 2000 Series'.

If you choose to adjust the temperature manually, the 1296 program prompts you to do this whilst the experiment is running. If no temperature control is required then select 'None'.

**Note:** The temperature controllers Lakeshore 340 and Oxford Instruments ITC503 are set up, prior to being operated, from their respective set-up files. These files, which are supplied with the impedance measurement software, are Lakeshore.set, oi_setup.set and eurotherm.set. Guidance on modifying the content of these files for your particular application is given in Section 6 of this user guide.

Address: For a Eurotherm 2000 Series Temperature Controller, you must enter either an RS232 or an RS485 serial port address. For Lakeshore 340 or Oxford Instruments ITC503 you must enter a GPIB address.

## 2.4 INITIAL CHECKOUT

To satisfy yourself that the 1296 unit is working, use the following checkout procedure:

1. Enter the hardware settings, as described in Section 2.3.

2. Take a 12961 Dielectric Reference Module and connect the Test terminals to the Sample terminals on the 1296.

3. Create a new project, entitled 'Initial Checkout'. *[For procedure, see Section 4.2 in the SMaRT User Guide.]*

4. Create an experiment that specifies:

   a. The data file (.DAT), and a frequency sweep between 1Hz and 1MHz, log scale, 5 points per decade,

   b. Impedance measurements, with an integration time of 1 second

   *[For procedure, see Section 5.1 in the SMaRT User Guide.]*

   (A sample definition is not required for the initial checkout, because permittivity will not be displayed.)

5. Start the experiment and allow the experiment to complete. *[For procedure, see Section 5.1 in the SMaRT User Guide.]* The Graph Set-up window then opens automatically.

6. Under ***Trace Set-up***, select the 'Initial Checkout' data file.

7. Click the LMB on '***OK***'. After a short initialising delay, the graph shown in Figure 2-6 should appear.

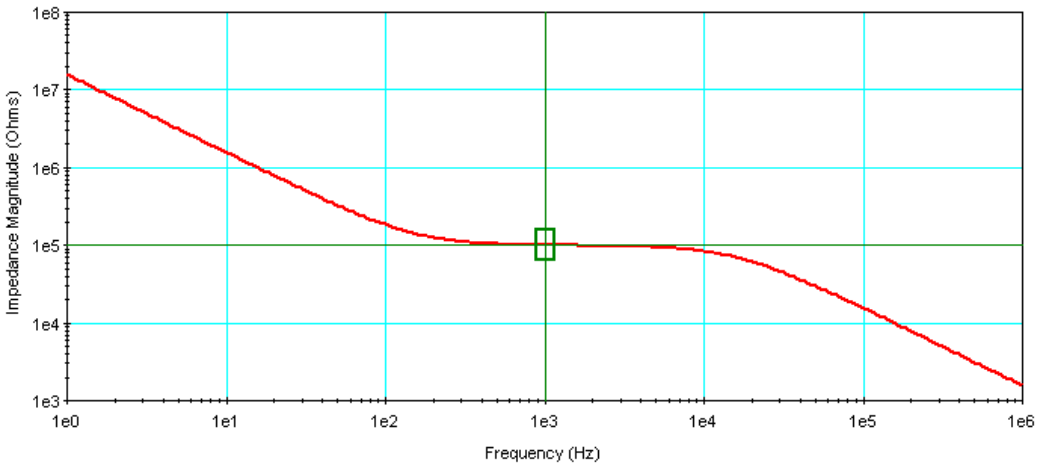



***Figure 2-6 Impedance plot of the 12961 test circuit.***

**8.** Click the RMB in the graph window and select '***Setup…***' This reopens the ***Graph Set-up*** window. Here, select '***Impedance Phase Degrees***' for the y-axis type and '***OK***'. After a short delay, the graph shown in Figure 2.7 should appear.

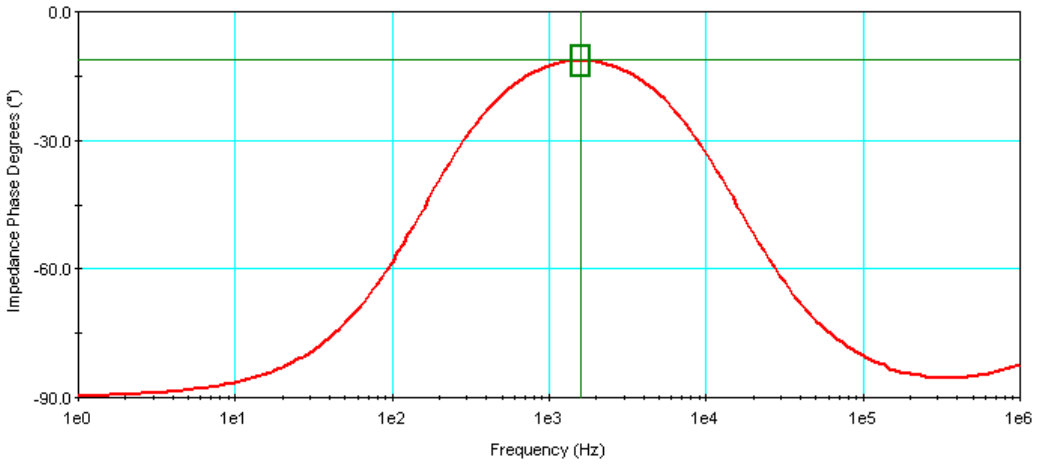



***Figure 2-7 Impedance phase plot of the 12961 test circuit***

**9.** Repeat step 8 as required, to select and plot various other characteristics of the test circuit. Figures 2.8 and 2.9 show plots of capacitance and capacitance tan delta against frequency.

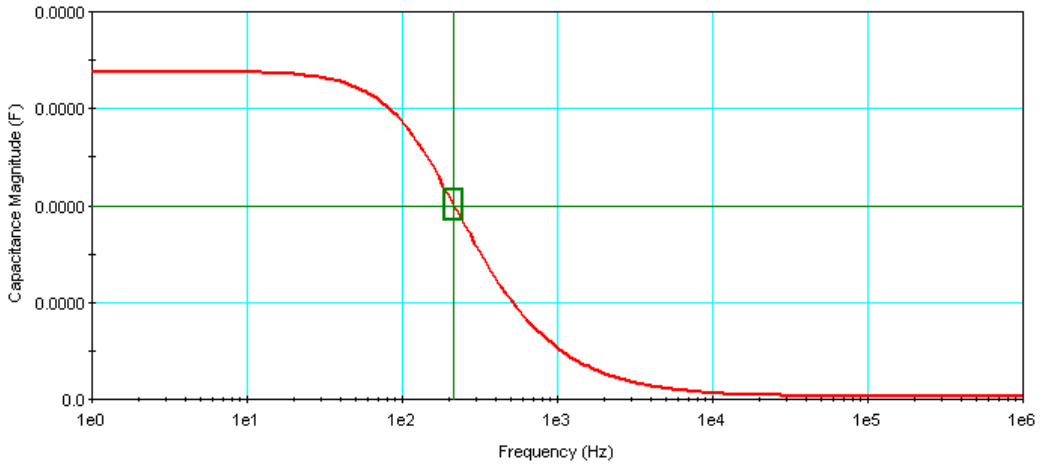



***Figure 2-8 Capacitance plot of the 12961 test circuit.***

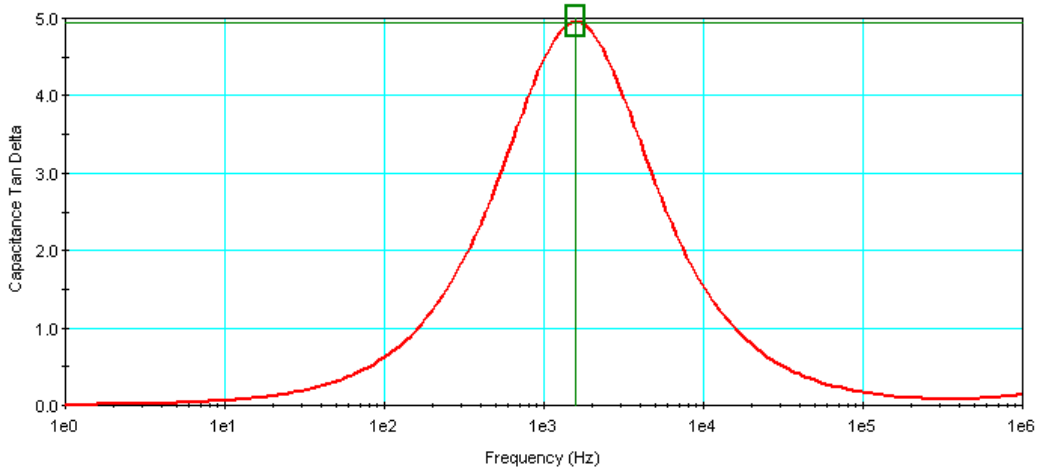



***Figure 2-9 Capacitance tan delta plot of the 12961 test circuit.***

# 3. 1296 Specification

Characteristics dependent on type of FRA used:

| 1296+FRA Characteristic | Type of FRA used with 1296 | | |
|---|---|---|---|
| | 1260 or 1255 | 1250 | 1253 |
| Frequency range | 10µHz to 10MHz | 10µHz to 65kHz | 10mHz to 20kHz |
| Signal amplitude | Up to 7Vrms* | Up to 10Vrms | Up to 10Vrms |
| DC bias | Up to 40V | Up to 10V | Up to 10V |

* For signals >3Vrms an internal amplifier is used: signal amplitude + dc bias must not exceed 10Vpeak.

Current Measurement: 1fA to 100mA.

Tan Delta Range: <10-4 to 103 (10-2 to 103 at >1MHz) in ref. mode.

Impedance Range: 100Ω to >100TΩ (1014Ω).

Capacitance Range: 1pF to >0.1F.

Software: Provides control of FRA, 1296, optional temperature controller, dc bias and provision for ac signal amplifiers.

Result Parameters: Admittance (Y*), Impedance (Z*), Complex Capacitance (C*), Permittivity (e*), Tan Delta (e"/e') plotted v. frequency, time, temperature, bias, ac level.

on Bode, complex plane.

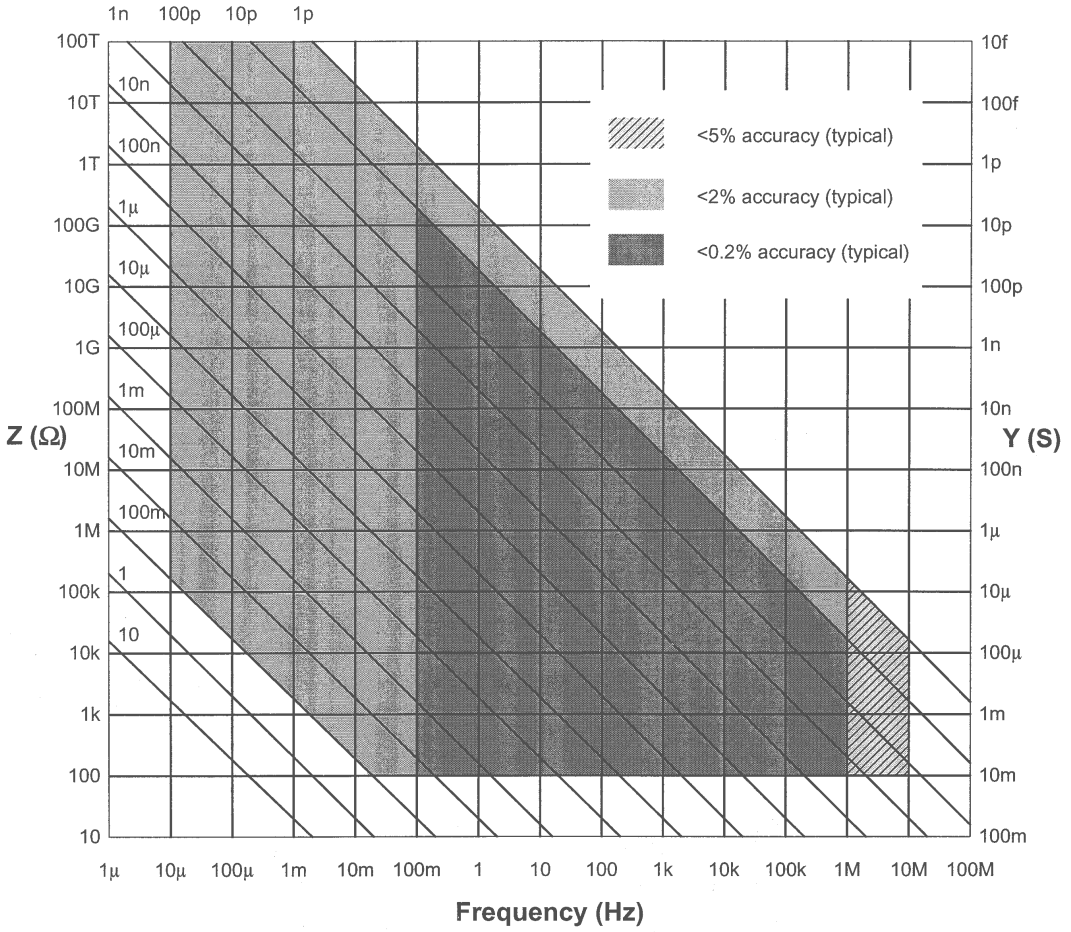
Power Supply: 85Vac - 264Vac (47Hz - 440Hz).
Fuse T1.25A.

Power Consumption: 30VA maximum.

Dimensions: 340mm (13.39in.) wide × 120mm (4.72in.) high × 300mm (11.81in.) deep.

Weight: 5.5kg (12.13lb)

Operating Temperature: 10°C to 30°C (50°F to 86°F).




**Accuracy of real capacitance values (typical), measured in the reference mode**

The measurement accuracy of the 1296 may be degraded should the instrument be subjected to high levels of radiated or conducted radio-frequency interference as defined in EN50082-1.

# 4. 1296 Sample Holders

## 4.1 INTRODUCTION

Three types of sample holders are available for use with the 1296 Dielectric Interface:

- **The 12962A sample holder** is designed for room temperature testing of solid samples, and has 20mm diameter electrodes. This consists of two parallel electrodes, one fixed and the other that can be moved into contact with the sample by adjusting the built-in micrometer.

- **The 12963A electrode kit** contains a set of additional fixed electrodes for testing solid samples of various sizes. The electrode diameters are 10mm, 30mm and 40mm. The kit must be used with the 12962A sample holder.

- **The 12964A liquid sample electrode kit** has electrodes for use with the 12962A for testing liquid samples.

Sample thickness can range from 0.2 to 25.4mm, and the impedance measurement range is 1Ω to >100TΩ. All 1296 sample holders utilise guard ring techniques to reduce the effect of stray fields at the edge of the sample (See section 4.2.3.) If you wish to improve the accuracy of your measurements, you can use measurement normalisation. (See section 4.4)

For information about sample holders for high and low temperature applications using cryostats and furnaces, please contact Solartron.

**Warning**

**Do not attempt to disassemble the fixed electrodes for solid samples. Any attempt to do so will adversely affect measurement accuracy, and will render the warranty invalid.**

## 4.2 12962A SAMPLE HOLDER

The 12962A sample holder is designed to allow accurate tests of high impedance solid materials at room temperature. The sample holder consists of two parallel electrodes, one of which is fixed in position (the 'Lo' electrode) and the other which can be moved into contact with the sample by adjustment of a micrometer (the 'Hi' electrode). The size of the active region of the Lo electrode for the standard sample holder is 20mm diameter. The 12963A electrode kit is available for testing materials of different sizes.

### 4.2.1 SAMPLE GEOMETRY

The 1296 software requires knowledge of the geometry of the sample. The thickness of the sample can be read from a micrometer digital display that is included as part of the sample holder. The area of the sample is defined by the area of the central Lo electrode. These dimensions are used in the 1296 PC software to calculate the permittivity of the material (which is independent of the size of the sample), using the formula:

$$\varepsilon = C.d/A$$

where: $\varepsilon$ = permittivity (F/m)
$C$ = capacitance (F)
$d$ = thickness (m)
$A$ = area ($m^2$)

This allows results from materials of different sizes to be compared.

Results can be displayed using the 1296 software as absolute or relative permittivity. Relative permittivity of the material, also known as the "dielectric constant" is calculated as:

$$\varepsilon\rho = \varepsilon / \varepsilon 0$$

where $\varepsilon_r$ = relative permittivity
$\varepsilon$ = permittivity of sample
$\varepsilon_o$ = permittivity of free space (8.85419E-12 F/m)

### 4.2.2 USING THE MICROMETER

For accurate measurements of the sample thickness, the micrometer should first be zeroed. This is done by removing the sample and moving the electrodes into contact with each other using the micrometer adjustment thimble. The micrometer is then zeroed by pressing the 'Zero/Abs' button. *(Refer to instructions supplied with the micrometer for details).*

The micrometer display can be configured for display of thickness in mm or inches as required.

### 4.2.3 GUARD RING

The 12962A makes use of a guard ring on the fixed electrode in order to reduce the effect of stray field lines at the edge of the sample that would otherwise lead to measurement errors. (See Figure 4-1.) The guard ring ensures that the electric field lines are parallel throughout the part of the sample that contributes to the impedance measurement.

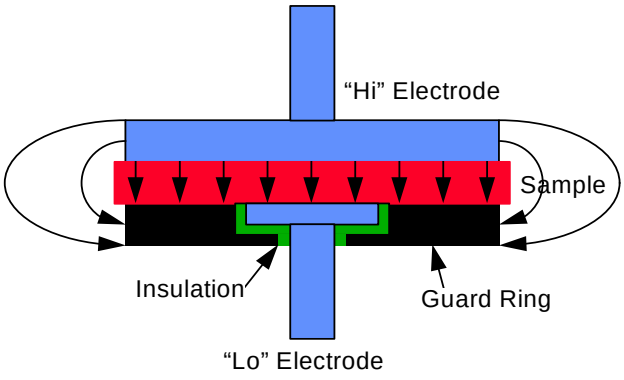



***Figure 4-1: Electrode arrangement of the 1296 2A sample holder***

The guard ring is connected to ground on the sample holder, while the Lo electrode is virtually the same ground potential because the current to voltage converter in the 1296 is a 'virtual earth' device. There is no potential difference between the Lo electrode and the guard ring and therefore the field lines at the edge of the "Lo" electrode are maintained parallel to each other. The impedance of the sample is calculated from the voltage drop across the sample and the current flowing through the central part alone of the sample where the field lines are parallel. The current flowing through the edge of the sample and the air surrounding the sample is not measured – it goes directly to earth and therefore does not contribute to the measurement.

### 4.2.4 CHANGING THE FIXED ('LO') ELECTRODE

1. Using the micrometer, move the travelling electrode as far away as possible from the fixed electrode, to give access to the fixed electrode.

2. Carefully unscrew the fixed electrode assembly (central electrode, guard ring and insulation) from the body of the sample holder, using finger pressure only. Store the electrode safely.

3. Screw the new electrode assembly into the body of the sample holder, taking care to keep the assembly properly aligned with the sample holder to avoid crossing the threads. Tighten the electrode gently against the body, using finger pressure only. DO NOT OVER TIGHTEN.

**Do not attempt to disassemble the electrode assembly.**

## 4.3 1296 4A LIQUID SAMPLE HOLDER

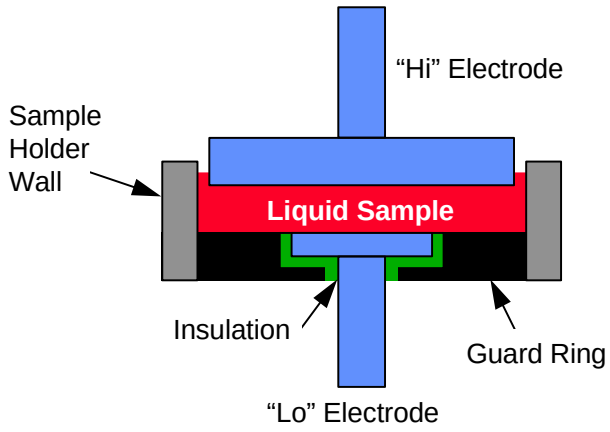



***Figure 4-2: Electrode arrangement of the 1296 4A liquid sample holder***

The 12964A electrodes replace the solid sample holder electrodes in the 12962A. The liquid sample holder is shaped as a dish in order to be able to measure small samples of liquid. Unlike the fixed electrode for solid samples, it is permissible to disassemble this electrode for cleaning. It makes use of the guard ring techniques described above.

### 4.3.1 USING THE 12964A

The upper electrode should be dipped into the liquid to make sure that air is removed from the sample being measured.

To create a particular sample thickness, follow this procedure:

1. Zero the micrometer, as described in section 4.2.2.

2. Using the micrometer, adjust the position of the upper electrode sufficiently, then place the sample liquid into the lower dish electrode.

3. Move the upper electrode carefully towards the lower electrode until the required sample thickness is displayed. The upper electrode is slightly smaller than the lower dish electrode so that excess liquid can escape around the side of the upper electrode. **Do not go past the required thickness, otherwise too much liquid will escape, and it will not be in contact with the upper electrode.**

## 4.4 NORMALISATION

For optimum accuracy when using liquid or solid sample holders, you should make use of the normalisation technique. This will reduce the errors due to fringing, connections, etc.

There are two possible methods of normalisation: matched cell geometry and matched cell capacitance. Both have advantages. Matched capacitance gives best cancellation of instrumentation errors, whereas matching the cell geometry optimises the cancellation of fringing effects. Select the one that gives you the best results.

### 4.4.1 METHOD 1 - MATCHED GEOMETRY

1. Take measurements on the sample in the way described above, making a note of the micrometer setting.

2. Remove the sample, clean the electrodes if necessary, and reset the micrometer to the setting noted above. This will create an air gap cell with the same dimensions as the cell used for the sample.

3. Measure the impedance of this air gap cell at the same frequencies as before, and use this data as the normalisation data.

### 4.4.2 METHOD 2 - MATCHED CAPACITANCE

1. Take measurements on the sample in the way described above and note the capacitance of the sample.

2. Remove the sample and clean the electrodes if necessary. Calculate the air gap, d (in m.), required to create an air gap cell with the same capacitance as the sample, using the equation:
   $d = A\varepsilon_0/C$
   where A is the area of the cell (in $m^2$), C is the required capacitance (in F), and $\varepsilon_0$ is the permittivity of free space (8.85419E-12 F/m)

3. Set the micrometer to the calculated air gap, measure the impedance of this air gap cell at the same frequencies as those used for the sample measurements, and use this data for normalisation.

# 5. Measurement Term Definitions

The following definitions relate to the various quantities calculated by the impedance measurement software.

## 5.1 ADMITTANCE

Admittance ($Y^* = I^*/V^* = G+jB$) is displayed in Bode or Nyquist plots as conductance G (real) and susceptance B (imaginary). This is the basic quantity calculated as each measurement is made. Admittance is the reciprocal of impedance. It can be represented by a parallel combination of resistive and reactive elements and is expressed in siemens (S). Admittance is also displayed on Bode plots, using the polar parameters of magnitude and phase.

## 5.2 IMPEDANCE

Impedance ($Z^* = V^*/I^* = R+jX$) is displayed as resistance R (real) and reactance X (imaginary). Impedance can be represented by a series combination of resistive and reactive elements and is expressed in ohms (Ω). Each result is held in the 1296 data output file in this form.

## 5.3 COMPLEX CAPACITANCE

The concept of complex capacitance is used in dielectric studies. Considering the admittance of a complex capacitor to be equivalent to the simple combination of R and C in parallel, gives:

$j\omega C^* = G+j\omega C$

$\backslash C^* = G/j\omega+C$

$= C- jG/\omega$

Note that the imaginary part is negative. The complex capacitance is defined as $C^* = C' - jC''$ so that, in this simple example, $C' = C$ and $C'' = G/\omega$, both positive.

## 5.4 PERMITTIVITY

The expression for permittivity can be derived as follows:

C = e.A/d where e = permittivity,

A = electrode area (m2)

d = thickness of dielectric (m)

also e = eo.er

where eo = permittivity of free space = 8.85419e-12 F/m

er = relative permittivity of dielectric material

From this e = C.d/A or, in complex form, e* = C*.d/A = (C'- jC")d/A = e'- je"

It then follows that er* = e*/eo = er'- jer" ; er' = e'/eo ; er" = e"/eo

## 5.5 MODULUS

Some researchers favour the use of dielectric modulus M* = 1/e*

## 5.6 TAN DELTA

The phase angle between voltage and current in a capacitor is nominally 90°. A (normally) small deviation d from 90° is caused by the power loss in the capacitor. The loss factor or tan delta of the capacitor or dielectric material is the ratio of this power, VIsind, to the purely reactive component VIcosd. It can be calculated from:

tan delta = C"/C' or e"/e'

This should be a positive value.

# 6. Temperature Controller Set-up Files

## 6.1 INTRODUCTION

The set-up files for the Lakeshore 340 and Oxford Instruments ITC503 temperature controllers, supplied with the Impedance Measurement software, are intended as examples. Although these files, as supplied, are fully workable, the object is that users should edit the files to suit their particular needs. An explanation of the commands and parameters for the Lakeshore340 and Oxford Instruments ITC503 can be found in the manuals provided with those instruments.

Sections 2 and 3 of this appendix give typical examples of the temperature controller set-up files and general advice on how to use them.

## 6.2 LAKESHORE 340 SET-UP FILE

A Cryostat has two temperature sensors, "Heater" and "Sample", whilst the Lakeshore 340 has two sensor inputs "A" and "B". (See Figure D.1.) The normal method of using the cryostat is to connect input "A" to "Heater" and input "B" to "Sample", and to control the cryostat heater from "A".

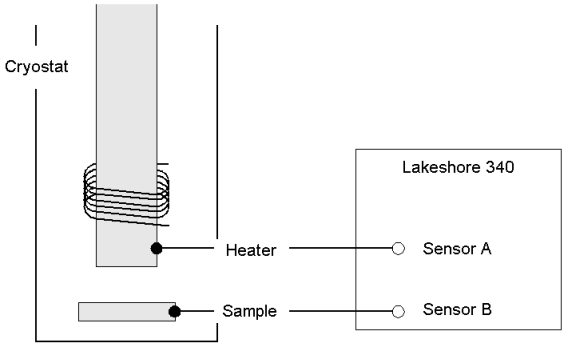



***Figure 6-1 Cryostat temperature sensing with Lakeshore 340.***

Since "B" lags behind "A", a settle period is configured to allow the temperature to stabilise at a temperature nearer to "A". The alternative allowed by the software is to control the cryostat with the sample temperature sensed at "B". Controlling the cryostat from the "B" input is a more accurate way of controlling the sample temperature, but it depends on there being a good thermal link between "A" and "B" in the cryostat. A poor thermal link can result in control loop instability and thermal runaway. You will need to decide which method is the best for your particular experiment. (If you control the cryostat temperature from the sensor "B" input, the sensor "A" input should be disabled.)

After transmitting the SETUP file to the controller, the DLL takes the following actions.

1. Sends CSET? to determine the control input. It assumes for now that the other input is on the sample.

2. Sends INSET? <Control Input> to verify that the control input is enabled.

3. Sends RDGST? <Control Input> to verify that the control Input is configured.

4. Sends INSET? <Sample Input> to verify that the sample input is enabled. If the sample input is not enabled, the control temperature is returned as the sample temperature.

5. If the sample input is enabled, sends RDGST? <Sample Input> to verify that the sample input is configured. If this is not so, the control temperature is used as the sample temperature. The sample temperature and the set point are both stored in the results file.

Below is a typical example of a Lakeshore set-up file. In this particular example, sense input "A" of the Lakeshore 340 is enabled and sense input "B" is disabled.

```
! Lines Beginning with ! are ignored
! All Commands are Converted to Upper Case before being
! Sent to the Temperature Controller.
!
! MAXIMUM PERMITTED LINE LENGTH IS 255 Chars
!
! The Only Query Allowed is BUSY?, which will wait
! For the response from the Controller to be 0
!
!
! Turn Off The Heater
RANGE 0
!
! Turn Off Ramping
RAMP 1,0,0
!
! Set Platinum 100(500Ohm) on Input A
INTYPE A,4
BUSY?
!
! Select Curve 4 for Input A
INCRV A,4
!
! Set Input A to be Enabled, B to be Disabled
INSET A,1,0
INSET B,0,0
!
!Setup The Display
DISPLAY 1,60
DISPFLD 1,A,1
!
! Set Limits dependant of Cryostat
CLIMIT 1,500,0,0,4,5
!
! Configure Control Loop 1,InputA, Units Kelvin, ON, Enable at power up
CSET 1,A,1,1,1
!
! Set Stability Condition = Within 1.5 Degrees for 30 Seconds
SETTLE 1.5,30
!
! Wait for not Busy
BUSY?
!
! Allow heater to go to Maximum range
RANGE 5
! End of Setup File
```

## 6.3 OXFORD INSTRUMENTS ITC503 SET-UP FILE

This section describes the set-up file that is used to initialise the Oxford Instruments ITC503 Temperature Controller.

### 6.3.1 FILE FORMAT

In the ITC503 set-up file there are lines of three types:

1. Lines beginning with ! are comments and are ignored.
2. Lines beginning with % define some driver parameters, as described below.
3. Other lines (including blank ones) are sent to the ITC503 as commands.

See the ITC503 manual for full details of the commands available.

Lines may contain up to 255 characters.

### 6.3.2 TEMPERATURE STABILISATION

The driver internally decides when the sample temperature has reached stability, before any impedance measurements are made. The stability test is very flexible and can be configured using the driver parameters listed in Section 3.3. The driver keeps an internal history of measured temperatures and uses these to decide when the sample temperature is stable, as follows:

1. The driver waits until the control channel temperature is within a small span of the requested temperature. The span is defined by %TStabTolerance. This test is optional and is done only if %TStabControlTarget is non-zero.
2. Similarly, the driver waits until the sample channel temperature is within a small span of the requested temperature. The span is defined by %TStabTolerance. This test is optional and is done only if %TStabSampleTarget is non-zero.
3. A moving standard deviation of the measured sample temperatures is calculated and the driver waits until this value is less than %TStabSD kelvin before deeming the temperature to be stable. The standard deviation is calculated over a time window of the most recent %TStabPeriod milliseconds of temperature readings.

### 6.3.3 DRIVER PARAMETERS

The driver parameters for the ITC503 are:

%ControlChannel = n This defines the channel (1-3) whose temperature is to be controlled by the temperature controller.

%SampleChannel = 2 This defines the channel (1-3) whose temperature is to be interpreted as the sample temperature. It can be the same as the control channel.

### 6.3.4 TYPICAL ITC503 SET-UP FILE

```
%ControlChannel = 1          ! Control temperature channel is 1.
%SampleChannel = 1           ! Sample temperature channel is 1.
%TStabPeriod = 10000         ! 10s stabilisation period.
%TStabSD = 0.1               ! Temperature SD to be less than 0.1 K.
%TStabControlTarget = 1      ! Wait for the control temperature to reach target.
%TStabSampleTarget = 0       ! Allow the sample temperature to differ from the
                                 ! target.
%TStabTolerance = 0.1        ! Temperatures must be within 0.1K of target.
!
! Go into manual mode:
A0
!
! Turn the heater off:
O0
!
! Turn the gas flow off:
G0
!
! Use the following command to set the maximum heater output (in volts) if
! required. Note that 0 has a special significance - see the ITC manual:
!
M20
!
! Set the PID controller terms if required:
!
P25
!
I0.5
!
D0
```

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