Spectrophotometer and Light Source

User Manual

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Metrohm
Autolab

Spectrophotometer and Light Source

1.0

User Manual

Metrohm Autolab
Metrohm Autolab B.V.
3543 AV Utrecht

Although all the information given in this documentation has been checked with great care, errors cannot be entirely excluded. Should you notice any mistakes please send us your comments using the address given above or at autolab@metrohm.com.

Table of contents

1   Autolab Spectroelectrochemistry product range

The Autolab Spectroelectrochemistry product range consists of modular spectrophotometers and light sources that can be controlled from the NOVA software. These devices can be used during most electrochemical measurements for hyphenated experiments .

Figure 1   The Autolab Spectrophotometer and Light Source

This manual provides information on the following products:

- **Spectrophotometers**
  - Autolab Spectrophotometer-UA for UV/VIS/NIR applications (200 - 1100 nm)
  - Autolab Spectrophotometer-UB for UV/VIS applications (200 - 850 nm)
- **Light sources**
  - Autolab Compact Deuterium/Halogen Light Source (200 - 2500 nm)
- **Optical fibers**
  - Optical fiber for UV/VIS/NIR, 2 m/200 μm
  - Optical fiber for UV/VIS, 2 m/200 μm

2 Hardware description

2.1 Autolab Spectrophotometers

The **Autolab Spectrophotometers** are compact instruments based on an optical bench with a focal length of 75 mm and a 50 μm entry slit developed in a symmetrical *Czerny-Turner* design. These instruments are fitted with a 2048 pixel CCD detector optimized for high-speed applications *(see figure 2, page 2)*.

Figure 2   The Autolab Spectrophotometer

The spectrophotometers are supported by the Autolab NOVA software and can be controlled at any time, with or without an Autolab potentiostat/galvanostat connected to the host computer.

The following features are included in the spectrophotometers:

- Order sorting coating to avoid stray light and second order effects.
- Detector Collection Lens for enhanced sensitivity.
- Deep UV coating to reduce second order effects from UV response.

The spectrophotometers can be connected to optical fibers using a standard **SMA** connection (SMA 905).

2.1.1 Scope of delivery

The Autolab spectrophotometer is supplied with the following parts:

- Autolab spectrophotometer unit
- USB cable
- SMA-905 cover
- DIO48.SPEC.TRIGGER.S trigger cable
- DIO12.SPEC.TRIGER.S trigger cable

ℹ️ Depending on the type of Autolab potentiostat/galvanostat used to control the spectrophotometer, one of the two types of trigger cables will be used. Please store the unused trigger cable for future use.

2.1.2 Spectrophotometer front panel

The front panel of the Autolab Spectrophotometer is shown in *Figure 3*.
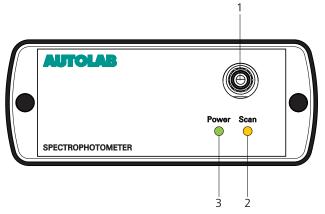
Figure 3   The front panel of the Autolab spectrophotometer

1   SMA-905 connector
Used to connect an optical fiber from the electrochemical cell.

2   Scan LED
Used to indicate if a spectrum is being acquired by the spectrophotometer (the LED is lit when a spectrum is acquired).

3   Power LED
Used to indicate if the spectrophotometer is powered (the LED is lit when this is the case).

2.1.3 Spectrophotometer back plane

The back plane of the Autolab Spectrophotometer is shown in *Figure 4*.

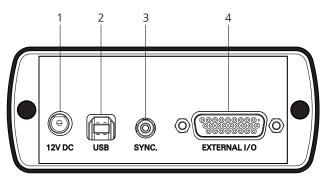*Figure 4   The back plane of the Autolab spectrophotometer*

1   12V DC plug
Can be used to power the spectrophotometer. This power plug is not required when the spectrophotometer is powered by a USB port on the computer.

2   USB connector
Used to interface the spectrophotometer with the computer and to provide power.

3   SYNC. connector
This connector can be used to synchronize the spectrophotometer with another spectrophotometer.

4   EXTERNAL I/O connector
26 pin, female SUB-D connector used to interface to the Autolab potentiostat/galvanostat.

2.1.4   Spectrum triggering

The Autolab spectrophotometer is designed to work in conjuction with the Autolab potentiostat/galvanostat. When used in this combination, the spectrophotometer will work as a slave unit. The spectrophotometer can be controlled in two different modes:

1.   **Software trigger mode:** the default control mode of the spectrophotometer. In this mode, the spectrophotometer will measure a single spectrum whenever a software trigger is received from NOVA through the host computer. The measured data is immediately transferred to the computer. When this is finished, the spectrophotometer can acquire a new spectrum if needed. This measurement mode is accessible using the **Manual control** and the **Spectroscopy** command, in software trigger mode (see *Chapter 4.1* and *Chapter 4.2.1, respectively)*.

2.   **DIO trigger mode:** in this mode, the spectrophotomer is configured to wait until a TTL trigger pulse, sent by the Autolab potentiostat/galvanostat, is received on pin 6 relative to pin 1 of the **EXTERNAL I/O** connector (item 4 in *Figure 4)*. NOVA automatically adresses the correct pins when sending the trigger pulse. A single spectrum is measured whenever the rising edge of a TTL trigger pulse is detected and the measured data is stored in the on-board memory of the spectrophotometer. This measurement mode is only available using the **Spectroscopy** command, in DIO trigger mode (see *Chapter 4.2.2)*.

When the spectrophotometer is used in **Software trigger mode**, the measurements are controlled by the host computer. The timing for triggering in this mode is aproximately 50 ms.

When the spectrophotometer is used in **DIO trigger mode**, the spectrophotometer reacts on the rising edge of the TTL trigger pulse *(see figure 5, page 5)*.
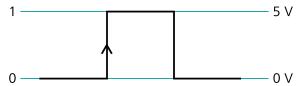

Figure 5 The spectrophotometer reacts on the rising edge of a TTL trigger pulse

This means that very accurate synchronization can be obtained as the triggering occurs on the timescale of 1 ms.

ℹ️ More information can be found in *Chapter 4*.

2.1.5 Specifications

The detailed specifications of the Autolab Spectrophotometers are provided in *Table 1*.

Table 1 Specifications of the Autolab Spectrophotometers

Specification	Autolab Spectro-photometer UA	Autolab Spectro-photometer UB
Wavelength range	200 - 1100 nm	200 - 850 nm
Resolution	2.5 nm	1.15 nm
Stray light	0.22 %	0.15 %
Sensitivity	310,000 counts/µW per ms integration time	
Detector	CCD linear array, 2048 pixels	
Maximum number of scans	1013 scans of 2048 pixels	
A/D converter	16 bit, 2 MHz	
Integration time	1.1 ms - 600 s	
Power requirements	12 V DC / 350 mA	
Dimensions	175 mm x 110 mm x 44 mm	

2.2   Autolab Light Source

The **Autolab Light Source** is a compact deuterium/halogen light source. It provides a good light output between 200 and 2500 nm for nearly all absorbance chemistry applications. Deuterium emits light between 200 and 550 nm, where the halogen takes over up to 2500 nm *(see figure 6, page 6)*.

Figure 6   The Autolab Light Source

This light source provides a TTL-controlled shutter, which can be controlled by the NOVA software.

The light source can be connected to optical fibers using a standard **SMA** connection (SMA 905).

⚠ CAUTION

Strong light source

This can be harmful to the eyes!

- Do not look at the light produced without approved eye protection gear.

⚠ WARNING

UV light source

This can be harmful to the eyes!

- Do not look at the light produced without approved eye protection gear.

2.2.1 Scope of delivery

The Autolab light source is supplied with the following parts:

- Autolab light source unit
- 12 V / 1.0 A power adaptor
- SMA-905 cover
- Power cable

2.2.2 Light source front panel

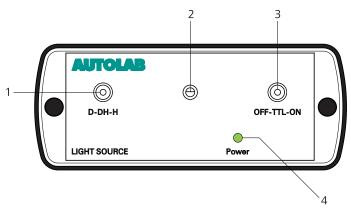The front panel of the Autolab Light Source is shown in *Figure 7*.

Figure 7 The front panel of the Autolab light source

1 D-DH-H switch
Used to select the active light bulb(s) used by the light source. In D position, only the deuterium light bulb is on. In H position, only the halogen light bulb is on. In DH position, both light bulbs are on at the same time.

2 SMA-905 connector
Used to connect an optical fiber to the electrochemical cell.

3 OFF-TTL-ON
Used to select the state of the light source and the light source shutter. In OFF position, the light source is powered off. In TTL position, the light source is on and the shutter is controlled by TTL. In ON position, the light source is powered on and the shutter is in the open position.

2.2.3 Light source back plane

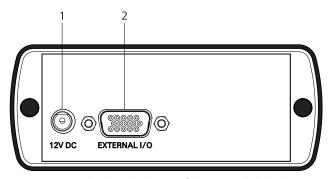The back plane of the Autolab Light Source is shown in *Figure 8*.

Figure 8   The back plane of the Autolab light source

1   12V DC plug
Used to power the light source.

2   EXTERNAL I/O connector
15 pin, female SUB-D connector used to interface to the Autolab potentiostat/galvanostat for TTL control of the shutter.

2.2.4   Unpacking and inspection instructions

Follow the guidelines below when unpacking and inspecting the light source:

1. Unpack your new power supply and Autolab Light Source carefully. Although the lamps are rigidly mounted in the light source, dropping this instrument can cause permanent damage.
2. Inspect the outside of the instrument and make sure that there is no damage to your unit. In case of damage contact Metrohm Autolab immediately and do **not** use the instrument!
3. Remove the SMA connector cover. Only do this if the light source is switched off.
4. Use this instrument in a clean laboratory environment only.

2.2.5   Shutter control

The Autolab Light Source is fitted with a metal pole switch (item 3 in *Figure 7* which can be set to three different positions:

- **OFF:** the light source is powered off and the shutter is closed.
- **TTL:** the light source is powered on and the shutter is open or closed depending on the input TTL signal provided on the EXTERNAL I/O connector, located on the back plane of the ligth source (item 2 in *Figure 8*).
- **ON:** the light source is powered on and the shutter is open.

When the shutter control is set to TTL, the Autolab can control its position by setting the voltage of pin 13 on the **EXTERNAL I/O** connector of the light source to 5 V with respect to pin 10 of the same connector (item 2 in *Figure 8*).

To close the shutter, pin 13 needs to be reset to 0 V with respect to pin 10. NOVA automatically adresses the correct pins when controlling the shutter *(see figure 9, page 9)*.
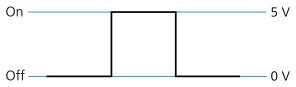
Figure 9   The shutter control through TTL

ℹ️ The shutter will remain in the specified position until changed by the user.

2.2.6   Specifications

The detailed specifications of the Autolab Light Source are provided in *Table 2*.

Table 2   Specifications of the Autolab Light Source

Specification	Deuterium lamp	Halogen lamp
Wavelength range	200 - 400 nm	400 - 2500 nm
Stability	< 1 mAU/h	< 1 mAU/h
Warm-up time	8 min	1 min
Drift	< 0.25 %/h	< 0.25 %/h
Optical power with 600 μm fiber	0.2 μW	7 μW
Lamp lifetime	1000 h	2000 h
Temperature range	5 °C - 35 °C	
Power requirements	12 V DC / 450 mA	
Dimensions	175 mm x 110 mm x 44 mm	

2.3   Optical fibers

The Autolab spectrophotometer and light source are designed to connect to the electrochemical cell using optical fibers. The standard dimensions of the optical fibers is 2 m in length and 200 μm in diameter.

⚠️ Handle the optical fibers with care. The fibers are flexible and can be bent. However, if this is done excessively, the fiber can break and will have to be replaced.

2.3.1 Optical fibers description

The Autolab optical fiber is shown in *Figure 2.3.1*.

Figure 10   The Autolab optical fibers

1 Label	**2 SMA-905 connector**
Specifies the type of fiber.	Used to connect to the light source, spectro-photometer and electrochemical cell.

2.3.2 Specifications

The detailed specifications of the Autolab optical fibers are provided in *Table 3*.

Table 3   Specifications of the Autolab Optical Fibers

Specification	AUT.FIBER.200.UVIR	AUT.FIBER.200.UV
Measurable range	250 - 2500 nm	200 - 850 nm
Length	2 m	
Diameter	200 µm	
Outer diameter	5.8 mm	
Minimum bending radius	18 mm	
Tensile strength	70 N	
Temperature range	-60 °C - 180 °C	

2.4   Trigger cables

In order to control the Autolab Spectrophotometer and Light Source, the supplied trigger cable is required.

Two different cables are available:

- **DIO48.SPEC.TRIGGER.S:** for all Autolab potentiostat/galvanostat instruments except the PGSTAT101, M101, PGSTAT204 or M204. Please refer to *Chapter 2.4.1* for more information.
- **DIO12.SPEC.TRIGGER.S:** for the Autolab PGSTAT101, M101, PGSTAT204 or M204. Please refer to *(see "Trigger cable for DIO12 instruments", chapter 2.4.2, page 12)* for more information.

🛈 The **PGSTAT302F** cannot be used in combination with the Autolab Spectrophotometer and Light Source.

2.4.1   Trigger cable for DIO48 instruments

Figure 11 shows an overview of the connections provided by the trigger cable for DIO48 Autolab instruments (part number: DIO48.SPEC.TRIGGER).
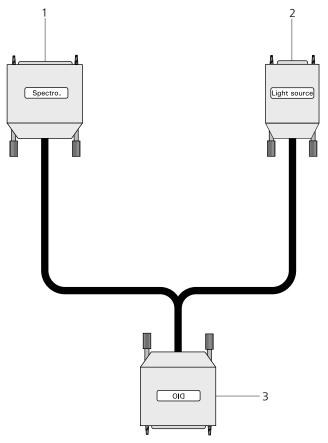
Figure 11   The trigger cable for DIO48 Autolab instruments

1   Spectro. connector
26 pin male SUB-D connector used to connect to the **EXTERNAL I/O** connector of the Autolab spectrophotometer (item 4 in *Figure 4*.

2   Light source connector
15 pin male SUB-D connector used to connect to the **EXTERNAL I/O** connector of the Autolab light source (item 2 in *Figure 8*.

3   DIO connector
25 pin male SUB-D connector used to connect to one of the **DIO** connectors of the Autolab potentiostat/galvanostat.

2.4.2   Trigger cable for DIO12 instruments

Figure 12 shows an overview of the connections provided by the trigger cable for DIO12 Autolab instruments (part number: DIO12.SPEC.TRIGGER.S).
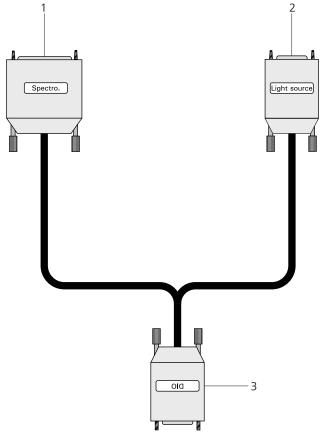
Figure 12   The trigger cable for DIO12 Autolab instruments

1   Spectro. connector
26 pin male SUB-D connector used to connect to the **EXTERNAL I/O** connector of the Autolab spectrophotometer (item 4 in *Figure 4*.

2   Light source connector
15 pin male SUB-D connector used to connect to the **EXTERNAL I/O** connector of the Autolab light source (item 2 in *Figure 8*.

3   DIO connector
15 pin (DIO12) male SUB-D connector used to connect to the **DIO** connector of the Autolab potentiostat/galvanostat.

3 Installation

This chapter describes the installation of the Autolab Spectrophotometer and Light Source.

3.1 Spectrophotometer and light source installation

In order to control the Autolab Spectrophotometer and Light Source, the supplied trigger cable must be connected to these devices and the Autolab potentiostat/galvanostat.

Two different cables are available:

- **DIO48.SPEC.TRIGGER.S:** for all Autolab potentiostat/galvanostat instruments except the PGSTAT101, M101, PGSTAT204 or M204. This cable is fitted with a male 25-pin SUB-D connector, which can be connected to one of the two DIO connectors of the Autolab, a 26-pin SUB-D connector, which can be connected to the back plane of the Spectrophotometer and a male 15-pin SUB-D connector, which can be connected to the back plane of the Light source. Each connector is suitably labeled.
- **DIO12.SPEC.TRIGGER.S:** for the Autolab PGSTAT101, M101, PGSTAT204 or M204. This cable is fitted with a male 15-pin SUB-D connector, which can be connected to the DIO connector of the Autolab, a 26-pin SUB-D connector, which can be connected to the back plane of the Spectrophotometer and a male 15-pin SUB-D connector, which can be connected to the back plane of the Light source. Each connector is suitably labeled.

ℹ The PGSTAT302F cannot be used in combination with the Autolab Spectrophotometer and Light Source.

3.1.1 Instruments with two DIO connectors

This section describes the installation of the Autolab spectrophotometer and light source for the following instruments:

- N Series Autolab
    - PGSTAT302N
    - PGSTAT128N
    - PGSTAT100N
- 7 Series Autolab
    - PGSTAT302
    - PGSTAT30
    - PGSTAT12
    - PGSTAT100

- µAutolab type II and III

1 Connect the PGSTAT labeled connector to **DIO** connector P1 or P2 on the Autolab potentiostat/galvanostat instrument. The corresponding connector will later be selected in the software, as described in *(see "Software trigger", chapter 4.2.1, page 28)*.

2 Connect the light source labeled connector to the **EXTERNAL I/O** connector located on the back plane of the light source (item 2 in *Figure 8*).

3 Connect the spectrophotometer labeled connector to the **EXTERNAL I/O** connector located on the back plane of the spectrophotometer (item 4 in *Figure 4*).

3.1.2   Instruments with a single DIO connector

This section describes the installation of the Autolab spectrophotometer and light source for the following instruments:

- Compact Series
  – PGSTAT101
  – PGSTAT204
- Multi Autolab Series
  – M101
  – M204

1 Connect the PGSTAT labeled connector to the **DIO** connector Autolab potentiostat/galvanostat instrument or module.

2 Connect the light source labeled connector to the **EXTERNAL I/O** connector located on the back plane of the light source (item 2 in *Figure 8*).

3 Connect the spectrophotometer labeled connector to the **EXTERNAL I/O** connector located on the back plane of the spectrophotometer (item 4 in *Figure 4*).

3.2   Software installation

To control the Autolab Spectrophotometer and Light Source, version 2.1.2 or later of NOVA is required.

ℹ️ Please refer to the NOVA user manual for more information.

❗ Make sure NOVA 2.1.2 or later in installed on the computer before continuing with the installation.

Connect the Autolab Spectrophotometer to the host computer using the provided USB cable. The installation of the spectrophotometer will be triggered on the computer *(see figure 13, page 16)*.
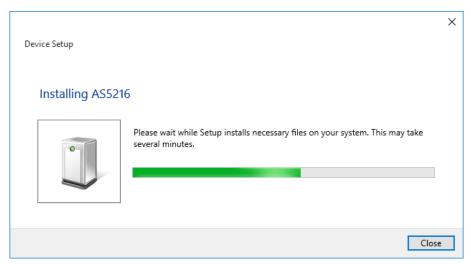

Figure 13   The Spectrophotometer installer

The installation will complete automatically.

Start NOVA 2.1.2 or later. The Spectrophotometer will be identified and listed in the **Instruments** panel of the **Dashboard** *(see "Spectrophotometer control", chapter 4, page 17)*.

4 Spectrophotometer control

Autolab spectrophotometers connected to the host computer can be controlled using the NOVA software. The instruments can be controlled in two different ways:

- Using the provided manual control *(see "Spectrophotometer manual control", chapter 4.1, page 17)*.
- Using a NOVA procedure *(see "Spectrophotometer procedure control", chapter 4.2, page 28)*.

4.1 Spectrophotometer manual control

Double clicking a spectrophotometer tile in the **Instruments** panel will open the manual control panel of the corresponding spectrophotometer *(see figure 14, page 17)*.
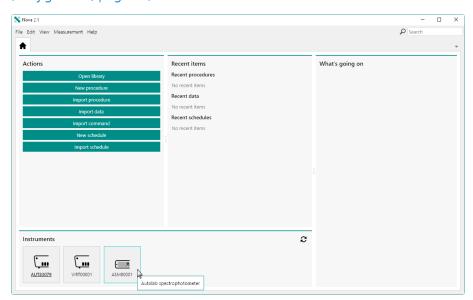

Figure 14   Opening the spectrophotometer manual control panel

The spectrophotometer manual control panel will be displayed in a new tab *(see figure 15, page 18)*.

Figure 15   The manual control panel of the spectrophotometer

The spectrophotometer manual control panel provides two sub-panels:

- **Hardware setup:** a panel displaying hardware setup information of the spectrophotometer.
- **Spectrophotometer display:** a panel displaying the measurement controls of the spectrophotometer.

4.1.1   Hardware setup panel

The configuration of the connected spectrophotometer can be adjusted in the **Hardware setup** panel *(see figure 16, page 18)*.
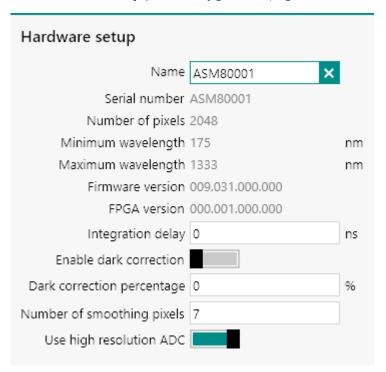

Figure 16   The Spectrophotometer Hardware setup panel

The **Hardware setup** panel displays information or properties of the connected spectrophotometer. The following properties are available:

- **Name:** an input field which can be used to give a dedicated name to the instrument. By default, the name of the instrument corresponds to the instrument serial number.

- **Serial number:** a read-only field that provides the serial number of the instrument.
- **Number of pixels:** a read-only field that provides the number of pixels of the detector of the instrument.
- **Minimum wavelength:** a read-only field that provides the lowest measurable wavelength of the detector the instrument.
- **Maximum wavelength:** a read-only field that provides the highest measurable wavelength of the detector the instrument.
- **Firmware version:** a read-only field that provides the firmware version of the instrument.
- **FPGA version:** a read-only field that provides the FPGA version of the instrument.
- **Integration delay:** an input field which can be used to specify the integration delay in ms (0 by default). This delay time corresponds to the time between the detection the TTL trigger pulse required to acquire a spectrum (in DIO trigger mode) and the start of the integration cycle of the spectrophotometer.
- **Enable dark correction:** a ▮▭ toggle which can be used to enable or disables the dark correction (default OFF).
- **Dark correction percentage:** an input field which can be used to specify the percentile value of dark correction (0-100 %). For more details, please continue to the section below about Dark Correction.
- **Number of smoothing pixels:** an input field which can be used to specify the number of pixels used in the smoothing algorithm. When this value is set to 0, no smoothing is used. The optimal value depends on the fiber diameter, pixel size and type of spectrophotometer.
- **Use high resolution ADC:** a ▭▮ toggle which can be used to enables or disables the high resolution ADC of the spectrometer. When enabled, the measured values are resolved using a 16 Bit ADC, when disabled a 14 Bit ADC is used instead (default ON).

Table 4 provides an overview of the optimal number of **Smoothing pixels** for the Autolab spectrophotometers in function of the fiber diameter.

Table 4   Optimal smooth pixel settings for the different optical fibre diameters

Fiber diameter (µm)	Optimal smoothing pixels
10	0
25	1
50	2
100	3
200 (default)	**7** (default)
400	14

Fiber diameter (µm)	Optimal smoothing pixels
500	17
600	21

ℹ The smoothing function will not apply to the first *x* and last *x* points, where *x* is the specified **Number of smoothing pixels**.

Dark Correction: The pixels of the CCD detector of the spectrophotometer are thermally sensitive, which causes a small dark current, even without exposure to light. The Autolab spectrophotometers are fitted with dedicated *black pixels*. To get an approximation of the dark current, these black pixels can be taken and subtracted from the raw data. This will happen if the **Enable dark correction** property is is enabled.

Since these optical black pixels have the same thermal behaviour as the active pixels, the correction is dynamic. Enabling dark correction will therefore result in a baseline fluctuating round zero, and measurement data will be less sensitive for temperature changes than with dark correction disabled.

The weight of the black pixels subtracted from the raw data can be set using the **Dark correction percentage** property. When this value is set to 0 %, no correction is performed. When this value is set to 100 %, the value of the black pixels is completely subtracted from the measured data.

ℹ This correction is different from the calculations of absorbance and transmittance that involve a dark spectrum.

4.1.2 Spectrophotometer display panel

The **Spectrophotometer display** panel provides manual control of the connected Autolab spectrophotometer *(see figure 17, page 20)*.
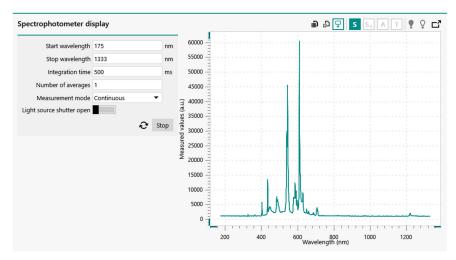

Figure 17 The Spectrophotometer display panel

The following properties are available *(see figure 18, page 21)*:

- **Start wavelength:** an input field which can be used to specify the start wavelength used in the measurement, in nm. This property is automatically set to the lowest possible value supported by the spectrophotometer.
- **Stop wavelength:** an input field which can be used to specify the stop wavelength used in the measurement, in nm. This property is automatically set to the highest possible value supported by the spectrophotometer.
- **Integration time:** an input field which can be used to specify the integration time, in ms (smallest possible value: 1.1 ms).
- **Number of averages:** an input field which can be used to specify the number of averaged spectra, as an integer.
- **Measurement mode:** a drop-down control that can be used to specify the measurement mode (continuous or single). In continuous mode, the spectrophotometer will acquire spectra until stopped by the user. In single mode, the spectrophotometer will acquire a single spectrum.
- **Light source shutter open:** a toggle that allows the light source shutter to be opened and closed with a TTL pulse from the default Autolab instrument. Please ensure that the hardware is set to the **TTL** shutter control position using the toggle on the front pannel of the Autolab light source when using this toggle.
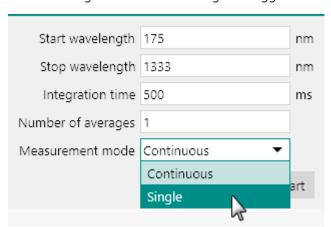

Figure 18   The measurement properties

To start the acquisition of a spectrum, the `Start` button can be pressed. Depending on the **Measurement mode** property, the spectrophotometer will acquire one or more spectra and display the measured data in the plot on the right hand side *(see figure 19, page 22)*.
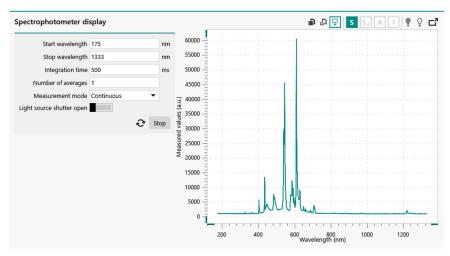
Figure 19   Measured spectra are displayed in the plot on the right-hand side

ℹ The measured data is displayed in arbitrary units.

ℹ While spectra are being acquired, the **Hardware setup** of the spec-trophotometer cannot be adjusted.

If needed, the measurement properties can be adjusted while spectra are acquired.

In continuous measurement mode, the acquisition of data can be stopped by pressing the Stop button.

After stopping the acquisition, it is possible to save the last measured spectrum as a *Dark spectrum* or as a *Reference spectrum*, by clicking the 💡 button or 💡 button, respectively *(see figure 20, page 22)*.
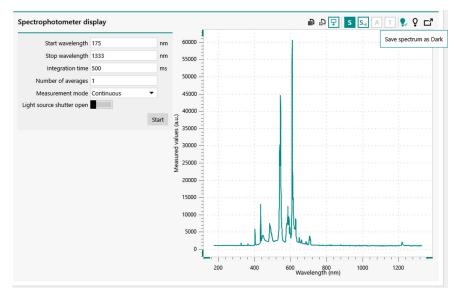
Figure 20   Saving a measured spectrum as Dark spectrum

When a *Dark* or *Reference* spectrum is saved, a check mark (💡✓ or 💡✓) will be visible in the top right corner of the **Spectrometer display** window *(see figure 21, page 23)*.
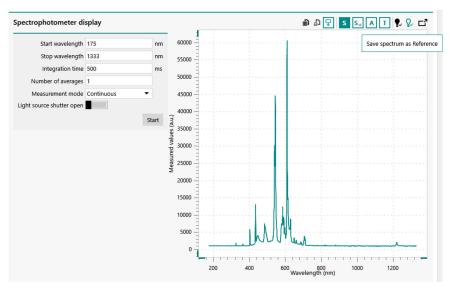

Figure 21   Saved spectra are indicated by a check mark

ℹ️ It is possible to overwrite the saved *Dark* or *Reference* spectrum by clicking the associated buttons again.

ℹ️ Changing the acquisition properties will discard the saved *Dark* and *Reference* spectrum.

The **Spectrophotometer display** panel provides the possibility to toggle between different display modes, using the buttons (S, S_d, A, T) located in the top right corner *(see figure 22, page 23)*.
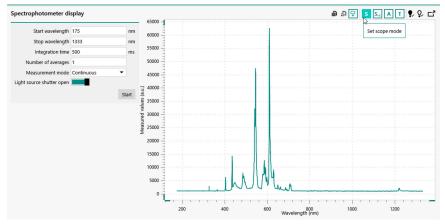

Figure 22   Controlling the display mode of the measured data

The following display modes are available:

- **Scope mode** (S)**:** this mode shows the raw data from the spectro-photometer in arbitrary units. This display mode is always available.

- **Dark corrected scope mode** ($\boxed{S_d}$)**:** this mode shows the raw data ($S_{Measured}$) from the spectrophotometer corrected with the stored *Dark* spectrum (S_{Dark}), in arbitrary units. This display mode is only available if a *Dark* spectrum is saved. The dark corrected scope data is calculated according to:

$$S_{-d} = S_{Measured} - S_{Dark}$$

- **Absorbance mode** ($\boxed{A}$)**:** this mode shows the absorbance values calculated from the measured data ($S_{Measured}$), the stored *Dark* spectrum (S_{Dark}) and the stored *Reference* spectrum ($S_{Reference}$). This display mode is only available if a *Dark* and a *Reference* spectrum are saved.

$$A = -\log\left(\frac{S_{Measured} - S_{Dark}}{S_{Reference} - S_{Dark}}\right)$$

- **Transmittance mode** ($\boxed{T}$)**:** this mode shows the transmittance values calculated from the measured data ($S_{Measured}$), the stored *Dark* spectrum (S_{Dark}) and the stored *Reference* spectrum ($S_{Reference}$). This display mode is only available if a *Dark* and a *Reference* spectrum are saved.

$$T = 100 \cdot \left(\frac{S_{Measured} - S_{Dark}}{S_{Reference} - S_{Dark}}\right)$$

ℹ️ The modes can be toggled while spectra are acquired.

4.1.2.1   Step through data

The **Spectrophotometer display** panel provides the possibility to toggle the *Step through data* mode on or off using the the ⬇️ button in the top right corner *(see figure 23, page 24)*.
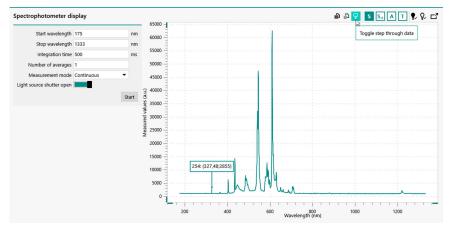

Figure 23   The Step through data option can be used in the Spectro-photometer control panel

When the *Step through data* mode is on, an additional indicator is added to the plot, showing the X and Y coordinates of the point indicated by the arrow, as shown in *Figure 23*.

ℹ️ The indicator is always shown for the first data point of the plot.

It is possible to relocate the indicator in the following ways *(see figure 24, page 25)*:

- By clicking anywhere in the plot area: the indicator is relocated to the closest data point of the plot.
- Using the **[←]/[→]**: the indicator can be moved by 1 point at a time.
- Using the **[←]/[→]** and **[CTRL]**: the indicator can be moved by 10 points at a time.
- **[←]/[→]** and **[CTRL]** and **[SHIFT]**: the indicator can be moved by 100 points at a time.
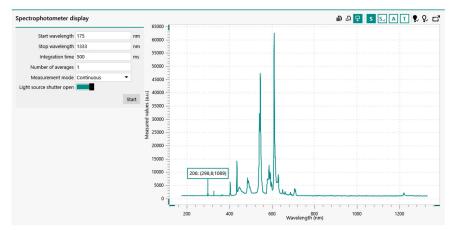
Figure 24   It is possible to relocate the indicator using the mouse or keyboard

4.1.2.2   Export data and plot

The **Spectrophotometer display** panel provides the possibility to export the measured data. Measured value can either be exported to ASCII or Excel format or as an image, using the provided 📊 and 🗐 buttons in the top right corner of the panel *(see figure 25, page 26)*.
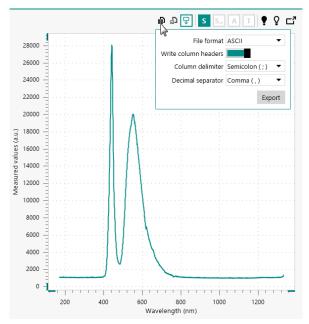
Figure 25   The measured data can be exported

Clicking the 🖹 button displays a pop-out menu providing controls of the format of the exported file *(see figure 26, page 26)*.
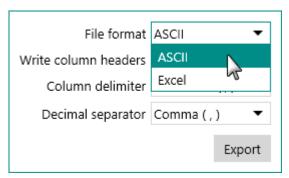

Figure 26   The data can be exported to ASCII or Excel

The data can be exported as ASCII or to Excel. The following properties can be specified:

- **File format:** specifies the format of the output file (ASCII or Excel), using the provided drop-down list.
- **Write column headers:** a ▬ toggle that can be used to indicate if the names of the signals need to be added to the output file.
- **Column delimiter:** specifies the symbol used as a column separator, using the provided drop-down list. This property is only available for ASCII output.
- **Decimal separator:** specifies the decimal separator symbol used in the output file, using the provided drop-down list. This property is only available for ASCII output.

Clicking the Export button displays a save dialog window which can be used to specify the filename and location *(see figure 27, page 27)*.

Figure 27   Specifying the filename and location

ℹ️ All of the available data is exported to the file.

Clicking the 🗗 button displays a pop-out menu providing controls of the format the size of the exported image file *(see figure 28, page 27)*.
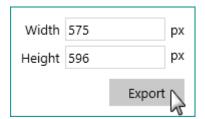

Figure 28   Exporting the data as image

Two types of image types can be used when exporting plots:

- **Pixel based output:** the data is exported to a pixel based file format, with or without compression (*.bmp, *.png, *.jpg, *.tiff, *.gif). When this type is used, the size of the image is specified in pixels.
- **Vector based output:** the data is exported to a vector file format (*.emf, *.svg, *.wmf). When this type is used, the size of the image is specified in arbitrary units.

Clicking the ⬚Export button displays a Windows explorer dialog which can be used to specify the path, name and file type used to create the output image file *(see figure 29, page 27)*.
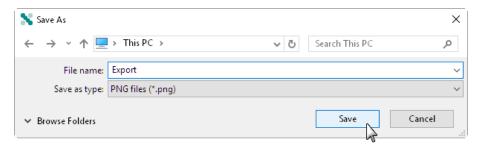

Figure 29   Specifying the name, location and type of output file

4.2   Spectrophotometer procedure control

The **Spectroscopy** command, available in the External devices group of commands can be used to control connnected Autolab spectrophotometers.

The **Spectroscopy** command can be used in two different modes, which can be selected using the provided drop-down list *(see figure 30, page 28)*:
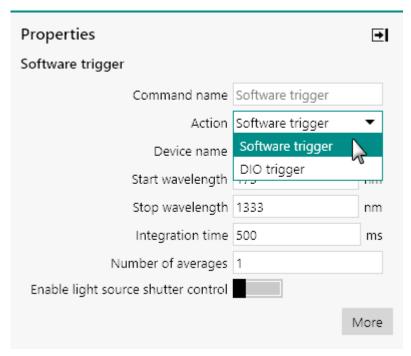

Figure 30   Two modes are provided by the Spectroscopy command

1.   Software trigger (default mode)
2.   DIO trigger

ℹ️ The **Spectroscopy** command description and properties available for editing are automatically adjusted according to the specified mode.

4.2.1   Software trigger

The following properties are available when the command is used in the *Software trigger* mode *(see figure 31, page 29)*:
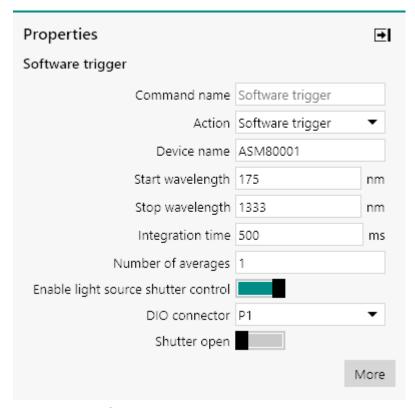
Figure 31   Software trigger mode properties

- **Command name:** a user-defined name for the command.
- **Device name:** specifies the name of the spectrophotomer used in the measurement. This property is automatically adjusted to the name of the connected spectrophotometer.
- **Start wavelength:** an input field which can be used to specify the start wavelength used in the measurement, in nm. This property is automatically set to the lowest possible value supported by the spectrophotometer.
- **Stop wavelength:** an input field which can be used to specify the stop wavelength used in the measurement, in nm. This property is automatically set to the highest possible value supported by the spectrophotometer.
- **Integration time:** an input field which can be used to specify the integration time, in ms (smallest possible value: 1.1 ms).
- **Number of averages:** an input field which can be used to specify the number of averages, as an integer.
- **Enable light source shutter control:** specifies if the command should control the light source shutter position using the provided ▮▭ toggle. This requires a physical connection between the Autolab potentiostat/galvanostat and the spectrophotometer. It is also necessary to set the shutter control to TTL mode on the connected light source.

- **DIO connector:** specifies which DIO connector is used to interface to the light source, using the provided drop-down list. This property is only visible if the *Enable light source shutter control* property is set to on. For the PGSTAT101, M101, PGSTAT204 and M204 instruments, this property is not shown.
- **Shutter open:** specifies the state of the light source shutter, using the provided ▮▭ toggle. This property is only visible if the *Enable light source shutter control* property is set to on.

ℹ The light source shutter will remain in the specified state until changed.

⚠ This mode requires a physical connection between the light source and the Autolab DIO connector if the *Enable light source shutter control* property is set to on. Please refer to *Chapter 3.1.1* and *Chapter 3.1.2* for more information.

4.2.2 DIO trigger

The following properties are available when the command is used in the *DIO trigger* mode *(see figure 32, page 30)*:
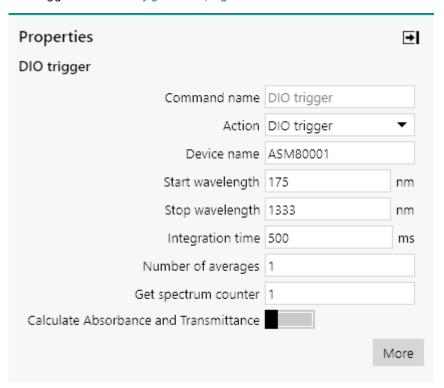

Figure 32   DIO trigger mode properties

- **Command name:** a user-defined name for the command.
- **Device name:** specifies the name of the spectrophotometer used in the measurement.

- **Start wavelength:** specifies the start wavelength used by the spectrophotometer, in nm.
- **Stop wavelength:** specifies the start wavelength used by the spectrophotometer, in nm.
- **Integration time:** specifies the integration time used by the spectrophotometer, in ms.
- **Number of averages:** specify the number of averages used by the spectrophotometer.
- **Get spectrum counter:** the counter value used by the triggering command.
- **Calculate Absorbance and Transmittance:** specifies if the measured values should be converted to absorbance and transmittance using values of a dark spectrum and reference spectrum using the provided ▮▭ toggle.

If the **Calculate Absorbance and Transmittance** property is on, it is necessary to link two single spectra to the **Spectroscopy** command. Two input anchoring points will be added to the command *(see figure 33, page 31)*.
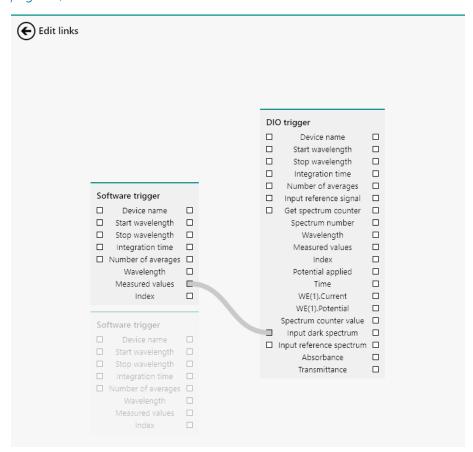

Figure 33   Dark and reference spectra can be linked to the Spectroscopy command

Using these two anchoring points, a dark spectrum and a reference spectrum can be linked to the **Spectroscopy** command in order to convert the measured values to absorbance and transmittance.

These values are calculated using the measured values ($S_{Measured}$), the linked *Dark* spectrum values (S_{Dark}) and the linked *Reference* spectrum values ($S_{Reference}$) according to:

- **Absorbance:**

$$A = -\log\left(\frac{S_{Measured} - S_{Dark}}{S_{Reference} - S_{Dark}}\right)$$

- **Transmittance:**

$$T = 100 \cdot \left(\frac{S_{Measured} - S_{Dark}}{S_{Reference} - S_{Dark}}\right)$$

The linked dark and reference spectra must be measured in the same conditions as those of the **Spectroscopy** command they are linked to.

The linked dark and reference spectra may be imported from saved data files using the **Import data** command. The spectroscopic measured values ($S_{measured}$) column from the imported data file can be linked to the appropriate anchoring point, either the **Input dark spectrum** or **Input reference spectrum**. The imported data must have been measured with an Autolab spectrophotometer using the same conditions as those of the **Spectroscopy** command they are linked to.

This mode requires a physical connection between the spectrophotometer and the Autolab DIO connector. Please refer to the Spectrophotometer User Manual for more information.

To use the **Spectroscopy** command in *DIO trigger* mode in a NOVA procedure, the command needs to be stacked onto the electrochemical measurement command that it is used with. *Figure 34* provides an example.
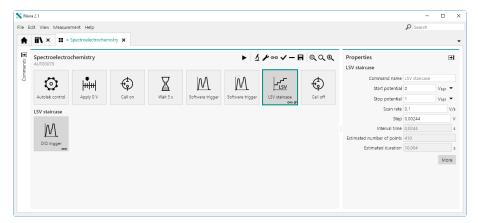
Figure 34   Stacking the spectroscopy command on a measurement command

Using this configuration, the **Spectroscopy** command used in *DIO trigger* mode will be executed whenever the parent measurement command (**LSV staircase** in *Figure 34*) will send a DIO trigger to the spectrophotometer.

At the end of a measurement, the electrochemical data will be provided by the parent measurement command and the spectroelectrochemical data will be provided by the **Spectroscopy** command *(see figure 35, page 33)*.
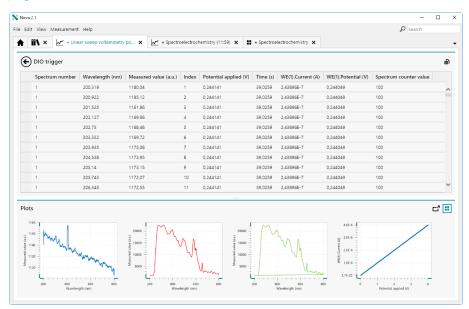

Figure 35   The spectroscopy and electrochemistry data is available in the Spectroscopy command

5 Storage instructions

When the Autolab spectrophotometer, light source, optical fibers and other parts used in spectroelectrochemical measurements are not used, it is recommended to protect the SMA-905 connections on all parts with the supplied covers.

The optical fibers should be stored in a protective container when not in use to avoid excessive bending.

All hardware should be protected from exposure to liquid and dust during use and storage.

6   Warranty

6.1   Warranty

The warranty on Autolab products is limited to defects or failures that are traceable to material, construction or manufacturing errors, which occur within 36 months from the day of delivery. In this case, the defects or failures will be rectified by Metrohm Autolab free of charge. Transport costs are to be paid by the customer, if applicable.

Glass breakage in the case of electrodes, cells or other parts is not covered by the warranty. Consumables (electrodes, optical fibers, the lamp within the light source, etc.) are not covered by the warranty.

If damage of the packaging is evident on receipt of the goods or if the goods show signs of transport damage after unpacking, the carrier must be informed immediately and a written damage report is demanded. Lack of an official damage report releases Metrohm Autolab from any liability to pay compensation.

If any instruments or parts have to be returned, the original packaging should be used. This applies to all instruments, electrodes, cells and other parts. If the original packaging is not available it can be ordered at Metrohm Autolab or at your local distributor. For damage that arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by Metrohm Autolab.

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