1 – The FI20 module

The FI20 is an optional module for the Autolab PGSTAT[1]. This module is a combined filter and analog integrator module. The non modular Autolab instruments cannot accommodate this optional module but they are fitted, by default, with an analog integrator with identical specifications[2].

1.1 – The FI20 – Filter

With the FI20 – Filter[3], it is possible to filter the current measured during electrochemical measurements. The filter can be used to remove noise on the measurements in cases where it is impossible to remove the noise by the use of proper shielding of the cell and electrodes or by using a Faraday cage.

The module uses a third order Sallen-Key filter, with three different filter time constants (0.1 s, 1 s and 5 s)[4]. The filter cannot be used in combination with the FRA2 or FRA32M module or in combination with the ADC750 or ADC10M module.

> **i** Note
>
> The filter is used to filter the current of the working electrode 1 (WE(1).Current).

1.2 – The FI20 – Integrator and on-board integrator

The FI20 – Integrator and on-board integrator[5] provides the means to integrate the measured current. The integrator can be used to perform chronocoulometric experiments and the so-called cyclic or linear sweep voltammetry current integration. The module consists of an analog integrator fitted with four different integration time constants (0.01 s, 0.1 s, 1 s, 10 s)[6].

> **i** Note
>
> The integrator is used to integrate the current of the working electrode 1 (WE(1).Current).

---

[1] This module is not available for the PGSTAT101, PGSTAT204 and the M101 or M204 module.
[2] This applies to the μAutolab II, μAutolab III, the PGSTAT101 and M101 module.
[3] In the rest of this tutorial, the FI20 – Filter options will be referred to as filter.
[4] The effective filter time constant corresponds to ten times the RC time constant (τ_{RC}), resulting in 99.995 % attenuation.
[5] In the rest of this tutorial, the both integrators (FI20 and on-board) options will referred to as integrator.
[6] More information is provided in the Integrator section of this tutorial.

> ### Scope of the tutorial
>
> The aim of this tutorial is to explain how to use the filter to remove noise from the current measured by the Autolab. It also provides information on the use of the integrator in chronocoulemetry and current integration CV and LSV experiments. Some examples using the Autolab dummy cell are provided.

2 – Hardware setup

In order to use the FI20 module, the hardware setup in NOVA must be configured accordingly. For instruments that can be fitted with the FI20 module, two FI20 modules (Filter and Integrator) are provided in the Additional module(s) section (see Figure 1).
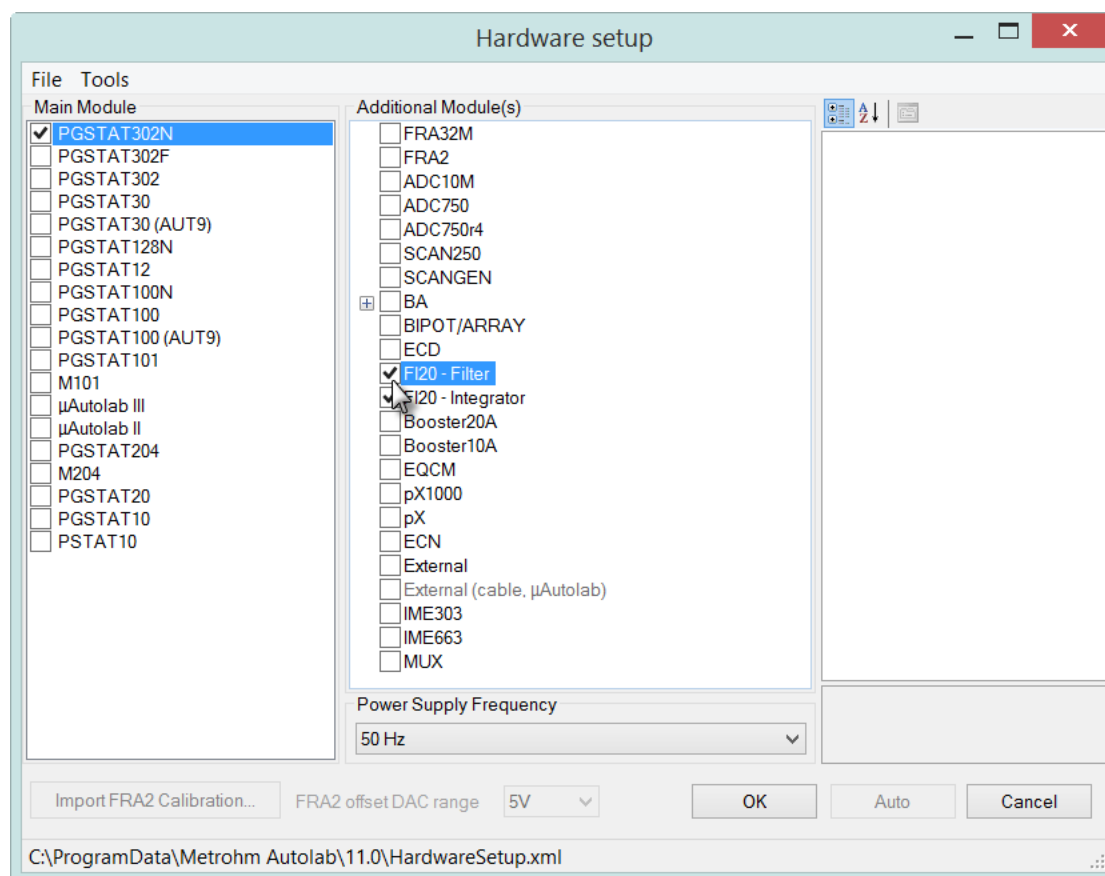

Figure 1 – Selecting the FI20 module(s)

When an instrument with on-board integrator is used, the FI20 – Integrator option is automatically selected in the hardware setup (see Figure 2).
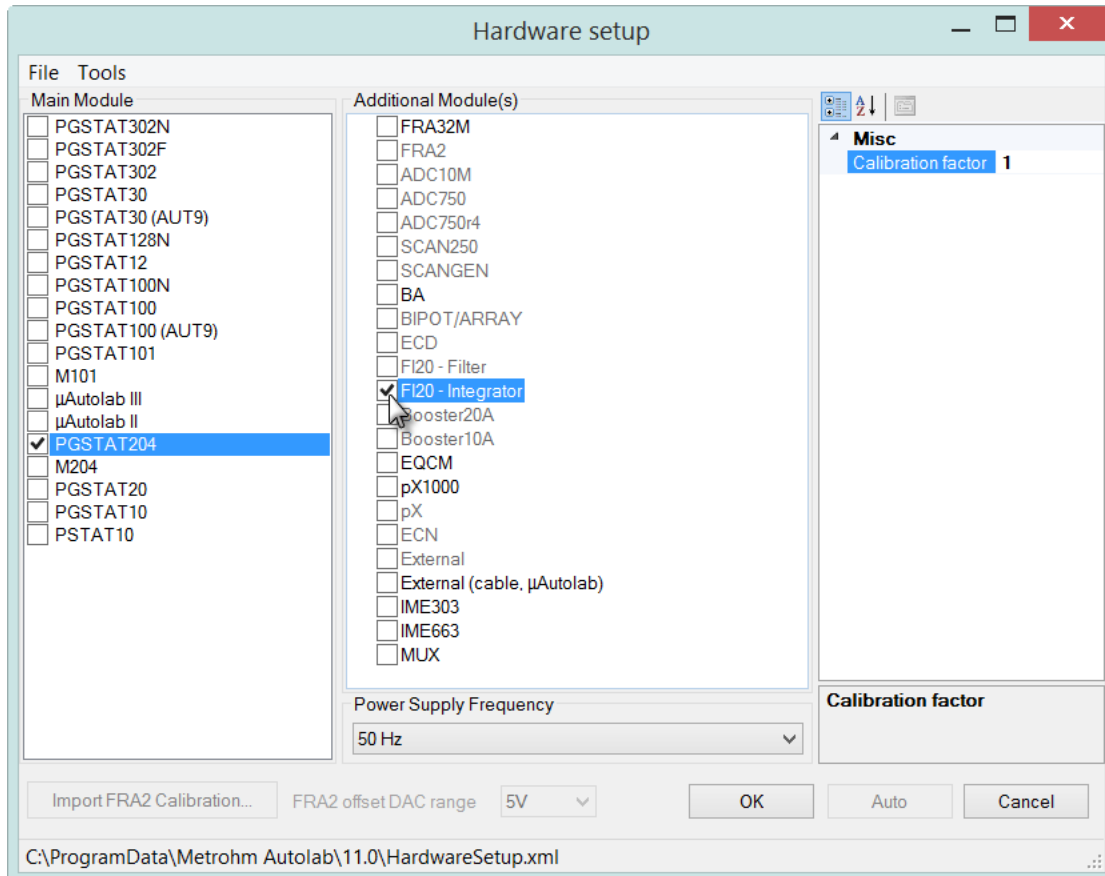
Figure 2 – The FI20 – Integrator module is automatically selected when using an instrument with an on-board intergrator

The use of the filter is explained in the first part of this tutorial. The second part of this tutorial provides more information on the use of the integrator.

3 – Using the filter

To use the filter in a procedure, the module must be switched on. Switching the filter on or off can be done during a measurement using the *Autolab control* command. The on/off control of the module is available in the Autolab control window, on the FI20 section (see Figure 3).
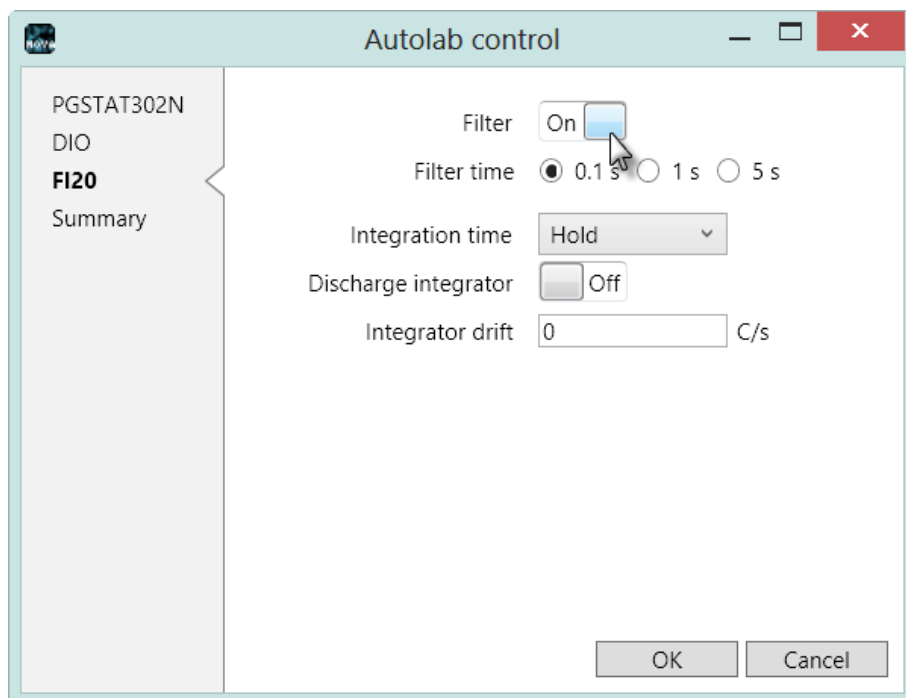
Figure 3 – The Autolab control window can be used to switch the FI20 module on or off

The Autolab control window is also used to set the filter time constant of the module. These settings can be found on the FI20 section (see Figure 4).
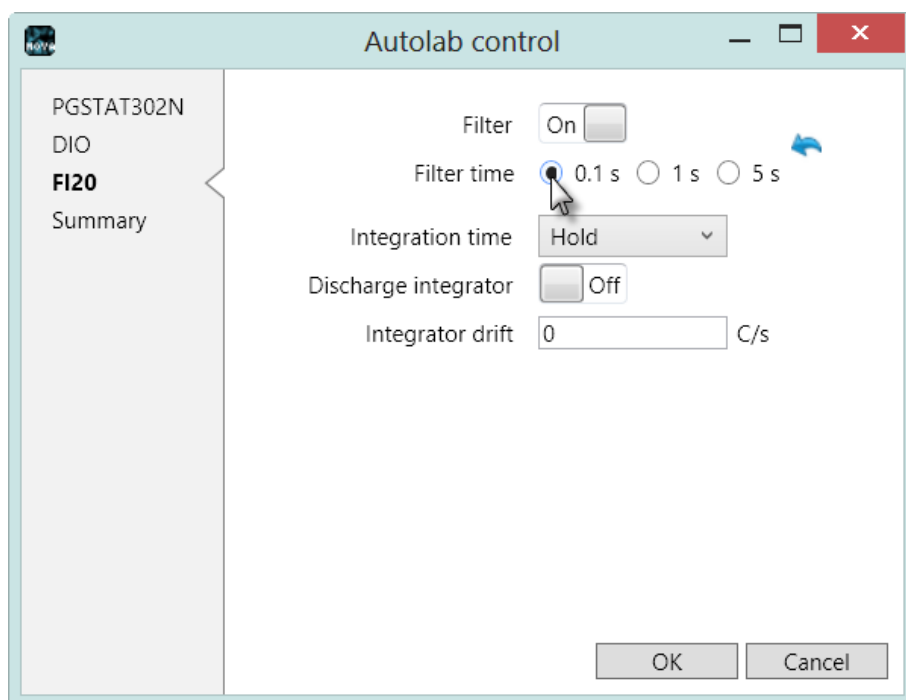

Figure 4 – The filter time constants can be found on the FI20 section

The following time constants are available: 0.1 s, 1 s, 5 s. The effect of a filter constant of x s is that x seconds after a potential perturbation has been applied, the

current response can be measured correctly. A high time constant results in heavy filtering.

3.1 – Automatic current ranging restrictions

When the FI20 module is used during a procedure, the Automatic current ranging option **cannot** be used. The validation process will display an error message whenever this situation is encountered (see Figure 5).
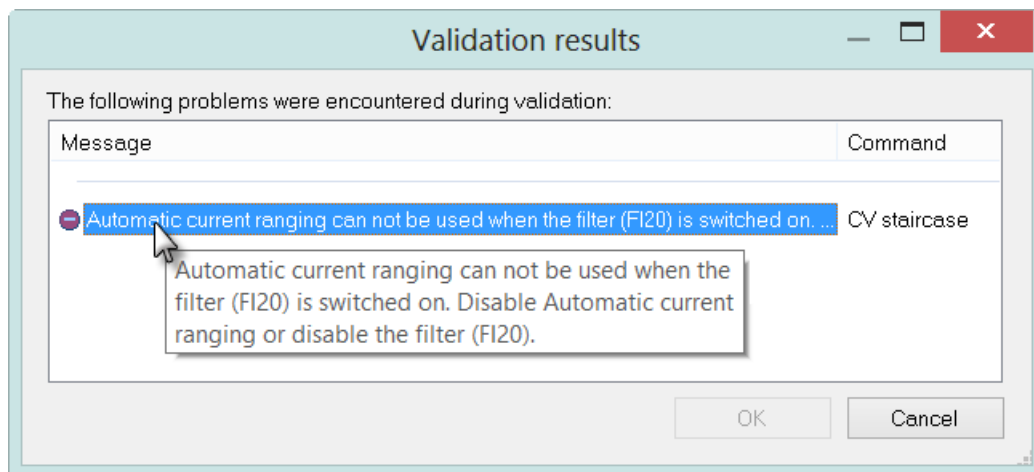

Figure 5 – The FI20 cannot be used in combination with automatic current ranging

3.2 – Bandwidth restrictions

The filter circuit on the FI20 module has a limited bandwidth. Depending on the value of the interval time, a warnings can be displayed in the in the procedure validation window, indicating that the selected filter time constant is too slow to measure properly (see Figure 6).
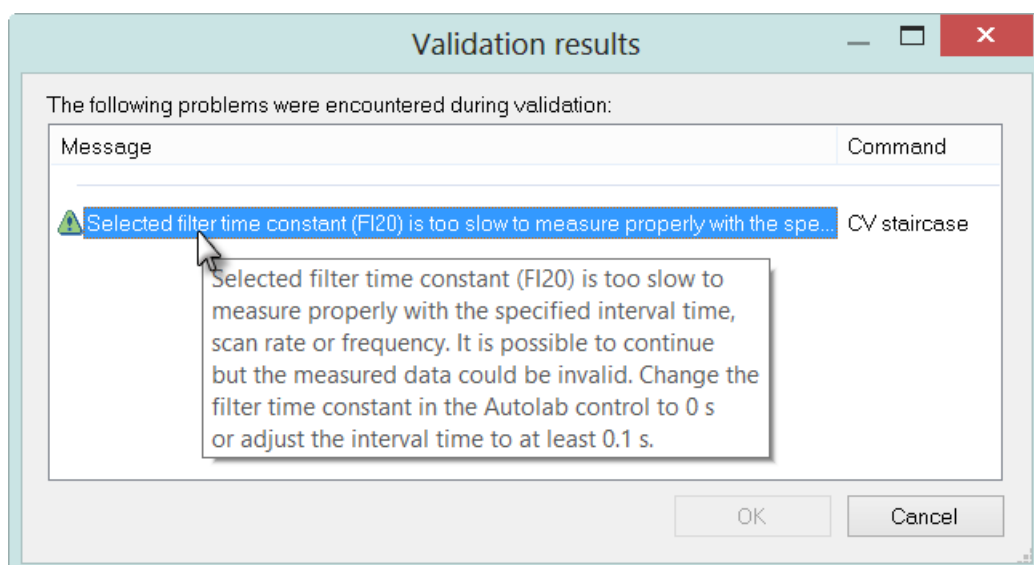

Figure 6 – A warning is displayed in the procedure validation when the selected filter time constant is too slow to measure properly

It is still possible to proceed with the measurement, but there is a possibility that the data will be invalid because of this limitation. It is therefore recommended to adjust the procedure parameters or to select another filter time constant, if possible.

3.3 – Measurements with the FI20 module on the dummy cell

A FI20 tutorial folder is located in the **Program Files\Metrohm Autolab\Nova 1.11\Shared Databases\Tutorials** folder (see Figure 7). Using the database manager, set this folder as the Standard database.
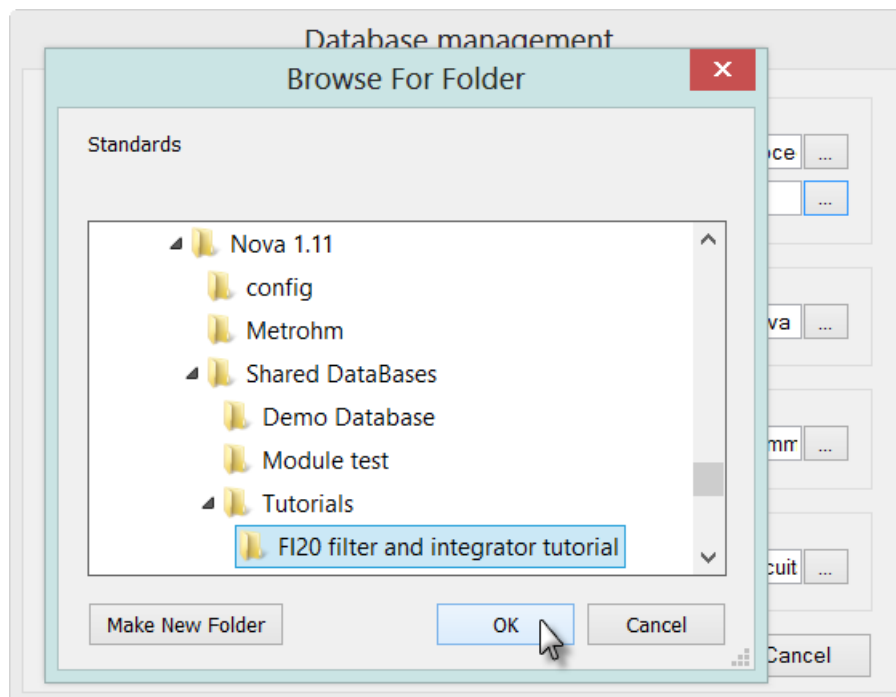

Figure 7 – Loading the FI20 tutorial database

The FI20 tutorial contains four procedures (see Figure 8). The first two procedures illustrate the use of the *Autolab control* command to control the filter during a measurement. The other two procedures are used to illustrate the use of the integrator module (see section 4.6).
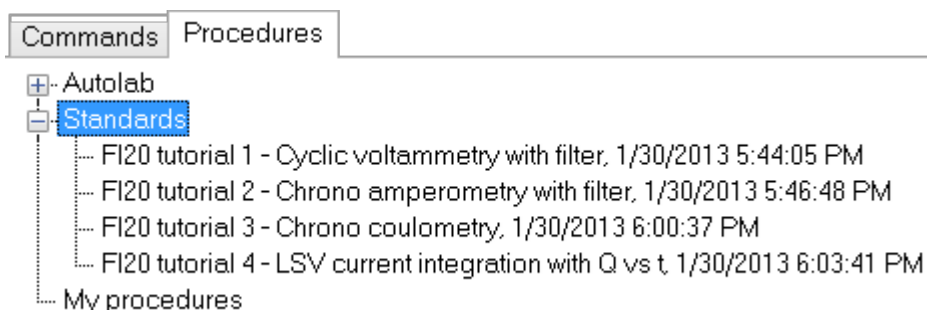

Figure 8 – The four FI20 tutorial procedures

3.3.1 – FI20 tutorial 1 – Cyclic voltammetry with filter

Load the FI20 tutorial 1 procedure. Figure 9 shows an overview of the procedure. This procedure is designed to perform a cyclic voltammetry staircase experiment. The FI20 – Filter is switched on at the beginning of the measurement using the *Autolab control* command. The FI20 is used to filter the WE(1).Current signal using a filter time-constant of 0.1 s (see Figure 9).

Commands	Parameters	Links
FI20 tutorial 1 - Cyclic voltammetry with filter		
Remarks	Example of FI20 use in CV.	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(1).Current	...
Options	0 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
WE(1).Mode	Potentiostatic	
WE(1).Bandwidth	High stability	
WE(1).Current range	1 µA	
Filter(1).Filter time	0.1 s	
WE(1).FI20-filter Mode	On	
Set potential	0.000	
Set cell	On	...
Wait time (s)	5	
CV staircase	[0.000, 1.000, -1.000, 0.000, 2, 0.0500000]	–
Set cell	Off	...
<..>		

Figure 9 – The FI20 tutorial 1 procedure

> **Note**
>
> Because the FI20 – Filter is used in this experiment, automatic current ranging is not possible.

Connect the cables from the PGSTAT to the Autolab dummy cell as shown in Figure 10 and press the start button to begin the measurement.
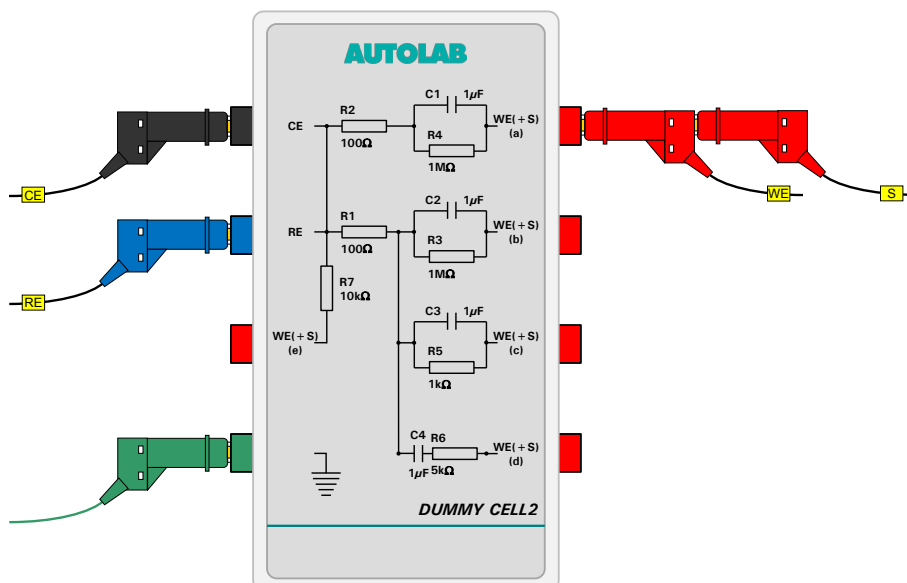
Figure 10 – Overview of the connections to the dummy cell

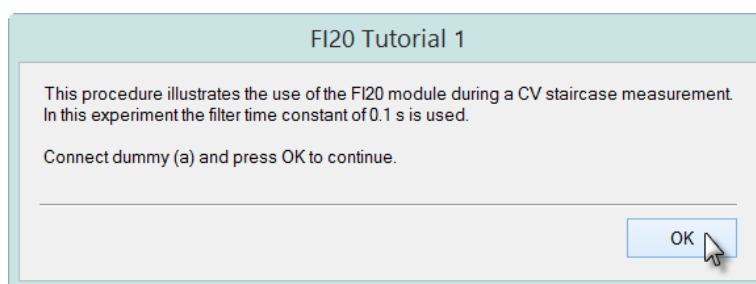A message box will be displayed (see Figure 11).

Figure 11 – A message box is displayed at the beginning of the measurement

Click the OK button to continue. At the beginning of the measurement, the FI20 is switched on and the filter time constant of 0.1 s is selected. A cyclic voltammetry measurement is performed on the dummy cell and the WE(1).Current values are plotted versus the applied potential (see Figure 12).
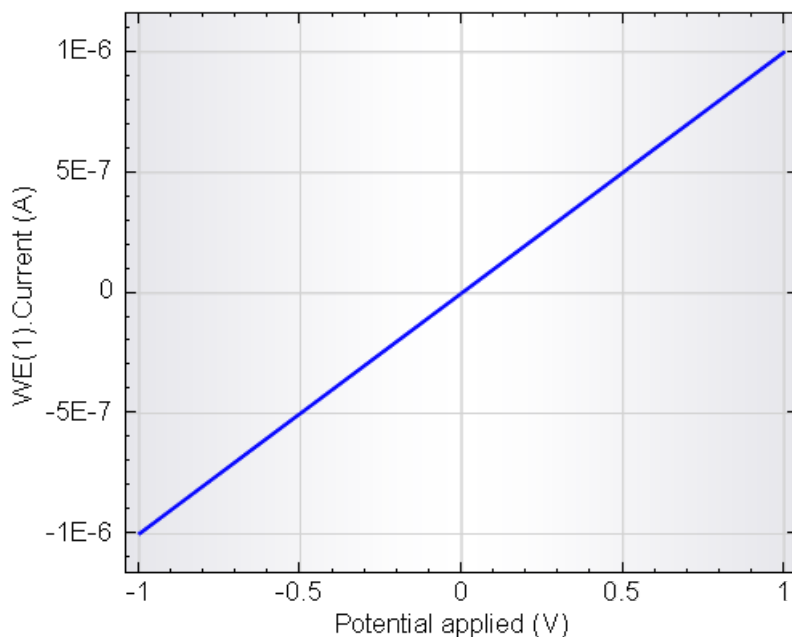
Figure 12 – The data recorded during the FI20 tutorial 1 procedure

Since dummy cell (a) is used in this test, the measured current values change between -1 µA and 1 µA as the potential is scanned between -1 V and 1 V. The current values displayed in Figure 12 are filtered using a 0.1 s time-constant.

	Note
i	At the end of the experiment, the FI20 – Filter is switched off using the End status Autolab settings.

3.3.2 – FI20 tutorial 2 – Chrono amperometry with filter

Load the FI20 tutorial 2 procedure. Figure 13 shows an overview of the procedure. This procedure is designed to perform two consecutive chrono amperometric steps at a constant potential of 0.3 V. The current is recorded using *Record signals (>1 ms)* commands. The FI20 – Filter is switched off during the first step. After the first *Record signals* command, the Filter is switched on and the 0.1 s time constant is selected using the *Autolab control* command.

Commands	Parameters	Links
FI20 tutorial 2 - Chrono amperometry with filter		
Remarks	Example of FI20 use in Chrono amperometry.	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(1).Current	...
Options	0 Options	...
Instrument		
Instrument description		
Message box		
Autolab control		...
Set potential	0.300	
Set cell	On	...
Wait time (s)	5	
Record signals (>1 ms)	[0.2]	–
Autolab control		...
WE(1).FI20-filter Mode	On	
Filter(1).Filter time	0.1 s	
Record signals (>1 ms)	[0.2]	–
Set cell	Off	...
<..>		

Figure 13 – The FI20 tutorial 2 procedure

Note

Because the FI20 – Filter is used in this experiment, automatic current ranging is not possible.

Connect the cables from the PGSTAT to the Autolab dummy cell as shown in Figure 10 and press the start button to begin the measurement.

A message box will be displayed (see Figure 14).

FI20 Tutorial 2

This procedure illustrates the use of the FI20 module during a chrono amperometry measurement. In this experiment the filter time constant of 0.1 s is used.
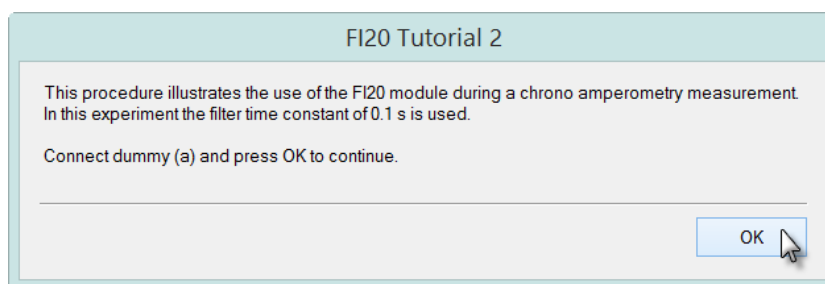
Connect dummy (a) and press OK to continue.

OK

Figure 14 – A message box is displayed at the beginning of the measurement

Click the OK button to continue. At the beginning of the measurement, the FI20 is switched off and the current is measured in the 100 nA current range. In the second part of the experiment, the current is filtered by the FI20 module, using the 0.1 s time-constant. The WE(1).Current values are plotted versus time (see Figure 15).
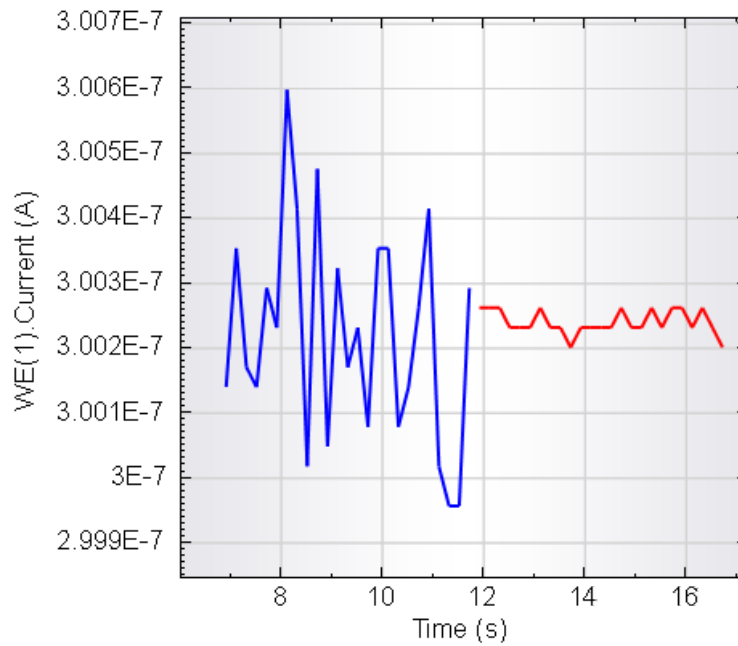
Figure 15 – The data recorded during the FI20 tutorial 2 procedure

Since dummy cell (a) is used in this test, the current values recorded at 0.3 V are ~ 300 nA. Switching the FI20 – Filter on during the measurement significantly increases the signal-to-noise ratio.

> **Note**
>
> At the end of the experiment, the FI20 – Filter is switched off using the End status Autolab settings.

4 – Using the integrator

The integrator can be used to measure the charge from the values of the WE(1).Current. The integration is performed real time and the results can be used for plotting purposes or can be tested with cutoff conditions, if required.

	Note
ℹ️	

The charge is available for all instruments as WE(X).Charge directly in the signal sampler. This signal is obtained by mathematical integration of the current versus time. This signal is available for all the working electrodes. The charge determined by the FI20 module or the on-board integrator is a measured signal, which is available only for the WE(1).

4.1 – Integrator calibration

	Warning
⚠️	

The integrator **must** be calibrated before it can be used.

The calibration of the integrator is performed in the diagnostics[7] application. Make sure that the **Integrator test** is selected (see Figure 16).
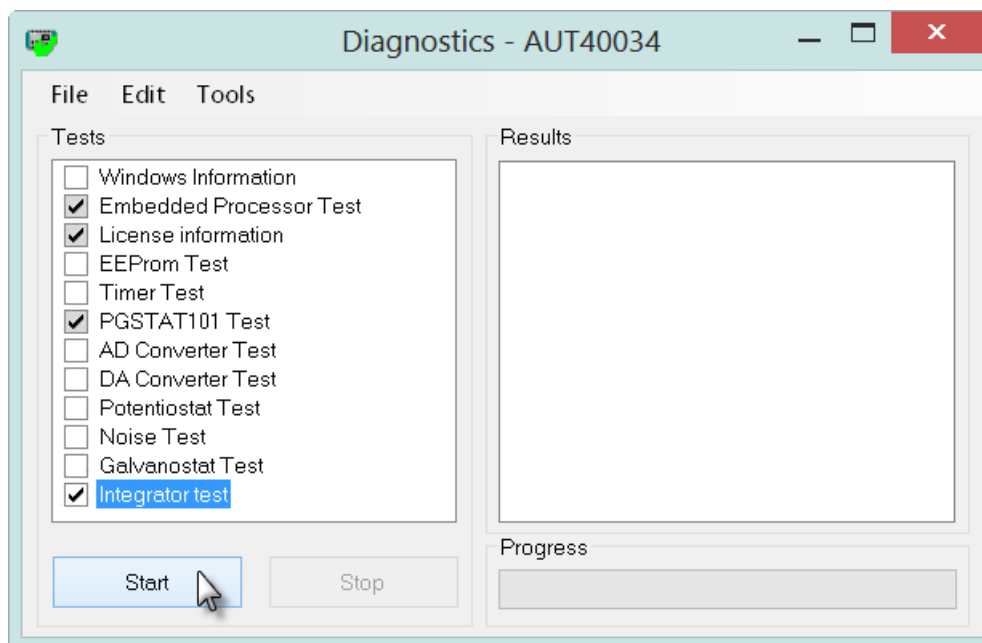

Figure 16 – The integrator test is used to determine the calibration factor of the integrator

[7] Refer to the Getting started manual for more information on the Diagnostics application.

> **Note**
>
> The other optional tests are not mandatory to calibrate the integrator.

The integrator calibration requires dummy cell (a) to be connected for all the instruments, except the PGSTAT101 and the M101 potentiostat/galvanostat module. For the PGSTAT101 and the M101, the internal dummy cell is used.

When the integrator test is finished, a window will be shown, indicating the currently used calibration factor and the measured calibration factor (see Figure 17).
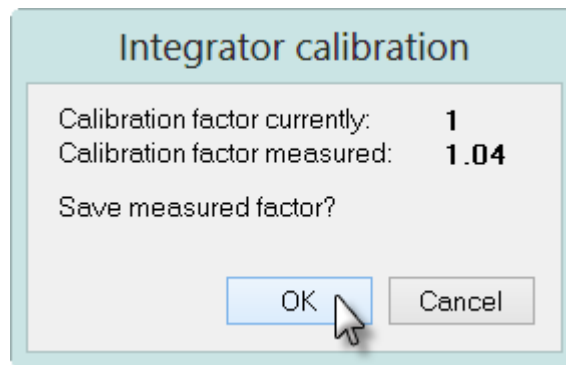
Figure 17 – The stored and measured calibration factors are shown at the end of the test

Click OK to store the new calibration factor or Cancel to keep the value stored in the hardware setup file.

The calibration procedure measures the deviation from the expected charge. A current of 1 µA is passed through the cell for 1 second, resulting in an expected charge of 1 µC. The calibration factor is determined by the ratio of the measured charge and the expected charge.

The measured calibration factors should be 1.0 ± 0.1[8].

---

[8] If the measured value exceeds these limits, contact you Autolab distributor.

4.2 – Signals provided by the integrator

The integrator provides two additional signals in the signal sampler (see Figure 18):

- **Integrator(1).Charge:** this signal corresponds to the measured charge, in coulombs.
- **Integrator(1).Integrated current:** this signal corresponds to the converted equivalent current obtained by deriving the measured charge over the interval time used in the measurement. This signal can be used in order to perform so-called **current integration cyclic and linear sweep voltammetry** measurements. In first approximation, at low scan rates, the results obtained with the current integration method can be compared to the results obtained with a linear scan generator.
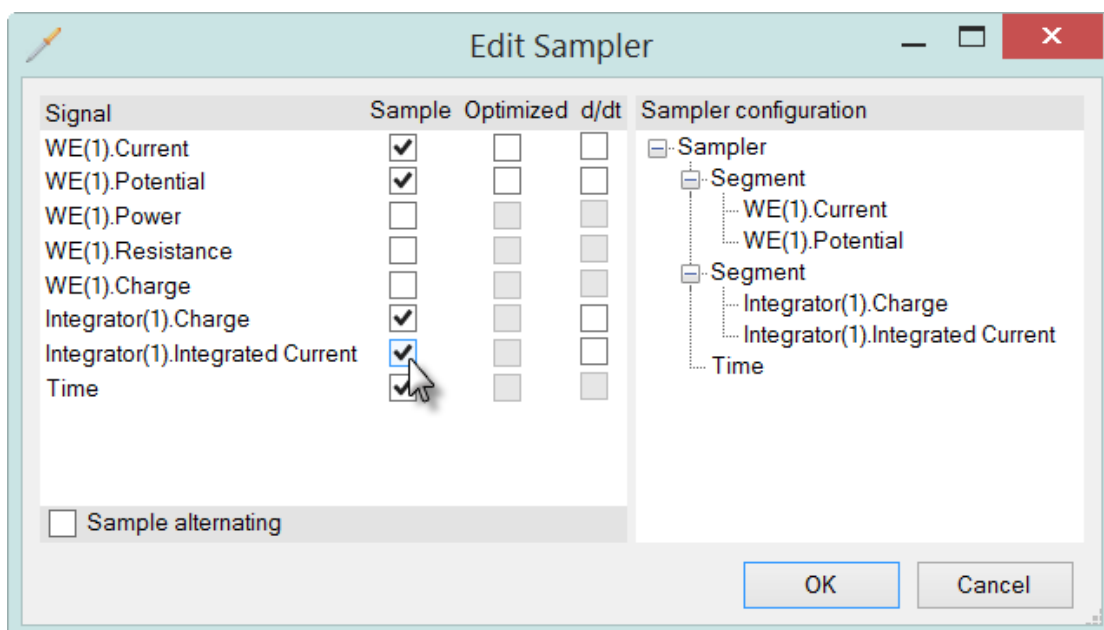

Figure 18 – The integrator module provides two additional signals in the sampler: Charge and Integrated current

In order to obtain the values of the charge and/or the integrated current, the WE(1).Current **must** be sampled. The signals to be measured can be selected using the signal sampler.

> **Note**
>
> The difference between the Integrator(1).Charge and the WE(1).Charge signal is that the latter is obtained from the mathematical integration of the WE(1).Current while the Integrator(1).Charge is determined by analog integration of the current.

4.3 – Integrator settings

For measurements with the integrator module, the settings for this module need to be specified. This can be done with the Autolab control command (see Figure 19).
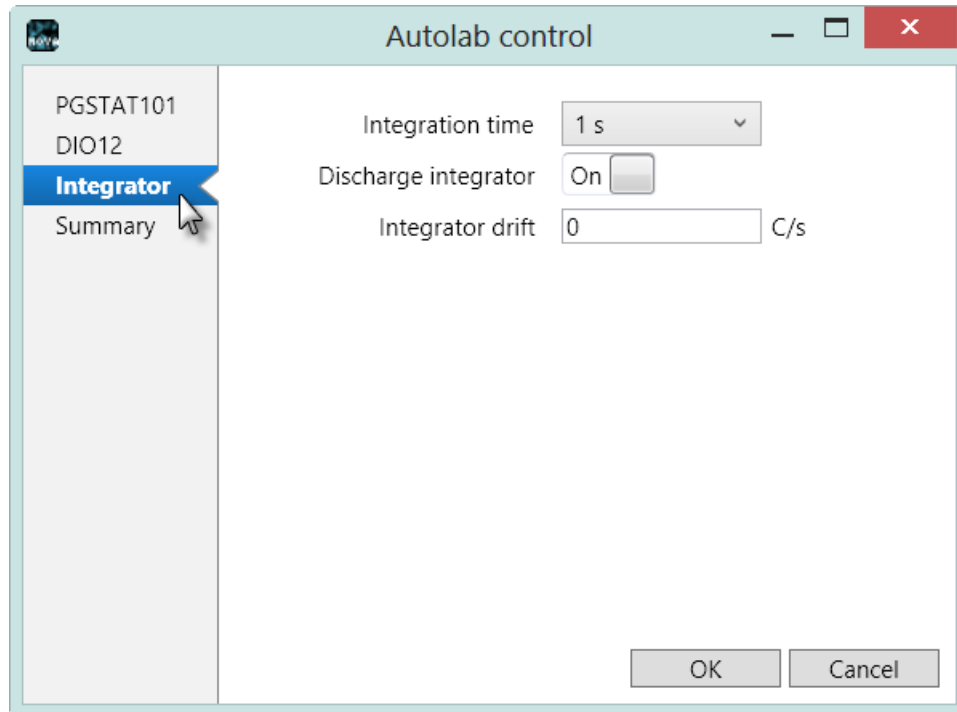

Figure 19 – The Autolab control window can be used to set the integration time

Three settings are available with the **Integrator** module:

- **Integration time:** used to specify the integration time used in the measurement.
- **Discharge integrator:** used to discharge the integrator completely.
- **Set drift:** specifies the amount of drift in the integrator response.

> **Note**
>
> It is highly recommended to set the Discharge integrator property to On at the end of each measurement involving the FI20 module or the on-board integrator, using the *End status Autolab* option. Leaving the integrator charged could introduce extra noise during consequent measurements.

4.3.1 – Integration time

The integration time should be selected carefully, taking the current range into account. If the current range is too low, the integrator will saturate and the measured charge will be lower than the real charge.

The saturation value is reached when the total charge is equal to approximately:

$$Q_{sat} = 10(CR \cdot \tau_{int})$$

Where CR is the selected current range and τ_{int} is the integration time. For example, in the 100 nA current range, in combination with an integration time of 1 s, the saturation charge will approximately equal to 1 µC.

> **Note**
>
> When the measured charge exceeds the maximum saturation capacity of the integrator, it is possible to use the mathematical integration of the current instead, by sampling the WE(1).Charge signal using the signal sampler.

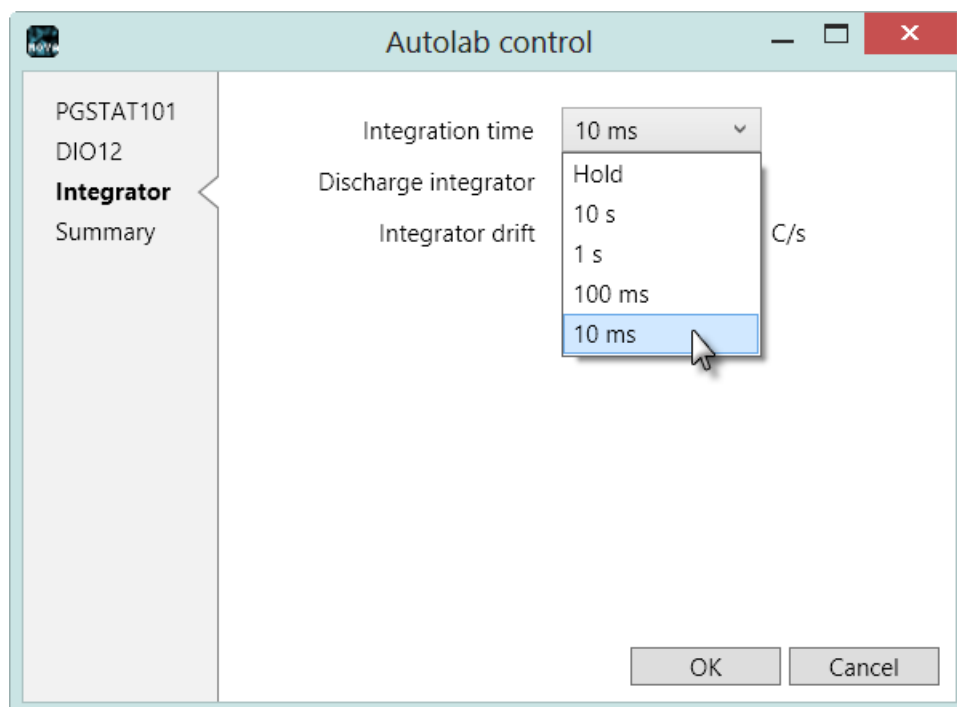The integration time can be fixed through the Autolab control window (see Figure 20).

Figure 20 – Setting the integration time constant for the integrator module

> **i**   **Note**
>
> It is possible to allow the software to adjust the integrator time constant automatically during a measurement (see Section 4.4).

The **Hold** setting, available from the Integration time drop down list is a special mode that can be used to isolate the integrator at any time during a measurement. When this mode is selected, the integrator is disconnected from the working electrode and the accumulated charge is stored. The integrator is not discharged when the Hold mode is active. The integrator can be reconnected to the working electrode at any time by selecting any of the available integration time values from the drop down list.

4.3.2 – Discharge integrator

The module is fitted with a reset switch that can be activated to fully discharge the integrator circuit. This can be done at any time during a measurement, independently of the activity of the working electrode. If the integrator is not discharged during long measurements, there is a saturation risk.

The Current integration staircase voltammetry method automatically discharges the integrator at the end of each potential step.

> **i**   **Note**
>
> It is highly recommended to set the Discharge integrator property to On at the end of each measurement involving the FI20 module or the on-board integrator, using the *End status Autolab* option. Leaving the integrator charged could introduce extra noise during consequent measurements.

4.3.3 – Drift

The drift setting, available in the Autolab control window, can be used to manually define the drift of the integrator. The drift is defined as the charge accumulation due to leakage current, in C/s. Setting the drift allows to compensate for the non-ideality of the current follower of the Autolab.

> **i**   **Note**
>
> The drift depends on the active current range.

The value can be adjusted manually using the Autolab control command or it can be adjusted automatically by using the *Determine integrator drift* command, available in the Measurement – general group of commands (see Figure 21).
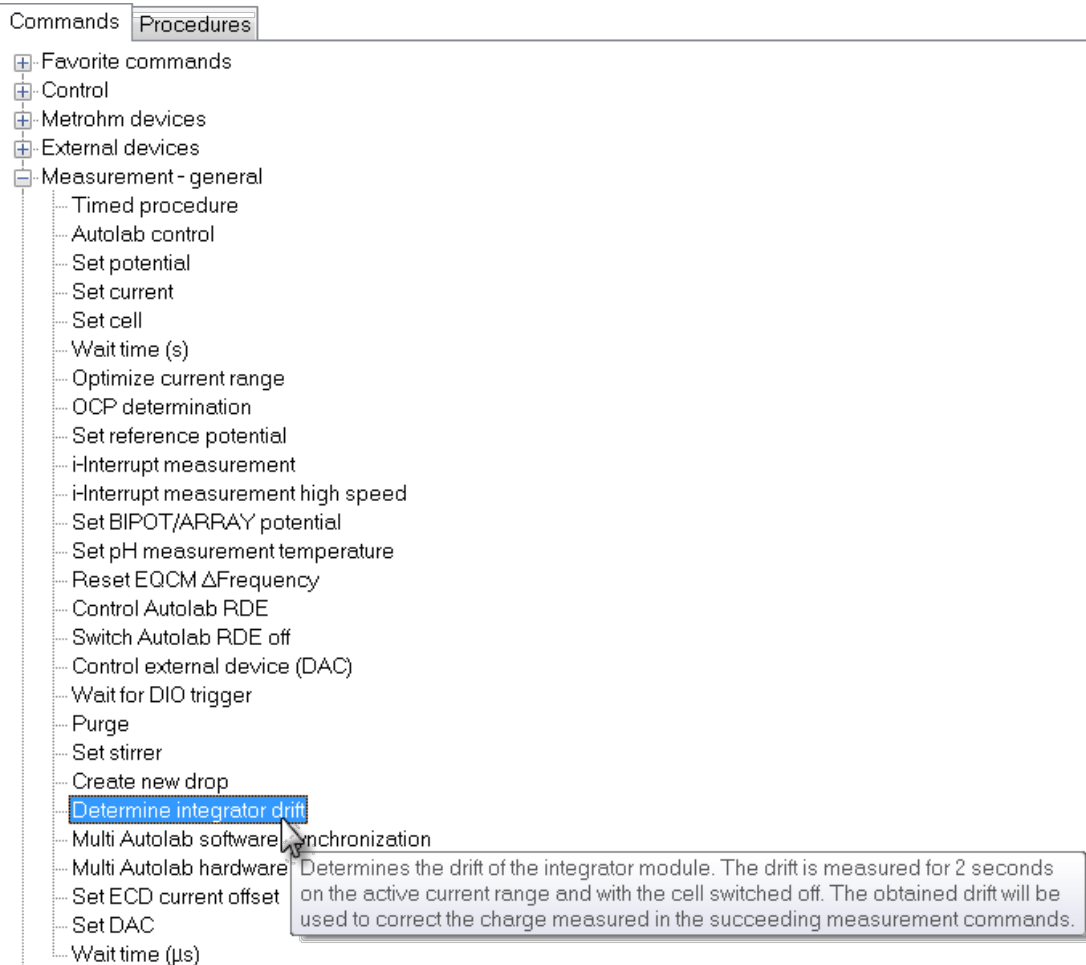
Figure 21 – The Determine integrator drift command can be found in the Measurement – general group

The *Determine integrator drift* command can be added to any procedure. It requires the current range to be specified (see Figure 22). This can be done through the Autolab control included in the command.
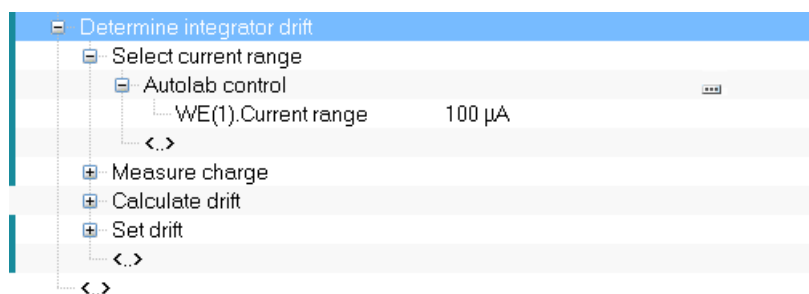

Figure 22 – The Determine integrator drift requires the current range to be specified (through the Autolab control command)

Note

The *Determine integrator drift* command automatically switches off the cell and measures the drift for two seconds. This value is automatically used as integrator drift. Empirically, it has been determined that the drift does not change significantly during the course of a day. For this reason, it is not mandatory to determine this value before each coulometric measurement, although it is advisable.

Warning

The drift should be determined whenever the current range is changed.

The *Determine integrator drift* command is included in the default Chrono coulometry fast procedure.

4.4 – Integrator options

The integration time can be automatically adjusted, if necessary. This option can be activated in the Options menu (see Figure 23).
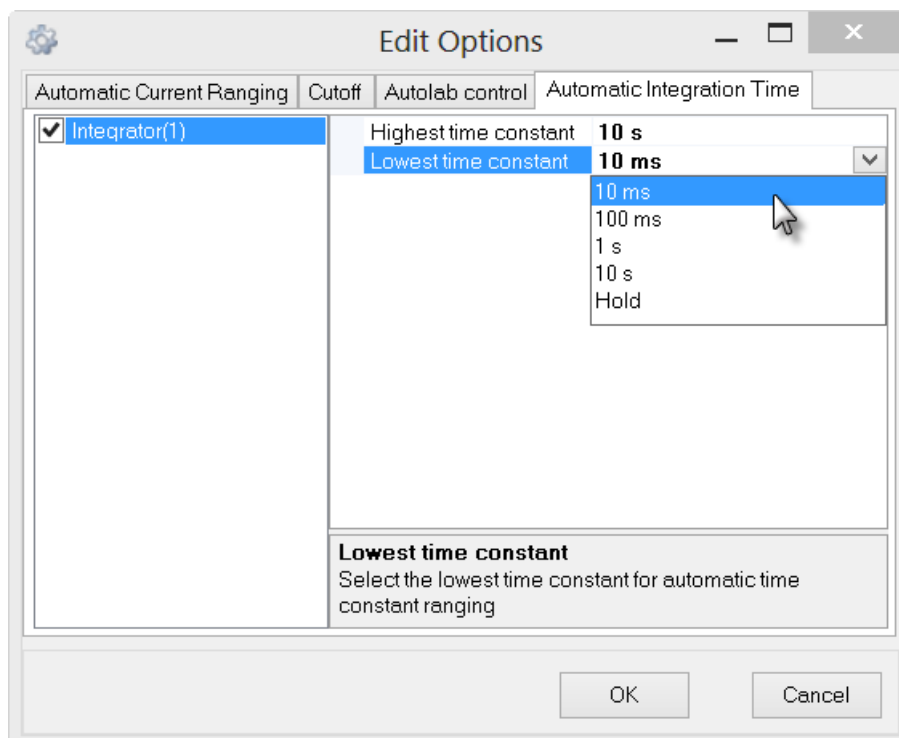

Figure 23 – The integration time can be adjusted automatically using the Automatic Integration Time option

4.5 – Automatic current ranging restrictions

When the integrator module is used during a procedure, the Automatic current ranging option **cannot** be used. The validation process will display an error message whenever this situation is encountered (see Figure 24).
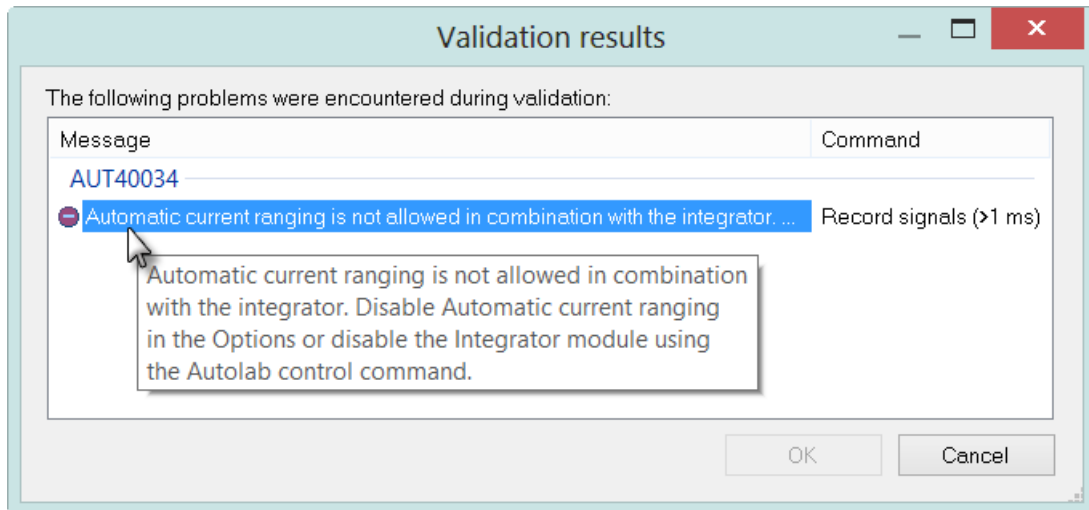

Figure 24 – The integrator cannot be used in combination with automatic current ranging

4.6 – Measurements with the integrator on the dummy cell

A FI20 tutorial folder is located in the **Program Files\Metrohm Autolab\Nova 1.11\Shared Databases\Tutorials** folder (see Figure 25). Using the database manager, set this folder as the Standard database.
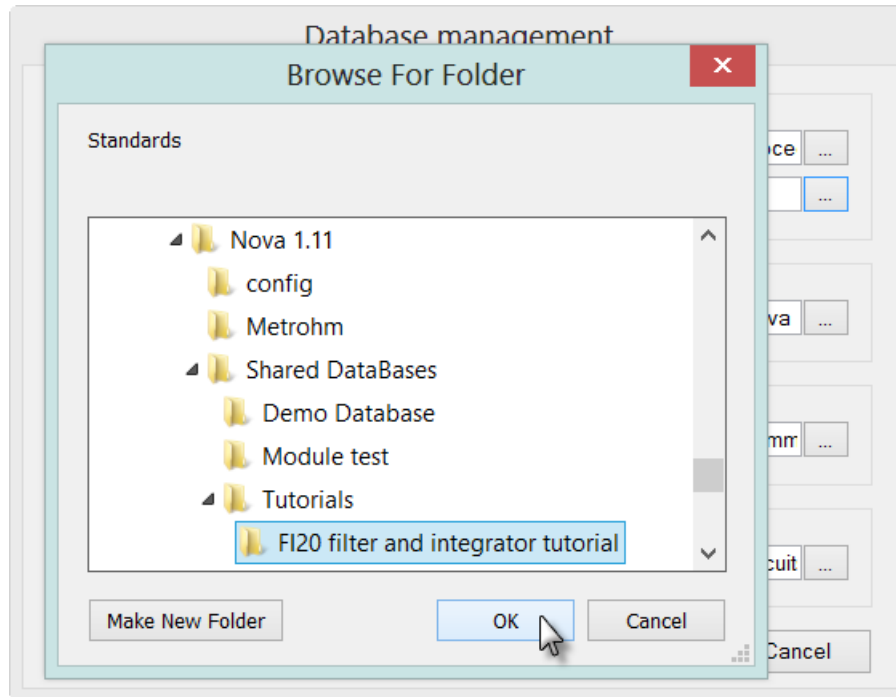
Figure 25 – Loading the FI20 tutorial database

The PGSTAT101 is not equipped with the Autolab dummy cell. An optional external dummy cell can be obtained[9]. It is also possible to activate the internal dummy cell, using the *Autolab control* command[10].

The FI20 tutorial contains four procedures (see Figure 26). The last two procedures illustrate the use of the *Autolab control* command to control the integrator during a measurement. The other two procedures are used to illustrate the use of the filter module (see Section 3.3).
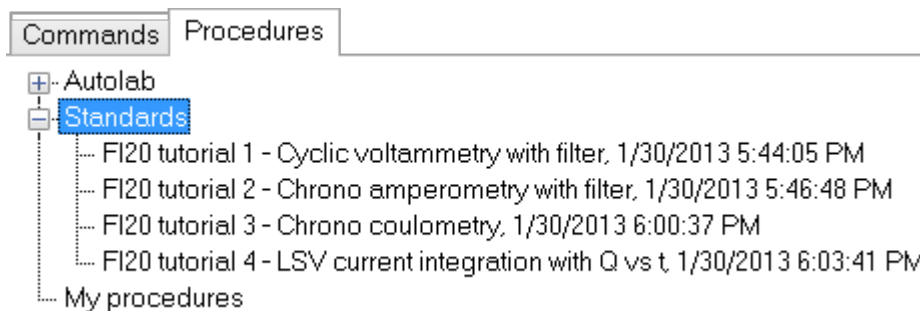

Figure 26 – The four FI20 tutorial procedures

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[9] Contact your Autolab distributor for more information.
[10] Please refer to the Autolab control tutorial, available from the Help menu, for more information.

4.6.1 – FI20 tutorial 3 – Chrono coulometry

Load the integrator FI20 tutorial 3 procedure. Figure 27 shows an overview of the procedure. This procedure is designed to perform a chrono coulometry experiment on the Autolab dummy cell. At the beginning of the procedure, the *Determine integrator drift* command is used to set the drift of the integrator module. The *Autolab control* command is used at the beginning of the procedure to discharge the integrator in order to remove any residual charge. The 1 s integration time is then selected using a second *Autolab control* command. The integrator is used to measure the charge passing through the working electrode (see Figure 27).

Commands	Parameters	Links
FI20 tutorial 3 - Chrono coulometry		
Remarks	Chrono coulometry with the Integrator	...
End status Autolab		...
Signal sampler	Time, WE(1).Potential, WE(1).Current, Integrator(1).Charge	...
Options	0 Options	...
Instrument	AUT40034	
Instrument description		
Message box		
Determine integrator drift		
Select current range		
Autolab control		...
WE(1).Current range	1 µA	
<..>		
<..>		
Autolab control		...
Set potential	0.000	
Set cell	On	...
Wait time (s)	5	
Autolab control		...
Record signals (>1 ms)	[2, 0.01]	–
Set potential	1.000	
Record signals (>1 ms)	[2, 0.01]	–
Set potential	-1.000	
Record signals (>1 ms)	[2, 0.01]	–
Set cell	Off	...
<..>		

Figure 27 – The FI20 tutorial 3 procedure

> **Note**
>
> Because the integrator is used in this experiment, automatic current ranging is not possible.

Connect the cables from the Autolab to the Autolab dummy cell as shown in Figure 28 and press the start button to begin the measurement.
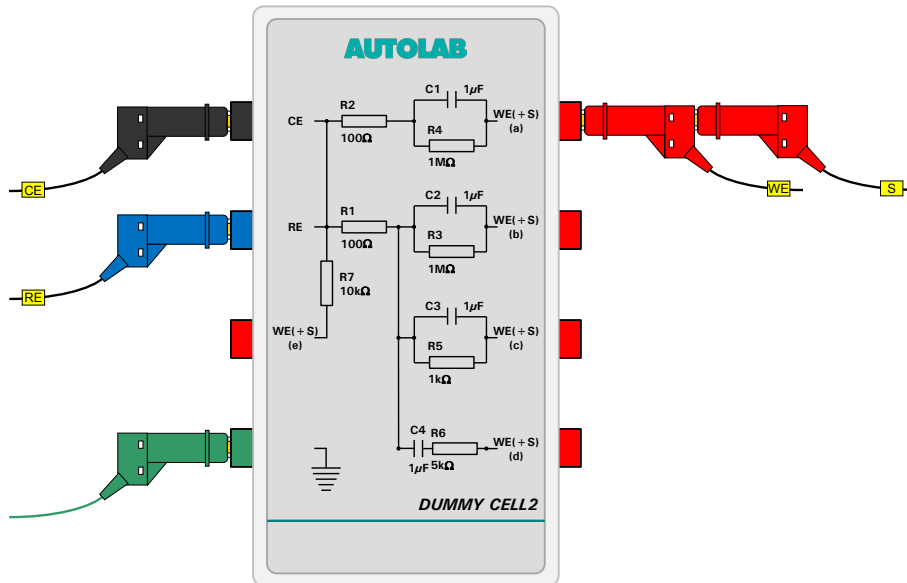
Figure 28 – Overview of the connections to the dummy cell

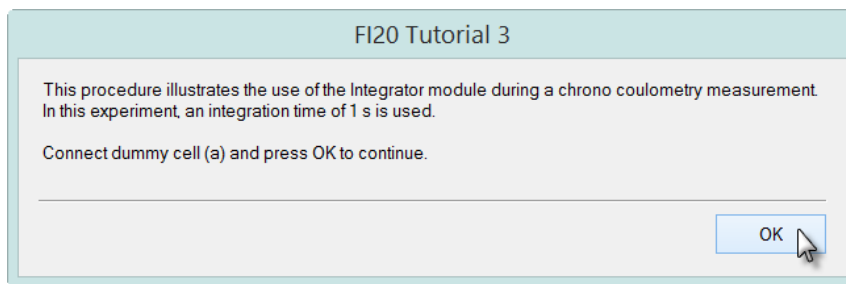A message box will be displayed (see Figure 29).

Figure 29 – A message box is displayed at the beginning of the measurement

Click the OK button to continue. At the beginning of the measurement, the drift is measured and set to 0. The integrator module is then set to a time constant of 1 s. A series of three potential values are applied to the cell and the WE(1).Current is measured for 5 seconds for each step. The measured current and the charge determined by the integrator is plotted versus time (see Figure 30).

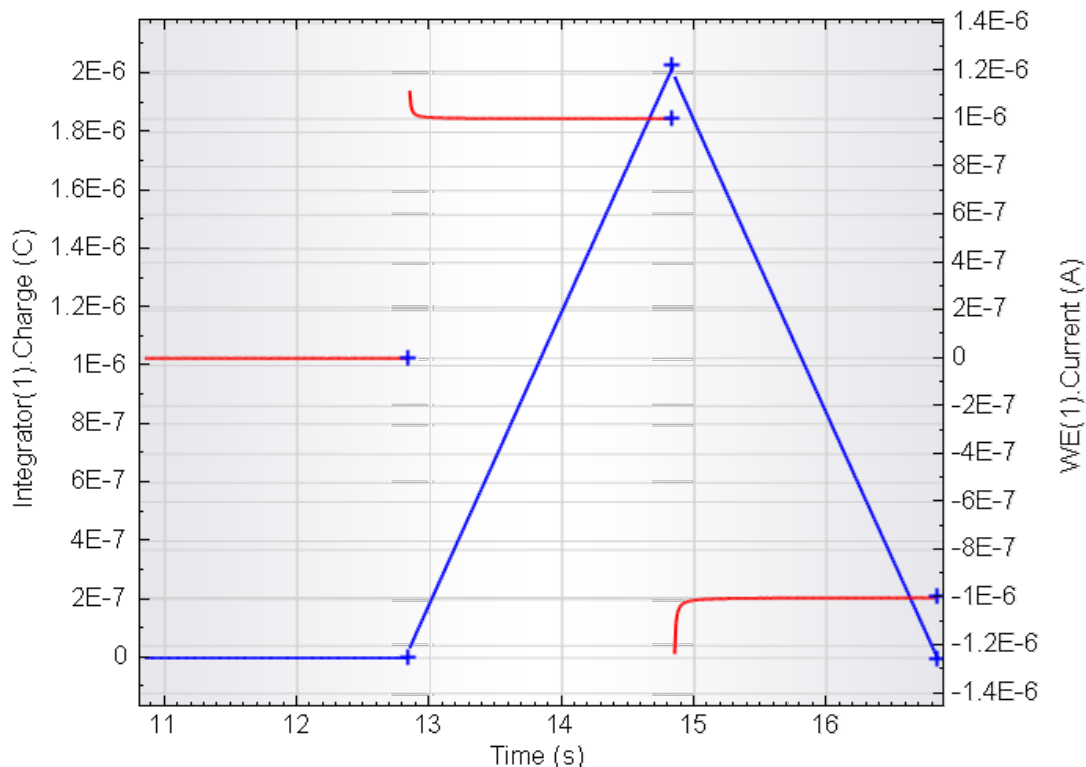**Figure 30 – The data obtained during the FI20 tutorial 3 procedure (left axis, blue curve: Q vs t; right axis, red curve: i vs t)**

The applied potential values are 0 V, 1 V and -1 V for the three consecutive steps. Since dummy cell (a) is used in this test, the measured current values corresponding to the three potential values will be 0 A, 1 µA and -1 µA, respectively. The duration of each potential step is 2 seconds. The maximum charge values recorded during the three steps are therefore 0 mC, 2 µC and -2 µC, respectively.

	Note
i	

The -2 µC variation of the charge measured during the last step bring the total charge back to 0 µC.

4.6.2 – FI20 tutorial 4 – Current integration linear sweep voltammetry

Load the integrator FI20 tutorial 4 procedure. Figure 27 shows an overview of the procedure. This procedure is designed to perform a current integration linear sweep voltammetry experiment on the Autolab dummy cell. At the beginning of the procedure, the *Determine integrator drift* command is used to set the drift of the integrator module. The *Autolab control* command is used at the beginning of the procedure to discharge the integrator in order to remove any residual charge. The 10 ms integration time is then selected using a second *Autolab control* command, just before the beginning of the LSV measurement. The integrator is used to measure the charge passing through the working electrode (see Figure 31).

Figure 31 – The FI20 tutorial 4 procedure

> **Note**
>
> Because the integrator is used in this experiment, automatic current ranging is not possible.

Connect the cables from the Autolab to the Autolab dummy cell as shown in Figure 32 and press the start button to begin the measurement.
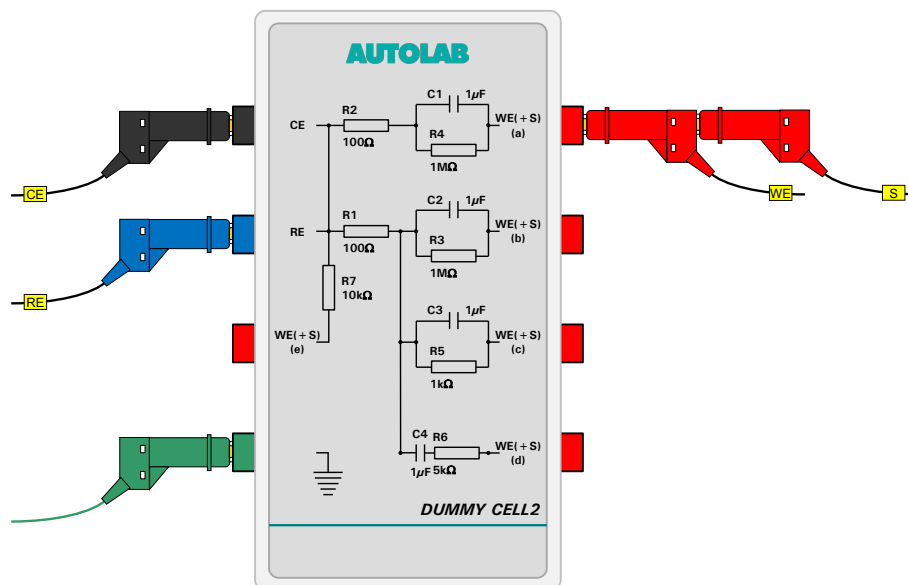

Figure 32 – Overview of the connections to the dummy cell

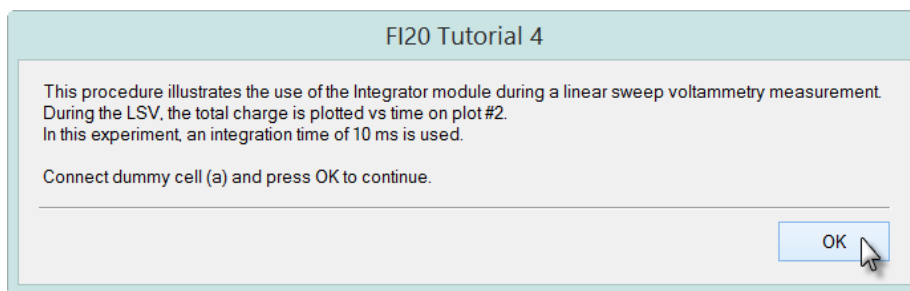A message box will be displayed (see Figure 33).

Figure 33 – A message box is displayed at the beginning of the measurement

Click the OK button to continue. At the beginning of the measurement, the drift is measured and set to 0. The integrator module is then set to a time constant of 10 ms. A linear sweep voltammetry measurement is then started, beginning at 0 V and stopping at 1 V. The integrated current is plotted vs the potential applied on plot #1 and the charge is plotted vs time on plot #2 (see Figure 34).
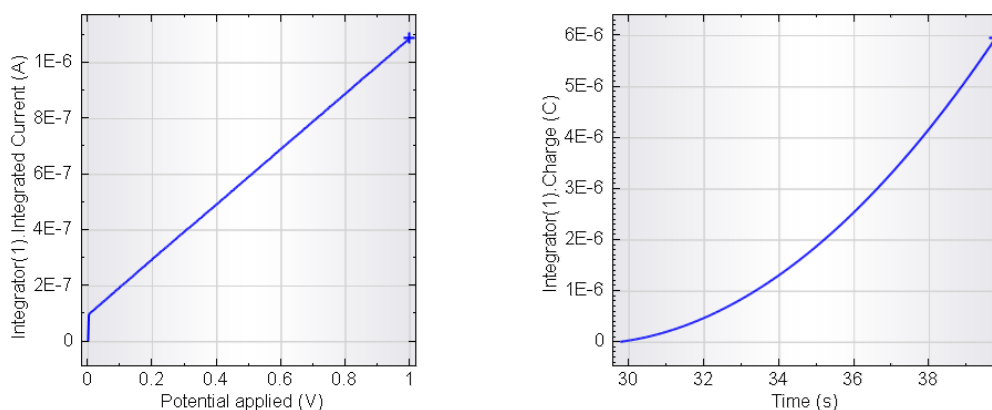

Figure 34 – The data obtained during the FI20 tutorial 4 procedure (left – integrated current vs applied potential, right – charge vs applied potential)

Note

Switch the measurement view to *Two plots vertically tiled* mode by pressing the button in the toolbar.

Hardware specifications

The FI20 module is an optional analog filter and integrator available for all the Autolab PGSTAT instruments with the exception of the PGSTAT101, PGSTAT204 and M101, M204 module and the PGSTAT302F. The filter circuit can be used to remove noise from the measured current when the experimental noise cannot be reduced by using a Faraday cage or by ensure proper shielding of the cell cables. The analog integrator can be used to perform direct coulometric measurements and so-called *Current integration* cyclic voltammetry measurements.

The PGSTAT101, PGSTAT204 and M101, M204 module, as well as the µAutolab type II and type III, are fitted, by default, with an on-board integrator with identical specifications as the FI20 – integrator.

Table 1 and 2 provide an overview of some of the module specifications.

Module	FI20 – Filter
Type of filter	3rd order Sallen-Key
Filter time constants	0 s, 10 ms, 100 ms and 500 ms
Output offset	± 2 mV

Table 1 – Overview of the specifications of the FI20 – Filter module

⚠️ **Important**

The filter can only be applied on the current measured through the main working electrode (WE(1).Current).

Module	FI20 – Integrator
Integration time constants	10 ms, 100 ms, 1 s, 10 s
Charge measurement accuracy	0.2 %

Table 2 – Overview of the specifications of the FI20 – Integrator module

⚠️ **Important**

The integrator can only be applied on the current measured through the main working electrode (WE(1).Current).