

1 – The BA, BIPOT & ARRAY module

The BA, BIPOT and ARRAY are optional modules that provide one additional working electrode, **WE(2)**¹. Depending on the type of module, the potential of the second working electrode can be expressed relative to the common reference electrode (Bipotentiostat mode) or relative to the first working electrode (Scanning bipotentiostat mode). The electrochemical signal provided by the additional working electrode, WE(2).Current, can be selected in the sampler.



Warning

The **BA** module is a switchable module. This means that the potential of the second working electrode can be expressed relative to both the reference electrode and the working electrode, WE(1). The **BIPOT** and **ARRAY** modules are not switchable and can only operate in a single mode: the BIPOT module is designed to work in Bipotentiostat mode, the ARRAY module is designed to operate in Scanning bipotentiostat mode.

2 – Using the second working electrode

The second working electrode, WE(2) provided by the BA, BIPOT or ARRAY² module does not require a dedicated measurement command. This means that the bipotentiostat module can be used with any measurement command which uses the sampler (CV staircase, LSV staircase, Chrono methods...).



Note

The bipotentiostat module can only work in potentiostatic mode. The main potentiostat can be set to galvanostatic mode. In BIPOT mode the potential of the second working electrode will be defined with respect to the reference electrode. In Scanning BIPOT mode, the potential of the second working electrode will be defined with respect to the first working electrode.

¹ NOVA also supports multiple BA modules. Please refer to the MultiBA tutorial for more information.

² In the rest of this tutorial, both the BA, BIPOT and ARRAY modules will be referred to as bipotentiostat module.

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Warning

The main difference between the BA and the BIPOT or ARRAY module is that the potential offset is set through the DAC164 #3 in the latter case, while in the former, the module has an on-board DAC. This means that when a BIPOT or ARRAY module is used, the DAC164 #3 is not available to control an external device.

In order to use the bipotentiostat module, the hardware setup must be configured accordingly (see Figure 1).

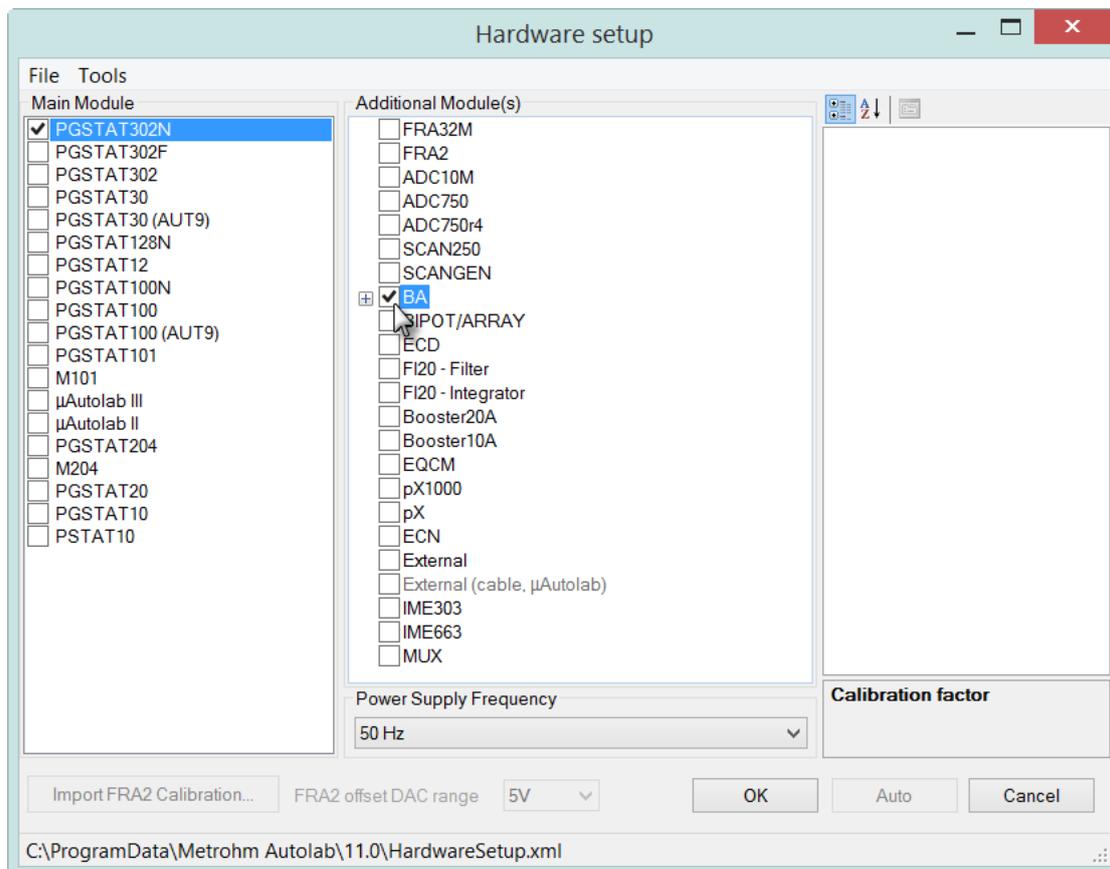


Figure 1 – Selecting the bipotentiostat module



Note

NOVA supports multiple BA modules. More information on instruments containing more than one BA module can be found in the MultiBA tutorial, available from the Help menu. The number of BA modules is automatically detected and the hardware setup self adjusts if necessary. BIPOT and ARRAY modules are not automatically detected and need to be specified manually in the hardware setup.

After the selection of the bipotentiostat module in the hardware setup, the **WE(2).Current** and the **WE(2).Charge** electrochemical signals becomes available in the signal sampler (see Figure 2).



Warning

The BIPOT and ARRAY modules and the ECD module are connected to the DAC164 channel #3. For this reason, they cannot be used at the same time. This DAC164 channel is also unavailable for controlling external devices like the Autolab RDE/RRDE whenever either one of these modules is used.

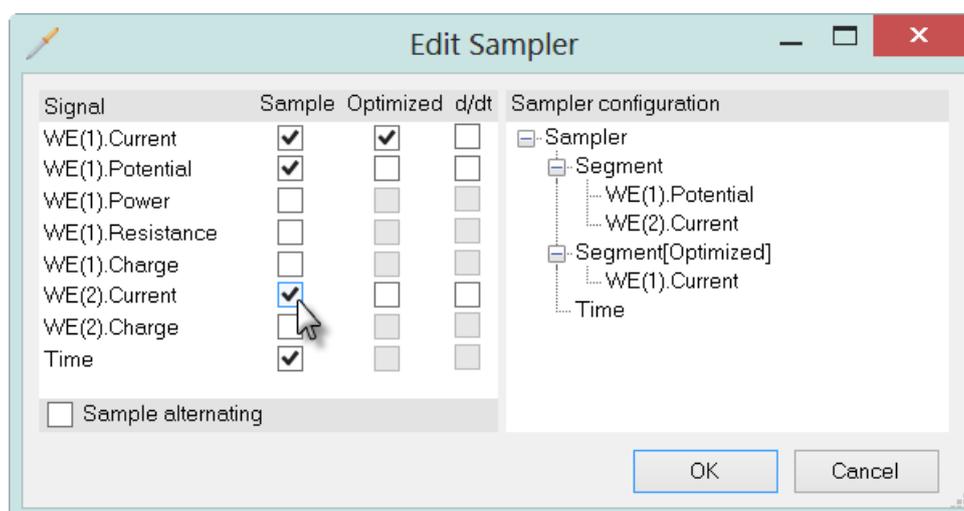


Figure 2 – The WE(2).Current and WE(2).Charge are available in the signal sampler

The WE(2).Current and WE(2).Charge signals can be sampled during the electrochemical experiment in the same way as the other available signals.



Note

It is not possible to sample the WE(2).Current and WE(2).Charge signals in FRA2 or FRA32M measurements or in measurements involving the fast sampling ADC (ADC10M or ADC750).

3 – Automatic current ranging

Independent Automatic current ranging options can be defined for each working electrode.

Clicking the Options button in the procedure editor frame displays the Automatic current ranging option window. If the bipotentiostat module is present, the automatic current ranging can be set for the second working electrode (see Figure 3).

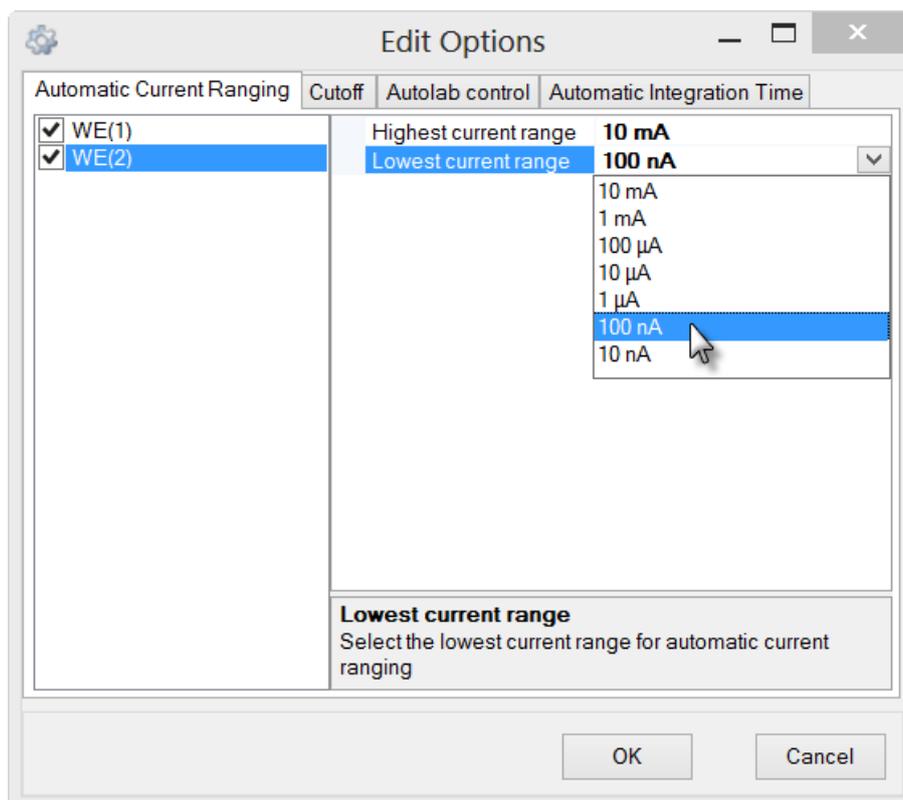


Figure 3 – The automatic current ranging settings for WE(1) and for WE(2)



Note

The automatic current ranging options can be defined for each working electrode, independently. For example, the automatic current option can be selected for WE(2) without having it selected for WE(1).

4 – Bipotentiostat module settings

All the settings of the bipotentiostat module can be defined using the *Autolab control* command. The settings for the second working electrode are listed in the BA or BIPOT/ARRAY section (see Figure 4 and Figure 5).

4.1 – BA module

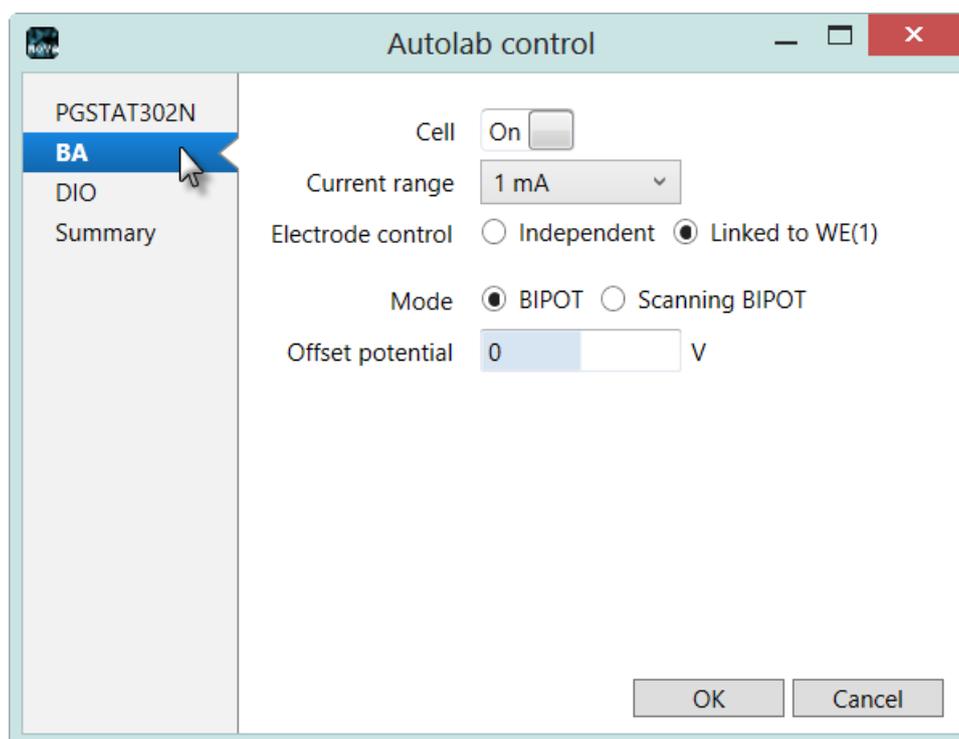


Figure 4 – The BA properties are set in the *Autolab control* command

Five settings are available with the **BA** module:

- **Cell:** used to set the cell switch for WE(2) on and off.
- **Current Range:** used to select the current range of WE(2).
- **Electrode control (independent, linked to WE(1)):** defines the relationship between the cell switch of WE(2) and WE(1). When this setting is set to *Linked to WE(1)*, the cell switch of WE(2) will automatically be set to the same status of the cell switch of WE(1). Using the *Set cell* command, both cell switches will be toggled at the same time. When this setting is set to *Independent*, the cell switch of WE(2) is decoupled from the cell switch of WE(1). Both switches must be set independently.
- **Mode:** defines the mode for the BA module (BIPOT/Scanning BIPOT).
- **Offset potential:** defines the potential difference between WE(2) and the reference electrode in BIPOT mode and between WE(2) and WE(1) in Scanning BIPOT mode.



Note

The default startup Electrode control setting is: linked to WE(1).



Warning

When the *electrode control* is set to *independent*, a specific order must be respected to avoid current leakage between WE(1) and WE(2). Always set the cell of WE(2) to ON **after** the cell has been set to ON for WE(1). Always set the cell of WE(2) to OFF **before** the cell has been set to OFF for WE(1).



Note

The *Set cell OFF* command switches the main potentiostat and the second working electrode off automatically, regardless of the *Electrode control* setting defined in the Autolab control window and the type of module.

4.2 – BIPOT or ARRAY module

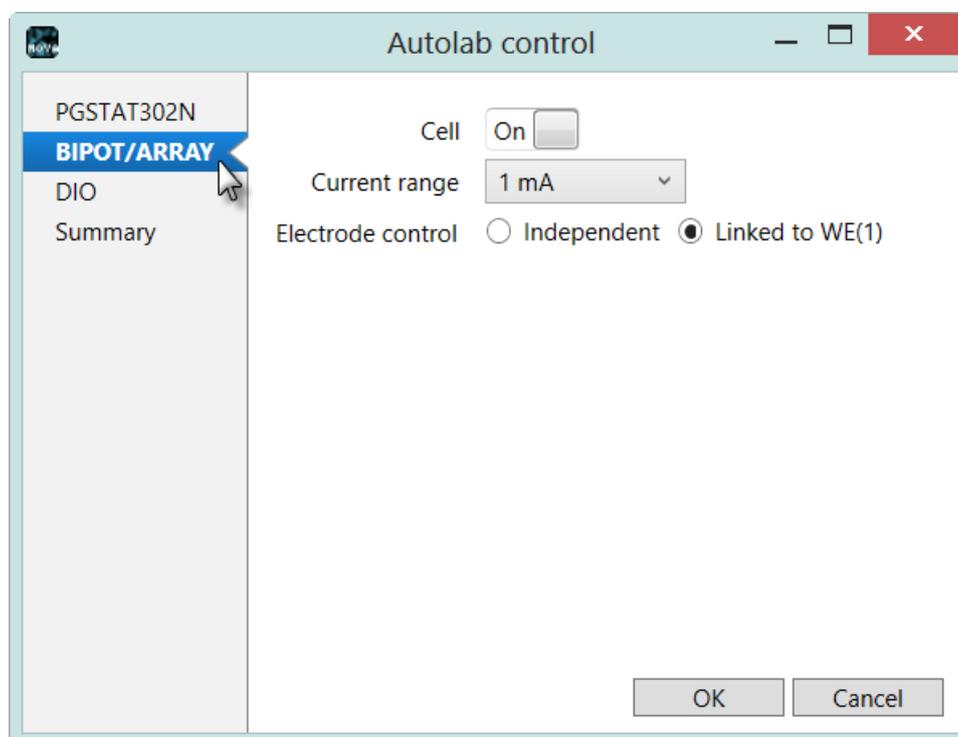


Figure 5 – The BIPOT or ARRAY properties are set in the *Autolab control* command

Three settings are available with the **BIPOT** or **ARRAY** module:

- **Cell:** used to set the cell switch for WE(2) on and off.
- **Current Range:** used to select the initial current range of WE(2).
- **Electrode control (independent, linked to WE(1)):** defines the relationship between the cell switch of WE(2) and WE(1). When this setting is set to *Linked to WE(1)*, the cell switch of WE(2) will automatically be set to the same status of the cell switch of WE(1). Using the *Set cell* command, both cell switches will be toggled at the same time. When this setting is set to *Independent*, the cell switch of WE(2) is decoupled from the cell switch of WE(1). Both switches must be set independently.



Note

The default startup Electrode control setting is: linked to WE(1).



Warning

When the *electrode control* is set to *independent*, a specific order must be respected to avoid current leakage between WE(1) and WE(2). Always set the cell of WE(2) to ON **after** the cell has been set to ON for WE(1). Always set the cell of WE(2) to OFF **before** the cell has been set to OFF for WE(1).



Note

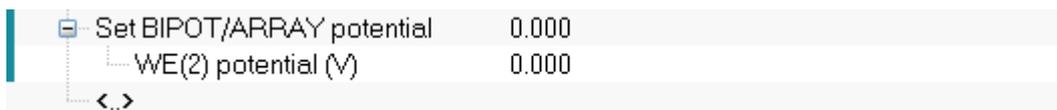
The *Set cell OFF* command switches the main potentiostat and the second working electrode off automatically, regardless of the *Electrode control* setting defined in the Autolab control window and the type of module.

The BIPOT or ARRAY module cannot be switched from BIPOT to Scanning BIPOT in the Autolab control window³. The offset potential with respect to the reference electrode (BIPOT) or the first working electrode (ARRAY) is defined by the DAC164 module of the Autolab. To set this potential, the *Set BIPOT/ARRAY potential* command must be used (see Figure 6). This command is located in the Measurement – General group⁴.

³ The operation mode is defined by the hardware. These modules can be fitted with a hardware switch. Contact your Autolab distributor for more information.

⁴ The Set BIPOT/ARRAY potential command is tagged as Intermediate and requires the BIPOT or ARRAY module in the Hardware setup.

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Set BIPOT/ARRAY potential	0.000
WE(2) potential (V)	0.000
<.>	

Figure 6 – The *Set BIPOT/ARRAY potential* command can be used to set the potential of WE(2) using the BIPOT or ARRAY module



Warning

The DAC164 is also connected to the ECD module and is available on the front panel of the Autolab. When the BIPOT or ARRAY module is used, it is not possible to use the ECD module or to control an external device like the Autolab RDE/RRDE through the DAC164 (using channel #1 on the front panel).

To define the settings for the bipotentiostat module, select the required item from the drop-down lists displayed in the Autolab control window. For example, to set the second working electrode mode, select the BIPOT or Scanning BIPOT using the radio buttons (see Figure 7).

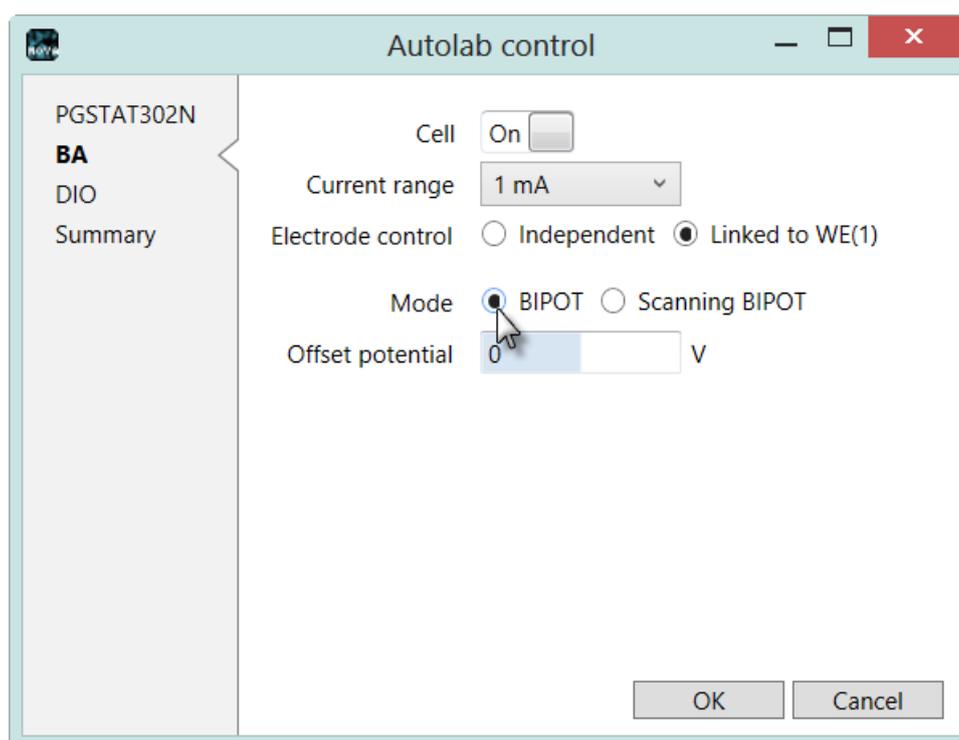


Figure 7 – Setting the mode for the second working electrode

To set the potential of the second working electrode, relative to the reference electrode or the first working electrode, change the Set Potential value using the corresponding drop-down list in the Autolab control window (see Figure 8).

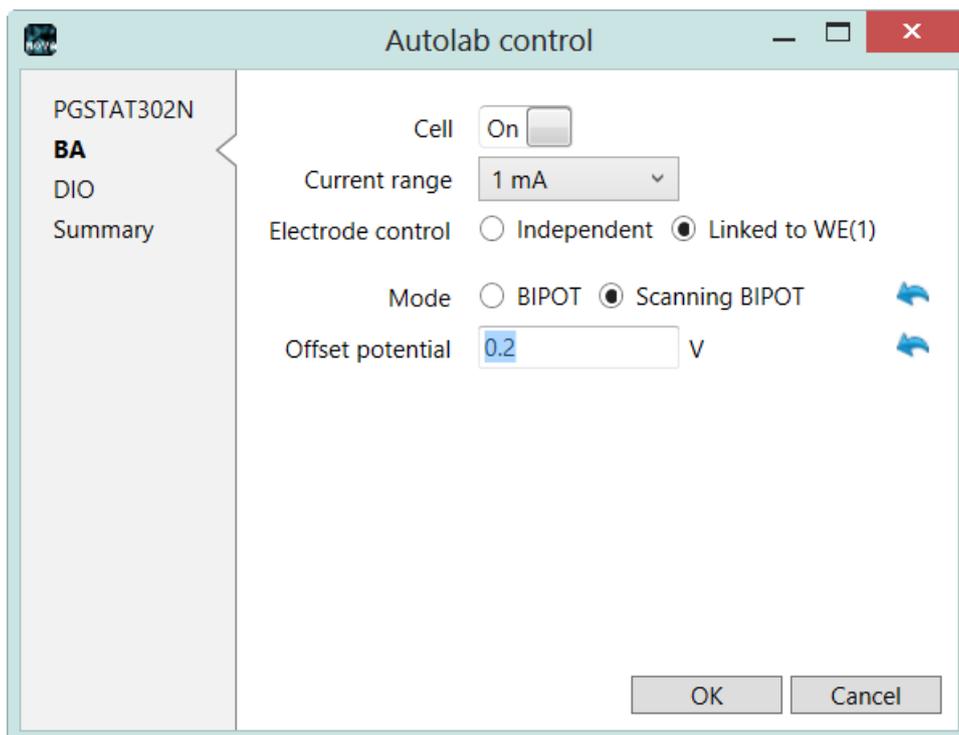


Figure 8 – Setting the potential of the second working electrode to 0.2 V

It is also possible to set the potential of the second working electrode using the slider provided for this property in the Autolab control Window (see Figure 9).

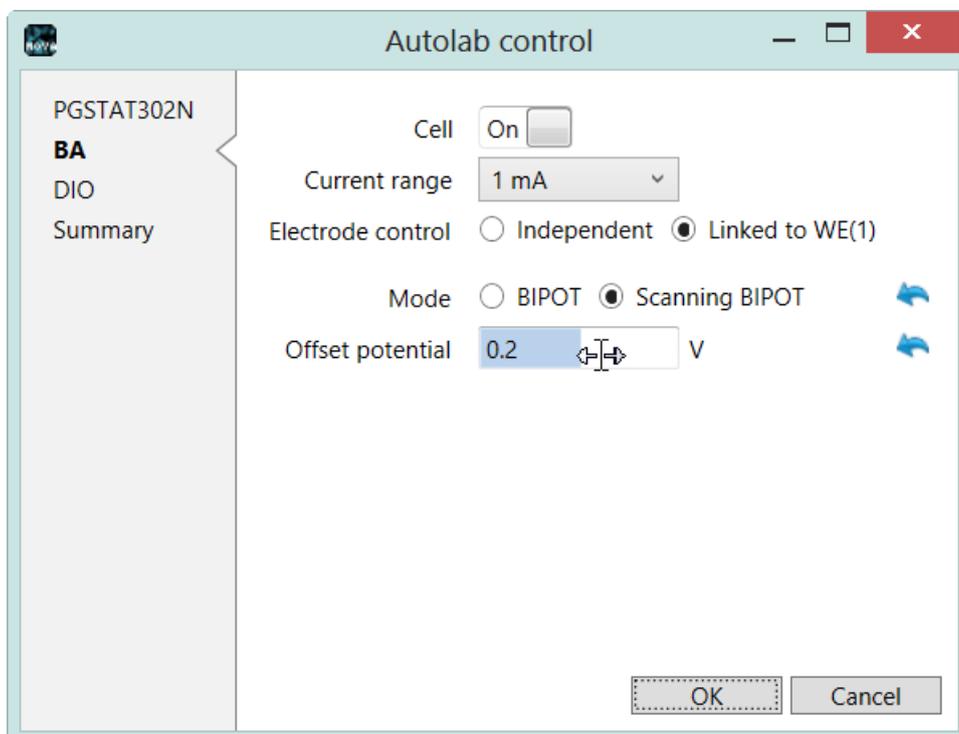


Figure 9 – It is also possible to set the potential using the slider provided in the Autolab control window

5 – A measurement using the Bipotentiostat module on the dummy cell

A Bipotentiostat tutorial folder is located in the **Program Files\Metrohm Autolab\Nova 1.11\Shared Databases\Tutorials** folder (see Figure 10). Using the database manager, set this folder as the Standard database.

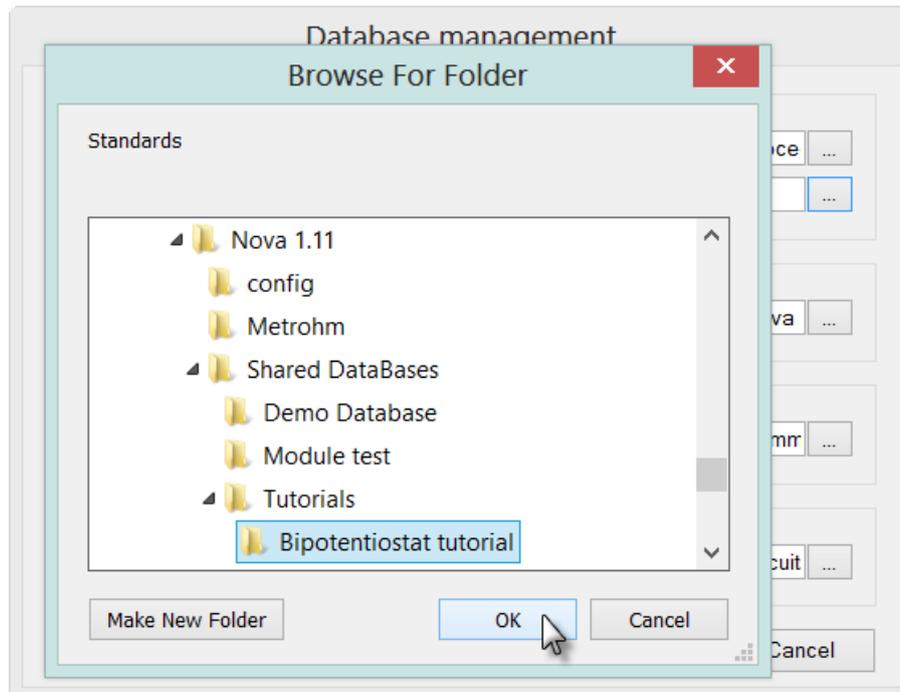


Figure 10 – Loading the Bipotentiostat tutorial database

Four procedures are included in this tutorial. All the procedures are intended to be used with the Autolab dummy cell. Two procedures are designed for the BA module, and the other two are for the BIPOT/ARRAY module (see Figure 11).

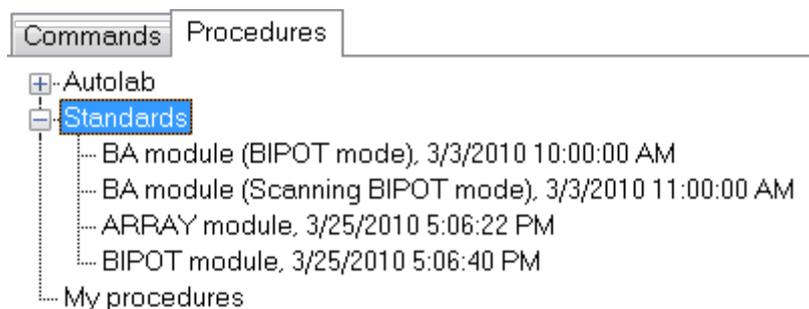


Figure 11 – The four bipotentiostat tutorial procedures

This section will provide some description regarding the two BA procedures⁵. Both procedures are designed to perform a standard staircase CV measurement with 1 V offset on the second working electrode. In BIPOT mode, the potential of the second electrode will be constant. In Scanning BIPOT mode, the potential of the second working electrode will change during the scan.

5.1 – Tutorial procedure #1 – BA module (BIPOT mode)

Load procedure *BA module (BIPOT mode)* procedure in the procedure editor. This procedure contains a single timed procedure. The *Autolab control* command is used to set the properties of the BA module at the beginning of the procedure (see Figure 12).

Commands	Parameters	Links
BA module (BIPOT mode)		
Remarks	BA tutorial: this procedure illustrates the use of the BA...	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(2).Current, WE(1).Current	...
Options	2 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
WE(1).Mode	Potentiostatic	
WE(1).Bandwidth	High stability	
WE(1).Current range	1 mA	
WE(2).BA Mode	Bipot	
WE(2).Potential	1	
WE(2).Electrode control	Linked to WE(1)	
Set potential	0.000	}
Set cell	On	
Wait time (s)	5	
Optimize current range	5	
CV staircase	[0.000, 1.000, -1.000, 0.000, 2, 0.1000000]	
Set cell	Off	...
<.>		

Figure 12 – The BA module (BIPOT mode) tutorial procedure

Connect the cables from the PGSTAT to the Autolab dummy cell as shown in Figure 13 and press the start button to begin the measurement.

⁵ The results are the same in the case of the BIPOT module.

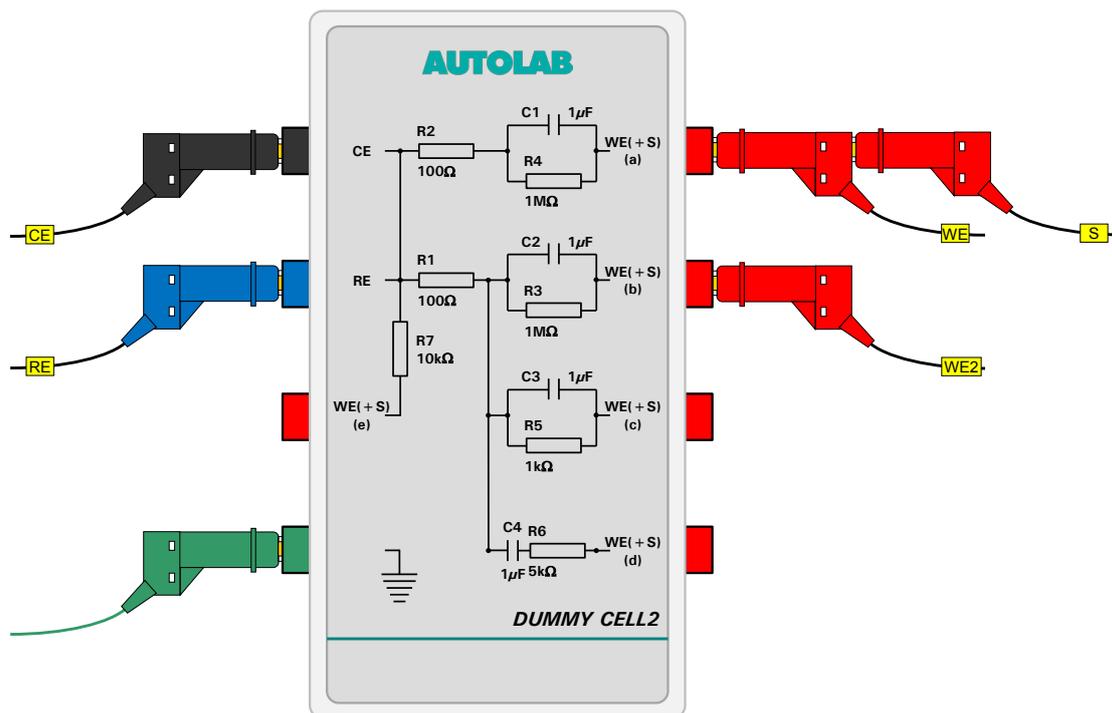


Figure 13 – The connections used for the BA module (BIPOT mode) procedure

Shortly after pressing the Start button, a message will be displayed (see Figure 14).

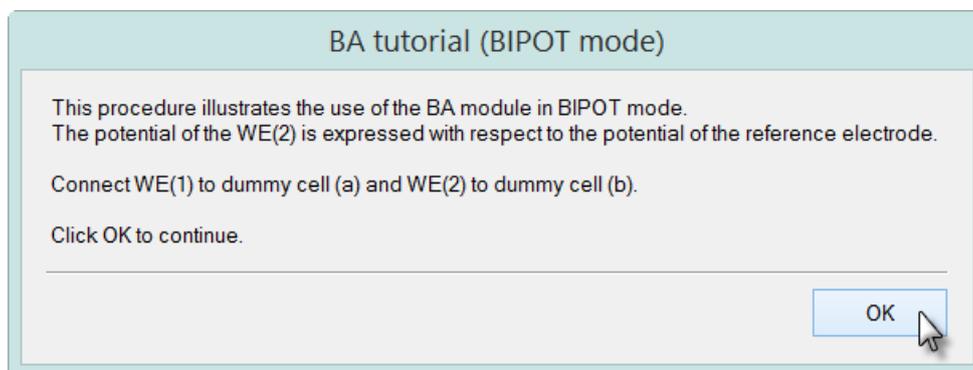


Figure 14 – The message displayed using the BA in BIPOT mode

Click the OK button to continue with the measurement. During the measurement, the WE(1).Current is plotted on the left Y-axis, while the WE(2).Current is plotted on the right Y-axis. Both signals are plotted versus the potential applied on WE(1) (see Figure 15).

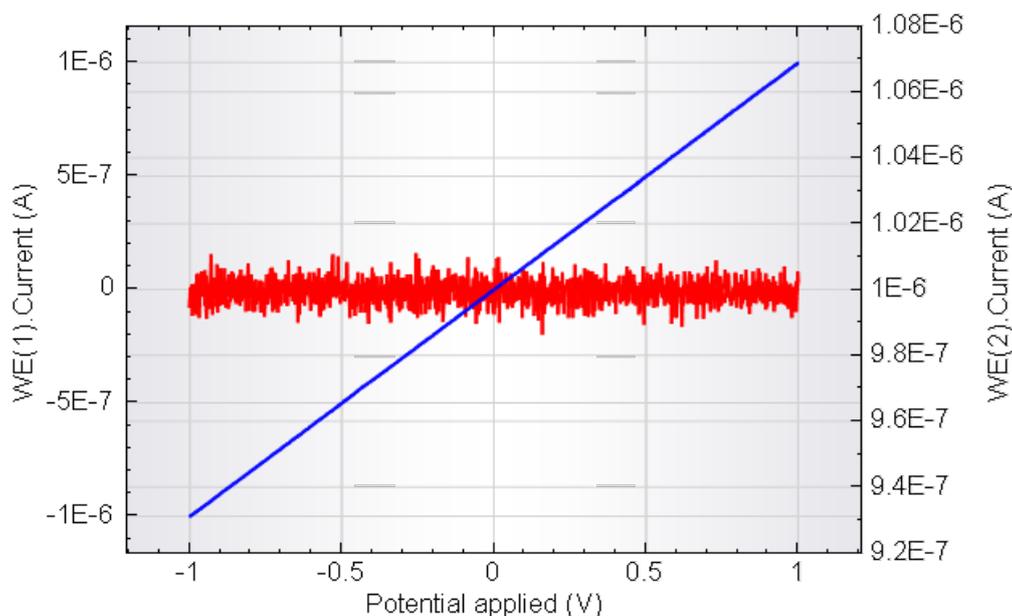


Figure 15 – BA tutorial (BIPOT mode) measured data – WE(1).Current plotted in blue (left axis) and WE(2).Current plotted in red (right axis)

As expected, the current of WE(1) changes from $-1 \mu\text{A}$ to $1 \mu\text{A}$ while the potential is scanned between -1 V and 1 V . The current of WE(2) remains at a constant $1 \mu\text{A}$ value during the measurement, since its potential is at 1 V relative to the reference electrode.

5.2 – Tutorial procedure #2 – BA module (Scanning BIPOT mode)

Select the *BA tutorial (Scanning BIPOT mode)* procedure from the Standards group. This procedure is identical to the previous one, with the exception that the BA mode defined in the Autolab control command is now set to Scanning BIPOT mode (see Figure 16).

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Commands	Parameters	Links
BA module (Scanning BIPOT mode)		
Remarks	BA tutorial: this procedure illustrates the use of the BA...	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(2).Current, WE(1).Current	...
Options	2 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
WE(1).Mode	Potentiostatic	
WE(1).Bandwidth	High stability	
WE(1).Current range	1 mA	
WE(2).BA Mode	Scanning bipot	
WE(2).Potential	1	
WE(2).Electrode control	Linked to WE(1)	
Set potential	0.000	
Set cell	On	...
Wait time (s)	5	
Optimize current range	5	
CV staircase	[0.000, 1.000, -1.000, 0.000, 2, 0.1000000]	
Set cell	Off	...

Figure 16 – The BA module (Scanning BIPOT mode) tutorial procedure

Connect WE(1) to the dummy cell (a) and WE(2) to the dummy cell (b), as shown in Figure 13 and press the start button to begin the measurement.

After pressing the Start button, a message will be displayed (see Figure 17).

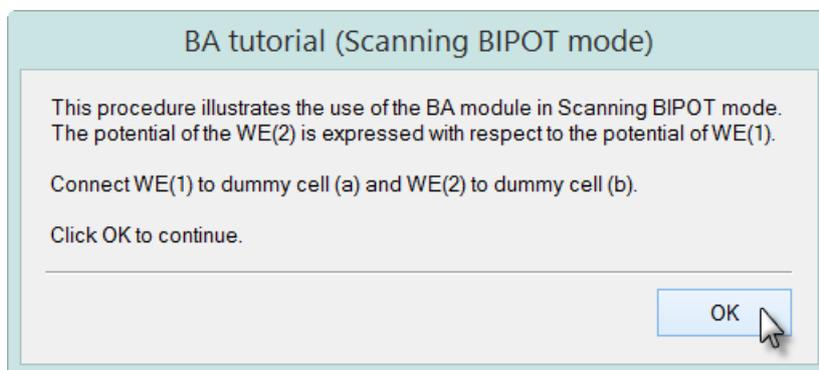


Figure 17 – The message displayed using the BA in Scanning BIPOT mode

Click the OK button to continue with the measurement. During the measurement, the WE(1).Current is plotted on the left Y-axis, while the WE(2).Current is plotted on the right Y-axis. Both signals are plotted versus the potential applied on WE(1) (see Figure 18).

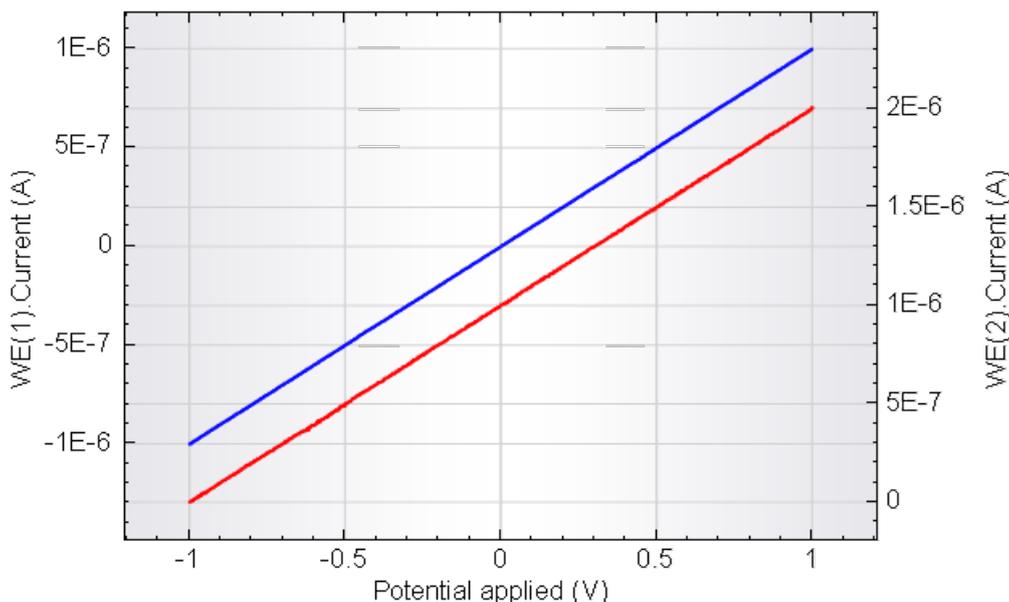


Figure 18 – BA tutorial (Scanning BIPOT) measured data – WE(1).Current plotted in blue (left axis) and WE(2).Current plotted in red (right axis)

As expected, the current of WE(1) changes from $-1 \mu\text{A}$ to $1 \mu\text{A}$ while the potential is scanned between -1 V and 1 V . The current of WE(2) changes from $0 \mu\text{A}$ to $2 \mu\text{A}$ while its potential is scanning between 0 V and 2 V , because of the 1 V offset relative to the potential of working electrode 1⁶.

5.3 – Tutorial procedure #3 – BIPOT module

Select the *BIPOT tutorial* procedure from the Standards group. This procedure is equivalent to the *BA tutorial (Bipot mode)* procedure for the BIPOT module. The Autolab control command is used at the beginning of the procedure to specify the settings of the BIPOT module (see Figure 19). Since the BIPOT module does not have an on-board DAC, the potential must be set using the dedicated *Set BIPOT/ARRAY potential* command (see Figure 19).



Note

DAC channel #3 is used by default. Since this DAC channel is externally connected to the $\leftarrow 1$ BNC plug on the front panel of the Autolab, this channel cannot be used to control an external device like the Autolab RDE/RRDE.

⁶ The WE(2).Potential value is not available.

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Commands	Parameters	Links
BIPOT module		
Remarks	BIPOT tutorial: this procedure illustrates the use of the...	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(2).Current, WE(1).Current	...
Options	2 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
WE(1).Mode	Potentiostatic	
WE(1).Bandwidth	High stability	
WE(1).Current range	1 mA	
WE(2).Current range	1 mA	
WE(2).Electrode control	Linked to WE(1)	
Set potential	0.000	
Set BIPOT/ARRAY potential	1.000	
Set cell	On	...
Wait time (s)	5	
Optimize current range	5	
CV staircase	[0.000, 1.000, -1.000, 0.000, 2, 0.1000000]	
Set cell	Off	...

Figure 19 – Overview of the BIPOT module tutorial procedure

Connect WE(1) to the dummy cell (a) and WE(2) to the dummy cell (b), as shown in Figure 13 and press the start button to begin the measurement.

After pressing the Start button, a message will be displayed (see Figure 20).

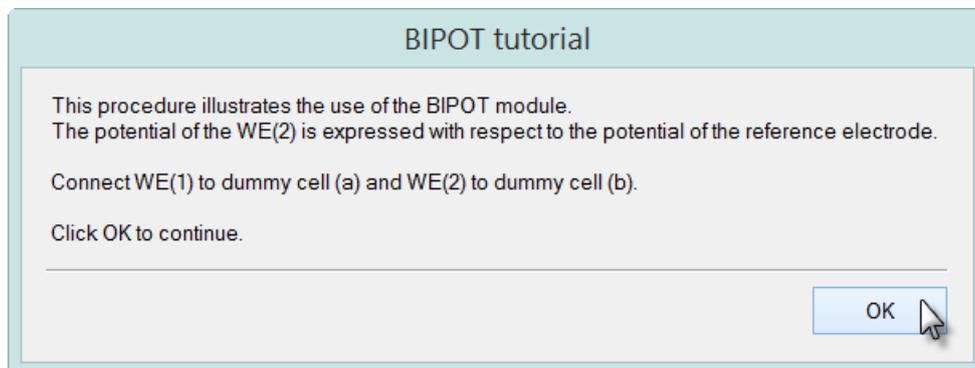


Figure 20 – The message displayed using the BIPOT module tutorial

Click the OK button to continue with the measurement. During the measurement, the WE(1).Current is plotted on the left Y-axis, while the WE(2).Current is plotted on the right Y-axis. Both signals are plotted versus the potential applied on WE(1) (see Figure 21).

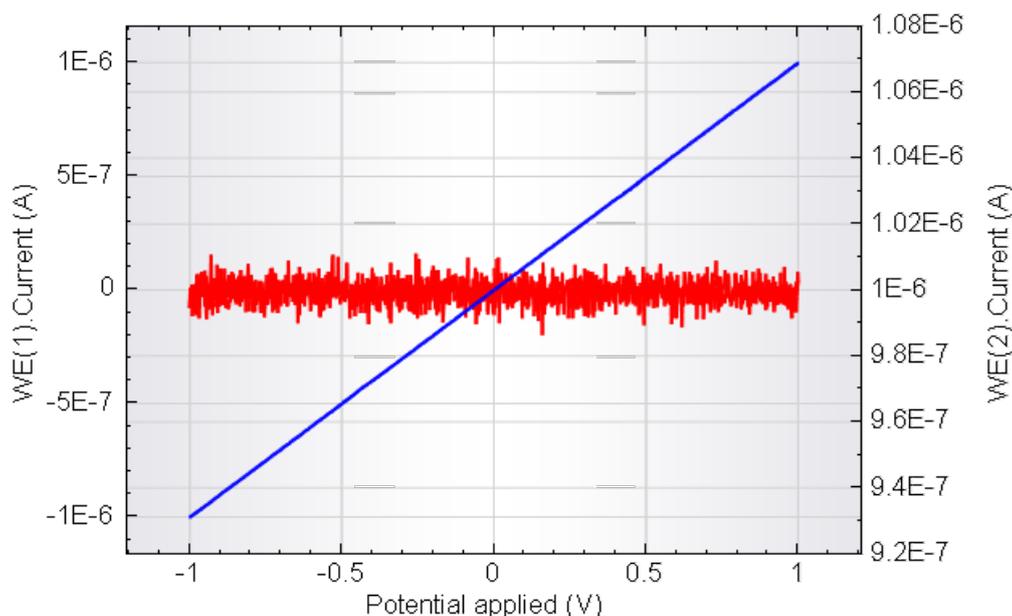


Figure 21 – BIPOT tutorial measured data – WE(1).Current plotted in blue (left axis) and WE(2).Current plotted in red (right axis)

As expected, the current of WE(1) changes from $-1 \mu\text{A}$ to $1 \mu\text{A}$ while the potential is scanned between -1 V and 1 V . The current of WE(2) remains at a constant $1 \mu\text{A}$ value during the measurement, since its potential is at 1 V relative to the reference electrode.

5.4 – Tutorial procedure #4 – ARRAY module

Select the *ARRAY tutorial* procedure from the Standards group. This procedure is equivalent to the *BA tutorial (Scanning Bipot mode)* procedure for the ARRAY module. The Autolab control command is used at the beginning of the procedure to specify the settings of the ARRAY module (see Figure 22). Since the ARRAY module does not have an on-board DAC, the potential must be set using the dedicated *Set BIPOT/ARRAY potential* command (see Figure 22).



Note

DAC channel #3 is used by default. Since this DAC channel is externally connected to the $\leftarrow 1$ BNC plug on the front panel of the Autolab, this channel cannot be used to control an external device like the Autolab RDE/RRDE.

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Commands	Parameters	Links
ARRAY module		
Remarks	ARRAY tutorial: this procedure illustrates the use of th...	...
End status Autolab		...
Signal sampler	Time, WE(2).Current, WE(1).Current	...
Options	2 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
WE(1).Mode	Potentiostatic	
WE(1).Bandwidth	High stability	
WE(1).Current range	1 mA	
WE(2).Current range	1 mA	
WE(2).Electrode control	Linked to WE(1)	
Set potential	0.000	
Set BIPOT/ARRAY potential	1.000	
Set cell	On	...
Wait time (s)	5	
Optimize current range	5	
CV staircase	[0.000, 1.000, -1.000, 0.000, 2, 0.1000000]	
Set cell	Off	...

Figure 22 – Overview of the ARRAY module tutorial procedure

Connect WE(1) to the dummy cell (a) and WE(2) to the dummy cell (b), as shown in Figure 13 and press the start button to begin the measurement.

After pressing the Start button, a message will be displayed (see Figure 23).

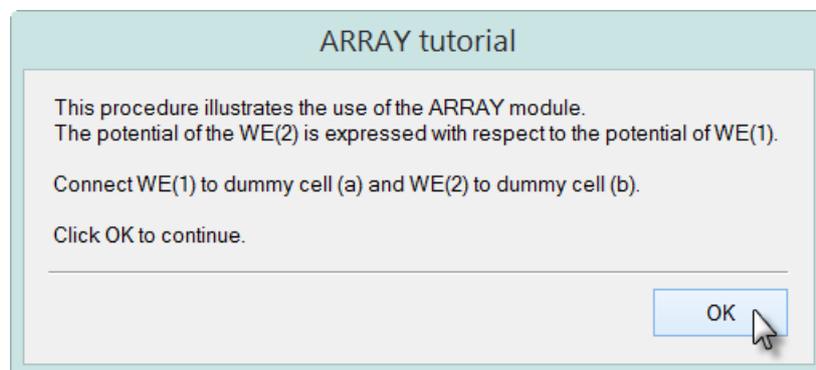


Figure 23 – The message displayed using the ARRAY module tutorial

Click the OK button to continue with the measurement. During the measurement, the WE(1).Current is plotted on the left Y-axis, while the WE(2).Current is plotted on the right Y-axis. Both signals are plotted versus the potential applied on WE(1) (see Figure 24).

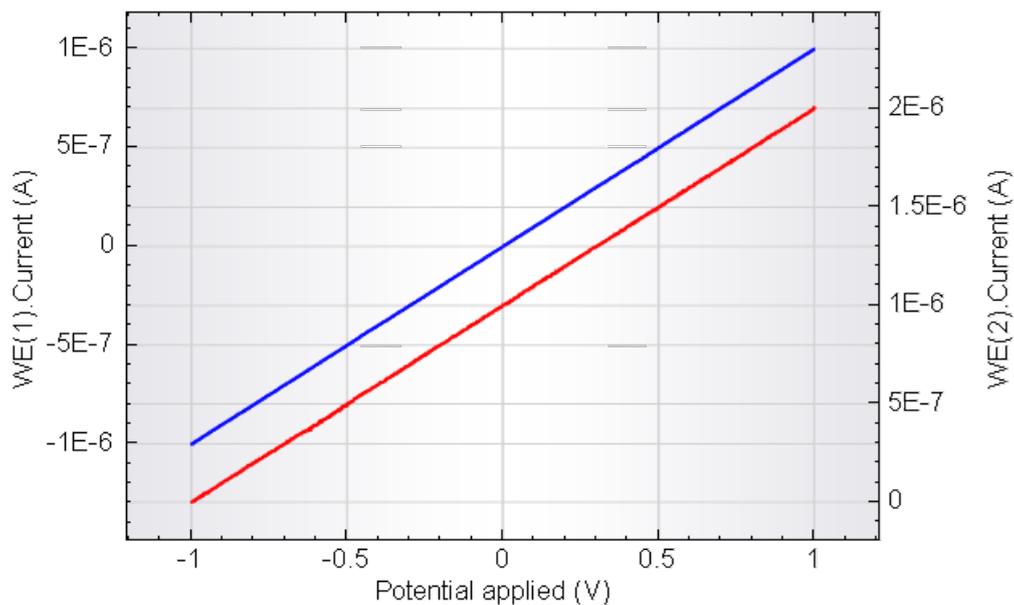


Figure 24 – ARRAY tutorial measured data – WE(1).Current plotted in blue (left axis) and WE(2).Current plotted in red (right axis)

As expected, the current of WE(1) changes from $-1 \mu\text{A}$ to $1 \mu\text{A}$ while the potential is scanned between -1 V and 1 V . The current of WE(2) changes from $0 \mu\text{A}$ to $2 \mu\text{A}$ while its potential is scanning between 0 V and 2 V , because of the 1 V offset relative to the potential of working electrode 1⁷.

⁷ The WE(2).Potential value is not available.

Hardware specifications

The BA, BIPOT and ARRAY modules provide the connections for the second working electrode. The BIPOT and ARRAY modules are modules for which the mode is hardware defined: the BIPOT can be used to control the potential of the second working electrode with respect the reference electrode (so-called **bipotentiostat** mode), while the ARRAY module can be used to control the potential of the second working electrode with respect to the first working electrode (so-called **scanning bipotentiostat** mode). The BA module is a switchable module which can be used in both modes, through a software switch.

Table 1 provides an overview of some of the module specifications.

Module	BA	BIPOT/ARRAY
Operation mode	Software	Hardware, fixed
Control DAC	On-board	DAC164
Maximum current	50 mA	35 mA
Current ranges	10 nA to 10 mA	100 nA to 10 mA
Current accuracy	± 0.2 % of CR	± 0.2 % of CR
Potential range	± 10 V	± 5 V
Potential accuracy	± 2 mV	± 2 mV

Table 1 – Overview of the specifications of the Bipotentiostat modules



Warning

The **BIPOT** and **ARRAY** modules are using the DAC164 to control the potential of the second working electrode. The same DAC164 circuit is used to control the compensation circuit of the **ECD** module. For this reason, the BIPOT or ARRAY module cannot be used at the same time as the ECD module. The Autolab RDE is controlled remotely through the DAC164. The restriction therefore applies to the use of the Autolab RDE/RRDE in combination with the BIPOT, ARRAY or ECD module. These restrictions do not apply to the **BA** module.



Note

The WE(2).Potential signal is not available in the signal sampler when the Bipotentiostat module is used.