

ModuLab XM PhotoEchem

USER MANUAL

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DECLARATION OF CONFORMITY

The directives covered by this declaration

CE

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)

ModuLab System (configured as DSSC*)

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:

BS EN61326:2013	Electrical equipment for measurement control and laboratory use -EMC requirements; including amendments A1:1998 and A2:2001.
BS EN61010-1:2010	Safety requirements for electrical equipment for measurement, control and laboratory use.

Accordingly the CE mark has been applied to this product.

Signed

For and on behalf of Solartron Analytical

Authority: Business Element Manager

Date: March 2015

* Certification does not apply to IPCE module

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. 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.

The equipment is intended solely for electronic measurement and should be used for no other purpose. The equipment protection may be impaired if it is not operated in a manner as specified by Solartron and detailed in this document. Solartron accept no responsibility for accidents or damage resulting from any failure to comply with these precautions.

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. System to be used with an IEC 60799 compliant mains supply cord, rated for the correct voltage and power rating as detailed on the rear panel.

FUSES

Before switching on the equipment check that the fuses accessible from the exterior of the equipment are of the correct rating. The rating of the AC line fuse must be in accordance with the voltage of the AC supply.

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. The replacement fuses are

T10A H250V (100-120V mains supply)

T5A H250V (220V-240V mains supply)

EXPLOSIVE ATMOSPHERES

You must NEVER OPERATE the equipment, or any sensors connected to the equipment, in a potentially explosive atmosphere. The equipment is NOT designed for use in these conditions and could possibly cause an explosion.

ACCESSORIES AND ANCILLARY EQUIPMENT

Only use accessories and ancillary equipment either delivered as part of the DSSC system or pre-approved for use with the DSSC system directly by Solartron. Use of unapproved accessories and ancillary equipment can cause damage to the equipment and potentially form an electrical hazard.

SAFETY SYMBOL

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

Symbol	*Meaning*
	General safety hazard. Refer to the operating manual for detailed instructions.
	Electrical safety hazard. This symbol may appear alongside the general safety hazard symbol, together with a voltage.

General Safety Precautions (*continued from previous page*)

Symbol	*Meaning*
	Safety hazard involving heavy weights. A numerical value for the amount of weight may appear alongside this symbol. Take care when handling and carrying. Follow the manufacturer's instructions, and ask for assistance if necessary.

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. Adjustments, maintenance or repair must only be done by qualified personnel, who should refer to the relevant maintenance documentation.

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 your Solartron service provider for service and repair.

DISPOSAL AND RECYCLING OF EQUIPMENT IN THE EUROPEAN UNION

Solartron is an environmentally friendly company, committed to the disposal and recycling of electrical and electronic waste according to procedures that have the least possible impact on health and the environment. The company supports the disposal of equipment according to the EU directive on Waste Electrical and Electronic Equipment (WEEE).



When you wish to dispose of any Solartron electrical and electronic equipment supplied in the EU, you should call your supplier and ask for advice about the disposal process. Do not dispose of any equipment in skips, rubbish bins or waste disposal sites.

MODULAB COMPONENTS, MATERIALS AND DISMANTLING

The following information will be of interest to parties who are involved in the disposal of equipment.

The ModuLab system consists of a metal chassis mounted in a polyurethane plastic case. The chassis contains a power supply and a number of PCBs including up to eight modules that can be removed and refitted. There is an LCD display mounted on the front of the metal chassis, behind the plastic front cover.

With the front cover removed, you can remove the chassis from its case as follows:

1. Undo three screws on each side of the case, at the bottom.
2. Slide the upper section of the case off to the rear, leaving behind the chassis and plastic base.
3. Lift the chassis off the plastic base.

The ModuLab system contains soldered components which may contain lead.

Contents

1. Introduction 1
2. Delivery Items 3
3. Hardware Installation 5
3.1 ModuLab Hardware 5
3.1.1 Configuring The PC Ethernet Interface 7
3.1.2 Configuring Ethernet for Windows 7 8
3.1.3 Installing the ModuLab software 10
3.1.4 Microsoft .Net Framework 11
3.2 Optical Bench and Interface Card 12
3.2.1 Interface Card 12
3.2.2 Optical Bench 12
3.2.3 Sample Holder 16
3.2.4 Mounting High Brightness LED onto Optical Bench 20
3.2.5 Optical Bench Connections for Testing Solar Cells 21
3.2.6 Optical Bench Calibration Connections 25
3.2.7 Manual Control of LED Driver 26
3.2.8 Care of Optical Bench 27
4. Software Installation 28
4.1 Installation of ModuLab XM PhotoEchem Software 28
4.2 Installation of LED driver software 28
4.2.1 Installation of DC2100 28
4.2.2 Installation of DC2200 Driver 28
5. ModuLab XM PhotoEchem User Interface 29
5.1 System Communication 29
5.1.1 Potentiostat Set-up 29
5.1.2 Photodetector Setup 34
5.1.3 Setting up LED driver 36
5.1.4 LED Calibration Routines 38
6. Creating Experiments 43
6.1 Getting Started 43
6.1.1 Auto vs. Advanced Experiments 44
6.1.2 AUTO 44
6.1.3 Advanced 45
6.2 Intensity ModuLab PhotoCurrent Spectroscopy 45
6.3 Intensity Modulated Photovoltage Spectroscopy 51
6.4 EIS 55

6.5 PhotoVoltage Decay 58
6.6 I-V Curves 61
6.7 Dark Charge Extraction 63
6.8 Short Circuit Charge Extraction 67
6.9 Other Techniques with LED control enabled 69
7. Analysis and Auto-Analysis 70
7.1 Analysis Tools 70
7.1.1 I-V analysis 70
7.1.2 EIS Analysis 72
7.2 Auto Analysis Tools 72
8. Low Light Intensity Studies Using an ND Filter 76
8.1 Calibration with ND Filter 76
8.2 Measurements with ND Filter 77
9. Miscellaneous 78
9.1 Cleaning of Optical Components. 78
9.2 Thermal Stability of the LED optics 78
10. System Specifications 79
10.1 Potentiotstat Card 79
10.2 Frequency Response Analyzer 80
10.3 Core Potentiostat Accuracy 81
10.4 FRA Accuracy 81
10.5 Chassis Specification 82
10.6 PC requirements 82
11. IPCE Module 84
11.1 TLS Module 84
11.1.1 Monochromator Theory of Operation 84
11.1.2 Setup and Operation of TLS Light Sources 84
11.1.3 Setup and Operation of Computer Controlled TLS Light Sources 86
11.1.4 Installing the TLS driver software 87
11.1.5 Bulb Replacement 87
11.2 Activation of License Code 87
11.3 Calibration of Optical Bench 88
11.3.1 Pre-Calibration checks 88
11.3.2 Calibration of Beam Splitter 91
11.4 Alignment of Samples 93
11.5 White Bias Light Source 95
11.5.1 Mounting White Bias Source 96
11.5.2 Adjusting White Bias Light Intensity 97

11.6 IPCE Experiments 100
11.7 Data Analysis 102
11.8 Specifications 104

1. Introduction

The ModuLab XM PhotoEchem is a fully integrated optical/electrical measurement system designed for the characterisation of photoelectrochemical systems such as DSSC's, Perovskite cells, solar water splitting applications and more. Based upon the core ModuLab ECS technology, the XM PhotoEchem system fully utilizes the powerful Solartron Frequency Response Analyzer technology for advanced frequency domain techniques that have been developed for this field of research.

The characterization of solar cells requires illumination by a light source and such sources must be stable, programmable in order to control intensity and of sufficient power to characterize the cell over a wide range of photovoltages. This system makes use of high brightness LED's which are optimised for power and excellent thermal stability.

A key feature of the system is the ability to auto-run and analyse data with little or no previous experience of measurements of DSSC's. The optical system contains a NIST traceable calibration routine that ensures measurements are repeatable and reproducible.

The optical system is fully compatible with all ModuLab ECS systems such that existing systems can be upgraded to include the software and hardware necessary for these measurements.

The following techniques are available with the system including;

- Polarization Curves
- IMPS (intensity modulated photovoltage spectroscopy)
- IMVS (intensity modulated photocurrent spectroscopy)
- EIS spectroscopy
- Charge Extraction Methods
- Photovoltage Decay
- IPCE

In addition to these techniques, the full suite of Electrochemical Techniques are available enabling comprehensive studies on a wide range of electrochemical systems.

This manual focuses on the features that are specific to the ModuLab XM PHOTOECHEM platform. It is advised that customer familiarise themselves in the use of the ModuLab software and therefore we recommend you consult the manual and system installation guide for a more detailed description of the ModuLab system.

The system has the ability to incorporate an IPCE module. The operation detail of this module are discussed at the end of this manual

2. Delivery Items

The system is configured for PhotoEchem experiments and includes the following ModuLab Hardware

- 4 or 8 slot Chassis
- Potentiostat Card with Auxiliary Card
- 300kHz or 1MHz FRA card
- PhotoEchem Card
- 3 off mains supply cords appropriate to local line voltage

The following cables associated with the measurement system are provided

Module / Channel	Part Number	Description
Pstat/HV main channels	20870301	RE cable from Pstat to cell
	20870323	CE / WE cable from Pstat to cell
Pstat/HV auxiliary channels	20870304	AUX A cable from Pstat to cell
	20870305	AUX B cable from Pstat to cell
	20870306	AUX C cable from Pstat to cell
	20870307	AUX D cable from Pstat to cell
FRA	20550301	Cable from any FRA socket to external monitoring device.

In addition to the ModuLab hardware, the system is delivered with the following items specifically for the optical bench;

- Optical Bench with Sample Holder
- Solaronix Reference Solar Cell
- BNC T-Piece
- Hexagon Key Set
- Thorlabs LED Driver with Power Supply and Software CD (DC2100 or DC2200 model)
- BNC LED connector cable
- BNC Photodetector cable (BNC cables are standard BNC plug – BNC plug cable, 50R impedance, 1m length)
- 1 x 590nm AMBER LED
- 1 x NIST Traceable Photodetector
- Spacing Bolts

The following LED's are available as options and can be ordered separately;

LED Options (nm)	Part Number
405	234253
420	234254
455	234255
470	234248
505	234256
530	234257
565	234259
590	System
617	234259
625	234261
Cold White	234332
Warm White	234331

An IPCE add-on module is available. The system is designed to work with the Optical Bench provided with the PhotoEchem system. The IPCE add – on module includes

- White light bias with power supply
- Monochromator
- White Light source for Monochromator
- Fibre Optic Cable
- Power Supply for Monochromator

3. Hardware Installation

3.1 ModuLab Hardware

The hardware modules must be installed in the following order as shown in Figure 1: ModuLab XM PhotoEchem Modules. The FREQUENCY RESPONSE ANALYZER must be placed in the first slot nearest the LCD display. The PhotoEchem card is placed next to the FRA in the second slot and the Potentiostat must be placed in the slot next to the PhotoEchem card. For details of the FRA and Potenstiostat cards, please refer to the ModuLab ECS manual section 2, which is supplied with this unit.



Figure 1: ModuLab XM PhotoEchem Modules. From Right to left on the screen, FRA, PhotoEchem Card and PStat.

ATTENTION ! Other ModuLab option cards may NOT be used in conjunction with the PhotoEchem card.

The ModuLab system consists of a metal chassis mounted in a plastic case and is designed as a desktop unit. To install the system, proceed as follows:

1. Choose a location with a firm solid surface, with easy access to the following:
   - A PC conforming to the system requirements. To get started, it is recommended that the Ethernet interface should be a spare socket not already connected to a network although a corporate network can be used if the configuration is correct.
   - An earthed mains power socket in the voltage range 100V to 120V AC and 220V to 240V AC and frequency range 50 to 60Hz).
2. Place the ModuLab system at your chosen location, making sure that both the front and rear panels are accessible and there are no obstructions that would prevent the circulation of air from the cooling fans at the rear.



Be careful when lifting and carrying the ModuLab system, as it is heavy and has no carrying handles. It should normally be carried by two people, gripping firmly under the footrests at the front and rear.

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3. The ModuLab is fitted with a power supply that automatically detects the mains voltage and there is no selector switch, but the correct fuses have to be fitted to the mains power input connector(Figure 2). The fuseholder (1) is on the rear panel between the mains input socket (2) and switch (3). There are two identical fuses, one on the live and one on the neutral side of the circuit. Pull out the fuse holder and check that the fuses are correct, and replace them if necessary.
4. The fuse ratings for the mains voltages are:

   220-240V mains supply - T5A H 250V (5A time lag, high breaking capacity, 250V rated)

   100-120V mains supply – T10A H 250V (10A time lag, high breaking capacity, 250V rated)

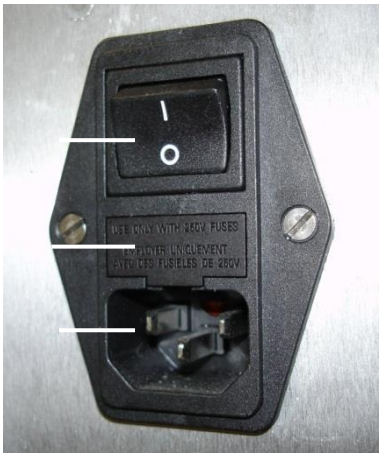


Figure 2: Mains Socket Rear Connector

5. Insert the mains power lead into the socket at the rear of the unit, shown in Figure 2: Mains Socket Rear Connector and connect to the mains supply.
6. Switch on the power at the mains, and at the switch above the power input socket. The power on status will be indicated on the front panel as follows:
   - The LEDs at the top of each module will light up.
   - The Solartron logo will appear on the display panel (Figure 3) This will remain permanently on display, as long as the system is switched on, until it comes under the control of the PC running the ModuLab software, then it is replaced by information about the system.



Figure 3: ModuLab Front Panel Display

7. The modules are identified by their front-panel labels and will have been installed by Solartron, according to your specification. If any of the modules need to be changed, switch off the system and unplug it from the mains, then proceed with the module installation.

3.1.1 Configuring The PC Ethernet Interface

To enable the PC to communicate reliably with the ModuLab system, the dedicated Ethernet card must be given a unique address that is outside the range of addresses used by the ModuLab. Windows XP, by default, uses automatic addressing on power-up, and in most circumstances this will enable the PC to recognise the ModuLab addresses and assign a different address to the dedicated Ethernet card. However, it is also possible to set the address manually.

The ModuLab system uses the default IP addresses given in the table below:

Device	IP Addresses	Subnet Mask
ModuLab System Display Panel	169.254.254.1	255.255.0.0 for all devices
ModuLab System Modules	169.254.254.*n+9* where *n* = Slot ID (1, 2, 3…). for example Slot 1 at the left of the chassis is: 169.254.254.10	

Each chassis has 4 or 8 slots, but the addresses ending in 10 to 119 should be reserved for the ModuLab in case they are needed for an expanded system. The dedicated Ethernet card can use any of the remaining addresses ending in 120 to 254 and the address can be set from the Windows Operating System.

NOTE: On a multi-user Operating System such as Windows 7, you have to be logged in as a user with sufficient privileges to set an IP address. If you cannot access all the features in this section, ask your system administrator to upgrade your user privileges or set the IP address for you.

The procedure for setting an IP address depends on the installed version of Windows, and the configuration of the Windows user interface. The following sub-sections describe typical procedures for Windows XP professional

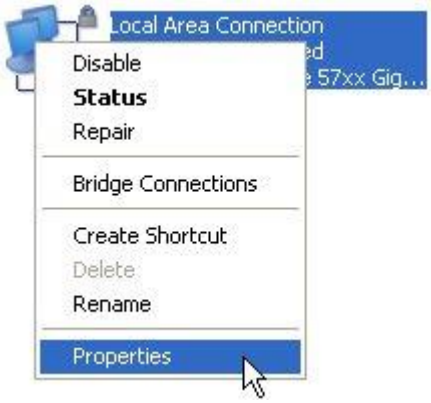
3.1.2 Configuring Ethernet for Windows 7

To configure the PC Ethernet interface for Windows 7 with the Control Panel set to Category view, proceed as follows:

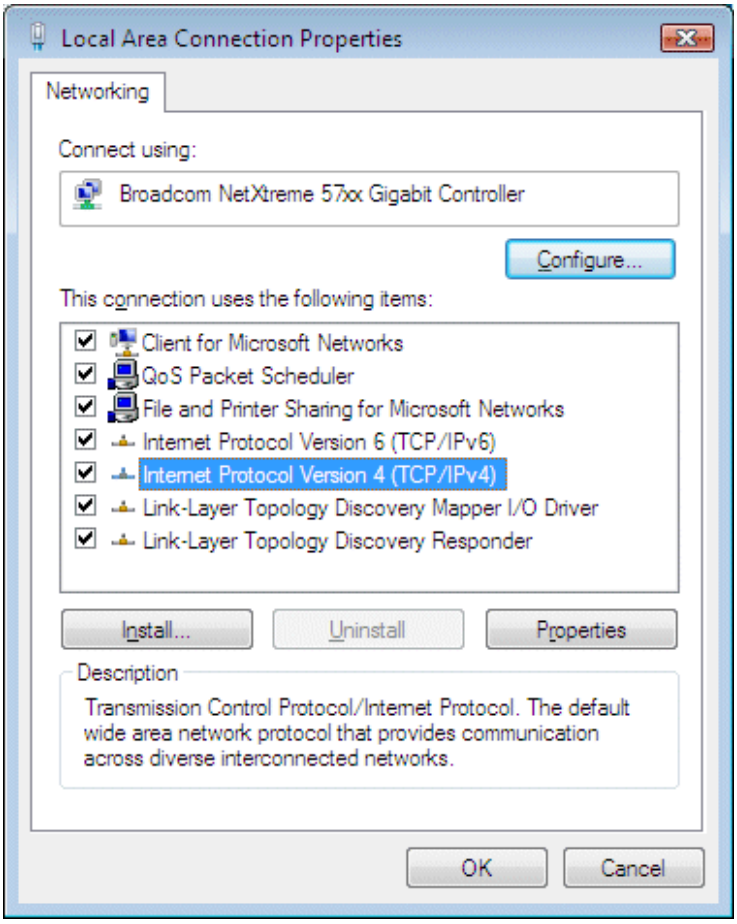
1. At the PC, click *Start* and Control Panel to display the Control Panel.
2. Click on Network and Internet
3. Click on Network connections to display the Network Connections panel containing the Local Area Connection icon as follows:



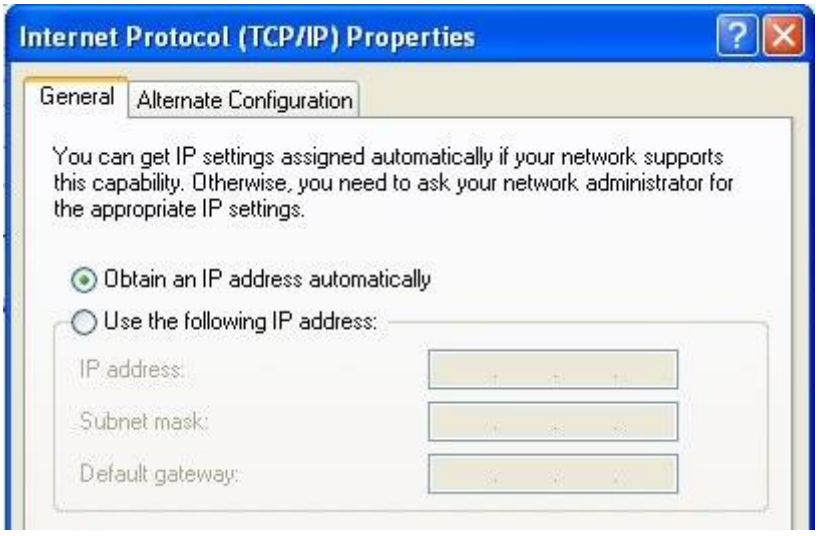
4. Right click on the icon to display the drop-down menu and select Properties:



This will display the dialog box entitled Local Area Connections Properties.

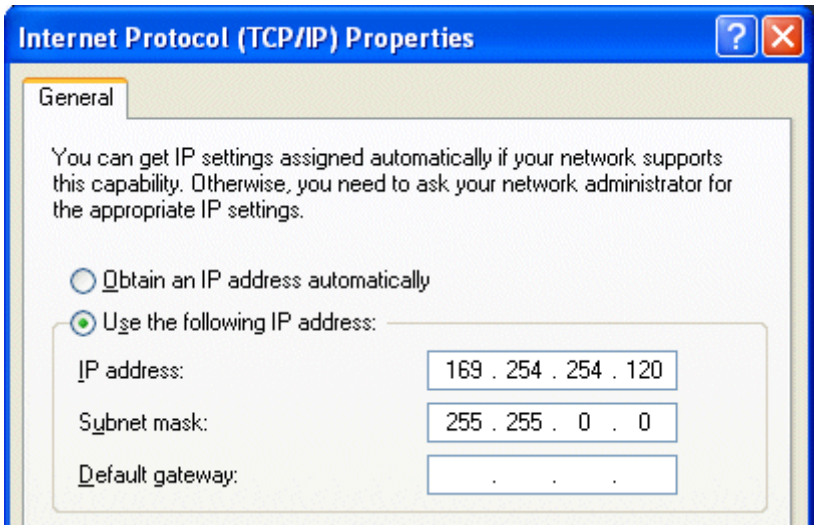


5. Select the Internet Protocol (TCP/IPv4) service and click on Properties, to display the dialog box entitled Internet Protocol (TCP/IP) Properties. This is initially set for automatic IP addressing as follows:



6. Normally the system will work correctly with automatic IP addressing, but if there are problems, select Use the following IP address, then enter the following values:
   - An IP address in the range 169.254.254.120 to 169.254.254.254.
   - A subnet mask 255.255.0.0

A dialog box appropriately filled in is as follows:



7. Click OK on both dialog boxes; and close the Network Connections panel.

NOTE: An alternative range of IP addresses will have to be used, for the PC and the ModuLab, if the PC is connected to a network that already uses the default range of addresses.

8. Click on Manage Network Connections to display the Network Connections panel with the Local Area Connection icon as follows:



9. Right click on the icon to display the drop-down menu and select Properties:

3.1.3 Installing the ModuLab software

The user controls the system from a PC running the ModuLab software, which is supplied on a single CD.

The ModuLab software is installed as follows:

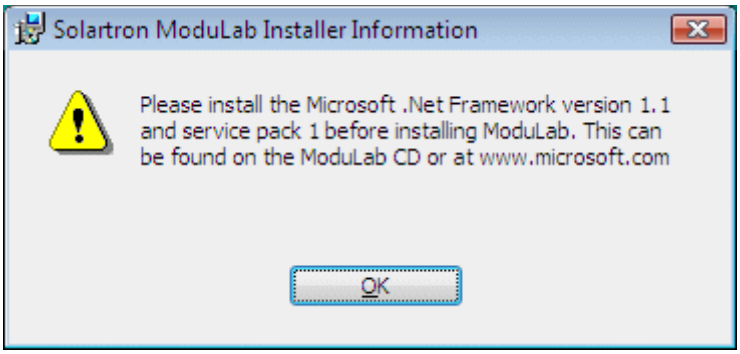
1. Make sure you are logged in to the PC as a user with Administrator privileges.
2. Close down all tasks.
3. Insert the CD into the relevant drive.
4. If the CD does not run automatically, select Start, Run… (in Windows XP), or the equivalent commands in Windows Vista, and enter the pathname of the setup file on your CD, e.g. d:\setup.exe
5. Follow the on-screen instructions.

3.1.4 Microsoft .Net Framework

The ModuLab application is built on a software development platform called .Net Framework, which must be installed on your computer to enable ModuLab to be installed. The required components are:

Microsoft .Net Framework Version 1.1
Microsoft .Net Framework 1.1 Service Pack 1

Other versions of .Net Framework may already be installed for use with other applications, but these are irrelevant to ModuLab, and the two components listed above are still required. If you attempt to install ModuLab on a PC that does not have these components, a prompt will appear as follows:



If this occurs, proceed as follows:

1. Click OK to close the prompt.
2. Close the installation wizard.
3. Run the file DotNetSetup.exe from the CD. This will run the Microsoft Component Installer for Windows.
4. Follow the prompts to install the required components.
5. Restart the ModuLab installation, by running setup.exe from the CD.

3.2 Optical Bench and Interface Card

3.2.1 Interface Card

The PhotoEchem interface card is specifically designed for testing DSSC's and is shown in Figure 4: PhotoEchem Card



POWER: Green Indicates power to the card

COMMS: The Comms light should be green during measurements

ERROR : will light RED if an ERROR is detected

LED on: Will light when the PhotoEchem card controls the LED Driver

The two BNC connectors are labelled LED and DET

LED: Connect this to the LED BNC input on the rear of the DC2100 OR DC2200LED driver (refer to Figure 6) using the 1m BNC / BNC cable provided

DET: Connect this to the BNC connector on the PhotoDetector (see Figure 13)

Figure 4: PhotoEchem Card

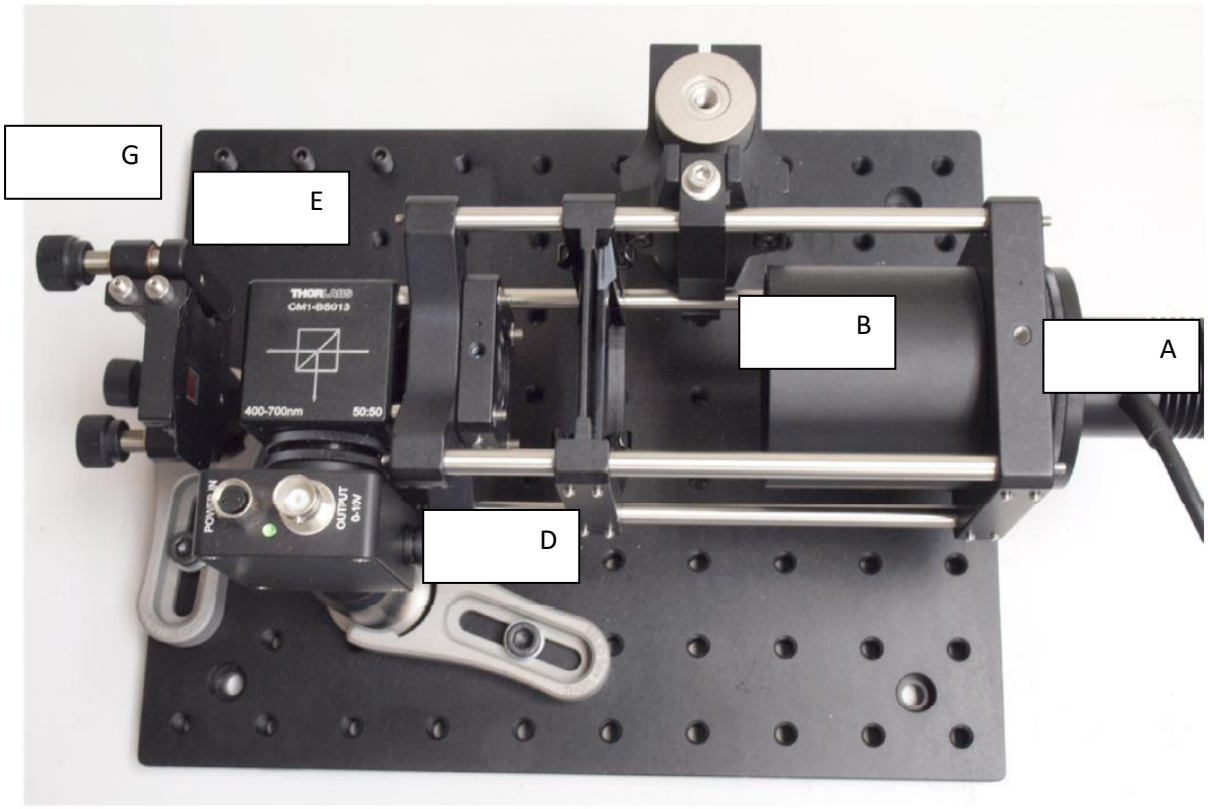
3.2.2 Optical Bench

The optical bench is shipped pre-assembled and therefore should not require major adjustments. In the unlikely event that the system requires reassembly, please contact Solartron Analytical Technical Support who can provide you with details of the assembly process.

The Optical Bench contains the following (shown in Figure 5: Optical Bench Assembly);

A) High Brightness LED
B) Collimating Lens Housing
C) Neutral Density Filter and Filter Holder
D) Focusing Lens
E) 50:50 Beam Splitter

F) Si NIST Traceable Fast Photodetector
G) Sample and Sample Holder



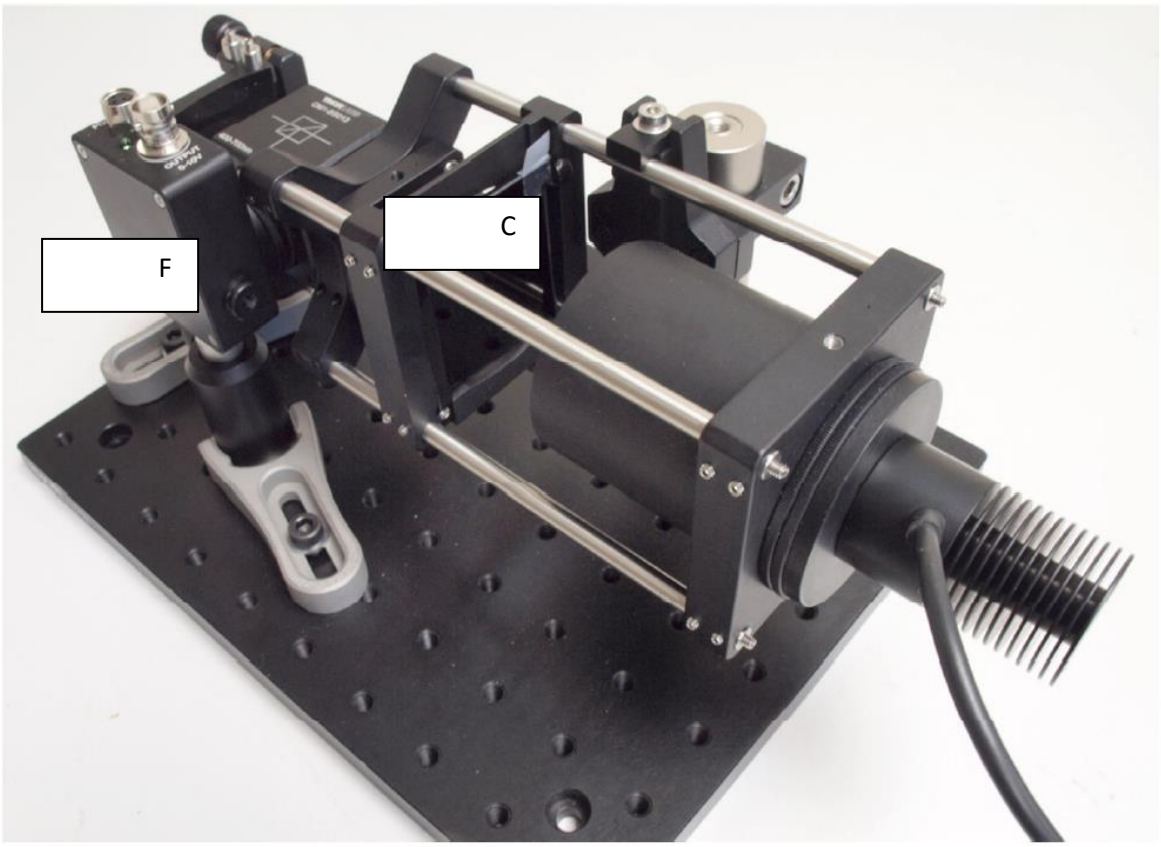


Figure 5: Optical Bench Assembly

The output of the high brightness LED (A) is collimated by the LENS in (B). This produces a uniform beam that can be focused onto the cell by the lens (D). Sometimes it may be necessary to attenuate the light intensity below the levels determined by the LED driver. In this case, one can use the 0.01 Neutral Density filter supplied in the kit (C). Low level intensity measurements will be described in subsequent sections.

The XM system is unique in the reference detection system for light intensity measurements is a permanent fixture and therefore should not be removed unless a fault is suspected with the photodetector. This configuration allows calibration of the output of the bench to be recorded conveniently and without the end user having to assemble complicated optical components. In order to achieve this, the beam is split into two beams of equal intensity through use of a 50:50 beam splitter (E). Please ensure that the arrows on the beam splitter point towards the sample and the photodetector as shown in Figure 5: Optical Bench Assembly.

3.2.2.1 Photodetector

The Photodetector is shipped with a power supply and these specification are shown below.

ATTENTION ! **USE Only this power supply provided. PLEASE CHECK THE VOLTAGE SETTING on the side of the power supply (RED TOGGLE). If you are using this system in a lower voltage region i.e. USA, then please switch to 110V. Failure to operate the system at the correct voltage will result in the damage of the power supply.**

Operating Temp	0 to 40 °C
Storage Temp	-55 to 125 °C
AC Power Supply	AC – DC Converter
Input Power	31 W 100 – 200 VAC (50 to 60Hz) 220 – 240 VAC (50 to 60 Hz)

The photodetector outputs a voltage (0-10V) and this is proportional to the light intensity that it measures. This output is fed into the DETECTOR input on the PhotoEChem card using a standard BNC cable (supplied with the unit).

3.2.2.2 LED Driver

The Thorlabs DC2100 OR DC2200LED driver is supplied with the following connections

- Computer USB cable
- Power Supply (specifications below)

To connect the LED to the LED driver , follow the instructions which are taken directly from the Driver Manual (Thorlabs, 2010) See Figure 6

- Connect LED head cable with socket labelled ‘LED’ at the back of the main control unit.
- Connect power supply cable with main control unit, use only the power supply supplied with the DC2100 OR DC2200 UNIT
- Plug in Power Supply

ATTENTION ! **Prior to switching on the device, please check if the line voltage corresponds to the input voltage range of the power supply.**



Figure 1 Connecting a LED to the DC2100

ATTENTION

Please, install the software prior to connect the instrument to your PC via USB interface.

Only use the supplied high speed (USB 2.0) cable not full speed (USB 1.1) cables or thin profile cables with increased resistance, this can cause transmission errors and improper instrument operation!

Figure 6: LED Driver set-up for DC2100

3.2.3 Sample Holder

Correct positioning of the sample holder with respect to the beam splitter is critical for repeatability. The following section describes how to mount the sample holder provided onto the optical bench. You may choose to use your own sample holders. The same set-up principles apply if you chose to follow this route.

- Figure 7: Assembly of Sample Holder shows how to mount the sample holder onto the post holder. Using the M4 Hex key (supplied with system), ensure that the holder is screwed tightly to the base to ensure that the sample does not move with respect to the beam during testing.

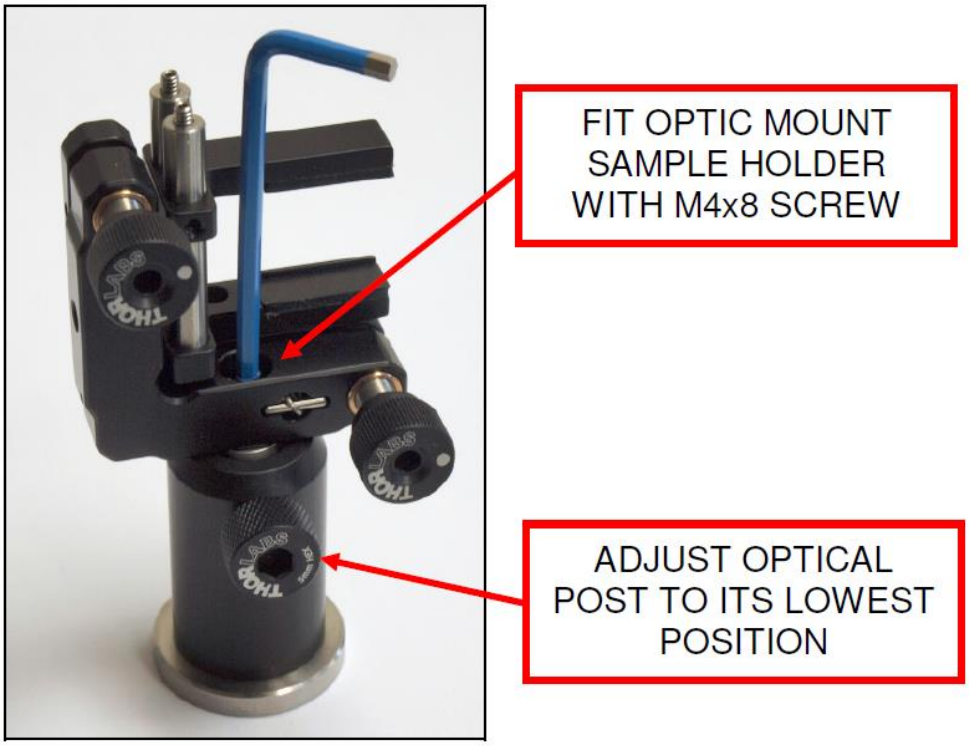


Figure 7: Assembly of Sample Holder

- Solar Cells can be mounted inside the sample holder using the 0.75mm HEX key (supplied). The height of the sample holder can be adjusted by loosening the small grub screws (illustrated in Figure 8: Sample Holder showing adjustable height screws). Note the screws are on BOTH sides of the holder.

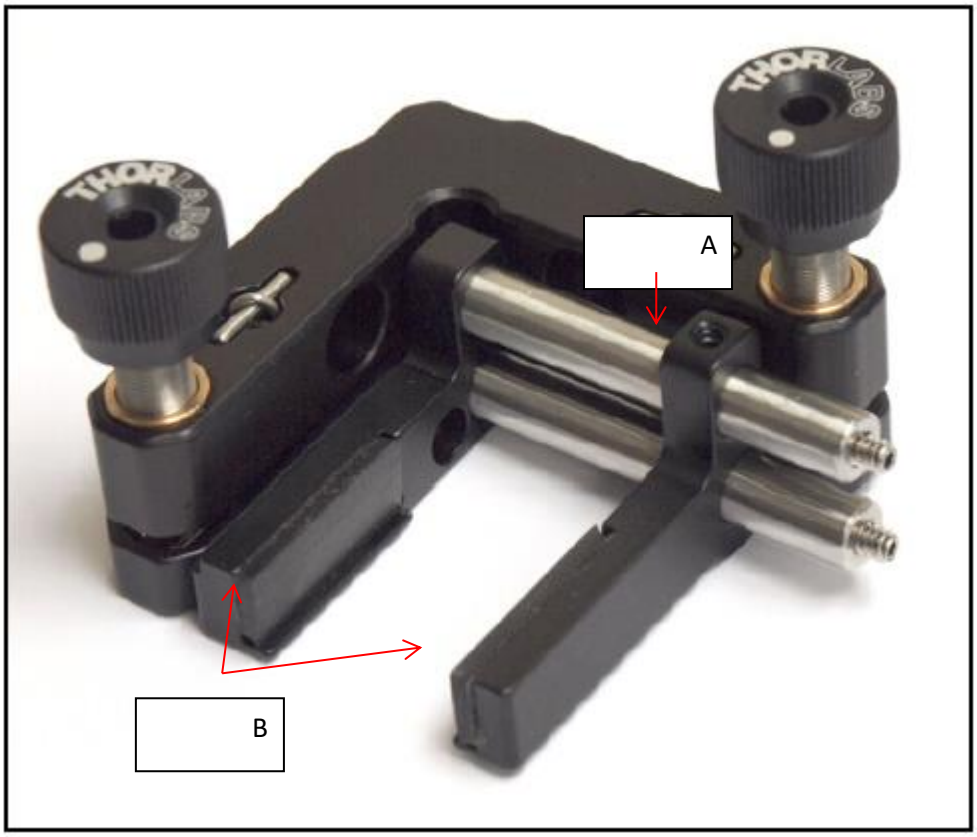


Figure 8: Sample Holder showing adjustable height screws (A) and rubber mats (B)

- The solar cell should be placed onto the rubber mats (shown in Figure 8: Sample Holder showing adjustable height screws (A) and rubber mats (B)) and clamped firmly in place.

ATTENTION ! ***It is important to mount the sample at the same distance from the beam splitter as the photodetector. The following section describes this process in detail.***

- In order to ensure correct calibration and calculation of light intensity, the distance from the sample to beam splitter should be 1.4cm. This is the same distance as the beam splitter to the photodiode detector.
- Using the M4 screws provided as a guide OR a pair of callipers, place the front of the solar cell 1.4mm from the housing of the beam splitter cube. This is shown in Figure 9: Placement of Sample Holder

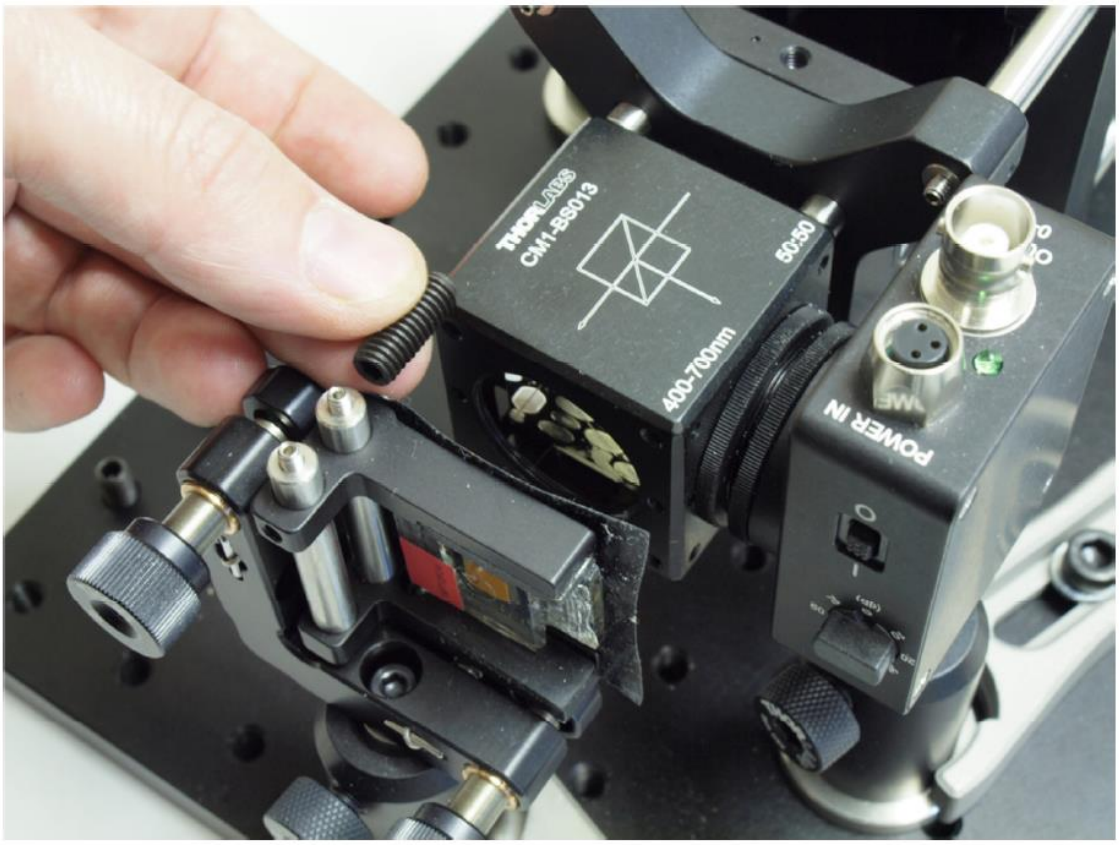


Figure 9: Placement of Sample Holder

- Once in position, place a post holder around the post and secure to the bench using the bolts provided as shown in Figure 10: Placement of Post Holder Clamps.



Figure 10: Placement of Post Holder Clamps

- The active area of the solar cell should be fully illuminated by the light beam. Therefore, it will be necessary to finely adjust the position on the cell within the holder and the

height of the sample holder. This is best achieved by manually adjusting the position of the holder in when the cell is under illumination (see Figure 11: Positioning of solar cell under illumination) The LED can be controlled manually with the DC2100 OR DC2200LED driver and this will be explained in Manual Control of LED Driver



Figure 11: Positioning of solar cell under illumination

3.2.4 Mounting High Brightness LED onto Optical Bench

The LED's are supplied in a Thorlabs carry case. Each LED has a built in chip for identification purposes and is a critical component of the system.



Figure 12: Typical High Brightness LED

ATTENTION ! ***DO NOT USE LED's OTHER THAN THOSE RECOMMENDED BY SOLARTRON AS THE LED DRIVER WILL NOT RECOGNIZE THE LED.***

- The LED's should screwed into the LED lens housing using a clockwise motion. Be careful not to touch the LED head on the PCB.
- Ensure that the LED is **FINGER** tight (do not over tighten)
- LED's can be removed by unscrewing – anti- clockwise. When not in use place the LED in the LED case

3.2.5 Optical Bench Connections for Testing Solar Cells

The following section describes how to connect the optical bench and LED driver to the ModuLab XM system. The set-up is used for all testing of solar cells and experiments using the light source. If calibration of the optical bench is required, please refer to Optical Bench Calibration Connections.

Equipment Required

- DC2100 OR DC2200Thorlabs LED Driver with Power Supply
- Fast Photodetector Power Supply
- 2 x BNC COAX cables (supplied)
- USB cable (supplied)
- WE/CE ModuLab measurement cable
- RE1/RE2 ModuLab measurement cable

The following schematic describes the System connections

(see Figure 13: System Connections)

- Using the USB cable, connect the LED driver to a USB port on the PC.
- Connect a BNC cable from the LED socket on the PhotoEchem Card to the BNC connection labelled Ext Trig on the LED driver.
- Connect the 4-pin LED cable from the High Brightness LED to the LED connector on the LED driver.

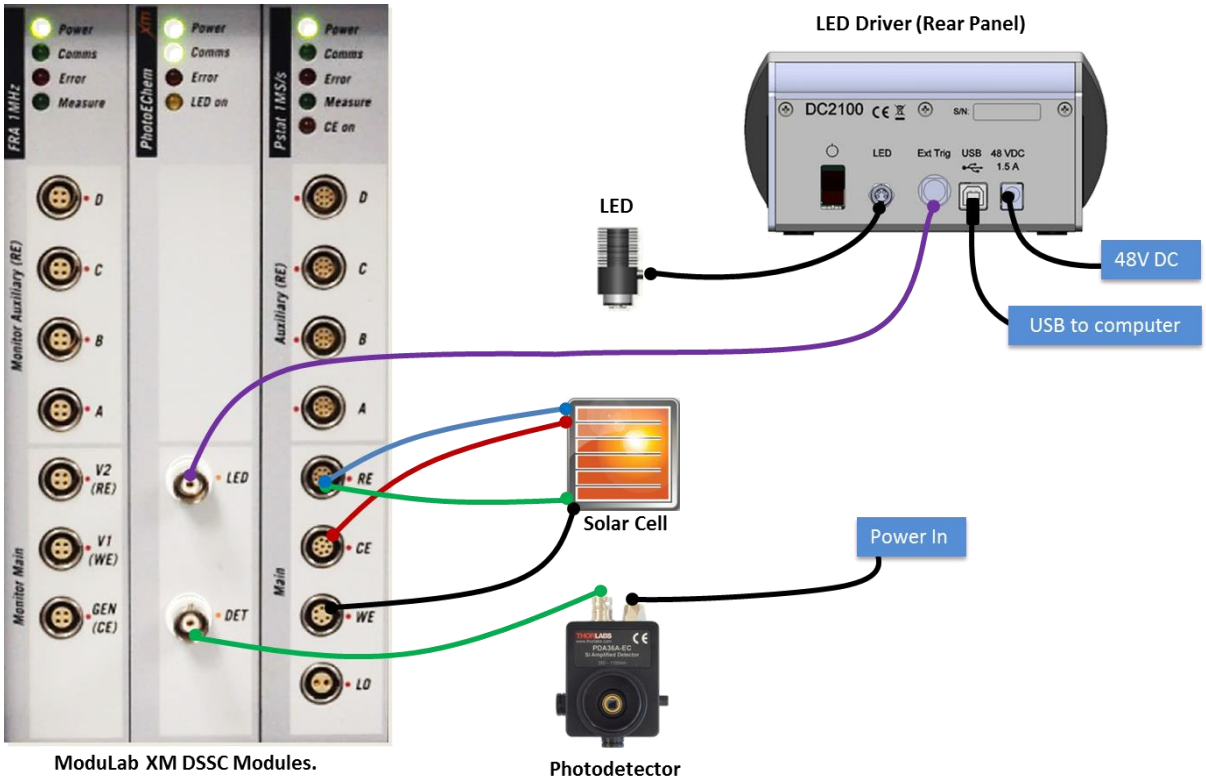


Figure 13: System Connections

ATTENTION ! PLEASE ENSURE THAT BOTH THE LED DRIVER AND THE PHOTODETECTOR ARE POWERED IS POWERED. USE THE POWER SUPPLY PROVIDED. PLEASE ENDURE THAT THE POWER BRICK FOR THE PHOTODETECTOR IS SET TO THE CORRECT VOLTAGE FOR YOUR REGION.

ATTENTION ! PLEASE ENSURE THAT IF YOU ARE USING THE DC2200 DRIVER, the SMA connector to the driver (Ext Trig) must be finger tight. Failure to do will cause offsets in some measurements

ATTENTION ! ***THE FAST PHOTODETECTOR MUST BE POWERED. A Green LED indicates power on the top of the device indicates power on.***

The Photodiode has 8 gain settings, 4 of which are in use with this system. The settings are as follows;

0 dB	x1 Gain
20 dB	x10 Gain
40 dB	x100 Gain
60 dB	x1000 Gain

The default setting is 0 dB and should be adequate for all experiments that do not make use of a Neutral Density filter for low light level measurements. The gain setting can be changed on the side of the detector with the wheel selector. The setting is determine by positioning the indentation on the desired setting. Figure 14: Gain Settings on Photodetector, shows the position for 0dB gain setting.

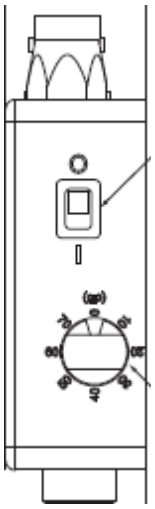


Figure 14: Gain Settings on Photodetector

The ModuLab system can perform two, three and four terminal measurements. For more details refer to the ModuLab ECS guide. For the majority of solar cell studies, two terminal measurements are required.

For Two terminal Measurements;

ANODE: The WE (BLACK) and RE2 (GREEN) cables are shorted together and connected to the Potentiostat card

CATHODE: The CE (RED)and RE1 (BLUE) are shorted together and connected to the Potentiostat card

As an example, the anode and cathode of the REFERENCE cell provided with this system is shown in Figure 15: Solar Cell Mounted in the Sample Holder. The Black Clip connects to the ANODE, the RED clip to the cathode.

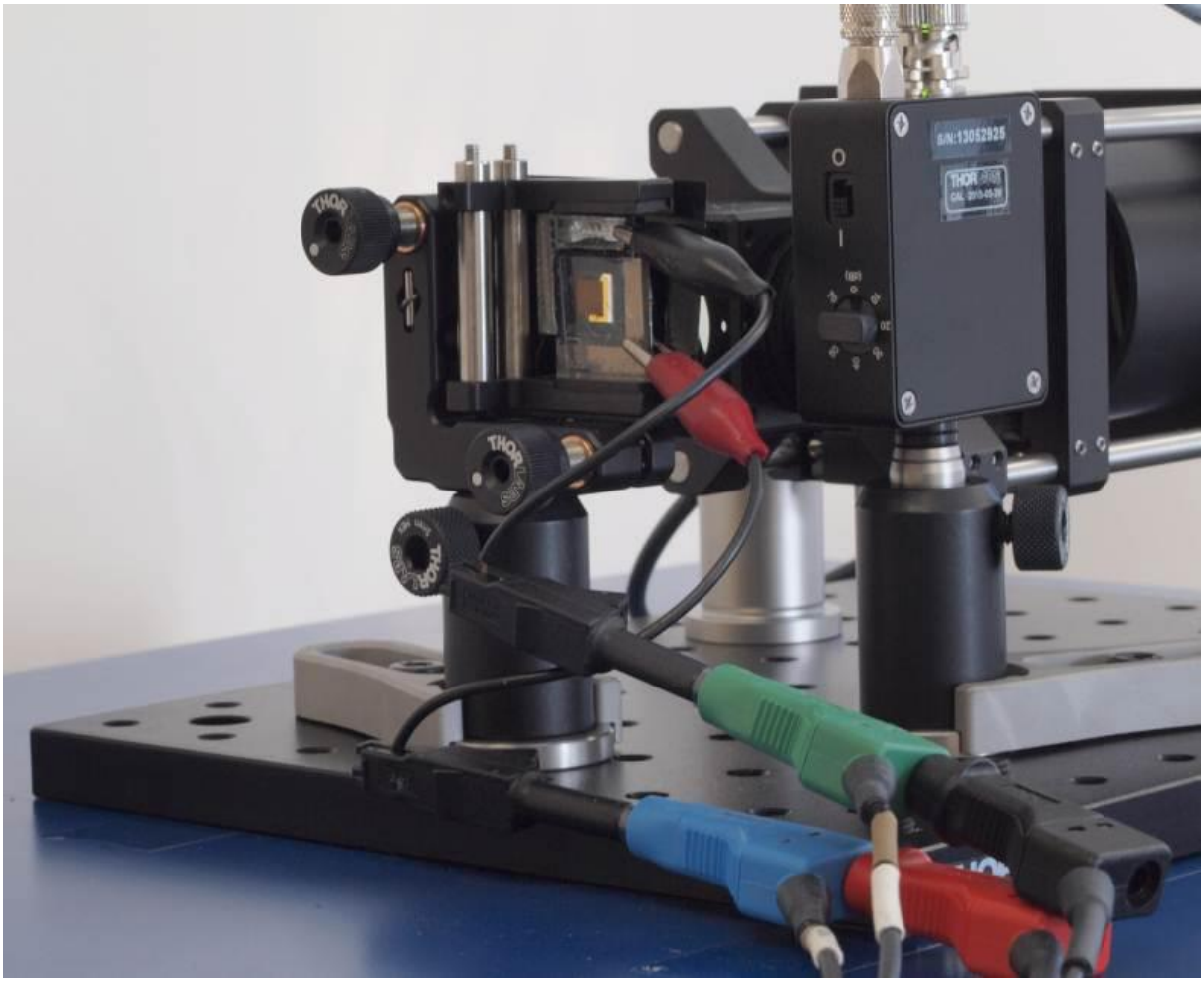
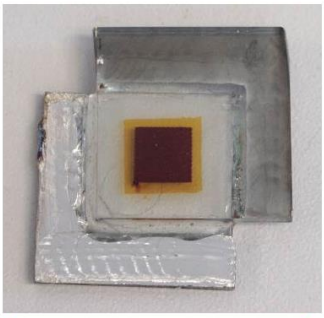
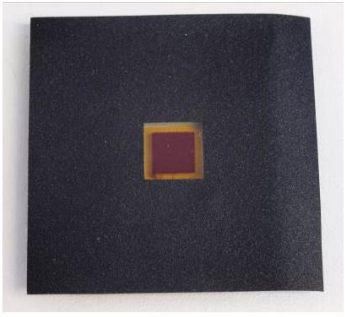


Figure 15: Solar Cell Mounted in the Sample Holder. The Black Clip connects to the ANODE, the RED clip to the cathode

The sample cells are shipped with a BLACK MASK as shown in Figure 16: Reference without and with MASK. The ANODE is the glass electrode to which the mask is attached. The cathode can also be identified by a small injection hole in the rear.



Solar cell sample



Solar cell sample with mask fitted

Figure 16: Reference without and with MASK

For Three Terminal Measurements (three electrode measurements)

ANODE: WE (BLACK) and RE2 (GREEN) shorted together

REFERENCE ELECTRODE: RE1 (BLUE)

COUNTER ELECTRODE: CE (RED)

3.2.6 Optical Bench Calibration Connections

The following connections apply for the CALIBRATION of the optical bench. The software calibration routine will be described in section 5.

Additional Equipment Required

2 X BNC T Piece

2 X BNC to Banana T-Piece

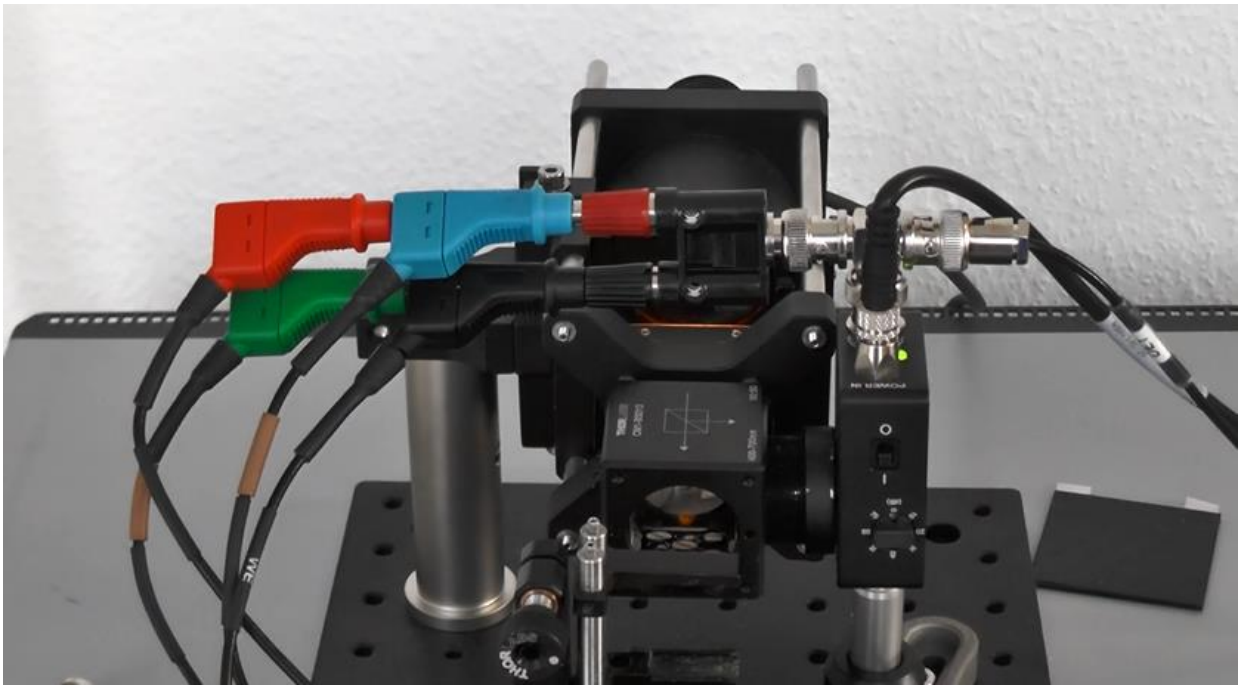


Figure 17: Calibration Cell Connections

- Insert BNC T Pieces into the BNC connectors on the Photodetectors (labelled 0-10V Output).

ATTENTION ! the BNC T piece shown here may differ in appearance to the one delivered with your system. However, both operate in exactly the same way.

- Connect the 'DET' BNC cable from the PhotoEchem Card to one end of the BNC T piece
- Connect the BNC to Banana T Piece to the other end of the BNC connector.

- Using the standard 4 terminal ModuLab Potentiostat cables, short the BLUE and RED cable (RE1 and CE) and BLACK and GREEN cables (WE and RE2) as shown in Figure 17: Calibration Cell Connections. Connect the WE and RE2 to the lo of the BNC (BLACK Banana) and the CE and RE1 to the Hi (RED). Insert the LEMO connector sockets into the LEMO connectors on the Potentiostat Card.
- The system also has provision to calibrate the beam splitter. The split ration of the beam splitter is wavelength dependent and therefore may not always be 50:50 for any given LED. Therefore, a second photodetector is supplied to measure the deviation.

3.2.7 Manual Control of LED Driver

It is sometimes useful to use the LED driver in manual mode. This allows the user to bypass the ModuLab software to control the LED. Situations in which this becomes useful include:

- Positioning and alignment of solar cell in the beam
- Conditioning of Solar Cells prior to testing. Some cells require an illumination soak period as part of the manufacturing process
- Basic System functionality testing

It is recommended that the end user consults with the DC2100 OR DC2200 Manual available from Thorlabs and can be downloaded from the following page

http://www.thorlabs.de/thorcat/18300/DC2100-Manual.pdf

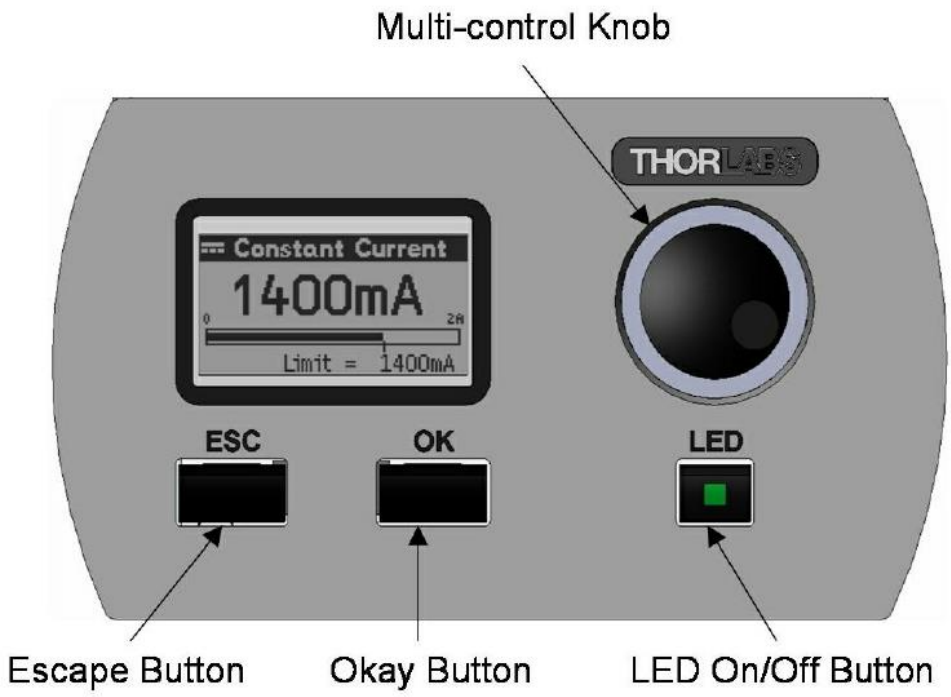


Figure 18: Thorlabs DC2100 - the DC2200 uses a touch screen panel.

To adjust the intensity of the light source using the DC2100 panel;

- Turn LED on using On/Off Button
- Select Constant Current Mode. The level can be adjusted using the Multi-control Knob

ATTENTION! Thorlabs have a built-in safety feature which does not allow you to apply a current ABOVE the operating range of the LED. Therefore, only use LED's specified by Solartron since the LED driver will need to identify the power rating of the LED in use

3.2.8 Care of Optical Bench

On occasion it will be necessary to clean the optical components. A box of optical cleaning tissues has been provided for this purpose. Thorlabs provide an excellent tutorial on care of optics and we advise the customer consult with their site.

http://www.thorlabs.de/tutorials.cfm?tabID=26066

4. Software Installation

4.1 Installation of ModuLab XM PhotoEchem Software

- Insert the CD into the appropriate drive
- If the CD does not run automatically , select, Start, Run….. and enter the pathname of the setup program on the CD
- Follow the on-screen instructions
- If a prompt appears asking for the installation of Microsoft.NET Framework, respond to the prompt and run DoNetSetup.exe from the CD. Then restart the ModuLab Installation.
  Details of Microsoft.NET are in the ModuLab ECS Installation guide

4.2 Installation of LED driver software

4.2.1 Installation of DC2100

Installing LED Driver

You can either install the drivers from the Thorlabs Disk or From the ModuLab installation

Installing from ModuLab Disk

1) Insert the CD into the appropriate drive
2) Open the Folder called DC2100 or DC2200
3) Run the setup.exe file.

Installing from Thorlabs Disk

1) You will first need to install the LED driver. Insert the Thorlabs LED CD model DC2100 into your disk drive.
2) Please search for the following folder ‘DC2100’
3) Open this folder and select either DPInst.exe for Windows 32 Bit users or DPInst_x64 for Windows 64 bit users. If in doubt click START --→ right click COMPUTER and click properties
4) Follow onscreen instructions to complete installation

4.2.2 Installation of DC2200 Driver

PLEASE DO NOTE CONNECT THE DRIVER TO THE PC BEFORE INSTALLATION

Installing from ModuLab Disk

1) Insert the CD into the appropriate drive
2) Open the Folder called DC2200
3) Double click on the icon  ”Download Drivers”
4) You will go directly to the Thorlabs website. Please follow the onscreen instructions.
5) If you cannot find the software, visit the Thorlabs website, www.Thorlabs.com and search for DC2200. The software update will be available for the driver on that page.

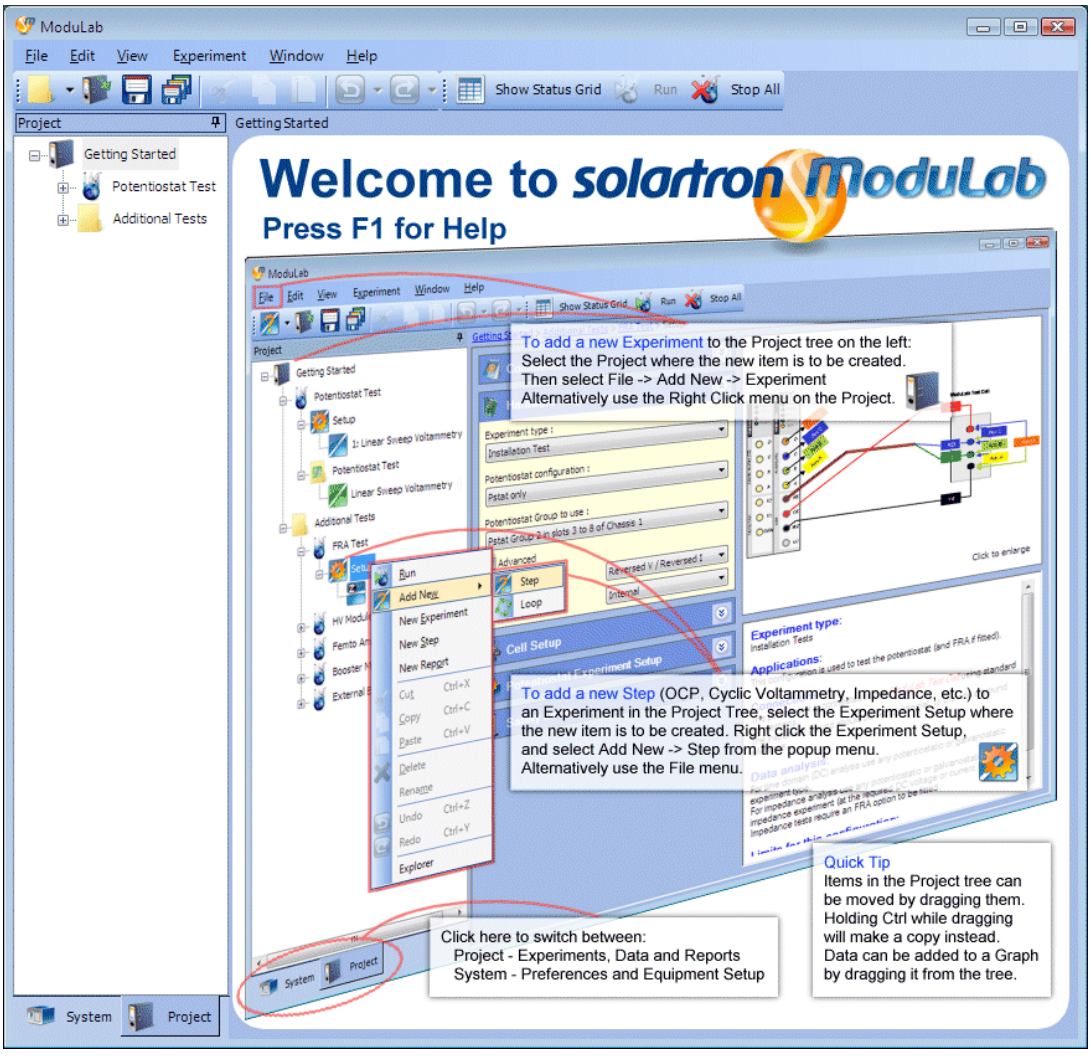
5. ModuLab XM PhotoEchem User Interface

The ModuLab XM PhotoEchem user interface has been specifically designed for experiments for DSSC's and other third generation solar cells. It builds upon the ModuLab ECS platform and therefore this manual will only focus on the features unique to DSSC. For all other applications, please refer to the ModuLab ECS manuals which are supplied with the system.

5.1 System Communication

5.1.1 Potentiostat Set-up

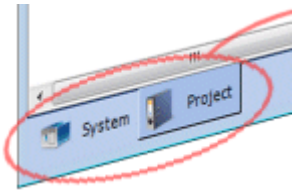
This is followed by the opening screen as follows:



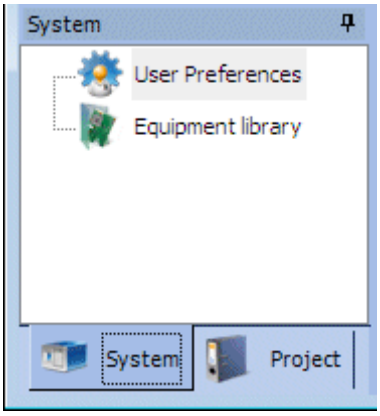
This screen has a left-hand navigation panel containing a structure of items representing the most recently opened *project*. The Getting Started *project* appears by default when ModuLab is first installed and run. The right-hand window displays a Welcome screen.

There are two tabs at the bottom of the left-hand navigation panel:

- **System.** This tab displays the User Preferences and Equipment Library.
- **Project.** This tab displays the currently open *project*.

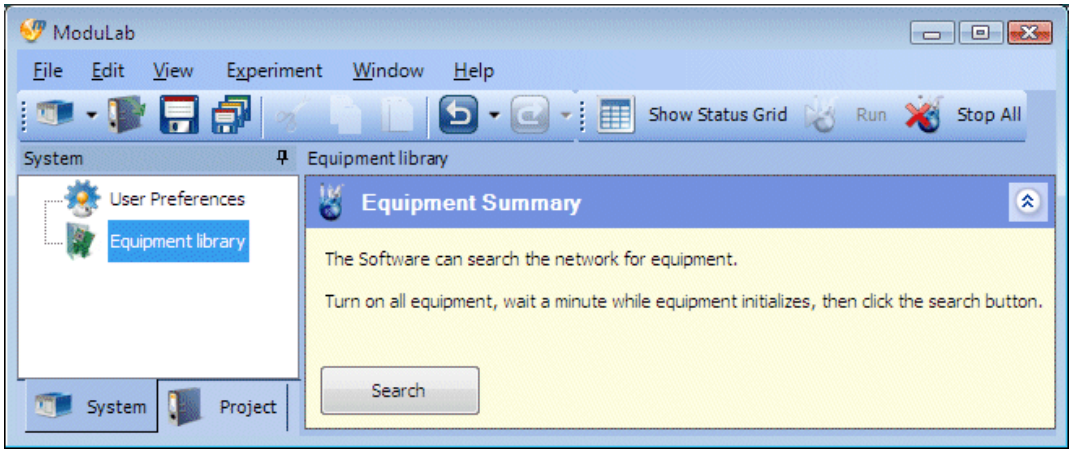


- Click on the System tab at the bottom of the left-hand navigation panel to display the User Preferences and Equipment library as follows:



The User Preferences define features of the ModuLab software that are specific to the user of Windows, and do not need to be changed at this stage. The Equipment Library is defined during this system installation.

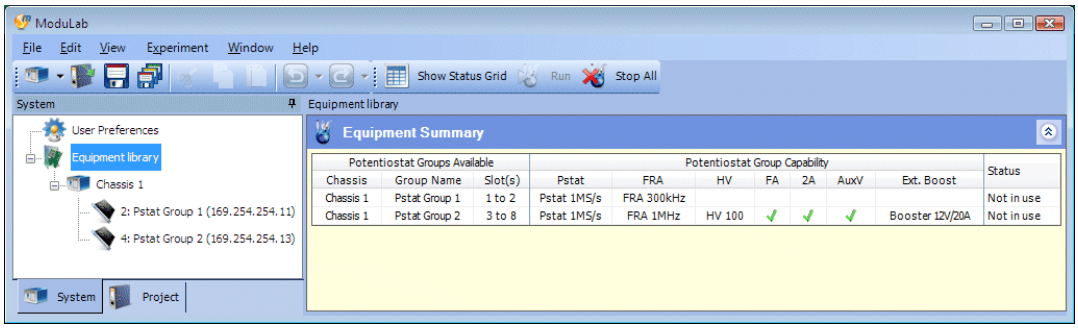
- Click on the Equipment Library to display the Equipment Summary. This will be initially empty because the system has not yet recognised any of the hardware, and there will be a Search button as follows:



- Click the Search button, and the system will identify all the items of equipment as follows:
  - The Search button will disappear and will be replaced with a tabulated list of equipment.
  - The Solartron logo will disappear from the ModuLab front panel and will be replaced by information about the modules.

  The appearance of a module in both of these lists indicates that the PC is able to communicate with the module.
- A plus symbol (+) will appear alongside the Equipment library in the left-hand navigation panel, to indicate that there is a structure of items underneath it,

and you can click the symbol on each item to expand the structure. An Equipment library with the expanded structure is as follows:



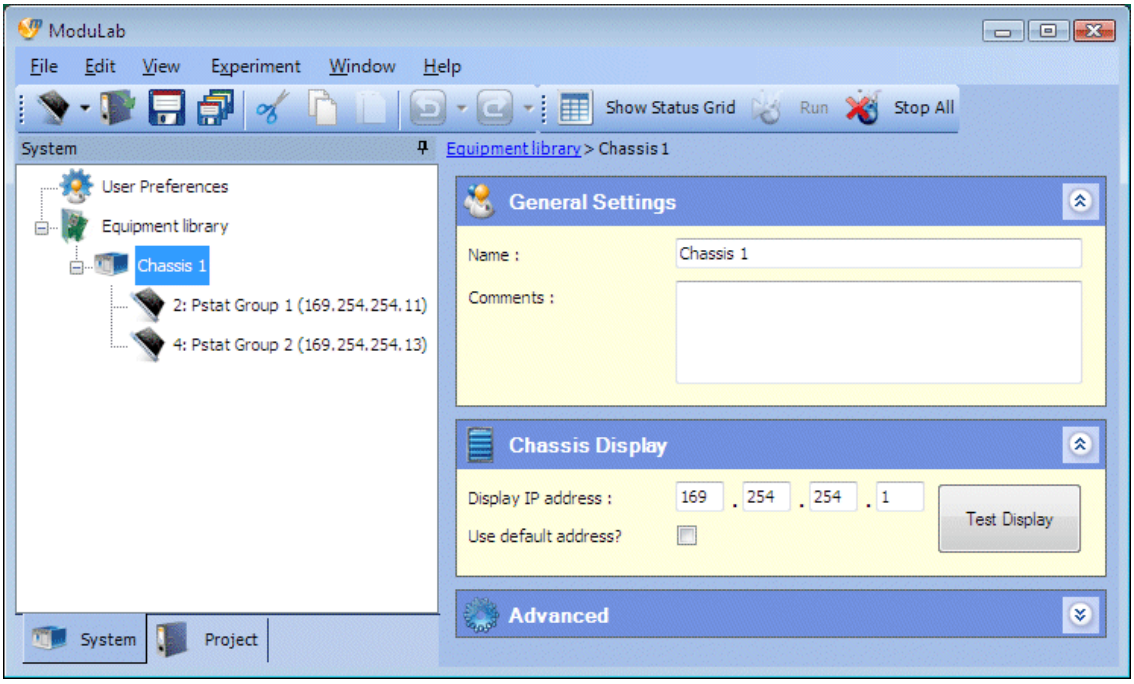
This shows a tabulated list of *potentiostat groups*, each consisting of a potentiostat and a number of optional modules, with one row for each group. The major column headers are as follows:

- **Potentiostat Groups Available.** The *chassis* and *potentiostat group* are identified by name, together with the slots occupied by the group.
- **Potentiostat Group Capability**. The potentiostat and optional modules are identified by model type. If an optional module is absent, the field is blank.
- **Status**. This column identifies the activity being performed by the *potentiostat group*.

Check that the tabulated list of equipment corresponds to your system hardware.

- If a chassis is missing, you can add one. However, there has to be at least one chassis to get an Equipment Summary, otherwise the Search button remains on display.

Click on a *chassis* name, for example Chassis 1, in the left-hand navigation panel, to display the *chassis* details as follows:

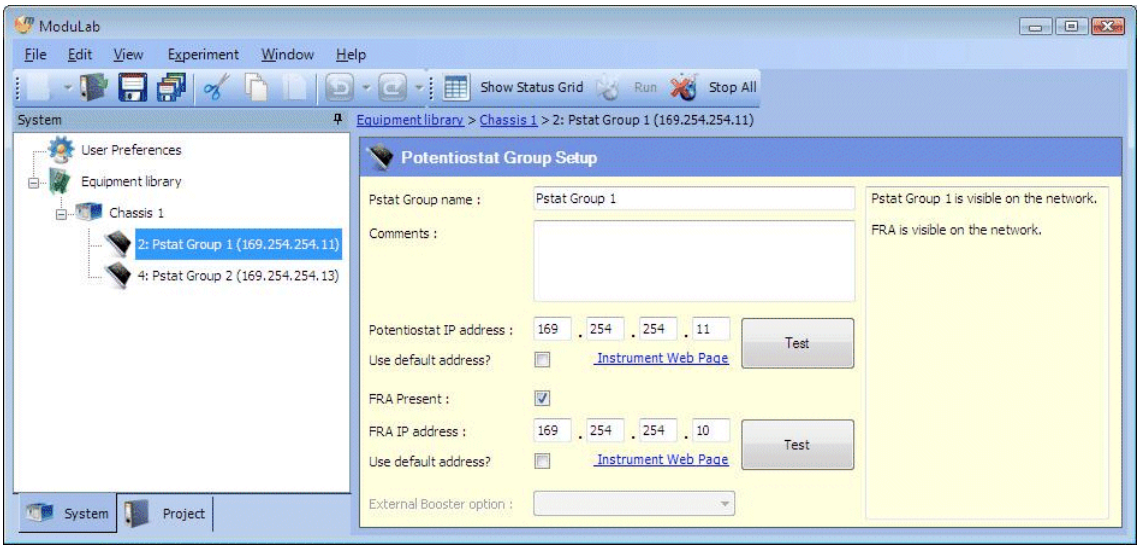


There are three sub-screens:

General Settings. You can specify a name for the chassis and make some comments. The default name is Chassis, but if it has been automatically detected by the system, it will be Chassis 1, Chassis 2, etc., for each successive chassis that is found. You can change it and the new name will appear in the left-hand navigation panel.

- **Chassis Display**. You can specify the IP address of the chassis front panel display. This IP address must correspond to the address that has been pre-programmed into the display panel hardware, to enable communication to occur. If you specify a different address, communication will fail, but the hardware address will not change. If the Use default address? box is checked, the default address for the display panel will appear, but again the hardware address will not change. You can click the Test Display button, and if communication is successful, a test message will appear on the chassis display.
- **Advanced**. This feature enables you to change the address of the chassis display panel hardware, and should normally only be used if there is a conflict of addresses within the system.

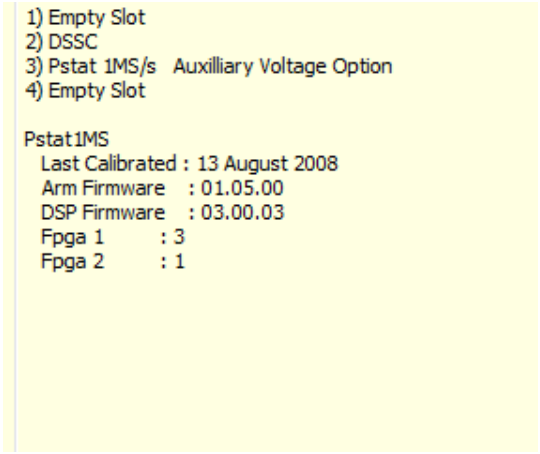
Click on a *potentiostat group*, for example Pstat Group 1, in the left-hand navigation panel, to display the Potentiostat Group Setup as follows:



This screen enables you to specify a name for the *potentiostat group* and make some comments, and then there are IP address fields and Test buttons for the potentiostat and FRA.

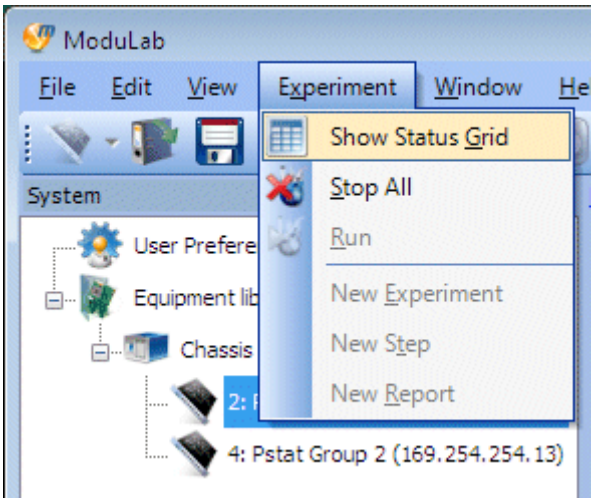
Make sure the FRA present box is checked. This should be checked by default, if an FRA is detected when the system searches for equipment (Step ?).

1. Make sure the addresses are correct for the potentiostat and the FRA if fitted. If a module uses the default address based on the slot number, select the Use default address? check box. Otherwise, if it has a different address, leave the box unchecked and make sure the address in the four-part field is correct, then click the Test button. This will set the address within the ModuLab software and will test the system, and if successful it will display details of the module. It will also display a list of other modules in the chassis, and their details. You can click the Test button at any time, to test the system, regardless of whether or not an address has been changed.
   When the Test button is pressed, a test message appears as follows:

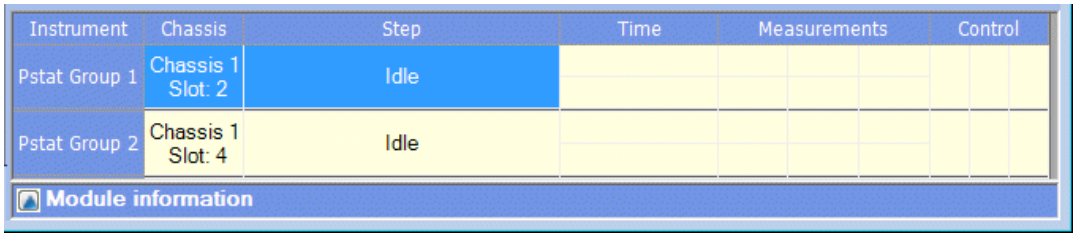


2. Click on the next *potentiostat group*, for example Pstat Group 2, in the left-hand navigation panel, and repeat these instructions from Step 0.

3. To display the activity of potentiostats, as the core modules of each group, click on Show Status Grid in the Experiment menu.

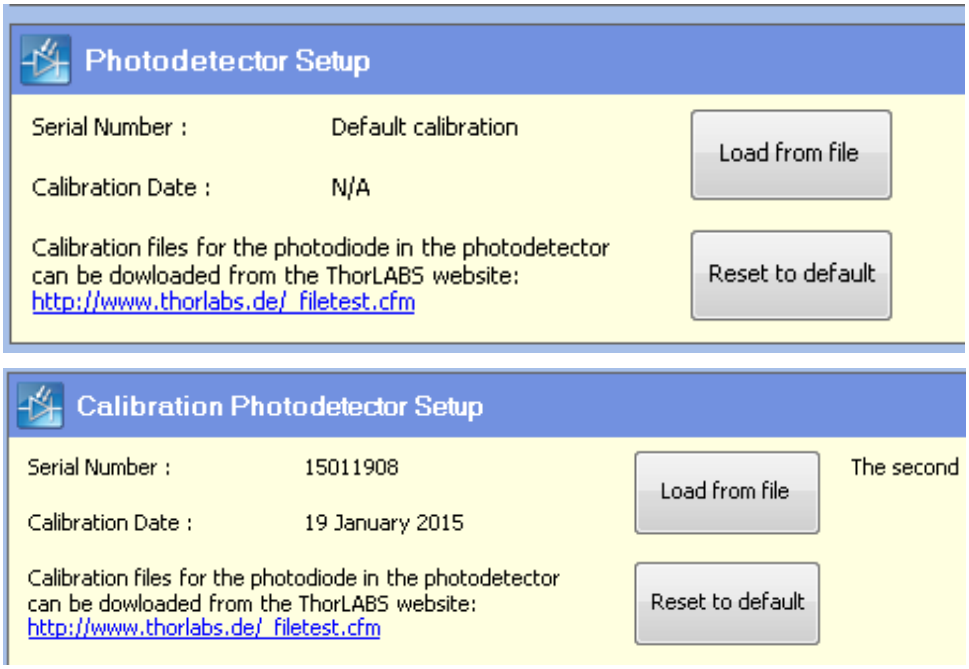


This will show the status of each potentiostat that has been tested using the Test button (Step 1), as follows:



5.1.2 Photodetector Setup

The system uses TWO NIST traceable Photodetectors for calibration (one for measurement). It is therefore necessary to load the two calibration files into the system. There are a number of methods of achieving this.



- The calibration file SPECIFIC to your system is available to download from Thorlabs using the link which is given in the software. Please make a note of the serial number of the photo-detectors since you will need this to retrieve the file information. The serial number appears on the REAR of the photo-detector and is shown in Figure 19. Note,
- The photo-detector that is pre-fitted to the bench is used for the PHOTODETECTOR SETUP box
- The second photo-detector is used for the CALIBRATION PHOTODETECTOR SETUP dialogue box.

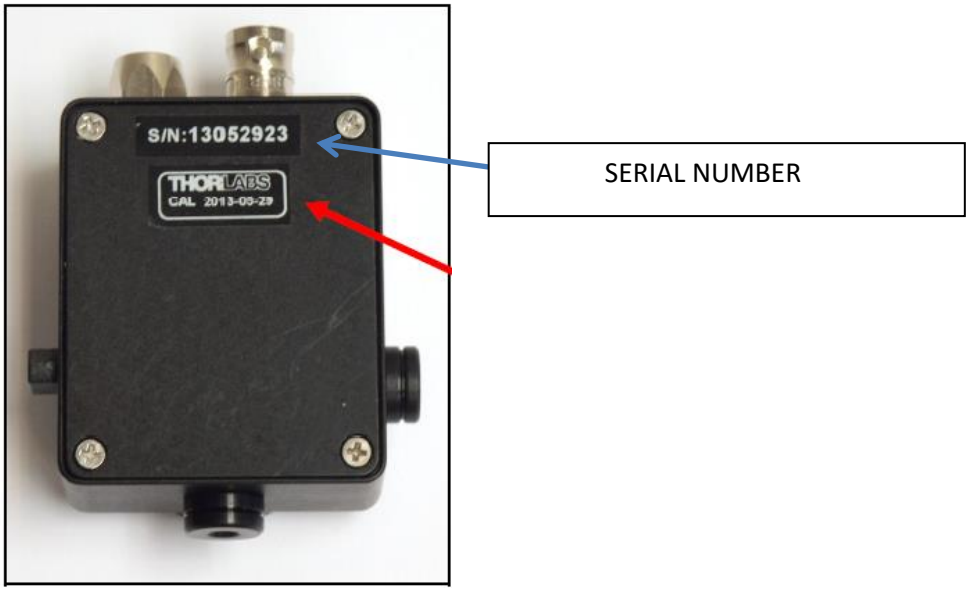
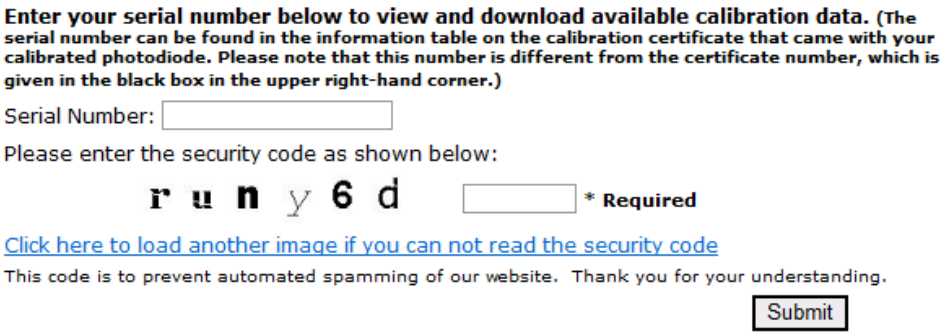


Figure 19: Photodetector Rear Panel Showing 1) Date of Calibration RED AROW and 2) Serial Number BLUE ARROW

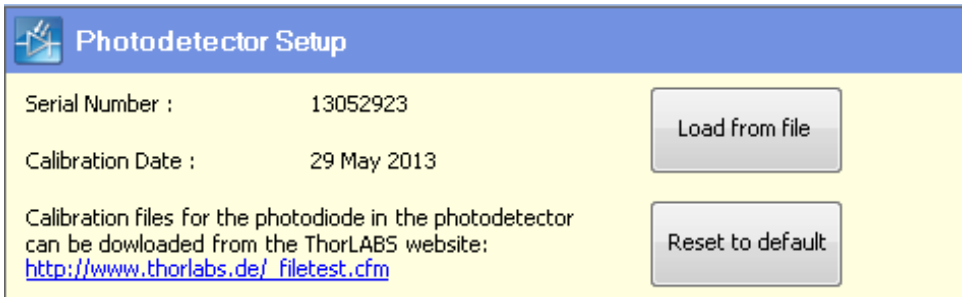
- Once in the site, you will be prompted to enter the serial number.



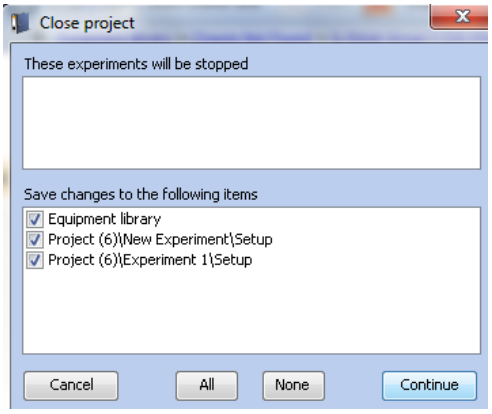
Follow the onscreen instructions

If you have entered the correct serial number, you will be given the option to save the file. Please make a record of the destination of the download. We recommend that you use C:\My Data\ModuLab DSSC

- Press the 'LOAD from file' button. You will need to search for the calibration file in this folder.
- You should now see that the Serial Number and Calibration Date fields are updated similar to that shown ;

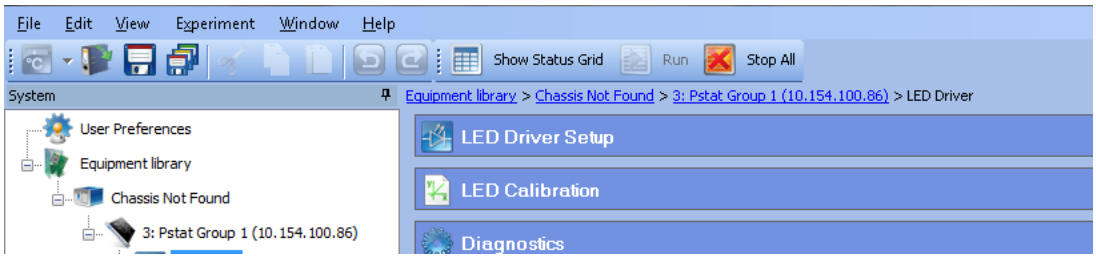


When exiting the project, you will be prompted to save the project. If you leave the ticks in the equipment library box, the set-up will be saved including the calibration file data. You will therefore not need to set-up the calibration setting each time you re-boot the software.

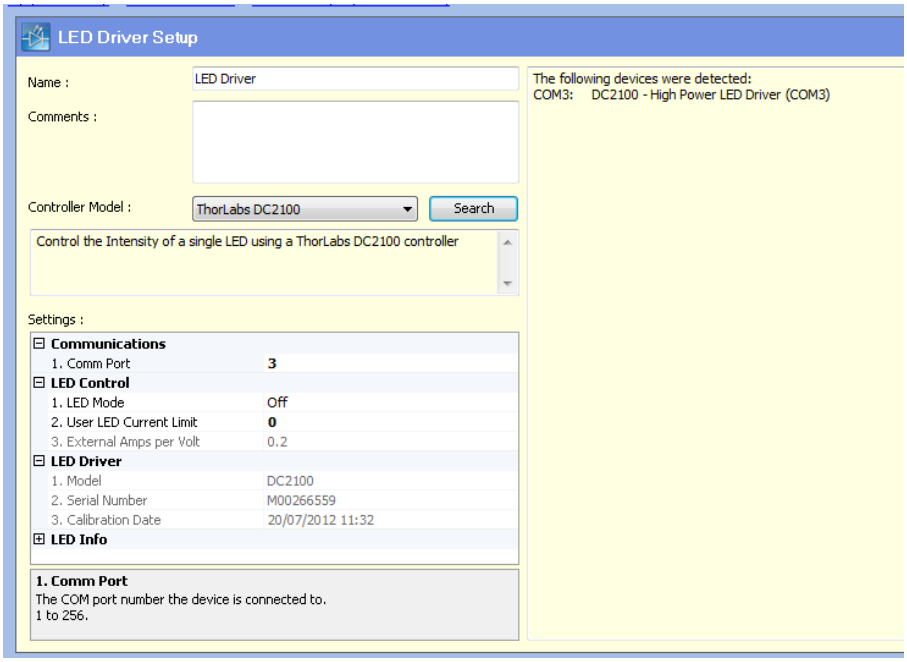


5.1.3 Setting up LED driver

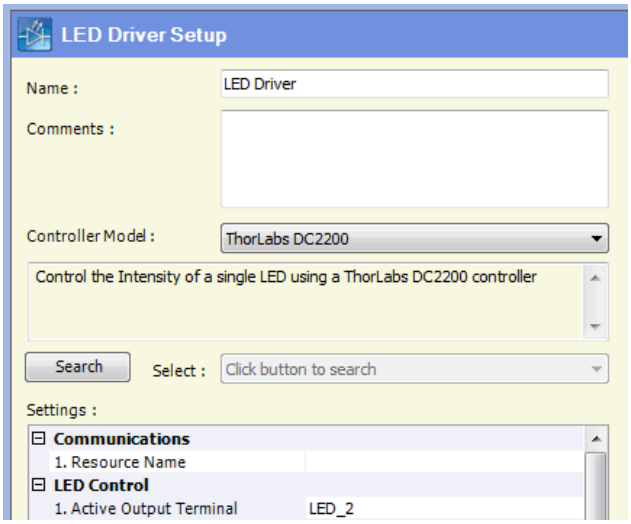
In order for the ModuLab software to control the LED driver it is necessary to setup the LED driver in the equipment library panel. In the system tree, you should notice a BLUE LED driver icon under the potentiostat group.



- Left click on this icon to reveal the three headings in the next pane; LED Driver Setup, LED Calibration and Diagnostics
- Please ensure that the LED driver is switched on and plugged into a USB port of the computer



- **Controller Model** – unless otherwise stated, the controller model will default to the Thorlabs DC2100 OR DC2200 Driver. The system is at present only designed to work with this driver. Use the search function to find the LED driver. If successful the message in the right hand pane will appear. If no device is detected ensure that the USB cable is connected properly
- **Settings for DC2100 Driver only –**
    1. FOR THE DC2100 driver - Check that the Comm Port number is the same as that stated after the Search test. In this instance, COM port 3 was found. This may change from one PC to another
    2. Enter the value of the Max Current Limit that can be applied to the LED. This will be contained in the LED manual
- **Settings for DC2200 Driver only-**
- Select Thorlabs DC2200 from the controller Model List



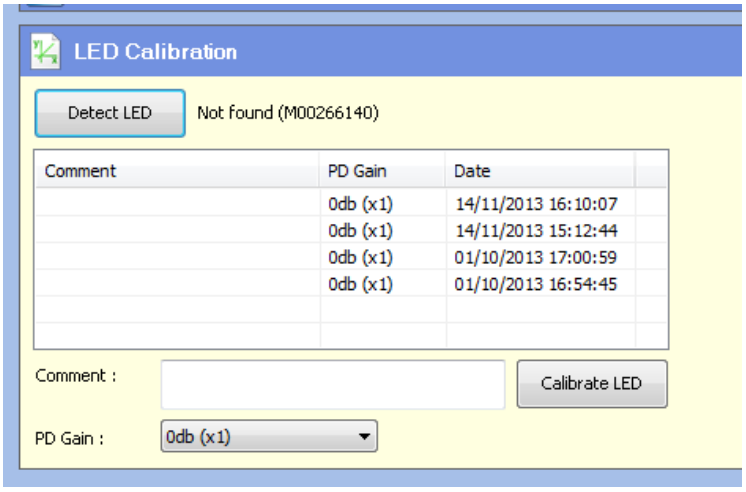
- Press the 'Search' Button – if a device is found, it's identity will be displayed on the right hand side of the screen in the form of a serial number e.g. DC 2200 (M00335958)
- From the 'Select' dropdown menu, select this device.

5.1.4 LED Calibration Routines

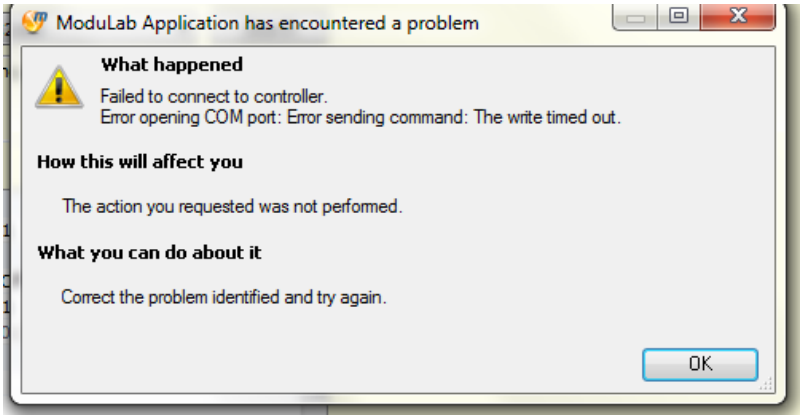
The following section describes how to calibrate the LED for routine operation and calibration of the beam splitter. The beam splitter characteristics as a function of wavelength are non-linear and therefore it may be necessary to calibrate the beam splitter for each LED.

5.1.4.1 LED calibration

This calibration is used for all measurements except for the IPCE module (see section 11). The system is calibrated using software and hardware control. This step is automated once the initial settings are determined. To set- up the system firstly clikc in the LED icon in the SYSTEM tree;



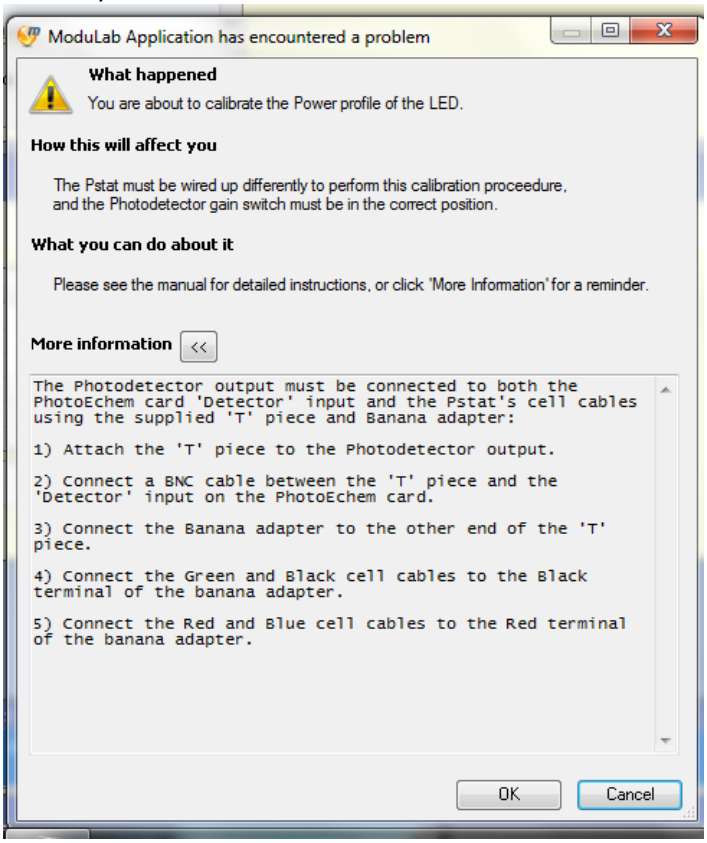
- Press the 'Detect LED' button
- If the COMM port is incorrect, the following message will appear. If so check that the COMM port entered in the Setup Box corresponds with the COMM port that was found in the search function



- If the COMM port is correct, an LED will be detected and ANY previous calibration files for that LED will also be shown.

ATTENTION ! It is recommended to include a comment in the comments box for future reference

- To create a NEW calibration file, connect the system as discussed in Chapter 3 and press the Calibrate Button
- You will receive the following warning which is a reminder to connect the hardware correctly. Press OK to continue



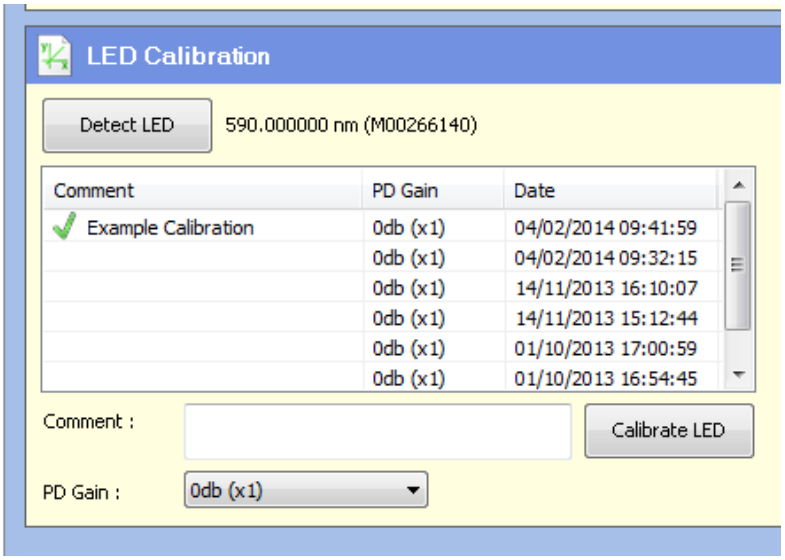
- The system will automatically perform the calibration routine and will take a few minutes

ATTENTION ! It is recommended that a calibration routine is performed once per month as the output of the LED will diminish with time

- It is optional to enter a comment in the ‘Comments Box’
- For the majority of testing, the PD gain setting in the LED calibration should be set to 0dB.

ATTENTION ! If you are using a Neutral Density Filter it will be necessary to change the gain setting in software AND hardware. The gain settings on both MUST match otherwise there will be significant errors in measurement. Calibration using Neutral Density Filters will covered in the next section

Once the calibration is complete, the LED Dialog box will appear as follows



- A tick mark will automatically appear against the latest CALIBRATION FILE. This file will be used for subsequent experiments. If you wish to use a previous CALIBRATION file as a method to cross check results, click the file of choice.

5.1.4.2 Beam Splitter Calibration

This procedure describes how to calibrate the beam splitter.

ATTENTION ! This procedure need only be performed ONCE per LED. When the calibration files have been generated, the beam splitter calibration file is valid for all experiments with that setting.

- Screw the second photo-detector onto the front port of the beam splitter. This need only be finger tight. This is the same arrangement as the Reference Photo-detector that is already aligned with the bench. This photodetector is called the CALIBRATION PHOTODETECTOR.
- Connect a BNC T – piece to the OUTPUT of the detector. On one end, place the 50 Ohm terminator BNC piece (see Figure 41: Connections to second PD amplifier for Beam Splitter Calibration

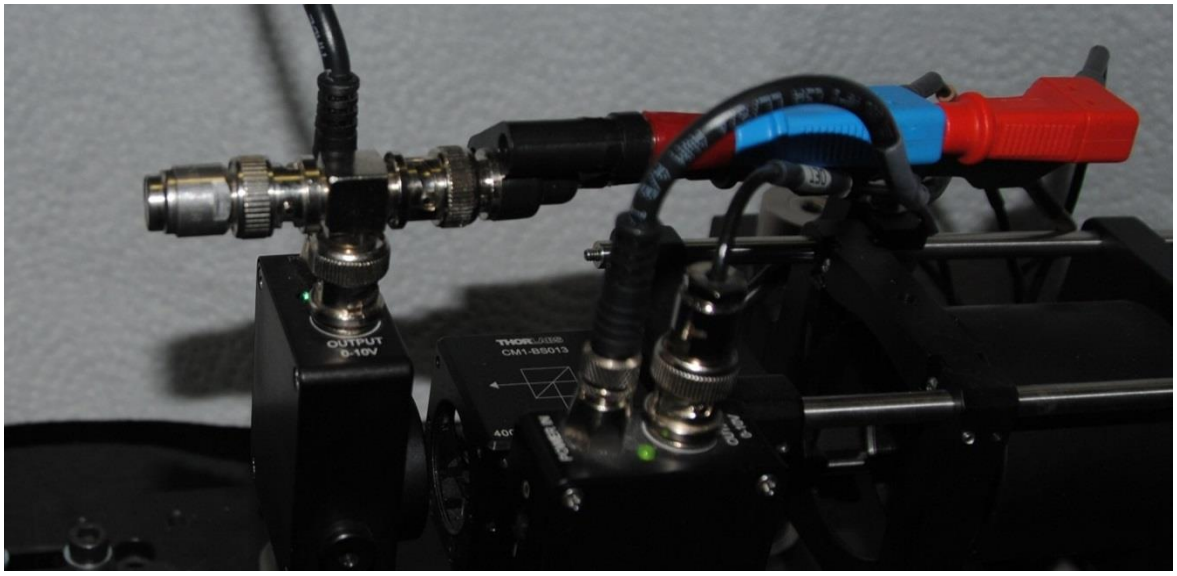
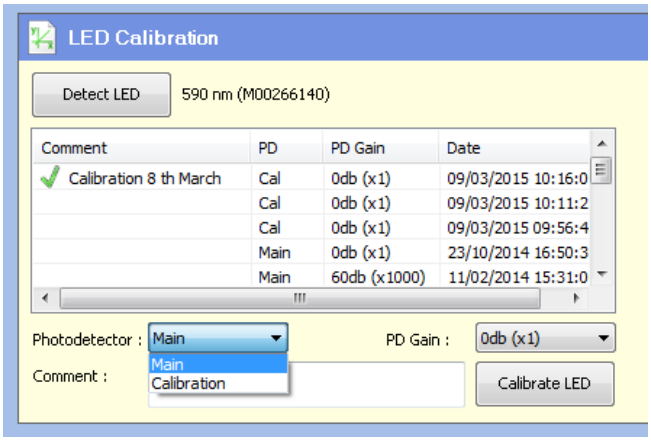


Figure 20: Connections to Calibration PD amplifier

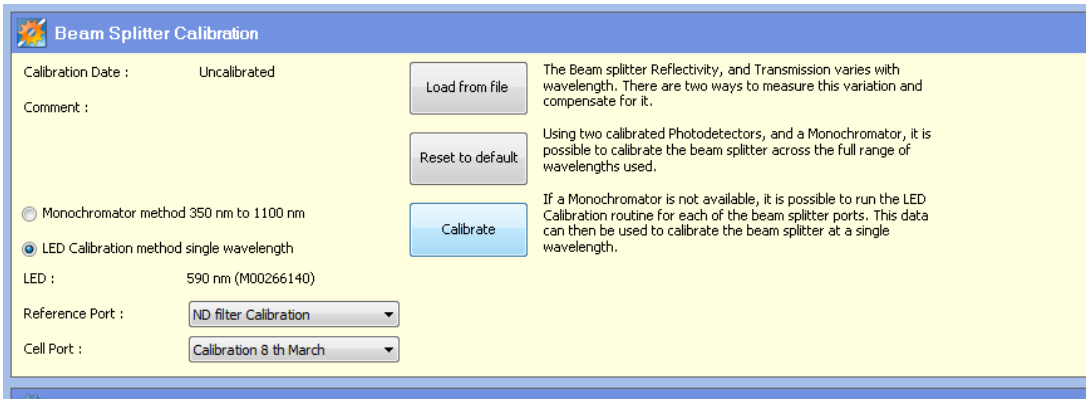
- To the other end, attach the BNC to Banana Plug adaptor.
- Place the Green and Black leads of the potentiostat card in the BLACK connector
- Place the Red and Blue leads of the potentiostat card into the RED connector.
- Please ensure that both photo-detectors are set at the same gain setting.
- Place the detector BNC cable from the PhotoEchem card to the 0-10V Output connection.

Note: it is only necessary to run this test with the PD set in the 0dB position.



To create the beam splitter calibration, it is necessary to generate TWO files.

- Calibrate the MAIN port of the beam splitter using the MAIN selection on the Photodetector (see above).
- Calibrate the CALIBRATION port of the beam splitter using the Calibration setting (see above).
- Once the calibration files have been generated, click on the pstat group in the system tree and select the BEAM SPLITTER DIALOGUE box.



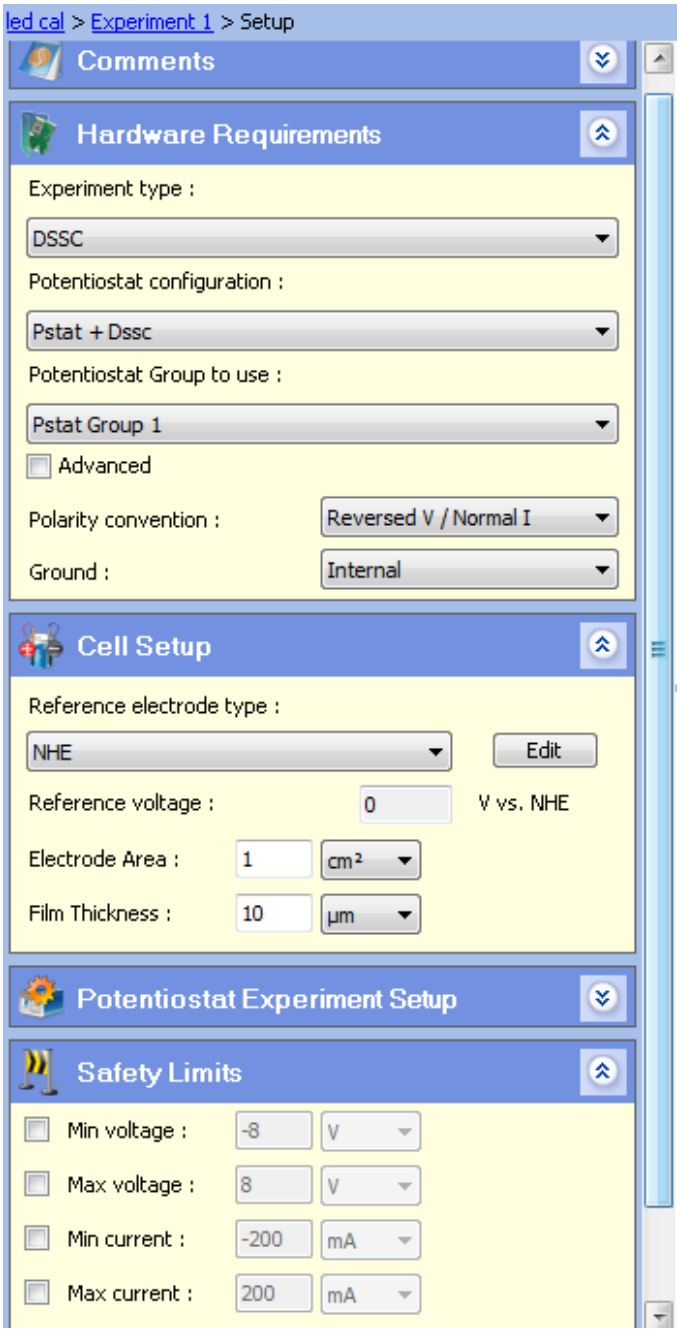
- Highlight the LED calibration method button and select the calibration files from the drop down lists. Both the Reference Port (Main Photo-detector and Cell port (Calibration photo-detector) need to be populated. The software automatically determines if a calibration file is from the main or calibration photo-detectors to avoid errors.
- Beam splitter calibrations can also be loaded from file.

6. Creating Experiments

The system is supplied with a range of experiments that have been specifically designed for the testing of DSSC's. The system contains several features that greatly ease the analysis and reduce complexity of the measurements. The following sections describes how to start experiments and provides details of the techniques specifically designed for testing DSSC's.

6.1 Getting Started

Before beginning any experiments it is important to enter specific information regarding the cell since much of the analysis described in later sections uses these values. In the Cell Setup Box, it is advisable to enter both the thickness of the Film and the surface area of the sample. We have entered a typical film thickness value of 10um.



6.1.1 Auto vs. Advanced Experiments

The ModuLab XM PhotoEchem was designed for both the newcomer to this field as well as expert users. Therefore, the system has two modes of operation.

For users new to this field of work we recommend the AUTO function

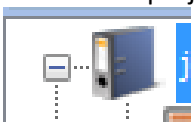
6.1.2 AUTO

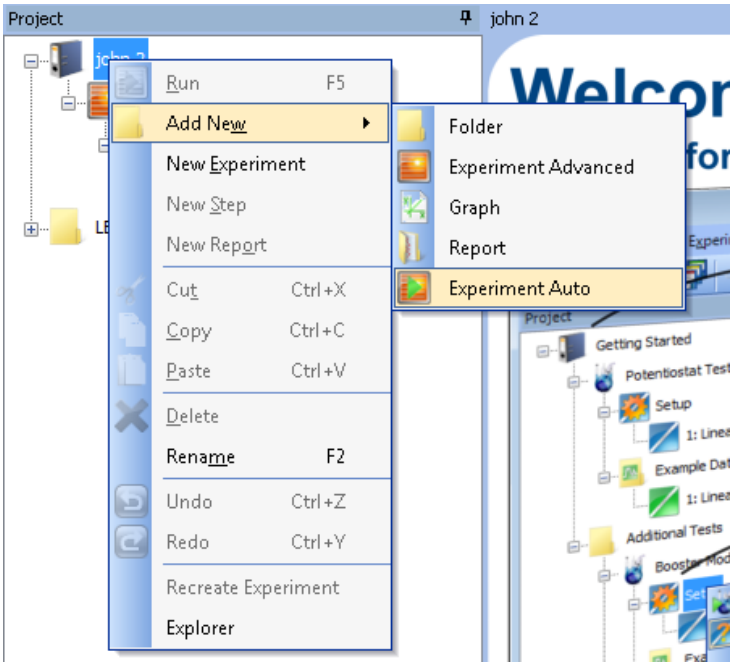
The features of this system include

AUTO generation of test results

AUTO analysis of the key data

Auto analysis of data is described in section Analysis and Auto-Analysis. The following section will describe how to start an AUTO experiment

- Once calibration is complete, click on the project tab
- Right click on the project icon 



- Click ‘Add New’
- Click ‘Experiment Auto’

The system will now run a complete set of DSSC experiments including IMVS, IMPS, EIS, Charge Extraction Dark and Light.

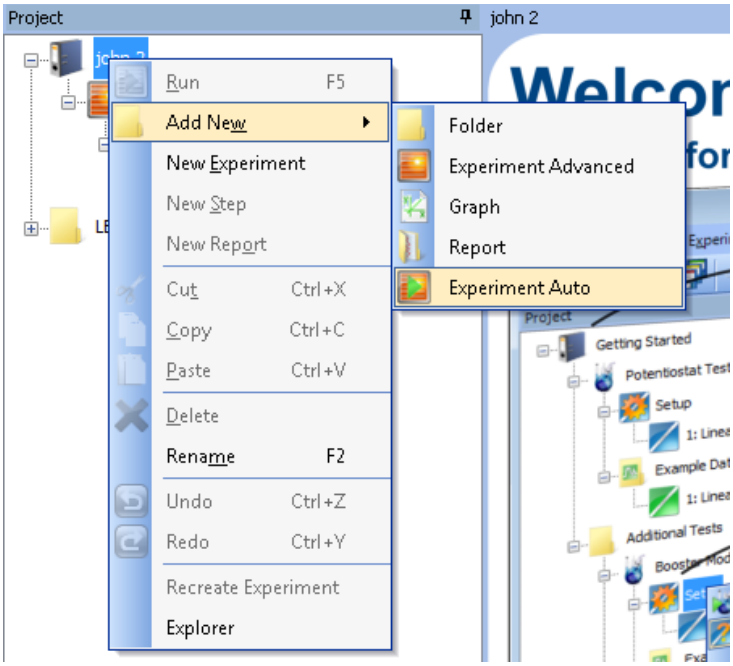
ATTENTION! Please ensure that the Optical bench is completely optically isolated from any external light. Use a suitable optical enclosure. Light pollution will affect results in particular the charge extraction techniques.

Using the AUTO step will NOT allow the user to change any of the pre-set levels. If you wish to change levels or perform only a few steps then you must chose the ADVANCED setting.

6.1.3 Advanced

In this modes, users can change any of the settings in each step type. These steps are described in the following sections.

- An advance experiment can be selected clicking the 'Experiment Advanced' setting as shown below. Follow the instructions for Auto setting to reveal this window



The following sections give details of the default settings for each step type.

6.2 Intensity ModuLab PhotoCurrent Spectroscopy

These measurements are used to determine the effective Diffusion Coefficient of electrons in the semiconductor layer.

The cell is held at short circuit and illuminated at a fixed wavelength of light. The diffusion coefficient (via measurements of short circuit current) changes as a function of light intensity. Therefore, measurements are made at a variety of bias illumination levels and the photo voltage is recorded.

The diffusion coefficient is determined by correlating the sine wave the short circuit ac photocurrent vs. the modulated intensity of light is measured as a function of frequency. The modulated light is generated by the FRA and is superimposed on the bias light. The level of the AC is light modulated signal is in the order of 10% of the DC Bias level. A schematic of the IMPS setup is shown in Figure 21: IMPS Schematic

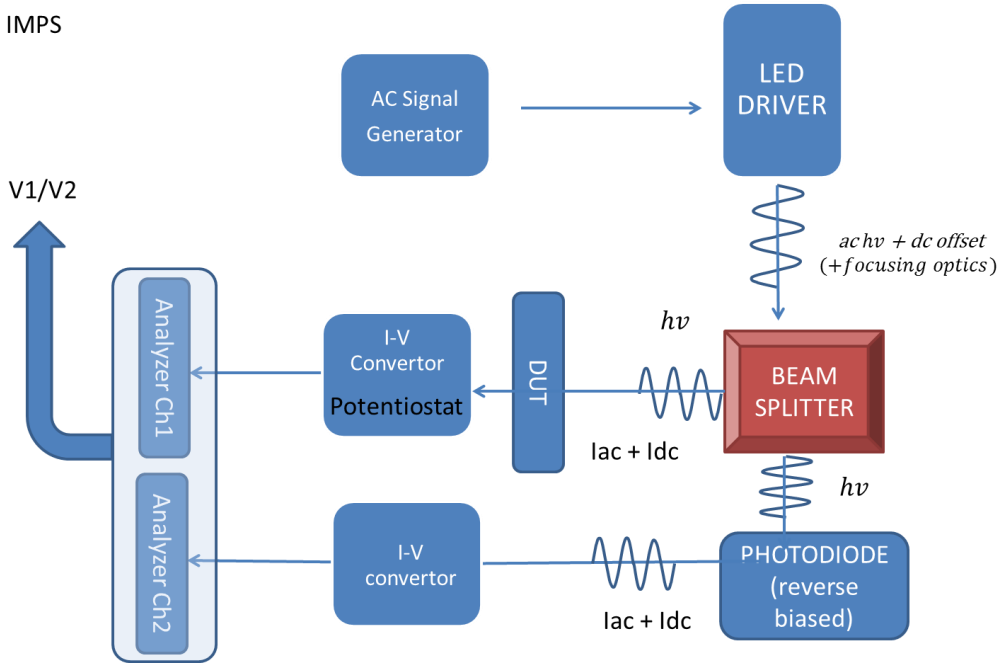
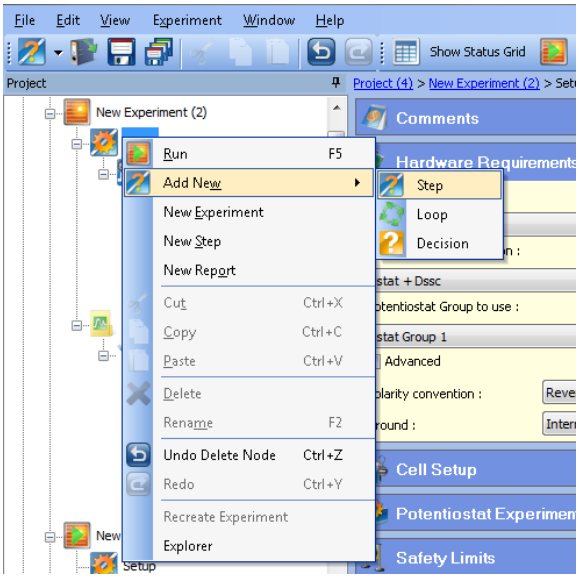
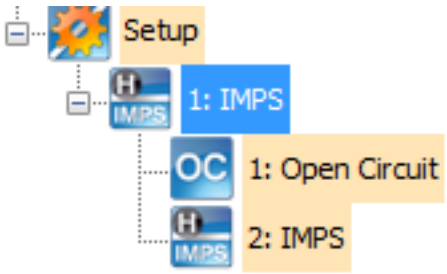


Figure 21: IMPS Schematic

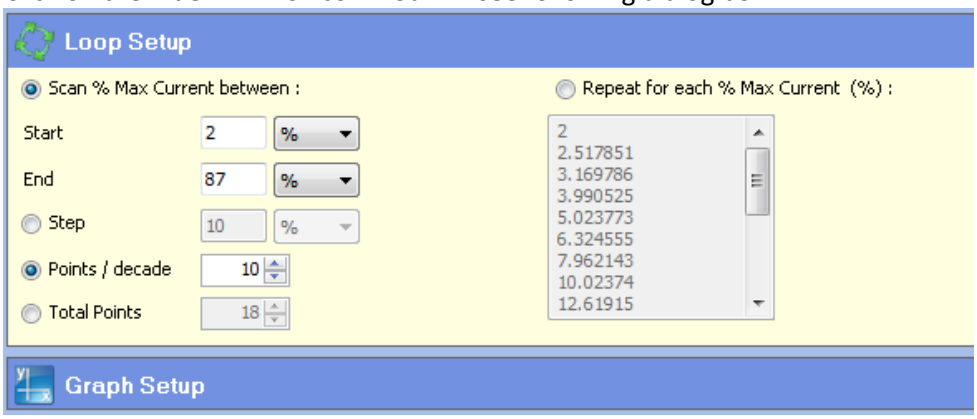
- To insert a new step , Right Click on the SET-UP ICON 

- Add New -- STEP
- 

When inserting a new IMPS step the following settings appear,



- Click on the Blue '1:IMPS' Icon. You will see following dialog box

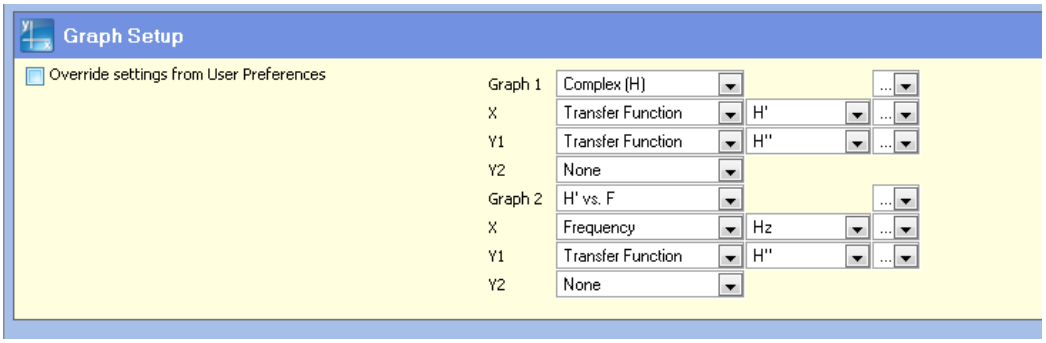


- Since the diffusion coefficient is determined as a function of light intensity, the IMPS step contains a Loop Set-up in which the BIAS light intensity is increased (or decreased) after each IMPS measurement. The end user does not need to manually change the light intensity for these measurements.
- 'Scan %Max Current between'
  In the Loop Setup dialog box, the user needs to enter the initial and final BIAS light intensity.
  The default settings used in software are suggested as starting and end values. In this example, the BIAS light intensity is scanned between 0.5% and 50% of the full power of the LED in use. The 0.5% setting is determined by the limits of the LED driver and as such we do not recommend setting this below 0.5%. The upper limit allows sufficient range for the addition of the AC signal. For example, At 50% DC Max Current Setting, the AC generator will apply 5% (10%) of the max power. The user can change the upper limits accordingly.

  The following four buttons determine the number of points (IMPS) measurements that will be performed. Please tick the preferred method
- 'Step'
  If the button is ticked, the experiment will increment the BIAS level by 10% after each step. For example, if the START % level is 2%, the next levels are 12% full power, 22% full power etc. Usually this button will NOT be ticked of used
- 'Points/Decade'. It is recommended that this is used as the default setting since the Effective Diffusion Coefficient exhibits a log relationship with the incident BIAS light intensity. The default setting is 10 points/decade
- 'Total Points' can be changed if the user is only interested in a few measurements and therefore will reduce total measurement time
- 'Repeat for each % Max current' should not be used unless the user wants to specifically change some of the applied levels.

- Each IMPS step contains an Open Circuit Step prior to the main measurement

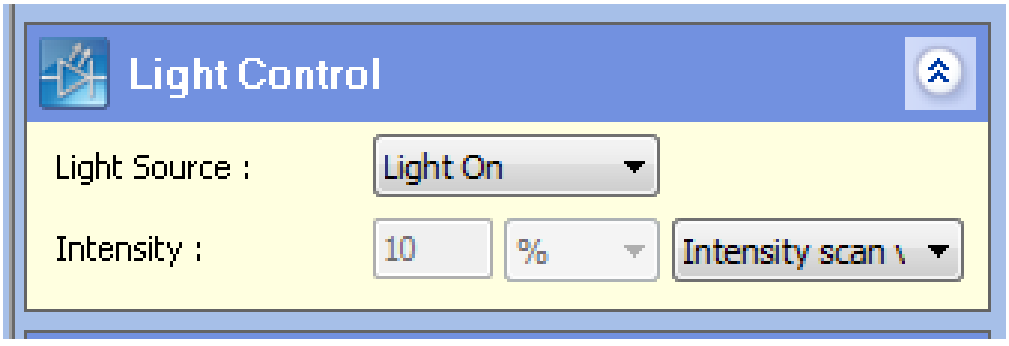
The default setting for the Graph Setup is as follows. It should not be necessary to change this.



Open Circuit

An open circuit step is included to allow the cell to attain steady state prior to the IMPS measurements. In addition, the SHORT CIRCUIT PhotoVoltage is recorded at the end of each step as this is required for analysis of data. It is recommended that you do not change these settings for IMPS measurement.

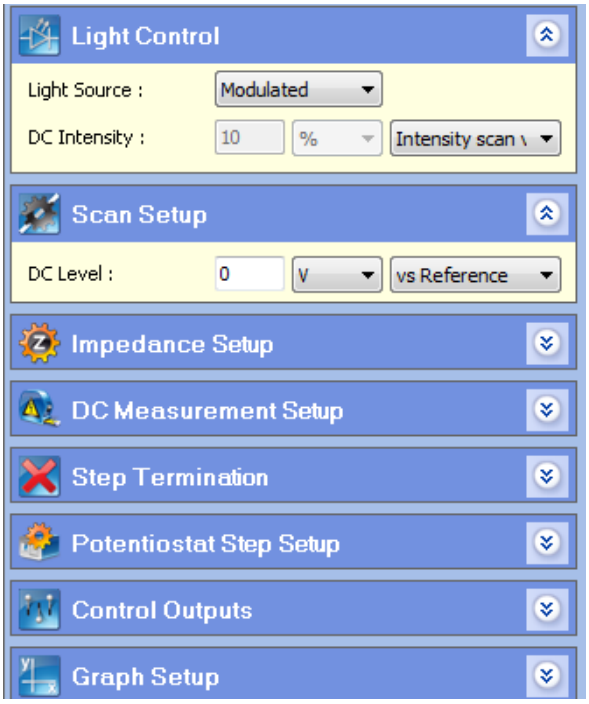
- Click on the 'Open Circuit' box
- The main dialog box is shown.
- The system loads the default settings. In this case are as follows. The Light Source is set to remain ON and the Intensity is set to remain at the Intensity Scan level.



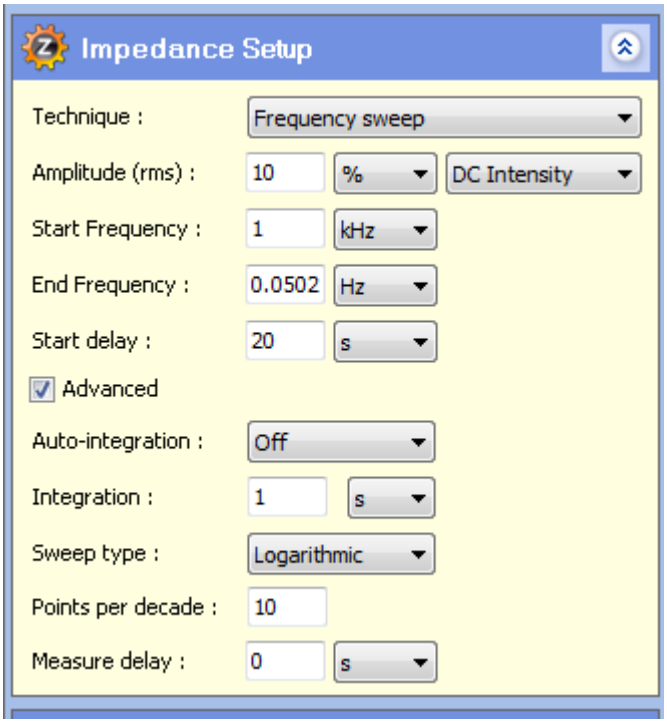
IMPS

The IMPS step contains the key control parameter for EACH individual IMPS measurement. Click on the IMPS icon to reveal the following Dialog box

- 'Light Control ' - do not change these settings
- 'Scan Set-up' . The DC level is set to 0V vs. Reference. This SHORT CIRCUITS the cell. Do not change these settings



- 'Impedance Set-up '- contains information about the frequency range, integration periods and delays to the start of the experiments



- **Technique –** please select Frequency Sweep. In addition you do have the choice of Multisine and Single Frequency measurements although these should only be used if you are confident in the analysis that you want to perform.

- **Amplitude (RMS) –** the default setting is 10% of the DC intensity. This ensures that the RMD value is the percentage of the Intensity Scan value determined in Scan-Setup dialog box. The RMS can be set to be a percentage of the max power of the LED, although we do not recommend this setting for inexperienced users.
- **Start and End Frequency** – this defines the max and min stimulation frequency of the measurement. The default range should be more than adequate for most cells. The max frequency is controlled by the LED generator and this is 100kHz.
- **Start Delay** – this determines the period in which the step begins BUT no measurements are taken. It ensure an additional stabilisation of the cell before measurements commence. The default setting is 20s and this can be changed if required
- **If the ADVANCED box is ticked** the used has the ability to change the integration period for each measurement. A detailed description of this functionality is contained in the ModuLab ECS manual and we encourage the end user to read through these sections.

To begin the experiments, click 'RUN' or press F5.

A typical set of IMPS responses are shown in Figure 22: Typical set of IMPS response as a function of Light Intensity

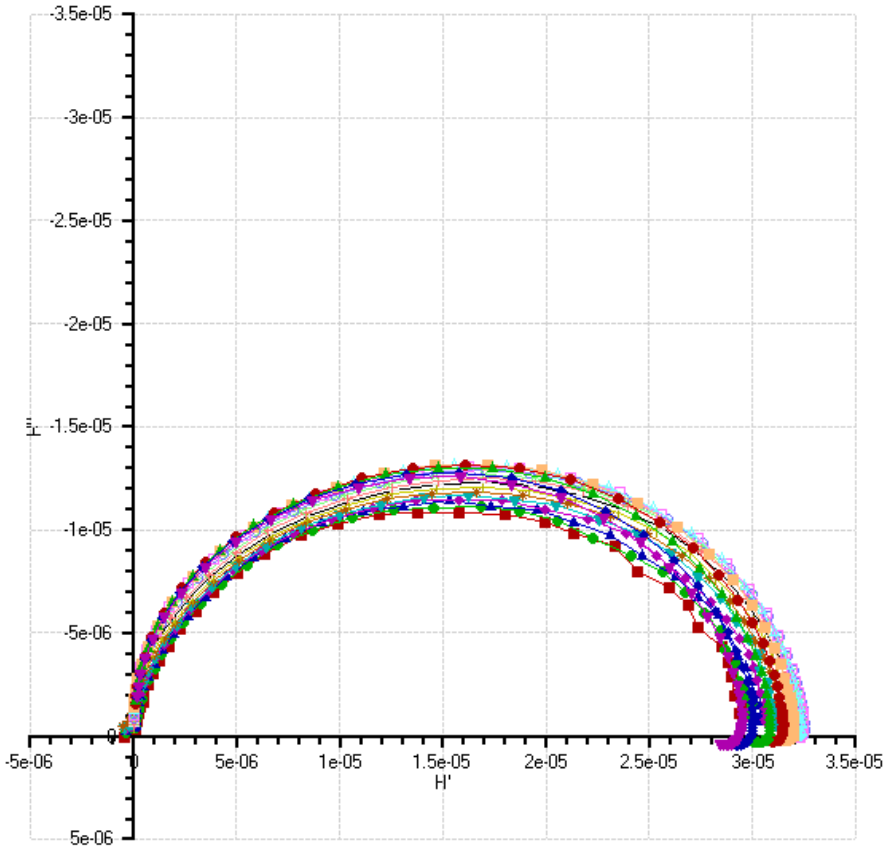


Figure 22: Typical set of IMPS response as a function of Light Intensity

6.3 Intensity Modulated Photovoltage Spectroscopy

These measurements are used to determine the effective Electron Lifetime. The cell is held at OPEN CIRCUIT and illuminated at a fixed wavelength of light. The Electron Lifetime (via measurements of open circuit) changes as a function of light intensity. Therefore, measurements are made at a variety of bias illumination levels and the photo voltage is recorded.

The electron lifetime is determined by correlating the sine wave of the open circuit ac photovoltage vs. the modulated intensity of light is measured as a function of frequency. The modulated light is generated by the FRA and is superimposed on the bias light. The level of the AC is light modulated signal is in the order of 10% of the DC Bias level. A schematic of the IMVS setup is shown in Figure 23: IMVS Schematic

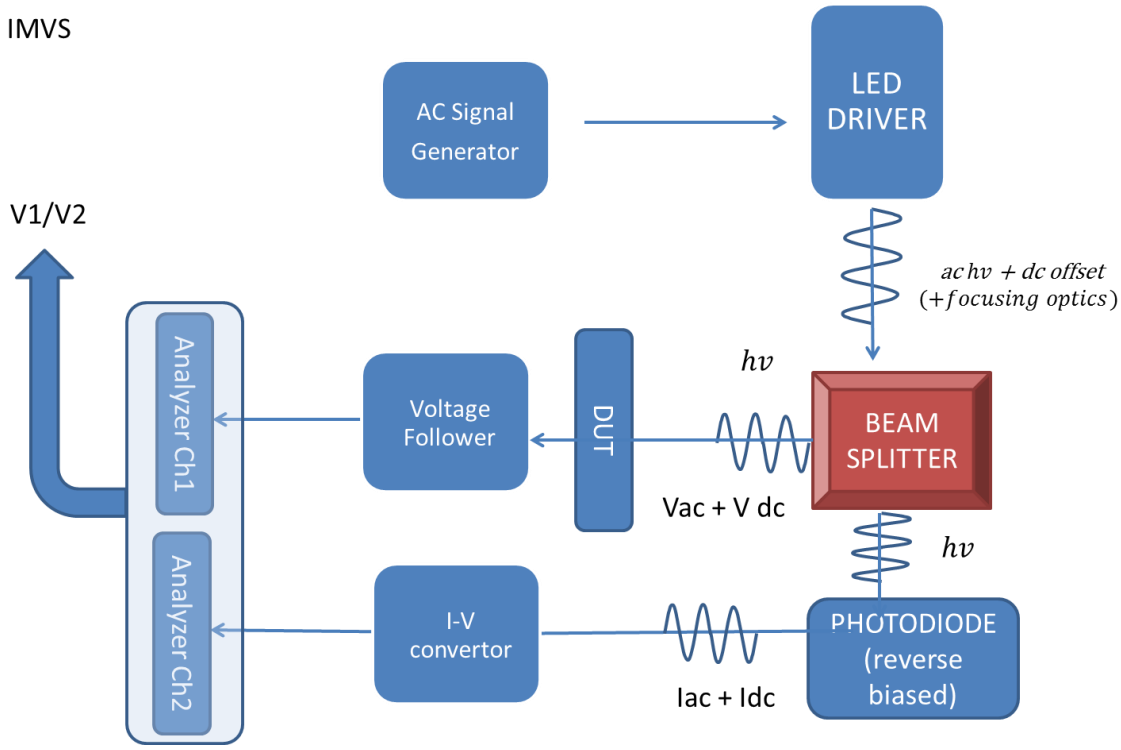
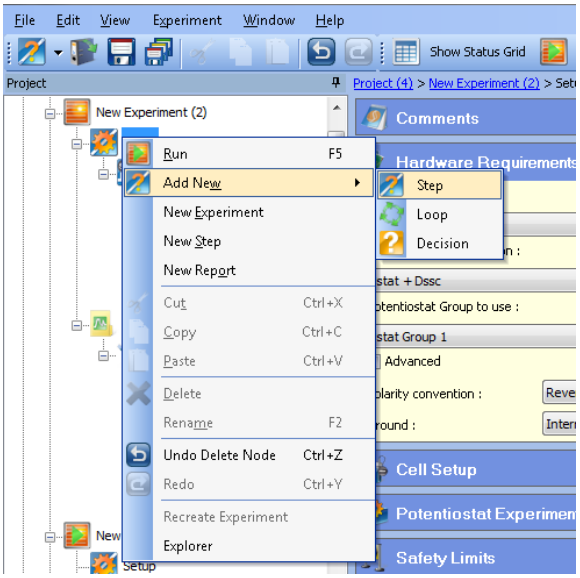


Figure 23: IMVS Schematic

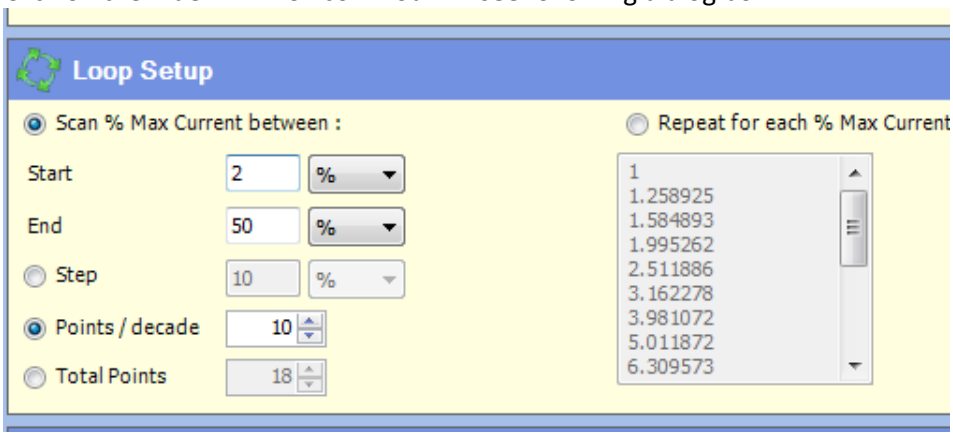
Insert a new IMVS step

- To insert a new step , Right Click on the SET-UP ICON 

- Add New -- STEP

- 

- Click on the Blue '1:IMVS' Icon. You will see following dialog box



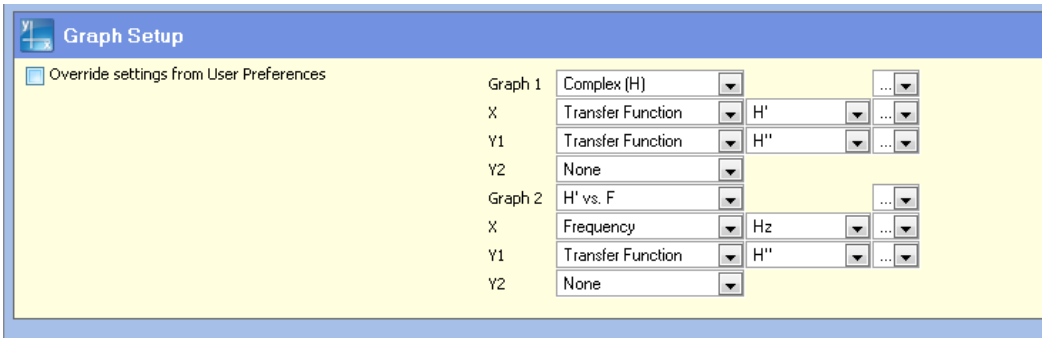
- Since the diffusion coefficient is determined as a function of light intensity, the IMPS step contains a Loop Set-up in which the BIAS light intensity is increased (or decreased) after each IMVS measurement. The end user does not need to manually change the light intensity for these measurements.
- 'Scan %Max Current between'
  In the Loop Setup dialog box, the user needs to enter the initial and final BIAS light intensity.
  The default settings used in software are suggested as starting and end values. In this example, the BIAS light intensity is scanned between 0.5% and 50% of the full power of the LED in use. The 0.5% setting is determined by the limits of the LED driver and as such we do not recommend setting this below 0.5%. The upper limit allows sufficient range for the addition of the AC signal. For example, At 50% DC Max Current Setting, the AC generator will apply 5% (10%) of the max power. The user can change the upper limits accordingly.

  The following four buttons determine the number of points (IMVS) measurements that will be performed. Please tick the preferred method
- 'Step'

If the button is ticked, the experiment will increment the BIAS level by 10% after each step. For example, if the START % level is 2%, the next levels are 12% full power, 22% full power etc. Usually this button will NOT be ticked .

- 'Points/Decade'. It is recommended that this is used as the default setting since the Effective Electron Lifetime exhibits a log relationship with the incident BIAS light intensity. The default setting is 10 points/decade
- 'Total Points' can be changed if the user is only interested in a few measurements and therefore will reduce total measurement time
- 'Repeat for each % Max current' should not be used unless the user wants to specifically change some of the applied levels.

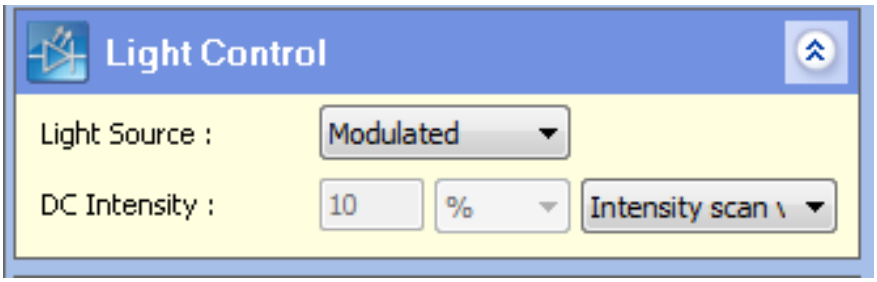
The default setting for the Graph Setup is as follows. It should not be necessary to change this.



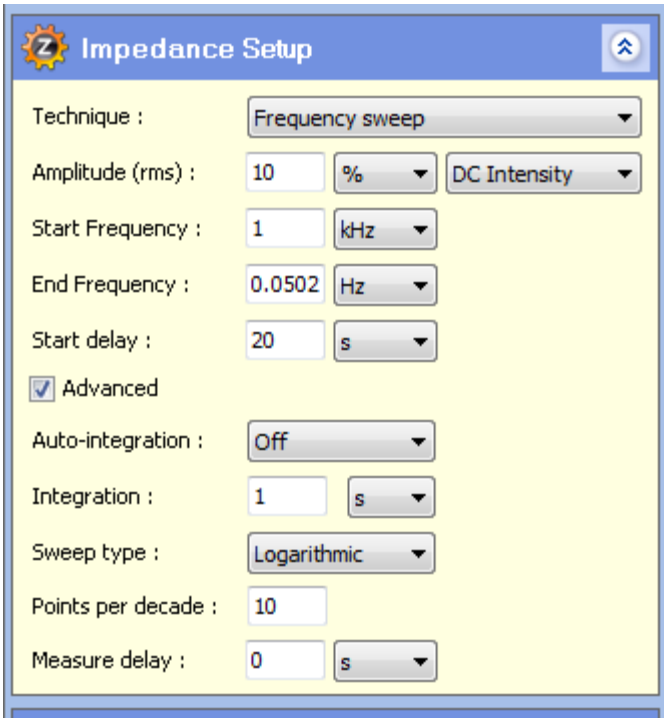
IMVS

The IMVS step contains the key control parameter for EACH individual IMVS measurement. Click on the IMPS icon to reveal the following Dialog box

- 'Light Control ' - do not change these settings



- 'Impedance Set-up '- contains information about the frequency range, integration periods and delays to the start of the experiments



- **Technique –** please select Frequency Sweep. In addition you do have the choice of Multisine and Single Frequency measurements although these should only be used if you are confident in the analysis that you want to perform.
- **Amplitude (RMS) –** the default setting is 10% of the DC intensity. This ensures that the RMD value is the percentage of the Intensity Scan value determined in Scan-Setup dialog box. The RMS can be set to be a percentage of the max power of the LED, although we do not recommend this setting for inexperienced users.
- **Start and End Frequency** – this defines the max and min stimulation frequency of the measurement. The default range should be more than adequate for most cells. The max frequency is controlled by the LED generator and this is 100kHz.
- **Start Delay** – this determines the period in which the step begins BUT no measurements are taken. It ensure an additional stabilisation of the cell before measurements commence. The default setting is 20s and this can be changed if required
- **If the ADVANCED box is ticked** the used has the ability to change the integration period for each measurement. A detailed description of this functionality is contained in the ModuLab ECS manual and we encourage the end user to read through these sections.

To begin the experiments, click ‘RUN’ or press F5.

A typical set of IMVS responses are shown in Figure 24: Typical Set of IMVS responses

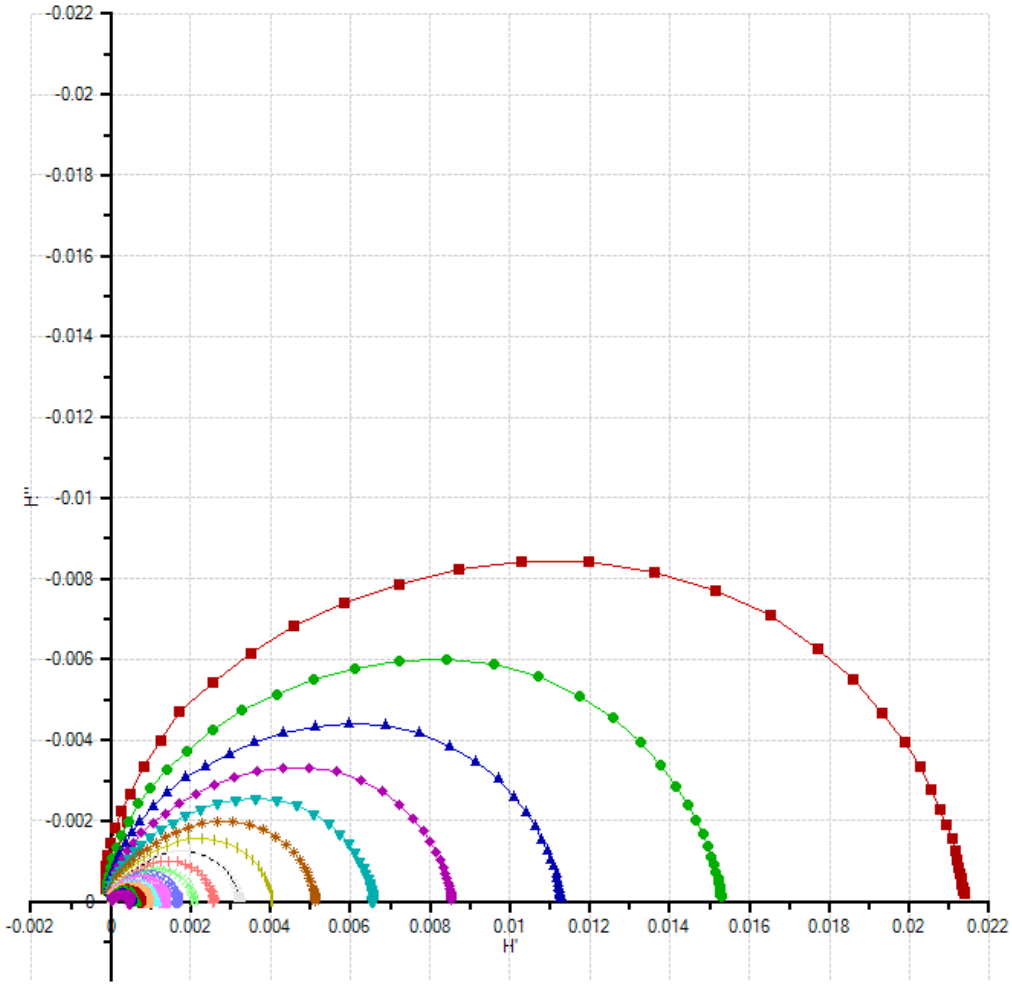


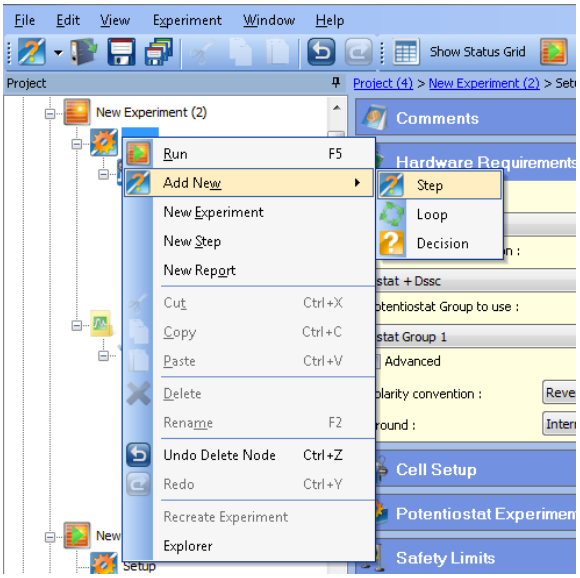
Figure 24: Typical Set of IMVS responses

6.4 EIS

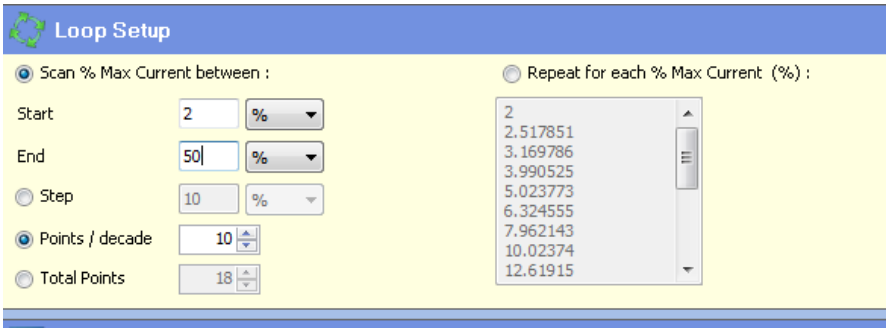
EIS measurements are usually performed at a variety of different light intensities. Therefore the loop Set-up used for both IMPS and IMVS measurements is used ad performs the same function. For further details please read the IMPS and IMVS sections (6.1) and(6.2).

- To insert a new step , Right Click on the SET-UP ICON 

- Add New -- STEP

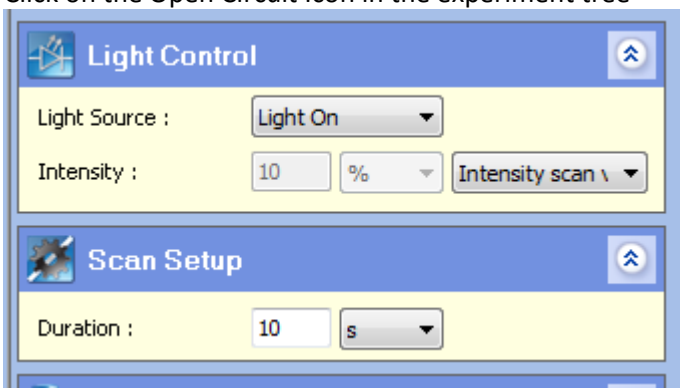
- 

In the EIS setting, click on the EIS i



- The default setting is 0.5% to 50% of full power

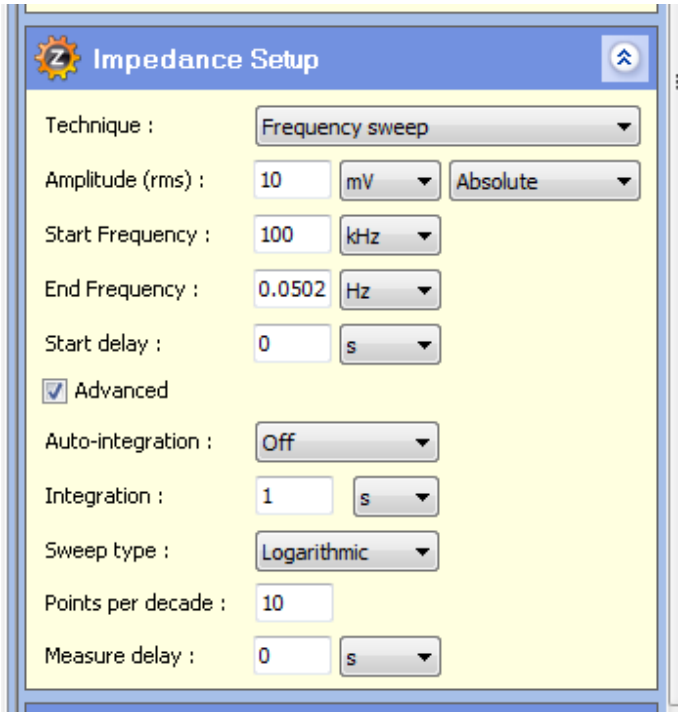
Open Circuit

- Click on the Open Circuit Icon in the experiment tree
- 

This step allows stabilisation of the cell AND records the open circuit of the cell when illuminated. It is recommended that these settings are kept.

Impedance Step

- Click on the Impedance Icon in the experiment tree. You will see the following experimental setup,



- **Technique –** please select Frequency Sweep. In addition you do have the choice of Multisine and Single Frequency measurements although these should only be used if you are confident in the analysis that you want to perform.
- **Amplitude (RMS) –** the default setting is 10mV absolute.
- **Start and End Frequency** – this defines the max and min stimulation frequency of the measurement. The default range should be more than adequate for most cells.
- **Start Delay** – this determines the period in which the step begins BUT no measurements are taken. It ensure an additional stabilisation of the cell before measurements commence. The default setting is 0s and this can be changed if required
- **If the ADVANCED box is ticked** the used has the ability to change the integration period for each measurement. A detailed description of this functionality is contained in the ModuLab ECS manual and we encourage the end user to read through these sections.

- To begin the experiments, click 'RUN' or press F5.

- A typical set of EIS responses are shown in Figure 24: Typical Set of IMVS responses

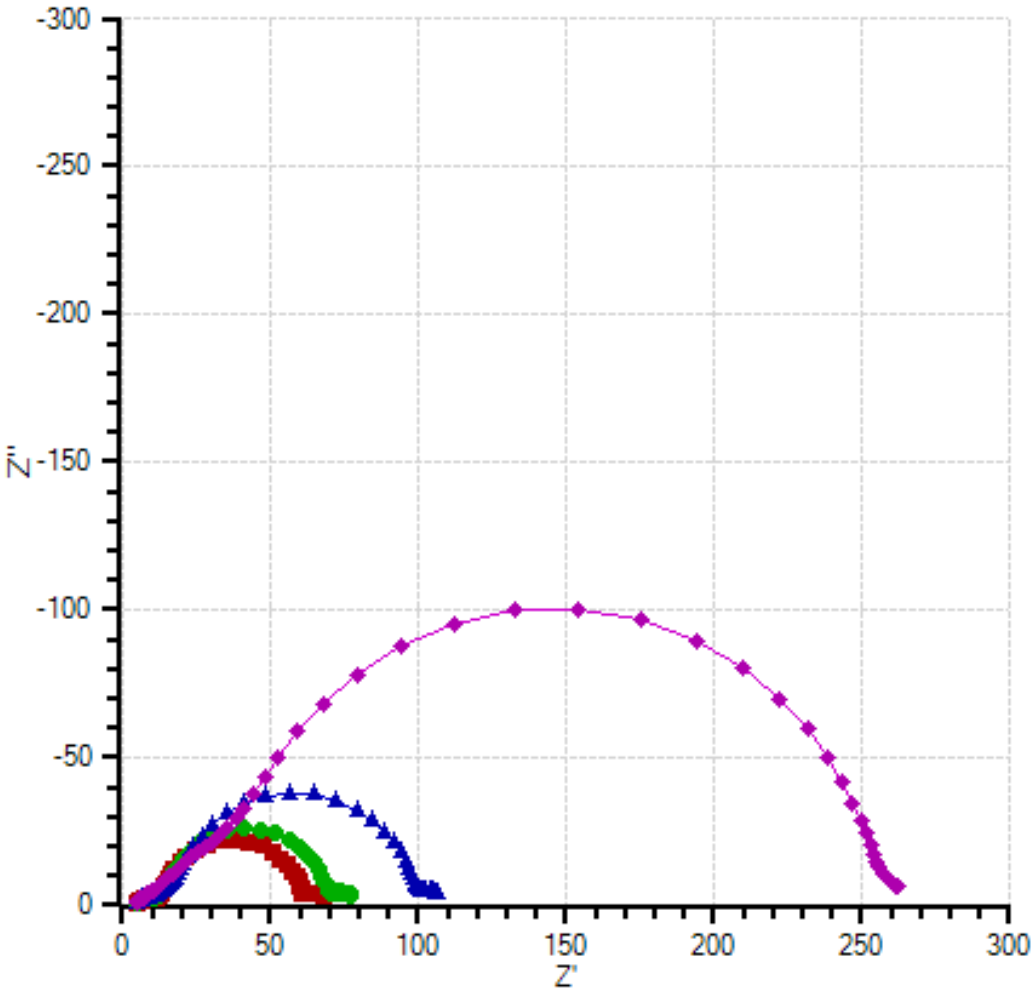


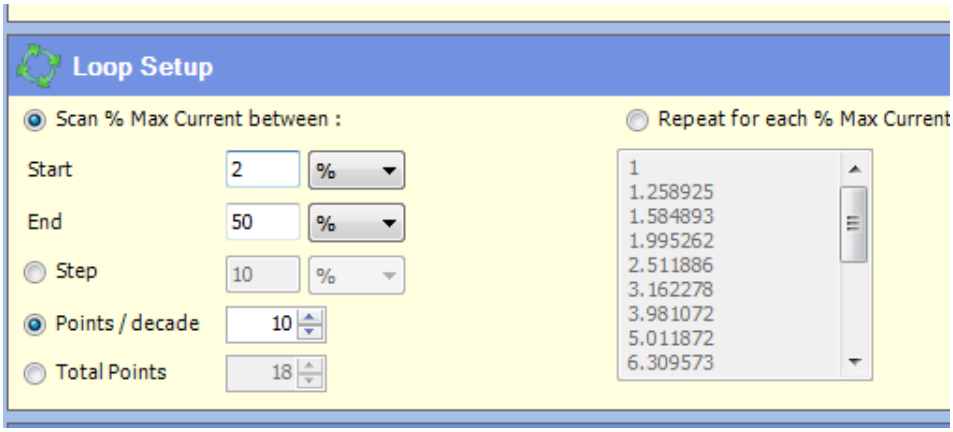
Figure 25: Typical EIS measurements as a function of Light Intensity

A key feature of the system is that it is designed for users NEW to this field and experienced users. A key component was the development of AUTO experiment and AUTO analysis features.

6.5 PhotoVoltage Decay

The technique was first described by Juan Bisqert and is used to determine the effective electron lifetime. This is a useful tool to compare with IMVS measurements. The cell is illuminated at open circuit at various intensities and after equilibration, illumination is interrupted and the Photovoltage decay is recorded.

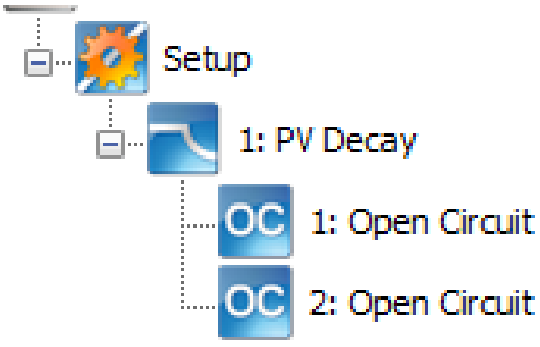
- To insert a Photovoltage decay test, click the photovoltage icon. You will see the following screen,

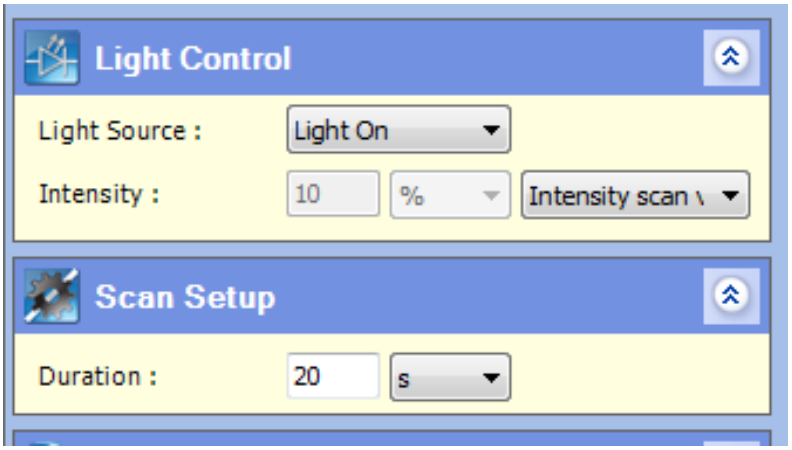


- 'Scan %Max Current between'
  In the Loop Setup dialog box, the user needs to enter the initial and final BIAS light intensity.
  The default settings used in software are suggested as starting and end values. In this example, the BIAS light intensity is scanned between 2% and 50% of the full power of the LED in use. The 2% setting is determined by the limits of the LED driver and as such we do not recommend setting this below 2%. The upper limit is set at 50% as this is the stability of the LED is excellent up 50% full power.

  The following four buttons determine the number of points PV measurements that will be performed. Please tick the preferred method
- 'Step'
  If the button is ticked, the experiment will increment the BIAS level by 10% after each step. For example, if the START % level is 2%, the next levels are 12% full power, 22% full power etc. Usually this button will NOT be ticked of used
- 'Points/Decade'. It is recommended that this is used as the default setting since the Effective Electron Lifetime exhibits a log relationship with the incident BIAS light intensity. The default setting is 10 points/decade
- 'Total Points' can be changed if the user is only interested in a few measurements and therefore will reduce total measurement time
- 'Repeat for each % Max current' should not be used unless the user wants to specifically change some of the applied levels.

The PV decay experiment contains two steps.





- The first Open Circuit Step applies the constant level BIAS light. The intensity of this light is determined by the Intensity Scan value and this was set in the Loop Setup section
- The duration of the illumination period is determined in the scan setup
- Please note that all experiments are recorded at open circuit



- The second Open Circuit step controls the decay step.
- The light is switched off and the decay is measured for two minutes
- To begin the experiments, click ‘RUN’ or press F5.

- A typical set of PV Decay responses are shown in Figure 26: Typical PV decay Curves

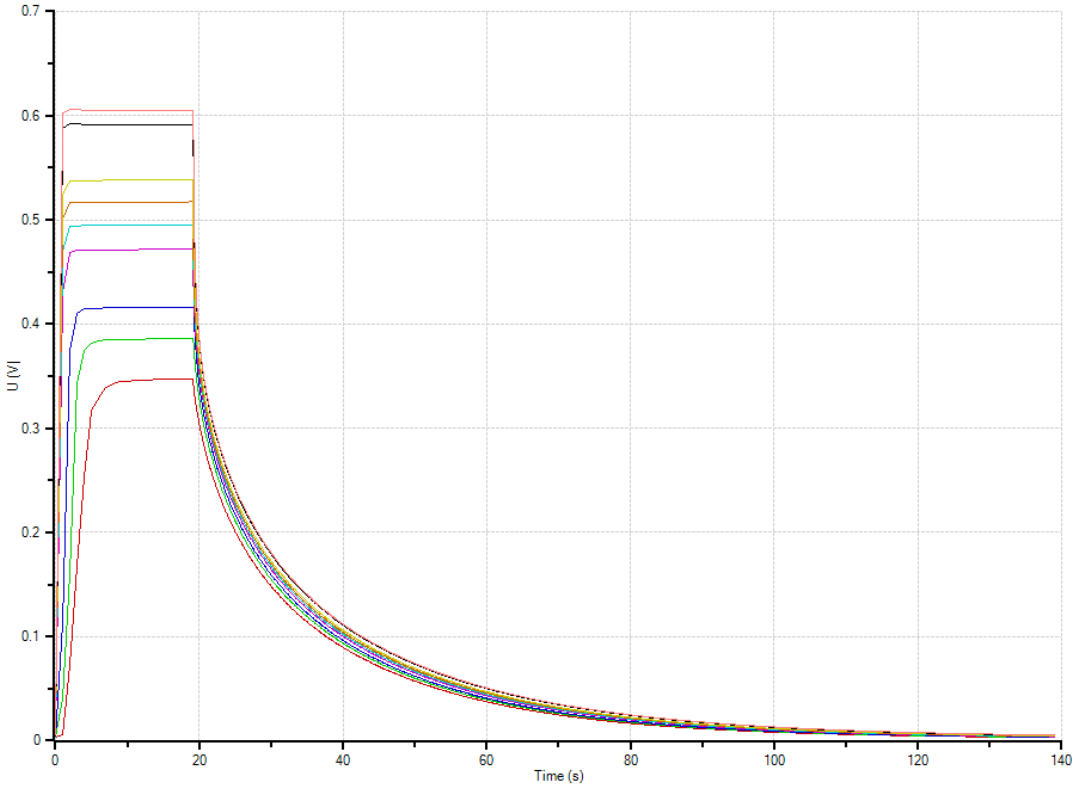
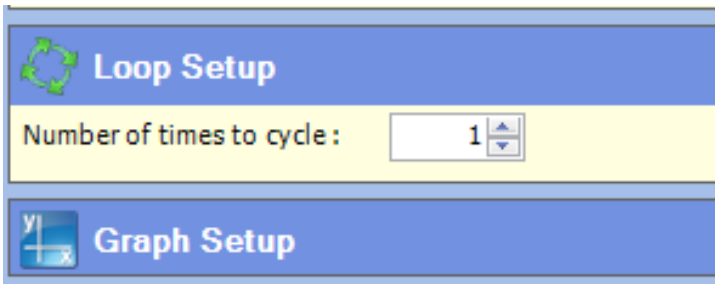


Figure 26: Typical PV decay Curves

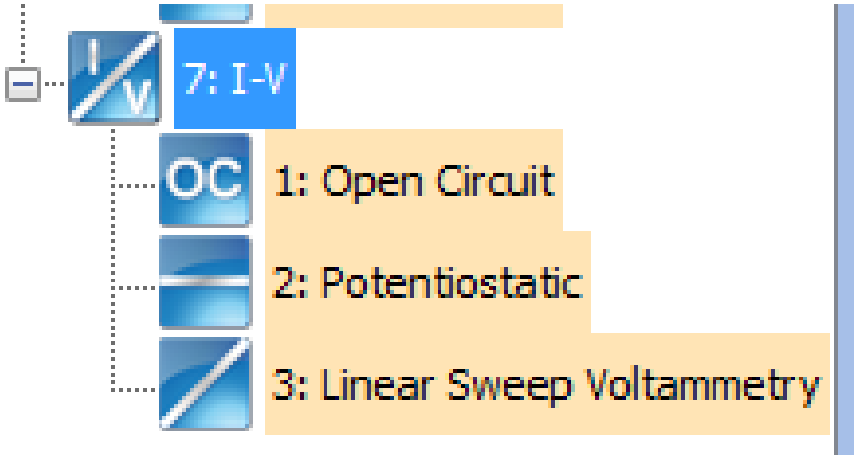
6.6 I-V Curves

By far the most common method used to test solar cells is to measure the I-V characteristics of the cell.

- Insert a new I-V step
- You will see the following screen,

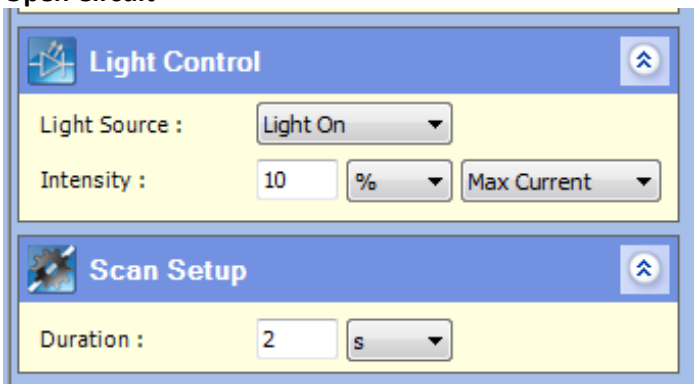


- It is sometimes useful to repeat the I-V characteristics to determine reproducibility. This step allows the user to specify the number of repeat steps. The default setting is one step.



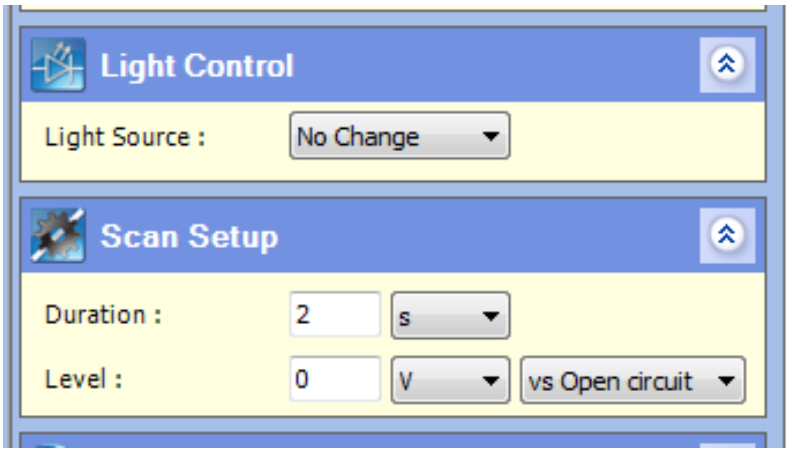
The I-V step consists of three stages

Open Circuit



- The cell is illuminated at open circuit. The default setting is 10% of the Max LED current.

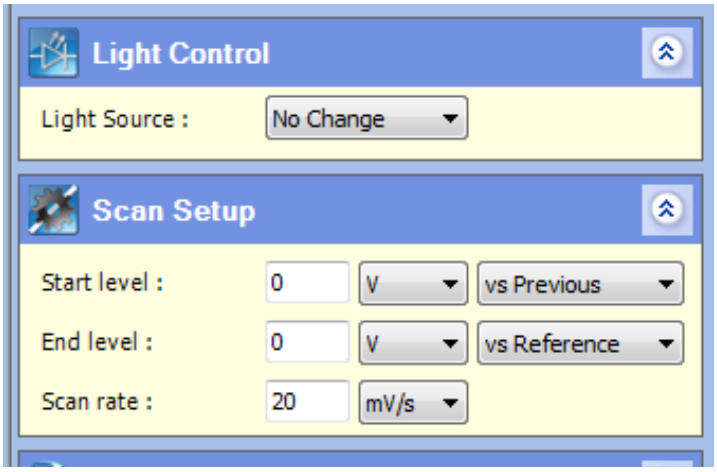
Potentiostatic



- This step allows the control loop of the potentiostat to achieve stability and enable the current measurement circuitry.

ATTENTION ! **IT IS HIGHLY RECOMMENDED THAT YOU DO NOT CHANGE THESE SETTINGS**

Linear Sweep Voltammetry



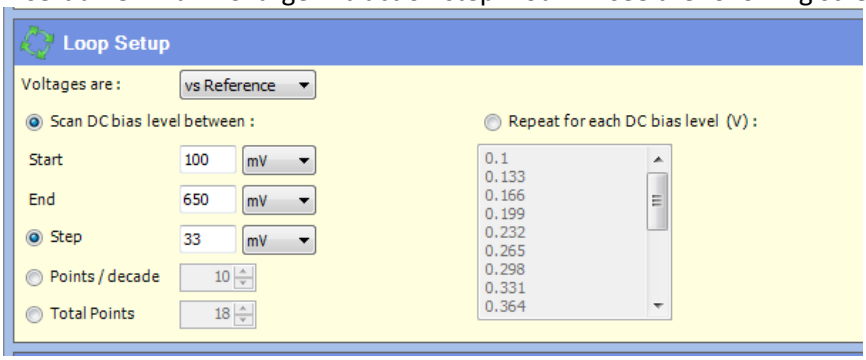
- The cell is swept from 0V vs the previous step (Open Circuit), to short circuit (0V vs. Previous). This step is the I-V measurement step.
- To begin the experiments, click 'RUN' or press F5.

6.7 Dark Charge Extraction

Charge extraction techniques were first developed by Prof. Laurie Peter to determine trapped charge density in the semi-conductor layers. The dark charge extraction technique determines the trapped charge by controlling the position of the quasi-Fermi level in the dark by using a potentiostat. The density of the trapped charge at any voltage is obtained by recording the short circuit current transient. For further details please refer to L.M.Peter. (2008). *Dye Sensitized Solar Cells.* Luasanne: EPFL Press and references within.

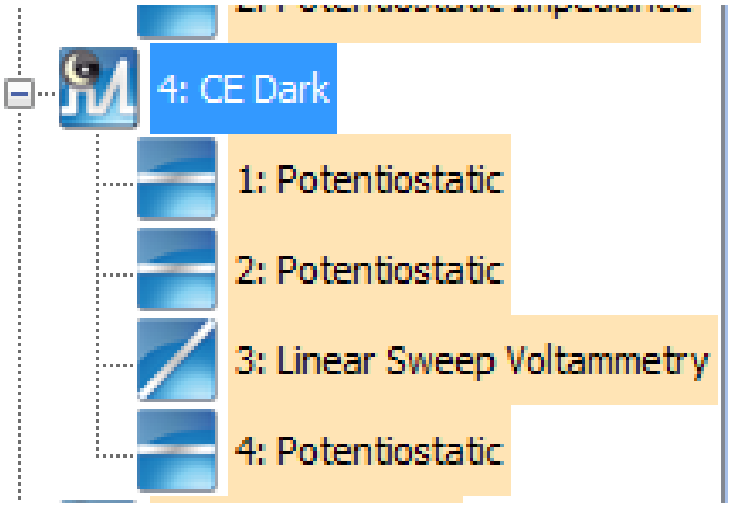
ATTENTION ! **IT IS CRITICAL THAT THESE MEASUREMENTS ARE PERFORMED IN THE DARK. ANY LIGHT POLLUTION WILL INVALIDATE THE RESULTS AND ANALYSIS**

- Insert a new Dark Charge Extraction step. You will see the following screen

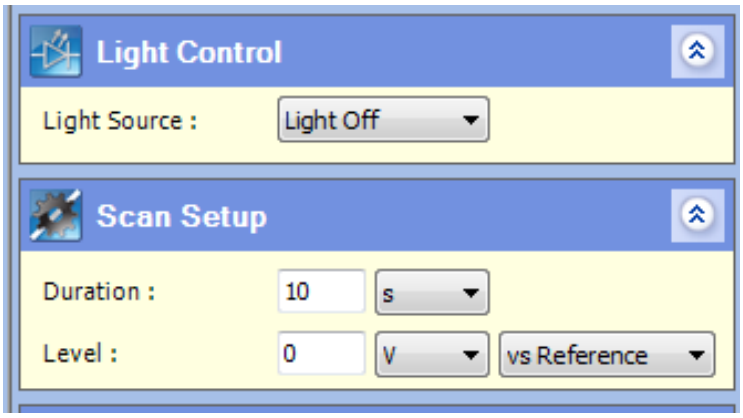


- The purpose of this box is to set the voltages that will be used for the charge extraction steps.
- 'Voltages are:' set to reference as default. It is recommended that the user does not change this setting
- 'Scan DC bias level between' – sets the lower and upper limits of the voltage for each charge extraction step. In this case, the lowest measurement is recorded at 100mV vs. reference and the upper limit is 650mV vs. Reference.
- 'Step' – these is the value in which the charge extraction steps increment. In the example shown, new charge extraction measurements are recorded at 33mV increments
- 'points per decade' the user may wish to specify the number of point per decade rather than a 'step' increment
- 'total points' the user may wish to specify the number of points rather than a 'step' increment

The Charge Extraction Step is made of 4 steps

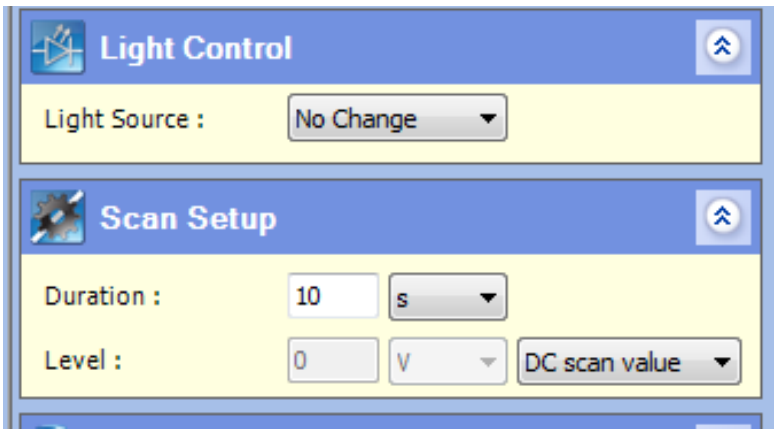


Potentiostatic



- This step short circuits the cell is used to remove any residual trapped charge. This does not form part of the measurement but should be considered as a pre-measurement conditioning step
- The light is ALWAYS off in this experiment
- The cell is held at 0V vs. ref (short circuit) to remove and residual trapped charge

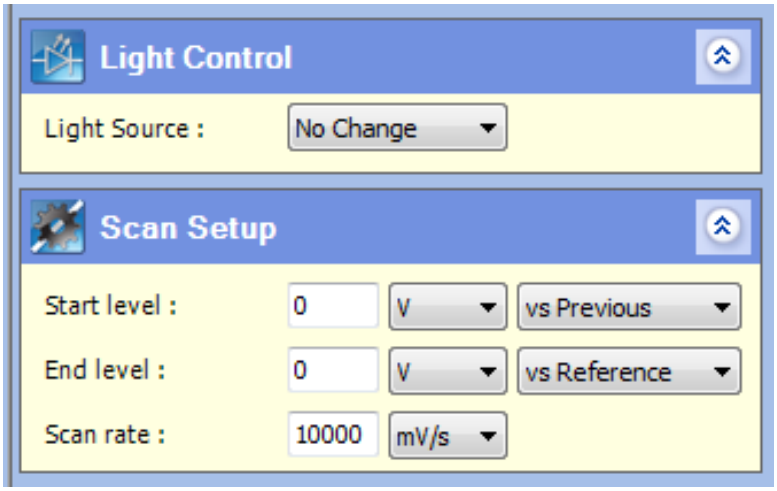
Potentiostatic



- This steps applies the voltage to the cell which is determined by the DC scan value. This value is set in the Loop Set-up section described earlier in this section

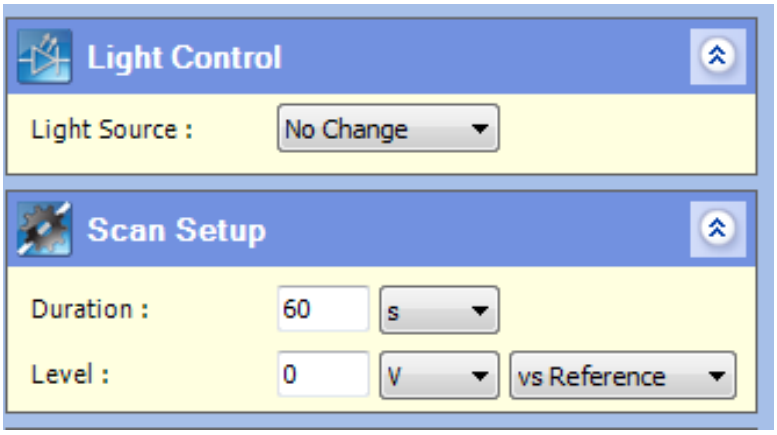
Linear Sweep Voltammetry

- This step sweep the voltage from the Previous Voltage level (determined in potentiostatic step) to short circuit. This step quickly removes the majority of the trapped charge



- It is recommended that users do not change the settings unless they are familiar with the technique

Potentiostatic



- This step holds the cell at short circuit for 60 seconds to remove all the remaining trapped charge. User may need to extend the step duration depending upon the rate of charge extraction. It may be necessary to extract charge in the order of 3 – 10 minutes.
- The results are automatically displayed as Q ext vs Time.

A typical set of Dark Charge Extraction Graphs are shown in Figure 27: Typical Dark Charge Extraction Measurements

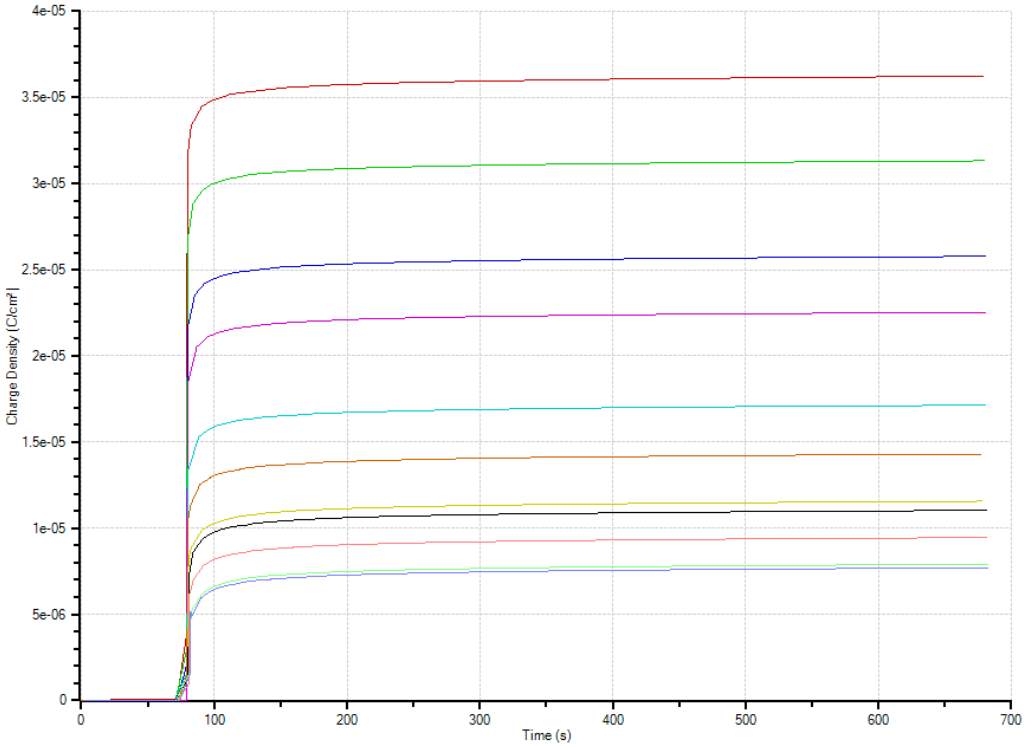


Figure 27: Typical Dark Charge Extraction Measurements

6.8 Short Circuit Charge Extraction

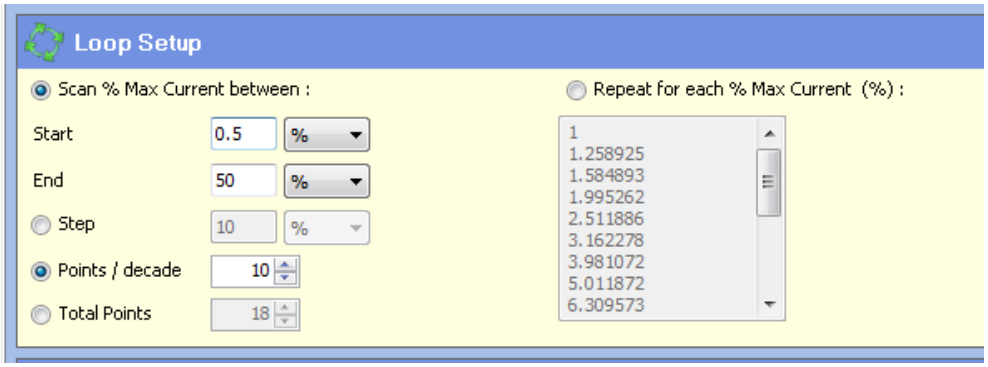
This is similar to the dark charge extraction technique with notable differences

i) the cell is held at short circuit
ii) the charge is measured after a period of illumination.

For further details please refer to L.M.Peter. (2008). *Dye Sensitized Solar Cells.* Luasanne: EPFL Press and references within.

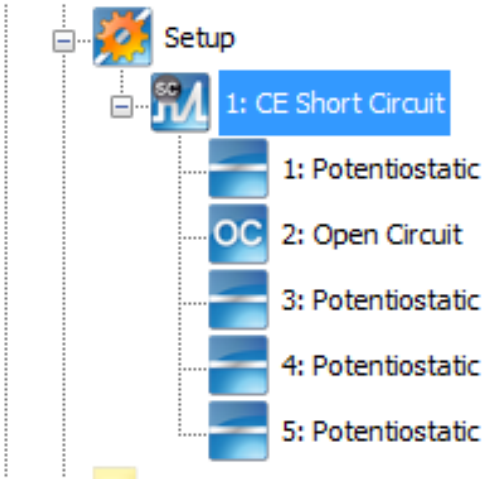
ATTENTION ! **IT IS CRITICAL THAT THESE MEASUREMENTS ARE PERFORMED IN THE DARK. ANY LIGHT POLLUTION WILL INVALIDATE THE RESULTS AND ANALYSIS**

- Insert a new Short Circuit Charge Extraction step. You will see the following screen



- The purpose of this box is to set the light intensities that will be used for the charge extraction steps.
- 'Scan DC bias level between' – sets the lower and upper limits of the voltage for each charge extraction step. In this case, the lowest measurement is recorded at 100mV vs. reference and the upper limit is 650mV vs. Reference.
- 'Step' – these is the value in which the charge extraction steps increment.
- 'points per decade' the user may wish to specify the number of point per decade rather than a 'step' increment
- 'total points' the user may wish to specify the number of points rather than a 'step' increment

The Charge Extraction is made of 5 steps;



- The first Potentiostatic step removes any residual charge from the cell
- The Open Circuit Step measures the Voltage of the cell under test during illumination
- The Potentiostatic Step holds the cell at SHORT CIRCUIT
- The subsequent two steps complete the charge extraction step. The light is switched off at this point and the trapped charge is recorded.

A typical set of Charge Extraction Plots are shown below in Figure 28: Short Circuit Charge Extraction

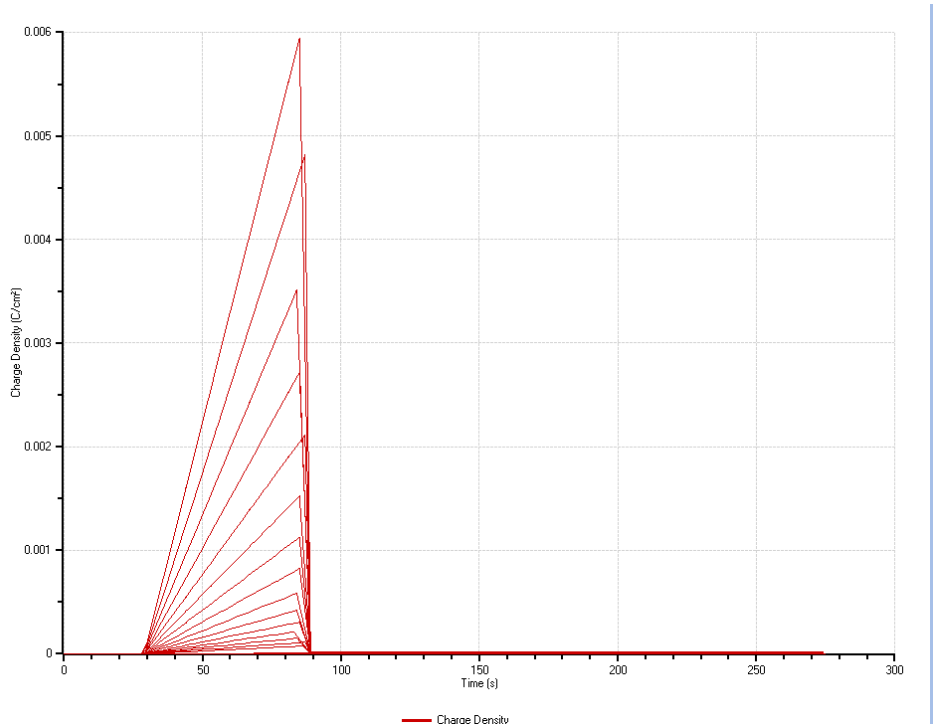


Figure 28: Short Circuit Charge Extraction

In order to view the extracted charge, highlight the LAST two steps in the experiment and drag and drop onto the graph page. (see Figure 29: Charge Extraction Steps

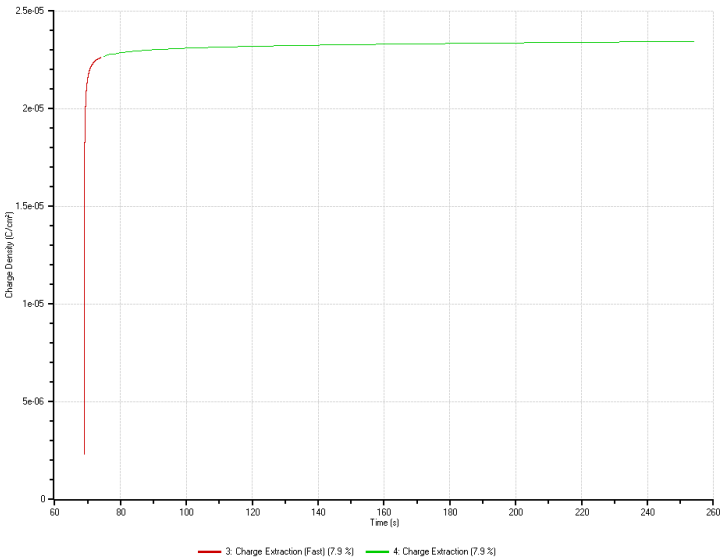


Figure 29: Charge Extraction Steps

6.9 Other Techniques with LED control enabled

All other Electrochemical techniques are offered with the ability to control an LED source. This will be useful for those researchers wishing to develop their own techniques. Details of the individual techniques are covered in depth in the ModuLab ECS manual.

7. Analysis and Auto-Analysis

7.1 Analysis Tools

ModuLab contains analysis tools that can be used for analysing the data. This section will focus on the analysis of I-V measurements and EIS data. The analysis of the other techniques can be performed automatically and is covered in the next section

7.1.1 I-V analysis

We have assumed that the user will perform I-V measurements using solar simulators. However, I-V measurement are possible with any light source including the LED provided with the bench.

- In order to analyse the data, select a file. Click on the third step ' Linear Sweep Voltammetry'
- Click on the FITTING tab in the and select I-V analysis
- Place two cursors on the graph as shown and press RUN

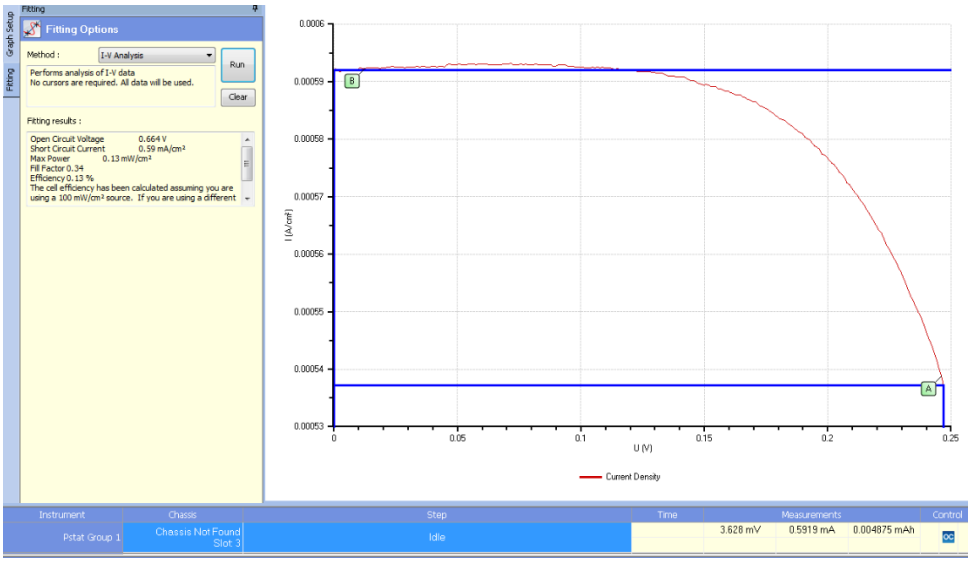


Figure 30: I-V analysis

The Text Dialog Box will display the following information:

Open Circuit Voltage 0.664 V

Short Circuit Current 0.59 mA/cm²

Max Power 0.13 mW/cm²

Fill Factor 0.34

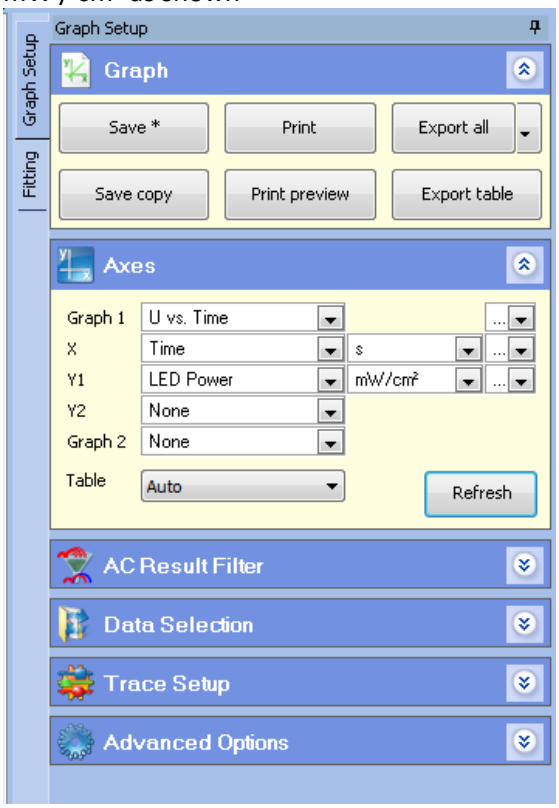
Efficiency 0.13 %

The cell efficiency has been calculated assuming you are using a 100 mW/cm² source. If you are using a different power source then you will need to recalculate the cell efficiency

If you are not using a 100mW cm^{-2} source, you will need to recalculate the cell efficiency (all other parameters are unaffected).

To apply the correction factor, determine the output of the of the LED or light source for the experiment in mW cm^{-2}. The LED output is recorded automatically and can be retrieved by following these steps,

- Select the 2nd step (potentiostatic)
- Plot U vs. Time
- In the X row, select TIME from the drop down list
- In the Y1 ROW, select the LED POWER from the drop down menu AND the units should be in mW / cm^2 as shown



- You will see an LED POWER response vs, Time as shown in Figure 31: LED POWER vs. Time.
- Read off the value of the LED POWER
- The correction factor is :

  100mW cm-2 / LED POWER (mW cm^{-2}) X Computed Cell Efficiency

- In the example shown, the cell efficiency was

  100 / 9.3 X 0.13 = 1.4%

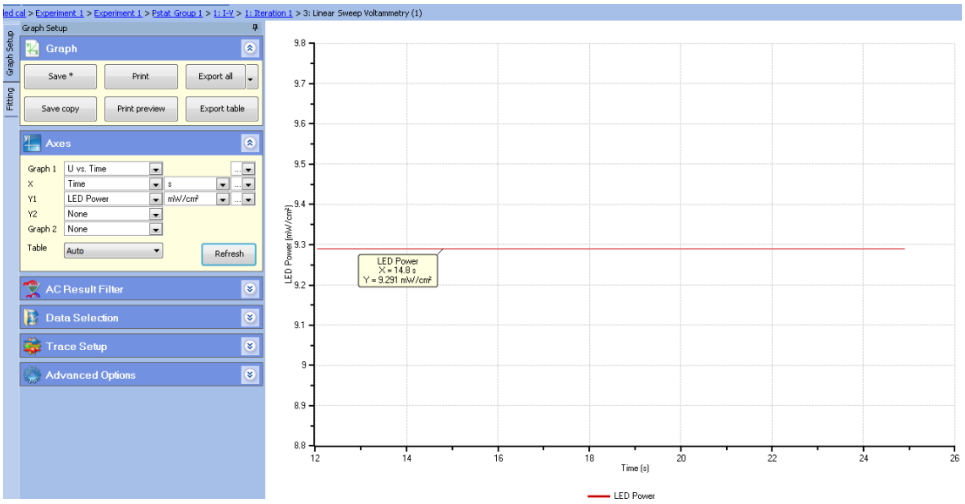


Figure 31: LED POWER vs. Time

7.1.2 EIS Analysis

It is recommended that customer analyse data in a third party package such as ZView since it contains pre-assembled equivalent circuit elements suitable for the analysis of DSSC's. Customers can shoes to analyse data in ModuLab but will be limited in the scope of the analysis.

7.2 Auto Analysis Tools

A key feature of the system is the ability to AUTO analyse much of the data. Auto analysis is applicable for

IMVS

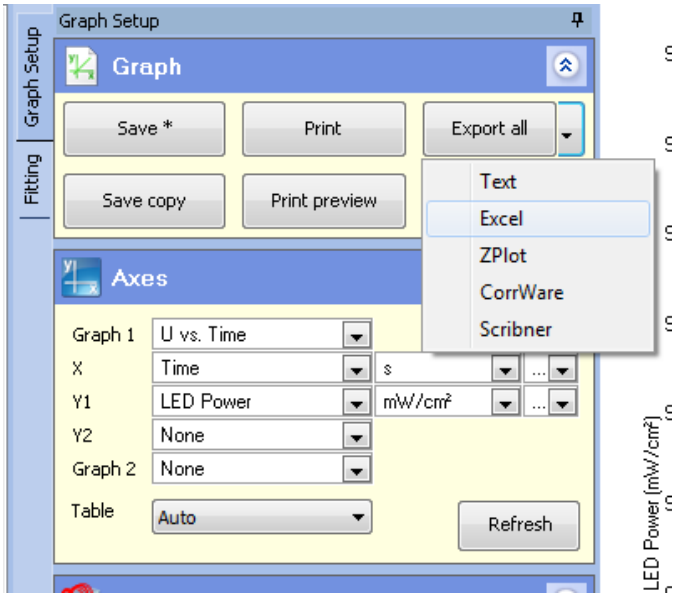
IMPS

PV Decay

Charge Extraction Techniques

In order to AUTO analyse data, first

- Select Experiment that you wish to analyse

- 

- In the Graph Setup box, select 'EXPORT ALL' and Click Excel
- An excel workbook will automatically load.
- The workbook will contains 6 tabs

  General, IMVS,IMPS, PV Decay, CE Short, CE Dark

  The General Column contains information pertaining to the cell under test. Much of this data will be automatically exported by ModuLab data

  The following fields are present,

  ATTENTION! Customers should pay particular attention to the comments section highlighted in RED

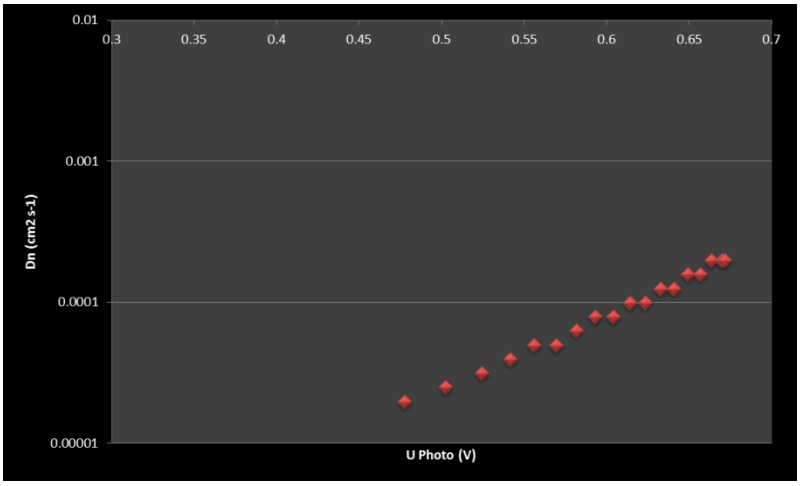
Field	Comment
Date/Time	Date of Experiment
Comment	LED Serial number or Comment in LED box
Calibration Date	
Cell Area (cm ^2)	Taken from Cell Set-up. We will assume an area of 1cm^2 if no value was entered
Film Thickness in cm	Taken from Cell Set-up. We will assume an a thickness of 1um if no value was entered
IMPS factor	The default value is 2.54. However the used should determine this for their cells. Details of this can be found in the Chapter 8 by L.M.Peter in 'Dye Sensitized Solar Cells, EPFL Press,

Series Resistance (Ohms)	The user should enter a series resistance value. This can be determined by examining the high frequency data in the EIS tests. The Series resistance is the point at which –Z" = 0 and Phase =0
1/Charge on Electron	Constant

If all of these values are entered, the system will automatically analyse the data and present it in the following formats

Step	Graph	Comment
IMPS	Dn vs U	Calculation of effective diffusion coefficient as a function of PhotoVoltage
IMVS	tn vs U	Calculation of effective diffusion coefficient as a function of PhotoVoltage
CE Dark	Charge vs U	Determination of Trapped Charge Density vs U
CE Light	Charge vs U	Determination of Trapped Charge Density vs U
PV DECAY	Charge vs U	Calculation of effective diffusion coefficient as a function of PhotoVoltage

A typical set of IMPS and IMVS analysis is shown in Figure 32: IMPS and IMVS analysis



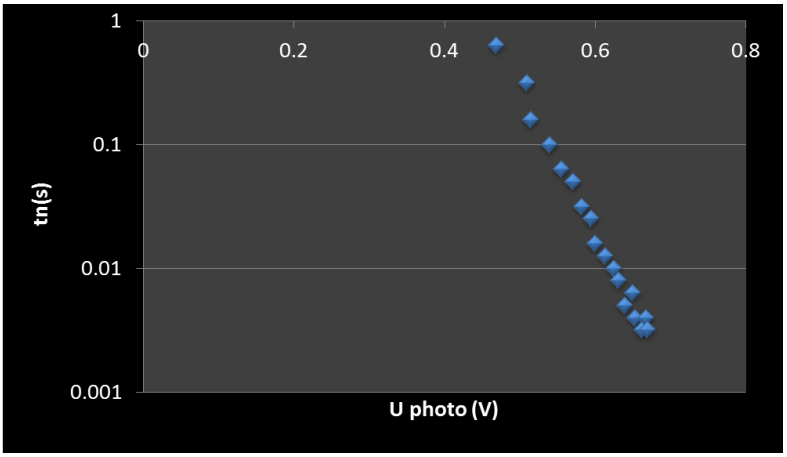


Figure 32: IMPS and IMVS analysis

8. Low Light Intensity Studies Using an ND Filter

The LED driver is calibrated to work down to 0.1% of full power. This represents three orders of magnitude change in intensity. However, the PhotoVoltage of a solar cell follows a log relationship with light intensity and changes by approximately 60-70mV per decade in light intensity. Therefore, in order to probe the characteristics of the cell at low light intensities, we have supplied a 0.01 Transmission ND filter. This reduces the incident intensity by a factor of 1000 and therefore extends the range of the optical bench to 6 orders of magnitude in light intensity. The following section describes the protocols to calibrate and use the ND filter.

8.1 Calibration with ND Filter

- Please ensure that the ND filter is clean and free from dust. Place the filter in the holder as shown in Figure 33: LED Optical Bench with ND filter in the holder

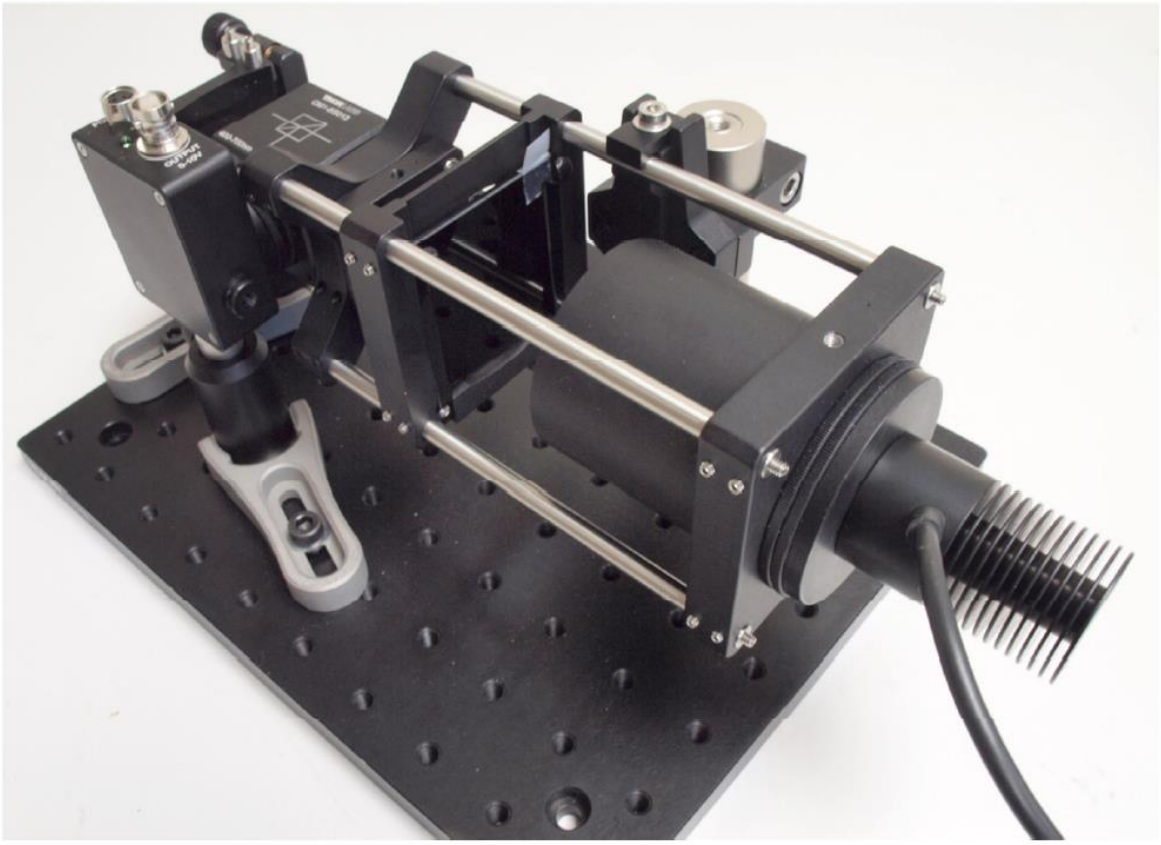
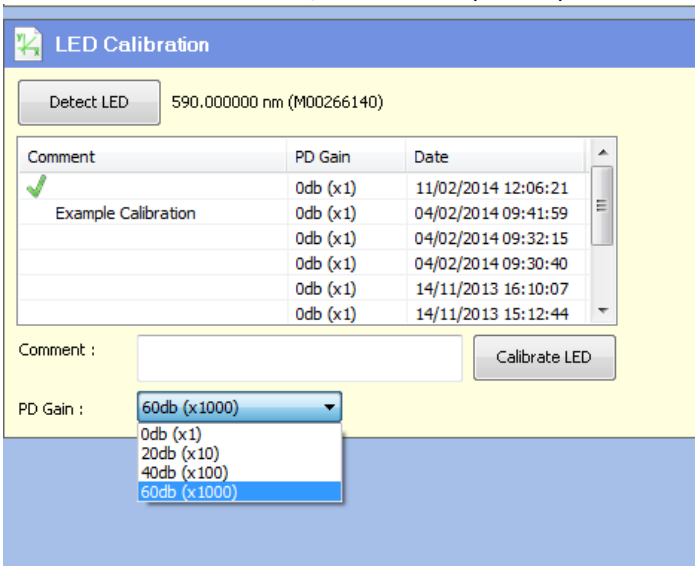


Figure 33: LED Optical Bench with ND filter in the holder

- Turn the wheel on the Fast Photodetector to position ‘60’. This is the 60dB setting

Please ensure that the measurements are performed in the DARK. ANY light pollution will severely affect results

- In the LED Calibration Box, select 60dB (x 1000) from the PD Gain drop down list



- Connect the system for the calibration routine as described earlier and press 'Calibrate LED' . Please refer to (Optical Bench Calibration Connections)
- It is advisable to enter a comment in the comments box

ATTENTION ! When using the ND filter calibration file ALWAYS remember to have the file TICKED in the LDE calibration box.

8.2 Measurements with ND Filter

All measurements can be performed with the ND filter. Please note that depending upon your cell, measurements at the lowest light intensity may produce scatter in the data.

9. Miscellaneous

9.1 Cleaning of Optical Components.

Where possible, the optical components should be free from dust and dirt. Please use the wipes provided to clean the bench. Do not use any abrasive cleaners as this will damage the optics.

9.2 Thermal Stability of the LED optics

The LED's have been designed for excellent thermal stability. Tests at our facility have shown that the systems are stable for many hours operating at full power

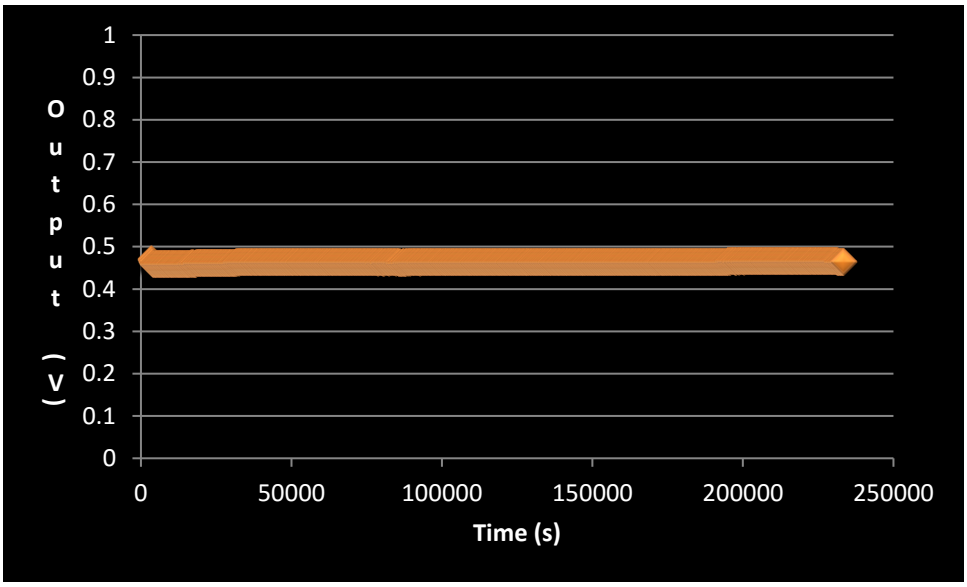


Figure 34: LED stability over 60 hrs at full power

For experiments requiring measurements above 50% of full power, it is advised to allow the LED to stabilise before commencing the measurement. Through extensive testing we have determined that the LED's attain stability after 25 mins continuous operation. Therefore, we advise that customers insert a start delay step prior to measurement in which the LED is powered to allow for equilibration.

10. System Specifications

10.1 Potentiotstat Card

Specification	Core
General	**Potentiostat**
Slots taken	1 slot
Cell connections	2, 3 or 4 terminal
Instrument Connections	CE, WE, RE, LO
Floating measurements	yes
Impedance measurement bandwidth	1MHz (via FRA)
Maximum ADC sample rate	1MS/s
Smooth scan generator	64MS/s interpolated and filtered
Maximum time record	Unlimited
DC scan rate (potentiostatic)	1.6MV/s to 1µV/s
DC scan rate (galvanostatic)	60kA/s to 200µA/s
Minimum pulse duration	1µs
IR compensation	yes
Counter Electrode (CE)	**Potentiostat**
Voltage polarization range	±8V
Current polarization range	±300mA
Maximum compliance (CE vs. LO)	±8V
Bandwidth (decade steps)	1MHz to 10Hz
Polarization V / I error (setting + range)	0.1% + 0.1%
Slew rate	>10V/µs
Reference Inputs (RE)	**Potentiostat**
Connections	Differential input
Cable shields	Grounded
Maximum voltage measurement	±8V
Ranges	8V to 3mV
Accuracy (reading % + range % + offset)	0.1%+0.05%+100µV
Maximum resolution	1µV
Input impedance	>100GΩ, <250pF
Input bias current	<10pA

Working Electrode (WE)	Potentiostat
Maximum current	±300mA
Ranges	300mA to 30nA
Accuracy (reading % + range % + offset)	0.1%+0.05%+30fA
Maximum resolution	1.5pA
Compliance voltage range (floating)	±8V
Auxiliary electrodes (A, B, C, D)	**Potentiostat**
Connections	4 (each differential)
Specification	Same as RE above***
DC Measurement	Synchronized to RE
Impedance measurement bandwidth	1 MHz (via FRA)

10.2 Frequency Response Analyzer

Specification	
Maximum sample rate	40 MS/s
Frequency Range - FRA 1MHz - FRA 300kHz	10 µHz to 1 MHz 10 µHz to 300 kHz
Frequency resolution	10 µHz (best)
Frequency error	±100 ppm
Minimum ∫ time per measurement (single sine, FFT or harmonic)	10 ms
Signal Output	
Waveform	Single sine, multi-sine
Single sine sweep	Linear / logarithmic
Multi-sine	All frequencies or selected frequencies
Analysis channels	
Accuracy (impedance)	±0.1%, ±0.1º
Anti-alias and digital filters	Automatic
Analysis channels	RE, WE, Aux A/B/C/D
Analysis modes:	Single sine, FFT, harmonic
DC Bias rejection	Automatic

10.3 Core Potentiostat Accuracy

This is the accuracy specification for the core potentiostat without the HV, femto ammeter, PhotoEchem and 2A options. The following conditions apply:

- 10mV AC stimulus
- Standard 2 meter cell connection cables
- 3 terminal connections above 1 kΩ impedance, 4 terminal for 1 kΩ and lower impedance
- Faraday cage for high impedance

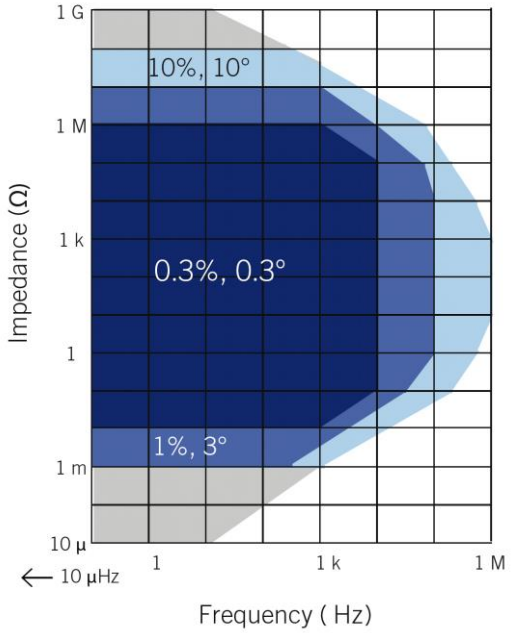


Figure 35: Impedance Contour Map

10.4 FRA Accuracy

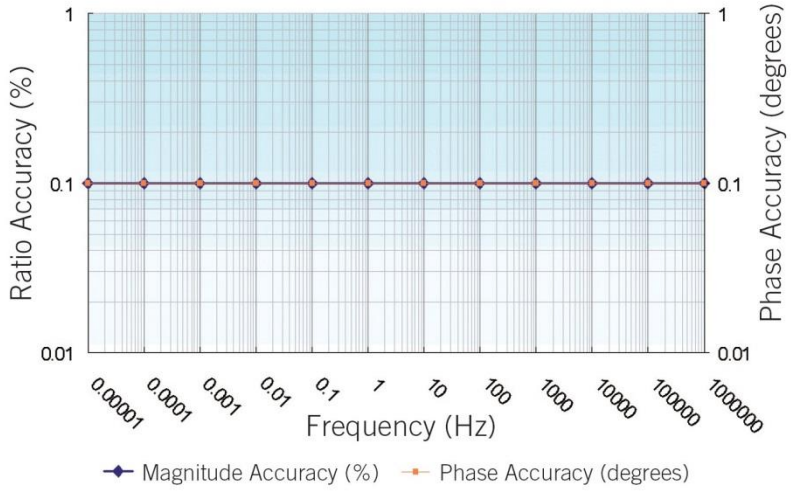


Figure 36: FRA accuracy map

10.5 Chassis Specification

General	
Chassis type	8 slot / 4 slot chassis
Potentiostats per chassis	8 maximum
Line Voltage	100V -120/ 220-240V ac to 264V, 50-60Hz
Power	600 (450) VA maximum
Dimensions w x h x d ( ) = 4 slot	450 (310) x 275 x 460 mm 17.7 (12.2) x 10.8 x 18.1 in
\ Weight	37 (21) kg - 82 (46) lb
Safety complies with:	BS EN 61010-1:2010
EMC complies with:	BS EN 61326-1:2013
Environmental	
Operating	5° to 40°C (41° to 104°F)
Specified Accuracy	10° to 30°C (50° to 86°F)
Storage	-25° to 70°C (-13° to 158°F)
IP Rating	None
Max Humidity	45% RH

10.6 PC requirements

Hardware / Software	Requirement
PC	Pentium IV or equivalent, 2GB RAM
Disk space	>1 Gbyte
PC Communications	Ethernet 100BaseT
Display monitor	17" or larger SVGA
Operating system	Windows 7 or 8


COOH
SCN
RU
SCN
SCN
COO
ModuLab XM PhotoEchem
solartron analytical
AMETEK

11. IPCE Module

The system can be upgraded to include an IPCE module. The following sections describe the use of the module. The system contains;

- Monochromator, lamp and power source
- 4 masks for the device under test
- Fibre Optic Cable and Holder
- White Light Bias Source and power supply.

11.1 TLS Module

The following section describes the use of the TLS module including

- Set-up of hardware
- Software installation

11.1.1 Monochromator Theory of Operation

The Solartron TLS sources are very compact and robust. The TLS light sources employ diffraction gratings to disperse the white light into its constituent colours. A diffraction grating based light source allows precise selection of wavelengths for measuring the absorption and reflection of materials. This is an important feature when studying the chemical and physical properties of materials.

The TLS source is an integrated package that includes a white light source and a monochromator to disperse and select the wavelengths.

The output from a 20 W lamp is collected by a lens that focuses the intense image of the filament on the entrance slit of the monochromator. After the light enters the monochromator the grating diffracts the light into its full spectrum of colours. By turning the grating selected band passes of light can leave the monochromator through an exit slit, or, more frequently, an optical fibre.

The amount of light that exits is determined by the characteristics of the grating and the widths of the entrance and exit slits or fibre optic. The TLS models are differentiated by the band pass of light that exits the monochromator through a 600 micron wide slit or fibre optic diameter. The grating in the TLS will allow 10 nm of light to pass through the slit or fibre optic cable

11.1.2 Setup and Operation of TLS Light Sources

A Schematic diagram of the IPCE hardware connections is shown in Figure 37: IPCE hardware connections

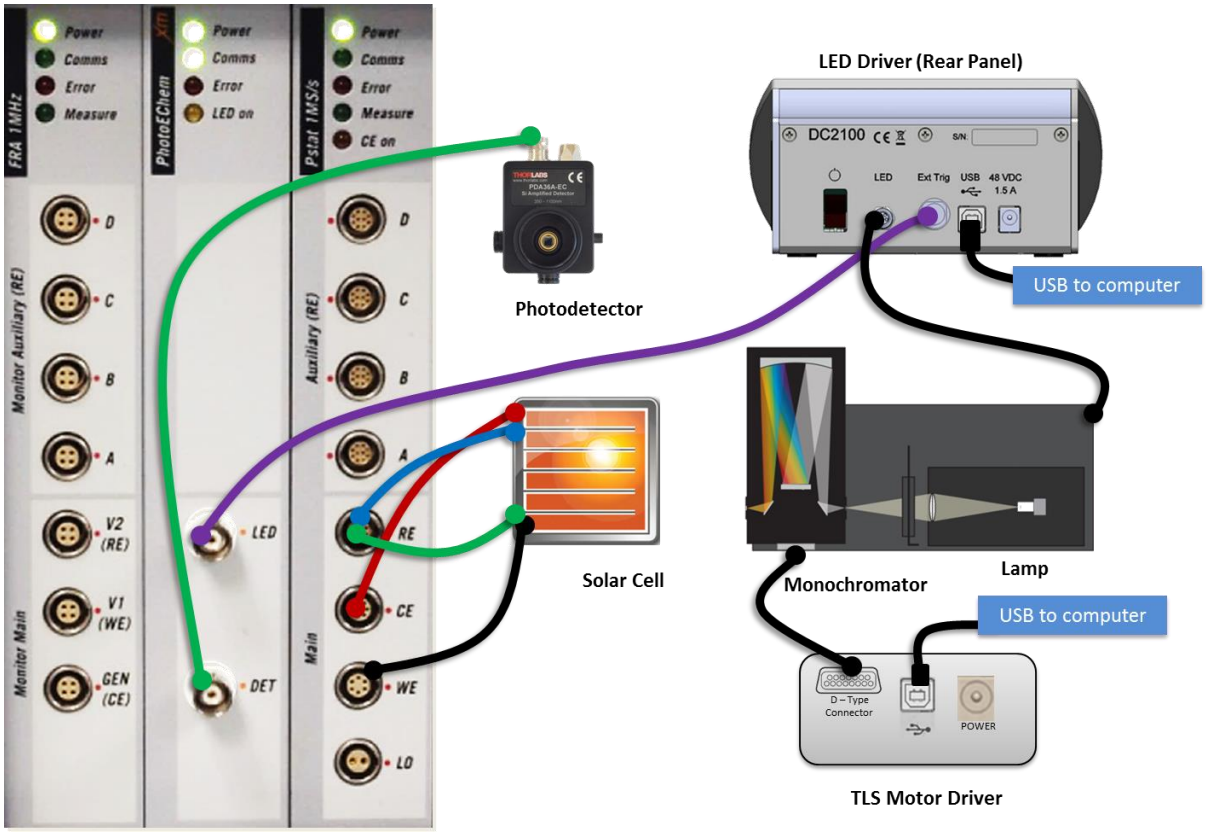


Figure 37: IPCE hardware connections

All TLS light sources come preassembled and optically aligned. They are packaged with one 600 micron slit installed on the monochromator. The fibre optic cable should be inserted into the exit side of the slit as shown in Figure 38: Fibre optic cable coupling to monochromator.



Figure 38: Fibre optic cable coupling to monochromator

Plug the power supply into the wall socket and into the lamp. The dial on the front of the monochromator is used to select the wavelengths exiting the monochromator. The counter indicates the central wavelength in nanometers.

11.1.3 Setup and Operation of Computer Controlled TLS Light Sources

The computer controlled TLS light sources include an interface box which contains a power supply for the monochromator and the USB interface between the source and computer. In addition, the lamp is powered by the Thorlabs LED driver (DC2100 or DC2200). Before connecting all of the cables, turn the knob on the monochromator until the counter reads 0000. Once the monochromator is under power it is not possible to turn the knob.

In order to provide power to the lamp, connect the LED drive to the lamp using the cable as shown in Figure 39: Power connection to light source The 4 pin plug should be inserted into the rear panel of the LED driver at the port labelled LED. (see Figure 6: LED Driver set-up)



Figure 39: Power connection to light source

The fibre optic cable is connected to the Optical Bench by means of a tube adaptor (see Figure 40: Fibre optic cable holder). Screw the



Figure 40: Fibre optic cable holder.

If an LED is screwed into the Optical Bench, remove by turning counter clock-wise. Screw the adaptor with the aluminium rod inserted into the main Lens housing of the optical bench (right hand side of Figure 40: Fibre optic cable holder.into the LED les housing until finger tight. Insert the end of the fibre optic cable into the adaptor until it will move no further. Tighten the screw on the adaptor to hole the fibre optic cable in place.

The hardware installation of the TLS is now complete

11.1.4 Installing the TLS driver software

ATTENTION ! Do not plug the USB cable from the monochromator power supply into the computer before installing the drivers

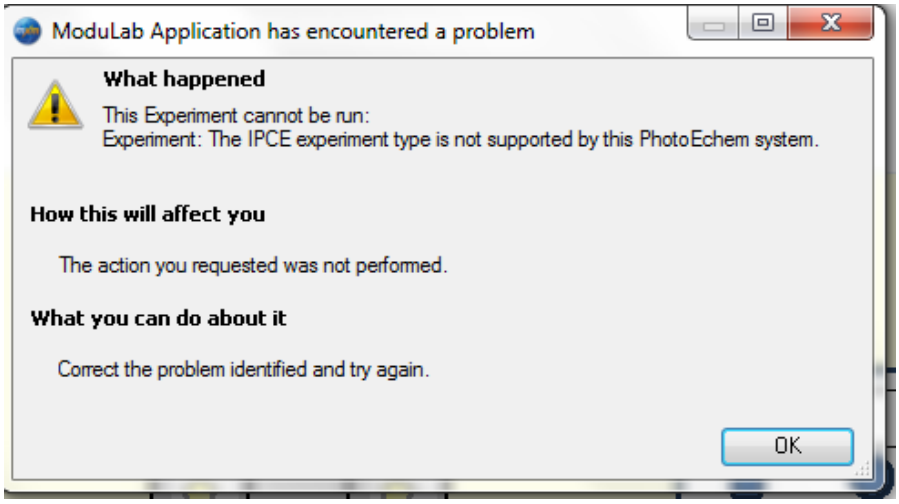
- Insert the ModuLab CD into the appropriate drive
- Search for the Folder labelled TLS – 10
- Run the setup.exe file and follow the on screen instructions
- Please note that the drivers for the Thorlabs DC2100 or DC2200 must be installed prior to this installation

11.1.5 Bulb Replacement

The 20 W tungsten halogen bulb has a lifetime of about 200 hours when used for IPCE measurements. The replacement part number**, (234486)**, can be ordered through the sales department at Solartron Analytical. Disconnect the lamp from the power supply before replacing the bulb. Unscrew the 5 hex nuts circled in the photo. Remove the back plate. Do not unscrew the knurled knobs as they are used for aligning the filament with the entrance slit of the monochromator and are set at the factory. Pull the defective bulb straight out of the socket. Insert the new bulb in its place but avoid touching its surface with your fingers. Wear gloves or use a soft cloth. Replace the back plate and replace the 5 hex screws.

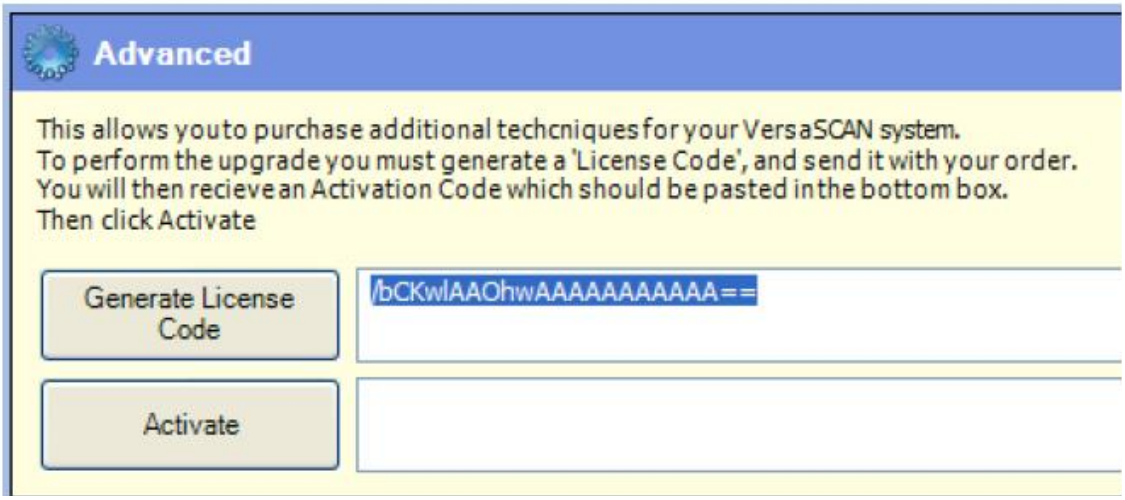
11.2 Activation of License Code

If the IPCE module is purchased with a ModuLab XM PhotoEchem system, the activation code will have been already supplied with the system and therefore no further action is required. However, if the IPCE is purchased after the PhotoEchem system, it is necessary to insert an activation code into the system. If the system does not have an activation code, the following error message will appear. Please speak with your local sales representative to request an activation code.



To request a code please follow the steps below

- In order for you to receive an activation code it is first necessary to generate an activation code. In the advanced section, click in the GENERATE LICENSE CODE TAB.



- Please copy the code and send to your local sales representative. They will provide an activation code via the Solartron factory.
- When you receive the code , cut and paste the code into the ACTIVATE box and press the ACTIVATE button.
- This should enable the IPCE measurements.

11.3 Calibration of Optical Bench

The system is equipped with a 50:50 beam splitter to provide a reference signal for all measurements. However, the split ratio is dependent upon wavelength and therefore for accurate determination of IPCE data, it is necessary to run a calibration. This is performed in two parts.

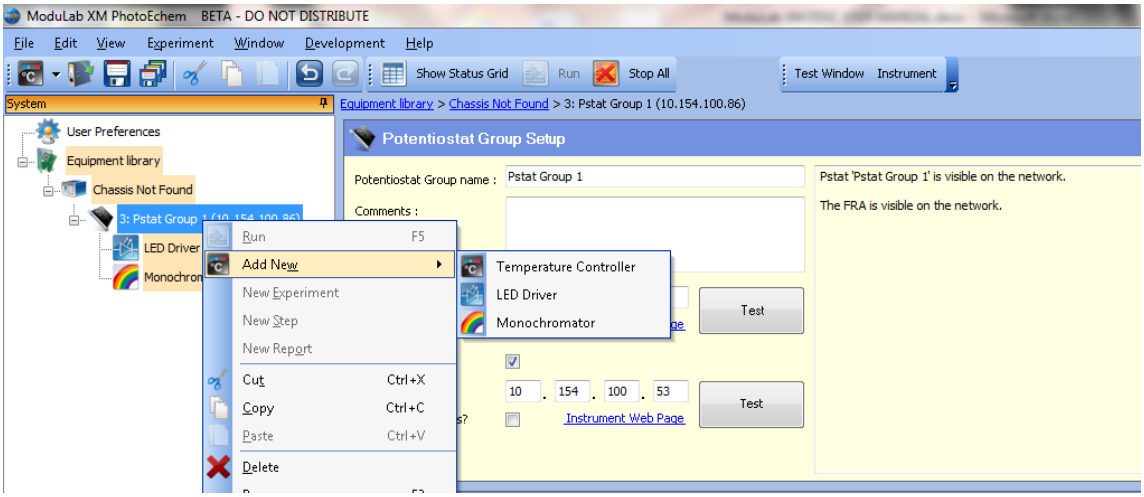
11.3.1 Pre-Calibration checks

The following section describes how to set-up the initial communications with the monochromator and to set the zero point of the system.

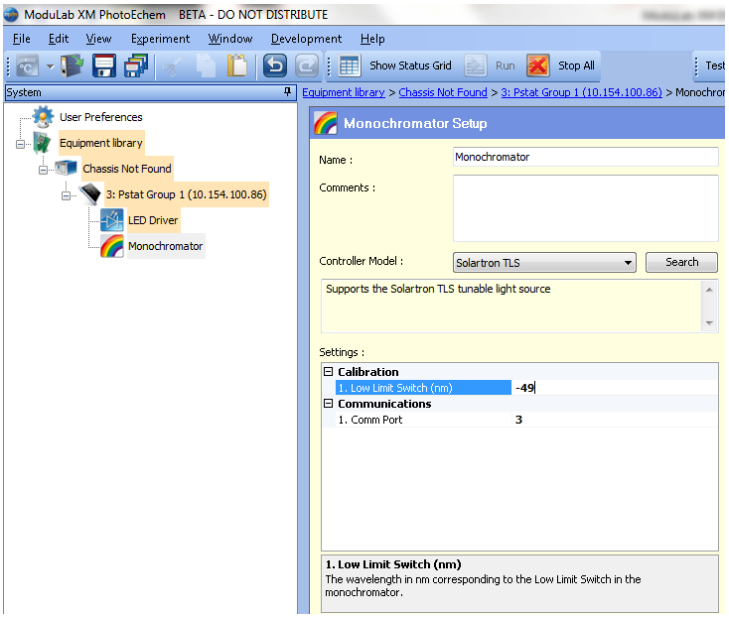
Communications with Monochromator

In order to run the Monochromator system through the software, follow these steps

- In the Equipment tree, right click on the PSTAT GROUP.
- Click 'ADD NEW'
- Click on MONOCHROMATOR as shown below

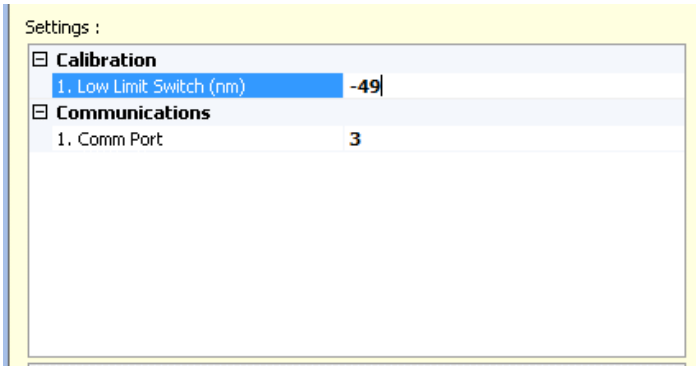


- Once the Monochromator icon appears in the system tree, left click on the monochromator icon. This will bring up the Monochromator dialogue box as shown below.
- Select SOLARTRON TLS from the controller model drop down list and press the search button.
- If the system is found, it will report a COM port number. Please ensure that the COM port number in the settings-→ communications box is the same



Calibration of Monochromator

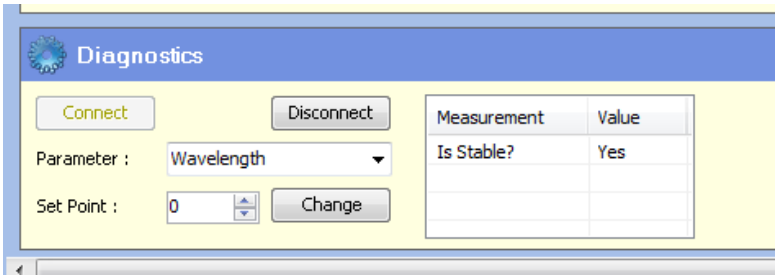
There is no positional feedback between the monochromator and the controller. The control software must be given an actual monochromator position upon start-up to reference subsequent movements. This is controller in software by the low limit switch in the setting dialogue box as shown below



- Ensure that the Monochromator power supply is switched on
- Using the diagnostics box, set the SET POINT to 0 and click CONNECT. This can be used to manually control the wavelength of the monochromator and is a useful tool for advanced studies.
- The monochromator has a counter on the front panel. This is the wavelength setting. Press CHANGE. The system will move to the low limit set point. You will hear the monochromator working and the counter will move towards and pass through 0.
- Make a note of the point at which the monochromator stops.
- To determine the low limit point, use the following expression

$$Low\ Limit = -(10{,}000 - endstop)$$

- For example, if the end stop was 9951, the Low Limit set point is –(10,000 -9951) = --49nm. DO NOTE FORGET TO INCLUDE THE MINUS SIGN
- Please enter this value into the LOW LIMIT SWITCH (nm) box in the Setting Dialogue box ((Communications with Monochromator)



The monochromator is now ready to use

11.3.2 Calibration of Beam Splitter

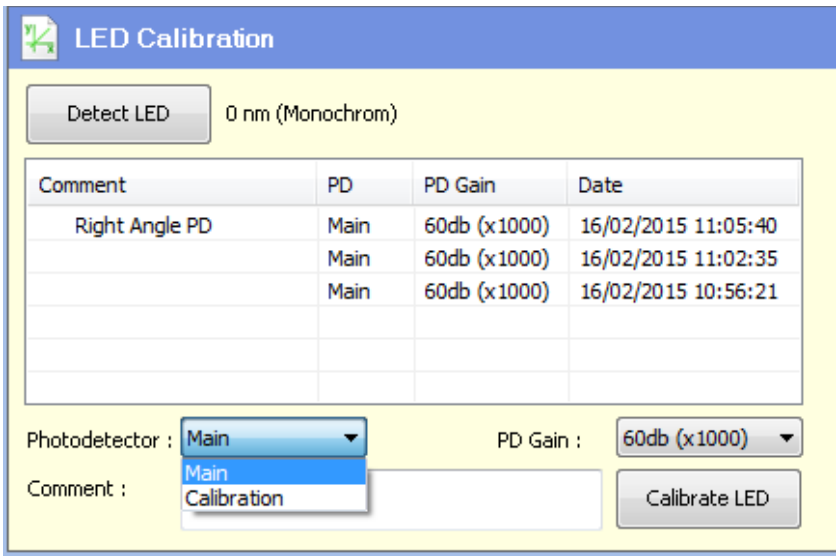
The following section describes the calibration of the beam splitter. Please open the Beam Splitter Calibration Dialogue box (click on the pstat group icon)

There are a number of options. We shall focus on the calibration of the monochromator only.

Creating new calibration file

You will first need to run an LED calibration using the monochromator Please follow the instructions for this highlighted in section 5 for connections to the PD amplifier.

- Change the wavelength of the monochromator to 500nm using the Diagnostics box in the monochromator settings (see section 11.3.1.1 for details).
- Run an LED calibration with photo-detector set to main and the PD gain setting at 60dB (as shown below)
- Press ‘Calibrate LED’



Once the calibration file has been measured, the next step is the calibration of the beam splitter.

You will need to set-up the hardware to perform this calibration.

- Screw the second photo-detector onto the front port of the beam splitter. This need only be finger tight. This is the same arrangement as the Reference Photo-detector that is already aligned with the bench. This photodetector is called the CALIBRATION PHOTODETECTOR.
- Connect a BNC T – piece to the OUTPUT of the detector. On one end, place the 50 Ohm terminator BNC piece (Figure 41: Connections to second PD amplifier for Beam Splitter Calibration

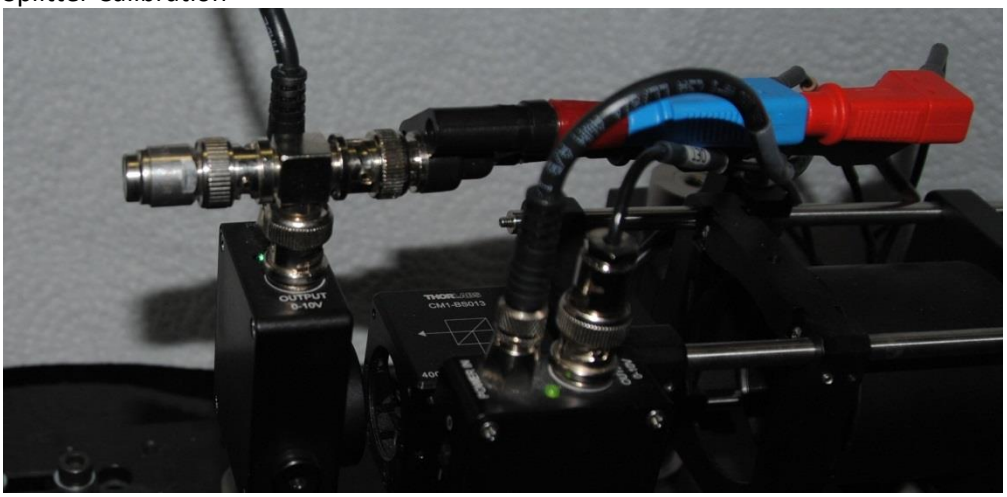
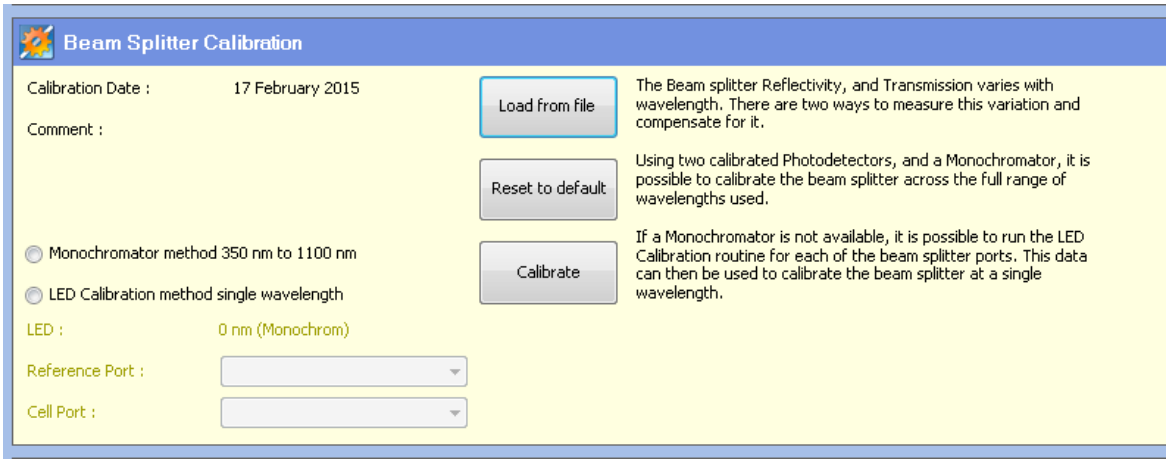


Figure 41: Connections to second PD amplifier for Beam Splitter Calibration

- Attach a BNC to Banana Plug adaptor to the other end,
- Place the Green and Black leads of the potentiostat card into the BLACK connector
- Place the Red and Blue leads of the potentiostat card into the RED connector.
- Please ensure that both photo-detectors are set at the same gain setting.
- Place the detector BNC cable from the PhotoEchem card to the 0-10V Output connection on the MAIN photo-detector.

- This process will create a new file.
- Select the MONOCRHOMATOR METHOD 350nm to 1100nm
- Click CALIBRATE



A Calibration file similar to the one show below is generated.

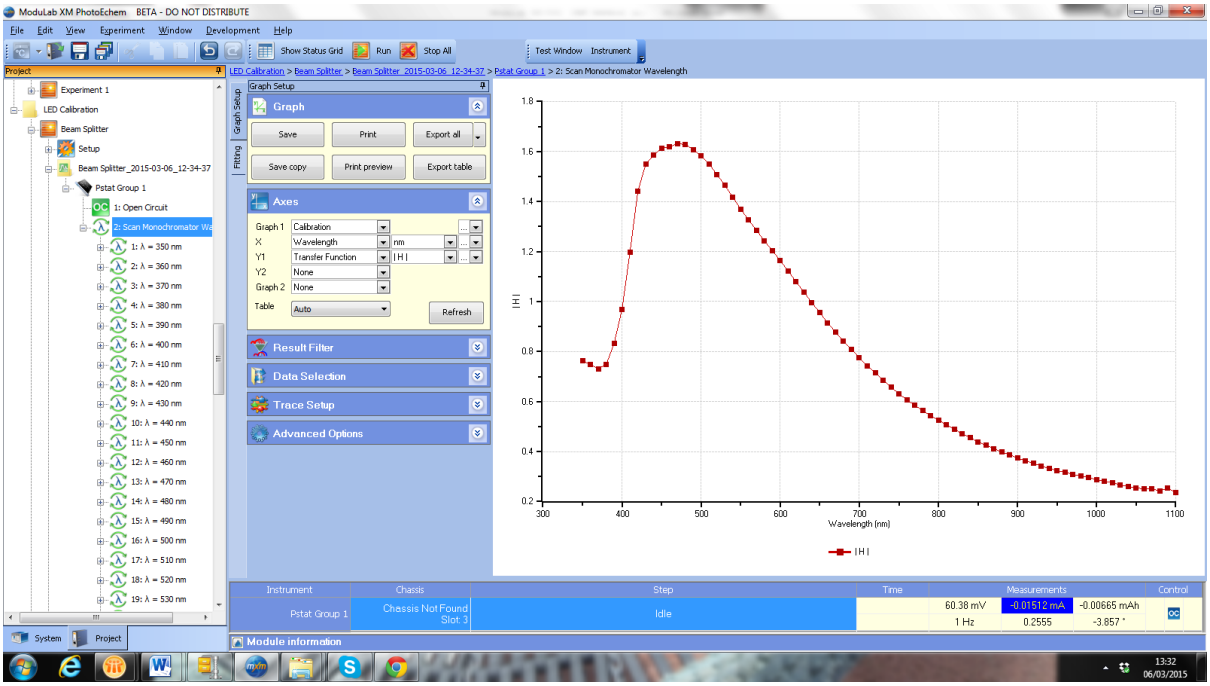


Figure 42: Typical Beam Splitter Calibration Curve

11.4 Alignment of Samples

Proper alignment of the sample with respect to the photo-detector is important to improve accuracy. The monochromatic light beam is non-homogeneous and therefore the sample must be aligned with respect to the photo-detector such that the photon flux at the photo-detector and the device under test are the same. The following procedure details how to achieve consistent results.

First, place the mask on the sample as shown in Figure 43: Placement of mask on solar cell.

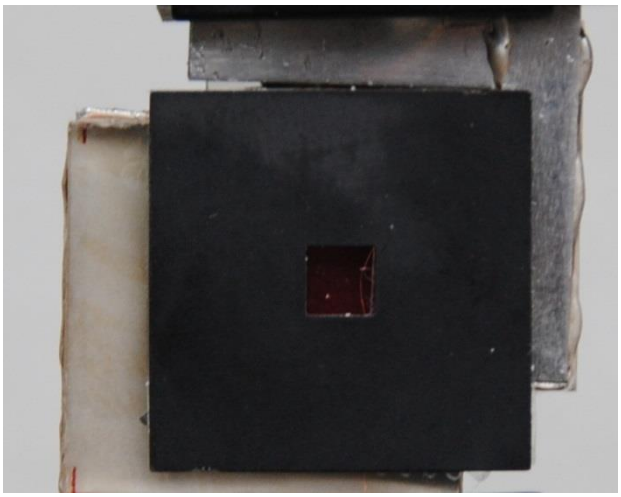


Figure 43: Placement of mask on solar cell

Please ensure that the mask is square with the sample. This ensures that when the sample is mounted in the sample holder, the exposed area can be aligned with the photo-detector. In order to help align the sample, it is advisable to illuminate the backside of the cell using the white bias provided or another light source. This will help project a clear image of the device under test on the photo-detector encapsulation. It may be necessary to adjust both the vertical and horizontal position of the cell for proper alignment.

NOTE: the distance between the cell and the beam splitter should be identical to that of the beam splitter and photo-detector.

It is easiest to see this alignment through the opposite side of the photo-detector. Examples of incorrect and correct alignment are shown in Figure 44: Incorrect Alignment and Figure 45: Correct Alignment. The image of the cell (orange) should lie within the boundary of the encapsulation of the photo-detector as illustrated in

Figure 46: Schematic of projection of BIAS source on the photo-detector.

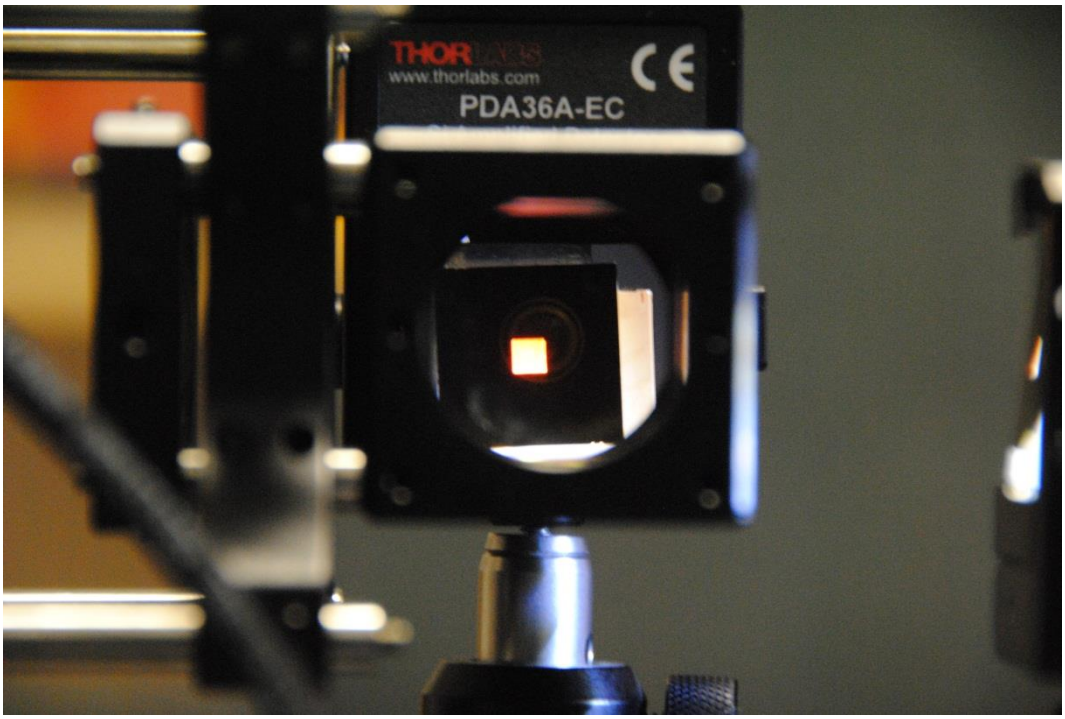


Figure 44: Incorrect Alignment

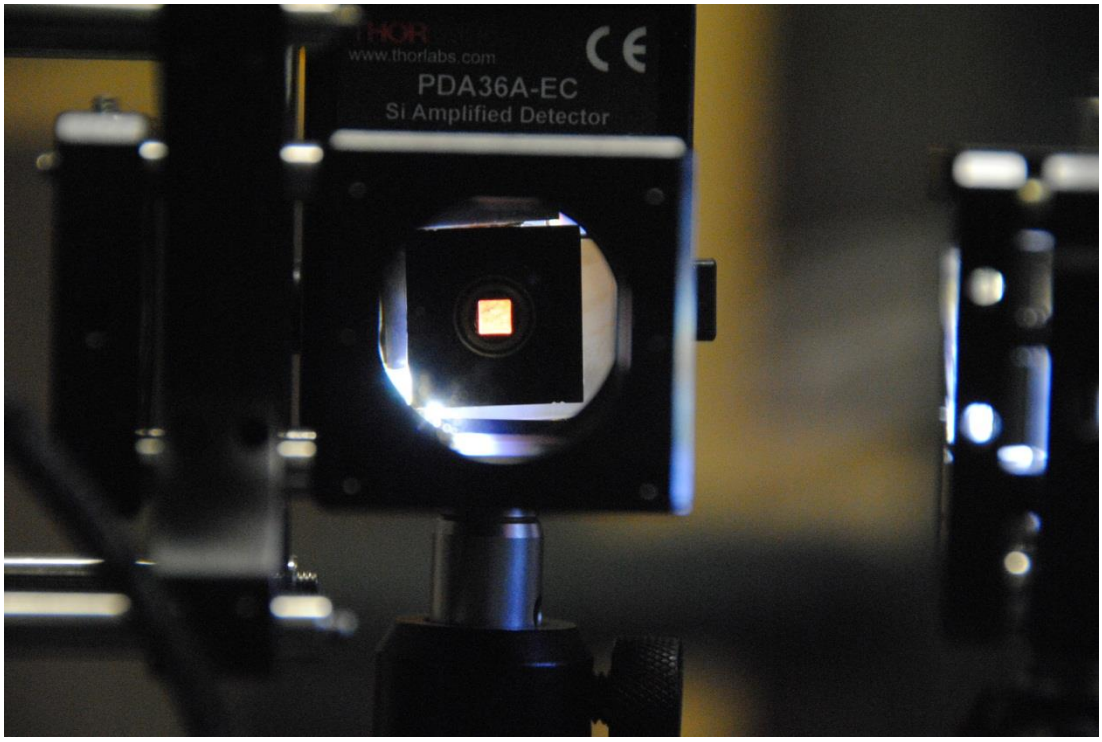


Figure 45: Correct Alignment

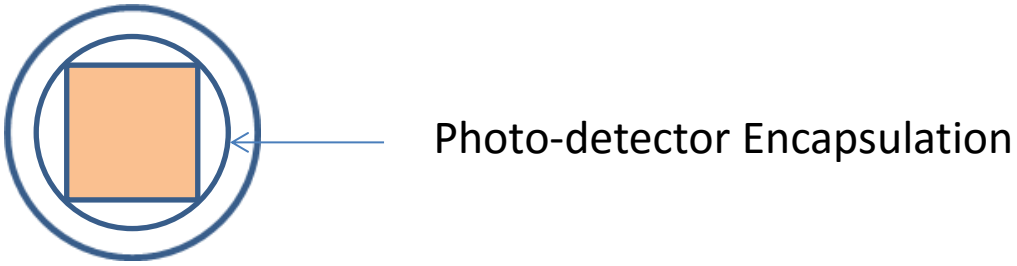


Figure 46: Schematic of projection of BIAS source on the photo-detector

11.5 White Bias Light Source

The following section describes the use of white bias light source. In some instances, the efficiency of the cell is intensity dependent i.e. the QE changes with changing light intensity. An example of this is shown in Figure 47: IPCE vs light intensity. The IPCE response is independent of light intensity only at higher intensities.

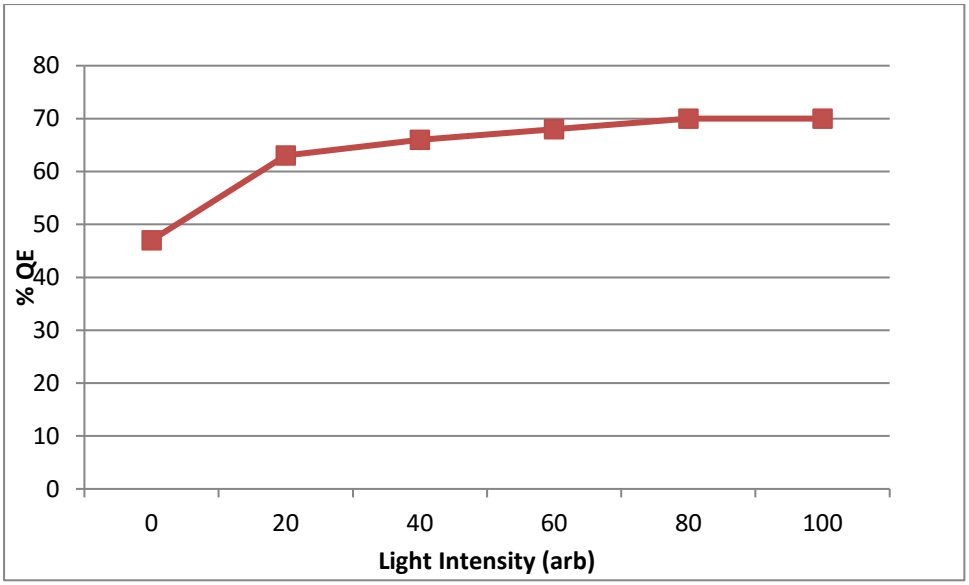


Figure 47: IPCE vs light intensity

This is clearly problematic especially when comparing results across groups and calculating the short circuit current density of the cell under AM1.5 conditions. It is therefore necessary to bias the cell with white light such that IPCE measurements are independent of the intensity of the probe (monochromatic) source. The system is shipped with a high brightness LED white light source.

11.5.1 Mounting White Bias Source

An additional breadboard is supplied with the system. The board should be bolted securely onto the main optical board using the bolts and connector bars supplied (refer to Figure 48: Breadboard mounting onto main Optical Bench Board)

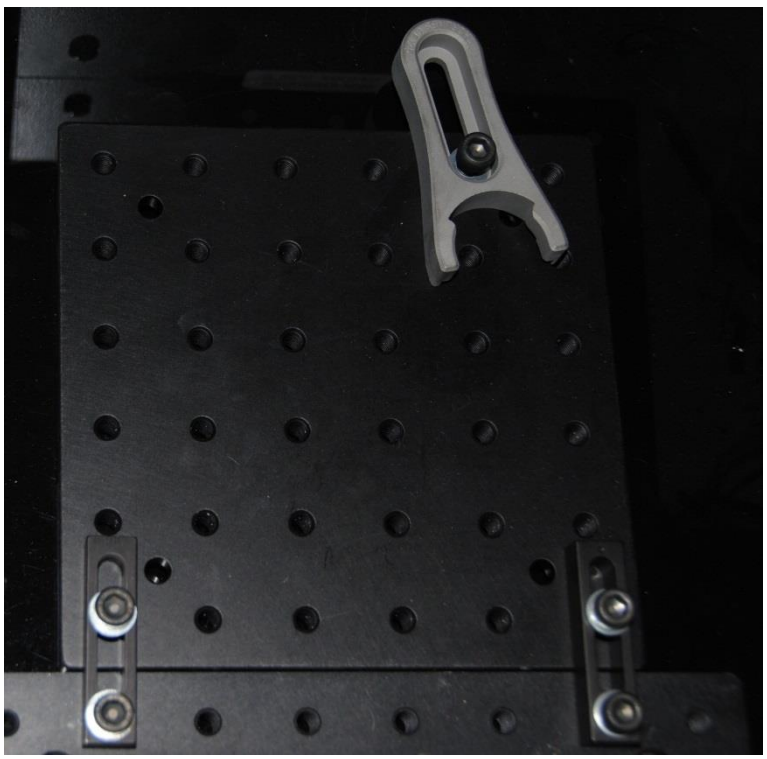


Figure 48: Breadboard mounting onto main Optical Bench Board

The White Bias Source can be mounted on the board as shown in Figure 49: White Bias Source. The bias source is supplied AC-DC switch mode power supply. Please chose the correct mains adaptor for your territory.

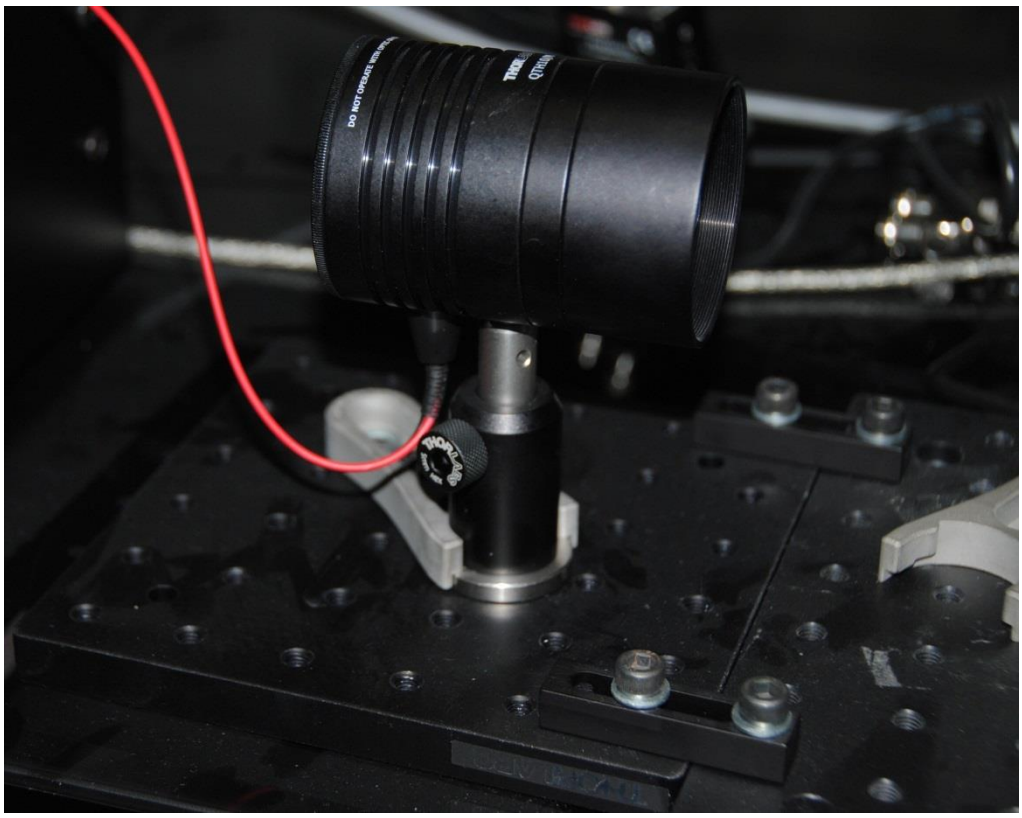


Figure 49: White Bias Source

11.5.2 Adjusting White Bias Light Intensity

The intensity of the white Bias Source is approximately 50mW cm^{-2} at the focal point of the beam. This will produce a large short circuit at the cell under test. This may be very large when compared with the AC current generated by monochromatic source. In general, the AC current should be no less than 0.1% of the current generated by the white bias source.

The I-V convertor measures both the DC and AC current components. The ranges of interest in these studies will be the 30mA, 3mA, 300uA and 3uA. Each range must be able to measure the DC component whilst able to resolve the AC component. The entire signal is passed to the analzyer of the FRA and therefore distortions due to poor signal/noise will reduce the efficacy of the system. Optimization of current range, White Bias intensity and AC signals is therefore an important part of the IPCE measurement.

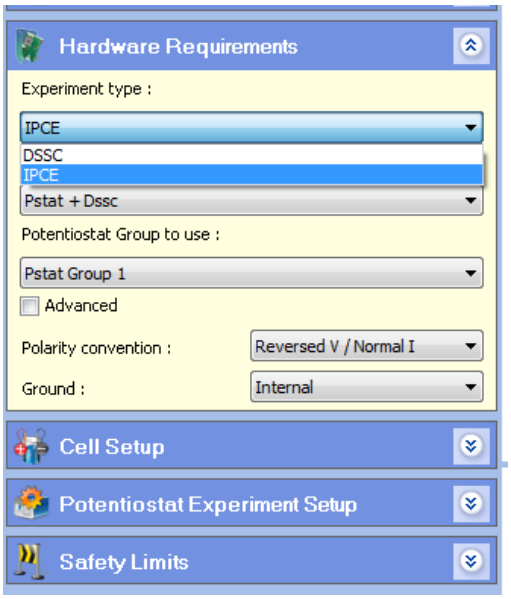
It will be necessary to attenuate the beam before IPCE measurements. This can be achieved with

- Neutral density filters (customer supplied)
- Positioning the light source at an angel to the device under test.

The section will focus on the second of these methods.

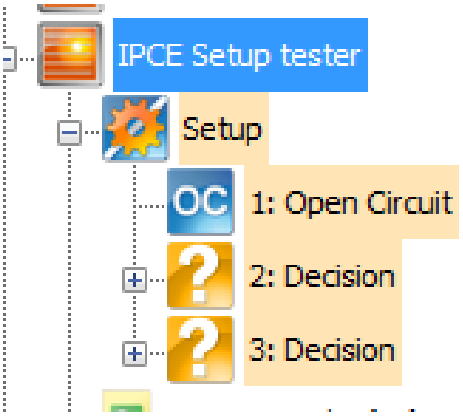
If one records the steady state photocurrent of the cell as a function of angle of incidence, it becomes apparent that the photocurrent falls with increasing angle of incidence. We suggest an angle of between 30 and 45 degrees.

We provide a test routine in software that determines if the bias light source is too strong for the measurement for the reference solar cell. These cells produce about 200uA of DC under low light illumination. Therefore, the I-V convertor will use the 300uA range. The test protocol determines if the measurements will be within this range.



Before using the software for measurements, it is necessary to select the IPCE experiment from the dropdown list type in the hardware requirements (as shown above)

Included in the getting started experiment files is an experiment called ‘IPCE Setup Tester’.



Run this test with the Bias Light at an angle to the device under test. If the current is too large the following screen appears (Figure 50: White Bias test result - FAIL)

If the test fails, adjust the position of the white bias light such that the steady state current < 250uA). Conversely, a PASS message will appear if the white bias light intensity is below the limits of the measurement system.

We recommend that the cell is allowed to thermally equilibrate with the lamp for at least ten minutes before measurements. Tests at the Solartron facility have show that the system is stable to within 1 deg C for over two hours after the initial warm up period.

For cells that are more efficient and produce high short circuit currents such as Peroskvite cells, it may be necessary to change the pass/fail parameters in the test. To achieve this , change the setting in OPTION 4 to a higher current. For example if your cell produces 2.5mA current under illumination then the I-V convertor will select the 3mA range. Set the current to > 2.5mA in option 4. If the current exceeds this, then reduce the intensity of the white bias source on the cell.

The key issue using these set up technical is to

- Ensure that the IPCE response is independent of white light bias intensity
- Use the minimum white light bias to achieve the above.

This has the benefit that the results will be less noisy than those obtained if the white light bias is too high. It is therefore recommended that you generate your own IPCE vs. Light intensity (current) plots as shown in Figure 47: IPCE vs light intensity. The following section describes how to run IPCE experiments.

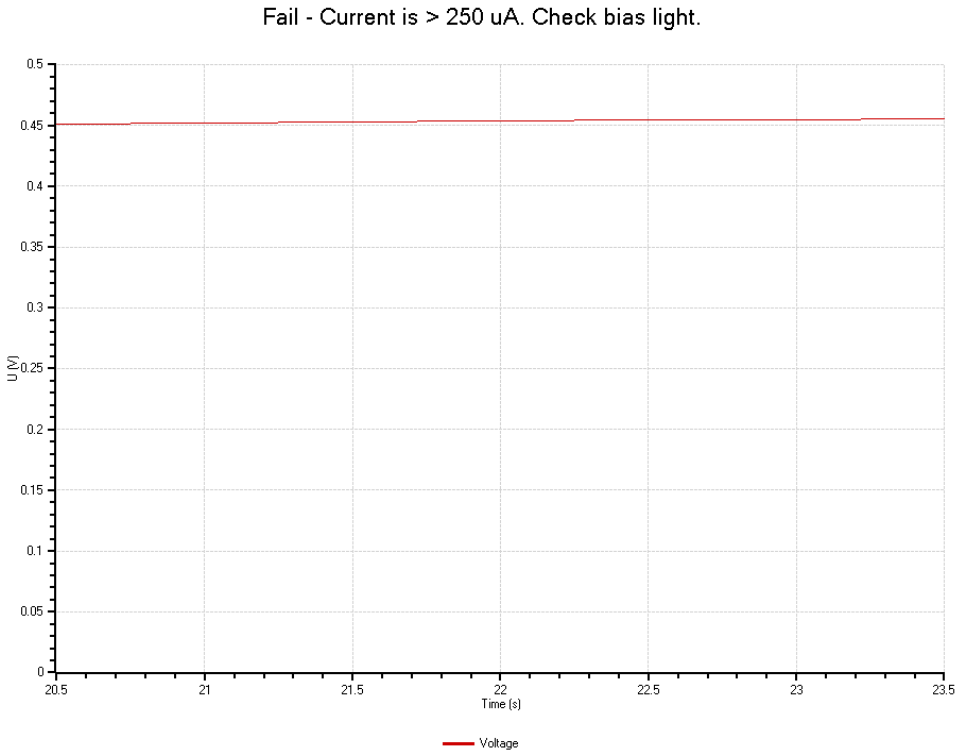
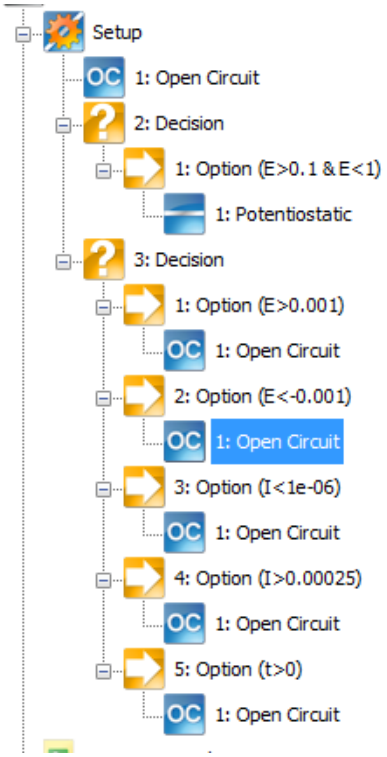
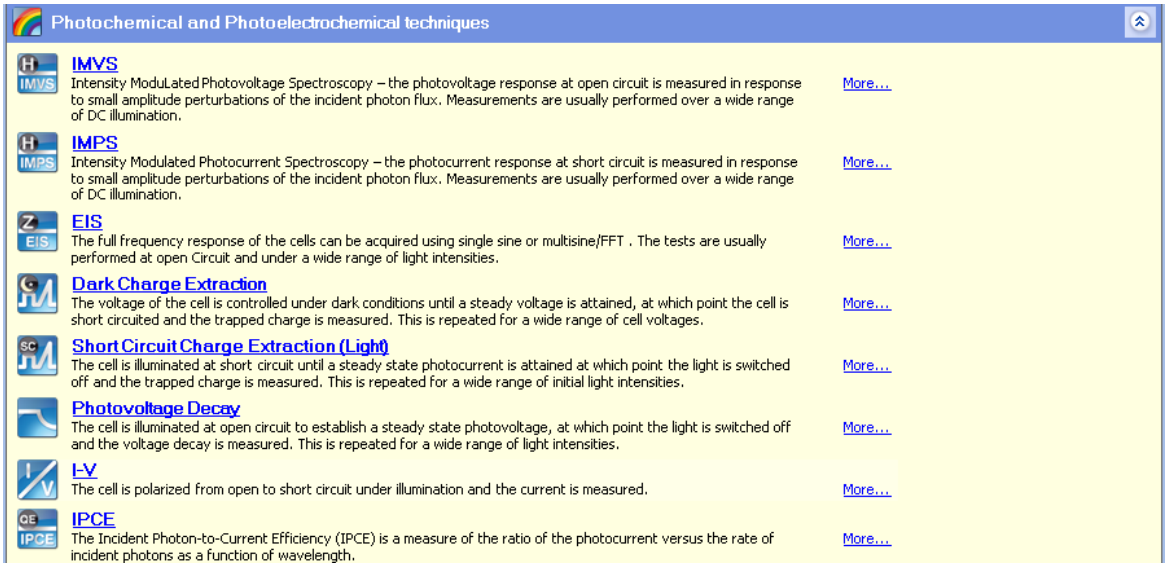


Figure 50: White Bias test result - FAIL

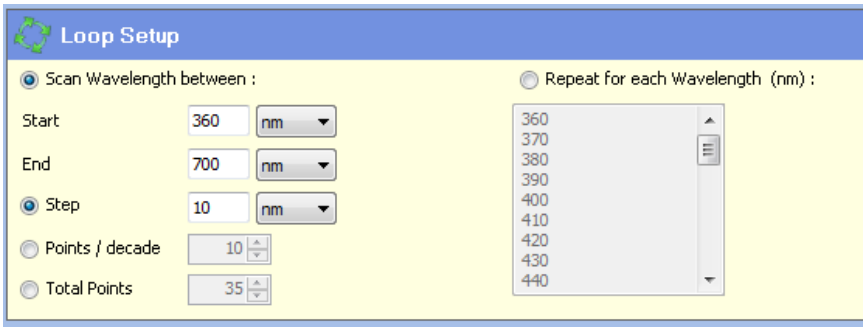


11.6 IPCE Experiments

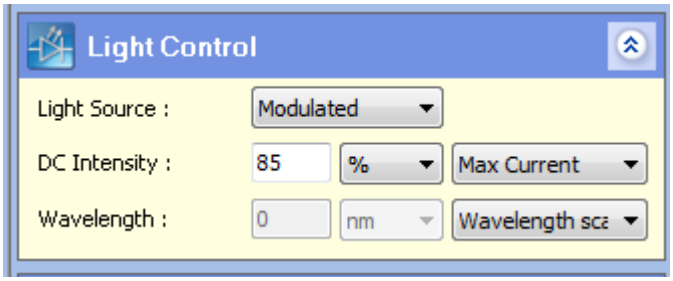
To run an IPCE experiment, select IPCE from the following list



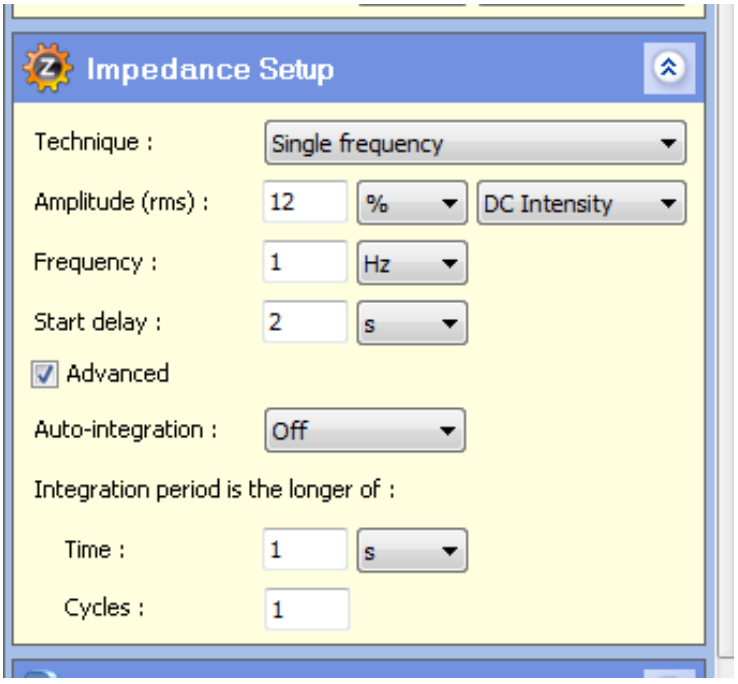
A predefined experiment is set up for your convenience. However, you are free to change the parameter settings if you wish. The main control parameters are split into three sections;



Loop Set-up: the sets the start and end wavelength and the step size in nm. The max resolution of the system is 10nm.



Light Control: This determines how the light is controlled for an IPCE step. The light is modulated using the FRA generator (see Impedance set up). It is advisable to LEAVE THESE SETTINGS. The DC intensity is the % of max current that can be supplied to the lamp.



Impedance Setup: This controls the modulation frequency and amplitude of the monochromatic source. It is suggested not to change these values. For some devices, it may be necessary to increase the Integration period to improve Signal/Noise e.g. increase the number of cycles to 3.

The experiment is started in the same manner as all PhotoEchem experiments. A typical IPCE graph is presented in Figure 51: IPCE graph. To select IPCE graphs, select the IPCE option from the drop down list in the GRAPH 1 settings within the AXES dialog box.

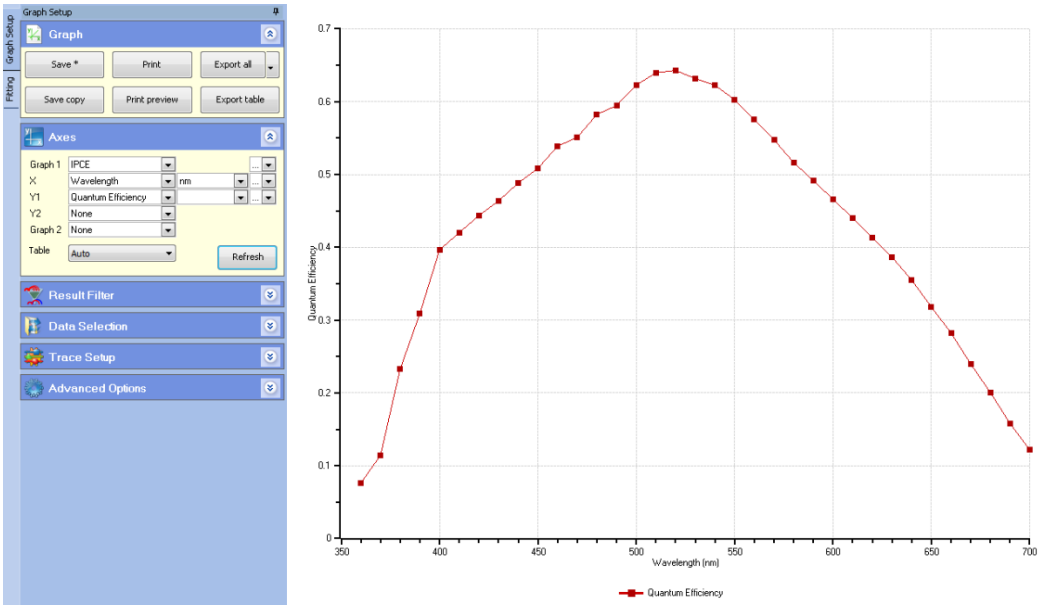


Figure 51: IPCE graph

11.7 Data Analysis

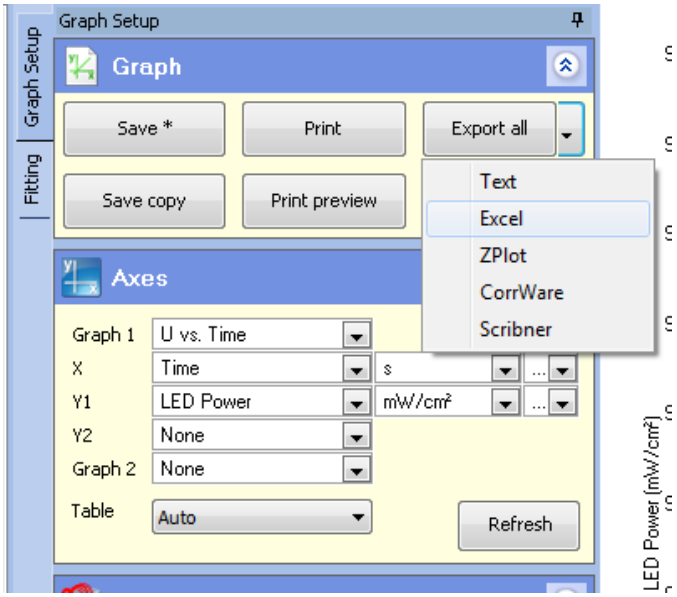
The software is equipped with an AUTO-ANALYSIS feature. To analyse a data set perform the following operations:

In order to AUTO analyse data, first

- Select Experiment that you wish to analyse by highlighting the data folder



Example of a data folder.



- In the Graph Setup box, select 'EXPORT ALL' and Click Excel
- An excel workbook will automatically load.
- The workbook will contains 8 tabs

  General, IMVS,IMPS, PV Decay, CE Short, CE Dark, IPCE and ASTMG173

A typical set of IPCE data is presented in Figure 52: IPCE spectrum of Ionic Liquid based cell.

In addition to the IPCE data, the short circuit current for the cell under AM1.5 sunlight is calculated. The data sets were derived from the spectral irradiance in sheet ASTMG713.

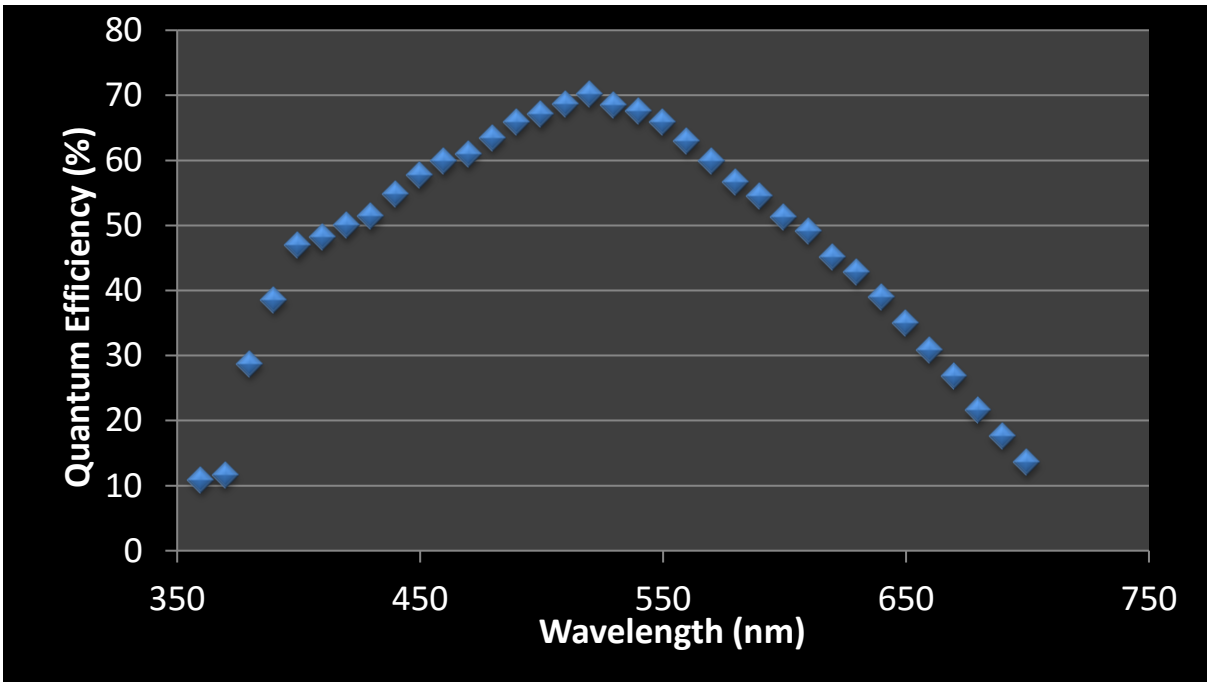


Figure 52: IPCE spectrum of Ionic Liquid based cell.

11.8 Specifications

General	
Monochromator Wavelength	350nm – 1100nm
System Wavelength (with Optical Bench)	350nm – 800nm
Min. Step Size	10nm
Typical Bulb Lifetime	➢ 100 hrs
Temperature Drift of Cell with Bias Light	< 1 Deg C after 10 min warm -up





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