Optical Bench

User Manual

Metrohm Autolab

Please read this manual carefully before starting to use the Autolab Optical Bench

The next sections deal with appearance and use of the equipment and contain necessary information regarding operation and installation.

SAFETY PRACTICES

General

- The following safety practices are intended to ensure safe operation of the equipment and must be observed during all phases of operation, service and repair of the instrument.
- Failure to follow these instructions may cause unsafe operation.
- Metrohm Autolab is not liable for any damage caused by not complying with the safety requirements.
- Failure to follow these instructions may void any warranty provided to this product.

Electrical hazards

- To avoid electric shock hazard, always ground the equipment by using the provided power adaptor.
- There are no user-serviceable parts. Equipment installation, component replacement and internal adjustments must be done only by qualified personnel.
- Opening the equipment poses a risk of exposure to potentially dangerous voltages.
- Please also refer to the Electrical Hazards described separately for the Autolab instrument used.

General precautions

- Use only stable surfaces for setting up the system.
- Do not look directly at the light coming out of the light source.

- Allow the light source to cool down after prolonged used at high driving currents.

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Autolab PGSTAT in combination with Optical Bench

The characterization of optical energy conversion devices (i.e., solar cells) requires controlled illumination from a light source. Depending on the experimental requirements, the light source can be a solar simulator, a simple LED, or a laser. This light source must be programmable in order to expose the device under test (DUT) to a user-defined light intensity. The type of light profile depends on the experimental conditions.

Information

This document provides basic information regarding the Autolab Optical Bench and the associated products. This accessory is controlled through the NOVA software. Additional resources are available online (www.metrohm.com).

This manual describes the use of the Autolab Optical Bench in combination with the Autolab PGSTAT.

Note

The Optical Bench is compatible with all Autolab PGSTAT instruments **except** the Autolab PGSTAT302F. In this manual, the term Autolab is used to describe any type of compatible instrument.

The following measurement techniques are possible with the Optical Bench in combination with the Autolab PGSTAT:

- Polarization curves and power density curves
- Charge extraction measurements
- Electrochemical impedance spectroscopy at constant illumination

- Intensity-modulated photovoltage spectroscopy (IMVS)
- Intensity-modulated photocurrent spectroscopy (IMPS)

It is also possible to combine all the measurement techniques in a single experiment.

Warning

Intensity-modulated measurements (IMPS, IMVS) are **not** possible with the µAutolab type III/FRA2, Autolab PGSTAT101 and the Multi Autolab with M101.

Warning

This device must be used carefully to prevent personal injury. Metrohm Autolab is not responsible for physical injuries sustained while using this product. It is advised to read this documentation very carefully before operating this equipment. Please contact Metrohm Autolab (autolab@metrohm.com) in case of problems.

Whenever applicable, a hot surface warning label or strong optical radiation warning label are used in this manual as reminder to the hazards that can be encountered when operating this equipment.

Warning

This warning symbol is used in this document to indicate a hot surface hazard related to the heat dissipation in the light source. Allow the system to cool down after prolonged use.

Warning

This warning symbol is used in this document to indicate an optical hazard related to highly focused beam of light generated by the LED light source. Do not look directly into the light beam when the light source is on.

1 – Scope of delivery

The Optical Bench is supplied with the following items:

- Autolab LED Driver box
- Power adaptor
- 50 Ω terminator plug
- 6 BNC to SMB adaptor plugs
- 3 SMB shielded cables (1 m)
- 1 BNC shielded cable (1 m)
- 1 BNC shielded cable (2 m)
- 3 BNC shielded cable (50 cm)
- DIO to BNC cable for Autolab N series PGSTAT instruments and µAutolab
- DIO to BNC cable for Autolab PGSTAT204 and M204 module
- 1 BNC splitter
- Optical rail

The Optical Bench is intended to be used with the Autolab LED light source, included with the Optical bench:

- 1 LED array cover (627 nm), 700 mA maximum current
- 1 LED cover holder
- 1 LED cover holder connection cable (2 m)
- 1 calibrated photodiode holder with calibration certificate

Additional accessories are available for the Autolab LED light source:

- Additional LED covers (see Section 5.2 for more information)
- Additional LED cover holders

Figure 1 shows a complete overview of the Optical Bench items.

Figure 1 – The Optical Bench, including the LED driver, optical rail, light source and photodiode holder

The three SMB/SMB shielded cables can be fitted with SMB to BNC adaptor plugs. Depending on the type of FRA module used in combination with the LED Driver, these cables can be modified accordingly:

- For the FRA2 module, the cables must be fitted with SMB to BNC adaptors on both ends (see Figure 2).

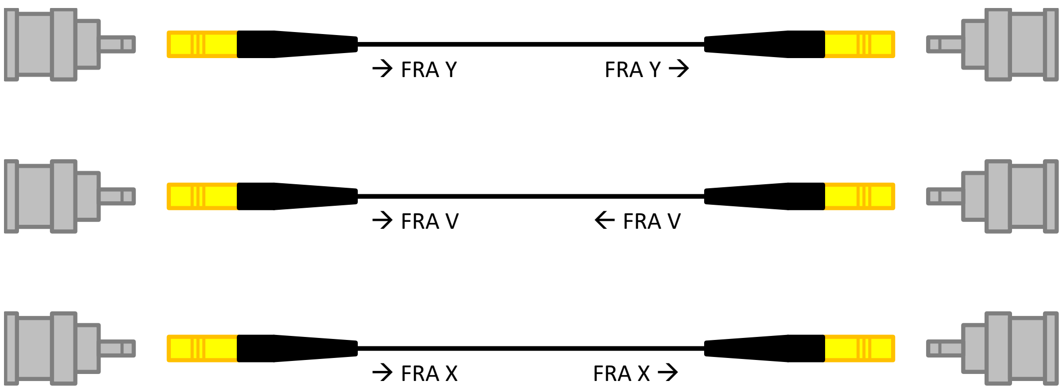


Figure 2 – Configuration of the SMB cables used in combination with the FRA2 module

- For the FRA32M module, the cables must be fitted with SMB to BNC adaptors on a single end (see Figure 3).

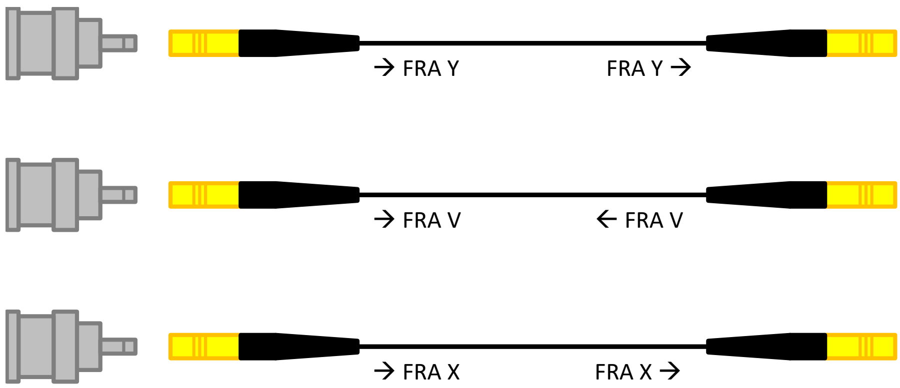


Figure 3 - Configuration of the SMB cables used in combination with the FRA32M module

2 – Hardware requirements

The experimental setups described in this manual require the following hardware:

- Autolab PGSTAT, μAutolab or Multi Autolab (for DC measurements)
- FRA2 or FRA32M module (for AC measurements)
- Autolab LED Driver
- Autolab LED cover with holder

Warning

The LED Driver has a working range of 0-10 V, which means that the FRA2 modules must be modified to the 0-10 V input range. The hardware setup must be adjusted accordingly (see Appendix 1).

3 – Software requirements

The experimental setups described in this manual require the following software:

- Latest version of NOVA

This manual assumes that the reader is familiar with the operation of the Autolab instrument in combination with the NOVA software. More information on the software tools used in this manual can be found in the NOVA manual.

4 – Principle of operation

The Autolab LED Driver is a controlled current source, able to supply a constant current between 0 A and 1 A. This current can then be used to control the light intensity of the LED light source.

The Autolab LED Driver is controlled by using an analog voltage value provided by the Autolab (through the DAC164 #1 or the Vout). This voltage value is converted by the LED driver to a constant current using the following relationship:

$$i_{LED} = \left(\frac{i_{Range}}{V_{Range}} V_{DAC164}\right) = \left(\frac{1\ A}{10\ V} V_{DAC164}\right) = 100\ mA/V \cdot V_{DAC164}$$

Where i_{LED} is the output driving current, in mA, i_{Range} is the current range of the LED driver (1 A), V_{Range} is the input range of the LED driver (10 V) and V_{DAC164} is the output value of the DAC164 #1, in V.

Setting the V_{DAC164} to 1 V will result in a driving current i_{LED} of 100 mA. Setting the V_{DAC164} to 0 V switches the light off and setting the V_{DAC164} to 10 V will generate the maximum light intensity.

Warning

Do not use DAC164 #2 to control the light intensity. The channel is internally connected to the PGSTAT and is not suitable for this application.

Note

When the input voltage is 90 mV or less, the current output will be switched off and the output current will be 0 mA. The minimum current is therefore limited to 9 mA.

Additionally, the LED Driver is also fitted with an extra input that can be coupled to the FRA2 or FRA32M module in order to modulate the light intensity of the LED connected to the LED Driver.

In this case, the driving current is defined by the output of the DAC164 #1 (or the Vout) and the output of the FRA2 or FRA32M module.

The difference between the DAC164 output and the FRA V output is converted by the LED driver to a modulated current using the following relationship:

$$i_{LED} = i_{DC} - i_{AC} = \left[\frac{i_{Range}}{V_{Range}}(V_{DAC164} - V_{FRA\,V})\right]$$

$$i_{LED} = 100\ mA/V \cdot (V_{DAC164} - V_{FRA\,V})$$

Where i_{LED} is the output driving current, in mA, i_{Range} is the current range of the LED driver (1 A), V_{Range} is the input range of the LED driver (10 V), V_{DAC164} is the output value of the DAC164 #1, in V and $V_{FRA\,V}$ is the output value of the FRA V, in V.

Warning

The V_{DAC164} value must always be larger than $V_{FRA\,V}$ so that the $V_{DAC164} - V_{FRA\,V}$ difference is always positive. When this difference is negative, the net output driving current i_{LED} is 0 A, regardless of the values of V_{DAC164} and $V_{FRA\,V}$.

Note

The output driving current, i_{LED} is given by the difference between the constant current, i_{DC} and the modulated current, i_{AC}.

Setting the V_{DAC164} to 1 V and the $V_{FRA\,V}$ to 100 mV will result in a DC driving current, i_{DC} of 100 mA, modulated by an AC current, i_{AC} of 10 mA. Setting the $V_{DAC164} - V_{FRA\,V}$ to 0 V switches the light off and setting the $V_{DAC164} - V_{FRA\,V}$ to 10 V will generate the maximum light intensity.

5 – Installation

This section describes the required connections between the Autolab PGSTAT and the Autolab LED driver.

The combination of the PGSTAT with a LED driver is a very useful hardware construction which allows measurements at controlled light intensity on solar cells. In this setup, the LED Driver is used to control the output of the light source while the Autolab PGSTAT is used to measure the potential and current on the device under test (DUT).

The LED Driver can be operated in constant output mode or in modulated output (which requires the FRA2 or FRA32M module). The Autolab PGSTAT can be operated in potentiostatic mode or in galvanostatic mode.

5.1 – Autolab LED driver

The Autolab LED driver is an analog and digitally programmable constant current source which can be used to supply a driving current to a LED or a LED array (see Figure 4). The maximum driving current which can be generated by the LED driver is 1 A DC.

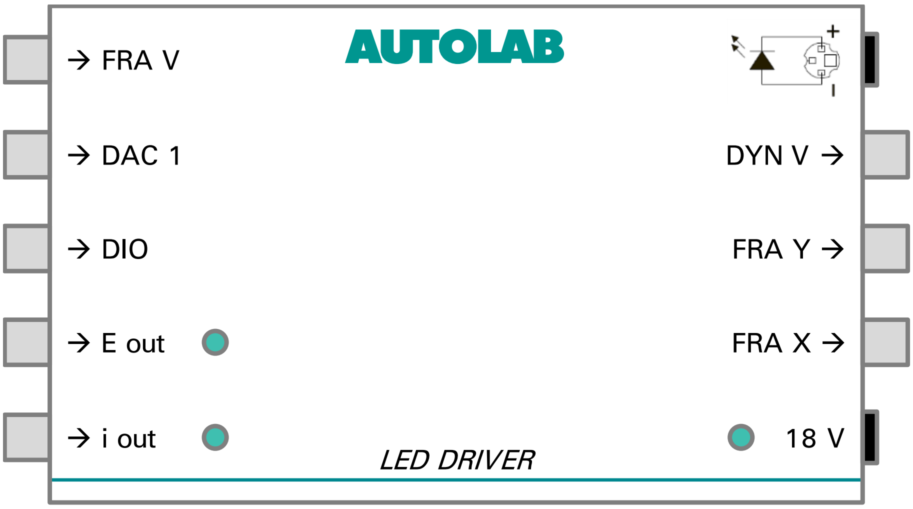


Figure 4 – Top view of the Autolab LED driver

The LED driver provides the following functionality:

1. A dual analog programmable input (→ FRA V and → DAC 1) for direct control of the LED current supplied by the driver.
2. A current to voltage converter for direct analog readout of the driving current (FRA Y →).
3. A dual input feedthrough for the Autolab E_{out} and i_{out} signals to the FRA → X output. The input is controlled by a DIO triggered switch (→ DIO input).

The LED Driver is designed to control a dedicated LED array of three, 700 mA rated LEDs, provided by Metrohm Autolab (see Section 5.2 for more information). This light source is recommended for this application and will be assumed in the rest of the document.

Note

The light source is a critical component of this hardware setup and it should be chosen carefully. Light sources like LEDs are economical and offer a narrow spectral distribution. Laser diodes can also be used. Both light sources have a low power output. Alternative light sources can be used; however, their use falls outside of the scope of this manual.

When working in combination with the FRA2 or FRA32M module, the maximum frequency that can be used in combination with the LED driver is 20 kHz. The maximum frequency is limited by the voltage-to-current converter in the LED driver.

5.1.1 – Basic connections

Figure 5 shows an overview of the basic connections to and from the LED driver. All the connectors located on the left-hand side of the driver are input connections from the Autolab PGSTAT to the LED driver. The BNC connectors located on the right-hand side of the driver are the output connections to the Autolab PGSTAT.

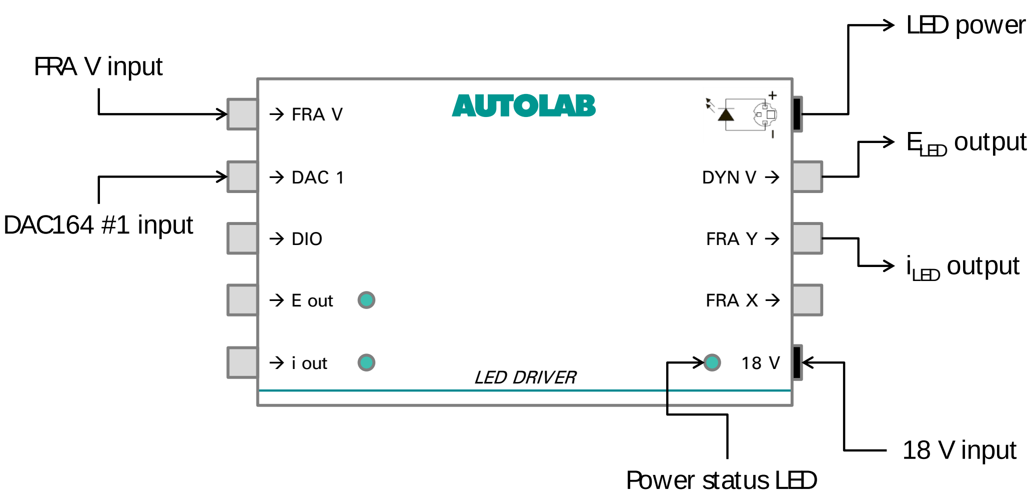
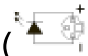


Figure 5 – Overview of the basic connections provided by the LED driver

The following connections are located on the left-hand side:

- **FRA V input (→ FRA V):** this input is used to supply the AC amplitude used to modulate the light intensity, in volts (0-10 V input range). See Section 5.3.2 for more information. When no signal is supplied to this input, this plug must be shorted using a 50 Ω termination plug (included).
- **DAC164 #1 input (→ DAC 1):** this input is used to supply the DC amplitude used to modulate the light intensity, in volts (0-10 V input range). See Section 5.3.1 for more information. This value must always be larger than the value supplied to the FRA V input.

The following basic connections are located on the right-hand side:

- **LED power ():** this connector is used to provide current to the LEDs through a dedicated cable. This cable is used to interface the driver and the holder described in Section 5.2.
- **E_{LED} output (DYN V →):** this output provides a voltage (0-10 V), corresponding to the difference between the value provided to the DAC1 input and the value provided to the FRA V input (on the left-hand side of the driver).
- **i_{LED} output (FRA Y →):** this output provides a voltage (0-1 V), proportional to the LED current. The output value, in mV, corresponds to the driving current, in mA (1000 mV → 1000 mA driving current). For IMPS and IMVS measurements, this output is fed into the FRA Y input.
- **18 V input:** this input is used to supply power the LED driver. The driver is powered when the power status LED located next to this input is lit.

5.1.2 – Optional connections

Figure 6 shows an overview of the optional connections to and from the LED driver (the basic connections are grayed out). All the connectors located on the left-hand side of the driver are input connections from the Autolab PGSTAT to the LED driver. The BNC connectors located on the right-hand side of the driver are the output connections to the Autolab PGSTAT.

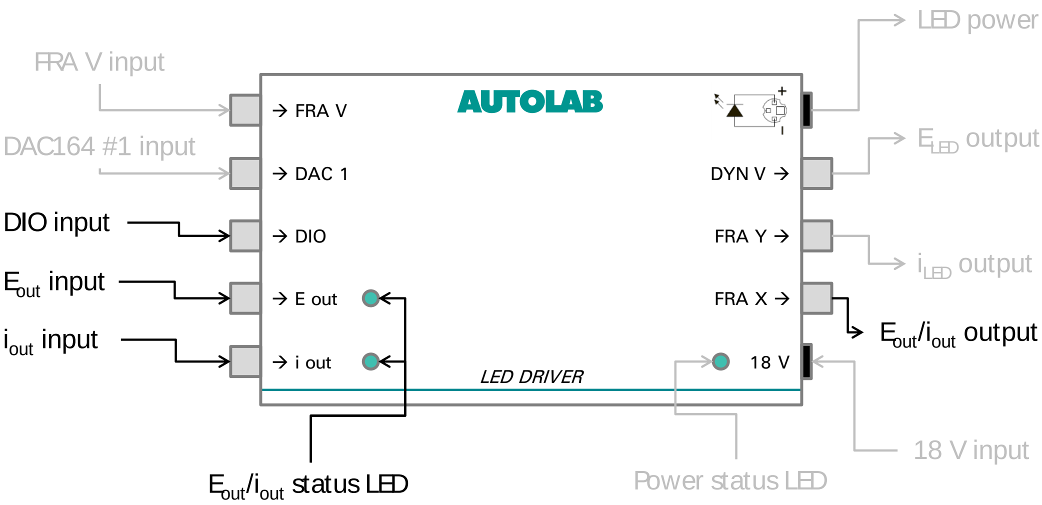


Figure 6 – Overview of the optional connections provided on the LED Driver (Basic connections are greyed out)

The following connections are located on the left-hand side:

- **DIO input (→ DIO):** this input can be used to connect to the DIO port(s) of the Autolab. By setting the DIO to the down status (default) or the up status, the switch connected to the E_{out} and i_{out} can be toggled[1]. The default position is set to i_{out}.
- **E_{out} input (→ E out):** this input can be used to connect to the E_{out} plug provided by the monitor cable of the Autolab (see Section 9.4.1 for more information). The E_{out} signal is required during IMVS measurements.
- **i_{out} input (→ i out):** this input can be used to connect to the i_{out} plug provided by the monitor cable of the Autolab (see Section 9.4.2 for more information). The i_{out} signal is required during IMPS measurements.

[1] The switch can also be toggled through an analog voltage. Supplying more than 2.5 V on this DIO input triggers the switch.

The following optional connections are located on the right-hand side:

- **E_{out}/i_{out} output (FRA X →):** this connector provides a direct connection to either the E_{out} or the i_{out} supplied on the left-hand side of the driver, through the E_{out} and i_{out} connectors, respectively. The default is i_{out} (indicated by the status LED). This output is connected to the FRA X for IMPS (i_{out}) or IMVS (E_{out}) measurements.

5.2 – Autolab LED array

The Autolab LED driver can be connected to a dedicated LED array. This array consists of a tri-focal LED assembly, collimated into a narrow beam with a lens. The LED array and the lens are enclosed in an anodized aluminum cover (see Figure 7).

The light beam width is 18°.

Warning

The lens mounted on the light source provided a highly focused beam of light. Do not look directly into the light source when it is operating, even if the driving current is small.

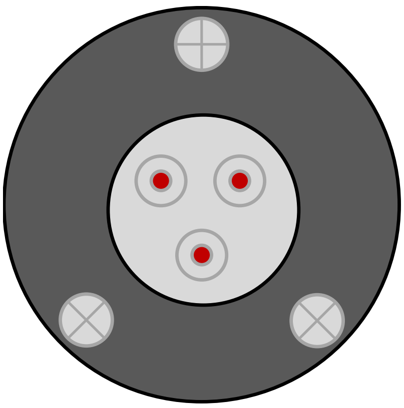


Figure 7 – Schematic top view of the LED array casing

The factory default cover, supplied with the Optical Bench, is fitted with three, 700 mA rated, red LEDs (wavelength: 627 nm).

Other covers are available, on request. The following wavelengths are available (see Table 1):

Table 1 – Overview of the available LED covers

Article code	Color	Wavelength (nm)	Maximum output (Lumen)
LDC655	Deep Red	655	n.a.[2]
LDC627	Red	627	306
LDC617	Red-Orange	617	402
LDC590	Amber	590	396
LDC530	Green	530	483
LDC505	Cyan	505	366
LDC470	Blue	470	210
LDCCW	White (Cool)	n.a.	660
LDCWW	White (Warm)	n.a.	435
LDCNW	White (Neutral)	n.a.	660

The back plane of the LED cover is fitted with two holes used to provide electrical contact to the LEDs enclosed in the cover (see Figure 8). Three screws are embedded into the cover to fasten it to the holder.

[2] The output in Lumen for the Deep Red source is not specified. The maximum output power is 1.74 W at 700 mA driving current.

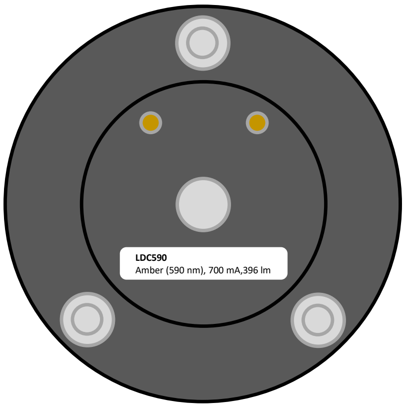


Figure 8 – Schematic bottom view of the LED array casing

A label is located on the back of the cover, indicating the type of LED included in the cover.

The cover can be mounted on a dedicated holder (see Figure 9). The holder is fitted with two spring mounted pogo pins that are intended to provide the electrical contact to the LEDs through the matching holes located in the back plane of the cover.

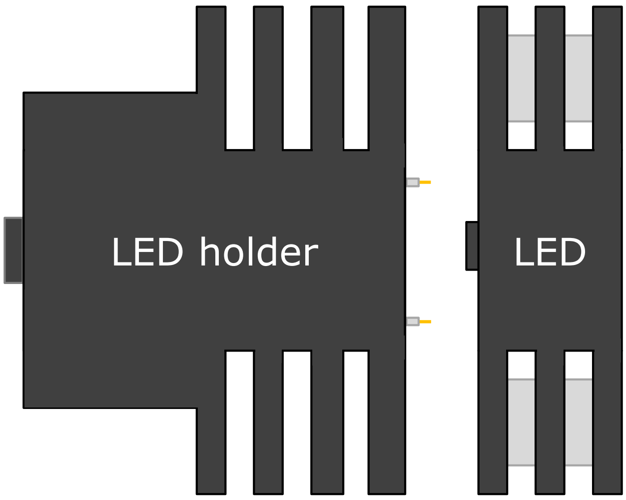


Figure 9 – The LED cover and the holder

To attach the cover to the holder, align the pogo pins with the matching holes in the back plane of the cover. The three screws located in the cover can be used to tighten the cover onto the holder.

Note

When tightening these screws, do not tighten one screw at a time but alternate between both screws in order to distribute the traction on each screw evenly. Do not over tighten the screws.

No soldering is required. Connecting the LEDs through pogo pins allows for a quick change of the wavelength of the light source, by simply replacing one cover by another.

Warning

Do not exchange the LED cover while the driver is in operation. Always power off the LED Driver before exchanging the LED cover.

The LED cover and the holder are fitted with six radial cooling fins, which allow evacuation of the heat generated by the light source. The higher the light intensity or the driving current supplied to the LEDs by the LED driver, the hotter the holder becomes (see Figure 10).

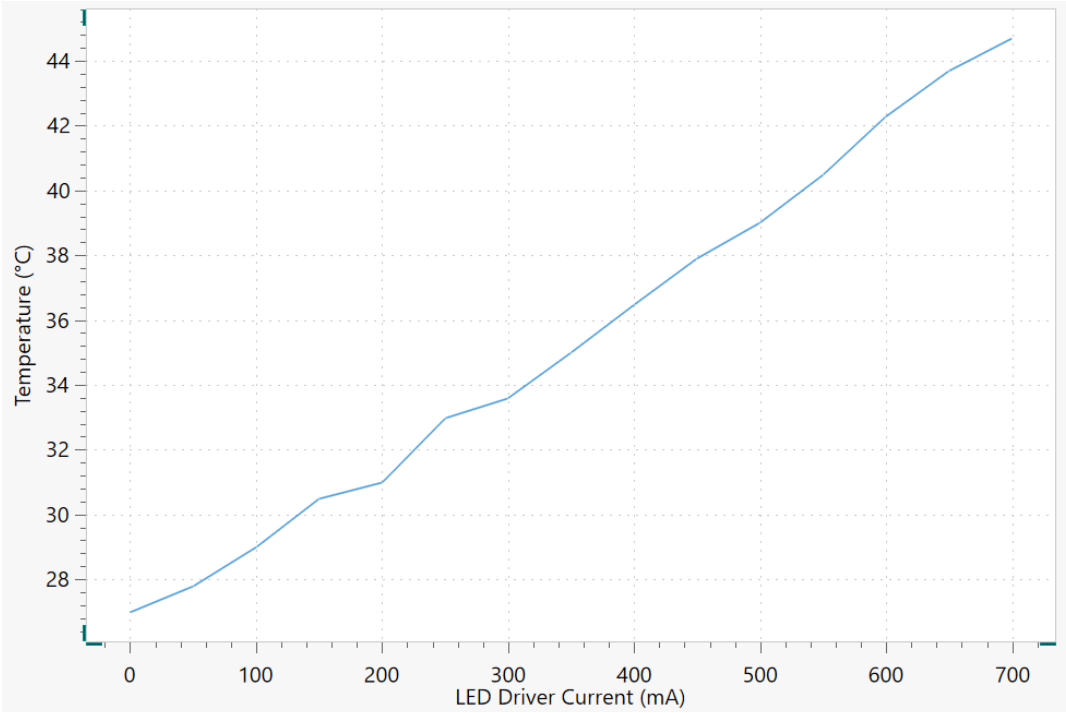


Figure 10 – Surface temperature of the LED cover in function of the driving current, in mA (measured with a 627 nm Red LED cover, settling time: 60 s)

Warning

When the light source is driven at high currents, the cooling fins can get very hot (up to 60 °C). Do not touch these fins and allow for enough cooling time to prevent injury.

Figure 11 shows the cover attached to the holder.

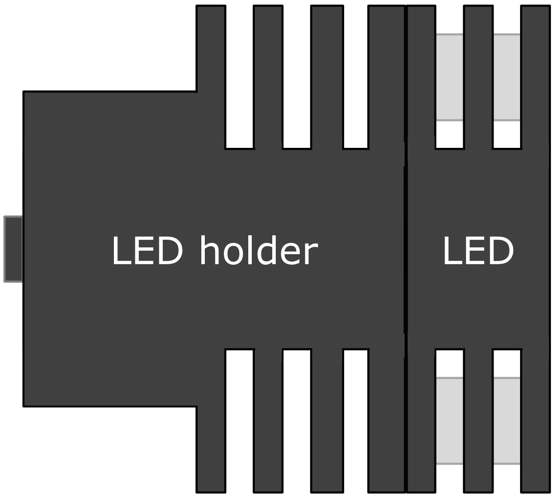


Figure 11 – The complete LED cover holder

5.3 – Autolab connections for light intensity control

Depending on the experimental setup, two connection schemes are possible between the LED driver and the Autolab:

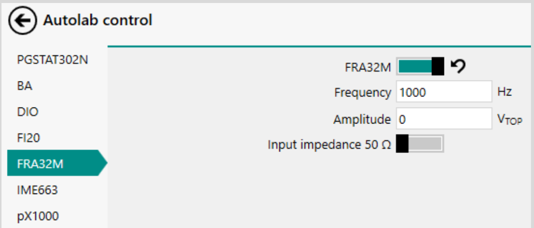
- **Measurements at constant light illumination:** these measurements are performed with the LED driver set to a fixed user-defined light intensity. The light source intensity can be changed during the experiment, but no additional modulation is used. For this setup, only the DAC 1 input of the driver is used. The → FRA V input **must be** shorted by a 50 Ω terminator plug (see Section 5.3.1 for more information).
- **Measurements at modulated light illumination:** these measurements are performed with the LED driver set to a fixed user-defined light intensity with an additional low amplitude intensity modulation. For these measurements, the DAC 1 and FRA V inputs are both connected to the Autolab (see Section 5.3.2 for more information).

Note

For modulated light illumination measurements, the monitor cable is required. This cable is provided with the N series Autolab and is optional for the Autolab PGSTAT204/M204 (see Figure 12).

Note

For measurements done with modulated light intensity (e.g., IMVS, IMPS), make sure that the external output of the FRA2 or FRA32M module is switched ON in the Autolab Control command in the procedure, before the measurement command starts.



Also, the external output of the FRA2 or FRA32M must be switched OFF in the Edit End Status of the procedure.

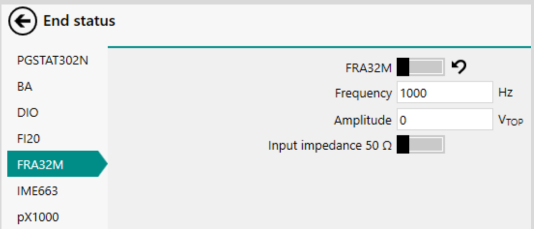


Figure 12 – The monitor cables for the N series Autolab (top), for the PGSTAT101 (middle) and for the Autolab PGSTAT204/M204/M101 (bottom)

Note

The PGSTAT204 and M204 module are not supplied with a monitor cable. This cable must be ordered separately (article code: **CABLE.MONITOR.MAC**). Please contact Metrohm Autolab (autolab@metrohm.com) or your local distributor for more information.

5.3.1 – Connections without a FRA module

Figure 13 shows the overview of the connections to the LED driver when no FRA module (FRA2 or FRA32M) is available.

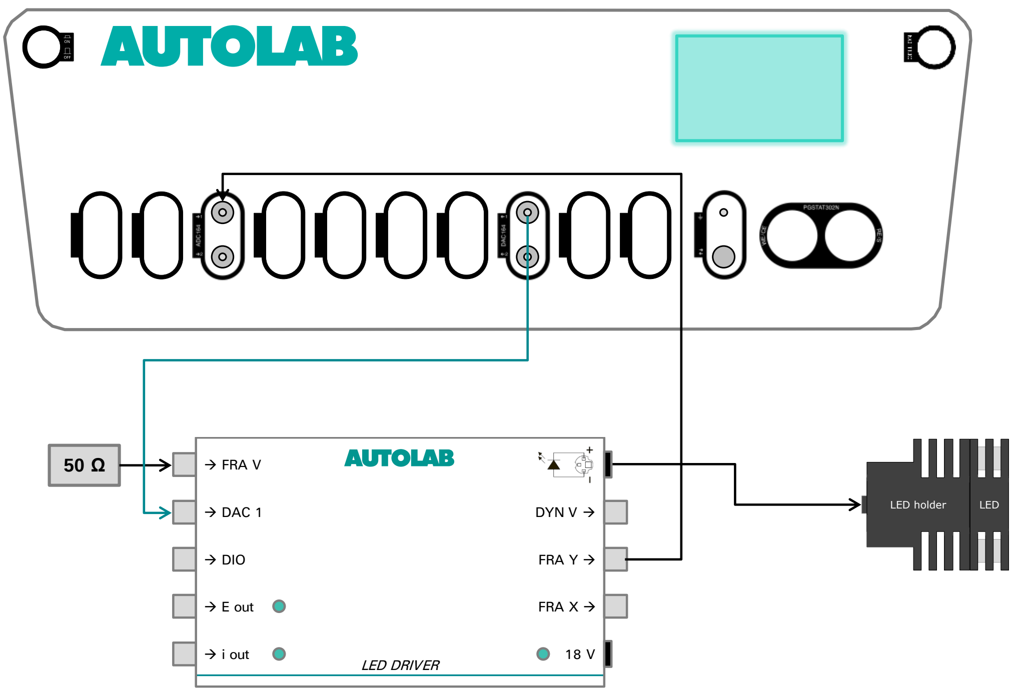


Figure 13 – Overview of the connections for constant illumination experiments

1. Connect the DAC164 ← 1 output[3] from the PGSTAT to the → DAC 1 input of the LED Driver.

[3] Or the Vout for the µAutolab, PGSTAT101/204 and M101.

2. Connect the FRA Y → output of the LED Driver to the ADC164 → 1 input[4] of the PGSTAT.
3. The → FRA V input **must be shorted** with the provided 50 Ω termination plug (see Figure 14).

Figure 14 – A 50 Ω terminator plug must be used to short the → FRA V input when this input is not used

Note

If a FRA2 or FRA32M module is present, it is possible to replace the 50 Ω terminator plug by a BNC or SMB cable from the ← V connector on the front panel of the FRA module and the → FRA V input.

5.3.2 – Connections with FRA2 module (Autolab N series)

Figure 15 shows the overview of the connections to the LED driver in combination with the FRA2 module.

[4] Or the Vin for the μAutolab, PGSTAT101/204 and M101.

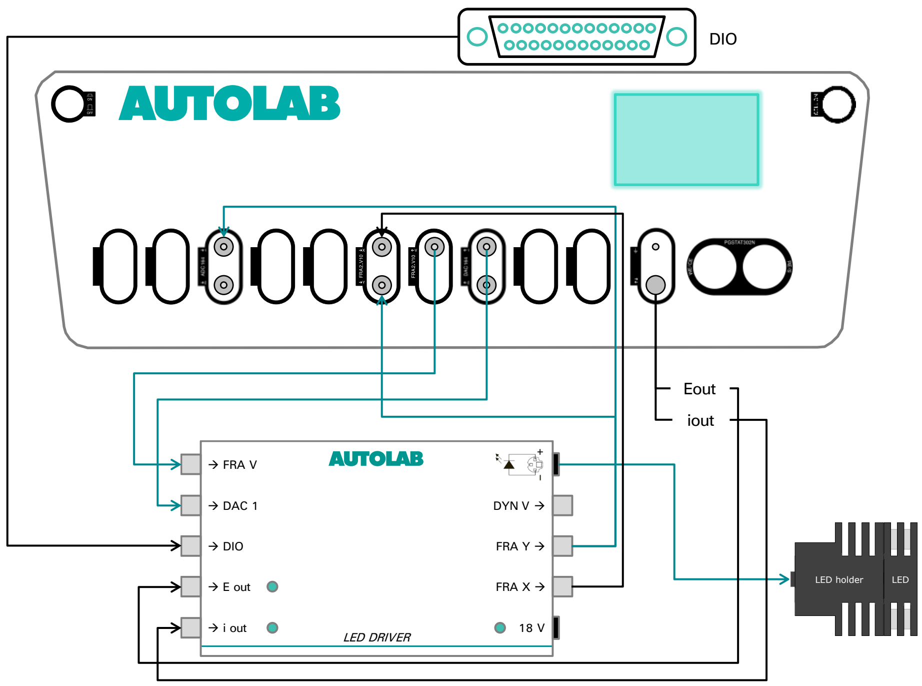


Figure 15 – Overview of the connections for modulated illumination experiments (FRA2)

1. Connect the DAC164 ← 1 output from the PGSTAT to the → DAC 1 input of the LED Driver.
2. Connect the ← V output of the FRA2 module to the → FRA V input of the LED driver.
3. Connect the FRA Y → output of the LED Driver to the ADC164 → 1 input of the PGSTAT and to the FRA → Y input, using the BNC splitter.
4. Connect the FRA X → output of the LED Driver to the → X input of the FRA2 module.
5. Connect the Eout and iout provided by the monitor cable to the → E out and → i out inputs located on the LED Driver.
6. Connect the provided DIO cable (25 pins) to the back of the Autolab, using connector P1 or P2. Connect the provided 2 m BNC cable from the DIO cable to the → DIO input on the LED Driver.

5.3.3 – Connections with FRA32M (Autolab N series)

Figure 16 shows the overview of the connections to the LED driver in combination with the FRA32M module.

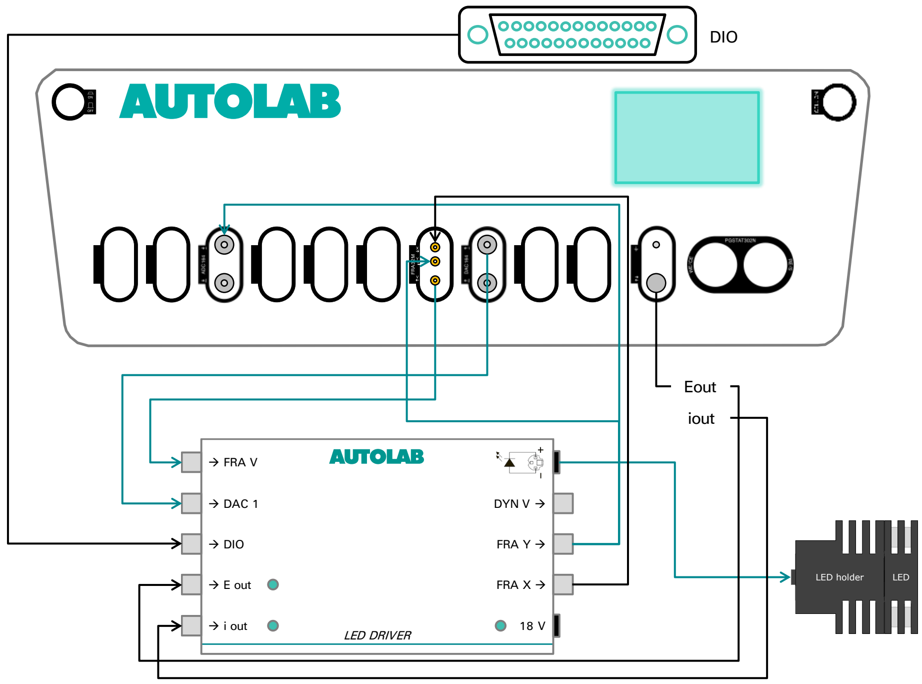


Figure 16 – Overview of the connections for modulated illumination experiments (FRA32M)

1. Connect the DAC164 ← 1 output from the PGSTAT to the → DAC 1 input of the LED Driver.
2. Connect the ← V output of the FRA32M module to the → FRA V input of the LED driver.
3. Connect the FRA Y → output of the LED Driver to the ADC164 → 1 input of the PGSTAT and to FRA → Y input, using the BNC splitter.
4. Connect the FRA X → output of the LED Driver to the → X input of the FRA32M module.
5. Connect the Eout and iout provided by the monitor cable to the → E out and → i out inputs located on the LED Driver.
6. Connect the provided DIO cable (25 pins for the N Series PGSTAT) to the back plane of the Autolab, using connector P1 or P2. Connect the provided 2 m BNC cable from the DIO cable to the → DIO input on the LED Driver.

5.3.4 – Connections with FRA32M (Autolab PGSTAT204, M204)

Figure 17 shows the overview of the connections to the LED driver in combination with the FRA32M module for a PGSTAT204. The connections for the M204 multi Autolab are similar and can be easily inferred from the connections for the PGSTAT204.

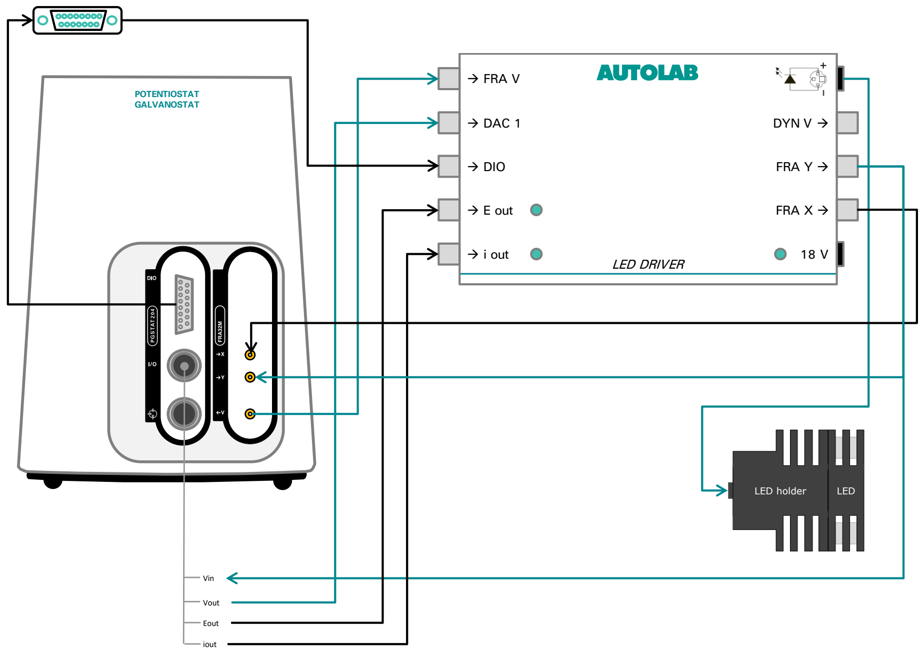


Figure 17 – Overview of the connections for modulated illumination experiments (PGSTAT204/FRA32M)

1. Connect the V out output from the monitor to the → DAC 1 input of the LED Driver.
2. Connect the ← V output of the FRA32M module to the → FRA V input of the LED driver.
3. Connect the FRA Y → output of the LED Driver to the Vin of the monitor cable and the FRA → Y input, using the BNC splitter.
4. Connect the FRA X → output of the LED Driver to the → X input of the FRA32M module.
5. Connect the Eout and iout provided by the monitor cable to the → E out and → i out inputs located on the LED Driver.

6. Connect the provided DIO cable (15 pins for the PGSTAT204) to the front panel of PGSTAT204. Connect the provided 2 m BNC cable from the DIO cable to the → DIO input on the LED Driver.

5.3.5 – DIO control of the Eout/iout inputs of the LED Driver

The Eout and iout outputs signals provided by the monitor cable are used for the IMVS and IMPS, respectively. By feeding these two signals into the driver, the user can pass either one of these signals to the FRA X output located on the right-hand side of the driver, using the either one of the compatible DIO cables (see Figure 18).

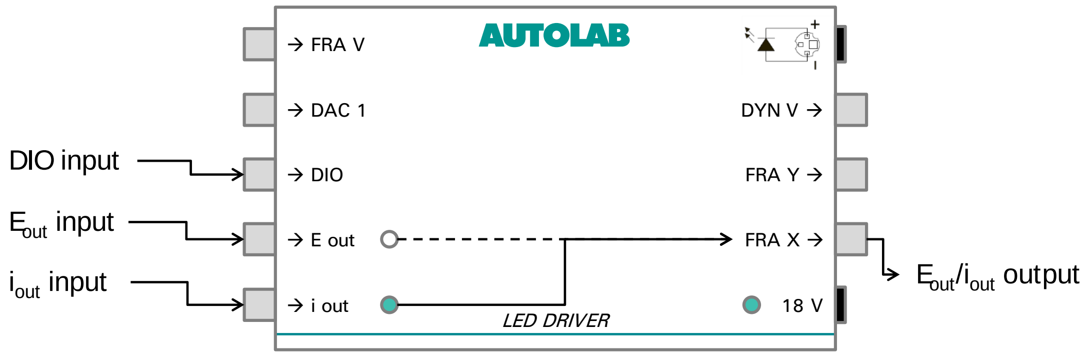


Figure 18 – Overview of the feedthrough connections for E_{out} and i_{out}

Two different DIO cables are supplied with the LED Driver:

- For all the supported Autolab instruments, except the PGSTAT204 and Multi Autolab M204, a male DIN25 to female BNC cable is supplied with the Optical Bench (see Figure 19). This cable can be connected to either P1 or P2 of the instrument.

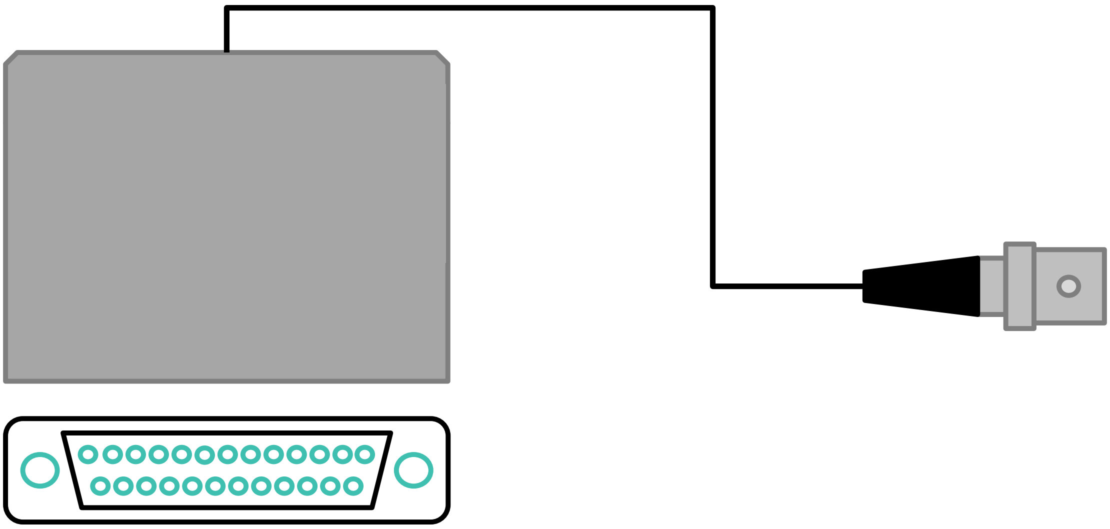


Figure 19 - DIO cable for the Series 7 and Series 8 PGSTAT instruments

- For the PGSTAT204 and M204, a male DIN15 to female BNC cable is available (see Figure 20). This cable can be connected to the DIO port of the instrument.

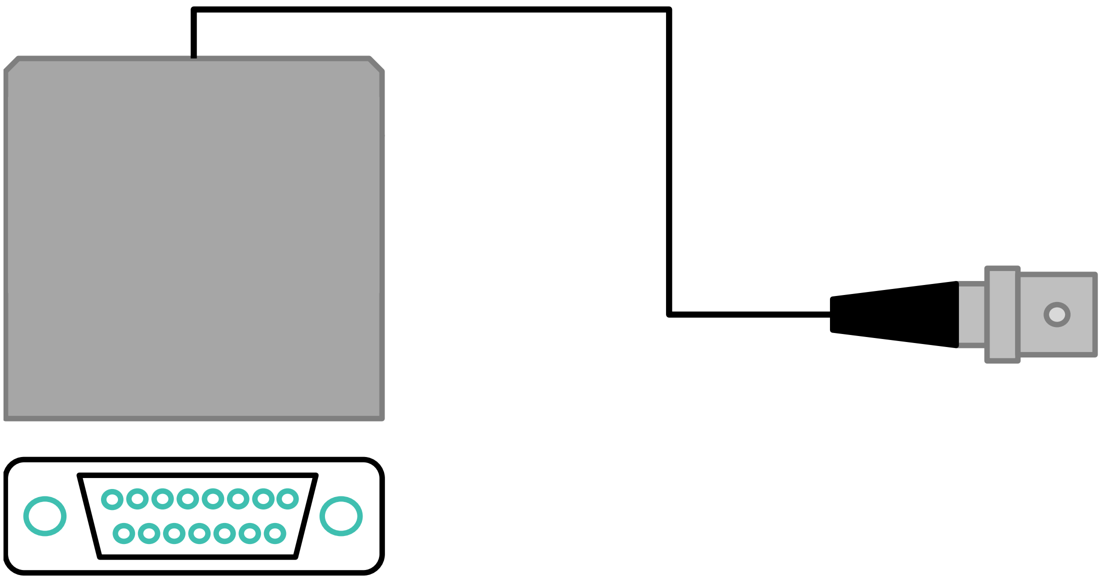


Figure 20 - DIO cable for the Autolab PGSTAT204 and M204

By setting the DIO input to high or low[5], the switch located in the driver can be toggled remotely. The status LEDs located on the left-hand side of the driver will indicate the selected input (see Figure 21).

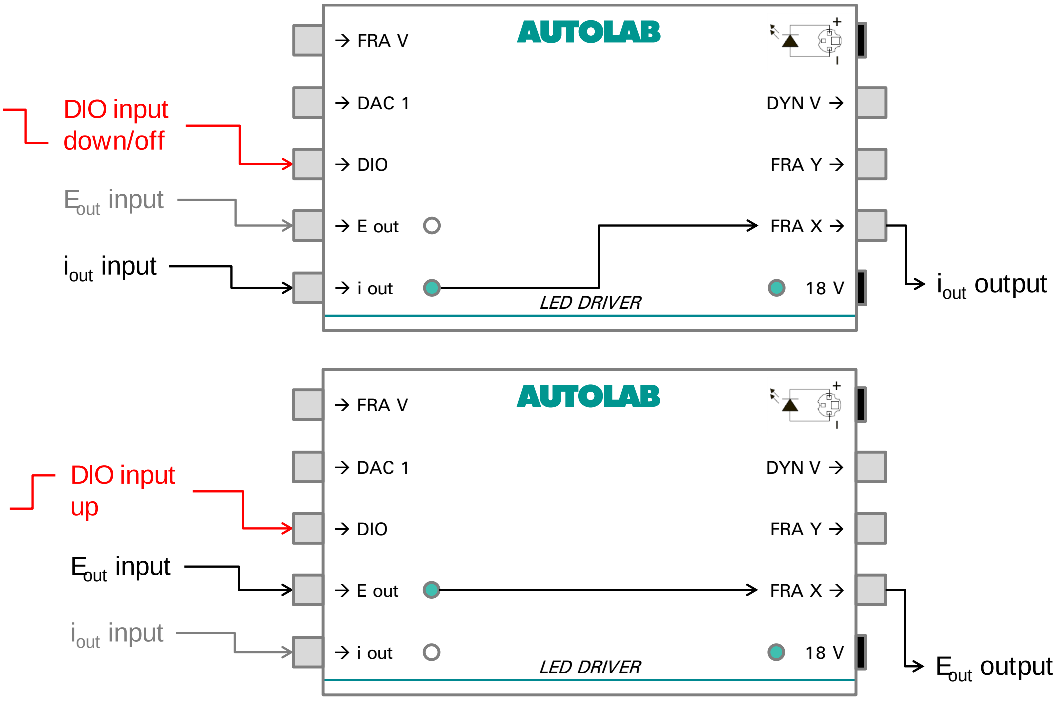


Figure 21 – The i_{out}/E_{out} switch can be set remotely through the DIO input (top, DIO status UP or disconnected – i_{out} selected, bottom, DIO status DOWN – E_{out} selected)

By default, when no connection to the DIO is present, the i_{out} is always selected, indicated by the status LED on the left-hand side. The switch can also be toggled through an analog voltage. Supplying more than 2.5 V on this DIO input triggers the switch.

[5] Please refer to the NOVA user manual for more information on the control of the DIO port(s) of the Autolab.

Note

Using the built in E_{out}/i_{out} switch is not mandatory for IMVS/IMPS measurements, but is recommended because it facilitates the wiring schemes.

6 – Software control

The Autolab LED driver is intended to be controlled by the NOVA software. The control of the driver is performed through the use of analog and/or digital settings:

- Analog control of the light intensity and modulation, if applicable.
- Digital control of the E_{out}/i_{out} through the DIO port.

This section provides details on the light intensity control.

6.1 – Hardware setup

The hardware setup of NOVA needs to be adjusted in order to indicate that the LED driver is being used in combination with the PGSTAT. Open the Hardware Setup from the Tools menu in NOVA and select the External module in the Additional Module(s) in the middle frame of the dialog (see Figure 22).

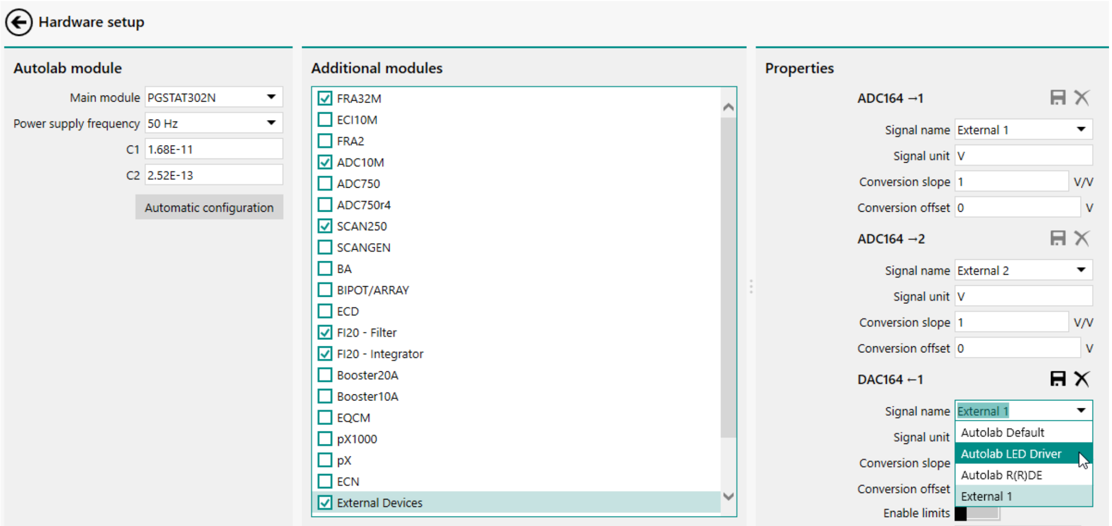


Figure 22 – Specifying the settings for the External module

Using the Signal drop-down boxes in the Hardware Setup dialog, specify the *Autolab LED Driver* setting for analog input and the analog output (see Figure 23).

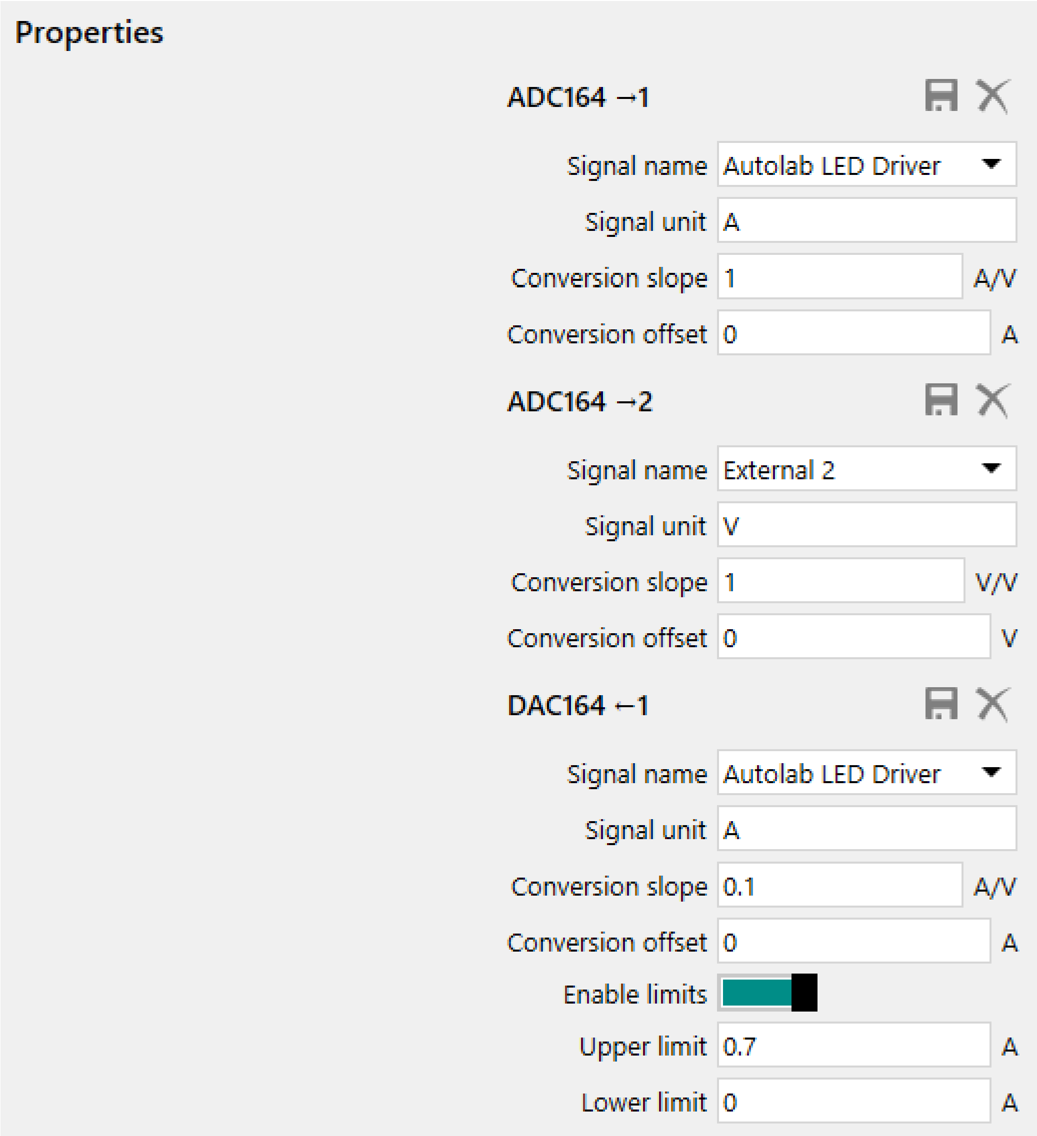


Figure 23 – Specifying the *Autolab LED Driver* in the External module setting

6.2 – Control of the DC light intensity

Using the settings defined in the Hardware setup, as shown in Section 6.1, it is possible to control the DC light intensity provided by the LED Driver in NOVA, either manually or within a NOVA procedure.

Warning

Do not look directly into the light generated by the LED array.

6.2.1 – Manual control of the DC light intensity

The LED Driver current can be adjusted manually by using the Autolab Manual control window. To display the Manual control panel see Figure 24.

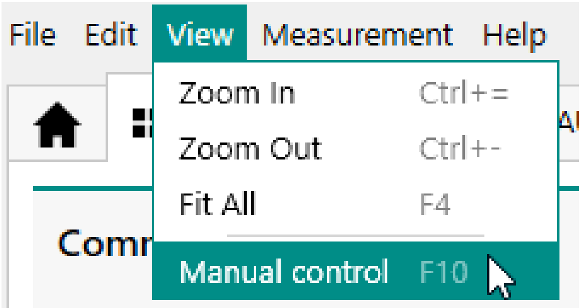


Figure 24 – Selecting the Manual control option from the View menu to display the Autolab control window

The External device control panel indicates the actual LED Driver current (see Figure 25).

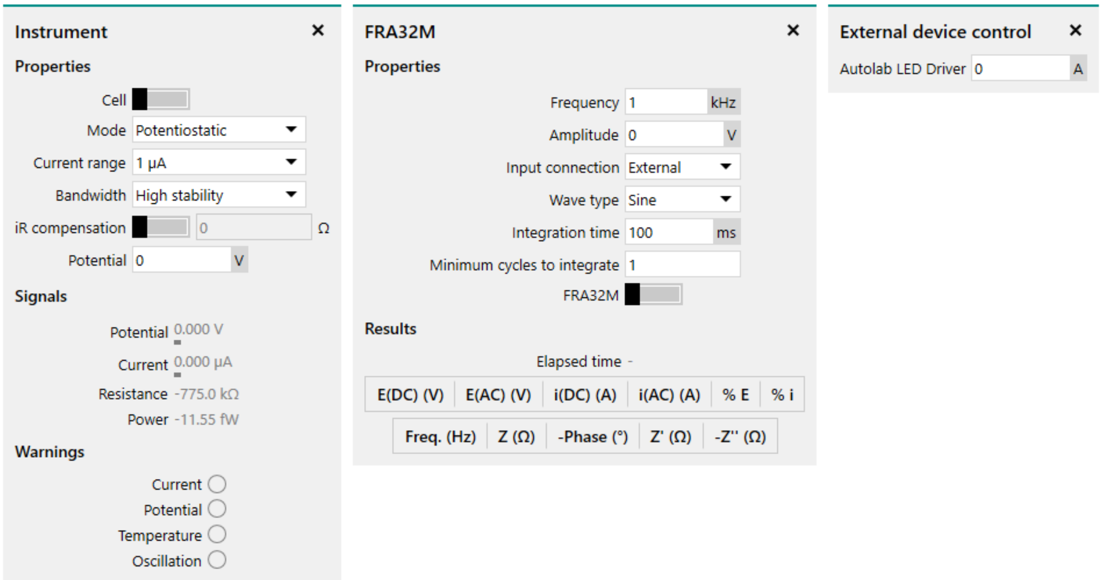


Figure 25 – The Autolab control window with the External device control panel

To define the LED Driver current, click the Autolab LED Driver label in the Autolab display window and specify the required value. Press the enter key to validate the value. The LED will immediately settle to a constant light output (see Figure 26).

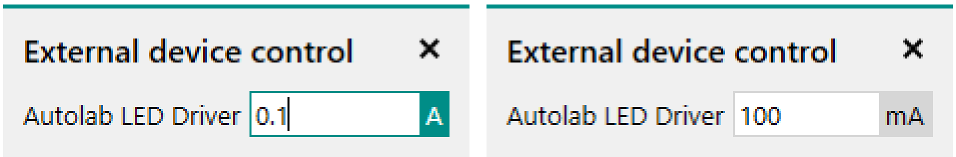


Figure 26 – Setting the driving current in the Autolab display window

The driving current can be readjusted at any time using the method described in this section.

6.2.2 – Settings for the DC light intensity

This setting is defined in the software through the *Autolab Control* command, available from the Measurement – general group of commands (see Figure 27).

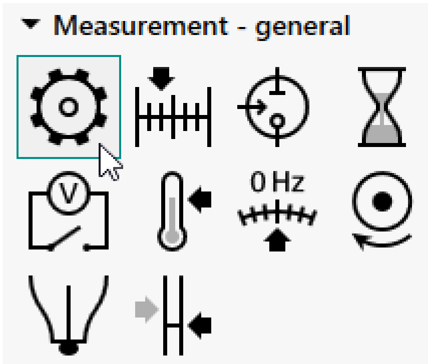


Figure 27 – The *Autolab Control* command is used to set the DC light intensity

The *Autolab Control* has the following parameter:

Parameter	Description
Autolab LED Driver Value	The Autolab LED Driver current, in A.

To modify the LED Driver current, click the More button located in the properties of the *Autolab Control* command (see Figure 28).

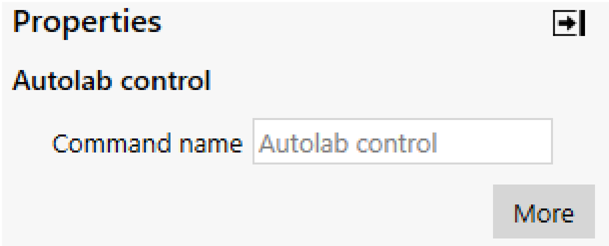


Figure 28 – The settings required for the *Autolab Control* command can be defined by clicking the More button

The Autolab LED Driver current can be specified, in A, in the Advanced part of the main PGSTAT section (see Figure 29).

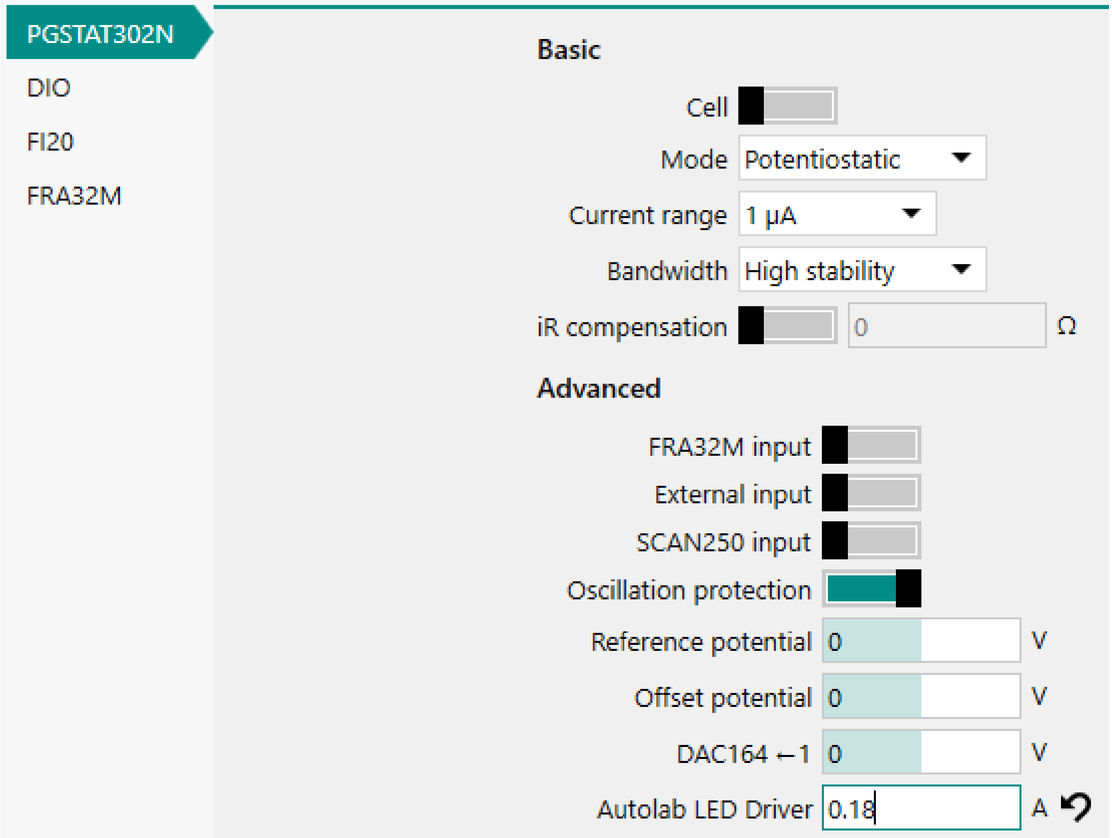


Figure 29 – Setting the LED Driver current in the Advanced part of the main PGSTAT section of the Autolab control command

It can also be specified by sliding the provided slider in the same editor (see Figure 30).

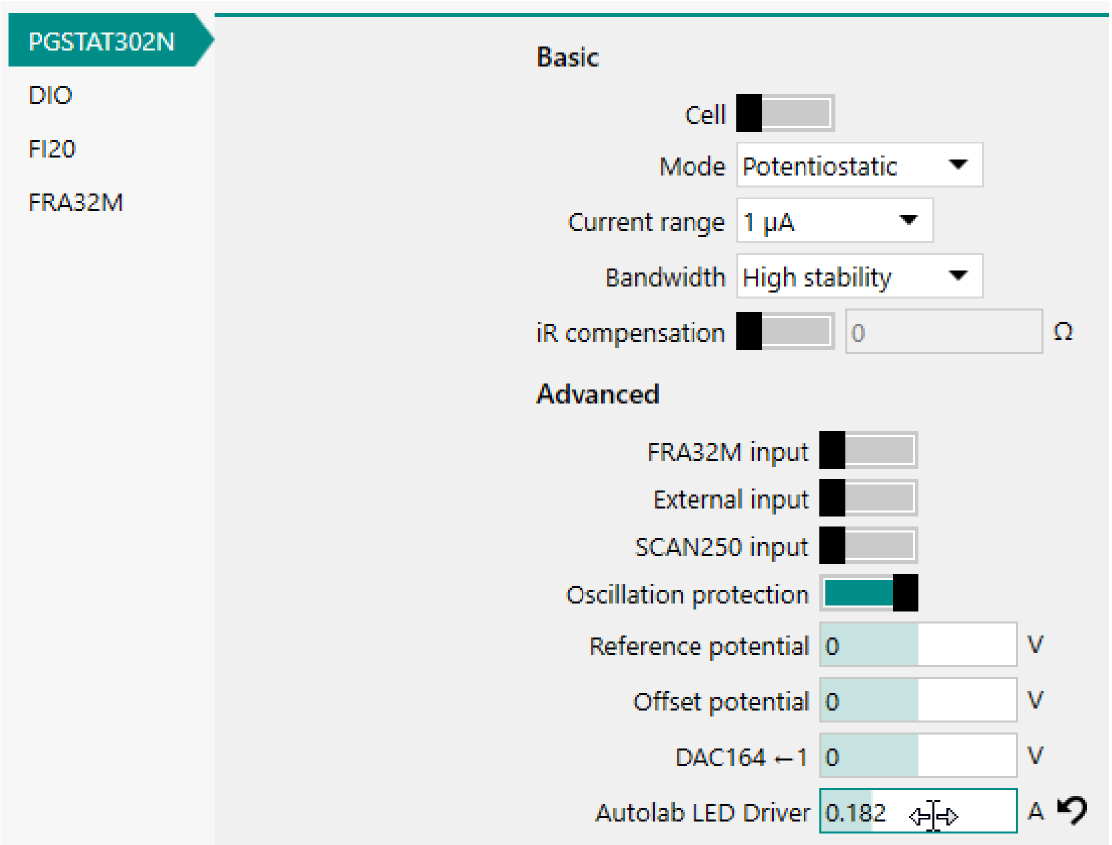


Figure 30 – Adjusting the driving current using the slider provided in the Autolab control dialog

Click the OK button to close the window. The procedure will be updated displaying the value specified in the Autolab control window (see Figure 31).

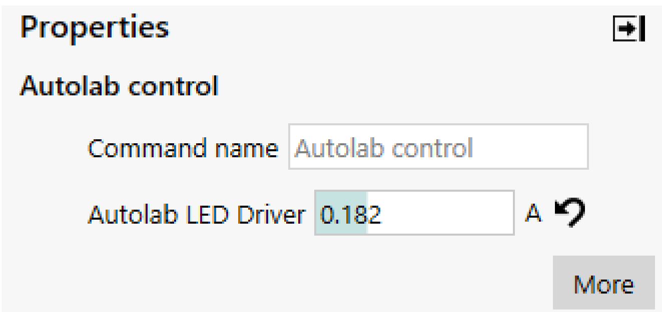


Figure 31 – The updated properties of the *Autolab control* command

The parameter provided by the *Autolab Control* command can be linked to other commands, like an *Input box* command or a *Repeat for each value* command, as show in Figure 32.

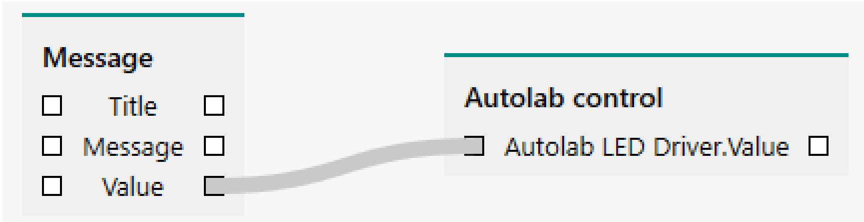


Figure 32 – Linking the driving current to an *Input box* command

6.2.3 – Settings for the DIO control

The → DIO BNC connector can be used to switch remotely between the → E out or →i out inputs on the LED Driver, using the DIO port(s) provided by the Autolab, as explained in Section 5.3.

To control the DIO of the Autolab, the *Autolab control* command[6] must be used to initialize the DIO port used in the experiment. The DIO port used must be initialized at the beginning of the procedure.

Note

Pin #1, located on DIO port A is used to control the switch.

To initialize the DIO port A, located on either P1 or P2, the port has to be set on, using the toggle provided in the Autolab control window (see Figure 33).

[6] More information on the settings of the DIO ports and on the Autolab control command can be found in the NOVA user manual.

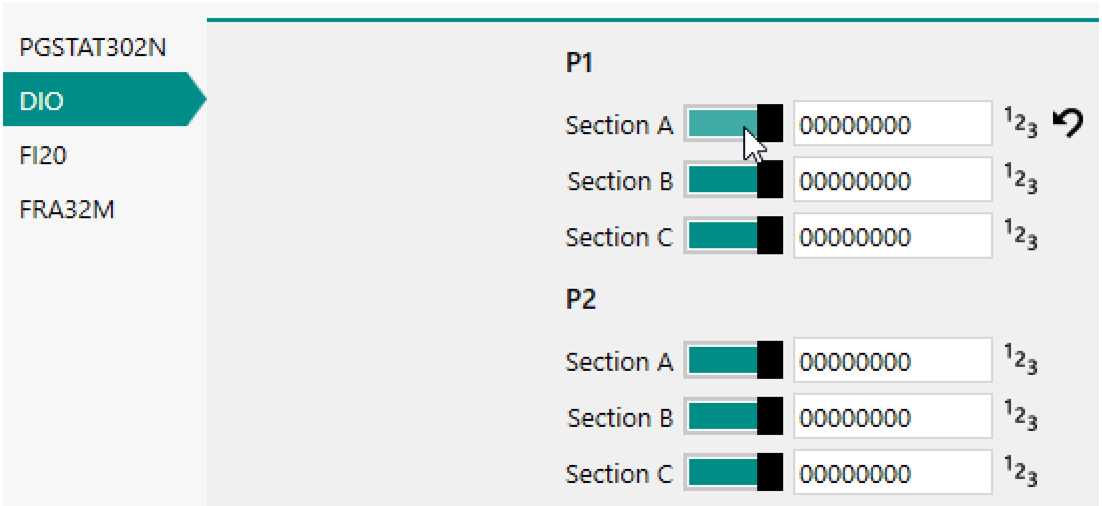


Figure 33 – Setting on Port A of connector P1

After the port is initialized, the status of Pin #1 can be set at any time to high (5 V) or low (0 V), using the *Autolab control* command.

- **Low (00000000):** when pin #1 is set to low status (0 V), the → E out input is active. This is also the default status when nothing is connected to the → DIO input of the LED Driver.
- **High (00000001):** when pin #1 is set to high (5 V), the → i out input is active. The switch will be kept in this position as long as pin #1 is set to high.

The status of pin #1 of the initialized port can be changed at any time using the Autolab control window. The status can be set as a decimal value (0 or 1), or as a binary string (00000000 or 00000001). The status defined is permanent until changed (see Figure 34).

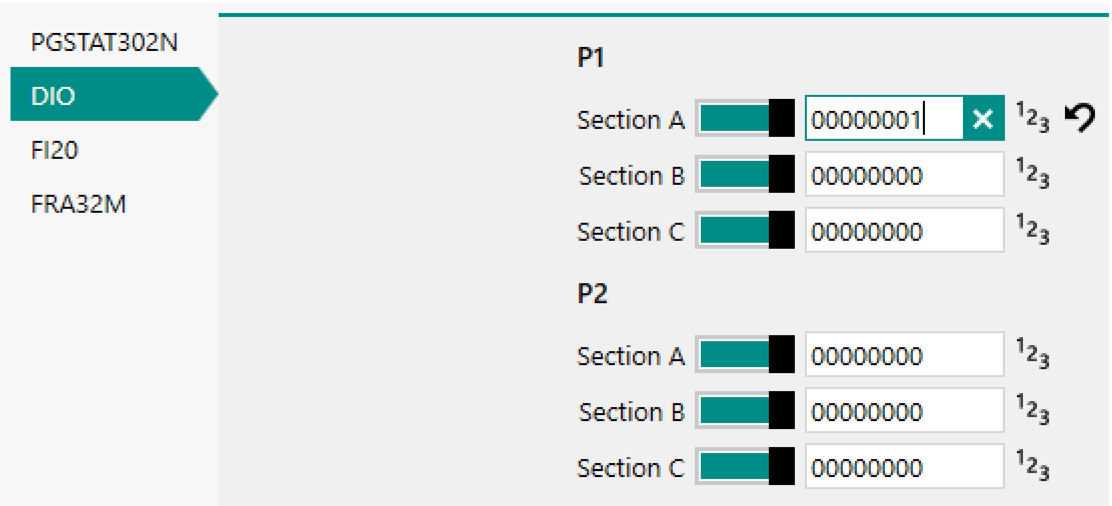


Figure 34 – Setting the status of pin #1

For the PGSTAT204 or M204 module, the same settings are used. However, in this case, only a single port is available (see Figure 35). Port A of the DIO connector of the, PGSTAT204 or M204 does not have to be initialized since it is hardwired to output.

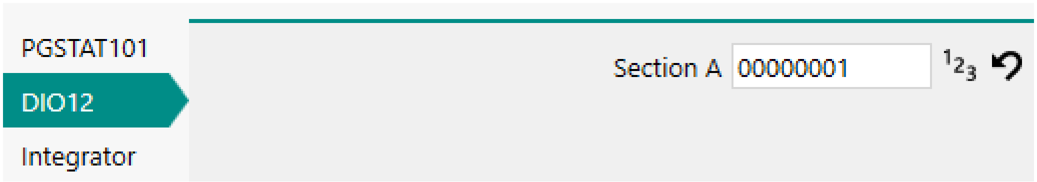


Figure 35 – Setting the status of pin #1 for the PGSTAT204 or M204 module

7 – Light source calibration

Quantitative results can only be obtained after the calibration of the light source. This can be done by using a calibrated photodiode exposed to the light source at a controlled distance on the optical bench. The calibration can be performed with the Autolab or with an external digital multimeter. In this manual, the procedure used in combination with the Autolab is described.

The Optical Bench is supplied with a calibrated photodiode, embedded into a cylindrical holder (see Figure 36). The measurement range of the photodiode is from 350 nm to 1100 nm.

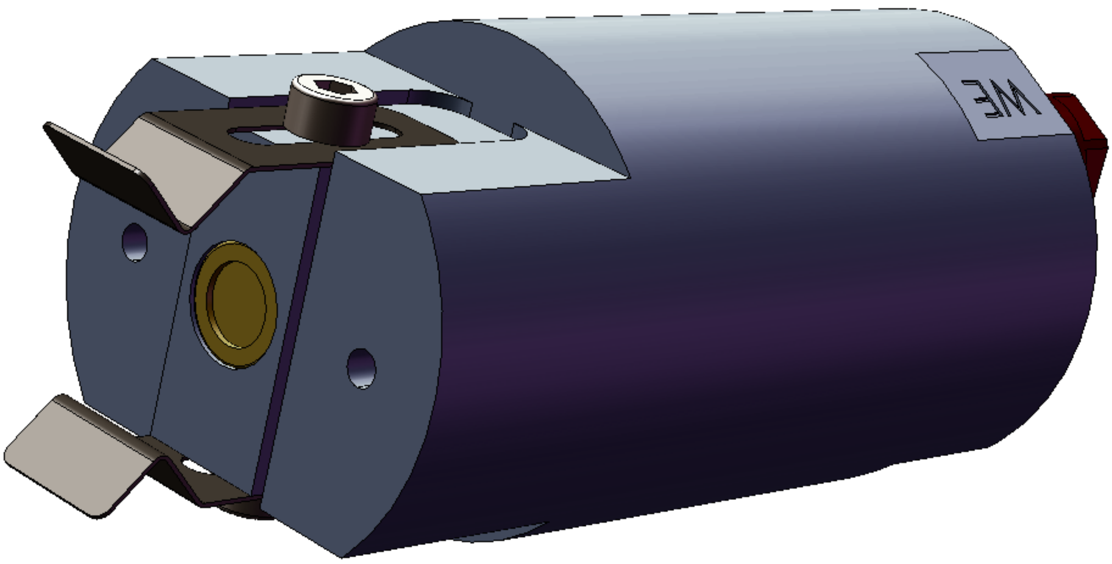


Figure 36 – The photodiode holder (front view)

The photodiode holder is fitted with three female 4 mm banana connectors, labeled WE (red), CE (black) and GND (green), respectively. These connectors are used to accommodate the cell connectors provided by the Autolab PGSTAT (see Figure 37).

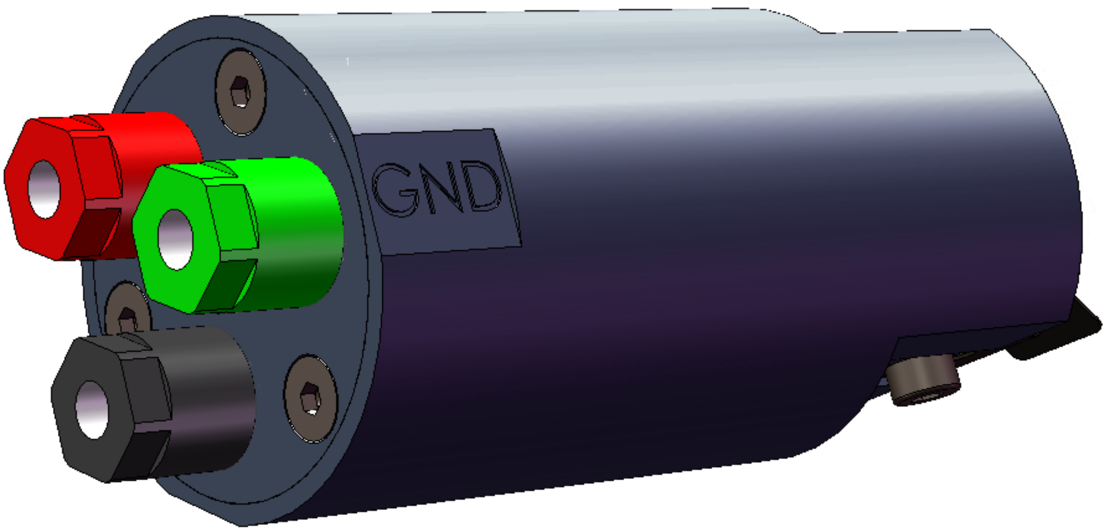


Figure 37 – The photodiode holder (back view)

The photodiode holder is also supplied with an additional plastic cover which can be mounted on top of the calibrated photodiode. This cover is designed to hold a filter[7] to protect the photodiode from exposure to light intensities beyond the damage threshold.

Warning

The photodiode will be irreversibly damaged when exposed to a light intensity of 100 mW/cm^2 or more.

The calibration procedure requires the calibrated photodiode to be placed at the required distance from the light source and to expose the photodiode to different light intensities (controlled by the driving current supplied by the LED Driver). The photodiode current can be converted to light intensity using the

[7] To protect the photodiode a reflective neutral density filter can be used. The size of the filter is 1.27 cm (1/2 inch).

conversion values reported in the calibration certificate of the calibrated photodiode supplied with the Optical Bench (see Figure 38).

Test Report

Model	FDS100-CAL	**Temperature**	25,0°C	**Test Date**	8-Jun-11
Serial No	11060821	**Humidity**	35%	**Tester**	Alexandra Ressel
Scan	CAL1				

λ [nm]	η [A/W]	λ [nm]	η [A/W]	λ [nm]	η [A/W]	λ [nm]	η [A/W]	λ [nm]	η [A/W]	λ [nm]	η [A/W]
350	6,95E-02	720	4,24E-01	1090	1,72E-01						
360	6,59E-02	730	4,35E-01	1100	1,37E-01						
370	6,38E-02	740	4,45E-01								
380	6,67E-02	750	4,56E-01								
390	7,27E-02	760	4,66E-01								
400	8,08E-02	770	4,76E-01								
410	9,02E-02	780	4,86E-01								
420	1,00E-01	790	4,96E-01								
430	1,10E-01	800	5,06E-01								
440	1,21E-01	810	5,16E-01								
450	1,31E-01	820	5,26E-01								
460	1,42E-01	830	5,35E-01								
470	1,52E-01	840	5,43E-01								
480	1,62E-01	850	5,52E-01								
490	1,72E-01	860	5,60E-01								
500	1,82E-01	870	5,68E-01								
510	1,93E-01	880	5,75E-01								
520	2,03E-01	890	5,83E-01								
530	2,14E-01	900	5,91E-01								
540	2,25E-01	910	5,99E-01								
550	2,35E-01	920	6,06E-01								
560	2,46E-01	930	6,13E-01								
570	2,58E-01	940	6,19E-01								
580	2,68E-01	950	6,25E-01								
590	2,79E-01	960	6,33E-01								
600	2,91E-01	970	6,36E-01								
610	3,02E-01	980	6,36E-01								
620	3,13E-01	990	6,31E-01								
630	3,24E-01	1000	6,22E-01								
640	3,35E-01	1010	6,04E-01								
650	3,47E-01	1020	5,73E-01								
660	3,57E-01	1030	5,28E-01								
670	3,70E-01	1040	4,68E-01								
680	3,81E-01	1050	3,99E-01								
690	3,92E-01	1060	3,23E-01								
700	4,03E-01	1070	2,59E-01								
710	4,14E-01	1080	2,12E-01								

Figure 38 – Calibration data provided on the calibration certificate

The calibration certificate provides a table of wavelengths (λ, in nm) and responsivity (η, in A/W). The responsivity values are reported in absolute values and should be normalized with respect to the active surface area of the photodiode (13 mm^2).

Using this strategy, it is relatively easy to establish a driving current-to-light intensity correlation, as shown in Figure 39. The data shown in this plot was obtained at a distance of 15 cm, using a 627 nm LED cover (Red).

The light intensity, P_{LED}, is calculated from the measured photodiode current, i_{SC}^{φ}, divided by the surface of the photodiode (0.13 cm^2) and divided by the responsivity reported in the calibration certificate (see Figure 38).

$$P_{LED} = \left(\frac{i_{SC}^{\varphi}}{A \cdot \eta}\right) = \left(\frac{i_{SC}^{\varphi}}{0.13\ cm^2 \cdot 0.318\ A/W}\right)$$

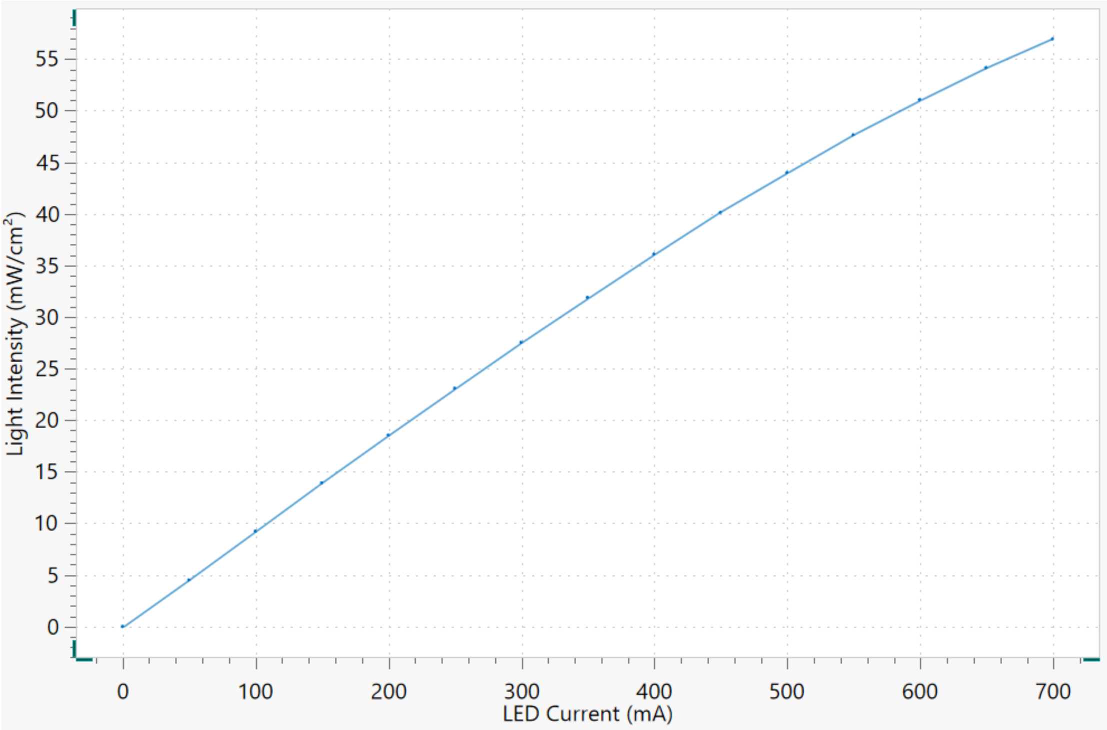


Figure 39 – Driving current to light intensity conversion curve, measured at 627 nm (Red)

To use the photodiode, connect the RE/CE leads from the PGSTAT to the CE plug on the photodiode holder and connect the WE/S to the WE plug on the photodiode holder. Connect the green ground plug embedded in the cell cables of the Autolab to the matching GND banana connector of the photodiode holder.

Note

The photodiode current must be measured at short-circuit conditions (0 V applied) and at known wavelength.

8 Cell holder

The calibrated photodiode holder is fitted with two sliding clamps that can be used to fix the cell onto the holder, provided that the cell is small enough to fit in this space. This holder is designed for a common form factor for experimental solar cells, like the one shown in Figure 39.

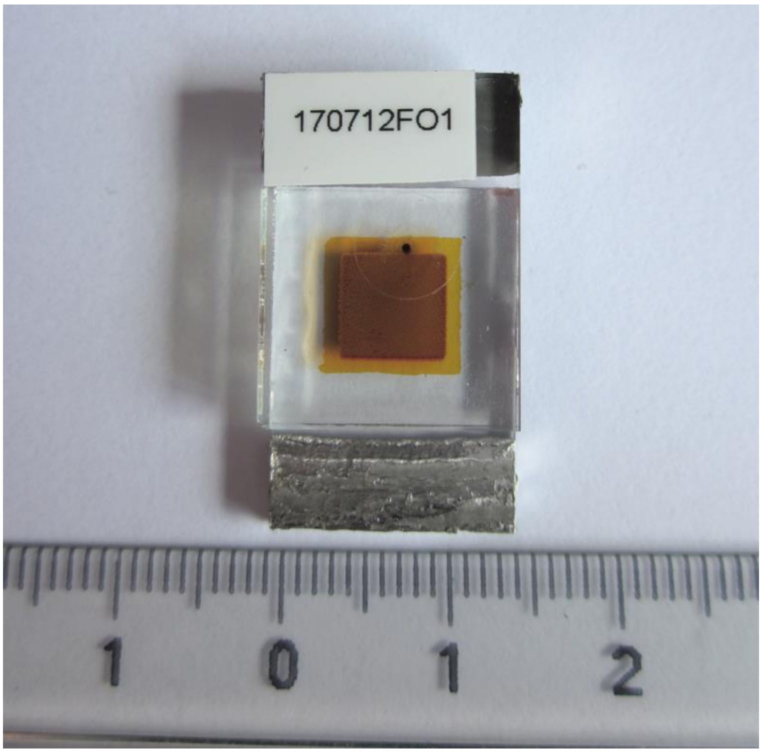


Figure 40 – Typical experimental cell design suitable for the Autolab Optical Bench

The clamps can be repositioned if necessary by loosening the two screws that hold them in place (see Figure 41).

Note

If the cell does not fit between the two clamps, an external holder can be used to place the cell close to the photodiode. Metrohm Autolab cannot anticipate all the possible cell configurations and it is left to the user to find a suitable solution, if necessary.

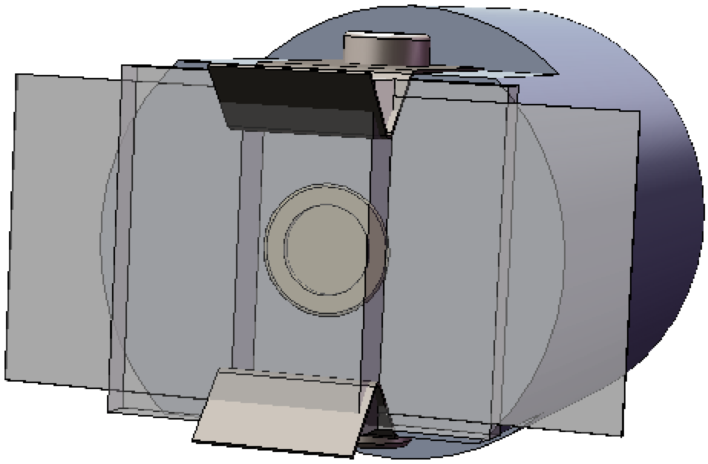


Figure 41 – The cell mounted on the photodiode holder

9 – Experiment description

This section provides a description of different experiments that can be carried out with the Optical Bench, using the information provided in the previous sections.

9.1 – Cell connections

Photovoltaic devices are usually characterized in the so-called two electrode mode. In this mode, the Autolab PGSTAT is connected to both the anode and the cathode using the CE/RE and WE/S, respectively (see Figure 42).

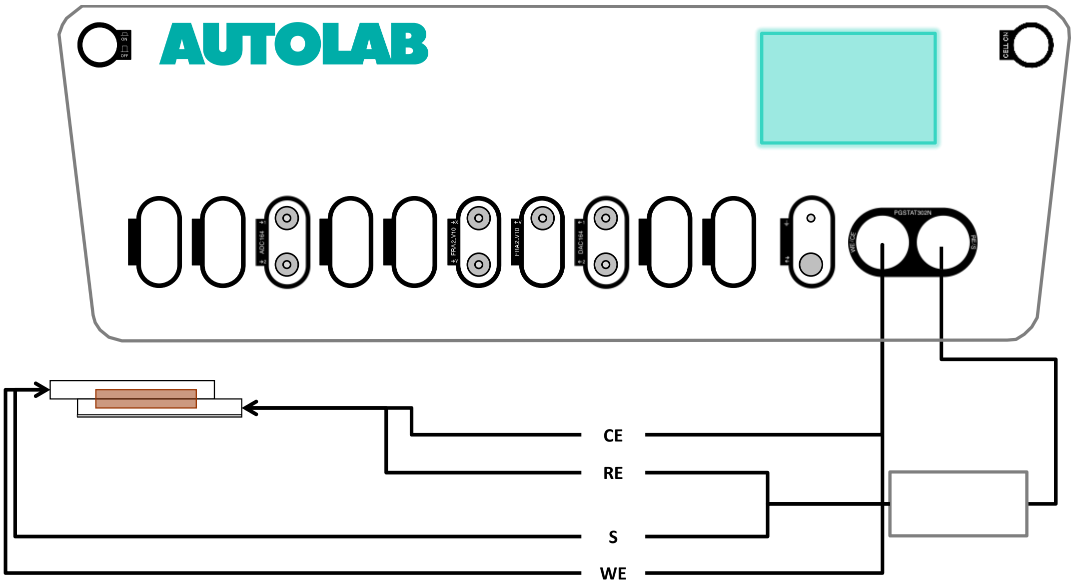


Figure 42 – Overview of the connections to the cell

In this configuration, the potential difference across the cell is measured between the RE and the S, using the differential amplifier electrometer of the Autolab PGSTAT. The current is measured between the CE and the WE.

Note

By convention, the potential of the cell under operating conditions is always positive and the current, under operating conditions is always negative.

9.2 – DC measurements at constant illumination

DC measurements at constant illumination are the simplest measurements possible with the PGSTAT in combination with the LED Driver. During these experiments, the light source is set to a user defined driving current to provide constant illumination on the cell, while the PGSTAT measures the i/V curve of the cell. The i/V curve can be recorded either in potentiostatic or in galvanostatic mode.

The following DC measurements are possible:

- Measurement of the i/V curve of the cell
- Charge extraction measurements

9.2.1 – i/V and power curves at constant illumination

Characterization of solar cells usually requires the determination of i/V curves. As the potential of the cell is scanned from 0 V (short circuit conditions) to the open circuit potential, the current changes from the maximum value (short-circuit current, i_{SC}) to OCP (0 A). A typical example is shown in Figure 43.

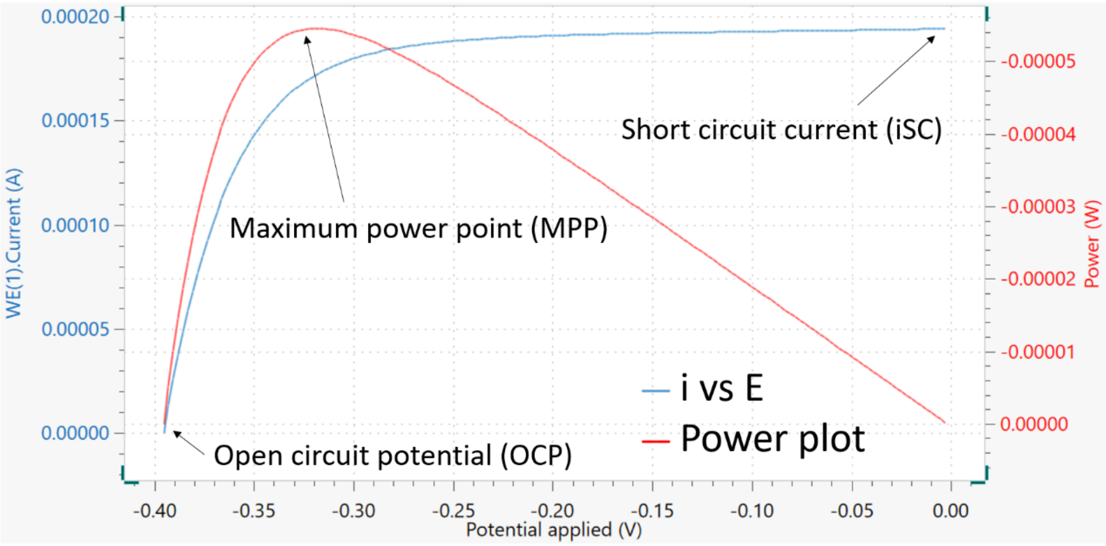


Figure 43 – i/V curve (blue) and power curve (red) obtained with the PGSTAT in combination with a 627 nm LED. The light intensity is 30 mW/cm², the cell is a Dye Sensitized Solar Cell

The i/V curves and power curves can be recorded at different light intensities, by varying the driving current. If the conversion of driving current to light intensity in W/cm² is known, then the light intensity to which the cell is exposed can be controlled directly (see Section 7).

From the short circuit current (i_{SC}), open circuit potential (V_{OCP}) and the maximum power point (P_{MAX}), the fill factor (FF) of the cell can be calculated.

$$FF = \left(\frac{i_{MAX} \cdot V_{MAX}}{i_{SC} \cdot V_{OCP}}\right) = \left(\frac{P_{MAX}}{i_{SC} \cdot V_{OCP}}\right)$$

$$FF = \left(\frac{54.602\ \mu W}{194.49\ \mu A \cdot 0.396\ V}\right) = 0.709$$

From the light intensity (P_{IN}), the wavelength (λ) and the short-circuit current density (j_{SC}) the incident photon-to-current conversion efficiency (ICPE) can be calculated.

$$IPCE = 1239\left(\frac{j_{SC}}{P_{IN} \cdot \lambda}\right) \cdot 100$$

$$IPCE = 1239\left(\frac{0.778\ mA/cm^2}{30\ \frac{mW}{cm^2} \cdot 627\ nm}\right) \cdot 100 = 5.12\%$$

9.2.1.1 – Software implementation

DC measurements with the PGSTAT in combination with the LED Driver can be performed using a NOVA procedure. The light intensity can be controlled using the *Autolab Control* command, as explained in Section 6.2.2.

This command can be combined in a measurement with a *LSV staircase* or *LSV staircase galvanostatic* command, depending on the experimental conditions.

Under potentiostatic condition, it is common practice to start the scan a 0 V (short-circuit conditions) and stop the scan when the current becomes zero (open circuit conditions). An example of a procedure for this type of measurement is shown in Figure 44.

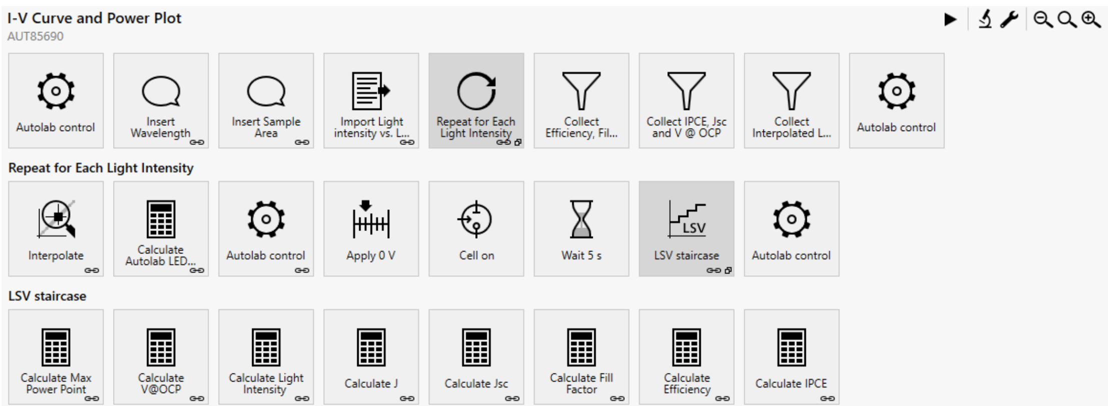


Figure 44 – An example of a procedure used to measure the i/V curve of a cell

Note

A cutoff condition can be used to detect the potential at which the current changes polarity (see Figure 45).

Cutoffs

Signal	When	Value	Action	Only once	Detections	Link as
WE(1).Current	<	0	Stop command		1	

Figure 45 – A cutoff condition can be used to detect the point where the current changes polarity

9.2.1.2 – Possible refinements

It is possible to combine this sequence with other NOVA commands in order to build a complete measurement sequence. Since the description of such a sequence is left to the requirements of the users, it falls outside of the scope of this document. A few tips are provided below:

- A *Repeat for each value* command, with the set current values defined in the values sequence of the repeat command can be used in combination with the *Autolab Control* command. This will repeat the whole measurement sequence for each pre-defined value (see Figure 44).
- The *Calculate signal* command can be added to the sequence in order to rectify the WE(1).Power signal (which is negative in the raw measurements). This is also shown in Figure 44.

9.3 – AC measurements at constant illumination

The LED Driver can be used in combination with the PGSTAT/FRA to obtain the electrochemical impedance spectrum of the device under test (DUT) under constant illumination. Electrochemical impedance measurements can be performed while illuminating the cell, in potentiostatic mode or galvanostatic mode. Typically, these measurements are performed at open circuit or at short-circuit.

The measurement strategy is the same as for a DC measurement at constant illumination. The light intensity is set to a fixed value using the *Autolab Control* command.

Warning

When performing AC measurements at constant illumination, the → FRA V input of the LED Driver must be shorted using the supplied 50 Ω terminator plug as explained in Section 5.3.1.

9.3.1 – EIS measurements at constant illumination

The standard Autolab procedure FRA impedance potentiostatic and FRA impedance galvanostatic procedures can be used to perform EIS measurements at constant illumination. The *Autolab Control* command can be added to the preconditioning stage in either one of the procedures. Figure 46 shows a possible procedure that can be used to perform EIS measurements on the DUT.

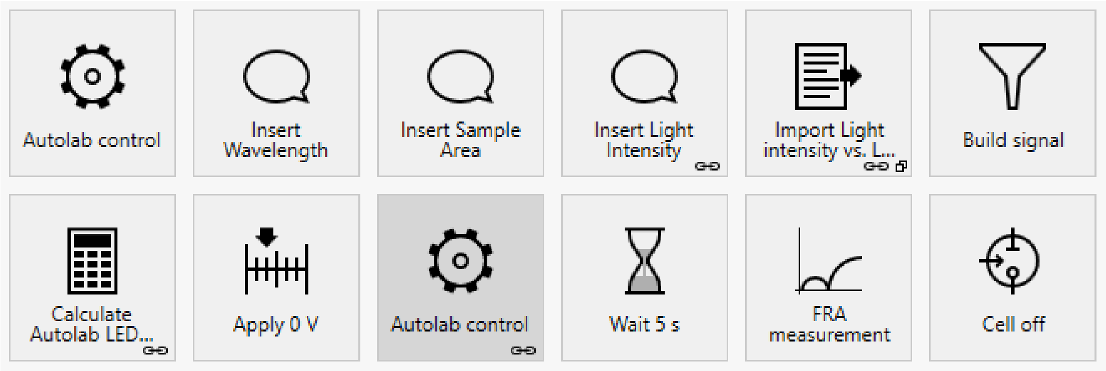


Figure 46 – The FRA impedance potentiostatic procedure modified using the *Autolab Control* command

Typical electrochemical impedance spectroscopy measured at different light intensities are illustrated in Figure 47. As the illumination level increases, the Nyquist plot indicates a lower total resistance.

The data shown in Figure 47 has been recorded at 627 nm, at open circuit potential, using an amplitude of 10 mV.

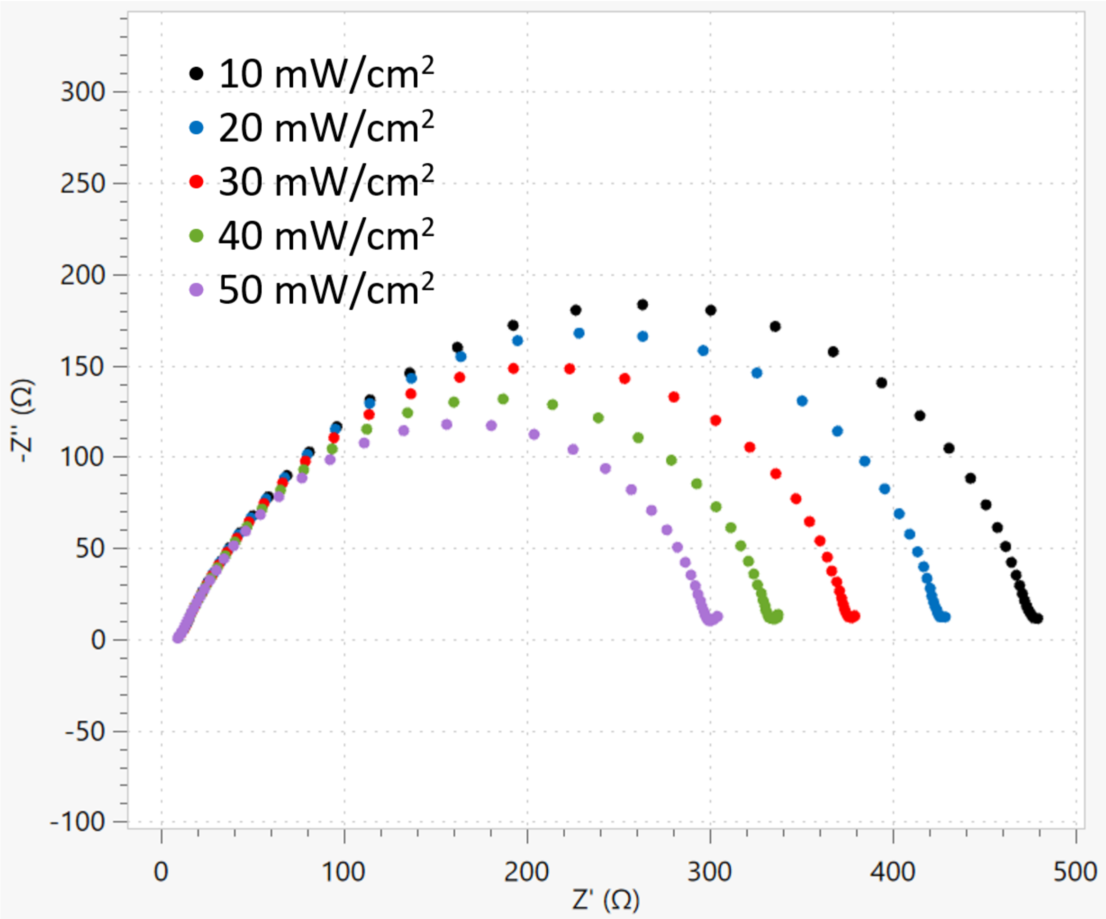


Figure 47 – Typical Nyquist plots recorded at different light intensities

9.4 – AC measurements at modulated illumination

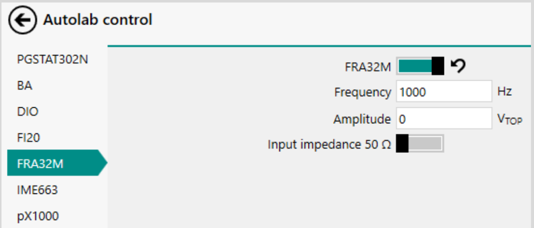
While it is possible to modulate the current or the potential in a classic electrochemical impedance spectroscopy measurement, as explained in Section 9.3.1, it is also possible to modulate the light intensity directly. Two measurement possibilities exist:

- Intensity-modulated photovoltage spectroscopy (IMVS)
- Intensity-modulated photocurrent spectroscopy (IMPS)

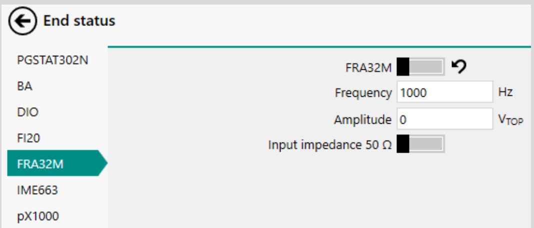
> **Note**
>
> The maximum modulation frequency is **20 kHz**.

Note

For measurements done with modulated light intensity (e.g., IMVS, IMPS), make sure that the external output of the FRA2 or FRA32M module is switched ON in the Autolab Control command in the procedure, before the measurement command starts.



Also, the external output of the FRA2 or FRA32M must be switched OFF in the Edit End Status of the procedure.



9.4.1 – Intensity modulated photovoltage spectroscopy (IMVS)

The LED Driver can be used in combination with the PGSTAT/FRA interface to record intensity modulated photovoltage spectroscopy measurements on the device under test (DUT).

During an intensity modulated photovoltage spectroscopy (IMVS) measurement, the DUT is exposed to a constant light intensity, φ_0, modulated by a small amplitude AC perturbation, $\Delta\varphi$. The DUT settles at a constant voltage, V_0, modulated by a small amplitude AC response, ΔV.

The IMVS impedance is the transfer function, H, between the AC voltage (ΔV) and the AC light modulation ($\Delta\varphi$).

Figure 48 shows a schematic overview of the experimental setup.

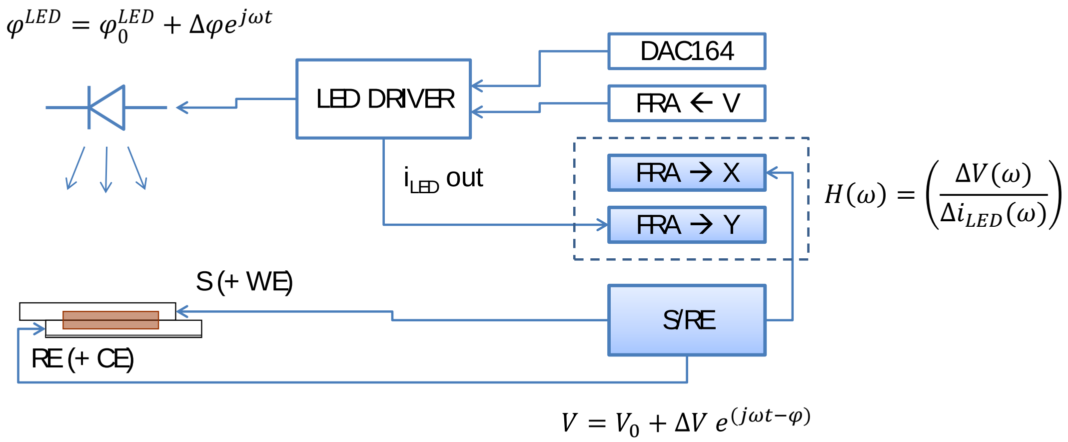


Figure 48 – Overview of the IMVS measurement setup

The fixed light intensity, φ_0 is provided by the DAC voltage using the *Autolab Control* command, as described in the Sections 5.3.1 and 6.2.2. The AC modulation, $\Delta\varphi$, is provided by the FRA ← V connector, as described in Section 5.3.2 and 5.3.3.

The transfer function, $H(\omega)$, is monitored using the external inputs of the FRA module:

- The Eout signal from the PGSTAT is fed into the FRA → X input connector of the FRA2 or FRA32M module[8].
- The FRA Y → output of the LED Driver is fed into the FRA → Y input connector of the FRA module.

[8] The E out signal can be connected directly to the FRA → X input or through the LED Driver, as described in Section 5.3. In this case, the DIO switch needs to be set in order to have the → E out input of the LED Driver active.

9.4.1.1 – IMVS measurements

IMVS measurements are usually carried out at open circuit potential (OCP).

Figure 49 shows an example of an IMVS procedure. In the first step of the procedure, the light intensity is set to a fixed DC level using the *Autolab control* command. The cell is then set open circuit conditions by applying 0 A in Galvanostatic mode.

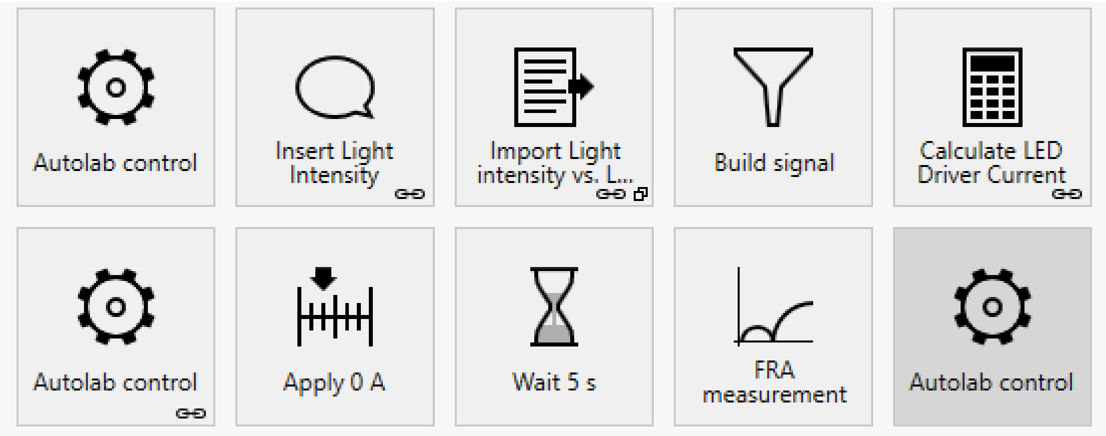


Figure 49 – An example of IMVS procedure

The second stage of the procedure performs the IMVS measurement using the *FRA measurement* command, External input connections. To specify the settings to this command, click the More button in the procedure editor (see Figure 50).

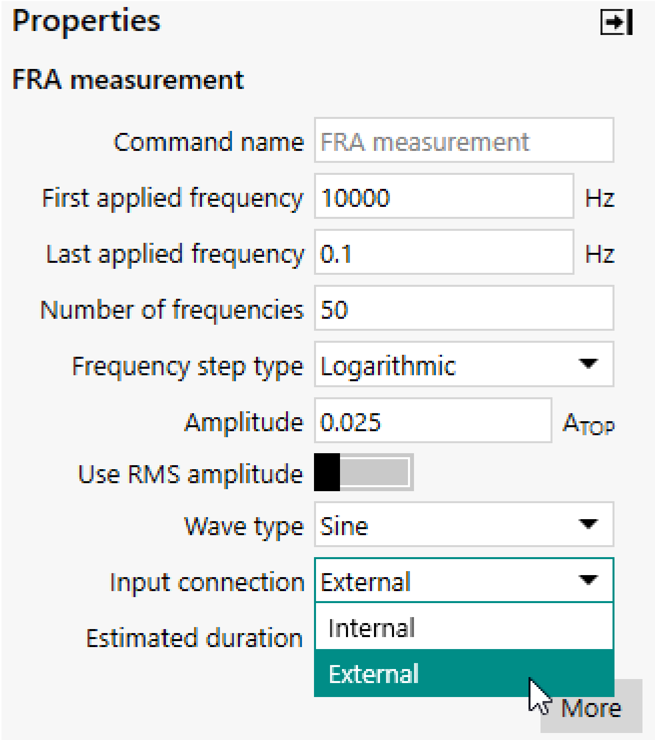


Figure 50 – The *FRA measurement* command, with the External input connection is used during the IMVS frequency scan

A new window will be displayed (see Figure 51).

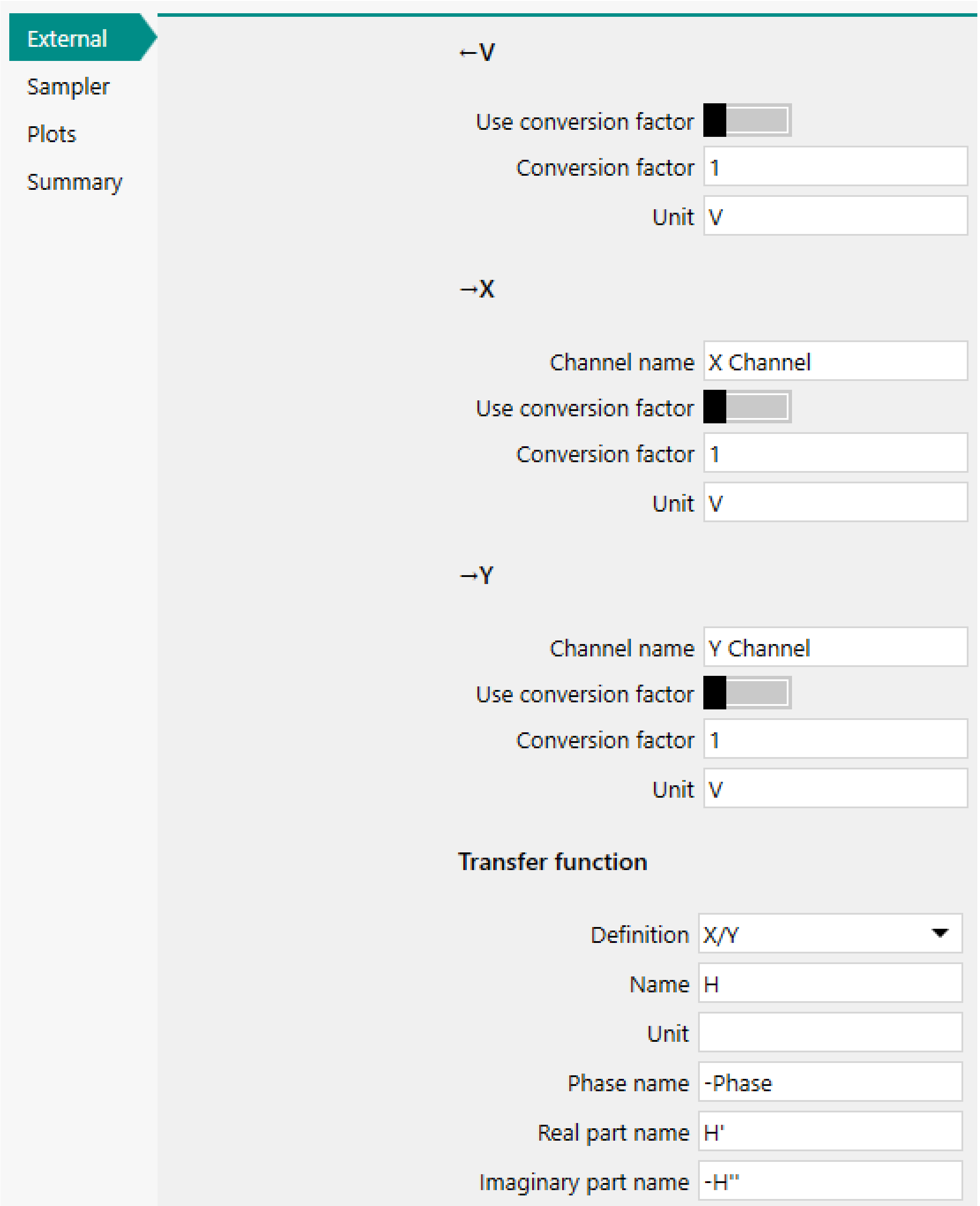


Figure 51 – The FRA editor window, when the external input connection is chosen

The settings defined on the **External** section of the FRA Measurement properties are used to specify the parameters of the IMVS transfer function section (see Figure 52 – Figure 55).

It is possible to define the IMVS transfer function as the ratio between the photovoltage and the light intensity, instead of photovoltage and LED Driver current, using the following conversion factor:

$$FRA\,Y\,conv.\ factor\left(\frac{mW/cm^2}{V}\right)=\frac{Light\ Intensity\ (mW/cm^2)}{LED\ Driver\ current\ (mA)/1000\left(\frac{V}{mA}\right)}$$

For example, if a light intensity of 20 mW/cm^2 is used, it can be seen from the light calibration plot (Figure 39, Section 7) that it corresponds to ≈ 205 mA. Using the equation above, the FRA Y conversion factor is calculated as being 97.56 $(mW/cm^2)/V$ (Figure 54).

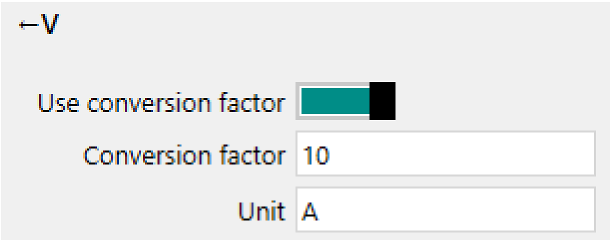


Figure 52 –Settings the parameters for the IMVS transfer function (1/4)

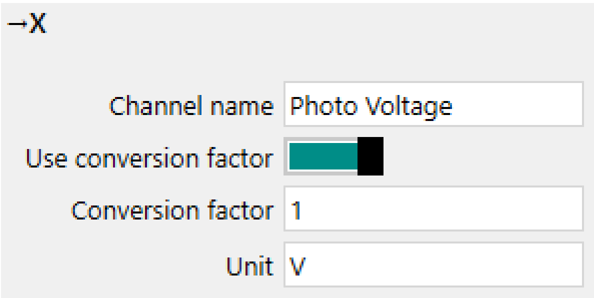


Figure 53 – Settings the parameters for the IMVS transfer function (2/4)

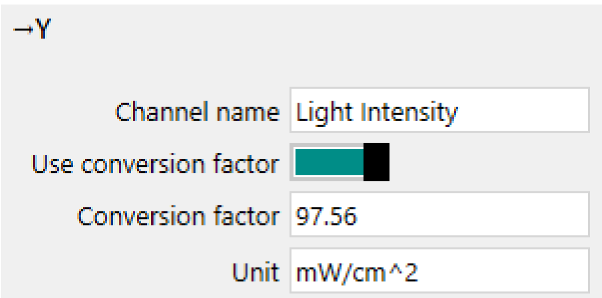


Figure 54 – Settings the parameters for the IMVS transfer function (3/4)

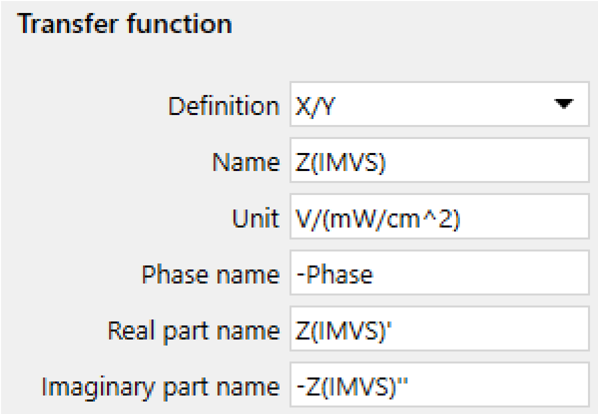


Figure 55 – Settings the parameters for the IMVS transfer function (4/4)

The frequency scan parameters are defined on the **Properties** section of the FRA Measurement command (see Figure 56).

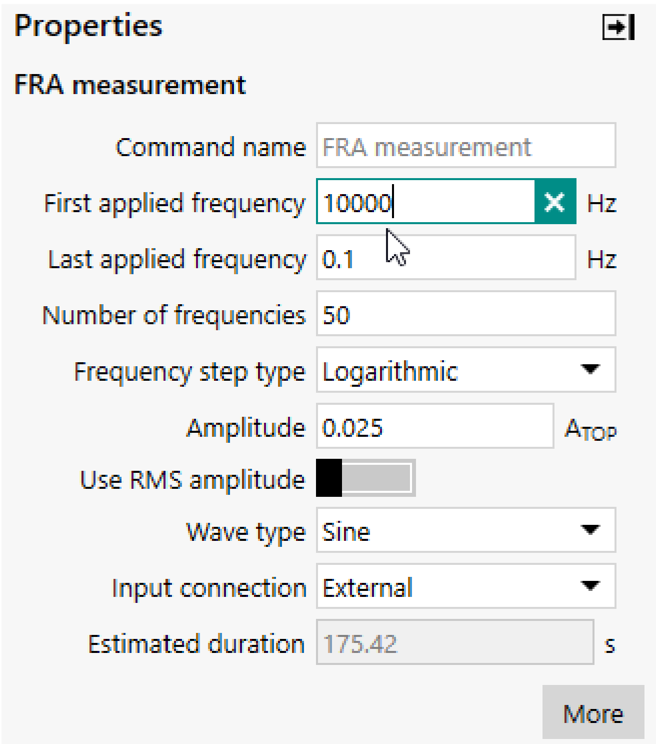


Figure 56 – Defining the frequency range

The amplitude specified in the **Frequency scan** corresponds to the amplitude of the modulated LED Driver current, in A.

On the **Sampler** section, the signals to sample during the frequency scan (see Figure 57).

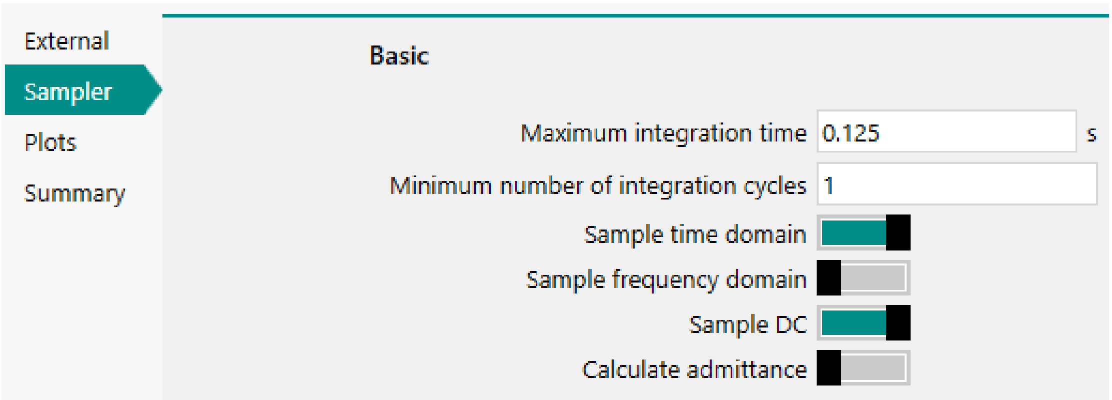


Figure 57 – Specifying the signals to sample during the frequency scan

Note

For this application, it is recommended to sample the time domain signals, as shown in Figure 57.

Finally, the **Plots** section allows the definition of the plots to display during the frequency scan. The availability of the plots depends on the settings defined on the Sampler section (see Figure 58).

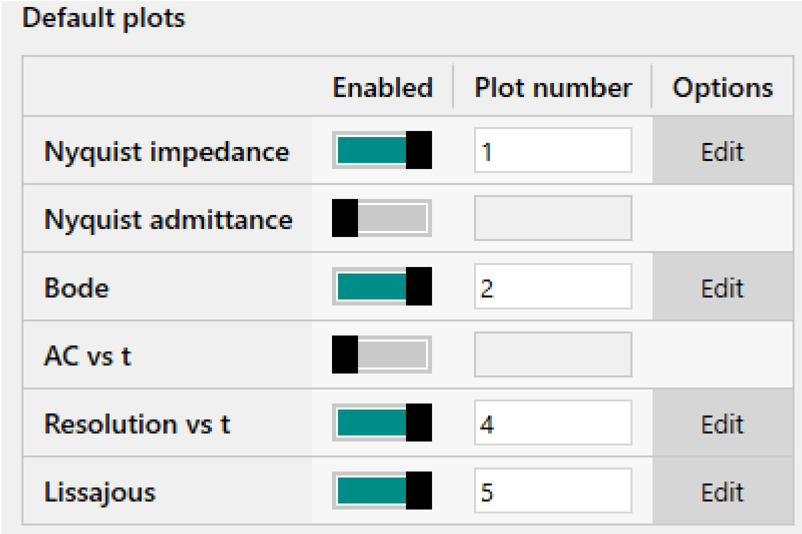


Figure 58 – Specifying the plots and the location for the plots

Click the  button to close the window. The procedure editor will be automatically updated.

Once the frequency range is defined, the measurement can be performed. Figure 59 shows a typical IMVS measurement on a DSC. The measurement is performed at 627 nm, at a constant light intensity of 20 mW/cm^2. The AC amplitude is 10% of the current corresponding at the DC light intensity.

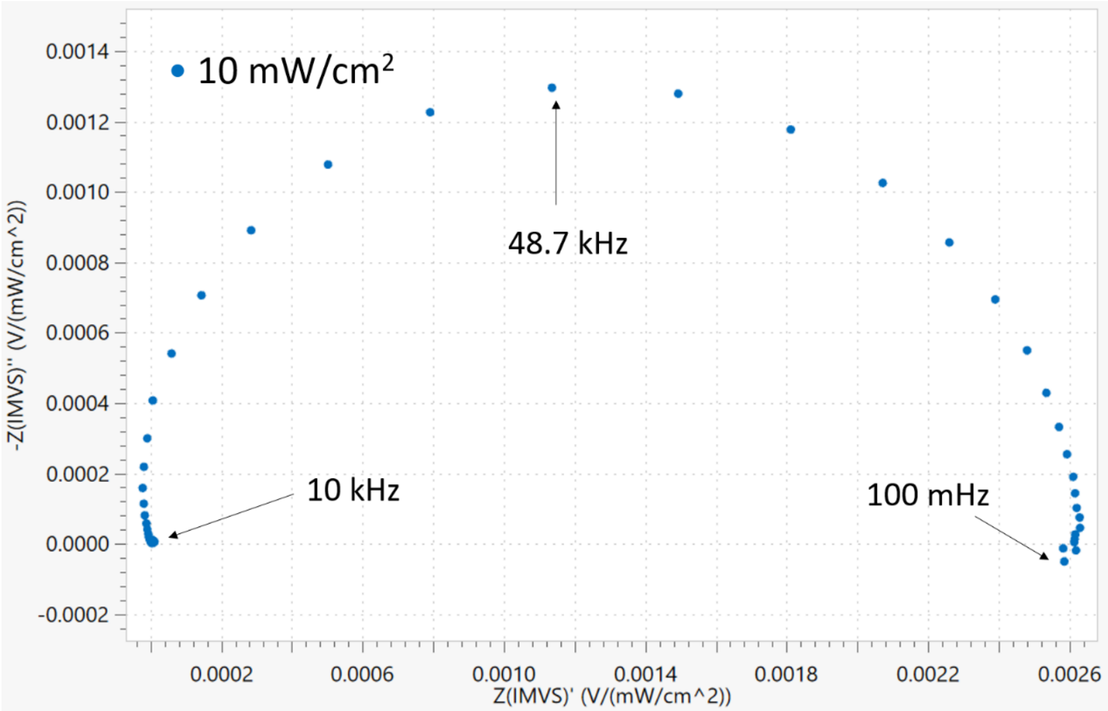


Figure 59 – A typical IMVS measurement on a dye-sensitized solar cell

9.4.2 – Intensity modulated photocurrent spectroscopy (IMPS)

The LED Driver can be used in combination with the PGSTAT/FRA to record intensity modulated photocurrent spectroscopy measurements on the device under test (DUT).

During an intensity modulated current spectroscopy (IMPS) measurement, the DUT is exposed to a constant light intensity, φ_0, modulated by a small amplitude AC perturbation, $\Delta\varphi$. The DUT settles at a constant current, i_0, modulated by a small amplitude AC response, Δi.

The IMPS impedance is the transfer function, H, between the AC current (Δi) and the AC light modulation ($\Delta\varphi$).

Figure 60 shows a schematic overview of the experimental setup.

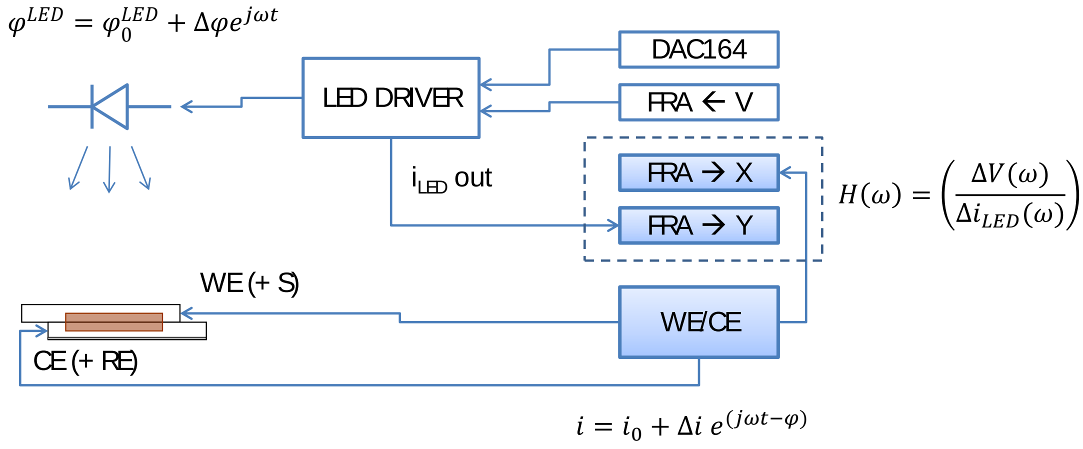


Figure 60 – Overview of the IMPS measurement setup

The fixed light intensity, φ_0 is provided by the DAC voltage using the *Autolab Control* command, as described in the Sections 5.3.1 and 6.2.2. The AC modulation, $\Delta\varphi$, is provided by the FRA ← V connector, using the *FRA measurement external* command, as described in Sections 5.3.2 and 5.3.3.

The transfer function, $H(\omega)$, is monitored using the external inputs of the FRA module:

- The iout signal from the PGSTAT is fed into the FRA → X input connector of the FRA module[9].
- The FRA Y → output of the LED Driver is fed into the FRA → Y input connector of the FRA module.

Warning

The automatic current ranging option <u>cannot</u> be used in an IMPS measurement.

[9] The iout signal can be connected directly to the FRA → X input or through the LED Driver, as described in Section 5.3. In this case, the DIO switch needs to be set in order to have the → i out input of the LED Driver active.

9.4.2.1 – IMPS measurements

IMPS measurements are usually carried out at short-circuit conditions.

Figure 61 shows an example of an IMPS procedure. In the first step of the procedure, the light intensity is set to a fixed DC level using the *Autolab Control* command. The potential is set to 0 V (short-circuit conditions).

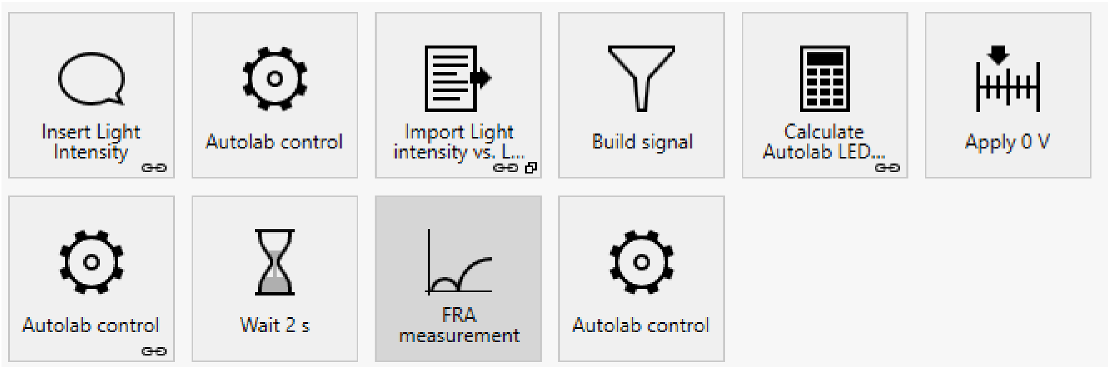


Figure 61 – An example of IMPS procedure

The second stage of the procedure performs the IMPS measurement using the *FRA measurement* command, external input connection. To specify the settings to this command, click the More button in the *Fra measurement* command properties (see Figure 62).

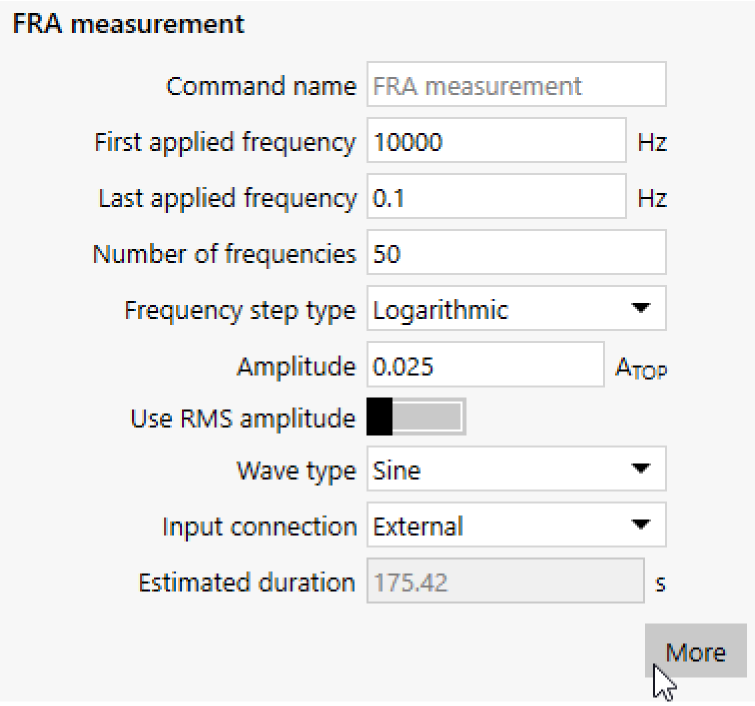


Figure 62 – The *FRA measurement external* command is used during the IMPS frequency scan

A new window will be displayed (see Figure 63).

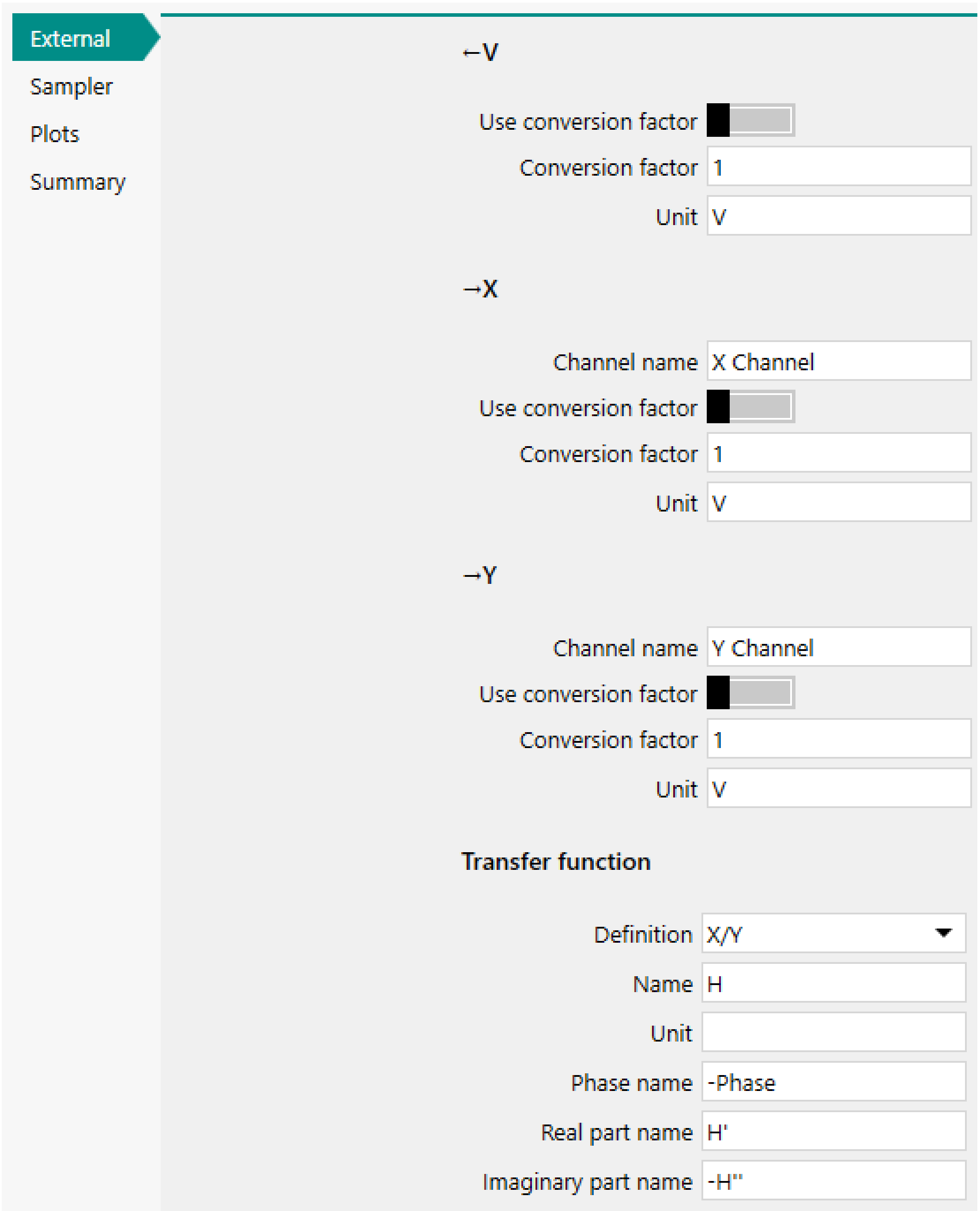


Figure 63 – The FRA editor window

The settings defined on the **External** section of the FRA editor window are used to specify the parameters of the IMPS transfer section (see Figure 64 – Figure 67).

It is possible to define the IMPS transfer function as the ratio between the photocurrent and the light intensity, instead of photocurrent and LED Drive current, using the following conversion factor:

$$FRA\,Y\,conv.\,factor\left(\frac{mW/cm^2}{V}\right) = \frac{Light\,Intensity\,(mW/cm^2)}{LED\,Driver\,current\,(mA)/1000\left(\frac{V}{mA}\right)}$$

For example, if a light intensity of 20 mW/cm^2 is used, it can be seen from the light calibration plot (Figure 39, Section 7) that it corresponds to ≈ 205 mA. Using the equation above, the FRA Y conversion factor is calculated as being 97.56 (mW/cm^2)/V Figure 54(Figure 66).

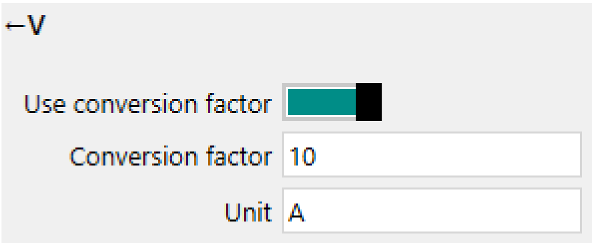


Figure 64 –Settings the parameters for the IMPS transfer function (1/4)

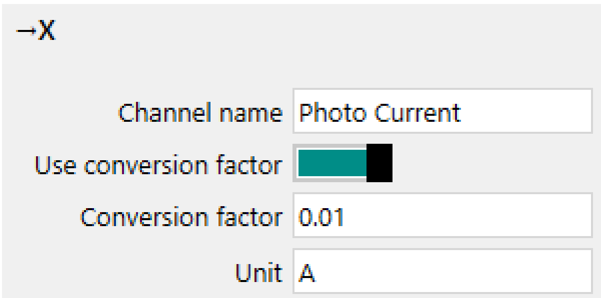


Figure 65 – Settings the parameters for the IMPS transfer function (2/4)

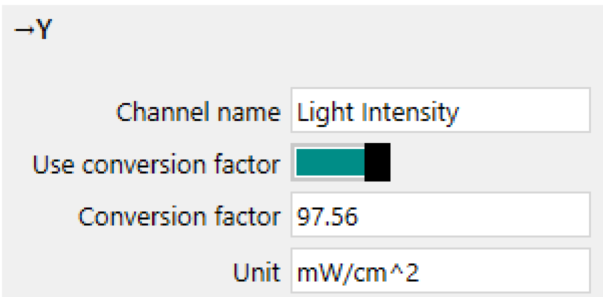


Figure 66 – Settings the parameters for the IMPS transfer function (3/4)

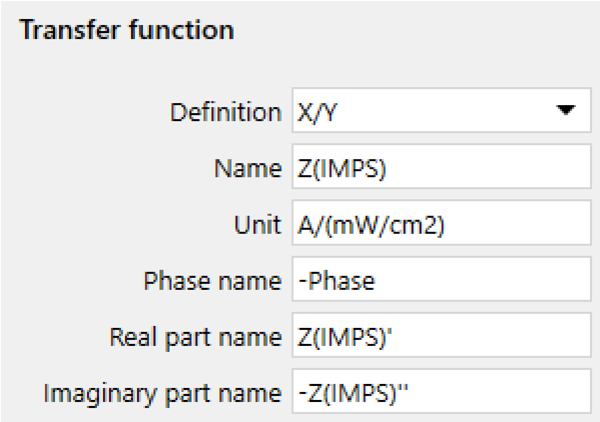


Figure 67 – Settings the parameters for the IMPS transfer function (4/4)

The multiplier specified for the → X input in the FRA editor, shown in Figure 65, depends on the current range used by the Autolab during the measurement. The signal measured on this input corresponds to the i out signal coming from the Autolab, which corresponds to the converted cell current (which depends on the selected current range[10]).

This input is therefore specified in A and requires a multiplier given by the measured current (in A) divided by the current range (in A/V). Table 2 shows the multiplier values depending on the selected current range. Figure 65 shows the settings for a measurement in the 1 mA current range.

Table 2 – Multiplier factors per current range

Current range	Conversion factor	Multiplier
1 A	1 A/V	1
100 mA	100 mA/V	0.1
10 mA	10 mA/V	0.01
1 mA	1 mA/V	0.001
100 µA	100 µA/V	0.0001
10 µA	10 µA/V	0.00001

[10] Please refer to the NOVA manual for more information.

1 µA	1 µA/V	0.000001
100 nA	100 nA/V	0.0000001
10 nA	10 nA/V	0.00000001

The frequency scan parameters are defined on the **Frequency scan** section of the FRA editor (see Figure 68).

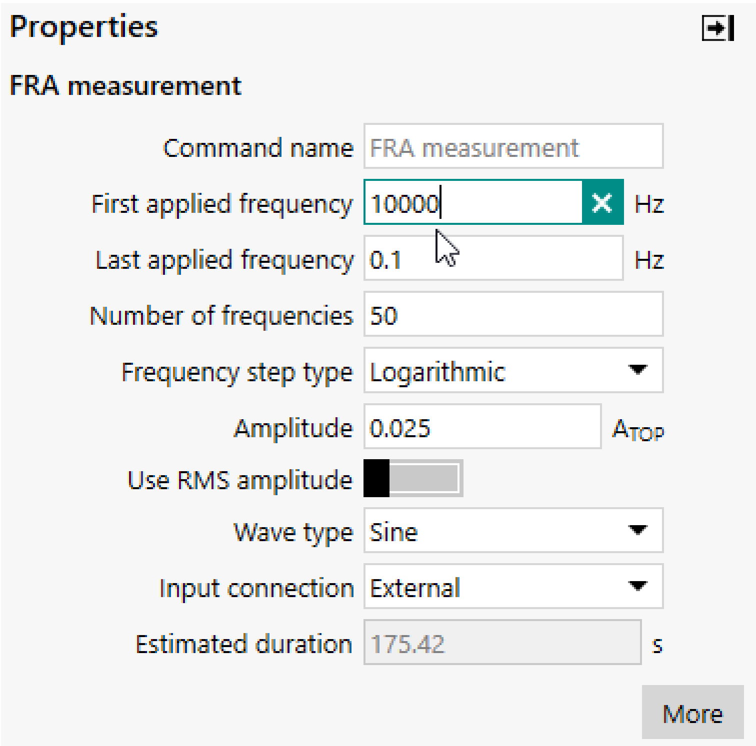


Figure 68 – Defining the frequency range

The amplitude specified in the **Frequency scan** corresponds to the amplitude of the modulated LED Driver current, in A.

On the **Sampler** section, the signals to sample during the frequency scan (see Figure 69).

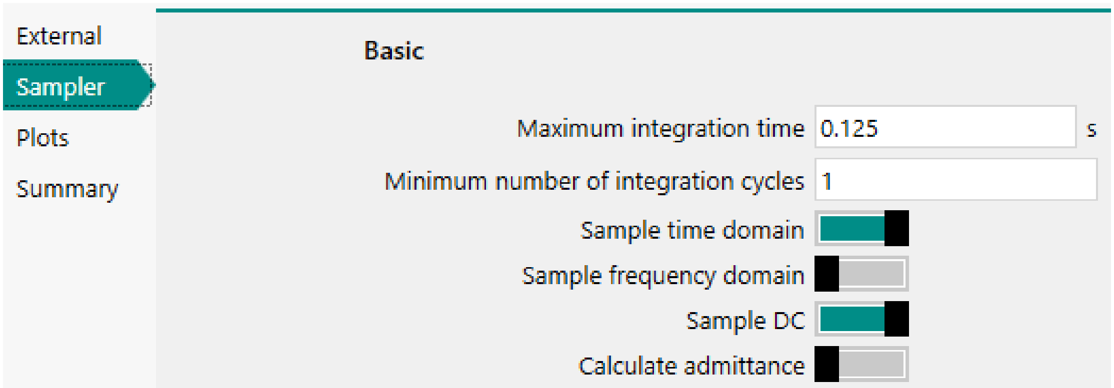


Figure 69 – Specifying the signals to sample during the frequency scan

Note

For this application, it is recommended to sample the time domain signals, as shown in Figure 69.

Finally, the **Plots** section allows the definition of the plots to display during the frequency scan. The availability of the plots depends on the settings defined on the Sampler section (see Figure 70).

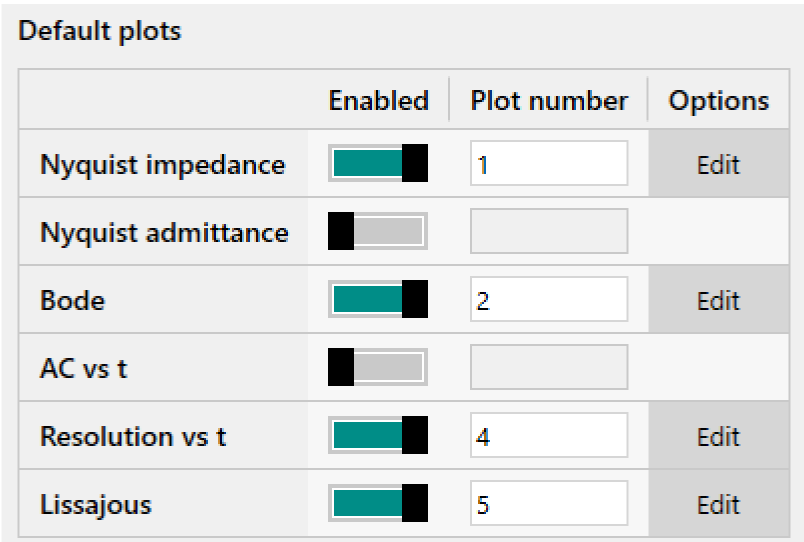


Figure 70 – Specifying the plots and the location for the plots

Click the button to close the window. The procedure editor will be automatically updated.

Once the frequency range is defined, the measurement can be performed. Figure 71 shows a typical IMPS measurement on a DSC. The measurement is performed at 627 nm, at a constant light intensity of 20 mW/cm^2. The AC amplitude is 10% of the DC light intensity.

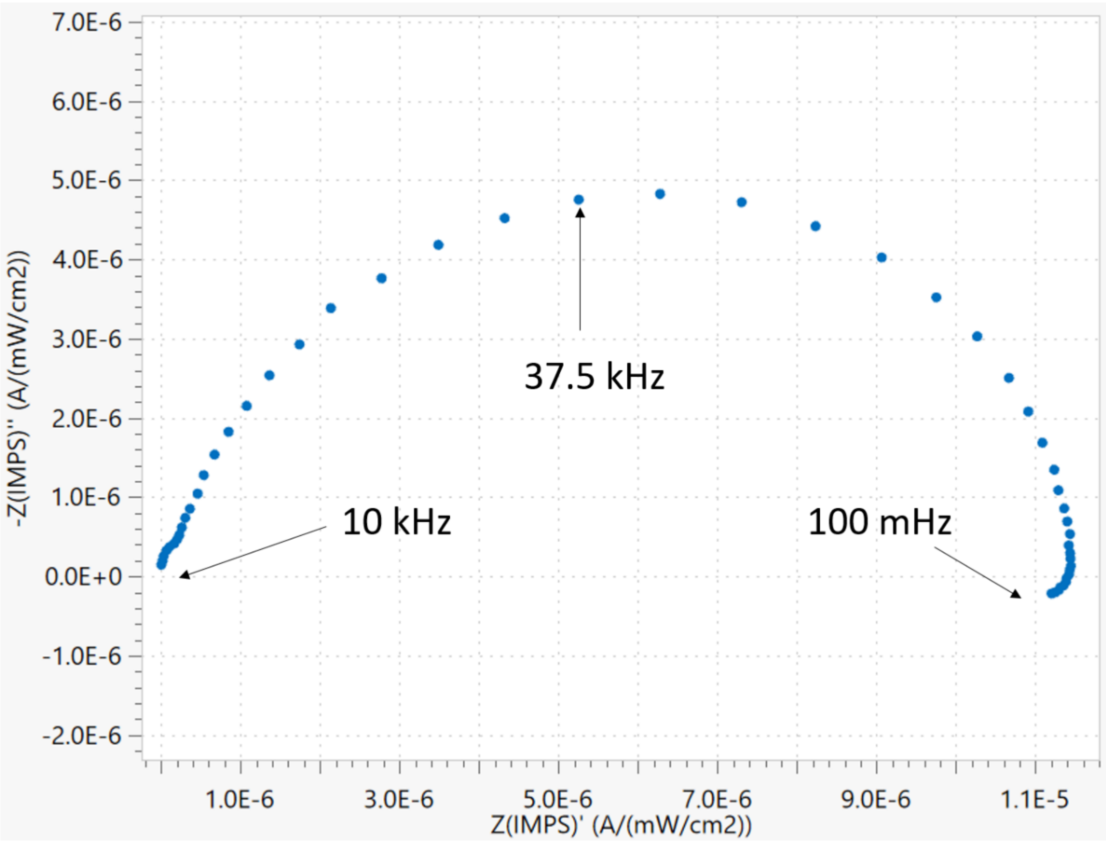


Figure 71 – A typical IMPS measurement on a DSC

9.5 – Safety settings when measurement is aborted

When measurements are manually interrupted, by clicking the Stop button, or when a cutoff condition is met that stops the complete procedure, it may be necessary to completely switch off the light source and the cell.

To define the end conditions for the Autolab Optical Bench and the cell, the End status Autolab settings can be defined in the procedure editor.

The end status of Autolab can be defined by clicking the title of the procedure, then the Edit End status button in the properties (see Figure 72).

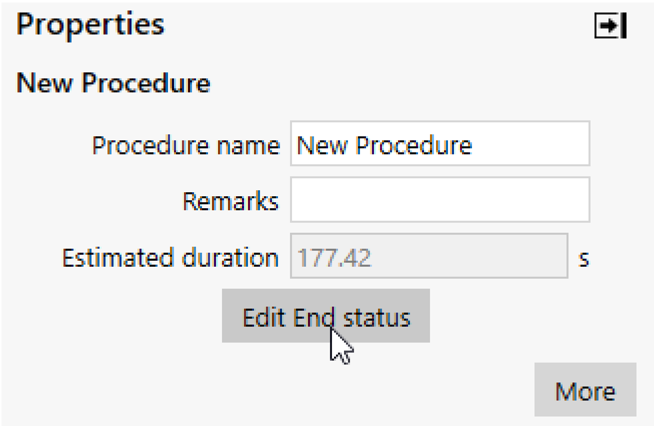


Figure 72 – the Edit End status

The Autolab control editor will be displayed (see Figure 73). Using the sections on the right, the required adjustments can be defined.

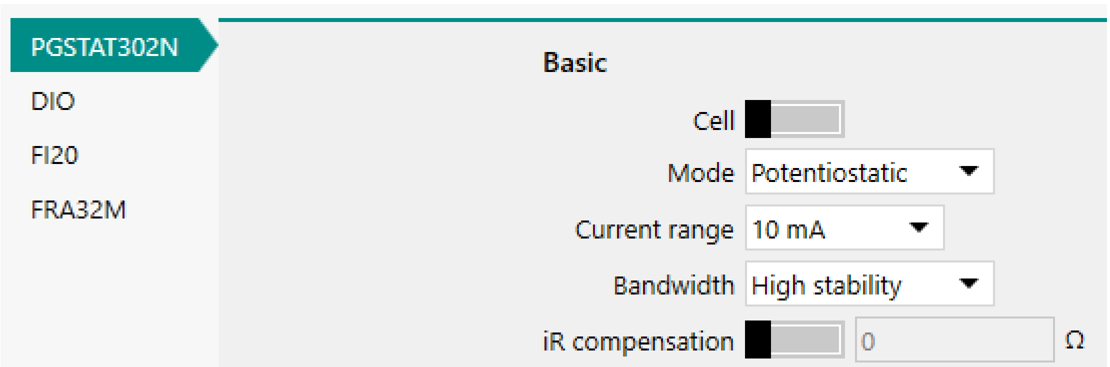


Figure 73 – The end status of Autolab is defined using the Autolab control window

The following settings can be relevant for this application:

- **Cell off:** to switch the cell off, set the Cell switch to Off (see Figure 74).

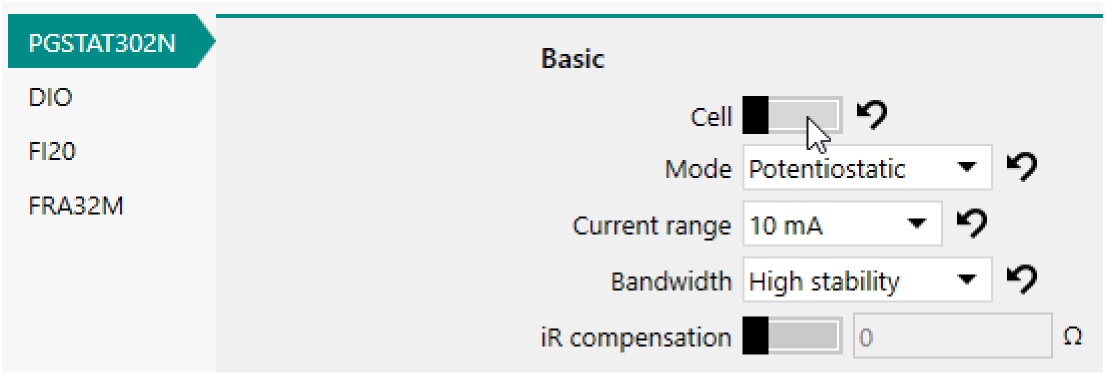


Figure 74 – Switching the cell off

- **Light off:** to switch the light source off, set LED Driver current to 0 A (see Figure 75).

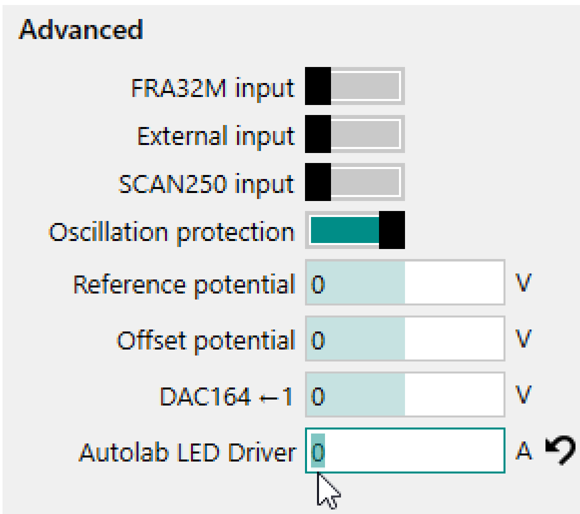


Figure 75 – Setting the LED Driver current to 0 A (DAC voltage = 0 V)

- **Modulation off:** to switch off the modulation generated by the FRA module, if applicable, set the FRA2 or FRA32M output off using the provided switch (see Figure 76).

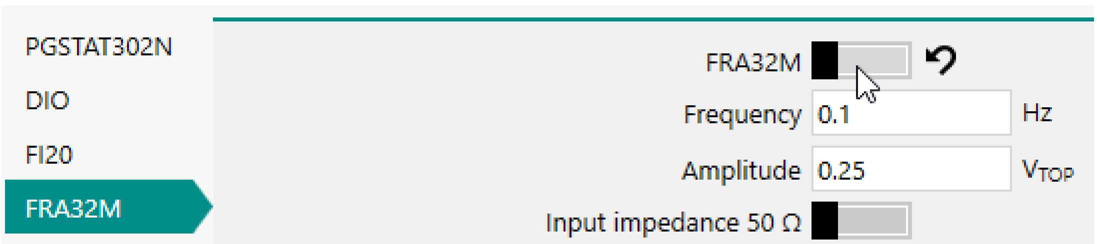


Figure 76 – Switching the output of the FRA module off

10 – Thermal considerations

Although the LED holder has been designed for optimal temperature management, the behavior of the LEDs located onto the PCB is affected by temperature variations. Since the overall temperature of the LED PCB increases as the driving current increases, the light output can be affected, leading to a non-linear relationship between driving current and light intensity.

Figure 77 and Figure 78 show how the relative light output of the LEDs is affected by the temperature. The light intensity is normalized with respect to the light intensity measured at 25 °C.

Warning

The LED light source can get hot when operated at high driving currents. Always allow the light source to cool down before touching it.

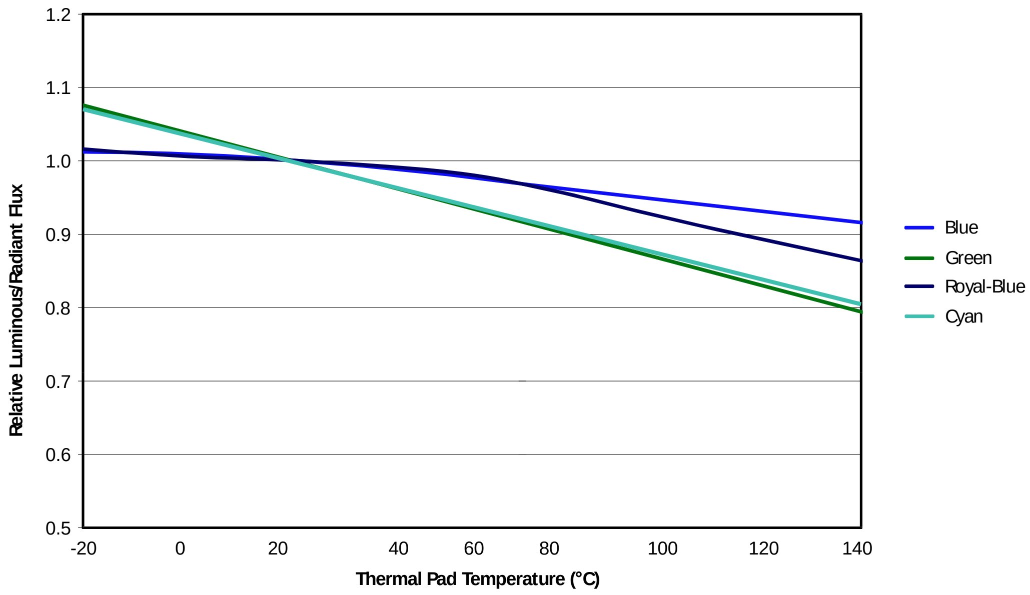


Figure 77 – Relative light output vs thermal pad temperature for green, cyan, blue and royal-blue[11]

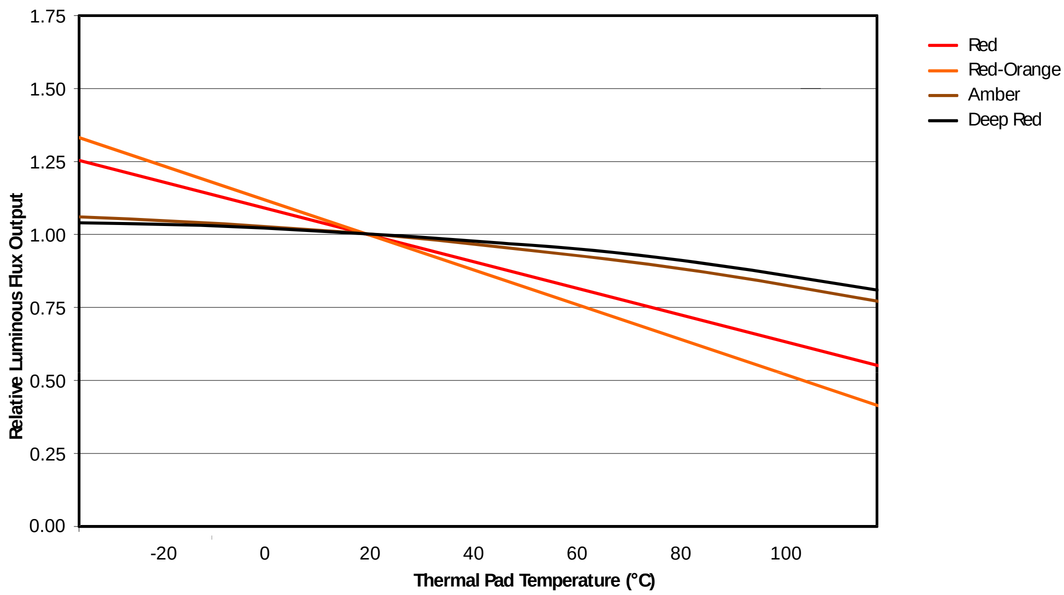


Figure 78 – Relative light output vs thermal pad temperature for red, red-orange and amber[12]

[11] Adapted from LUXEON Rebel, Direct Color Portfolio, High power, coloured LEDs, Technical Datasheet DS65.

[12] Adapted from LUXEON Rebel, Direct Color Portfolio, High power, colored LEDs, Technical Datasheet DS65.

Appendix 1 – Modification of the input range of the FRA2 module

By default, the external inputs of the FRA2 modules shipped before July 2009 (revision number 8.0 and lower) can be used to record analog signals in the ±5 V range. For some applications, analog signals in the ±10 V range are required. In order to be able to record voltages between 5 and 10 V, the FRA2 modules with revision numbers lower than 8.1 need to have the extended range offset DACs activated. This requires a simple hardware modification described in this document. Please contact Metrohm Autolab (autolab@metrohm.com) or your local distributor in case of any doubts.

The modification described in this document requires the following items:

1. ESD safety kit (see Figure 79)
2. Soldering iron and solder

The modification requires soldering **JP2** and **JP3** of the analog board of the FRA2 module. Figure 79 shows a picture of the Analog board. The two jumpers are highlighted.

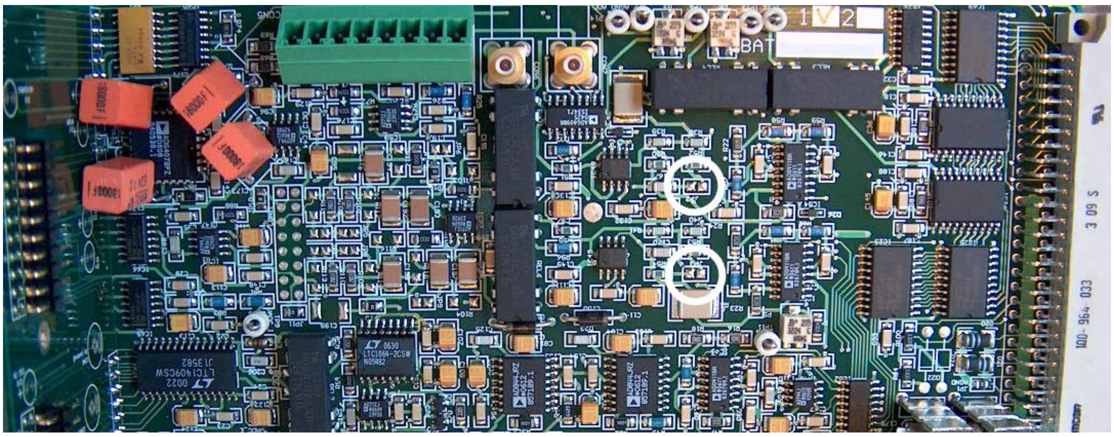


Figure 79 – The FRA2 Analog board (JP2 and JP3 are highlighted)

The two jumpers need to be soldered together.

Warning

Take all the necessary steps to avoid ESD damage to the module by grounding yourself during the handling of the module and the soldering of the jumpers. The use of an ESD safety kit is highly recommended.

Remove the FRA2 module from the Autolab frame. To remove the FRA2 module follow the instructions reported the following documents:

The modification described in this document requires the following items:

1. For the PGSTAT128N, PGSTAT302N, PGSTAT302F and PGSTAT100N: *All modules – Insert new module in 8-series cabinet.pdf*
2. for the PGSTAT12, PGSTAT30, PGSTAT302 and PGSTAT100 *All modules – Insert new module in 7-series cabinet.pdf*

The FRA2 module consists of 2 PCBs (see Figure 80). One PCB is the digital signal generator (DSG) PCB and the second one is the Analog PCB.

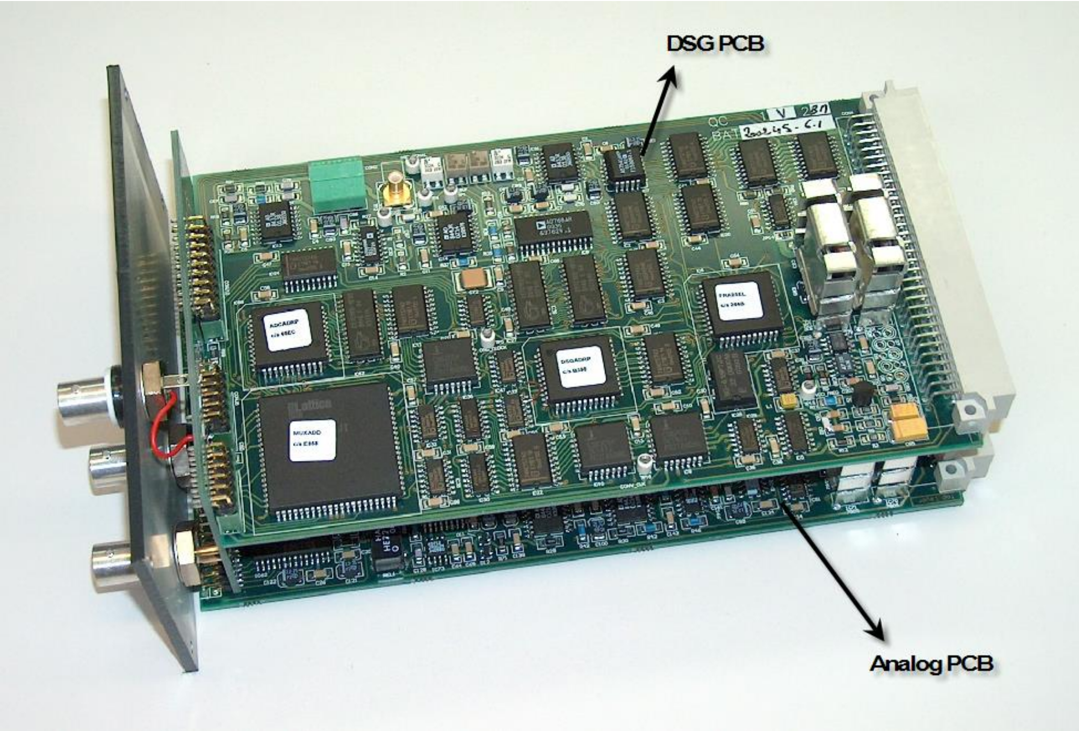


Figure 80 – The FRA2 consists of two PCBs (the DSG on top, and the Analog below)

To access the Analog PCB, the DSG PCB must be detached. To do this, gently push on both sides of the board as shown in Figure 81 and Figure 82.

Note

It is recommended to store the DSG PCB in an ESD safe bag during the modification.

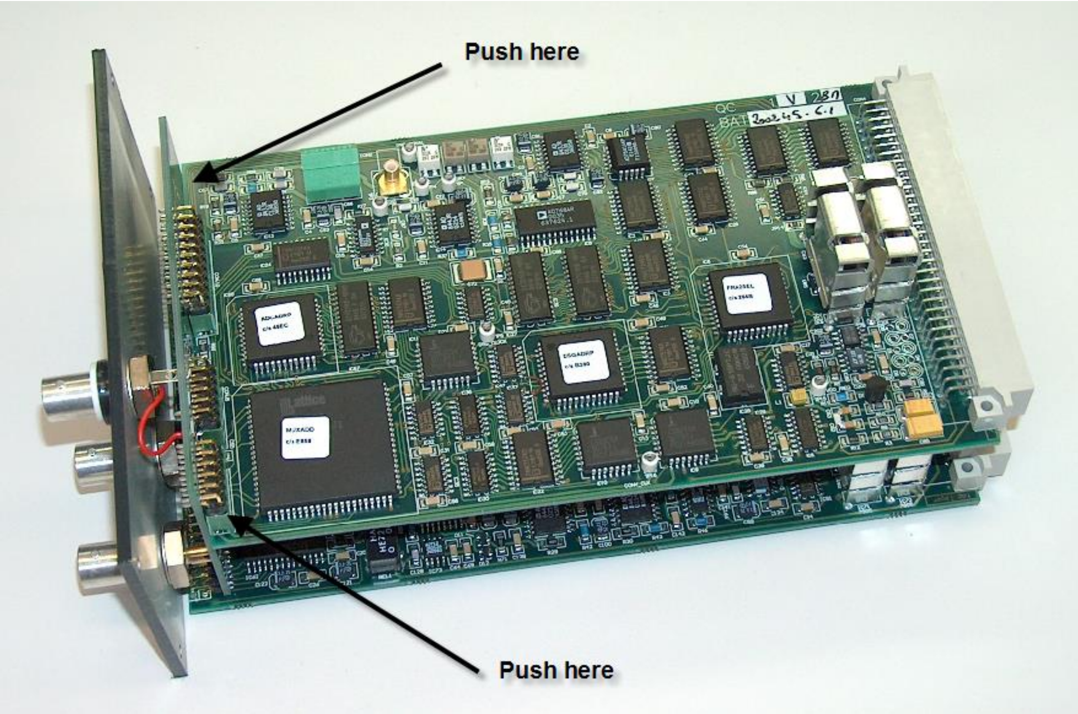


Figure 81 – Pressure points on the DSG PCB

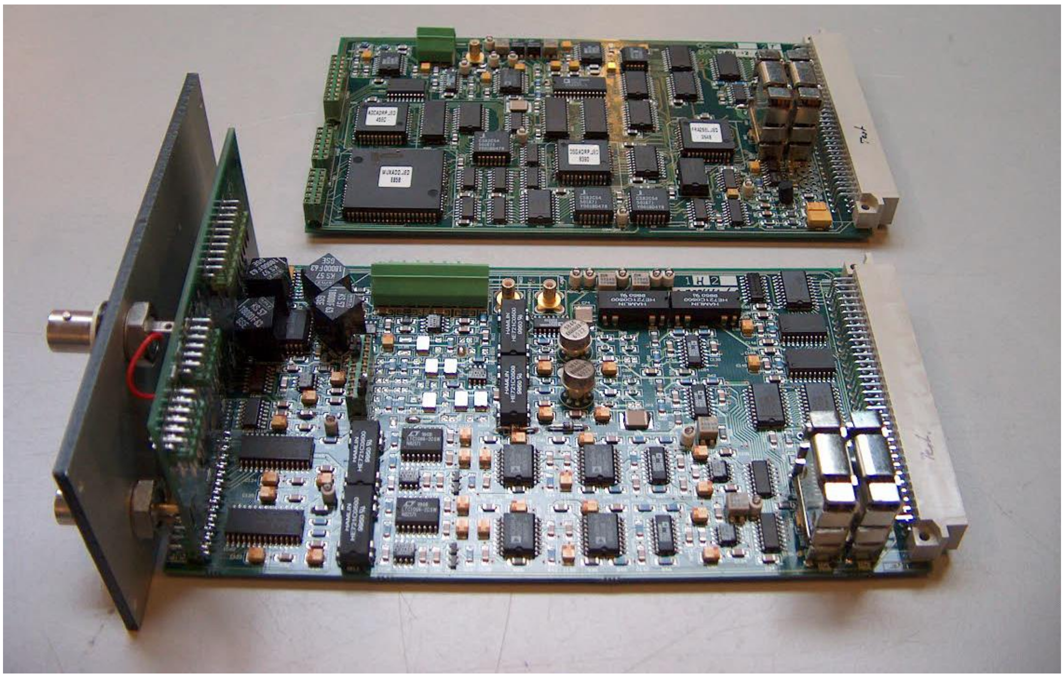


Figure 82 – The removed DSG PCB and the Analog PCB of the FRA2 module

Figure 82 shows the FRA2 module, with the removed DSG PSB. Locate JP2 and JP3 on the Analog PCB and close both jumpers by applying a bit a solder.

To reassemble the 2 boards, all the holes in the 3 green connectors of the DSG PCB have to be aligned with the matching pins on the FRA2 module assembly (see Figure 83).

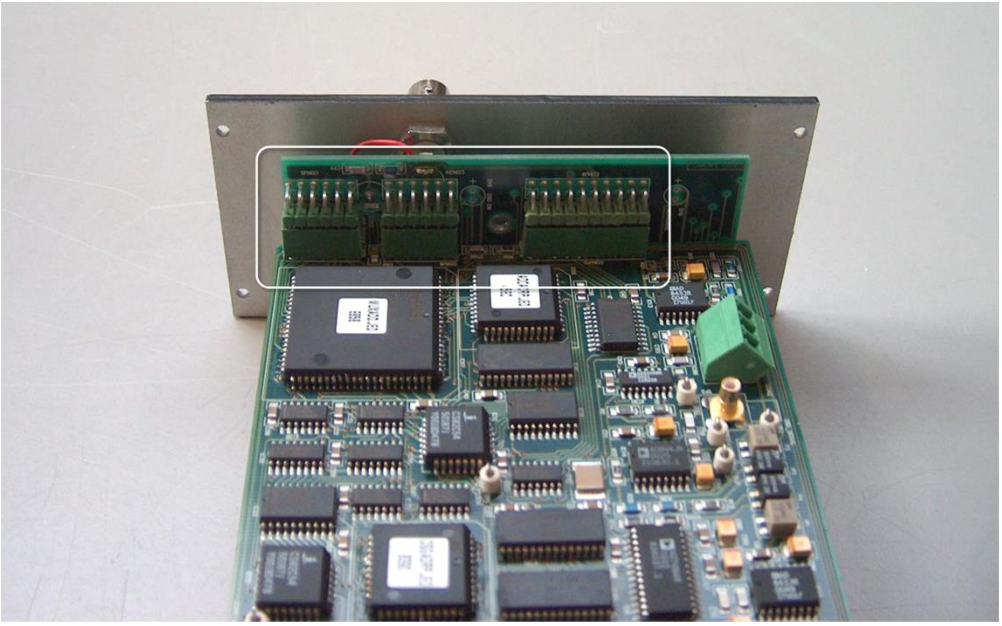


Figure 83 – Reassembling the FRA2 module

Note

The factory FRA2CAL.INI file can still be used. No recalibration is required.

Reinstall the module in the Autolab following the instructions provided in the installation guides mentioned at the beginning of this section.

After modification of the FRA2 module has been modified, the software needs to be adjusted.

The FRA2 input range is directly specified in the Hardware setup. Start NOVA and open the Hardware setup. Click on the FRA2 module in the list of Additional modules and then the FRA offset DAC range toggle at the right of the Hardware setup window (see Figure 84).

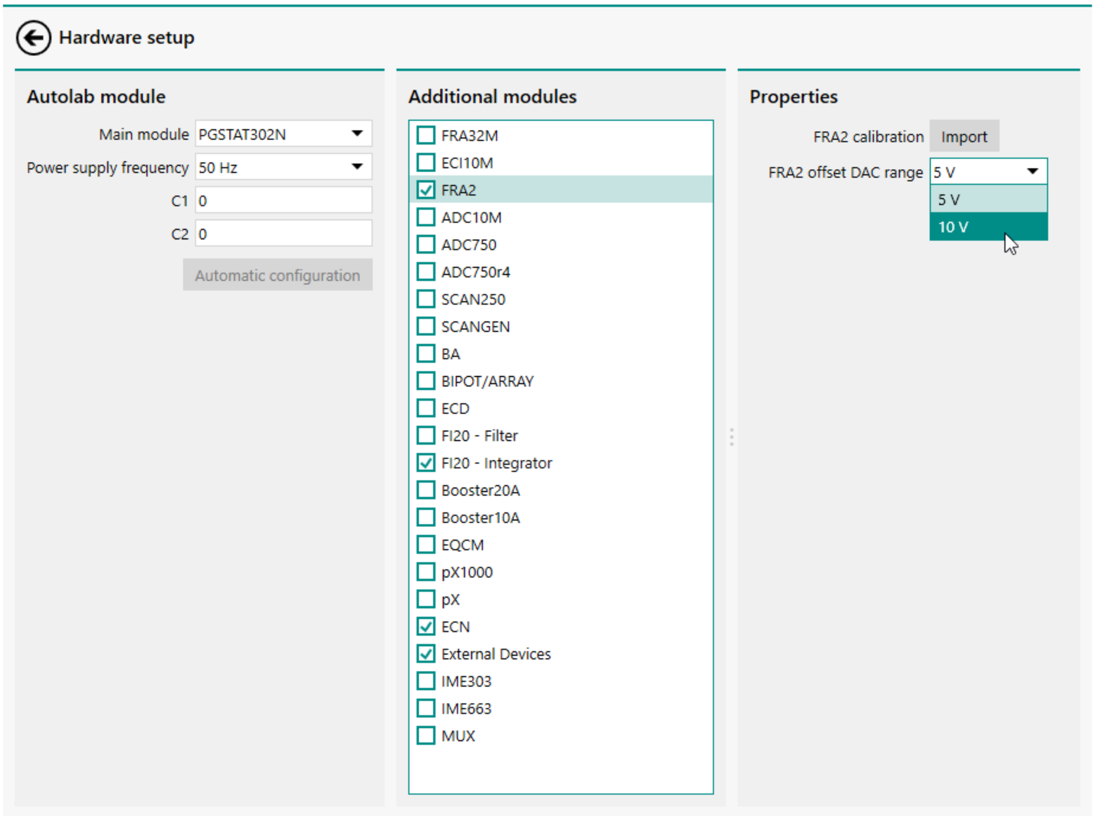


Figure 84 – The 10 V input range can be specified in the Hardware setup directly

Set this toggle to 10 V as shown in Figure 84. Click OK to close the Hardware setup and save the modifications when prompted.

This modification is permanent.

If necessary, new labels (article codes: CAB.LABEL.FRA2.V10.V and CAB.LABEL.FRA2.V10.XY) can be ordered for the modified FRA2 module (see Figure 85).

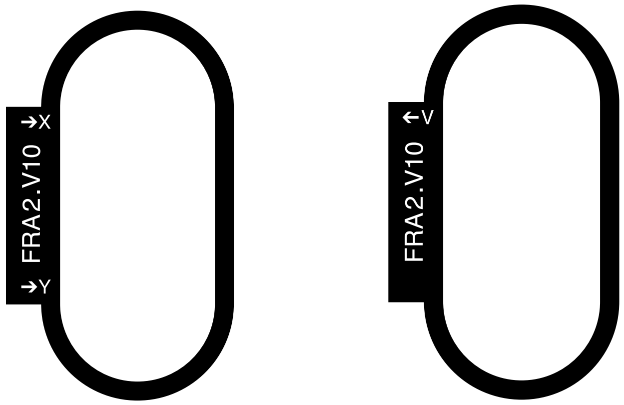


Figure 85 – FRA2 10 V input range labels

Appendix 2 – Calibrated Photodiode

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Ph. 973-579-7227
FAX 973-300-3600

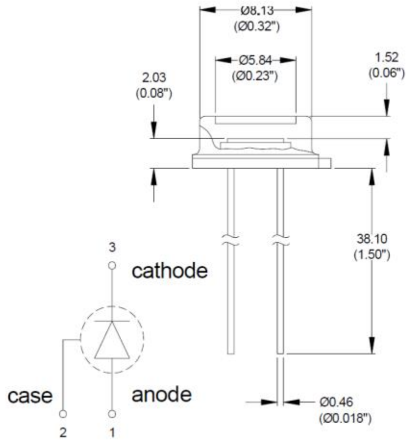
FDS100 Si Photodiode

High Speed
Large Active Area

The FDS100 is a high-speed silicon photodiode with a spectral response from 350nm to over 1100nm. This photodiode has a PIN structure that provides fast rise and fall times with a bias of 20V.

Electrical Characteristics

Spectral Response:	350-1100nm
Active Area:	13.0mm²
Rise Time (RL=50Ω):	10ns (20V bias)
Fall Time (RL=50Ω):	10ns (20V bias)
NEP@900nm:	1.2×10^{-14} W/√Hz (@20V bias)
Dark Current:	20nA max (20V)
Package:	T05, 0.36" can

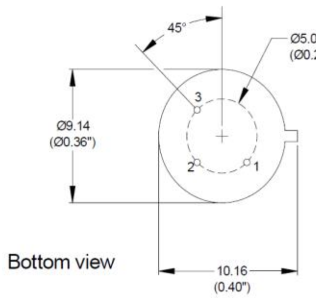


Maximum Ratings

Damage Threshold CW:	100 mW/cm^2
Damage 10ns Pulse:	500mJ/cm²
Max Bias Voltage:	25V

Pin Description

1. Laser anode
2. Laser case
3. Laser cathode



The Thorlabs FDS100 photodiode is ideal for measuring both pulsed and CW light sources, by converting the optical power to an electrical current. The Si detector is housed in a T05 can, with an anode, cathode and case connection. The photodiode anode produces a current, which is a function of the incident light power and the wavelength. The responsivity $\Re(\lambda)$, can be read from **Figure 1** to estimate the amount of photocurrent to expect. This can be converted to a voltage by placing a load resistor (R_{LOAD}) from the photodiode anode to the circuit ground. The output voltage is derived as:

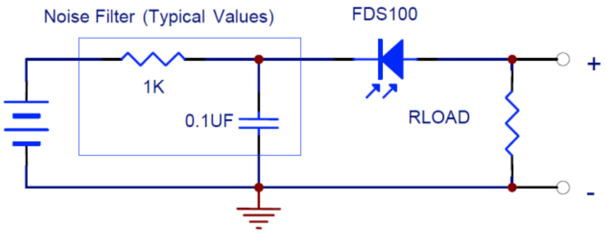
$$V_O = P * \Re(\lambda) * R_{LOAD}$$

The bandwidth, f_{BW}, and the rise time response, t_R, are determined from the diode capacitance, C_J, and the load resistance, R_{LOAD}, as shown below. Placing a bias voltage from the photo diode cathode to the circuit ground can lower the photo diode capacitance.

$$f_{BW} = 1/(2\pi * R_{LOAD} * CJ),\ t_R = 0.35/f_{BW}$$

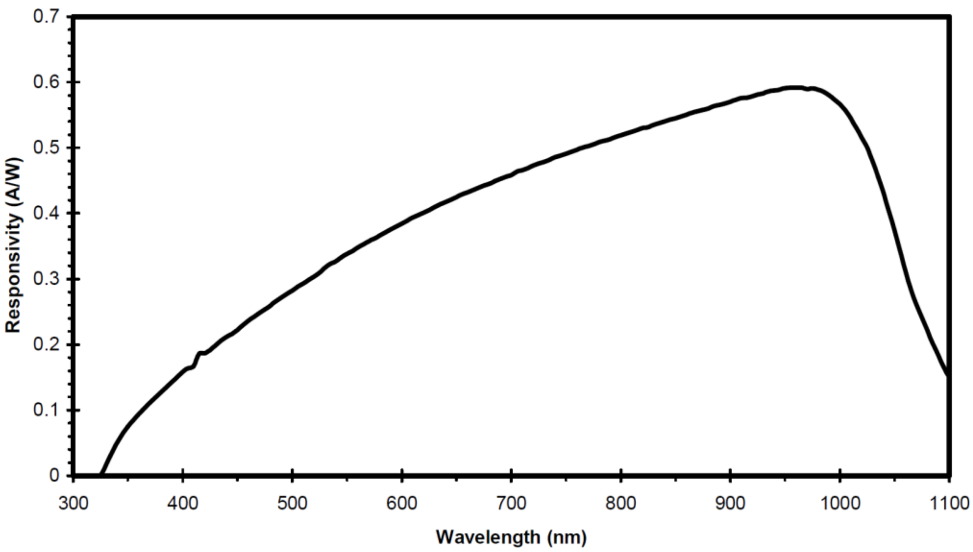
0637-S01 Rev D 4/4/03

Typical Circuit Diagram



Typical Plots

Figure 1 - FDS100 Spectral Responsivity Curve



Typical Responsivity Curve using Thorlabs calibration services.

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Appendix 3 – LED Driver hardware specifications

The specifications of the LED Driver are listed in Table 3.

Power supply	18 V, 1.2 A
Maximum current	1 A
Maximum LED current	700 mA
Input voltage range	90 mV – 10 V
Output voltage range	0 V – 1 V
Maximum modulation amplitude	5 V (TOP)
Maximum modulation frequency	20 kHz
Operating temperature	0 – 40 °C

Table 3 – Specifications of the Autolab LED Driver

Appendix 4 – LED specifications

The LEDs used in the Autolab LED covers are produced by Philips LUMILEDS. These LEDs have an estimated lifetime of more than 10000 hours, provided that the operating conditions are respected. This is especially the case for the operating temperature since prolonged exposure to high temperature values negatively affects the lifetime of the LEDs. If the LED covers are used as described in this manual, the expected lifetime specified by Philips can be expected.

For the colored LEDs, the spectral distribution depends on the color. The typical relative light intensity and spectral distribution of the different LED colors is shown in Figure 86.

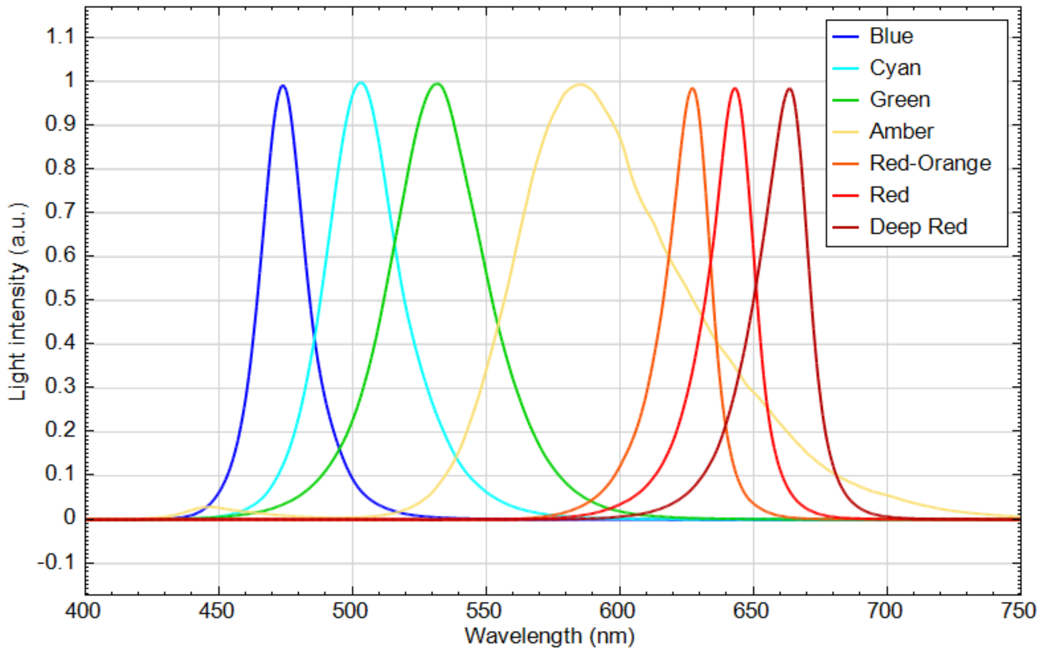

Figure 86 – Typical spectral distributions of the coloured LEDs

For the white LEDs, the spectral distribution depends on the white temperature. The typical relative light intensity and spectral distribution of the different white LEDs are shown in Figure 87–Figure 89.

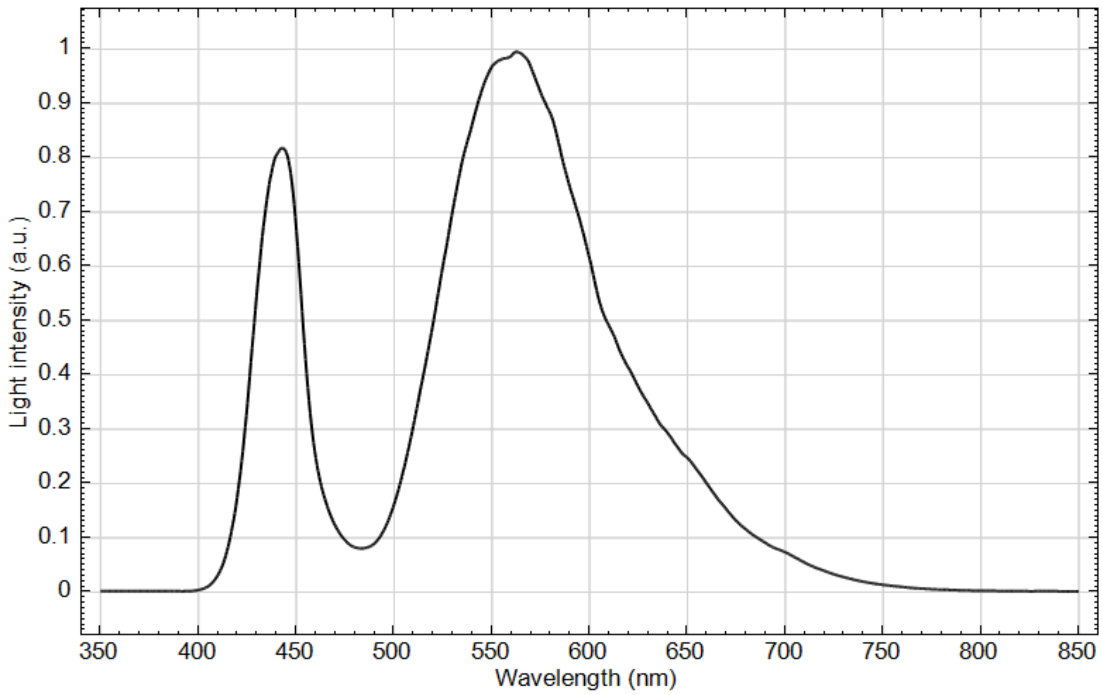

Figure 87 – Typical spectral distribution of the neutral white LED

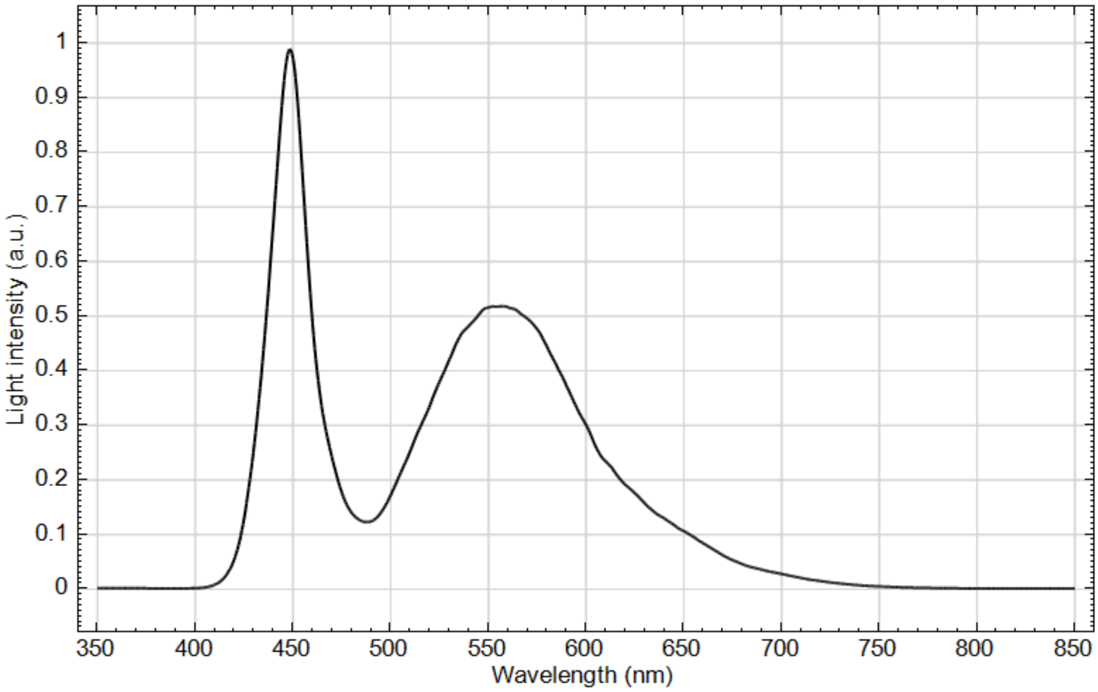

Figure 88 – Typical spectral distribution of the cool white LED

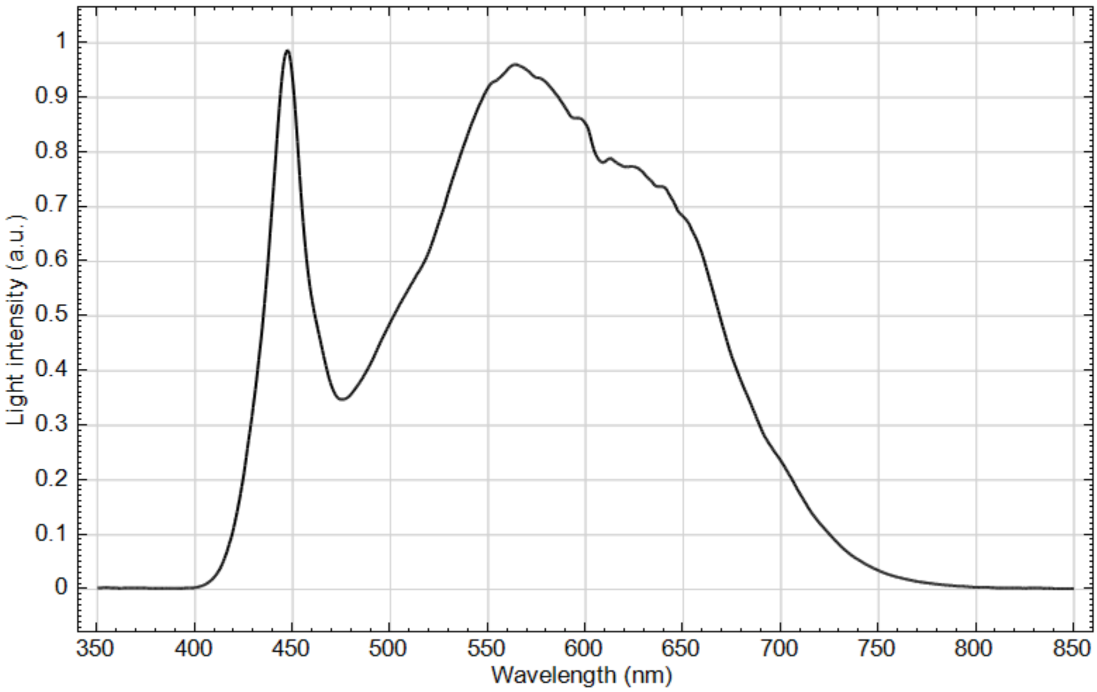

Figure 89 – Typical spectral distribution of the warm white LED

Note

The information in this appendix is derived from the specifications provided by Philips LUMILEDS and are subject to change without notice.

The Philips LUMILEDS article codes used in the Autolab LED Covers are provided below for reference:

- **LXML-PWC1-0120**: Cool white, 220 lm, 700 mA
- **LXW9-PW30**: ANSI (Warm) white, 145 lm, 700 mA
- **LXML-PWN1-0120**: Neutral white, 220 lm, 700 mA
- **LXM2-PD01-0050**: Red, 102 lm, 700 mA
- **LXM3-PD01-0260**: Deep Red, 580 mW, 700 mA
- **LXML-PB01-0040**: Blue, 70 lm, 700 mA
- **LXML-PM01-0100**: Green, 161 lm, 700 mA
- **LXML-PE01-0070:** Cyan, 122 lm, 700 mA
- **LXM2-PH01-0070:**, Red-Orange, 134 lm, 700 mA
- **LXM2-PL01-0000**: Amber, 132 lm, 700 mA

Appendix 5 – Mechanical specifications

This appendix provides a schematic overview of the items supplied with the Autolab Optical Bench. The dimensions of these items are also provided here.

The dimensions of the LED holder and LED cover are shown in Figure 90.

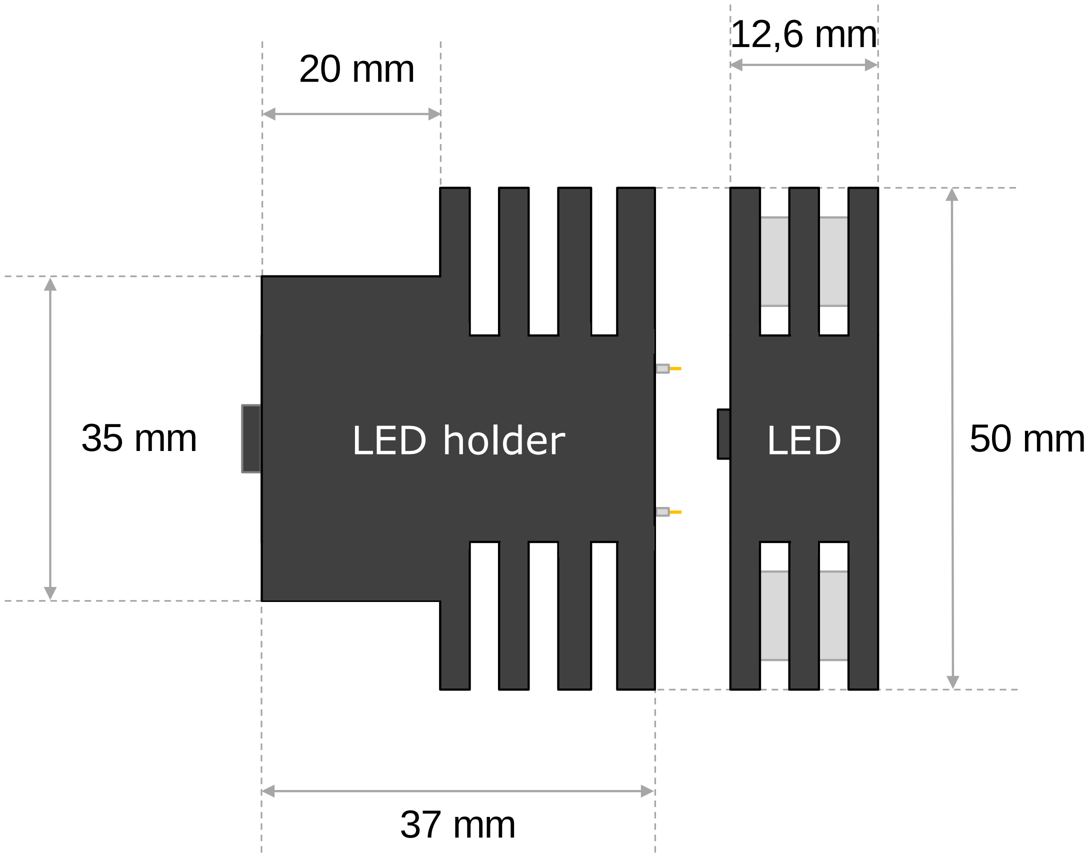

Figure 90 – Dimensions of the LED holder and LED cover

The dimensions of the optical rail and its accessories is shown in Figure 91.

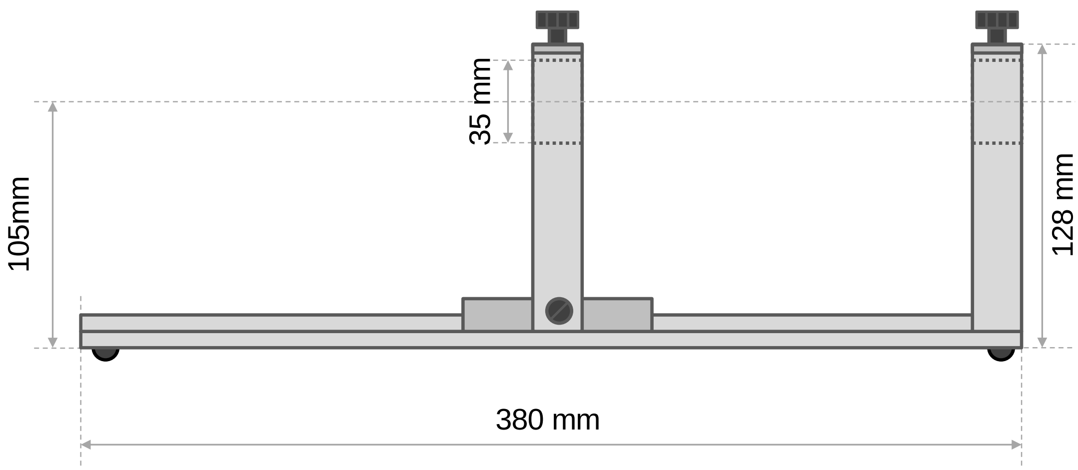

Figure 91 – Dimensions of the optical rail

The dimensions of the photodiode holder are shown in Figure 92.

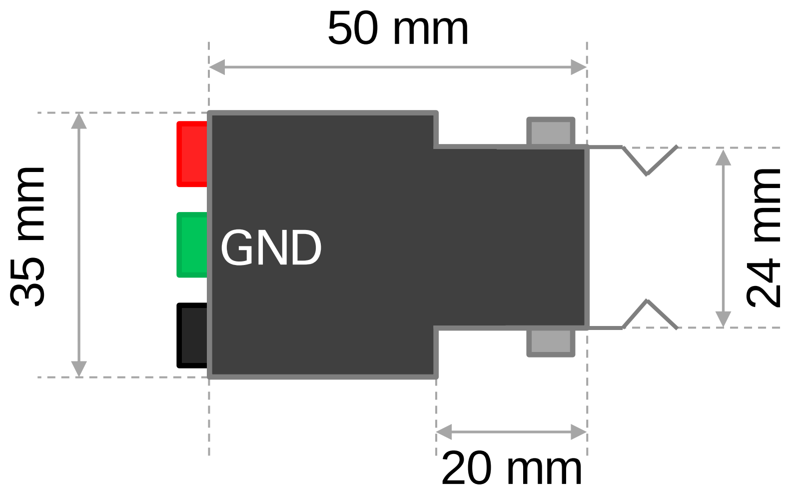

Figure 92 – Dimensions of the photodiode holder

05/2025

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